

## **EXHIBIT B.3**

### **Zeolite Information and MSDS for chemicals and reagents used in process**

# MATERIAL SAFETY DATA SHEET

An explanation of terms used may be found in OSHA  
29 CFR 1910.1200 available from OSHA regional or area offices.

October 1, 2000

## 1. PRODUCT IDENTIFICATION

PRODUCT: St. Cloud Natural Clinoptilolite Zeolite CAS No. 12173-10-3  
CHEMICAL NAME: Potassium-calcium-magnesium-aluminosilicate  
EMPIRICAL FORMULA: (K,Ca,Mg) 20-AL2O3-10SiO2-6H2O  
SYNONYMS: Clinoptilolite

## 2. COMPANY IDENTIFICATION

COMPANY NAME: ST. CLOUD MINING COMPANY  
MAILING ADDRESS: P.O. Box 1670 - Truth or Consequences, New Mexico 87901, U.S.A.  
PHYSICAL ADDRESS: Winston M3 Site - Winston, New Mexico 87943, U.S.A.  
PHONE NUMBER: 505-743-8218 FAX NUMBER: 505-743-3333 WEBSITE: www.stcloudmining.com

## 3. HAZARDOUS INGREDIENTS AND HEALTH HAZARD DATA

HAZARDOUS INGREDIENTS: May Contain 0.07-0.10% Free Silica ROUTES OF ENTRY: Inhalation  
HEALTH HAZARDS: Granular powder, airborne dust particles are irritating to respiratory tract. Contact with eyes and skin may cause irritation. May be harmful if inhaled. Clinoptilolite with a particle size in the respirable range was tested for carcinogenicity in rats by intratracheal instillation. No significant increase in the incidence of tumors was found. Hazard Symbols for this product: Xn.  
HAZARDOUS COMPONENTS: Clinoptilolite zeolite / potassium, calcium, sodium, aluminosilicate, hydrated (s) Percent by weight: 100%  
CAS #: 12173-10-3 EINECS #: Not found Hazard Symbol: Xn Risk Phrases: R 20, 36/37/38  
CARCINOGENICITY: NTP? No IARC Monographs? No OSHA Regulated? No  
SIGNS & SYMPTOMS OF EXPOSURE: Coughing, Irritation of respiratory tract.

## 4. FIRST AID MEASURES

SWALLOWING: If ingested in large quantities, contact physician for permission to induce vomiting.  
SKIN CONTACT: Rinse with water if irritation ensues. INHALATION: Remove the person to fresh air; rinse mouth and nasal passages with fresh water. Seek medical attention if irritation persists.  
EYE CONTACT: Check for and remove contact lenses. Flush eyes with water for 15 minutes. Seek medical attention if irritation persists.  
NOTES TO PHYSICIAN: St. Cloud Natural Zeolite has no additives or materials that require special treatment. However some products of St. Cloud Natural Zeolite are treated to enhance their use for special applications. Should a specialty product with additives to St. Cloud Natural Zeolite, a separate MSDS is available from the supplier.

## 5. PHYSICAL & CHEMICAL CHARACTERISTICS

COLOR: White, to off white with dark specks BOILING POINT: Not applicable  
ODOR: None FREEZING POINT: Not applicable  
MELTING POINT: 1600 F VAPOR PRESSURE: Not applicable  
VAPOR DENSITY: Not applicable SPECIFIC GRAVITY: 2.2 - 2.4  
SOLUBILITY IN WATER: 0 EVAPORATION RATE: Not applicable

## 6. FIRE AND EXPLOSION DATA

FLASH POINT: Not applicable FLAMMABLE LIMITS: Not applicable  
LEL: Not applicable UEL: Not applicable  
EXTINGUISHING MEDIA: Not applicable  
SPECIAL FIRE FIGHTING PROCEDURES: Not combustible  
UNUSUAL FIRE AND EXPLOSION HAZARDS: None

## 7. REACTIVITY DATA

STABILITY: Stable CONDITIONS TO AVOID: Not applicable  
INCOMPATIBILITY (Materials to avoid): None  
HAZARDOUS POLYMERIZATION: Will not occur CONDITIONS TO AVOID: Not applicable

## 8. SAFE HANDLING AND USE

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED: Physical clean up is required only to prevent sediment to surface water courses, depending upon the particle size of the product. St. Cloud Natural Zeolite will not react or release any harmful substances to the environment.  
WASTE DISPOSAL METHOD: Solids of St. Cloud Natural Zeolite should be cleaned up in a manner that minimizes the generation of dust as a potential nuisance, or may be dispersed of in any garden areas, lawn or lands.  
PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE: Avoid breathing dust, may irritate the respiratory tract.  
OTHER PRECAUTIONS: None

## 9. EXPOSURE CONTROL MEASURES

<b>RESPIRATORY PROTECTION:</b> Wear OSHA approved dust respirators to avoid nuisance dust and possible irritation.	
<b>VENTILATION</b>	<b>LOCAL EXHAUST:</b> Exhaust ventilation is recommended for operations to avoid nuisance dust and possible irritation.
	<b>MECHANICAL (general):</b> Not applicable
	<b>SPECIAL:</b> Not applicable
	<b>OTHER:</b> Not applicable
<b>EYE PROTECTION:</b> Safety glasses or goggles should be worn to prevent dust from entering eyes.	
<b>PROTECTIVE GLOVES:</b> Optional. May cause drying of skin with prolonged contact.	
<b>OTHER PROTECTIVE CLOTHING OR EQUIPMENT:</b> Dust Respirator	

## 10. ECOLOGICAL CONSIDERATIONS

<b>WATERFOWL TOXICITY:</b>	Not Applicable
<b>AQUATIC TOXICITY:</b>	Not Applicable
<b>FOOD CHAIN CONCENTRATION POTENTIAL:</b>	Not Applicable
<b>BIOCHEMICAL OXYGEN DEMAND:</b>	Not Applicable
<b>ATMOSPHERIC:</b>	Contain no ozone depleting substances.

## 11. TRANSPORTATION INFORMATION

Proper Shipping Name, Non-Hazardous for Transport	IATA Hazard Class/Packaging Group: Not Regulated
DOT Hazard Class/Packaging Group: Not Regulated	IMDG Hazard Class: Not Regulated
Reference: Not Applicable	RID/ADR Dangerous Goods Code: Not Regulated
UNNA Identification Number: None	UN TDG Class/Packaging Group: Not Regulated
Label: None Required	Hazard Identification Number (HIN): None
Hazard Symbol: None	

## 12. REGULATORY INFORMATION

CERCLA: None.

SARA Title III Reportable Ingredients: None

California Prop 65: There are no chemicals present in product known to the state of California to cause cancer or reproductive toxicity.

CPR (Canadian Controlled Products Regulations): This product has been classified in accordance with the hazard criteria of the CPR and the MSDS contains all the information required by the CPR. WHMIS Classification: Not Controlled.

IDL (Canadian Ingredient Disclosure List): Components of this product identified by CAS number and listed on the Canadian IDL are shown in Section 3.

DSL / NDSL (Canadian Domestic Substances List / Non-Domestic Substance List): Components of this product identified by CAS number are not listed on the DSL or NDSL, or are otherwise in compliance with the New Substances Notifications (NSN) regulations.

EINECS (European Inventory of Existing Commercial Chemical Substances): Components of this product identified by CAS numbers are on the European Inventory of existing Commercial Chemical Substances unless indicated as "not found".

WQH Water Quality Index: nvg

VCK Index: not applicable

HHS HAZARD RATINGS		HEALTH	1	Definitions:	* = Chronic Health Hazard	2 = MODERATE
	FLAMMABILITY	0			0 = INSIGNIFICANT	3 = HIGH
	PHYSICAL HAZARD	0			1 = SLIGHT	4 = EXTREME
	PERSONAL PROTECTIVE EQUIPMENT	E			E: Safety Glasses, Gloves, Dust Respirator	

It is recommended that all recipients of this MSDS become familiar with and understand any hazards associated with this product. This information should then be made available to employees and customers who may come in contact with this material.

## Appendix B. Zeolite Specifications

### ***St. Cloud Mining Company***

P.O. Box 1670 Truth or Consequences, New Mexico 87901

Phone: 505-743-5215 Fax: 505-743-3333 website: [www.stcloudmining.com](http://www.stcloudmining.com)

Email: [info@stcloudmining.com](mailto:info@stcloudmining.com)

### **St. Cloud Natural Zeolite (Clinoptilolite)**

#### **Product Information Sheet**

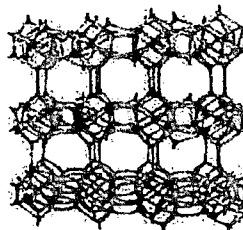
Natural zeolites are a unique type of microporous volcanic mineral with sieving and cation exchange properties for use in agriculture, environmental and industrial applications. St. Cloud Mining Company is the largest producer of natural zeolite in North America.

#### **• Uses of St. Cloud Zeolite**

St. Cloud Zeolite has hundreds of proven uses, and the list continues to grow. St. Cloud Zeolite is a natural mineral produced by all natural means, and is used as an animal feed supplement that has been shown to improve health and reduce the negative effects of animal waste; as an additive to animal stalls and pens to reduce ammonia and other emissions to air and water; as a soil amendment to retain water and plant nutrients in root zones, which conserves both; for a number of other horticultural and agricultural uses, including being the only zeolite endorsed for use in space by NASA; as a water filtration media that removes ammonia, some heavy metals and other constituents; and dozens of other uses.

#### **• Physical Properties**

Bulk Density (In Place, dried)	87 lbs/ft <sup>3</sup> (1,390 kg/m <sup>3</sup> )
Bulk Density (Aggregate, dried. Common sizes)	
4 X 6 Mesh	44-48 lbs/ft <sup>3</sup>
6 X 14 Mesh	45-47 lbs/ft <sup>3</sup>
14 X 40 Mesh	46-49 lbs/ft <sup>3</sup>
-40 Mesh	48-52 lbs/ft <sup>3</sup>
-100 Mesh	49-53 lbs/ft <sup>3</sup>
-325 Mesh	43-47 lbs/ft <sup>3</sup>
Clinoptilolite content	75 to 85%
Cation Exchange Capacity (CEC)	0.8 - 1.2 meq/g
Surface Charge density	10.1E-23 meq/Å <sup>2</sup>
Color	White (85 optical reflectance)
Crushing Strength	2,500 lbs/in <sup>2</sup> (176 kg/m <sup>2</sup> )
Hardness	3.5-4.0 Mohs
LA Wear (Abrasion Index)	24
Molecular Ratio	5.1 (Si/Al)
pH (natural)	7.5 to 8.0
pH Stability	0-13
Permeability	10 <sup>-3</sup> m/sec (1.4 - 0.4 mm particles)
Pore Size (diameter)	4 - 7 angstroms
Pore Volume	≤ 52%
Resistivity	~ 9,000 ohms/cm
Specific Gravity	2.2 - 2.4:1
External Surface Area	14 to 15 m <sup>2</sup> /g
Total Surface Area	≤ 800 m <sup>2</sup> /g
Swelling Index	nil
Thermal Stability	1,202° F (650° C)
Other	non-soluble, non-slaking, free flowing, readily mixable





• **Chemical Analysis**

(Ca, Na, K)(Al<sub>6</sub>SiO)<sub>2</sub> 24H<sub>2</sub>O; Calcium-sodium-potassium aluminosilicate). In approximate weight percent for major oxides:

CaO	3.4%	MgO	1.5%	Na <sub>2</sub> O	0.3%	Fe <sub>2</sub> O <sub>3</sub>	1.6%
K <sub>2</sub> O	3.0 %	P <sub>2</sub> O <sub>5</sub>	0.05%	Al <sub>2</sub> O <sub>3</sub>	12.1%	SiO <sub>2</sub>	70.0%

• **Chemical Composition** for selected elements by x-ray fluorescence (in weight % or ppm, as noted)

Ca	2.4 %	Mg	0.9 %	Na	0.1 %	Fe	0.6%	Cu	10 ppm
K	1.2 %	P	0.01%	Al	3.1 %	Si	32.9%	Zn	59 ppm

• **Cation Exchange Capacity**

Total exchange capacity of 0.8 – 1.2 meq/g total, and an external exchange capacity of about 0.1 meq/g.

• **Major Exchangeable Cations**

Rb, Li, K, Cs, NH<sub>4</sub>, Na, Ca, Ag, Cd, Pb, Zn, Ba, Sr, Cu, Hg, Mg, Fe, Co, Al, Cr. (selectivity of above cations is a function of hydrated molecular size and relative concentrations).

• **Primary Adsorbing Gases:**

CO, CO<sub>2</sub>, SO<sub>2</sub>, H<sub>2</sub>S, NH<sub>3</sub>, HCHO, Ar, O<sub>2</sub>, N<sub>2</sub>, H<sub>2</sub>O, He, H<sub>2</sub>, Kr, Xe, CH<sub>3</sub>OH, Freon, formaldehyde, and mercaptans.

• **Environmental Properties**

Zeolites are natural, inert, non-toxic substances which are federally classified as GRAS (Generally Regarded As Safe) in most applications and exempted from most regulations and reporting when used in accordance with good agricultural practice and when less than 2% in animal feed products (40 CFR, Part 180.1001 and elsewhere). St. Cloud Zeolite is USDA approved as an absorbent for spills in food processing facilities and complies with federal and state environmental requirements. No fibrous silicates have been detected and crystalline silica is present in quantities of less than 0.01%. Reference MSDS Sheet (product CAS No. 1318-02-1) for additional information.

• **Location** - St. Cloud's Zeolite operation is centrally located near Winston, New Mexico, approximately 35 miles west of Interstate 25 midway between Albuquerque, New Mexico and El Paso, Texas.

• **Reserves** - An 18.3 million ton zeolite reserve base mined from surface exposures 1.5 miles from plant site.

• **Delivery and Freight** - Motor carriers for packaged and bulk products for domestic and international destinations. Bulk rail facilities are available by special arrangement. UPS, LTL, and regional warehousing services also available. Warehouse customer pick-ups 8:00 am-3:00 PM, 5 days per week, or 24 hrs per day or by prior arrangement.

• **Trade Names**

Sold by St. Cloud as Stone House® Cat Litter, Floor Drying Material and Natural Zeolite Crystal, and Heaven Scent®. Marketed or included in products by brokers, manufacturers and distributors as Ammo® Chips, Litter and Rocks, Ammonia-X®, A-Stone®, Biolite®, Clinolite®, EcoFresh®, Ecolite®, E-Z Dry®, Hydrosil®, PDZ® Sweet Stall®, Stall Fresh®, Zeofresh®, ZeoPro and others.

• **Packaging**

Standard, private label and custom packaging and palletizing available including 10, 25, 50 lb. or other units in paper or plastic with stitch, taped, valve packed or heat sealed closures. Other off-site special packaging services available on request. Bulk packaging in approximately 1-ton and other supersacks or directly loaded in any bulk truck or rail car configuration including top loading or pneumatic carriers.

*Information herein is accurate to the best of our knowledge, but may be subject to change without notice. Suggestions are made without warranty or guarantee of results. Before using, user should determine the suitability of the product for its intended use and user assumes the risk and liability in connection herewith.*

st. cloud<sup>TM</sup>  
zeolite

### St. Cloud Zeolite Screen Size Analysis

Nominal Particle Size Mesh	Approx. Particle Size Inches	Microns (um) Approx.	Millimeters (MM) Approx.	Lab Opening US Std. (Inches)	Plant Opening Mill Gr. (Inches)	Percentage (%) Retained on Laboratory Screens													
						Powders			Sands		Granules				Aggregates				
						-325 Mesh	-100 Mesh	-40 Mesh	40x60 Mesh	14x40 Mesh	8x14 mesh	6x14 mesh	6x8 mesh	4x8 Mesh	4 mesh x 1/2 in	1/2 in x 3/4 in	3/4 in x 1 in		
1" Inch	1	25,400	25.40	1.000	1.000	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0	
3/4 Inch	3/4	19,050	19.00	0.750	0.750	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	49.9	
1/2 Inch	1/2	12,700	13.00	0.500	0.500	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	64.47	44.0	
4 Mesh	7/32	4,760	4.70	0.157	0.215	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.04	50.59	35.13	3.4		
6 Mesh	8/34	3,360	3.40	0.132	0.139	0.00	0.00	0.00	0.00	0.00	0.00	0.21	1.08	75.67	46.93	0.08	0.5		
8 Mesh	7/84	2,380	2.40	0.094	0.100	0.00	0.00	0.00	0.00	0.00	1.23	30.03	70.58	23.32	n/a	n/a	n/a		
14 Mesh	3/84	1,410	1.40	0.055	0.054	0.00	0.00	0.00	0.00	0.12	87.05	64.70	27.58	0.58	4.72	0.05	0.8		
20 Mesh	1/32	841	0.84	0.033	0.038	0.00	0.00	0.00	0.55	41.88	10.67	n/a	n/a	n/a	n/a	n/a	n/a		
40 Mesh	1/64	420	0.42	0.017	0.017	0.00	2.00	0.70	5.01	63.68	0.47	4.31	n/a	n/a	0.11	0.03	0.4		
70 Mesh	n/a	210	0.21	0.008	n/a	0.00	8.00	30.40	91.71	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a		
100 Mesh	n/a	149	0.15	0.008	n/a	0.00	8.20	14.80	2.04	4.82	0.10	0.09	0.21	0.07	0.28	0.04	0.4		
<100 Mesh	n/a	<149	<.15	<.008	n/a	0.00	83.70	54.20	0.68	0.61	0.49	0.68	0.55	0.34	1.88	0.22	0.7		
140 Mesh	n/a	105	0.10	0.004	n/a	0.00	7.70	14.50	0.00	0.00	0.00	0.00	0.00	0.00	0.00	n/a	0.0		
200 Mesh	n/a	74	0.07	0.003	n/a	0.00	6.50	7.70	0.00	0.00	0.00	0.00	0.00	0.00	0.00	n/a	0.0		
325 Mesh	n/a	44	0.04	0.002	n/a	1.50	10.90	8.40	0.00	0.00	0.00	0.00	0.00	0.00	0.00	n/a	0.0		
<325 Mesh	n/a	<44	<0.04	<.0017	n/a	98.50	58.50	23.80	0.00	0.00	0.00	0.00	0.00	0.00	0.00	n/a	0.0		
Typical Density (lbs/m3)						45.0	51.5	50.3	50.1	49.0	46.0	48.4	48.0	44.7	48.0	50.5	48.5		
Typical Moisture %						8.5	7.0	8.8	5.8	3.8	2.9	4.8	3.5	3.5	6.3	7.6	7.2		

Notes: All laboratory analysis performed similar to ASTM D392, Method A. Laboratory Sieves are U.S. Standard mesh and plant screens are standard "mill grade".

St. Cloud Zeolite - [www.stcloudmining.com](http://www.stcloudmining.com)  
P.O. Box 1670 - Truth or Consequences, New Mexico 87901 - Ph: 505.743.5215



## Material Safety Data Sheet

Date Prepared: 05/22/2014

Supersedes: 09/03/2013

## Section 1 – Product &amp; Company Identification

**Product Name:** Zeolite (Chabazite, Clinoptilolite)  
**Part Numbers:** Winston Clino (NM-Ca), Ash Meadows Clino (NV-Na), Rome Mordenite  
 Bowtie Chab: (AZUB), (AZLB-Na) & (AZLB-Ca)  
**General Uses:** Water Filtration, Sorbent, Odor Control/Desiccant, Animal Feed Additive  
**Product Description:** White, Off-White, or Tan Granules & Powders w/Dark Specs  
**Manufacturer's Name:** St. Cloud Mining Company OR SCM - Ash Meadows LLC  
**Street Address:** 761 St. Cloud Rd. E. Spring Meadows Rd.  
**P.O. Box:** P.O. Box 196 HCR 70-Box 7006  
**City, State, Zip:** Winston, NM 87843 Amargosa Valley, NV 89020  
**Phone:** 575-743-5215 Phone/Fax: 775-372-5524  
**Fax:** 575-743-3393 Country: U.S.A.

## Section 2 – Hazardous Ingredients

**Hazardous Ingredients:** May contain greater than 0.10% free silica  
**Hazardous Components:** Clinoptilolite Zeolite/potassium, calcium, sodium, aluminosilicate, hydrated (a)  
**Percent by Weight:** 100%  
**CAS #:** 12173-10-3 (Clinoptilolite), 1318-02-1 (Chabazite)  
**EINECS #:** Not found  
**Hazard Symbol:** Xn  
**Risk Phrases:** R20, 36/37/38  
 (a) Clinoptilolite is not listed on the Canadian DSL or NDSL.

## Section 3 – Hazards Identification

**Carcinogenicity:** NTP? No IARC Monographs? No OSHA Regulated? No  
 Clinoptilolite with a particle size in the respirable range was tested for carcinogenicity in rats by intratracheal instillation. No significant increase in the incidence of tumors was found.  
**Eye Contact:** May cause irritation.  
**Skin Contact:** May cause irritation.  
**Inhalation:** Causes irritation.  
**Ingestion:** No hazards known or suspected.  
**Chronic Hazards:** No known chronic hazards. Not listed by OSHA, NTP or IARC as a carcinogen. Does not contain asbestos or fibrous zeolite minerals. Crystalline silica was not detected by X-Ray Diffraction Analysis, but may be present above the notification level of 0.10%. (Crystalline silica is considered a hazard by inhalation. During October 1996, IARC reviewed the literature for polymorphs of crystalline silica and determined that there is sufficient evidence in humans for the carcinogenicity of inhaled crystalline silica in the form of quartz or cristobalite from occupational sources.)

## Section 4 – First Aid Measures

**Ingestion:** If ingested in large quantities, contact physician for permission to induce vomiting.  
**Inhalation:** Move subject to fresh air. Rinse mouth & nasal passages with fresh water. If irritation persists, seek medical attention.  
**Skin Contact:** Rinse with water. If irritation persists, seek medical attention.  
**Eye Contact:** Remove contact lenses. Flush eyes with water for 15 minutes. If irritation persists, seek medical attention.  
**Notes to Physician:** St. Cloud Natural Zeolite has no additives or materials that require special treatment. Some St. Cloud products, however, are treated to enhance their use for special applications. A separate MSDS is available from the supplier for additives to the St. Cloud Natural Zeolite.

## Section 5 – Fire Fighting Measures

**General Hazards:** Product is not considered flammable or combustible.  
**Flash Point (°F):** N/A **Flammable Limits (Vapor in Air, Vol. %):** N/A  
**Fire Extinguishing Media:** N/A **Unusual Fire & Explosion Hazards:** None

## Section 6 – Accidental Release Measures

**Environmental Hazards:** No known adverse effects.  
**Spillage:** Sweep, scoop, or vacuum discharged material.  
**Waste Disposal Method:** Landfill according to local, State & Federal regulations.

## Section 7 – Handling &amp; Storage

**Precautions:** Keep container closed when not in use. Protect containers from abuse; store in a cool, dry area. Keep out of reach of children.

## Section 8 – Exposure Controls / Personal Protection

**OSHA Permissible Exposure Limit (or ACGIH):** Values have not been established.  
**Recommended Threshold Limit:** Ceiling Limit = 15 mg/m<sup>3</sup> Total Dust  
 5 mg/m<sup>3</sup> Respirable Fraction  
**Exposure Analysis Methods:** Respirable Sampler or Midget Impinger see: J. Am. Ind. Hyg. Assoc. 28:554 (1967)  
**Respiratory Protection:** Use NIOSH approved dust mask or respirator where dust occurs. Refer to 29 CFR 1910.134 or European Standard EN 149 for regulations.  
**Gloves:** Plastic, rubber or cotton.  
**Eye Protection:** Safety glasses or chemical goggles. Refer to 29 CFR 1910.133 or European Standard EN166.  
**Other Protective Equipment:** N/A  
**Personal Hygiene:** Avoid breathing dust.  
**Engineering Control:** Use with adequate ventilation.

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Product Name: Zeolite (Chabazite, Clinoptilolite, Mordenite)

**Material Safety Data Sheet**

Date Prepared: 05/22/2014

Supersedes: 09/23/2013

**Section 9 – Physical & Chemical Properties**

<b>Appearance &amp; Odor:</b>	Chabazite = Dry, tan granules & powders. Odorless. Clinoptilolite = Dry, white to off-white granules & powders with specs.
<b>Specific Gravity:</b>	Chabazite = N/A (Liquids Only), Clinoptilolite = 2.2 – 2.4
<b>Solubility in Water:</b>	Chabazite = Negligible, Clinoptilolite = 0
<b>Vapor Density:</b>	N/A
<b>Vapor Pressure:</b>	Chabazite = N/A (mm Hg at °F, Nonaqueous Liquids Only) Clinoptilolite = N/A
<b>Evaporation Rate:</b>	Chabazite = N/A (Butyl Acetate = 100, Nonaqueous Liquids Only) Clinoptilolite = N/A
<b>Solids Content:</b>	Chabazite = N/A (Solutions, Dispersions or Pastes Only) Clinoptilolite = N/A
<b>Melting Point:</b>	Chabazite = N/A, Clinoptilolite = 1800°F
<b>Boiling Point:</b>	Chabazite = N/A (°F, Nonaqueous Liquids Only), Clinoptilolite = N/A
<b>pH:</b>	Chabazite = N/A (Aqueous Liquids Only), Clinoptilolite = N/A

**Section 10 – Stability and Reactivity**

<b>Stability:</b>	Stable	<b>Conditions to Avoid:</b>	N/A
<b>Incompatibility (Materials to Avoid):</b>	N/A	<b>Hazardous Decomposition Products:</b>	None
<b>NFPA Hazard Ratings:</b>	Health Hazard: 1 Reactivity: 0	<b>Flammability Hazard:</b> 0 <b>Special Hazard:</b> None	

**Section 11 – Toxicological Information**

<b>Chabazite:</b>	CAS #1318-02-1	<b>EINECS # (not found)</b>	<b>LC/LD50 - N/A</b>
<b>Clinoptilolite:</b>	CAS #12173-10-3	<b>EINECS # (not found)</b>	<b>LC/LD50 - N/A</b>

**Section 12 – Ecological Information**

No data available. None of the ingredients are classified as a Marine Pollutant.

**Section 13 – Disposal Considerations**

Dispose of in accordance with Local, State and Federal Regulations. Product is classified as non-hazardous, however, non-hazardous materials may become hazardous waste upon contact with other products. Refer to "40 CFR Protection of Environment Parts 260-299" for complete waste disposal regulations. Consult your local, state or Federal Environmental Protection Agency before disposing of any chemicals. According to the European Waste Catalogue, waste codes are application-specific and should be assigned by the user based on the application for which the product is used.

**Section 14 – Transport Information**

<b>DOT Shipping Name:</b>	Non-Hazardous for Transport	<b>IMDG Hazard Class:</b>	Not Regulated
<b>DOT Hazard Class:</b>	Not Regulated	<b>IATA Hazard Class:</b>	Not Regulated
<b>UN/NA ID No.:</b>	None	<b>RID/ADR Code:</b>	Not Regulated
<b>UN TDG Class:</b>	Not Regulated	<b>Label:</b>	None Required
<b>Hazard ID No. (HIN):</b>	None	<b>Hazard Symbols:</b>	None

**Section 15 – Regulatory Information****TSCA (U.S.A. – Toxic Substances Control Act)**

All components of this product are listed on the U.S. Toxic Substances Control Act Chemical Inventory or are exempted from listing because a Low Volume or Polymer Exemption has been granted in accordance with 40 CFR 723.50.

**SARA TITLE III (U.S.A. – Superfund Amendments & Reauthorization Act)**

311/312 Hazard Categories-Immediate Health, 313 Reportable Ingredients-None

**CERCLA (U.S.A. – Comprehensive Response Compensation & Liability Act)**

None

**California Prop 65, Safe Drinking Water & Toxic Enforcement Act of 1986**

There are no chemicals present known to the State of California to cause cancer or reproductive toxicity.

**CPR (Canadian Controlled Products Regulations)**

This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations and the MSDS contains all the information required by the Controlled Products Regulations. WHMIS Classification: Not controlled

**IDL (Canadian Ingredient Disclosure List)**

Components of this product identified by CAS number and listed on the Canadian Ingredient Disclosure List are shown in Section 2.

**DSL/NDSL (Canadian Domestic Substances List / Non-Domestic Substances List)**

Components of this product identified by CAS number are not listed on the DSL or NDSL, or are otherwise in compliance with the New Substances Notification (NSN) regulations. Only ingredients classified as "hazardous" are listed in Section 2 unless otherwise indicated.

**EINECS (European Inventory of Existing Commercial Chemical Substances)**

Components of this product identified by CAS numbers are on the European Inventory of Existing Commercial Chemical Substances unless indicated as "not found".

**WGK Water Quality Index: nwg**

Vbk Index: not applicable

**Section 16 – Other Information****Notes & Full R-Phrase Text:**

R20 Harmful by inhalation  
R36/37/38 Irritating to eyes, respiratory system and skin

**HMIS Hazard Ratings:**

Health = 1  
Physical Hazard = 0  
Flammability = 0  
Personal Safety Equipment = E

**Definitions:** \* = Chronic Health Hazard, 0 = Insignificant, 1 = Slight, 2 = Moderate, 3 = High,  
4 = Extreme, E = Safety Glasses, Gloves, Dust Respirator

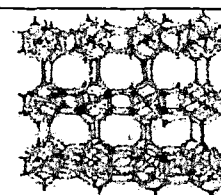
**Revision Summary:** This MSDS has been revised as follows - Updated Format & Data

**Additional Contact Information**

**Contact:** Daniel T. Eyde, Chief Technology Officer  
**Email:** [dteyde@stdcloudmining.com](mailto:dteyde@stdcloudmining.com)  
**Phone:** (520) 744-8845

**Contact:** Joseph P. McEnaney, Vice President-Sales  
**Email:** [jmcenaney@stdcloudmining.com](mailto:jmcenaney@stdcloudmining.com)  
**Phone:** (203) 761-1291

**URL:** <http://www.stdcloudmining.com>





## Univar USA Inc Safety Data Sheet

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HOMESTAKE MINING CO.

ALBQ. WEST ON I-40

SEE INSTRUCTIONS ON

GRANTS

NM 87020

SDS No:

CDS2441

Version No:

001 01/15/15

Order No:

AQ322380

3075 Highland Pkwy, Ste 200, Downers Grove, IL 60515  
(425) 889 3400

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### Emergency Assistance

For emergency assistance involving chemicals call  
Chemtrec - (800) 424-9300

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COMPANY IDENTITY: Univar  
PRODUCT IDENTITY: SULFURIC ACID 93%  
SDS NUMBER: CDS-2441

SDS DATE: 01/15/2015  
ORIGINAL: 01/15/2015

**SAFETY DATA SHEET**

This Safety Data Sheet conforms to ANSI Z400.5, and to the format requirements of the Global Harmonizing System.  
THIS SDS COMPLIES WITH 29 CFR 1910.1200 (HAZARD COMMUNICATION STANDARD)  
IMPORTANT: Read this SDS before handling & disposing of this product.  
Pass this information on to employees, customers, & users of this product.

**SECTION 1. IDENTIFICATION OF THE SUBSTANCE OR MIXTURE AND OF THE SUPPLIER**

PRODUCT IDENTITY: SULFURIC ACID 93%  
PRODUCT USES: Mineral Acid

COMPANY IDENTITY: Univar  
COMPANY ADDRESS: 17425 NE Union Hill Road  
COMPANY CITY: Redmond, WA 98052  
COMPANY PHONE: 1-425-889-3400  
EMERGENCY PHONES: CHEMTREC: 1-800-424-9300 (USA)  
CANUTEC: 1-613-996-6666 (CANADA)

**SECTION 2. HAZARDS IDENTIFICATION****DANGER!!****2.1 HAZARD STATEMENTS: (CAT = Hazard Category)**

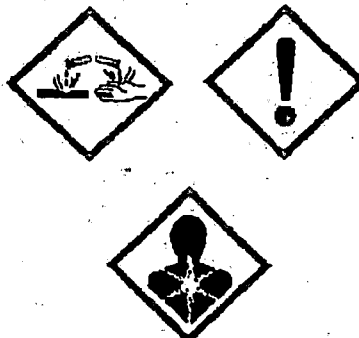
(H200s) PHYSICAL: Corrosive To Metals:  
H290 MAY BE CORROSIVE TO METALS.(CAT:1)  
(H300s) HEALTH: Skin Corrosion/Irritation:  
H314 CAUSES SEVERE SKIN BURNS AND EYE DAMAGE.(CAT:1)  
(H300s) HEALTH: Acute Toxicity, Inhalation:  
H332 HARMFUL IF INHALED.(CAT:4)

**2.2 PRECAUTIONARY STATEMENTS:**

EXPOSURE PREVENTION: AVOID ALL CONTACT!  
PREVENT DISPERSION OF MISTS OR DUST!

P100s = General, P200s = Prevention,  
P300s = Response, P400s = Storage, P500s = Disposal  
P234 Keep only in original container.  
P260 Do not breathe dust/fume/gas/mist/vapors/spray.  
P262 Do not get in eyes, on skin, or on clothing.  
P264 Wash hands thoroughly after handling.  
P280 Wear protective gloves/protective clothing/eye protection/face protection.  
P301+330+331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.  
P303+361+353 IF ON SKIN (OR HAIR): Remove/Take off immediately all contaminated clothing. Rinse skin with water/shower.  
P304+340 IF INHALED: Remove victim to fresh air & keep at rest in a position comfortable for breathing.  
P305+351+338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses if present & easy to do - Continue rinsing.  
P310 Immediately call a POISON CENTER or doctor/physician.  
P363 Wash contaminated clothing before reuse.  
P390 Absorb spillage to prevent material damage.  
P404 Store in a closed container.  
P405 Store locked up.  
P501 Dispose of contents/container to an approved waste disposal plant.

SEE SECTIONS 8, 11 & 12 FOR TOXICOLOGICAL INFORMATION.



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## SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

MATERIAL	CAS#	EINECS#	WT %
Sulfuric Acid	7664-93-9	231-639-5	93
Water	7732-18-5	231-791-2	7

TRACE COMPONENTS: Trace ingredients (if any) are present in < 1% concentration, (< 0.1% for potential carcinogens, reproductive toxins, respiratory tract mutagens, and sensitizers). None of the trace ingredients contribute significant additional hazards at the concentrations that may be present in this product. All pertinent hazard information has been provided in this document, per the requirements of the Federal Occupational Safety and Health Administration Standard (29 CFR 1910.1200), U.S. State equivalents, and Canadian Hazardous Materials Identification System Standard (CPR 4).

## SECTION 4. FIRST AID MEASURES

## 4.1 MOST IMPORTANT SYMPTOMS/EFFECTS, ACUTE &amp; CHRONIC:

See Section 11 for symptoms/effects, acute & chronic.

## 4.2 GENERAL ADVICE:

First Aid responders should pay attention to self-protection and use the recommended protective clothing (chemical resistant gloves, splash protection). If potential for exposure exists, refer to Section 8 for specific personal protective equipment.

## 4.3 EYE CONTACT:

If this product enters the eyes, check for and remove any contact lenses. Open eyes while under gently running water. Use sufficient force to open eyelids. "Roll" eyes to expose more surface. Minimum flushing is for 15 minutes. Seek immediate medical attention.

## 4.4 SKIN CONTACT:

If the product contaminates the skin, immediately begin decontamination with running water. Minimum flushing is for 15 minutes. Remove contaminated clothing, taking care not to contaminate eyes. If skin becomes irritated and irritation persists, medical attention may be necessary. Wash contaminated clothing before reuse, discard contaminated shoes.

## 4.5 INHALATION:

After high vapor exposure, remove to fresh air. If breathing is difficult, give oxygen. If breathing has stopped, trained personnel should immediately begin artificial respiration. If the heart has stopped, trained personnel should immediately begin cardiopulmonary resuscitation (CPR). Seek immediate medical attention.

## 4.6 SWALLOWING:

If swallowed, CALL PHYSICIAN OR POISON CONTROL CENTER FOR MOST CURRENT INFORMATION. If professional advice is not available, give two glasses of water to drink. DO NOT INDUCE VOMITING. Never induce vomiting or give liquids to someone who is unconscious, having convulsions, or unable to swallow. Seek immediate medical attention.

## 4.7 RESCUERS: Victims of chemical exposure must be taken for medical attention. Rescuers should be taken for medical attention, if necessary. Take a copy of label and SDS to physician or health professional with victim.

## 4.8 NOTES TO PHYSICIAN:

There is no specific antidote. Treatment of overexposure should be directed at the control of symptoms and the clinical condition of the patient. Any material aspirated during vomiting may cause lung injury. Therefore, emesis should not be induced mechanically or pharmacologically. If it is considered necessary to evacuate the stomach contents, this should be done by means least likely to cause aspiration (such as: Gastric lavage after endotracheal intubation).

## SECTION 5. FIRE FIGHTING MEASURES

## 5.1 FIRE &amp; EXPLOSION PREVENTIVE MEASURES:

Isolate from alkalis, oxidizers, organics, extreme heat and open flames.

## 5.2 SUITABLE (&amp; UNSUITABLE) EXTINGUISHING MEDIA:

Use extinguishing agent appropriate for surrounding fire.

## 5.3 SPECIAL PROTECTIVE EQUIPMENT &amp; PRECAUTIONS FOR FIRE FIGHTERS:

Water spray may be ineffective on fire but can protect fire-fighters & cool closed containers. Use fog nozzles if water is used. Do not enter confined fire-space without full bunker gear. (Helmet with face shield, bunker coats, gloves & rubber boots).

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**SECTION 5. FIRE FIGHTING MEASURES (CONTINUED)****5.4 SPECIFIC HAZARDS OF CHEMICAL & HAZARDOUS COMBUSTION PRODUCTS:****SLIGHTLY COMBUSTIBLE!**

Reacts with most metals producing hydrogen which is extremely flammable & may explode. Keep container tightly closed. Isolate from oxidizers, alkalis, heat, & open flame. Applying to hot surfaces requires special precautions. Closed containers may explode if exposed to extreme heat. Continue all label precautions!

**SECTION 6. ACCIDENTAL RELEASE MEASURES****6.1 SPILL AND LEAK RESPONSE AND ENVIRONMENTAL PRECAUTIONS:**

Uncontrolled releases should be responded to by trained personnel using pre-planned procedures. Proper protective equipment should be used. In case of a spill, clear the affected area, protect people, and respond with trained personnel. ELIMINATE all ignition sources (no smoking, flares, sparks, or flames in immediate area). Prevent additional discharge of material, if possible to do so without hazard. For large spills, implement cleanup procedures and, if in public area, advise authorities.

**6.2 PERSONAL PRECAUTIONS, PROTECTIVE EQUIPMENT, EMERGENCY PROCEDURES:**

The proper personal protective equipment for incidental releases (such as: 1 liter of the product released in a well-ventilated area), use impermeable gloves, they should be Level B: triple-gloves (rubber gloves and nitrile gloves over latex gloves), chemical resistant suit and boots, hard-hat, and Self-Contained Breathing Apparatus specific for the material handled, goggles, face shield, and appropriate body protection. In the event of a large release, use impermeable gloves, specific for the material handled, chemically resistant suit and boots, and hard hat. Self-Contained Breathing Apparatus or respirator may be required where engineering controls are not adequate or conditions for potential exposure exist. When respirators are required, select NIOSH/MSHA approved based on actual or potential airborne concentrations in accordance with latest OSHA and/or ANSI recommendations.

**6.3 ENVIRONMENTAL PRECAUTIONS:**

Stop spill at source. Construct temporary dikes of dirt, sand, or any appropriate readily available material to prevent spreading of the material. Close or cap valves and/or block or plug hole in leaking container and transfer to another container. Keep from entering storm sewers and ditches which lead to waterways, and if necessary, call the local fire or police department for immediate emergency assistance.

**6.4 METHODS AND MATERIAL FOR CONTAINMENT & CLEAN-UP:**

Absorb spilled liquid with polypads or other suitable absorbent materials. If necessary, neutralize using suitable buffering material, (acid with soda ash or base with phosphoric acid), and test area with litmus paper to confirm neutralization. Clean up with non-combustible absorbent (such as: sand, soil, and so on). Shovel up and place all spill residue in suitable containers. Dispose of at an appropriate waste disposal facility according to current applicable laws and regulations and product characteristics at time of disposal (see Section 13 - Disposal Considerations).

**SECTION 7. HANDLING AND STORAGE****7.1 PRECAUTIONS FOR SAFE HANDLING:**

Isolate from oxidizers, alkalis, heat, & open flame. Use only with adequate ventilation. Do not get in eyes, on skin or clothing. Wear OSHA Standard full face shield. Consult Safety Equipment Supplier. Wear goggles, face shield, gloves, apron & footwear impervious to material. Wash clothing before reuse. Continue all label precautions! NEVER pour water into this substance. When dissolving or diluting, always add it slowly to the water.

**7.2 CONDITIONS FOR SAFE STORAGE, INCLUDING ANY INCOMPATIBILITIES:**

Keep separated from strong oxidants, strong bases, combustible & reducing substances, metals, food & feedstuffs, incompatible materials. May be stored in stainless steel containers. See: Section 10, <Materials to Avoid>. Do not store above 49 C/120 F. Keep container tightly closed & upright when not in use to prevent leakage. Reacts with most metals producing hydrogen which is extremely flammable & may explode. Wear full face shield, gloves & full protective clothing when opening or handling. When empty, drain completely, replace bungs securely.

**7.3 NONBULK: CONTAINERS:**

Store containers in a cool, dry location, away from direct sunlight, sources of intense heat, or where freezing is possible. Material should be stored in secondary containers or in a diked area, as appropriate. Store containers away from incompatible chemicals (see Section 10, Stability and Reactivity). Post warning and "NO SMOKING" signs in storage and use areas, as appropriate. Empty containers should be handled with care. Never store food, feed, or drinking water in containers which held this product.



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**SECTION 7. HANDLING AND STORAGE (CONTINUED)****7.4 BULK CONTAINERS:**

All tanks and pipelines which contain this material must be labeled. Perform routine maintenance on tanks or pipelines which contain this product. Report all leaks immediately to the proper personnel.

**7.5 TANK CAR SHIPMENTS:**

Tank cars carrying this product should be loaded and unloaded in strict accordance with tank-car manufacturer's recommendation and all established on-site safety procedures. Appropriate personal protective equipment must be used (see Section 8, Engineering Controls and Personal Protective Equipment.). All loading and unloading equipment must be inspected, prior to each use. Loading and unloading operations must be attended, at all times. Tank cars must be level, brakes must be set or wheels must be locked or blocked prior to loading or unloading. Tank car (for loading) or storage tanks (for unloading) must be verified to be correct for receiving this product and be properly prepared, prior to starting the transfer operations. Hoses must be verified to be in the correct positions, before starting transfer operations. A sample (if required) must be taken and verified (if required) prior to starting transfer operations. All lines must be blown-down and purged before disconnecting them from the tank car or vessel.

**7.6 PROTECTIVE PRACTICES DURING MAINTENANCE OF CONTAMINATED EQUIPMENT:**

Follow practices indicated in Section 6 (Accidental Release Measures). Make certain application equipment is locked and tagged-out safely. Always use this product in areas where adequate ventilation is provided. Collect all rinsates and dispose of according to applicable Federal, State, Provincial, or local procedures.

**7.7 EMPTY CONTAINER WARNING:**

Empty containers may contain residue and can be dangerous. Do not attempt to refill or clean containers without proper instructions. Empty drums should be completely drained and safely stored until appropriately reconditioned or disposed. Empty containers should be taken for recycling, recovery, or disposal through suitably qualified or licensed contractor and in accordance with governmental regulations. DO NOT PRESSURIZE, CUT, WELD, BRAZE, SOLDER, DRILL, GRIND, OR EXPOSE SUCH CONTAINERS TO HEAT, FLAME, SPARKS, STATIC ELECTRICITY, OR OTHER SOURCES OF IGNITION. THEY MAY BURST AND CAUSE INJURY OR DEATH.

**SECTION 8. EXPOSURE CONTROLS/PERSONAL PROTECTION****8.1 EXPOSURE LIMITS:**

MATERIAL	CAS#	EINECS#	TWA (OSHA)	TLV (ACGIH)	IDLH (NIOSH)
Sulfuric Acid	7664-93-9	231-639-5	1.0 mg/m <sup>3</sup>	1.0 mg/m <sup>3</sup>	15 mg/m <sup>3</sup>
Water	7732-18-5	231-791-2	None Known	None Known	

This product contains no EPA Hazardous Air Pollutants (HAP) in amounts > 0.1%.

**8.2 APPROPRIATE ENGINEERING CONTROLS:****RESPIRATORY EXPOSURE CONTROLS**

Airborne concentrations should be kept to lowest levels possible. If vapor, dust or mist is generated and the occupational exposure limit of the product, or any component of the product, is exceeded, use appropriate NIOSH or MSHA approved air purifying or air-supplied respirator authorized in 29 CFR 1910.134, European Standard EN 149, or applicable State regulations, after determining the airborne concentration of the contaminant. Air supplied respirators should always be worn when airborne concentration of the contaminant or oxygen content is unknown. Maintain airborne contaminant concentrations below exposure limits. If adequate ventilation is not available or there is potential for airborne exposure above the exposure limits, a respirator may be worn up to the respirator exposure limitations, check with respirator equipment manufacturer's recommendations/limitations. For particulates, a particulate respirator (NIOSH Type N95 or better filters) may be worn. If oil particles (such as: lubricants, cutting fluids, glycerine, and so on) are present, use a NIOSH Type R or P filter. For a higher level of protection, use positive pressure supplied air respiration protection or Self-Contained Breathing Apparatus or if oxygen levels are below 19.5% or are unknown.

**EMERGENCY OR PLANNED ENTRY INTO UNKNOWN CONCENTRATIONS OR IDLH CONDITIONS**

Positive pressure, full-face piece Self-Contained Breathing Apparatus; or positive pressure, full-face piece Self-Contained Breathing Apparatus with an auxiliary positive pressure Self-Contained Breathing Apparatus.

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## SECTION 8. EXPOSURE CONTROLS/PERSONAL PROTECTION (CONTINUED)

## VENTILATION

LOCAL EXHAUST: Necessary  
SPECIAL: None  
MECHANICAL (GENERAL): Necessary  
OTHER: None  
Please refer to ACGIH document, "Industrial Ventilation, A Manual of Recommended Practices", most recent edition, for details.

## 8.3 INDIVIDUAL PROTECTION MEASURES, SUCH AS PERSONAL PROTECTIVE EQUIPMENT:

## EYE PROTECTION:

Safety eyewear complying with an approved standard should be used when a risk assessment indicates this is necessary to avoid exposure to liquid splashes, mists or dusts. If contact is possible, chemical splash goggles should be worn, when a higher degree of protection is necessary, use splash goggles or safety glasses. Face shields are recommended when the operation can generate splashes, sprays or mists.

## HAND PROTECTION:

Use gloves chemically resistant to this material. Preferred examples: Butyl rubber, Chlorinated Polyethylene, Polyethylene, Ethyl vinyl alcohol laminate ("EVAL"), Polyvinyl alcohol ("PVA"). Examples of acceptable glove barrier materials include: Natural rubber ("latex"), Neoprene, Nitrile/butadiene rubber ("nitril") or ("NBR"), Polyvinyl chloride ("PVC") or "vinyl", Viton. Chemical-resistant, impervious gloves complying with an approved standard should be worn at all times when handling chemical products if a risk assessment indicates this is necessary. Considering the parameters specified by the glove manufacturer, check during use that the gloves are still retaining their protective properties. It should be noted that the time to breakthrough for any glove material may be different for different glove manufacturers. In the case of mixtures, consisting of several substances, the protection time of the gloves cannot be accurately estimated.

## BODY PROTECTION:

Use body protection appropriate for task. Cover-all, rubber aprons, or chemical protective clothing made from impervious materials are generally acceptable, depending on the task.

## WORK &amp; HYGIENIC PRACTICES:

Wash hands, forearms and face thoroughly after handling chemical products, before eating, smoking and using toilet facilities and at the end of the working period. Provide readily accessible eye wash stations & safety showers. Remove clothing that becomes contaminated. Destroy contaminated leather articles. Launder or discard contaminated clothing.

## SECTION 9. PHYSICAL &amp; CHEMICAL PROPERTIES

APPEARANCE:	Oily Liquid, Water-White to slightly yellow
ODOR:	None
ODOR THRESHOLD:	Not Available
pH (Neutrality):	< 1
MELTING POINT/FREEZING POINT:	-29 C / -20 F
BOILING RANGE (IBP, Dry Point):	276 to 281 C / 528 to 538 F
FLASH POINT (TEST METHOD):	Not Applicable
EVAPORATION RATE (n-Butyl Acetate=1):	Not Applicable
FLAMMABILITY CLASSIFICATION:	Noncombustible
LOWER FLAMMABLE LIMIT IN AIR (% by vol):	10.0 (Lowest Component)
UPPER FLAMMABLE LIMIT IN AIR (% by vol):	Not Available
VAPOR PRESSURE (mm of Hg)@20 C	< 0.3
VAPOR DENSITY (air=1):	3.4
GRAVITY @ 68/68 F / 20/20C:	
DENSITY:	1.830
SPECIFIC GRAVITY (Water=1):	1.835
POUNDS/GALLON:	15.3
WATER SOLUBILITY:	Complete
PARTITION COEFFICIENT (n-Octane/Water):	Not Available
AUTO IGNITION TEMPERATURE:	Not Applicable
DECOMPOSITION TEMPERATURE:	Not Available
VOCs (>0.044 Lbs/Sq In) :	0.0 Vol% / 0.0 g/L / 0.000 Lbs/Gal
TOTAL VOC'S (TVOC)*:	0.0 Vol% / 0.0 g/L / 0.000 Lbs/Gal
NONEXEMPT VOC'S (CVOC)*:	0.0 Vol% / 0.0 g/L / 0.000 Lbs/Gal
HAZARDOUS AIR POLLUTANTS (HAPS):	0.0 Wt% / 0.0 g/L / 0.000 Lbs/Gal
NONEXEMPT VOC PARTIAL PRESSURE (mm of Hg @ 20 C)	0.0
VISCOSITY @ 100 C (ASTM D445) 514.0	
VISCOSITY @ 20 C (ASTM D445):	Not Available
* Using CARB (California Air Resources Board Rules).	

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**SECTION 10. STABILITY & REACTIVITY****10.1 REACTIVITY & CHEMICAL STABILITY:**

Stable under normal conditions, but Reacts with most metals producing hydrogen which is extremely flammable & may explode.

**10.2 POSSIBILITY OF HAZARDOUS REACTIONS & CONDITIONS TO AVOID:**

Isolate from sources of ignition, heat, & open flame. Reacts vigorously with water.

**10.3 INCOMPATIBLE MATERIALS:**

The substance is a strong acid, reacts violently with bases and is corrosive. Upon heating, irritating and toxic fumes are formed including sulfur oxides. The substance is a strong oxidant & reacts violently with combustible & reducing materials. Corrosive to most common metals, forming flammable/explosive gas (hydrogen). Sulfuric acid reacts violently with water & organic materials with much heat. Hazardous gases are evolved on contact with chemicals such as cyanides, sulfides, and carbides.

**10.4 HAZARDOUS DECOMPOSITION PRODUCTS:**

Upon heating, irritating and toxic fumes are formed including sulfur oxides.

**10.5 HAZARDOUS POLYMERIZATION:**

Will not occur.

**SECTION 11. TOXICOLOGICAL INFORMATION****11.1 ACUTE HAZARDS****11.11 EYE & SKIN CONTACT:**

Severe burns to skin, defatting, dermatitis.  
Animal testing indicates this material is corrosive to the eye.  
Severe burns to eyes, redness, tearing, blurred vision.  
Liquid can cause severe skin & eye burns. Wash thoroughly after handling.

**11.12 INHALATION:**

Severe respiratory tract irritation may occur. Vapor harmful.

**11.13 SWALLOWING:**

Harmful or fatal if swallowed.

**11.2 SUBCHRONIC HAZARDS/CONDITIONS AGGRAVATED****MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE:**

Pre-existing disorders of any target organs mentioned in this Document can be aggravated by over-exposure by routes of entry to components of this product. Persons with these disorders should avoid use of this product.

**11.3 CHRONIC HAZARDS****11.31 CANCER, REPRODUCTIVE & OTHER CHRONIC HAZARDS:**

PROVEN Carcinogen, Human, Group 1 (IARC), SUSPECTED Carcinogen, Human, Group A2 (ACGIH).

**11.32 TARGET ORGANS:** May cause damage to target organs, based on animal data.

**11.33 IRRITANCY:** Irritating to contaminated tissue.

**11.34 SENSITIZATION:** No component is known as a sensitizer.

**11.35 MUTAGENICITY:** No known reports of mutagenic effects in humans.

**11.36 EMBRYOTOXICITY:** No known reports of embryotoxic effects in humans.

**11.37 TERATOGENICITY:** No known reports of teratogenic effects in humans.

**11.38 REPRODUCTIVE TOXICITY:** No known reports of reproductive effects in humans.

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COMPANY IDENTITY: Univar  
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SDS NUMBER: CDS-2441SDS DATE: 01/15/2015  
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## SECTION 11. TOXICOLOGICAL INFORMATION (CONTINUED)

A MUTAGEN is a chemical which causes permanent changes to genetic material (DNA) such that the changes will propagate across generational lines. An EMBRYOTOXIN is a chemical which causes damage to a developing embryo (such as: within the first 8 weeks of pregnancy in humans), but the damage does not propagate across generational lines. A TERATOGEN is a chemical which causes damage to a developing fetus, but the damage does not propagate across generational lines. A REPRODUCTIVE TOXIN is any substance which interferes in any way with the reproductive process.

## 11.4 MAMMALIAN TOXICITY INFORMATION

LD50 (Oral, Acute): 2140 mg/kg (Rat)  
LC50 / 2 hours: 510 mg/m<sup>3</sup> (Rat), 320 mg/m<sup>3</sup> (Mouse)

## SECTION 12. ECOLOGICAL INFORMATION

12.1 ALL WORK PRACTICES MUST BE AIMED AT ELIMINATING ENVIRONMENTAL CONTAMINATION.

## 12.2 EFFECT OF MATERIAL ON PLANTS AND ANIMALS:

This product may be harmful or fatal to plant and animal life if released into the environment. Refer to Section 11 (Toxicological Information) for further data on the effects of this product's components on test animals.

## 12.3 EFFECT OF MATERIAL ON AQUATIC LIFE:

The substance is harmful to aquatic organisms.

LC50 / 48 hours: 49 mg/L, Tap Water, 20 C (Bluegill sunfish)  
LC50 / 48 hours: 100 - 330 mg/L, Aerated Water (Flounder)

## 12.4 MOBILITY IN SOIL

Mobility of this material has not been determined.

## 12.5 DEGRADABILITY

This product is completely biodegradable.

## 12.6 ACCUMULATION

Bioaccumulation of this product has not been determined.

## SECTION 13. DISPOSAL CONSIDERATIONS

Processing, use or contamination may change the waste disposal requirements. Do not dispose of on land, in surface waters, or in storm drains. Waste should be recycled or disposed of in accordance with regulations. Large amounts should be collected for reuse or consigned to licensed hazardous waste haulers for disposal. ALL DISPOSAL MUST BE IN ACCORDANCE WITH ALL FEDERAL, STATE, PROVINCIAL, AND LOCAL REGULATIONS. IF IN DOUBT, CONTACT PROPER AGENCIES. EPA CHARACTERISTIC: D002

## SECTION 14. TRANSPORT INFORMATION

MARINE POLLUTANT: No  
DOT/TDG SHIP NAME: UN1830, Sulfuric acid, 8, PG-II  
DRUM LABEL: (CORROSIVE)  
IATA / ICAO: UN1830, Sulfuric acid, 8, PG-II  
IMO / IMDG: UN1830, Sulfuric acid, 8, PG-II  
EMERGENCY RESPONSE GUIDEBOOK NUMBER: 137



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**SECTION 15. REGULATORY INFORMATION****15.1 EPA REGULATION:**

SARA SECTION 311/312 HAZARDS: Acute Health, Chronic Health, Reactivity

All components of this product are on the TSCA list.

SARA Title III Section 313 Supplier Notification

This product contains the indicated <\*> toxic chemicals subject to the reporting requirements of Section 313 of the Emergency Planning & Community Right-To-Know Act of 1986 & of 40 CFR 372. This information must be included in all MSDSs that are copied and distributed for this material.

SARA TITLE III INGREDIENTS	CAS#	EINECS#	WT%	(REG.SECTION)	RQ(LBS)
*Sulfuric Acid	7664-93-9	231-639-5	93	(302,311,312,313)	1000

**15.2 STATE REGULATIONS:**

CALIFORNIA SAFE DRINKING WATER & TOXIC ENFORCEMENT ACT (PROPOSITION 65):

This product contains no chemicals known to the State of California to cause cancer or reproductive toxicity.

**15.3 INTERNATIONAL REGULATIONS**

The identified components of this product are listed on the chemical inventories of the following countries:

Australia (AICS), Canada (DSL or NDSL), China (IECSC), Europe (EINECS, ELINCS), Japan (METI/CSCL, MHLW/ISHL), South Korea (KECI), New Zealand (NZIoC), Philippines (PICCS), Switzerland (SWISS), Taiwan (NECSI), USA (TSCA).

**15.4 CANADA: WORKPLACE HAZARDOUS MATERIALS INFORMATION SYSTEM (WHMIS)**

D2B: Irritating to skin / eyes.

E: Corrosive Material.

This product was classified using the hazard criteria of the Controlled Products Regulations (CPR). This Document contains all information required by the CPR.

**SECTION 16. OTHER INFORMATION****16.1 HAZARD RATINGS:**

HEALTH (NFPA): 3, HEALTH (HMIS): 3, FLAMMABILITY: 0, PHYSICAL HAZARD: 2  
(Personal Protection Rating to be supplied by user based on use conditions.)  
This information is intended solely for the use of individuals trained in the NFPA & HMIS hazard rating systems.

**16.2 EMPLOYEE TRAINING**

See Section 2 for Risk & Safety Statements. Employees should be made aware of all hazards of this material (as stated in this SDS) before handling it.

**16.3 SDS DATE: 01/15/2015**

## Univar USA Inc Safety Data Sheet

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For Additional Information contact SDS Coordinator during business hours, Pacific time: (425) 889-3400

### Notice

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Do not use ingredient information and/or ingredient percentages in this SDS as a product specification. For product specification information refer to a product specification sheet and/or a certificate of analysis. These can be obtained from your local Univar sales office.

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This information relates only to the product designated herein, and does not relate to its use in combination with any other material or in any other process

**Exhibit B.4**  
**Standard Operating Procedure, Kinetic**  
**Phosphorescent Analyzer**

# HOMESTAKE MINING COMPANY OF CALIFORNIA

## Grants Reclamation Project



### STANDARD OPERATING PROCEDURE Kinetic Phosphorescent Analyzer

Written By:  
Michael Hay  
Jesse R. Toepfer  
Kyle Martinez

Current Revision:  
Rev. 0

Date of Most  
Recent Revision:  
21 December 2015

APPROVED FOR USE BY: JRT, DATE: 21 Dec 2015  
Closure Manager and RSO

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## Specialized Equipment Standard Operating Procedure (SOP)

For the use of the

### Kinetic Phosphorescence Analyzer (KPA) for Dissolved U(VI) Analysis

#### Operations Reminders

Replace the reference solution in the instrument:	Once per day
Generate a new calibration curve (i.e., run the calibration standards):	Once per week
Prepare fresh dilute Uraplex solution from the stock:	Once every 2 months or more frequently
Prepare fresh calibration standards:	Once every 4 to 6 months
Replace the laser dye:	Once every 6-8 months (Check periodically for evaporation/precipitation)

#### 1. Starting the Instrument and Software

- 1.1) Ensure that the KPA is turned ON. Check for the green light on the front panel of the instrument (the green light will be on when the instrument is on). The power switch is located on the back panel immediately above the power cord.
- 1.2) Let the instrument warm up for at least 1 hour. You can consider turning the instrument on the night before to save time.
- 1.3) Turn the computer on and log in: Select: "Chemchek"

User: Chemchek  
Password: kpa11

If the password expires, change it back to "kpa11".

- 1.4) Start the KPA software by double-clicking the "KPAWin" icon on the desktop or single-clicking the icon on the task bar.

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1.5) Click on "Security" → "Login" to log into the software:

User: Supervisor  
Password: Supervisor (note capital S)

1.6) If the computer and the KPA were off when you started, test the connection between the computer and KPA. Click on "Tools" → "Diagnostics" → "Probe KPA Unit". Ensure that there are no error messages and the light on the front of the KPA is still green.

1.7) Click on the "Analysis Input Form" icon (red vertical bar just to the right of the stop sign) in the upper left to start analyses. (You can also click on "Analyze" → "Sample Input".)

- This will open up the previous "Sample Input Configuration" window, including the calibration and the standards that were processed during the last run. If you wish to use the previous calibration curve, do not remove any of the samples from this input form; just continue to add samples to the current form.
- If you wish to create a new calibration curve, then clear the current grid and enter the parameters for the new calibration curve (see below). Clear the current grid by clicking on "Analyze" → "Remove All", and clicking "Yes" at the prompt.

## 2. Replacing the Reference Solution

At the beginning of each day's run, the reference solution needs to be changed.

**NOTE:** Pipettors and any other debris or waste generated should only be discarded in the trash can in the lab. Do not discard any materials in any other trash can.

2.1) Wear gloves when handling solutions and cuvettes.

2.2) Remove the reference (quartz glass) solution cuvette from the KPA. The reference solution spot is the one closer to the door hinge.

2.3) Ensure that the cuvette is clean. Rinse the inside with distilled water (fill with distilled water then evacuate the water using a pipette; repeat three times) and wipe the outside with a kimwipe.

2.4) Transfer 1.5 mL of dilute Uraplex solution into the cuvette using the adjustable pipetter.

**NOTE:** The Uraplex solution is kept in the refrigerator in the men's restroom. Be sure to check the date of the Uraplex solution. If the Uraplex solution is older than 60 days, prepare a new solution using the procedure in Attachment 1.

2.5) Transfer 1.0 mL of 100 ppb uranium standard solution into the cuvette using the adjustable pipetter and a clean pipette tip.

2.6) Mix thoroughly by replacing the cap on the cuvette and turning upside down several times, or by withdrawing and replacing solution 3 to 4 times with the adjustable pipetter.

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- 2.7) Place the cuvette back into the proper position (*i.e.*, the slot closest to the hinge in the front door) in the KPA.

### 3. Running the Calibration Standards

Uranium concentration is determined in samples by comparison against a calibration curve. This calibration curve is generated by running calibration standards. A new calibration curve should be generated by re-running the calibration standards at least once per week. It may be useful to get into the following routine:

- If the instrument is being run once per week or less, plan on running a calibration set every time you run.
- If the instrument is being run more than once per week, plan on running a calibration on the first day the instrument is run in a given week.

Add the calibration standards to the sample input form using the following procedures:

- 3.1) If you wish to run a new calibration standard, clear the current sample analysis grid by clicking on "Analyze" → "Remove All", and clicking "Yes" at the prompt.
- 3.2) The standard calibration set can be loaded into the sample analysis form by clicking on "File" → "Open", highlighting "WeekCal", and then clicking "Load". This should load the following set of calibration standards into the sample analysis form:

<u>Sample ID:</u>	<u>Description:</u>	<u>Sample Type:</u>	<u>Range:</u>
weakHNO3	2% HNO <sub>3</sub> background	BckGnd	Both
5ppb_std	5 ppb standard	CalStd	Both
10ppb_std	10 ppb standard	CalStd	Both
50ppb_std	50 ppb standard	CalStd	Both
100ppb_st	100 ppb standard	CalStd	High
1000ppb_st	1000 ppb standard	CalStd	High
100ppb_ch	100 ppb check standard	CChkStd	High

If the form was populated correctly, proceed to step 3.8 below.

If the "WeekCal" file is not present or if a modified calibration set is to be used, the calibration set can be entered manually:

- 3.3) Add a new sample to the input list by clicking the "+" icon. The first sample should be "BckGnd". Provide the following information for the sample:
- Sample ID                      example: weakHNO3 (don't use spaces)
  - Sample Type                  Leave this as "BckGnd" and plan on running a 2% HNO<sub>3</sub> blank.
  - Description                    Your preference, spaces allowed.
  - Preferred Range              Both

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Note that all RED fields need an entry, but you will leave the defaults for most of them (Alarms/Warnings, Calibration Config, KPA Unit Settings, Performance Limits, QC Limits, and Concentration Units).

- 3.4) Click on the "+" icon again, and provide the following information for the first calibration standard:

- |                                 |   |
|---------------------------------|---|
| ○ Sample ID                     | example: 5ppb_std (don't use spaces)      |
| ○ Sample Type                   | CalStd (choose from drop-down)            |
| ○ Description                   | Your preference, spaces allowed           |
| ○ Standard ID                   | Keep this simple; example: 5              |
| ○ Standard Concentration (ug/L) | example: 5                                |
| ○ Preferred Range               | Low/High/Both/Auto (see guidelines below) |

Leave other RED fields at their default values.

- 3.5) Repeat step 3.4 for each of the calibration standards.
- 3.6) Before moving on to sample analyses, it is recommended that a calibration check standard be run. This may consist of any of the calibration solutions. Click on the "+" icon, set the Sample Type to "CChkStd", and follow the steps above to fill in the sample information.
- 3.7) If desired, save the details of the calibration set to a file so they can be quickly recalled the next time you run a calibration set. Click on "File" → "Save As", specify a file name in the "Enter Sample Name ->" box, and click "Save All" (be careful to click Save All instead of Save, or else it will only save details for the last sample in the list).

Prepare and run the calibration standards using the following procedures:

**NOTE:** For the first standard for the weak HNO<sub>3</sub> solution, use 1.5 mL Uraplex and 1.0 mL of 2% HNO<sub>3</sub>.

- 3.8) Add 1.5 mL of dilute Uraplex solution + 1.0 mL of the desired standard solution to the cuvette.
- If using the quartz glass cuvette, be sure it is thoroughly cleaned with distilled water before use.
  - If using a plastic cuvette, use a clean fresh cuvette and dispose after use. Use a kimwipe to lightly wipe away any dust or Styrofoam particles. Do not rub too vigorously, or you may create an electrostatic charge on the plastic.
- 3.9) Insert the cuvette into the sample position in the KPA (slot furthest from the sample chamber door hinge). Ensure that the Reference Solution is also in place (slot closest to the door hinge).
- 3.10) Click on the "Analyze Selection" (lightning bolt) icon to start the run. Follow the prompts as they appear on the screen, loading each calibration in sequence into the KPA.



Assess the quality of the calibration curve:

- 3.11) Once all of the calibration standards have been run, click on the "Display Calibration" icon to view the calibration (it is the icon that looks like a book, next to the lightning bolt). This brings up the "Select Calibration Samples" window.
- 3.12) Review the data. If satisfied with the calibration, **PRINT the screen for recordation and file appropriately**. If not satisfied, repeat calibration. If not satisfied after multiple attempts, see Troubleshooting Steps.
- 3.13) Inspect the  $R^2$  values for both the low range and high range calibration curves. This is a measure of the goodness-of-fit of the line fitted to the individual calibration data points. The  $R^2$  value should be very close to 0.999 or higher for both curves; if it is close to 0.98 or below, the calibration may be poor. Repeating the calibration process with new standards may be required.
- 3.14) Inspect the "% Disc" for each of the individual calibration standards. This is a measure of how much a given data point differs from the fitted line. If any of these values are substantially outside  $\pm 10\%$ , that particular calibration standard may not be accurate. The calibration process may need to be repeated with freshly-prepared standards.
- 3.15) If you want, you can view a plot of the calibration curve by clicking the "Plot" button. Return to the previous view by clicking the "Grid" button.
- 3.16) Return to the "Sample Input Configuration" window by closing the "Select Calibration Samples" window.

#### 4. Running Samples

- 4.1) Before sample analyses, ensure that the following pre-operation checks have been made:
  - o Did you replace the Reference Solution?
  - o Do you need to re-run the Calibration Standards?
  - o Is the dilute Urplex solution fresh?
  - o Have the samples been properly digested?
- 4.2) Add a new sample to the input list by clicking the "+" icon within the "Sample Input Configuration" window.
- 4.3) Add information for the following (note that all RED fields require a value, but some you will not change from the default):

o Sample ID	Use sample name (no spaces)
o Sample Type	Sample (pick from drop-down)
o Description	Your preference, spaces allowed
o Prediluted Volume (mL)	<u>see below (Sample Volume Information)</u>
o Diluted Volume (mL)	<u>see below (Sample Volume Information)</u>

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- o Sample Volume (mL) see below (Sample Volume Information)
- o Analysis Volume (mL) see below (Sample Volume Information)
- o Preferred Range Low or High (see guidelines in step 6 below)

4.4) Add 1.5 mL of dilute Uraplex solution and 1.0 mL of the desired sample solution into the cuvette (quartz or disposable plastic) using the adjustable pipetters.

4.5) Click on the "Analyze Selection" (lightning bolt) icon to start the run.

You have the option of adding multiple samples to the input list before clicking the "Analyze Selection" option (click the "+" before clicking the lightning bolt). (**NOTE:** The volumes need to be entered before the "Analyze Selection" button is pushed.) The software will then prompt you when it is time to load each sample into the instrument. However, it is advisable to run each sample before adding the next sample to the input list, rather than adding several samples to the input list at once. There are two reasons for this:

- If there is a problem with the current sample being run, you may wish to modify the planned sample sequence (for example, re-run the current sample).
- It will be easier to view the results of the current sample in the results window before it is displaced by the result of the next sample (this is described in more detail below).

Sample Volume Information

The KPA software contains four input fields for sample volumes, which automatically accounts for two sample dilutions.

The first two fields should be used to account for dilution during sample preparation:

Prediluted Volume = Volume of sample added to the digestion vial

Diluted Volume = Final solution volume in the vial after digestion

Example: You could perform a digestion by adding 3 mL of your sample to a digestion vial, plus some acid, plus some peroxide. Then you heat the sample to complete dryness during the digestion, and then you add 5 mL of acid solution to redissolve. The prediluted volume would be 3 mL and the diluted volume would be 5 mL.

See **Attachment 1** for more detail on sample preparation and dilution procedures.

The second two fields should be used to account for any additional dilutions that are made following the initial sample preparation.

Sample Volume = Volume of sample added to the vial

Analysis Volume = Volume of solution in the vial after adding sample and diluent

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Example: You could prepare a 10x dilution by adding 0.5 mL of sample to 4.5 mL of 2% nitric acid (for a total volume of 5 mL): Sample Volume = 0.5 and Analysis Volume = 5.0.

Example: You can also dilute directly into the cuvette for the analysis. Instead of adding 1.5 mL dilute Uraplex + 1.0 mL of sample to the cuvette, you could add 1.5 mL of dilute Uraplex, 0.2 mL of sample, and 0.8 mL of 2% nitric acid. This would yield a 5:1 sample dilution: Sample Volume = 0.2 mL, Analysis Volume = 1.0 mL (= 0.2 mL + 0.8 mL).

If no dilution is made in the second step, then Sample Volume and Analysis Volume should be the same (you can simply enter 1.0 and 1.0).

## 5. Running Calibration Checks

While you are running the instrument, it is recommended that you run a calibration check once per every 8 to 10 samples, as well as at the beginning and end of the sample run.

- The calibration check standard may be something different each time
- The calibration check standard can either be one of the calibration standards you prepared in-house, or one of the PerkinElmer lab-prepared standards. It is recommended that you alternately run both.
- Calibration check blanks should also be run periodically, consisting of 2% nitric acid.
- As with all samples and standards, the calibration checks (standards and blanks) must be prepared with 1.0 mL sample and 1.5 mL dilute Uraplex.

The following is a hypothetical sample list for a given day's run:

	<u>Sample Type:</u>
• Background (2% nitric acid)	BckGnd
• Calibration standards (typically 5)	CalStd
• Calibration Check (1 std; e.g., 100 ppb)	CChkStd
• Sample set (8 to 10 or less)	Sample
• Calibration Check (1 std; e.g., 10 ppb)	CChkStd
• Sample set (8 to 10 or less)	Sample
• Calibration Check (1 std; e.g., 100 ppb)	CChkStd
• Calibration Check Blank (2% HNO <sub>3</sub> )	CChkBlk

Use the following procedures to add calibration checks to the sample run:

5.1) Add a new sample to the input list by clicking the "+" icon.



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5.2) Add information for the following (note that all RED fields require a value, but some you will not change from the default):

- |                                 |  |
|---------------------------------|--|
| ○ Sample ID                     | example: 100ppb_Chk                        |
| ○ Sample Type                   | CChkStd or CChkBlk                         |
| ○ Description                   | Your preference, spaces allowed            |
| ○ Standard ID                   | example: 100                               |
| ○ Standard Concentration (ug/L) | example: 100                               |
| ○ Preferred Range               | Low or High or Both (see guidelines below) |
|                                 | Both (if running a check blank)            |

5.3) Add 1.5 mL of dilute Uraplex solution and 1.0 mL of the desired calibration standard/blank into the cuvette (quartz or disposable plastic).

5.4) Click on the "Analyze Selection" (lightning bolt) icon to start the run.

## **6. Choosing the Appropriate Analysis Range**

The cutoff for operating in the low range of the KPA is approximately 50 to 100 ppb uranium. Above this value, the intensity is too high for the detector to measure accurately. For samples above this cutoff, an aperture is placed in the path of the phosphorescence to limit the amount of light reaching the detector – this allows the KPA to operate in the high range.

The following are some guidelines for choosing the appropriate range:

- Because the highest concentration standard within the low range is 50 ppb, the low range should only be used up to that concentration. For samples with higher concentration, the high range should be chosen.
- If the sample concentration is higher than the highest standard used in the high range (1000 ppb), the sample must be diluted such that it falls either within the low range or the high range.
- For many concentration standards and blanks, the range will be set to "Both" (such that the sample is run under both the high and low range).
- If it is unknown whether the sample concentration falls within the high or low range, the range can be set to "Auto". However, this is generally not recommended, as this does not always work (sometimes the sample is run in the low range even though it is more appropriate in the high range).
- If you are unsure which range is appropriate, a good policy is to run it in the high range. If you find that the raw concentration (i.e., concentration measured by the instrument before correction for dilution) is below 100 ppb, re-run the sample in the low range to get a more reliable concentration.

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## 7. Viewing Results during the Run

At the end of each sample analysis, the results of that analysis are automatically displayed in a results window. It may be labeled "high" or "low" depending on the range run; if both ranges were run, two results windows will be present. However, this window is often hidden beneath the "Sample Input Configuration" window.

- 7.1) Minimize the Sample Input Configuration window and inspect the results window. Note that this window will not update until the current sample is done processing (this may take several seconds).
- 7.2) Record the information by outputting to a designated Excel file at the end of the run (See Section 8).
- 7.3) Note that TWO concentrations will be displayed in this window:
  - The "Concentration" value, which is included in the "Results" outlined box. This is the concentration measured by the instrument (non-dilution corrected)
  - The "Final Result" is the concentration corrected for dilutions. **This is the actual uranium concentration in the original sample.**

Check the "Concentration" value and make sure that it is within range of the calibration used:

- If the "Concentration" is higher than the highest standard used (typically 1000 ppb), the sample will need to be diluted and re-run. If dilution is required to get below the highest standard used, refer to Attachment 1.
  - If the sample was run in the high range and the "Concentration" is below 100 ppb, consider re-running the same sample in the low range.
- 7.4) Check the sample lifetime. The sample lifetime should be between approximately 250 to 350  $\mu$ s. Lifetimes below 250  $\mu$ s indicate substantial quenching, and the uranium concentrations obtained may be highly erroneous.
  - 7.5) Check the reference ratio. This number should be very close to 1; if it's not, this may indicate that the reference solution may need to be changed, or the calibration standards may need to be re-run.
  - 7.6) The Sample  $R^2$  value should be very close to 1; if not, the uranium concentration obtained is likely erroneous (it is likely that the Sample Lifetime will also be low).
  - 7.7) Check for any alarms/warnings that went off during the sample run, and determine the extent to which data quality was impacted. A guide to the alarms and warnings is included in the Chemchek attachments.

Note that the results window only contains information for the most recent sample analysis completed. When another sample is run, the results for this sample will displace the results for the previous sample;



therefore, you will want to **view and record** the results for a given sample before it is displaced by the next sample in line.

## 8. Downloading and Printing the Data

At the completion of the day's run, particularly if concentration information for each sample was not written down during the run, export the concentrations to a text file using the following procedure.

- 8.1) Click on the Sample Reports icon (white paper icon just to the right of the upper stop sign icon) to open the Sample Reports window.
- 8.2) Choose the date range for which you want to retrieve samples. If you only want to download the current day's samples, click the "Between" button in the "Date" box and enter the current day's date in both boxes.
- 8.3) Leave all other fields in their default settings.
- 8.4) Click the "Retrieve Samples" button. The sample list of interest should appear in the window. If the samples of interest are not in the window, double-check the date range and click "Retrieve".
- 8.5) To export the data, the number next to each sample in the "Sample Key" column needs to be highlighted. To export all samples in the window, click on the first "Sample Key" value (it should then be highlighted blue), use the window scroll bar to scroll down to the bottom of the list, hold the Shift key down, and click on the last "Sample Key" value. This should highlight all of the Sample Key values blue.
- 8.6) Click the "Export Reports" button. This should bring up a new window.
  - o **IMPORTANT:** Ensure that both "Result-AnalyticalResult" AND "Result-FinalResult" are included in the "Included Export Fields" list (see step 8.12 below). By default, the Analytical Result may be in the list, but not the Final Result. If the Final Result is not listed, then find it in the "Available Export Fields" list, highlight it, and then click the "Add Field(s)" button.
- 8.7) Click the "Export Data" button. If this button is grayed out, you may need to click the "Apply" button first.
- 8.8) This should bring up a "Save As" window. Give the file a name of your preference (a date stamp is a good choice; e.g., KPAEXPORT\_07Jan2014.TXT), but retain the ".TXT" extension. It is recommended that you save the file to the computer in a common folder (check the "Documents" → "KPA Output" folder).
- 8.9) Once saved, open Windows Explorer and then copy the file from the common folder on the computer to a USB key.

This procedure saves a comma-delimited text file. The results are most easily viewed by importing the data into Microsoft Excel.



- 8.10) Open Microsoft Excel, and go to [Windows Icon] → "Open".
- 8.11) In the "Open" window, be sure that "All Files (\*.\*)" is highlighted in the drop-down, and then open the KPA export text file.
- 8.12) This should bring up a "Text Import Wizard" window. The text file is comma delimited. To import the text, highlight "Delimited" (rather than "Fixed width"), click the "Next" button, and then check the "Comma" box. Then click "Finish".
- 8.13) Sample results and statistics should now be listed in separate Excel columns. Note the following:
- As described above, the "Analytical Result" is the concentration measured in the solution by the instrument – this does not correspond to the uranium concentration in your sample if any dilutions or digestions have been performed. Based on the dilution information entered when you ran the sample, the "Final Result" is calculated – this is the actual concentration of uranium in your sample.
  - This should also contain other important details on your sample run:  $R^2$  value, sample lifetime, etc. Review these parameters for quality as outlined above.
- 8.14) Save the Excel file by clicking "File" → "Save As"; with "Save as type:" set to "Excel Workbook" (with the ".xls" or ".xlsx" extension). Additional recommendations for data analysis are as follows:
- Do not modify the data in the original worksheet tab that you imported; instead, make a copy of the worksheet within the same workbook for data analysis and plotting. That way, the original tab includes all of the "raw" data. To do this, right-click on the tab at the bottom of the worksheet, click "Move or Copy...", check the "Create a copy" box, then click "OK". Do this as many times as you like to have separate data analysis/plotting tabs.
  - When sample names only consist of numbers, Excel will interpret that as a number. This can be switched back to text by putting an apostrophe at the beginning of the number (Excel then interprets it as text).

## 9. Shutting Down the Instrument

- 9.1) Remove the sample cuvette from the instrument. If using the quartz cuvette, thoroughly rinse with distilled water and wipe the outside dry with a kimwipe. The cuvette can be stored in the instrument for later use.
- 9.2) Remove the Reference Solution cuvette, dispose of the solution, and clean the cuvette. Store in the instrument.



- 9.3) After downloading the data (step 8 above), close all open windows within the main KPA Win window by clicking the X's in the upper right. Do not close the main KPA Win window with the X.
- 9.4) Once all open windows are closed, click on "File" → "Exit" to close the software.
- 9.5) If you plan to run the next day, you can leave the computer and the KPA instrument on. If you do not intend to run the instrument again soon, shut down the computer and turn off the KPA instrument (the power switch is located on the back panel immediately above the power cord).

## 10. GENERAL TROUBLESHOOTING:

### Calibration Issues:

1. Check the **Standard**.
  - a. If the calibration date is suspect, try:
    - i. Using an auxiliary Standard
    - ii. Order a new Standard from the lab.
  - b. If the integrity of the bottle is suspect; look for contamination or fouling in the solution. If found:
    - i. Use the auxiliary Standard
    - ii. Order a new Standard from the lab.
2. Check the **Reference Solution**
  - a. The reference solution should be replaced each day. If a fresh reference solution was not used, or is suspect, replace the reference solution and repeat the calibration procedure.
  - b. If the reference solution is believed to be contaminated, replace the reference solution and repeat the calibration procedure.
  - c. Review the steps in the sample preparation. Ensure the agents into the solution were properly dosed.
    - i. Check that the HNO<sub>3</sub> concentration is accurate; if a formula was used to determine the dilution ratio, then:
      1. Verify the original concentration of the HNO<sub>3</sub> solution.
      2. Re-check the calculation.
    - ii. Prepare a new solution
3. Check the **Quartz Vial**
  - a. The quartz vial needs to be clean. This means: Free of scratches and free of air bubbles when the solution is being analyzed. Inspect the quartz vial:
    - i. If scratches are found, discard the container and replace with a new one.
    - ii. If air bubbles are present in the solution, gently agitate until the air bubbles are released to the surface of the solution. Be sure to wipe the outside of the container with a kimwipe (or other lint free wipe) before replacing.



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Grants Reclamation Project

KPA Standard Operating Procedure (Rev. 0)

- b. The quartz vial needs to be positioned properly. This means: Squarely in position, and fully seated. Check to ensure both of these items and correct as necessary.
4. Check the **Laser Dye**.
  - a. The laser dye needs to be replaced every 6 to 8 months. Check to ensure this has been done.
  - b. Check for evaporation or precipitation. If either is present, replace the dye.
5. If calibration issues still persist:
  - a. Consult the KPA User's Manual.
  - b. Call technical support.

**Other Issues:**

For additional technical support, it may be necessary to call an outside consultant for help.

**Attachments**

- Attachment 1: Procedures for Sample and Reagent Preparation
- Attachment 2: Procedure for Preparation of Calibration Standards
- Attachment 3: Procedure for Replacement and Preparation of Laser Dye
- Attachment 4: List of Supplies and Vendors
- Attachment 5: KPA Instrument Operation Manual (Chemchek)
- Attachment 6: KPAWin Software User's Guide (Chemchek)
- Attachment 7: Potential consulting contacts for technical support

# Attachment 1

## Sample Preservation, Digestion, Dilution, and Reagent Preparation

### Units:

Volume: 1 liter (L) = 1,000 milliliters (mL)  
1 mL = 1,000 microliters (μL)

Concentration: 1 milligram per liter (mg/L) = 1 part per million (ppm)  
1 mg/L = 1,000 micrograms per liter (μg/L)  
1 μg/L = 1 part per billion (ppb)

### Contents:

- Preservation of Freshly-Collected Samples
- Sample Digestion on the Hot Plate
- Sample Dilution
- Reagent Preparation: 50% HNO<sub>3</sub>, 2% HNO<sub>3</sub>, and 20x Dilution Uraplex

### 1) Preservation of Freshly-Collected Samples

Aqueous samples should be preserved with acid to prevent potential metals precipitation and adsorption to storage bottles. Acid preservation involves acidification of the sample to below pH 2 using concentrated nitric acid (HNO<sub>3</sub>). This is typically achieved by addition of 1.5 mL of 70% w/w nitric acid per liter of sample.

Acid dosing for sample preservation (use EITHER concentrated HNO<sub>3</sub> OR 50% HNO<sub>3</sub>, not both):

<u>Sample Volume</u>	<u>Acid Dose</u> <u>(Concentrated HNO<sub>3</sub>)</u>	<u>OR</u>	<u>Acid Dose</u> <u>50% HNO<sub>3</sub></u>
20 mL	0.03 mL		0.06 mL (60 μL)
50 mL	0.075 mL		0.15 mL (150 μL)
100 mL	0.15 mL		0.3 mL (300 μL)
250 mL	0.375 mL	OR	0.75 mL (750 μL)
500 mL	0.75 mL		1.5 mL
1000 mL	1.5 mL		3.0 mL

## 2) Sample Digestion on the Hot Plate

Sample "digestion" may need to be performed for several reasons, primarily to remove chloride from the solution because it interferes with the uranium analysis. Digestion to remove chloride involves addition of acid and evaporating the sample to complete dryness; this causes chloride to evaporate with water as hydrochloric acid (HCl), thereby removing it from the sample. Other dissolved solids (including uranium) do not evaporate; they precipitate in soluble salts at the bottom of the digestion vial and are redissolved with addition of 2% HNO<sub>3</sub>.

- Label a set of fresh 20 mL scintillation vials according to the sample names using a permanent marker.
- Transfer sample solutions to the scintillation vials using the adjustable pipetter. See the suggested volumes below.
  - It is recommended that a new pipette tip be used between each sample. An acceptable alternative may be to rinse the pipette tip between each use by pulling clean water into the pipette.
- Add 50% HNO<sub>3</sub> solution to each of the scintillation vials containing the aqueous sample (see suggested volumes below).
- Remove the cap from the vials and place the samples to be digested on the hot plate and turn the hot plate on until all water has evaporated. The dial setting should be at approximately 2.5 or 3.0.

### Suggested volumes for digestion (adjust as necessary):

Add to vial before heating:

Sample volume: 2 mL

50% HNO<sub>3</sub> volume: 0.3 mL

3% H<sub>2</sub>O<sub>2</sub> 1 mL

Use ONLY if first attempt at digestion yielded a low solution lifetime on the KPA

Add to vial after heating to complete dryness:

2% HNO<sub>3</sub> volume: 3 mL

Add to sample after heating to complete dryness.

## 3) Sample Dilution

To obtain accurate measurements with the KPA, the sample concentration needs to be lower than the highest concentration standard used, which is typically 1,000 ppb. If the sample concentration measured by the KPA is above 1,000 ppb, then the sample must be diluted and re-run. The following procedures should be used:

- Run the sample on the KPA using the procedures outlined in the Main SOP. Be sure to account for any dilutions performed during the digestion step (see Section 4, "Sample Volume Information", Main SOP), but start by running the sample without additional dilution.

- After the sample run is completed, check the "Concentration" value in the results window (see Section 7.2, Main SOP). If this concentration is above 1,000 ppb, dilute the sample and re-run. The following guidelines are suggested:

Measured Concentration (ppb)	Suggested Dilution	Dilution Into Vial			Dilution Directly Into Cuvette		
		Sample Volume (mL)	2% Nitric Acid Volume (mL)	Total Volume (mL)	Sample Volume (mL)	2% Nitric Acid Volume (mL)	Total Volume (mL)
1,000 to 2,000	5x	1	4	5	0.2	0.8	1
2,000 to 5,000	10x	0.5	4.5	5	0.1	0.9	1
5,000 to 10,000	20x	0.5	9.5	10	not recommended		
10,000 to 50,000	50x	0.2	9.8	10	not recommended		

- Account for the dilutions above in the "Sample Volume" and "Analysis Volume" boxes in the KPA window, where the "Analysis Volume" is the Total Volume indicated in the table.
- For concentrations above 50,000 ppb (50 ppm), a serial dilution may be required (e.g., perform a 100x dilution by performing a 10x dilution on the sample, then another 10x dilution on the diluted sample).

#### 4) Reagent Preparation: 50% HNO<sub>3</sub>, 2% HNO<sub>3</sub>, and 20x Dilution Uraplex

##### Preparation of 50% Nitric Acid

The 50% nitric acid solution is prepared on a volume basis, simply by adding 1 part concentrated acid to 1 part water. Strictly speaking, this yields a solution less than 50% nitric acid by volume (since concentrated nitric acid is typically 70% HNO<sub>3</sub> by volume, a 1:1 dilution in water yields a 35% solution by volume). More accurately, the acid stock used (70% HNO<sub>3</sub>) is a 16 M (moles per liter) nitric acid solution, and the "50% Nitric Acid" solution prepared below is an 8 M nitric acid solution.

Procedure for preparation of 100 mL of 50% (8 M) nitric acid:

- Add 50 mL of clean water to a clean, empty 100 mL or 250 mL plastic bottle. Label as "50% Nitric Acid (8 M)" using a Sharpie pen. This can be done in one of two ways:
  - Fill a 50-mL volumetric flask with clean water up to the line, then pour from the flask into the clean plastic bottle, OR
  - Place the plastic bottle onto the electric balance, Tare (zero) the balance, then fill with clean water up to 50 g.
- Using the rubber bulb, pull acid from the jar into the graduated acid dispenser such that more than 50 mL is present in the dispenser.
- Dispense 50 mL acid into the plastic bottle containing water, using the graduations on the acid dispenser as a guide. (REMEMBER: Always add acid to water, not the other way around.)

##### Preparation of 2% Nitric Acid

The 2% nitric acid solution is prepared via 25X dilution of the 50% nitric acid stock.

Procedure for preparation of 100 mL of 2% nitric acid (Volumetric Flask method):



- Fill a clean 100 mL glass volumetric flask approximately halfway with clean water.
- Using the adjustable pipetter with a clean tip, add 4 mL of 50% nitric acid stock to the water within the volumetric flask.
- Continue to fill the volumetric flask to the white line with clean water.
- Mix well (end-over-end), then transfer the solution to a clean 100 mL or 150 mL plastic bottle.

#### Preparation of 20x Dilution Uraplex

Fresh dilute Uraplex solution should be prepared at least once every one to two months. When not in use, it may be stored in the refrigerator, but it is advisable to remove it from the refrigerator the day before use so that it may come up to room temperature.

Procedure for preparation of 100 mL of 20x Dilution Uraplex:

- Fill a clean 100 mL glass volumetric flask approximately halfway with clean water.
- Using the adjustable pipetter with a clean tip, add 5 mL of concentrated Uraplex solution to the water within the volumetric flask. (The concentrated Uraplex solution should always be stored in the refrigerator.)
- Continue to fill the volumetric flask to the white line with clean water.
- Mix well (end-over-end), then transfer the solution to a clean 100 mL or 150 mL plastic bottle.
- Add date to the bottle of all newly prepared solutions using a permanent marker.

## Attachment 2

### Preparation of Uranium Calibration Standards

Updated: 10/26/2015

Standard solutions must be prepared at least once every 4 to 6 months and stored tightly capped, in a cool place (ideally in the refrigerator) to avoid evaporation. Wear disposable gloves and eye protection while handling stock and standard solutions.

The procedure outlined is for the preparation of uranium calibration standards from a 1000 ppm stock at the following concentrations:

- 5 ppb ( $\mu\text{g/L}$ )
- 10 ppb
- 50 ppb
- 100 ppb
- 1000 ppb

Use the pre-labeled 100-mL volumetric flasks for preparation of the standards. You will also need distilled water (1-gallon plastic jugs purchased from Walmart) for glassware rinsing as well as deionized water (supplied by ARCADIS laboratory) for preparation of solutions. Although the distilled water is very clean and appropriate for rinsing of glassware before use, the deionized water is even cleaner and should be used for preparation of solutions.

- 1) Ensure that glassware is clean and dry, following rinsing with tap water and then with distilled water. Ensure adjustable pipetter is functional and accurate, and there are enough disposable tips available before starting work. Tips should be changed after every use.
- 2) Check to make sure that you have six 100-mL volumetric flasks with glass stoppers, labeled "10 ppm", "1000 ppb", "100 ppb", "50 ppb", "10 ppb", and "5 ppb".
- 3) Fill the "10 ppm" (not the "10 ppb") 100-mL volumetric flask about halfway with deionized water. Using the adjustable pipetter, add 1 mL of the stock uranium solution (1000 ppm U) to the 100 mL volumetric flask.
- 4) Continue to fill the flask to the white line on the neck using deionized water.

When completing any volumetric glassware, slowly fill it until water reaches the neck of the flask, or gets closer to the marked volume line. Then, use a disposable transfer pipet or squirt bottle to transfer more water into the flask, until the bottom of the meniscus reaches the marked line exactly, without passing it. If too much water is added and the overall volume surpasses the marked line, the procedure will need to be repeated.

- 5) Cap the volumetric flask and gently turn it upside down twice to mix the solution. Transfer the solution to a clean, dry glass beaker labeled "10 ppm U". This solution will be used to make the five standards used for instrument calibration.

1000 ppb solution:

- 6) Fill the "1000 ppb" 100-mL volumetric flask about halfway with deionized water.
- 7) Using the adjustable pipetter, add 10 mL of the 10 ppm U solution to the volumetric flask.
- 8) To the same volumetric flask, add 2 mL of 50%  $\text{HNO}_3$  solution. Complete the flask up to the marked line with deionized water, cap and mix.
- 9) Transfer the solution to a series of 20 mL scintillation vials for storage and use. Label the scintillation vials with the uranium concentration and date. Note that some of the 1000 ppb solution will be used to prepare the 50 ppb, 10 ppb, and 5 ppb solutions below.

100 ppb solution:

- 10) Fill the "100 ppb" 100-mL volumetric flask about halfway with deionized water.
- 11) Add 1 mL of the 10 ppm U solution to the volumetric flask.
- 12) To the same volumetric flask, add 2 mL of 50%  $\text{HNO}_3$  solution. Complete the flask up to the marked line with deionized water, cap and mix. Transfer the solution to 20 mL scintillation vials for storage and use.

50 ppb solution:

- 13) Fill the "50 ppb" 100-mL volumetric flask about halfway with deionized water.
- 14) Add 5 mL of the 1000 ppb U solution to the volumetric flask.
- 15) To the same volumetric flask, add 2 mL of 50%  $\text{HNO}_3$  solution. Complete the flask up to the marked line with deionized water, cap and mix. Transfer the solution to 20 mL scintillation vials for storage and use.

10 ppb solution:

- 16) Fill the "10 ppb" 100-mL volumetric flask about halfway with deionized water.
- 17) Add 1 mL of the 1000 ppb U solution to the volumetric flask.
- 18) To the same volumetric flask, add 2 mL of 50%  $\text{HNO}_3$  solution. Complete the flask up to the marked line with deionized water, cap and mix. Transfer the solution to 20 mL scintillation vials for storage and use.

5 ppb solution:

- 19) Fill the "5 ppb" 100-mL volumetric flask about halfway with deionized water.
- 20) Add 0.5 mL (500  $\mu\text{L}$ ) of the 1000 ppb U solution to the volumetric flask.

21) To the same volumetric flask, add 2 mL of 50%  $\text{HNO}_3$  solution. Complete the flask up to the marked line with deionized water, cap and mix. Transfer the solution to 20 mL scintillation vials for storage and use.



## Attachment 3

### Preparation of Laser Dye

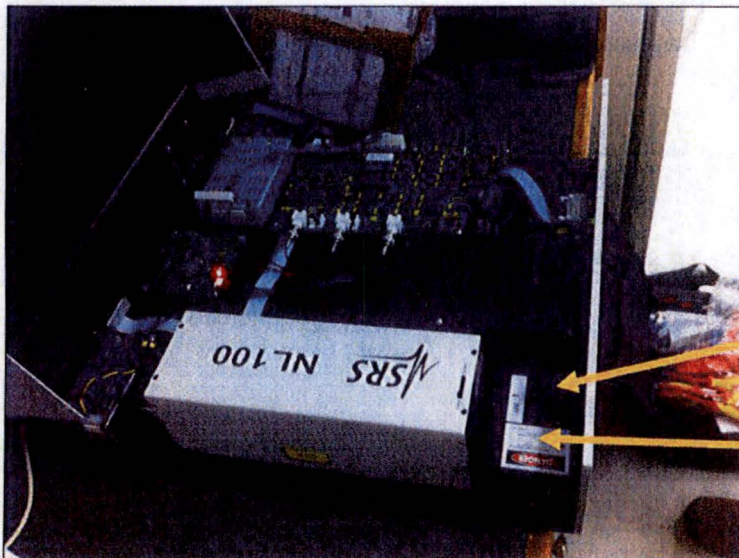
Updated: 10/27/2015

#### Contents:

- Preparing and Storing the Laser Dye Solution
- Replacing the Laser Dye Solution

The laser dye cell, which contains the laser dye solution, is located inside the KPA. The laser dye solution consists of an organic dye dissolved in methanol. This dye solution is necessary for producing the proper wavelength of light for excitation and measurement of uranium in the sample. Over time, the organic dye breaks down and must be replaced by emptying and refilling the laser dye cell. It is recommended that the laser dye solution in the instrument be replaced every 6 to 8 months, but consider changing more frequently (every 3 months) if the instrument is being used often (daily or weekly).

The Dye Module is shown in the figure below. The laser dye cell is located inside the Dye Module. To access the module, the KPA cover must be removed, as described below. For additional information, refer to Section 2.3.3 of the KPA Instrument Operation Manual.



Round black cap  
covering the laser  
dye cell holder

Black box with "Danger"  
sticker is the Dye Module

#### Preparing and Storing the Laser Dye Solution:

The laser dye solution is prepared by dissolving the dye powder (Stilbene-420, obtained from ChemChek) in methanol. The dye solution is prepared in a 20 mL scintillation vial (the same ones used for sample and calibration standard preparation and storage). The dye solution in the instrument breaks down over time due to exposure to laser light. However, once prepared, the dye solution is more stable in the dark and in the refrigerator, as long as the methanol does not evaporate.



1) Locate all of the supplies needed:

- Laboratory gloves
- 20 mL scintillation vial
- Laboratory balance
- Methanol
- Stilbene-420 dye (1 g powder in a small vial, should be stored in the cabinet in a box)
- 5-mL adjustable pipette and transfer pipettes
- Weigh-boat or weigh-paper and small scoop. Aluminum foil will also work for both.

2) Fill the scintillation vial with 20 mL of methanol using the 5-mL adjustable pipette.

3) Place the weigh paper or small (2-inch x 2-inch) piece of aluminum foil on the analytical balance and tare/zero the balance.

4) Measure out precisely 19.1 mg laser dye powder onto the weigh paper/foil using a scoop or small shred of aluminum foil. Note: this will seem like a small mass of powder.

5) Transfer the powder into the methanol solution using the weigh paper/foil. Cap and label the vial (include today's date in the label), shake, and allow the powder to dissolve completely. It may take an hour or more for the powder to completely dissolve.

6) After replacing the dye in the instrument (procedure below), cap the vial tightly and store the remainder of the solution in the refrigerator. Consider wrapping the vial in aluminum foil to minimize light exposure.

**Replacing the Laser Dye Solution:**

1) Prepare fresh laser dye solution using the procedure above, or check to see if there is any previously-prepared laser dye solution in the refrigerator.

- If using previously-prepared and refrigerated dye solution, inspect the bottle to ensure that there was no spillage or evaporation and that the solution is clear (no precipitation of dye). Consider preparing fresh solution if the refrigerated solution is more than 1 year old.

2) Remove the KPA cover (four screws located on the bottom-side edges of the instrument)

3) Locate the Dye Module and remove the laser dye cell cap. You may need to push down and twist.

4) Slide the laser dye cell up and out of the module. NOTE the location of the label on the dye cell - the cell must be placed back in the same orientation. Note also that the dye cell slides in and out of the module at a slight angle - not straight up-and-down.

5) Inspect the dye cell for any precipitation in the solution. If a precipitate is present or if ethanol has evaporated, consider replacing the dye solution more frequently.



- 6) Remove the cap on the cell. Using a transfer pipette, remove the solution from the cell and dispose. Be careful to avoid touching the walls of the cell.
- 7) Rinse the inside of the cell with methanol using a transfer pipette and dispose of rinse solution.
- 8) Add fresh laser dye solution using a transfer pipette.
- 9) Place the cell back into the module. It should slide easily into the holder at a slight angle. Verify that the cell label is directed toward the arrow and the label on the Dye Module cover.
- 10) Place the black cap back onto the cell holder, and then reinstall the KPA cover.



## Attachment 4

### KPA Equipment Purchase List

Updated: 10/27/2015

Product	Detail	Vendor	Catalog Number	Unit	Cost	Qty	Total Cost
	= Not yet ordered, optional						
	= Not yet ordered, HIGHLY RECOMMENDED						
<b>KPA Consumables:</b>							
Uraplex stock solution		Chemchek Instruments	9110049	25 mL bottle	\$95.00	2	\$190.00
Laser dye powder		Chemchek Instruments	9110008	1 g powder	\$60.00	1	\$60.00
Concentrated uranium stock solution	500 mL bottle, 1,000 ppm U in 2% nitric acid	Inorganic Ventures	CGu1-5	500 mL			
Dilute uranium check standard	Five 100-mL bottles, 50 ppb U in 2% nitric acid	PerkinElmer	Custom Quote	5 x 100 mL	\$256.50	1	\$256.50
<b>der Periodically As-Needed):</b>							
	Type 3P Disposable Acrylic, 4 clear windows	NSG Precision Cells	3PMMMA	Box of 100	\$43.00	4	\$172.00
Scintillation vials	Borosilicate glass with attached cap (polyethylene liner)	VWR Scientific	66022-065	Case of 500	\$284.84	1	\$284.84
Kimwipes	11 cm x 21 cm	VWR Scientific	470173-504	box	\$4.65	10	\$46.50
Wash bottles	500 mL, green lid	VWR Scientific	16650-024	Pack of 6	\$47.88	1	\$47.88
Transfer pipettes	Extended fine tip, large bulb	VWR Scientific	414004-023	Pack of 250	\$39.58	1	\$39.58
Pipette tips	Finntip 250 Universal: 0.5 - 200 uL (10 x 96 rack)	VWR Scientific	53511-901	Case of 960	\$78.34	1	\$78.34
Pipette tips	Finntip 1000: 100 - 1000 uL (10 x 96 rack)	VWR Scientific	14232-914	Case of 960	\$82.40	1	\$82.40
Pipette tips	Finntip 5 mL: 1 - 5 mL (5 x 54 rack)	VWR Scientific	53515-980	Case of 270	\$96.77	1	\$96.77
<b>er):</b>							
	Type P66 Cell Holder	NSG Precision Cells		each	\$26.00	2	\$52.00
Bottle-Top Dispenser	VWR Labmax, 1-5 mL volume basic dispenser	VWR Scientific	40000-062	each	\$363.29	1	\$363.29
Glass reservoir	500 mL amber glass reservoir	VWR Scientific	53502-520	each	\$33.57	2	\$67.14
Hot Plate, Large	10" x 10", 120 V, 1500W, 12.5A, Ceramic top	VWR Scientific	97042-650	each	\$539.23	1	\$539.23
Scintillation vial block	Aluminum block for scintillation vial heating	Chemchek Instruments	P/N 9110008	each	\$495.00	1	\$495.00
Scintillation vial rack	Wheaton Vial Rack	VWR Scientific	66022-556	Case of 5	\$107.00	1	\$107.00
							<b>Total Cost</b>





*Kinetic Phosphorescence Analyzers for trace level detection of Uranium and Lanthanides*

1845 Terminal Dr. Suite 101 • Richland WA 99354 • USA • Tel: 509-943-5000 • Fax: 509-946-3368 • Website: [www.chemchek.com](http://www.chemchek.com)

Mr. Adrian Venable  
Homestake Mining Co.  
Hwy 605  
Grants NM 87020

October 22, 2015  
Phone: 505-287-4456  
Fax: 505-287-9289  
E-mail: [avenable@barrick.com](mailto:avenable@barrick.com)

### Quote # DS15-64P

ITEM	QUANTITY	DESCRIPTION	US\$ PRICE
1	1	P/N 9110008 Heat Block (64 vial capacity)	475.00
2	1	Handling charge \$20 per published price list 15 May 2015 required.	20.00.
TOTAL			\$495.00.

Delivery: 10 days ARO or ASAP

Shipping: UPS Ground Pre Pay and Add or on Customer's courier account.  
(Insurance coverage recommended)

Terms: Net 30 per published price list 15 May 2015. Checks or ACH. Credit cards not accepted.

**Note:** U.S. ECCN #3A999 (f) prohibits export or re-export to E country groups (under embargo or any country that supports terrorism) without U.S. Export License. U.S.D.O.C. deemed export license may be required if the use of this technology is by a citizen from country group E.

FOB: Richland, WA 99354

Quote valid: 60 days

Quoted by: Barbara Miller-Collins, President



PAGE 1 of 1

## Quotation

**PerkinElmer Health Sciences Inc.**  
710 Bridgeport Avenue  
Shelton, CT 06484-4794

**Phone: 1-800-762-4000**  
**Fax: 203-944-4904**

To: Adrian Venable  
Homestake Mining Co.  
Hwy 605  
GRANTS NM 87020

QUOTE NO.:	45001997
QUOTE VALID TO:	12/26/2015
QUOTE DATE:	10/26/2015
PAY. TERMS:	Due Upon Receipt
FREIGHT TERMS:	FOB Factory - Prepay & Add
ULTIMATE DEST.:	UNITED STATES OF AMERICA

TELEPHONE NO. 505-287-4456  
FAX NO. 505-287-9289  
YOUR REFERENCE

ITEM	MATERIAL	DESCRIPTION	QTY/EA	UNIT PRICE	TOTAL
1	SPECIALSTD	PEPLUS-251-500	1	290.00	290.00
		Material Discount			72.50-
		PERKINELMER PURE PLUS			
		50 UG/L U			
		MATRIX: 2% HN03			
		VOLUME: 500 ML (5 X 100 ML)			
		+/-5% ACCURACY, 6 MONTH EXPIRATION			
		Freight/Handling:			39.00
		Total Net Price in USD:			256.50
<p>Please direct orders to <a href="mailto:LAS.Customs@perkinelmer.com">LAS.Customs@perkinelmer.com</a> or fax 203-402-1912.</p> <p>CUSTOM STANDARDS MAY NOT BE CANCELED OR CHANGED ONCE THE ORDER HAS BEEN PROCESSED</p>					
<hr/> <p>MARGARET CLEARY</p>					

SEND PURCHASE ORDERS TO:

PerkinElmer Health Sciences, Inc.  
710 Bridgeport Ave.  
Shelton, CT 06484-4794  
Phone: 1-800-762-4000  
Fax: 203-944-4904  
Email: [LAS.Customs@perkinelmer.com](mailto:LAS.Customs@perkinelmer.com)

SALES REPRESENTATIVE: MARGARET CLEARY  
PREPARED BY: Natasha Harrison

**ACCEPTANCE OF THIS QUOTATION IS LIMITED TO THE ATTACHED TERMS AND CONDITIONS**  
**TERMS SUBJECT TO CREDIT APPROVAL**

**PerkinElmer Health Sciences, Inc.**  
**Terms and Conditions of Sale**

1. **PARTIES.** "Buyer" as used herein means the addressee of this quotation. "PERKINELMER" as used herein means PerkinElmer Health Sciences, Inc. Buyer and PERKINELMER are individually referred to as a "Party" and collectively referred to as "Parties".
2. **PRECURSOR.** Buyer is required to supply chemically pure compounds, purified biological material, and/or cell lines (hereinafter "precursor(s)") for the products to be generated by PERKINELMER pursuant to this quotation. In the event the precursor(s) is(are) destroyed in whole or in part during manufacture of the products by PERKINELMER, Buyer shall have no claims whatsoever, including, without limitation, claims for damages of any kind, against PERKINELMER or any of its employees or representatives as a result of such destruction. At Buyer's election, PERKINELMER will dispose of or return to Buyer, at Buyer's sole expense, all unused precursor(s) provided by Buyer when the work under this quotation is completed or otherwise terminated.
3. **CONFIDENTIALITY.** Without prior approval, each party will keep all information concerning this project made available to such party by the other party including the existence of this Agreement, as far as such information is not common knowledge, strictly confidential and will in no event provide the same to a third party other than its professional advisors, and then only subject to the same obligation of confidentiality as applies to the parties of this agreement. Further, without the prior written permission of a party ("Disclosing Party"), the other party ("Receiving Party") will not disclose any information pertaining to any service outlined in this Proposal unless such information (i) is already known to Receiving Party before Disclosing Party's entering into this Proposal, (ii) is or becomes publicly available through no fault of Receiving Party, (iii) is received from a third party which has the legal right to disclose it to Receiving Party (iv) is required by any law, rule, regulation, decision, order, subpoena or other process to be disclosed; or (v) is shown through sufficient evidence to be independently developed by the Receiving Party without reference to the information. If disclosure by subpoena, order, etc., is requested, Receiving Party will notify Disclosing Party promptly of such request.
4. **COMPLETION DATE.** The date for the completion of the products is estimated from the later of the date of receipt by PERKINELMER of (a) the purchase order, (b) the precursor(s), or (c) all information required to produce the product(s) from the precursor(s). This date is an estimate and subject to change. Whenever possible, PERKINELMER will provide Buyer with advance notice of such a change; however, PERKINELMER shall have the right to change the estimated completion date, and Buyer shall have no claims whatsoever, including, without limitation, claims for damages of any kind, against PERKINELMER or any of its employees or representatives as a result of such a change.
5. **CANCELLATION.** PERKINELMER may encounter unforeseen difficulties in the preparation and purification of products which conform to the specifications and terms quoted herein. In such event, PERKINELMER shall have the right to cancel this quotation, and Buyer shall have no claims whatsoever, including, without limitation, claims for damages of any kind, against PERKINELMER as a result of such a cancellation. Whenever possible, PERKINELMER will offer a modified quotation.
6. **PAYMENT.** Buyer shall remit payment to PERKINELMER within thirty (30) calendar days of the date of invoice.
7. **PERMITTED USE OF PRODUCT.** Buyer shall use the products and any derivatives thereof for research purposes only, and shall not use the products (or derivative thereof) for any other purposes, including, without limitation, for in vitro diagnostic purposes or in foods, drugs, biologics, medical devices or cosmetics, for humans or animals. Buyer shall not use, transfer, sell, or otherwise distribute the products or derivatives thereof to any third party, whether alone or in combination with other materials. Buyer also shall comply with any instructions or other limitations furnished by PERKINELMER relating to use of the products (or derivatives thereof) and not misuse the products (or derivatives thereof) in any manner.
8. **COMPLIANCE.** Without limiting the use restrictions of Section 7 above, Buyer covenants to PERKINELMER that Buyer shall test the precursor(s) and/or the products for any risks to health or the environment, comply with all applicable regulatory requirements, take appropriate precautions to protect persons and the environment from such risks; enforce appropriate methods of research to minimize such risks, implement safety assessments and procedures relative to the procurement, storage, use and disposal of the materials involved, and issue all appropriate warnings and information to users of the precursors(s) and/or products.
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10. **INDEMNIFICATION.** In case of any actual or alleged third party claim of infringement against Buyer or PERKINELMER and its affiliates in the performance of the work described in this quotation, Buyer will indemnify and hold harmless PERKINELMER and its affiliates, and their officers, directors, employees, agents and reimburse such parties for any expenses incurred in connection with such claim. The parties will immediately inform each other of any of such claims.
11. **ACCEPTANCE; MODIFICATION OF TERMS.** Buyer's acceptance of this quotation is expressly subject to Buyer's assent to each and all of the terms and conditions set forth herein, and Buyer's assent to these Terms and Conditions of Sale shall be conclusively presumed from Buyer's submission of its purchase order. No addition to or modification of said Terms and Conditions of Sale shall be binding upon PERKINELMER unless specifically agreed to by an authorized representative of PERKINELMER in writing. If Buyer's purchase order or other correspondence contains terms or conditions contrary to or in addition to the terms and conditions contained herein, acceptance of any order by PERKINELMER shall not be construed as assent to such contrary or additional terms and conditions or constitute a waiver by PERKINELMER of any of the terms and conditions contained herein. PERKINELMER's acceptance of Buyer's purchase order is expressly conditioned on Buyer's assent to these Terms and Conditions of Sale.
12. **INTERPRETATION.** These Terms and Conditions of Sale shall be governed by and construed in accordance with the laws of the Commonwealth of Massachusetts, without regard to the conflicts of law principles thereof.

## **Attachment 7**

### **Potential Consulting Contacts for Technical Support**

Shannon Ulrich  
630 Plaza Drive, Suite 100  
Highlands Ranch, CO 80129  
[shannon.ulrich@arcadis-us.com](mailto:shannon.ulrich@arcadis-us.com)  
303-471-3465

Michael Hay  
4999 Pearl East Circle, Suite 200  
Boulder, CO 80301  
[michael.hay@arcadis-us.com](mailto:michael.hay@arcadis-us.com)  
303-544-0043 x22

Jeff Gillow  
630 Plaza Drive, Suite 100  
Highlands Ranch, CO 80129  
[jeff.gillow@arcadis-us.com](mailto:jeff.gillow@arcadis-us.com)  
303-471-3446

If you run into difficulties while running the instrument, it may be useful to send a copy of the database and some screen-grab images to ARCADIS personnel to aid in troubleshooting.

The procedures for exporting the database are as follows:

- 10.1) Close all open windows within the main KPA Win window by clicking the X's in the upper right. Leave the main KPA Win window open.
- 10.2) Insert a USB key into the USB drive.
- 10.3) Click on "Database" → "Open". This will bring up a window called "Looking for KPA Database File".
- 10.4) In the list of files on the right-hand side, find the file with the most recent "Date modified" (likely today's date).
- 10.5) Right-click on the file, point to "Send to", locate the USB key in the list, and click on it. This will create a copy of the database file on the USB key.
- 10.6) Click "Cancel" on the "Looking for KPA Database File" window. Click the "OK" button on the "NO Database Assigned" window.
- 10.7) Email the database file that was copied to the USB key.

Use the following procedures for collecting screen grabs (an image of what is currently displayed on the monitor) using the following procedures:

- 10.8) With the KPA Win software displayed on the screen, press the "Print Screen" button in the upper right of the keyboard.
- 10.9) Open Microsoft PowerPoint, and paste the screen-grab image onto the slide ("Paste" is in the upper left).
- 10.10) Add additional PowerPoint slides for screen-grabs by clicking on "New Slide".
- 10.11) Save the PowerPoint file to the USB key and email it along with the database file to ARCADIS.

**Attachment 5**  
**KPA Instrument Operation Manual (Chemchek)**

**OPERATION  
MANUAL**

**MANUAL & AUTOMATIC  
KINETIC PHOSPHORESCENCE ANALYZER  
KPA-11**



**| 1845 Terminal Drive |**

**Richland, WA 99354 U.S.A.  
www.chemchek.com  
(509) 943-5000**

**Revised August 2006**

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# **Kinetic Phosphorescence Analyzer**

## **Model 11 (KPA-11)**

### **1.0 RECEIVING THE KPA**

#### **1.1 Introduction**

You have the opportunity to use the latest in electro-optic technology, data analysis, and patented technology in your analytical work as incorporated within the CHEMCHEK *Kinetic* Phosphorescence Analyzer. The KPA-11, a commercial kinetic phosphorimeter, offers new and unique features that help produce better results.

#### **1.2 Warrantee**

CHEMCHEK INSTRUMENTS, INC. warrants this instrument to be free from defects in materials and workmanship for a period of twelve (12) months from delivery. Should defects develop within this time, CHEMCHEK will, at its option, either repair or replace the product without charge. However, the laser plasma cartridge is warranted for  $2 \times 10^7$  pulses or one (1) year, whichever comes first. Replacement of the plasma cartridge is on a prorated basis.

CHEMCHEK INSTRUMENTS will also indemnify our customers against infringement resulting from our activities or resulting from our product as provided. Specifically, CHEMCHEK shall retain the options of procuring appropriate rights for its customer in order to avoid infringement, modifying its product to avoid the infringement, or requesting the return of the product to CHEMCHEK for full refund of purchase price and shall have no other liability.

#### **1.3 Liability Disclaimer**

Liability to CHEMCHEK INSTRUMENTS, INC. for any defect in this instrument is limited to the invoice price of the defective instrument. CHEMCHEK INSTRUMENTS, INC. has no control over the set-up, use, connection to other equipment, or data generated by means of CHEMCHEK products. Therefore, in no event shall CHEMCHEK INSTRUMENTS, INC. be liable for any incidental or consequential damages, losses, or liability which may result from improper use of its products, either in connection with other equipment or in the generation, reporting, or application of data and results.

#### **1.4 Receiving The KPA-11**

The KPA-11, phosphorescence cells, software, laser dye, analyte complexant, power and serial cables, and this manual comprise the KPA system. A computer capable of running Windows 95/98/NT/2000/XP is required. Either Chemchek or the customer provides the computer and any necessary peripherals.

##### **1.4.1 Unpacking**

Unpack the equipment immediately after receiving and check for evidence of damage. Remove the cover of the KPA and check inside for concealed damage, which may show up only during actual operation.

The cells are shipped in a Styrofoam box. The laser dye and URAPLEX™ are packaged inside plastic bags. The laser dye should be stored in a dark, dry location and the URAPLEX™ requires immediate refrigeration upon arrival.

#### **1.4.2 Damage Notification**

The carrier making delivery should be notified immediately of any detected damage, and is normally liable for damage in shipment. Packing materials, shipping papers, etc., should be retained in order to establish claims. After notification of the carrier, please inform CHEMCHEK of the circumstances so that assistance in making damage claims and in providing replacement equipment, if necessary, may be provided.

#### **1.5 Repair Service**

If it becomes necessary for instrument repair, please contact CHEMCHEK INSTRUMENTS. The repair may be most expeditiously accomplished by returning either the part or the complete unit for repair or replacement. With first-day delivery transportation, a repair can be completed within a week. First, obtain a Return Authorization Number (RA#) and communicate the nature of the fault, either in writing or telephone, and the model and serial number. Returned instruments must be packed to withstand normal transit handling and shipped PREPAID via air. The address label should include the RA# assigned. Returned instruments that are damaged in transit due to improper packing are repaired at the sender's expense, and the sender is responsible to make damage claims with the carrier. Instruments not in warranty will be repaired at the standard rates. In unusual warranty repair cases, such as misuse, the sender will be notified prior to the repair and given a repair quotation.

##### **1.5.1 Packaging The KPA For Return**

To avoid damage and consequent repair charges, please observe the following:

1. Remove the cells from the sample compartment and retain.
2. Remove and empty the dye cell. Place the cell in its box or another protective container.
3. Either place the KPA in a plastic bag or wrap it in plastic.
4. Package the KPA in a box having at least 5 cm (2 inches) of packing material on all sides. Seal the box and ship by air.

NOTE: Ideally, save the original KPA box and packing materials. Manual KPA-11s are now shipped inside a case to be used for return.

## **2.0 KPA SPECIFICATIONS AND SET-UP**

### **2.1 Specification For The KPA-11**

Kinetic Phosphorescence Analyzer, model 11. Computerized kinetic phosphorimeter.

#### **Illumination Source**

Pulsed Laser	nitrogen pumped dye laser
Peak Output Power	5.0 kW
Average Power	0.5 mW
Pulse Length	3 ns
Pulse Energy	20 J
Repetition Rate	1-20 pps.
Nitrogen Laser Wavelength	337 nm
Dye Laser Range	360-900 nm

#### **Detection System**

Interference Filters	10 nm Bandpass
Photomultiplier Tubes	300-650 nm
Mode Of Operation Of PMTs	photon counting
Sensitivity Range	high, low - each 10,000

#### **Additional Specifications**

Power Source	115/220 VAC, 50-60 Hz
	200 W
Data Communication Port	RS232
Weight	14 kg

#### **Standard Accessories Included**

Enhancing Reagent	URAPLEX™ for uranium
	EDTA for lanthanide elements
Cuvette	2, Low Fluorescence
Laser Dye	1 g

#### **Additional Accessories Available**

PERSONAL COMPUTER (IBM compatible)

AUTOMATIC SAMPLE CHANGER: automatic analysis of 42 or 108 samples. Samples are loaded in sample vials suitable for sample preparation. Sample and enhancing reagent are automatically mixed before each measurement. Cell flushing and decontamination are automatic and complete, with a sample-to-sample decontamination of about 1000.

REMOTE SAMPLE CHAMBER: for analysis in hostile environment, with limited space availability in the hood.

## **2.2 Description Of Components**

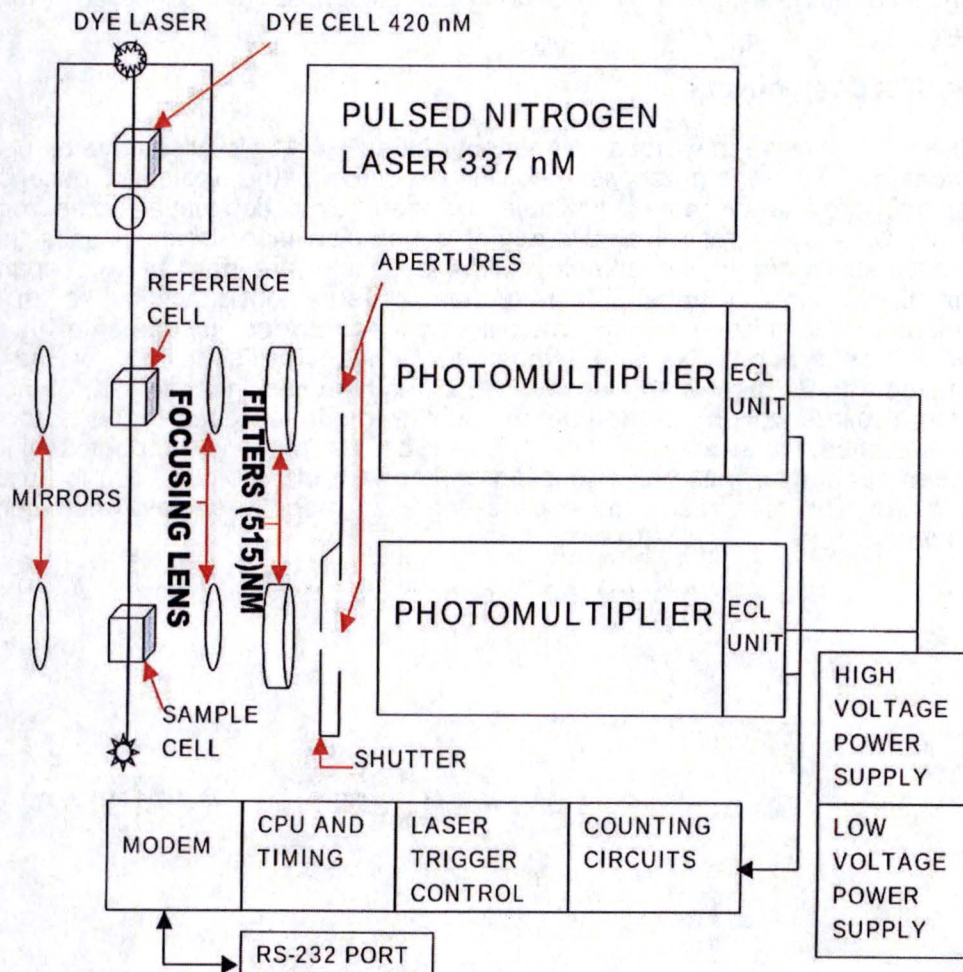
The KPA-11 is a bench-top instrument that rapidly performs single-measurement, quench corrected analysis by measuring sample phosphorescence as a function of time.

### **2.2.1 KPA Components**

Figure 2.2.1 shows the various component of the KPA-11. A pulsed dye nitrogen laser provides the wavelength necessary for the excitation of the photoluminescence of the sample. The dye laser makes available a range of excitation wavelengths from 360 to 900 nm with high spectral and temporal purity. The laser beam passes through a reference cell prior to the sample cell. These cells are part of two separate and identical detection systems. The reference measurement functions as an external standard in normalizing sample measurements to correct instrumental fluctuations such as laser brightness and temperature drifts. The sensitivity of the KPA is optimized with  $f/1$  mirrors and lenses, as well as high performance interference filters. Photomultipliers working in the photon counting mode are used to detect the sample and reference luminescence. The KPA-11 counts photons of photoluminescence between each laser pulse as a function of time with the choice of two to hundreds of time gates. The time resolution can be varied in order to achieve multi-component resolution.



**Figure 1. Diagram of the Kinetic Phosphorescence Analyzer  
(Uranium Configuration)**





## **2.3 INSTRUMENT SET-UP**

### **2.3.1 Location**

The KPA-11 was designed to permit module stacking to conserve bench space. A 19 x 21 inch space, preferably near a sink, is adequate for the KPA. Space is also necessary for a computer (adjacent or remote) and peripherals. A computer terminal or a printer, for example, may be placed on top of the KPA-11.

The location should have clean temperature controlled air and not be near a source of organic fumes, such as stored urine samples.

### **2.3.2 Connections**

The computer is connected to the KPA-11 by means of the serial cable. Connect the 25-pin connector end of the serial cable to the KPA and the 9-pin connector to the computer serial port. When multiple serial ports are present, select the operating port for KPA using the KPA software. (See KPAWin User's Guide)

The KPA-11 automatically operate from either 90-132 or 175-264 VAC within the range of 47-63 Hertz. No switching is necessary.

### **2.3.3 Laser Dye Cell**

Remove the quartz dye cell (cell with screw cap) from its box and clean carefully, using good optical technique. Let cell dry.

**NOTE: HANDLE THE CELL CAREFULLY TO AVOID BREAKAGE. IT IS AN OPTICAL ELEMENT OF THE SYSTEM. A REPLACEMENT CELL MAY PRODUCE HIGHER BACKGROUND, LOWER SENSITIVITY AND REQUIRE DYE MODULE REALIGNMENT.**

Using a Pasteur pipette, fill the dye cell with methanol solution of stilbene-420 (23.9 mg of stilbene-420 in 25 mL methanol) or your other choice of dye solution. Screw on the cap.

Remove the cover of the KPA. In the left front corner is the dye module, which has a cover with a laser warning sign and a black cap over the dye cell holder. Always remove the cap with a turning-twisting motion. The cell should smoothly slide into the holder at a 5° angle slanted toward the left side of the KPA. **DO NOT FORCE!** The cell label must be directed toward the arrow and the label on the dye module cover.

**NOTE: BE SURE THE CELL IS IN ITS TRACKS.**

Replace the black cap and KPA cover. The dye solution is long lasting because of the low laser power. Dye replacement should be every 2 to 3 months, when it evaporates, or when it develops a precipitate from decomposition.



### **2.3.4 Tunable Dye Module (Optional Equipment)**

A grating, which is rotated by means of a micrometer control, is used in place of the rear mirror in the dye module to permit infinite excitation wavelength tuning throughout the range of a particular dye. Use of the micrometer setting of 0 nm is the broadband mode and yields laser power similar to the peak when wavelength selection is used. The 0 nm setting is also used when the dye module is being tuned. The beam at the aperture may be viewed by following the instructions in Section 8.1. If the best beam is obtained at a setting somewhat different than 0, this difference may be used to correct nm readings throughout the range of use. See the following pages.

Most dyes produce adequate intensities over about 40 nm. See the dye vendor for detailed information. Stilbene-420, which is frequently used in the KPA-11, has an intensity peak near 425 nm. Intensities may be measured as a function of the excitation wavelength (dye module micrometer setting) by taking repeated "background" measurements with an appropriate concentration of analyte in the sample cell. Operation of the dye module is by turning the micrometer to the desired wavelength, which is indicated on the dial read-out. The read-out is in micrometers, or, if the decimal point is ignored, nanometers.

The micrometer wavelength setting is locked by means of the ring just above the digital read-out. Turn the ring counter-clockwise to loosen before changing the wavelength setting. Turn it clockwise to lock the micrometer setting at a wavelength selection.

### **2.3.5 Emission Filters**

The light emitted from the substance being determined is filtered through interference filters, so that the photomultiplier tubes will detect only the wavelength bandpass that provides the best signal to noise ratio.

The KPA is normally provided with high performance filters having a bandpass of 10 nm, which are in an optics holder at the rear of the sample chamber. The optics holder is friction mounted; its adjustment screw is in the center of the sample chamber cover. To remove the optics holder, reach into the sample chamber and grasp the knob, which is above and between the lenses. The optics holder should snap out at its top. To replace it, carefully position the bottom of the holder on the pointed guide pins before snapping it into place. A second optics holder simplifies frequent conversions of the KPA between analytes.

Emission filters are normally retained in the holder by internal snap rings. Removal of the rings permits switching filters in the optics holder.

**NOTE: FILTERS ARE OPTICAL ELEMENTS; handle and clean them accordingly.**



## 2.4 Applications: Methods and KPA Set-Up

Method				
Analyte	Laser Dye	Complexant	Buffer	Detection Limit (µg/L)
Dysprosium	BPBD	EDTA	Acetate	0.2
Europium	PBBO	EDTA	Acetate	0.05
Samarium	PBBO	EDTA	Acetate	2
Terbium	BPBD	EDTA	Acetate	0.01
Thulium	BPBD	EDTA	Acetate	1
Uranium	Stilbene-420	URAPLEX™	--	0.01

Spectroscopy					
Analyte	Wavelength (nm):		Signal Timing:		Lifetime (µs)
	Excitation	Emission	StartGate	Duration (µs)	
Dysprosium	402	580	4	13	20
Europium	398	620	4	13	350
Samarium	405	589	2	2	8
Terbium	375	550	4	13	1200
Thulium	362	450	2	2	18
Uranium	425	515	4	13	200-300

### Analytical Range

Over 1,000,000 for uranium (from 0.01 to 50,000 ppb).



## 3.0 LASER SPECIFICATIONS AND SAFETY

### 3.1 Description

The KPA-11 meets all requirements of 21 CFR 1040.10 for a Class I laser product in that there is no laser radiation accessible by the operator. The laser incorporated within the KPA-11 is a Class III-b, pulsed nitrogen ion laser (337 nm), which pumps a dye to produce a beam. This beam is totally enclosed and inaccessible except during service.

### 3.2 Active Laser Warnings

The laser emits an audible warning, a "clicking" sound, which warns when the laser is on and pulsing. In addition, a warning light on the front panel of the KPA changes from green to red whenever the laser is on and pulsing or enabled so it can come on.

### 3.3 Laser Specifications

Parameter	Nitrogen	Dye
Maximum Power Output (mW)	2.0	0.5
Peak Output Power (kW)	40	5.0
Pulse Length (ns)	3	3
Pulse Energy (μJ)	120	20
Repetition Rate (pps)	1-20	1-20
Wavelength (nm)	337	400-700
Warranted Lifetime of Plasma Chamber (pulses) (years)	2 x 10 <sup>7</sup> 1	
Beam Dimensions	3 x 8 mm	
Laser Head Power Requirements	12 VDC @ 2A	



### 3.4 Interlocks

The KPA-11 has several interlocks to protect personnel and also the sensitive photomultiplier tubes (PMTs) from excessive light levels (see Figures 1 and 2). The KPA interlocks utilize infrared optorelector sensors. These emit infrared light toward a protective housing, which reflects it back to a sensor, a part of the device. They not only interrupt normal operation when a protective housing is not in place, but they are checked by the internal computer at the beginning of each measurement to ensure that any detected light is reflected by the housing rather than from an external source. The interlocks are described below.

- a. Primary Door Interlock. The door is interlocked to prevent laser and PMT high voltage actuation when the door is not latched. The optorelector sensor is pointed upward toward the door latch, which is rotated to horizontal in latching. The interlock is actuated to prevent laser and PMT operation when the KPA door is unlatched. Actuation is intended during operation, maintenance, and service.
- b. Secondary Door Interlock. The door is interlocked to prevent laser and PMT high voltage operation when the door is open. The optorelector sensor is mounted on the front panel and behind the door, where it is actuated by the door opening and closing during operation, maintenance, and service.
- c. Cover Interlock. Maintenance and/or service personnel must be protected from accidental exposure to laser radiation when protective housings are removed. The optorelector sensor is mounted on the inside top of the front panel, where it is directed at the KPA cover. It prevents activating the laser or PMT high voltage. Actuation is during maintenance and service, when the protective housing (KPA cover) is not in place.
- d. Other protective housings exposed by removal of the KPA cover are exclusively over the laser, over the dye or 337 nm modules, and the beam trap, which eliminates the used laser beam. The O.E.M. laser cover is interlocked with microswitches. Actuation interrupts power to the laser.

Computer Interlock. The computer controls all instrumental operations, thereby fulfilling a pseudo laser interlock role. The laser cannot be operated except through deliberate use of the KPA computer program. Even then, the laser will only operate for a preset maximum time, usually 50 seconds. The computer interlock is actuated during normal operation and prevents laser operation except through program commands.



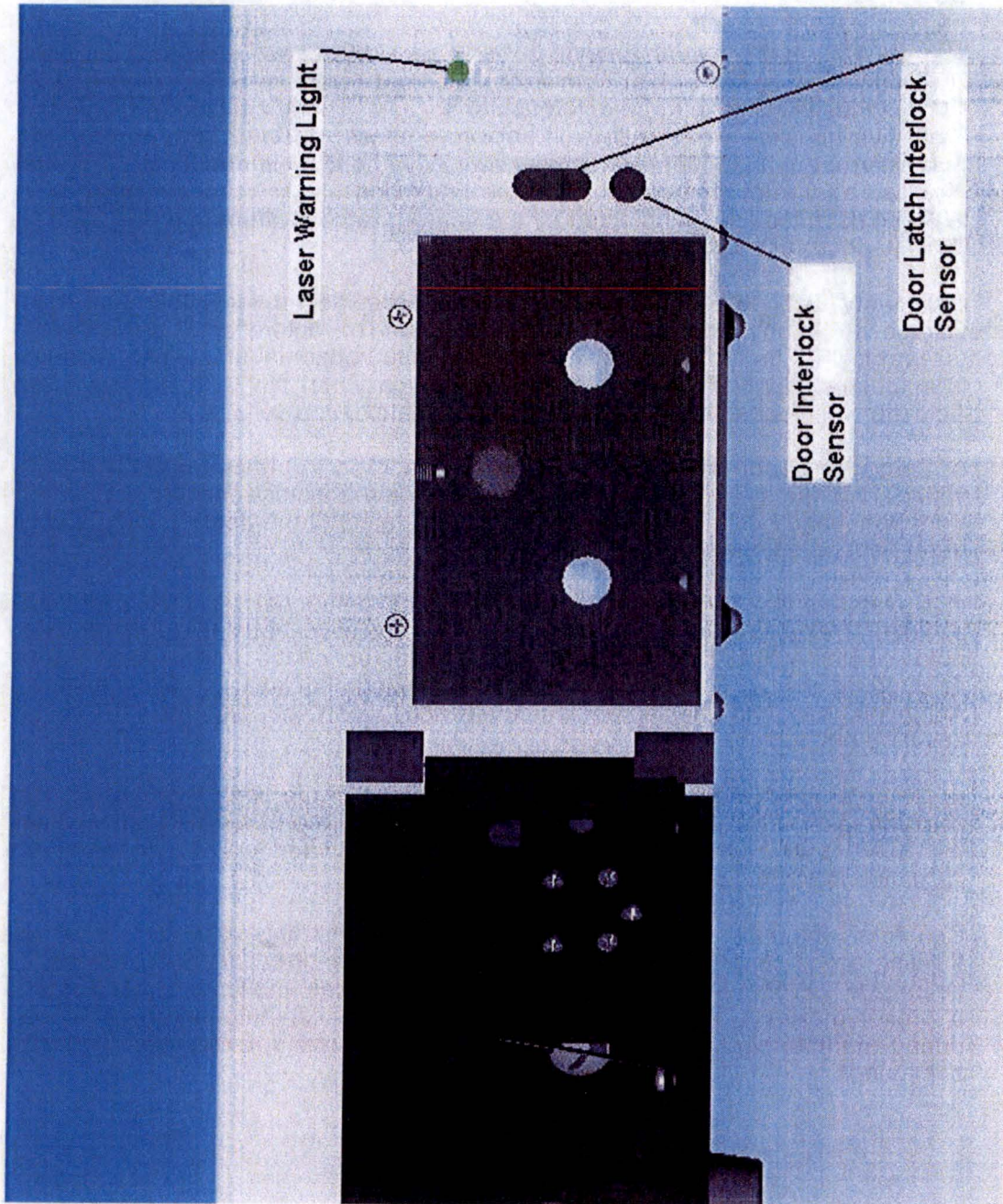
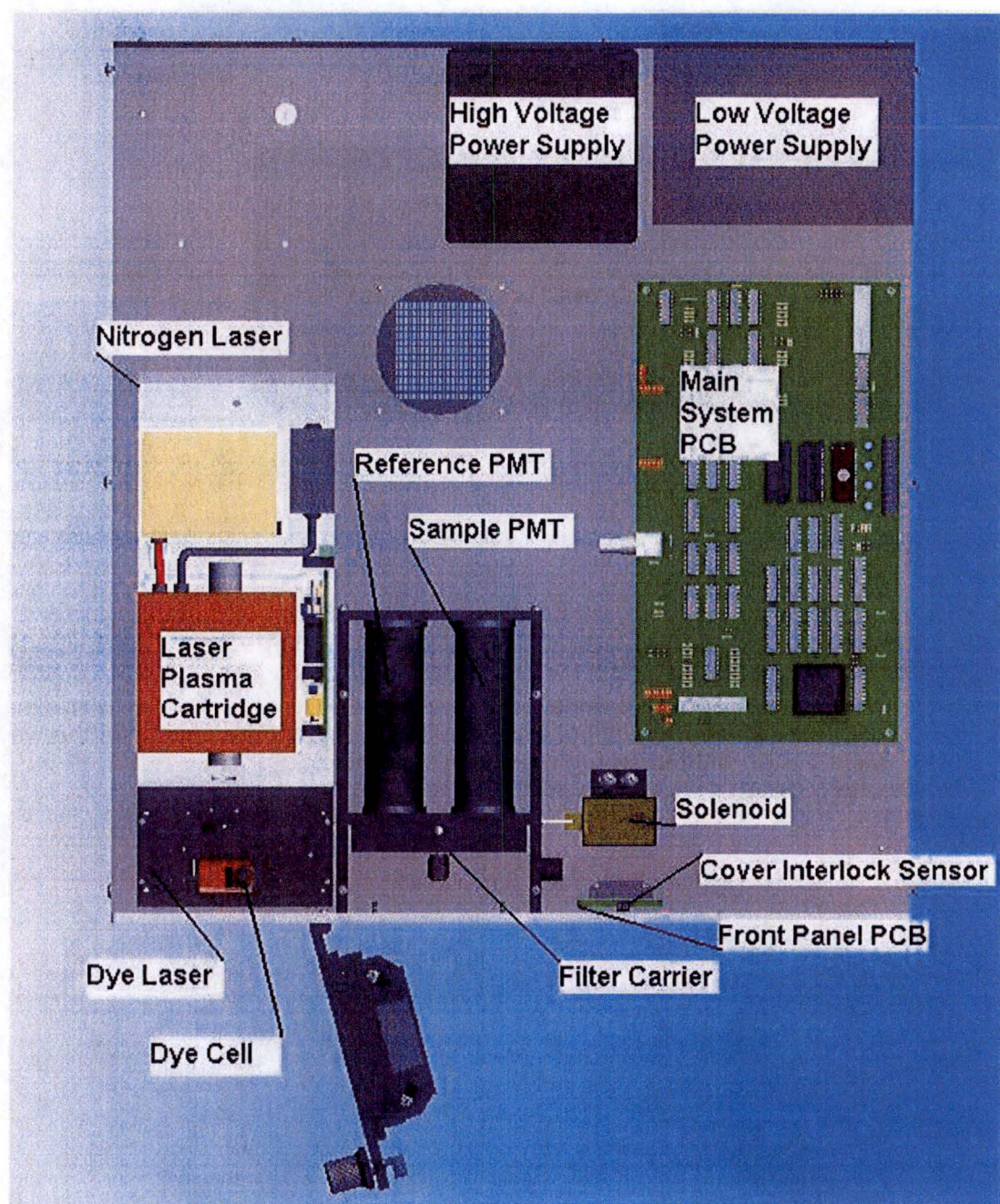


Figure 2. Exterior View of Laser Interlocks and Warning Light



Figure 3. Interior View of Cover Interlock Sensor





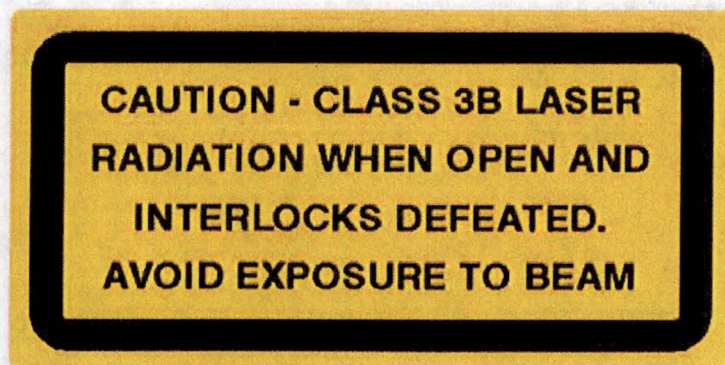
### 3.5 Laser Product Labels

In conforming to 21 CFR 1040.10, the KPA has the following labels:

- a. **Certification and Identification labels.** These are combined and located on the rear panel.



- b. **Protective Housing Labels.** Removal of the KPA cover may permit exposure to Class IIIb laser radiation. Therefore, each side of the cover has the warning label shown below. A protective housing label can also be found on the laser cover, dye module cover, and the black beam trap.



## 4.0 PRINCIPLE OF OPERATION

### 4.1 Theory

Nearly all molecules scatter or emit light after having been struck by a photon. Fluorescence involves the absorption of a photon, which electronically excites the molecule. Return to ground state occurs by relaxation of the intermediate singlet electronic state with the emission of a photon. The relaxation lifetime typically is of the order of nanoseconds. Phosphorescence involves a similar process, except the relaxation incorporates a triplet electronic state. The subsequent return to ground state with emission of a photon is "forbidden", so the relaxation lifetimes range from microseconds to hours. For more detail, the reader should refer to one of many texts on the subject.

With a pulsed excitation source, phosphorescence emissions may be observed in the absence of other light so that measurement conditions are optimum. Pulsed lasers (20 pulses/second maximum in the KPA-11) provide the best temporal resolution for this purpose. In *kinetic phosphorimetry*<sup>(a)</sup>, the pulse of source light is assigned an occurrence time of zero. Intensity measurements are taken at fixed intervals in time, called time gates, following the pulse. Intensity values from these intervals are summed over the number of pulses used in each measurement. In this way, excellent statistics and repeatability can be obtained.

The first order kinetic decay of an excited phosphor, for example uranyl ions, can be represented by the equation:

$$(4.1) \quad \ln U_i = \ln U_0 - (k_p + k_q)t \quad \text{where,}$$

$U_i$  = population of excited uranium ions at time  $i = t$  or  $0$ ,

$k_p$  = rate constant for phosphorescent decay, and

$k_q$  = rate constant for all other relaxation processes.

The number of detected photons at any time,  $t$ , is proportional to the number of excited uranyl ions. As a result, the least squares fitting of the logarithm of detected intensity versus time-after-pulse yields an intercept proportional to the number of excited uranyl ions. This intercept is independent of nonradiative effects such as quenching. The decay lifetime,  $\tau$ , is defined as the reciprocal of  $k_p + k_q$ , which is the time required for the phosphorescent intensity to decrease by the factor  $1/e$ .

Nonradiative relaxation processes generally involve collisional interactions and transfer of energy. These processes must be minimized by stabilizing or otherwise protecting the molecules of interest. Cryogenic temperatures, conversion to solid state, formation of micelles, and complexation are effective methods. Complexation of the uranyl ion with URAPLEX<sup>TM(b)</sup>, at room temperature, is utilized for uranium. Lifetimes as long as 340 microseconds ( $\mu s$ ) are observed under optimum conditions. Lifetimes greater than 100  $\mu s$  are required for best measurements. In practice, the instrumental plus reagent background must be recorded and subtracted from the measured sample intensities, because background luminescence may be significant and does not follow first order decay kinetics.

(a) B.A. Bushaw, Progress in Analytical Spectroscopy, Proceedings of the 26th Oak Ridge Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, October 1983.

(b) URAPLEX™ is a proprietary complexing agent for uranium. URAPLEX™ may be obtained from: CHEMCHEK INSTRUMENTS, INC., 1845 Terminal Dr., Richland, WA 99354

## 4.2 Principles of KPA Hardware and Operation

The KPA operates on emission and detection of light in the visible spectrum as a function of time. In particular the KPA utilizes equation 4.1 in section 4.1. This equation simply states that after excitation of the sample, the natural logarithm of the light emitted by the sample will decay linearly with time from its starting value, which is when the population of excited analyte molecules is at a mathematical maximum. This is at time  $t = 0$ , when the exciting light is terminated. The intensity of light emitted from the excited analyte molecules at any time is directly related to the concentration of the analyte and the intensity of excitation for that particular sample. Assuming the excitation process is reproducible, then concentrations can be determined simply by measuring emitted light intensity, either steady state during the time of excitation or later, after a delay time. Unfortunately, short lived luminescence, varying relaxing processes and excited state lifetimes, stray excitation light, and other uncontrollable variables make it extremely difficult to accurately quantify the analyte of interest by emission intensity.

However, using the decay equation 4.1, the amount of light emitted at  $t = 0$  can be accurately determined by observing the light emitted over a period of time after the unwanted light sources have decayed. Equation 4.1 describes the rate of decay of a population of electronically excited analyte molecules. Since intensity,  $I$ , of emitted light is proportional to concentration, we can substitute intensity for concentration in equation 4.1 and obtain:

$$(4.2) \quad \ln I_0 = \ln I_t + (k_p + k_q)t$$

This equation can be used for sample analysis if we provide:

- a. A means of excitation of the sample.
- b. A method of measuring the emitted light.
- c. A method of correlating light intensity to time.
- d. A method of relating light intensity to concentration of the analyte.

In the KPA these are accomplished as follows:

- a. Excitation of the sample is provided by a pulsed nitrogen dye laser. Laser wavelength and power are dependent on the laser dye and tuning. Average power is in the 0.1 - 0.5 mWatt range. Pulse duration is 3 ns at a frequency of 20 pulses/second or less.
- b. Light intensity is measured with a photomultiplier tube, which is connected to a photon amplification and discriminating circuit. This combination yields an electronic signal whose frequency is directly proportional to the intensity of

detected light.

In the KPA, the electronic signal just described is fed into a counting circuit, which is read and reset by the microprocessor. Each successive reading of the counter occurs after a constant interval and forms a time gate. The individual contents of the counter are stored in consecutive memory locations in the on-board microcontroller. In other words, this circuitry provides a series of numbers in memory, which correspond to emitted light displaced in time. The chosen increment of time is the duration of the time gates. The number of time gates is predetermined in the KPA. The result is emitted light intensity as a function of time. For uranium, 128 time gates, each 13  $\mu$ s long, are read for every laser pulse.

Since the photomultiplier tubes of the KPA produce statistical signals, the precision of measurement can be increased by increasing the number of observations. With the KPA, reasonable results can be obtained with as few as 200 laser pulses. The software is defaulted at 1000 pulses per measurement for better precision.

- c. Time is measured by clock cycles in a microprocessor circuit to establish the time gates. Intensity measurements begin with the exciting laser pulse, which defines time zero,  $t = 0$ .

The light intensities for each time gate are summed over all laser pulses to form an intensity sum for each time gate. These values are stored in memory. Their logarithms become the dependent variable in the subsequent least squares regression, where time is the independent variable. The KPA, with the aid of a digital computer, utilizes this method to find the best linear fit of the data and obtain the slope of the decay line and its intercept, where  $t = 0$ .

For uranium, the KPA program utilizes the intensities taken from time gates 5 through 49 in the least squares calculation. By delaying  $\sim 60 \mu$ s until time gate 5, intensities from most short-lived luminescence sources are eliminated from the calculations. Forty-five values provide a considerable amount of data for calculating precise results without overly long data processing time. In addition, intensities over this period are generally well above noise levels except near the KPA detection limits.

- d. The analyte luminescence is related to the concentration of analyte in the KPA sample cell by an empirical calibration procedure. During calibration, the emission intensities from a standard solution of known concentration are measured and stored. The intercept of the data at  $t = 0$  is calculated according to equation 4.2. That value is the quantitative parameter used to establish the calibration equation relating signal to concentration, and to calculate analyte concentrations during sample measurements.
- e. The method just described is nominally sufficient to determine the concentration of analyte in samples. The KPA corrects for several other variables in order to improve accuracy and precision of the results. These are as follows:
  - 1. Instrumental Background. This is a combination of low-level signals produced by sources other than the analyte, including: photomultiplier tube dark count (electronic noise), luminescence from the quartz cells and



optics, impurities in the reagent, and stray ambient light. These sources become particularly important at the lower detection levels of the KPA. Since they will contribute signal to all measurements, they are classified as background.

The instrumental background is measured during calibration and stored for subtraction from the intensities obtained in subsequent measurements. The background is determined with a sample cell filled with BLANK, which is deionized or distilled water and reagent to simulate a measurement on an analyte-free sample.

2. Reference System. Optical instruments are sensitive devices that are affected by various sources of instability. Since such signal deviations cause variable results, the KPA includes a second photomultiplier and electronic amplification and discrimination system, which is identical to the sample system. This is called the REFERENCE channel.

A second, reference cell containing the uranium reference solution (see page 24) is placed in the laser beam just prior to the sample cell, in front of its respective optics and detection channel. Detected luminescence is processed in an identical fashion to the sample measurements except that there is no background correction. Background is insignificant because a pinhole aperture is used. The calculated intercept for the reference measurement is used to normalize the corresponding sample data as described in the next subsection. The result is a type of external standardization that eliminates or reduces the following sources of error, which are listed below in order of importance and associated mode of correction:

Laser power:

Both cells have common laser beam.

Ambient temperature change:  
intercept.

Lifetimes are affected, but not the

Electrical line surges, and  
high voltage drift.

Common electrical sources.

- f. Limitations in Detection Electronics. Photomultiplier tubes and electronic circuitry have an upper limit to their frequency response. Since photomultipliers exposed to light emit pulses which are not periodic with time, some pulses will be spaced very close to the previous pulse, others will be further apart. Pulses that occur before the electronic circuitry has recovered from the previous event will not be counted. The result is a decrease in detection efficiency at high counting rates and a nonlinear response. The KPA avoids using data in the nonlinear region by actively monitoring the detected signal. Intensity values are not used in calculations when they exceed a preset level set in the *Settings* → *General Configuration* → *Performance Limits* section of the program.

During measurements at "high" concentrations, the set group of time gates (defaulted at 45) comprise a time window, which will shift out in time until a lower intensity within the linear response range of the KPA is reached. The default time gates 5-49 may shift to, for example, 80-124 at a very high level of analyte. The extrapolation becomes too long to be accurate, but the result is still semi-quantitative. Calculation of concentration is terminated when the time

window passes beyond the range of available time gates (see also Section 4.3.3, 3).

- g. **Analytical Range.** The linear response range of electronics and photomultiplier tubes limits the range of concentrations that can be determined with optical instruments. In the KPA, the range has been substantially extended by using apertures to limit signal light. The result is two ranges:

**Low Range:** 0.625 inch aperture. From detection limit to within nonlinear region of detection system where time gate shifting occurs (see Section 4.3.3, 3). The concentration where gate shifting starts to occur varies from instrument to instrument. For uranium, a concentration around 10-15  $\mu\text{g/L}$  most commonly represents the upper limit of the linear interval in the low range. Concentrations higher than 20  $\mu\text{g/L}$  may cause gate shifting and should be analyzed in the HIGH range.

**High Range:** 0.02 inch aperture. From within low range to nonlinear region of detection system where time gate shifting occurs in high range (see also Section 4.3.3, 3). The concentration where gate shifting will start to occur varies from instrument to instrument. For uranium, a concentration around 5 mg/L most commonly represents the upper limit of the linear interval in the high range. Concentrations higher than 5 mg/L may cause gate shifting, and should be analyzed after proper dilution. The 0.02 inch aperture is in place except during a low range measurement.

- h. **PMT Protection.** The photomultiplier tubes are protected from excessive light levels by turning off the high voltage and closing a shutter. Each light detection system has a pin hole for transmission of filtered emitted light to the photomultiplier tubes. Since the high voltage is off except during a measurement, the small amount of light transmitted when the door is open is not detrimental. On the sample side, the pinhole is in a movable shutter. This shutter opens for each low range measurement to make the 0.625 inch aperture available for much greater sensitivity.

## 4.3 Calculations

The following explanation of KPA calculations is not detailed, and is only intended to aid you in understanding the various aspects of the data treatment.

### 4.3.1 Principles

Since for photoluminescence the logarithm of intensity is a linear function of time (see equation 4.2), each quantitative array of data for the preset number of time gates (default gates are 5-49) undergoes a linear least squares regression (LSR). The result yields the slope, which is rate of decay, and the intensity intercept, which is at zero time. The intercept is a fixed, reproducible quantity for a given concentration of luminophor. The slope is  $-1/\text{lifetime}$ , therefore it is characteristic of the luminophor and varies with its chemical environment and with temperature.

The function of the REFERENCE measurement, which is part of each set of KPA data, is to provide the means to adjust all sample data to a fixed reference point (the REFERENCE MARK) for the purpose of removing instrumental deviations, primarily due

to laser power fluctuations. LSR is applied to the reference intensities. The resulting intercept, at  $t = 0$ , is the factor used for normalization of all data.

The first measurement in each range (i.e. the background measurement) yields the REFERENCE MARK, the intercept to which all other measurements are normalized. The Reference Ratio for this measurement is defined as 1.00. All subsequent measurements have their sample data adjusted (normalized) to the reference mark. This is done as described in the following section, under 2.

#### 4.3.2 Algorithm

Calculations are described in operational order.

1. The instrumental BACKGROUND is measured and stored. LSR is performed over set time gates of reference data to obtain lifetime and intercept (RI). RI becomes the REFERENCE MARK, RM.
2. Calibration Standards are measured and the intensities are normalized to the RM. All subsequent measured intensities are normalized by:

$$(4.3) \quad (RM/RI) I_s = I_{s_i}$$

where  $I_s$  is luminescence intensity at each time gate, and  $I_{s_i}$  represents normalized intensities.

Stored background is subtracted, gate intensity by gate intensity, from the normalized standard intensities to obtain net intensities. LSR is performed on standard net intensities to obtain lifetime ( $\tau$ ), correlation coefficient ( $R^2$ ), and intercept (SI) in counts per unit of concentration.

The calibration equation is produced by LSR of  $SI_i$ , the intercept values for the calibration standard(s) and their concentrations. The program offers the choices of:

$$(4.4) \text{ Linear fit of the form } SI = aC + b$$

$$(4.5) \text{ Quadratic fit, } SI = aC^2 + bC + c, \text{ and}$$

$$(4.6) \text{ Linear fit with forced zero intercept } SI = aC,$$

where  $C$  is concentration and the constants  $a$ ,  $b$ , and  $c$  are determined by the LSR.

Sample data is reduced by a process identical to the standards. Quantification for concentration  $C$  is by entering the SI from the sample measurement into the calibration equation.

#### 4.3.3 Nuances

The following features simplify operation and aid in producing quality analytical results.

1. Calibration: Up to seven calibration points may be established in each range of the KPA. The linear fit with zero intercept should be used when analyses will be performed near the detection limit of the KPA. At least five calibration concentrations should be used to describe a quadratic (curved) fit.
2. Each range (High or Low) is entirely independent of the other range, although both are handled in identical manner. Low Range is highly linear, while High Range is also linear, but may be curved near the upper end. When calibrating both ranges, choose calibration concentrations to provide overlap between the ranges.
3. Maximum count rate: The electronics provide linear response with intensity up to about 40 megaHertz. Measurements are protected from errors by excluding higher count rates from the calculations.

At higher concentrations where intensities exceed the limit, the first time gate for LSR is shifted out in time to where the intensities fall within the limits of the electronics. This is usually referred to as "gate shifting". Gate shifting automatically increases the delay time and extends the analytical range, while keeping the data within electronic linearity. The longer time extrapolation reduces accuracy and is only approximate for delays longer than twice the time window (the time interval of intensities used in the LSR). No gate shifting should occur in calibration. When calibrating the KPA, **the concentration of the standard solutions should be well within the linear interval for the particular range selected.** See Section 4.2, f and g. Gate shifting is detected because the time gates indicated in the top line of the display header are not the usual 5-49.

4. Net Data Threshold: Since luminescence intensities decrease in time, they may reach the noise level where they will be detrimental to the measurement by skewing the LSR fit. Thresholds or minimum intensities have been established for which sample net intensities must be greater in order to be included in LSR. Minimum intensities that have been established are:

Low Range: Square root of number of laser pulses

High Range: Third root of number of laser pulses.

The minimum intensity threshold can be software adjusted with the minimum intensity factor available in *Settings → General Configuration → KPA Unit Settings*. When the minimum intensity threshold is exercised, the number of intensities used in LSR is reduced. This normally only occurs near the detection limit. The time gates used are indicated in the top line of the display header.

5. Non-Detectable Concentration: The first five time gates must have net intensities above the threshold in order to obtain a positive result. Otherwise, the report will have all zeroes for all calculated values.
6. Non-Linearities: Occasionally sample data may be curved for any of several reasons, or the analyte concentration may be near the detection limit with scattered data points. In such cases, the plot of net data should be inspected. For this reason, the plot is automatically displayed when  $R^2$ , the coefficient of linearity, is less than 0.96.

**5.0 OPERATION AND OVERVIEW**  
**(See KPAWin User's Guide)**

## **6.0 DIAGNOSTICS FOR BEST RESULTS**

### **6.1 Parameters For Analysis Diagnostics**

A combination of data parameters are available from each measurement, which provide diagnostic information about the validity of the result, hence, "Analysis Diagnostics." These are listed below with qualifying statements concerning their interpretation. Remember, the ideal set of data has a highly linear relationship between the logarithm of intensity and delay time, with a nearly ideal lifetime. The following refer to uranium as an example.

#### **6.1.1 $R^2$**

Values greater than 0.99 are typical. A small value is caused by a nonlinear plot and/or scattered data, such as near the detection limit. Curvature is caused by other sample phosphors, dirty cell windows, or suspended particles.

#### **6.1.2 Lifetime**

Lifetimes are temperature dependent, decreasing as temperature increases. See Section 2.4 for analyte-specific information.

Uranium lifetimes in samples are usually shorter than 350  $\mu\text{s}$ . For uranium, be aware that lifetimes less than 100  $\mu\text{s}$  occur when acidity exceeds two molar. At lower acidities, URAPLEX™ typically provides lifetimes over 250  $\mu\text{s}$ . URAPLEX™ improves the resistance of uranyl phosphorescence to quenching. For best results, lifetime should be >200  $\mu\text{s}$ . Uranium-specific characteristics follow.

1. If the lifetime is short, <100  $\mu\text{s}$  and/or curved, with  $R^2$  less than 0.96, consider diluting the sample if the concentration is high enough. Check the lifetime with a standard addition, especially if a nondetectable result is obtained. The luminescence may be completely quenched. Otherwise, wet-ash the sample to destroy interfering organics. Eliminate many reducing elements, halides, and excess acidity by boiling to dryness with nitric acid.
2. If the lifetime is long (>350-600  $\mu\text{s}$ ) and the decay plot is curved, particulates and/or organic phosphors may be present or the cell is dirty. Centrifuging, cell cleaning, and/or wet-ashing are in order.
3. If the result is near the analytical detection limit with a curved decay plot or short lifetime, dilution is barely an option. Take background measurements to verify that the cell is clean, i.e., blank net count within perhaps 300 counts of zero. If the cell is contaminated or dirty, clean and then repeat questionable sample measurements. Check lifetimes with standard additions, e.g. 0.1 mL of uranium standard in the cell.

For lanthanides, lifetimes will be close to the values in Section 2.4, unless a different complexing medium is present, acidity is too high, or a spectral interference with a substantially different lifetime is present.

### 6.1.3 Uncertainty

The KPA is capable of excellent (1-3%) precision. Replicated results within the uncertainty are easily obtained. An unusually large uncertainty indicates the concentration is either near the detection limit or a measurement problem. Either data is scattered, curved, or the concentration is out of the analytical range.

## 6.2 CONCENTRATION ISSUES

Occasionally, individual samples or a source of sample are analyzed and inaccurate or no results are obtained. An alternative procedure of preparing the sample for analysis can solve the problem and give you the analytical results needed.

### 6.2.1 Problems

Examples are:

*Color (inner filter effect):* Colored agent absorbs excitation or emission light.

*Quenching agent:* Substance that causes short or zero lifetimes. Acidity may appear to quench because the enhancing complex may be dissociated.

*Fluorescent agent:* Fluorescence from a contaminant may be strong enough to disrupt the normal decay curve of the analyte. This is indicated by curvature and a misplaced intercept.

*Particles:* Suspended particles interfere by strongly reflecting the laser beam. The reflected light will disrupt the normal decay curve of the analyte. This is indicated by curvature and a misplaced intercept.

*Major constituents:* Concentrated solutions of dissolved minerals, metals, or other substances may interfere by some of the above effects or by preventing complexation of the analyte with the enhancing reagent.

*Complexants:* A chemical may interfere by complexing the analyte differently than the chosen reagent. The effect will be noted by a different lifetime. Example: The EuEDTA complex, pH $\approx$ 4, has a lifetime near 311  $\mu$ s; the presence of citrate increases both the lifetime and quantum efficiency of europium.

*Static quencher:* A substance that holds the analyte out of the photoluminescence process causes static quenching and low results. An example is a complexant that maintains uranium as U(IV). Photoluminescent uranium is the  $\text{UO}_2^{++}$  ion.

*Precipitant:* A precipitate with either the analyte or enhancing reagent will interfere by loss and the particle effect. An example is phosphate when lanthanides are being determined.

Following are some actions that may be taken to correct faulty determinations. They are ordered by technical difficulty and labor required.

### 6.2.2 Actions

1. *Dilution.* Dilution is effective when the analyte concentration is well above the detection limit of the KPA. Substance(s) that detrimentally affect the KPA measurement may be diluted to where their effect is negligible. Use a dilution as large as practical.

2. *Centrifugation.* The interference due to the presence of suspended particles can be eliminated by centrifuging the sample before analysis.

3. *Determination by Method of Standard Addition.* This method of determination places a known amount of analyte in the sample matrix. The idea is that the sample matrix will affect the added analyte similarly to the native sample analyte. This option of quantitation is available in the program both in Automatic Analysis and in the Special Analysis menu of the manual analysis version of the KPA program.

4. *Evaporation to Dryness.* Taking the sample to dryness with an oxidizing acid (e.g., nitric acid) has the beneficial effects of eliminating some quenching agents and fluorescers. Particles/colloids may also be coagulated or eliminated. Substantial amounts of organic materials as from plants will not be eliminated.

5. *Wet-Ashing.* Samples of urine, vegetation, and soil or water-bearing decayed vegetation must be thoroughly wet-ashed to eliminate substances that may either strongly fluoresce or, in the case of uranium, quench. Follow the procedure for urine in section 7.0.

6. *Chemical Separation.* Analysis of bulk materials such as dissolved minerals, soil, or metal will require chemical separation from trace levels of the analyte. This method may be desired as an alternative to *Actions* 1-5 when the analyte concentration is near the detection limit. Likely separation types are ion exchange, ion chromatography, and solvent-solvent extraction.

## 6.3 Analysis, Verification, And High Precision

### 6.3.1 Dilution And Cell Cleanliness

The usual aliquot for measurement is 1.0 mL combined with 1.5 mL of complexing reagent. A ten-fold dilution may be easily obtained by adding 0.1 mL of sample to 1.0 mL of D.I. water in the cell. An additional 4% dilution of the 2.5 mL total volume is incurred.

Always inspect the cell against a light to detect smudges or acid/water droplets on the cell exterior and particulates on the inside. Water droplets may be touched off, but acid droplets and smudges must be rinsed off. Particulates in the cell may settle out. If they do not settle rapidly, it is best to centrifuge the sample before making the determination.



### 6.3.2 Verification

The application of good analytical practices is especially important for determinations near the detection limit. Periodic sample and instrumental blanks, quality control standards, and standard additions all have important roles in producing reliable analytical results.

In *kinetic* phosphorimetry, the standard addition has a special use. Sometimes interfering phosphors are present, and their presence must be identified. In other words, the detected phosphorescence must be verified as being due to analyte. A 0.1 mL or less addition of analyte providing an increase in concentration approximately equal to that indicated by the result, may be injected directly into the cell. The resulting recovery is then easily evaluated. A comparison of the lifetimes between the two measurements is important. If they are markedly different, then the sample result may in part be subject to interference and the sample must be treated. The frequency of such additions can be reduced at higher levels, where the analyte phosphorescence overwhelms any signal from interferences.

### 6.3.3 Precision

Excellent precision of 1-3% RSD is common with the standard techniques described already. However, by incorporating gravimetric measuring of aliquots, relative standard deviations better than 1% may be possible at concentrations in midrange and with lifetimes longer than 200  $\mu$ s. Samples digestions should be very slow to avoid spattering. Multiple analyses are necessary, of course, to establish the precision.

### 6.3.4 Detection Limit Measurement

Prepare a solution in concentration near the KPA detection limit. Analyze it ten or more times and calculate the standard deviation. This estimates the standard deviation of the background, that is, the noise level. The detection limit may be defined as three times this standard deviation.

## **7.0 SAMPLE PREPARATION: URANIUM ASSAY OF URINE**

### **7.1 Principle**

Proper preparation of samples is of utmost importance to the analysis. Raw urine cannot be analyzed without pretreatment except at levels well above 20  $\mu\text{g/L}$ . A large dilution, >1000, is necessary to dilute away the matrix effects. The reasons for this are: the organic constituents fluoresce, may complex uranium, and along with chloride, quench uranyl phosphorescence.

Sample treatment, i.e., wet-ashing, becomes increasingly important as the needed detection limit is lowered. Residual unsaturated organics in a poorly ashed sample may raise the detection limit by a factor of ~100. Aliquots of samples from the same bottle may ash differently.

This procedure is written for urine analyses near the KPA detection limit of about 0.02  $\mu\text{g/L}$ . The requirements for ashing are less stringent at higher levels. However, good ashing technique is still needed for good precision.

### **7.2 Reagents**

Deionized Water: Preferably over 10 megohms.

URAPLEX™: A superior uranium complexant that provides longer phosphorescent lifetime than others for better data, stronger complexing, and lower background level of uranium.

### **7.3 Equipment**

Acid Dispenser - Such as 500 mL Repipet; set the dispensed volume to 1.5 mL. Glass reservoirs should be acid-leached; plastic reservoirs should be immersed in boiling water.

Liquid Scintillation Vials, 20 mL. For analyses below ~0.2  $\mu\text{g/L}$ , vials must be leached in either 1 M phosphoric or 4 M nitric acid at sub-boiling temperature for two or more days to remove leachable uranium. Rinse well with deionized water.

Hot Plate

Heating Block, optional - Made from 40 - 45 mm (1.5 inch) thick aluminum with a number of holes such as 50 holes to accept vials, 20 mm (0.75 inch) deep, 28 mm (1.1 inch) diameter. Handles for carrying may be placed at each end.

Muffle Furnace.

## **7.5 Alternative Ashing Procedure**

1. Add weight or volume aliquot of sample (2-10 mL) to vial. NOTE: Samples with solids must be homogenized (vigorously stirred), because uranium concentrates in solids.
2. Add 2-3 mL 16 M nitric acid with swirling and 0.5 mL of 30% hydrogen peroxide.
3. Place vials on hot plate at moderate heat and NEAR boiling for several hours. Hydrogen peroxide may be effectively added after partially cooling the samples.
4. Increase heat to boil dry. Replenish oxidants 1-2 times until the residue is pale yellow to white. Cool samples somewhat before adding hydrogen peroxide.
5. When the vials are dry, place in muffle furnace at 500-550° C. for at least 1/2 hour.

NOTE: WET-ASHING MUST BE COMPLETE TO AVOID RESIDUAL ORGANICS. FURNACE TREATMENT WILL NOT COMPENSATE FOR POOR WET-ASHING.

The presence of carbon black after the furnace step indicates incomplete wet-ashing. For samples less than 0.2 µg/L, the ashing should be restarted with a fresh aliquot.

6. When cool, dissolve the urine salts in 1/2 to 1 mL of 4 M nitric acid with warming. Dilute to desired volume with deionized water. Swirl to mix. Final volume may be determined by weight.

NOTE: For efficiency, steps 2-6 can be accomplished in a heating block.

7. For low level samples, it is beneficial to let solutions set overnight to let micro-particulates settle out. Centrifuging is an alternative.
8. Analyze resulting solutions with the KPA. Avoid picking up solids with the sample aliquot.

## **7.5 Alternative Ashing Procedure**

Perchloric acid, 0.5 mL, may be used instead of the 30% hydrogen peroxide. Step 4 can be eliminated.

## **7.6 Uranium Assay Of Water**

Most clean water samples can be analyzed directly. Other water samples may just require a simple acidification with nitric acid and boil down to dryness. If significant organic matter such as decayed vegetation is present, a wet-ashing similar to urine may be required. Because chloride and reducing metals quench strongly, some waters cannot be analyzed without pretreatment.

**OTHER APPLICATIONS - Contact CHEMCHEK INSTRUMENTS, Inc.**

## **8.0 SERVICE INSTRUCTIONS**

### **8.1 Dye Module Alignment**

The tuning of the laser system in the KPA needs to be performed only by trained personnel. If the analytical sensitivity seems low, first make sure the dye cell is correctly orientated. If the dye cell has correct orientation, then contact CHEMCHEK with any questions.

### **8.2 Replacement of Nitrogen Laser Plasma Cartridge**

This procedure should be followed when the laser power degrades to marginally useful levels or otherwise fails.

**EXERCISE CAUTION** when removing the KPA cover: Residual high voltage can exist inside! The bleeder resistor used on the high voltage circuits requires a short period of time (about one minute) to drain any remaining voltage.

**CAUTION:** THE LASER PRODUCES INVISIBLE ULTRAVIOLET RADIATION. Make sure the laser beam will not contact skin, highly reflective surfaces, or especially the eyes!

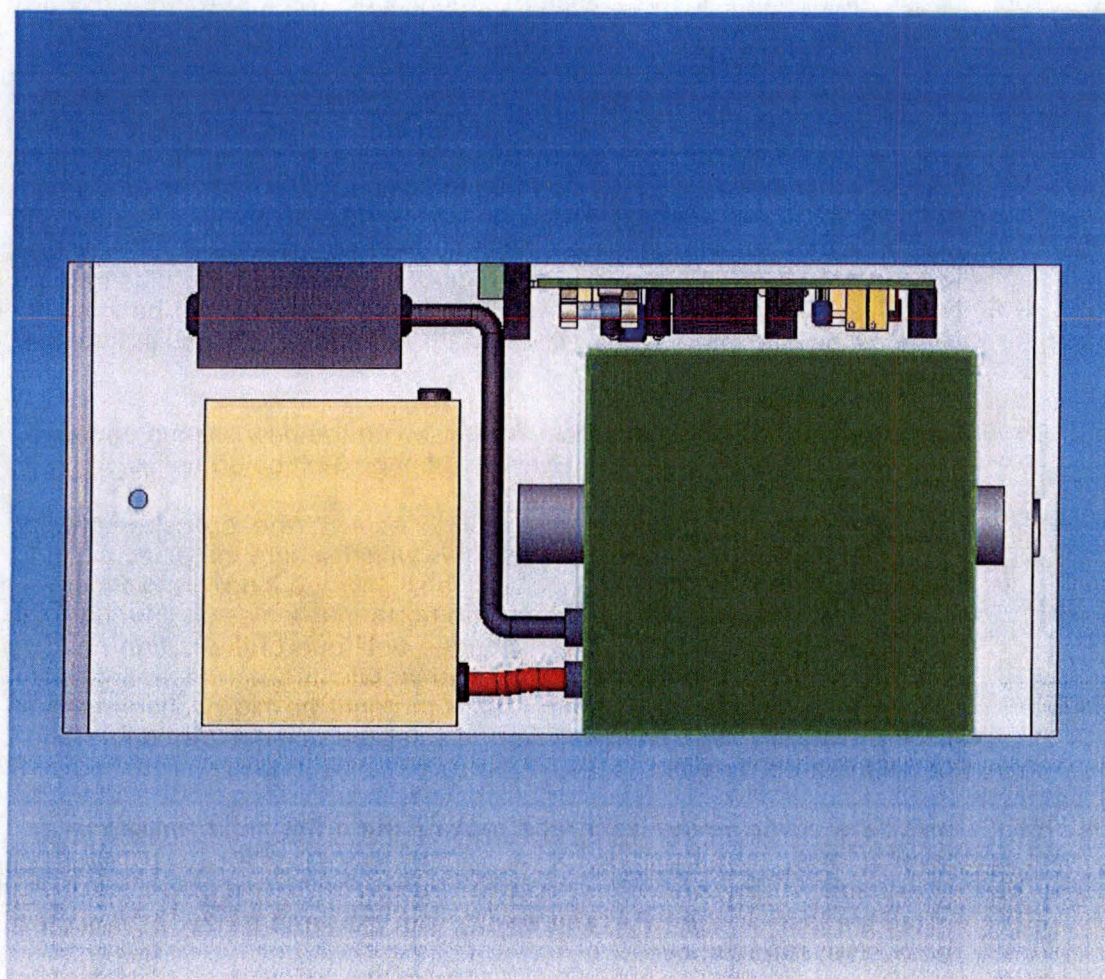
This procedure is used when installing a plasma cartridge with black sides.

01. Turn off the KPA and remove its cover.
02. Remove the 3 round headed laser hold-down screws from the top of the laser. Disconnect the 3 connectors from the back end of the laser. Rotate counterclockwise and remove the BNC connector for the laser trigger (marked "TRIG" on the back of the laser). The second connector (marked "INT") is the interlock; push down on the latch on top of this connector and remove the connector. The third connector (marked "12VDC") is for 12 volt power; pull it from the back of the laser.
03. Remove the laser from the KPA, and place it on a flat surface.
04. Remove the 4 flat head phillips screws from the laser cover sides, and lift the laser cover away.
05. Turn the laser over, and remove the 4 button head screws which hold the plasma cartridge to the laser base. Use a 3/32 inch hex wrench to loosen the button head screws.
06. Turn over the laser carefully while holding the plasma cartridge, so it will not fall out of the laser. Lift up the front of the cartridge, and rotate it out of the laser. As the cartridge comes out, remove the cables in the rear of the cartridge by gently pulling them as the cartridge is removed.
07. Examine the connectors on the ends of the high voltage leads for evidence of

burning or arcing. If significantly burned, call CHEMCHEK for replacement connectors.

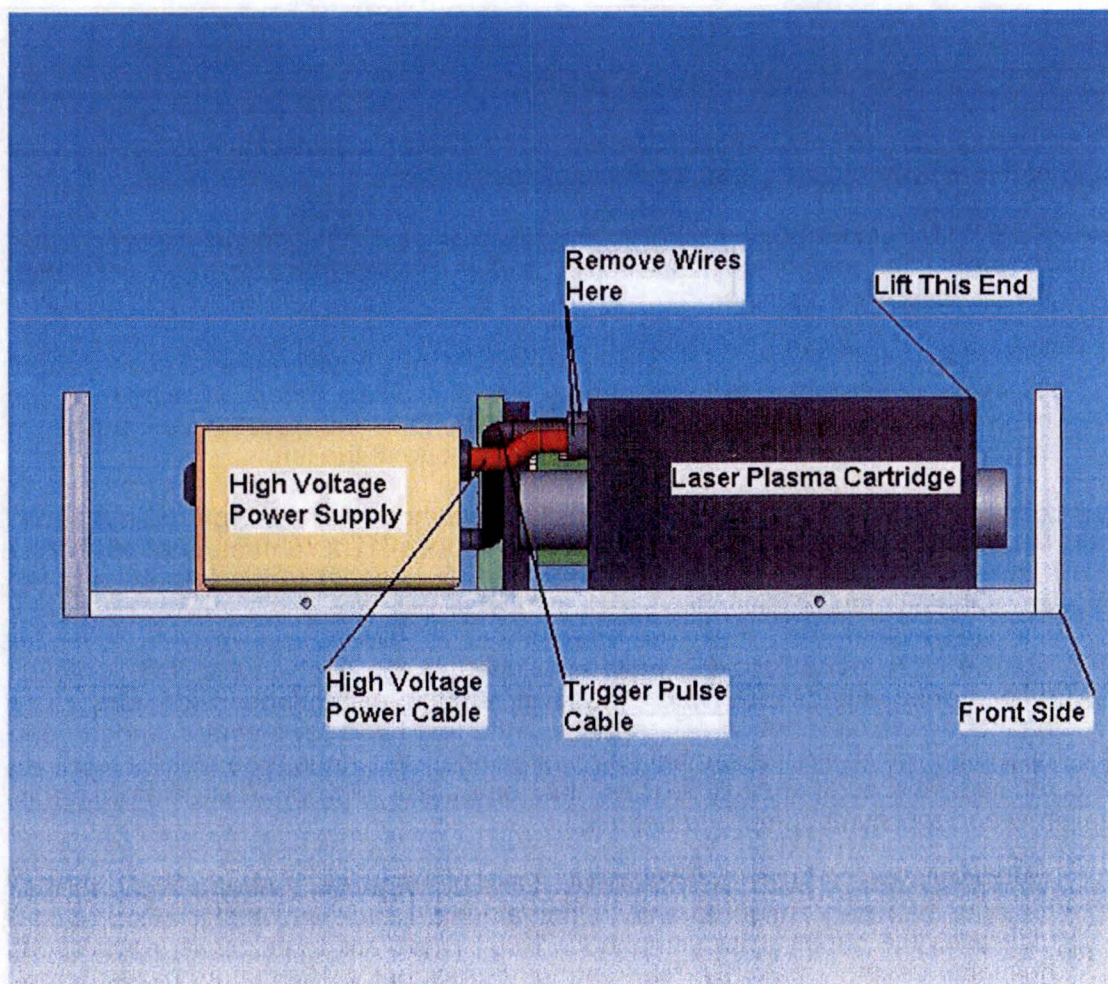
08. Unwrap the replacement plasma cartridge, and install the high voltage cable and trigger cable into the new plasma cartridge. Push both leads in firmly until they seat in the plasma cartridge (about 35 millimeters into the cartridge). Make sure that the leads are all the way into the plasma cartridge. Measure and mark the length of the leads if necessary, to insure complete insertion into the plasma cartridge. In some cases it may be necessary to apply silicone high voltage grease to the leads, to help inserting them into the plasma cartridge. Note that the high voltage cable and the trigger cable cannot be interchanged. If the leads are interchanged, the plasma cartridge will not fit into the laser properly.
09. Remove the protective caps from the lenses on the new plasma cartridge. After removal of the caps, use caution not to damage the coated optics.
10. Turn the laser over, taking care not to scratch the optics on the plasma cartridge. Install the 4 button head screws which secure the plasma cartridge to the laser base. If the high voltage lead is slightly too long, preventing the holes in the laser base from lining up with the holes in the plasma cartridge, then do the following. Rotate the plasma cartridge until one of the button head screws can be inserted. Install that screw, then rotate the cartridge and install the remaining screws. It may be necessary to bend the high voltage lead slightly per the diagram to aid in installation. Center the plasma cartridge in the laser, then tighten the screws.
11. Install the cover on the laser, and replace the 4 flat head Phillips laser cover screws.
12. Install the laser in the KPA, and secure with the three 6-32 x 2 1/2 inch screws removed in step 2 above.
13. Install the 3 connectors on the back of the laser into their proper sockets.
14. Turn the KPA on, and check for proper operation of the laser.





**Figure 4. Plasma Cartridge Replacement, Laser Top View**





**Figure 5. Plasma Cartridge Replacement, Laser Side View**



## **9.0 AUTOMATIC SYSTEM**

### **9.1 Components of Automatic System**

The components of the Automatic Kinetic Phosphorescence Analyzer System consists of the KPA, a syringe pump, and a sample changer.

**SYRINGE PUMP, Model 402 (Dilutor).** The dilutor is equipped with a 5-ml syringe for aspirating or dispensing solutions, and a valve that controls flow of either reagent/diluent or sample.

**SAMPLE CHANGER, Model 233.** At the KPAWin© software's direction, the Model 223 selects each sample to be analyzed by the KPA. Selection is by means of xyz movement of a needle probe. The Model 223 holds four racks, which are positioned on the polypropylene tray, beginning with rack one at the left.

Samples are held in three Code 24 racks, each holding 14 sample vials (20 ml). The fourth rack, Code 29, holds 60 culture or test tubes (12 x 75 mm). Vial positions in the Code 24 rack start with #1 nearest the sample changer in the left row and proceed through each row of seven for a total of 14 vials per rack.

The 60 tube rack, Code 29, must be placed at the right on the tray. Dilution and mixing of sample aliquots with enhancing reagent takes place in this rack. The rack has four rows of 15 tubes each. Each of the first three rows corresponds to one rack of sample vials with the number one vial being mixed in the tube closest to the sample changer, the number 14 tube is the last one used. The number 15 position in each row and row number four are not used.

**ALTERNATIVE RACK CONFIGURATION, by request.** Alternatively the sample changer can be configured with a non-standard rack and vial configuration. This increases the capacity for samples to 108. There are three racks, each capable of holding 44 sample vials (6.8 ml). The fourth rack holds 108 culture or test tubes (10 x 75 mm). This configuration, however, does reduce the amount of available sample for repeat analysis.

**FLOW CELL, and associated tubing.** The flow cell has a sample chamber that is less than 100  $\mu$ L in volume. Tubing and cell are chosen to optimize analytical quality: sample size, speed, detection limit, and low cross-contamination. Cross-contamination from previous samples is less than 0.1%.

The dilutor and sample changer are controlled along with the KPA by the KPAWin© Software. More information about these two modules is in the Gilson manuals, which are provided separately.

**READ THE GILSON MANUALS FOR UNPACKING AND ASSEMBLY INSTRUCTIONS FOR THE 402 DILUTOR AND THE 223 SAMPLE CHANGER.**



## 9.2 Electrical Connections and Set-up

For mechanical connections, please refer to the Gilson user guides.

Electrical connections are as follows. The GSIOC cable is a Y-cord with two female, 9-pin connectors and one male 9-pin connector that provides communication between the sample changer and the dilutor. Standard 25-pin by 9-pin serial cables are used to provide communications between the KPA and the computer and between the sample changer and the computer. Additionally, newer computers do not have two serial port connectors. For these types of computers, a USB Dual Serial Adapter is used.

**GSIOC Cable:** This is the Y-cable with one 9-pin connection (single end) at one end and two 9-pin connections (double end) at the other end. Connect the single connection to the back of the Sample Changer. Connect the one of the double end connections to the back of the Dilutor.

**USB Serial Adapter (Optional for computers with a USB port and less than 2 serial ports):** Please refer to the installation guide provided with the product for proper installation.

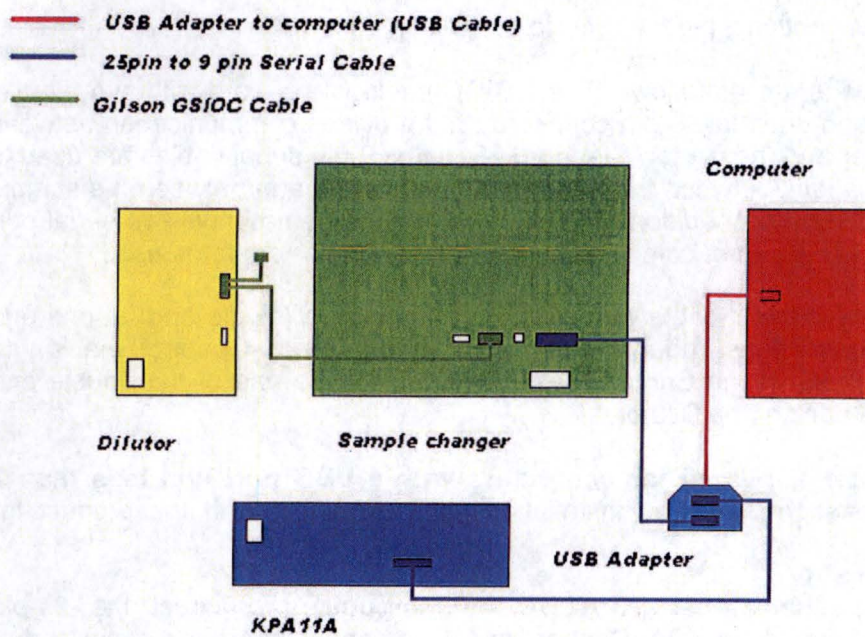
**9-pin to 25-pin Serial Cable (KPA11A to Computer):** Connect the 25-pin connection to the back of the KPA. Connect the 9-pin connection to top serial port of the USB serial adapter, or Com Port 1 on the computer.

**9-pin to 25-pin Serial Cable (Sample Changer to Computer):** Connect the 25-pin connection to the back of the Sample Changer. Connect the 9-pin connection to bottom serial port of the USB serial adapter, or Com Port 2 on the computer.

**NOTE:** The 402 Dilutor has 8 dip switches numbered from bottom to top. In normal operation, switches 6, 7, and 8 are closed (positioned to the right when viewed from the back of the pump) and switches 1, 2, 3, 4, and 5 are open (positioned to the left).



Figure 6: Cable connections





### 9.3 Tubing Connections

**Syringe Pump (Dilutor)** – The dilutor draws diluted URAPLEX™ reagent from the reservoir through the porous metal filter and tubing as described in the dilutor manual. Solution transfer to the sample changer is through the 4 m length of 1-mm I.D. Teflon tubing. This length has a volume exceeding 3 mL to minimize the chance that sample might enter the dilutor syringe. The tubing passes reagent and sample solution to the sample changer probe. It also serves as a reservoir for aspirated solution to the sample changer probe. The tubing should be arrayed to prevent entanglement with the changer arm, eg., suspend it from the ceiling or fasten the excess coil down at a suitable location.

**Sample Changer to KPA** – The sample changer injection port, is connected to the stainless steel tubing in the KPA door by means of a short length of 1-mm I.D. Teflon tubing. This tubing must be kept short for a minimum internal volume. Therefore, the KPA should be positioned so its door is near the injection port of the sample changer. Connection between the Teflon and steel tubing is by means of a short length of the white C-flex tubing.

**NOTE:** The injection port contains a rubber and Teflon seal. If a leak should occur during sample injection, first try tightening the nut. **CAUTION!** Over tightening will hinder the probe entering the seal and overload the changer motor (it will sound different so you will know if it is tightened too much).

**Sample Cell** – The flow cell is placed in the cell holder in the KPA door. Connections to the 1/16 inch O.D. stainless steel tubes, which pass through the door, are with elastic C-Flex tubing. The flow cell should be orientated with the writing on the cell facing the reference cell. The c-flex from the input stainless steel tubing connects to the cell tube leading to the u-shaped channel in the flow cell (the cell tube furthest from the door). The outlet cell tube connects to the output stainless steel tubing.

**Drain Connections** – The output stainless steel tube is connected to the drain tubing in the rinse station by means of 1-mm I.D. Teflon tubing. A short length of c-flex tubing connects the Teflon and steel tubing. The first 5/16 inch O.D. drain tubing connects to the rinse station and extends to a suitable receptacle. The second drain tubing is connected to an overflow at the side of the sample tray and extended to the same receptacle. This is in case of spillover.



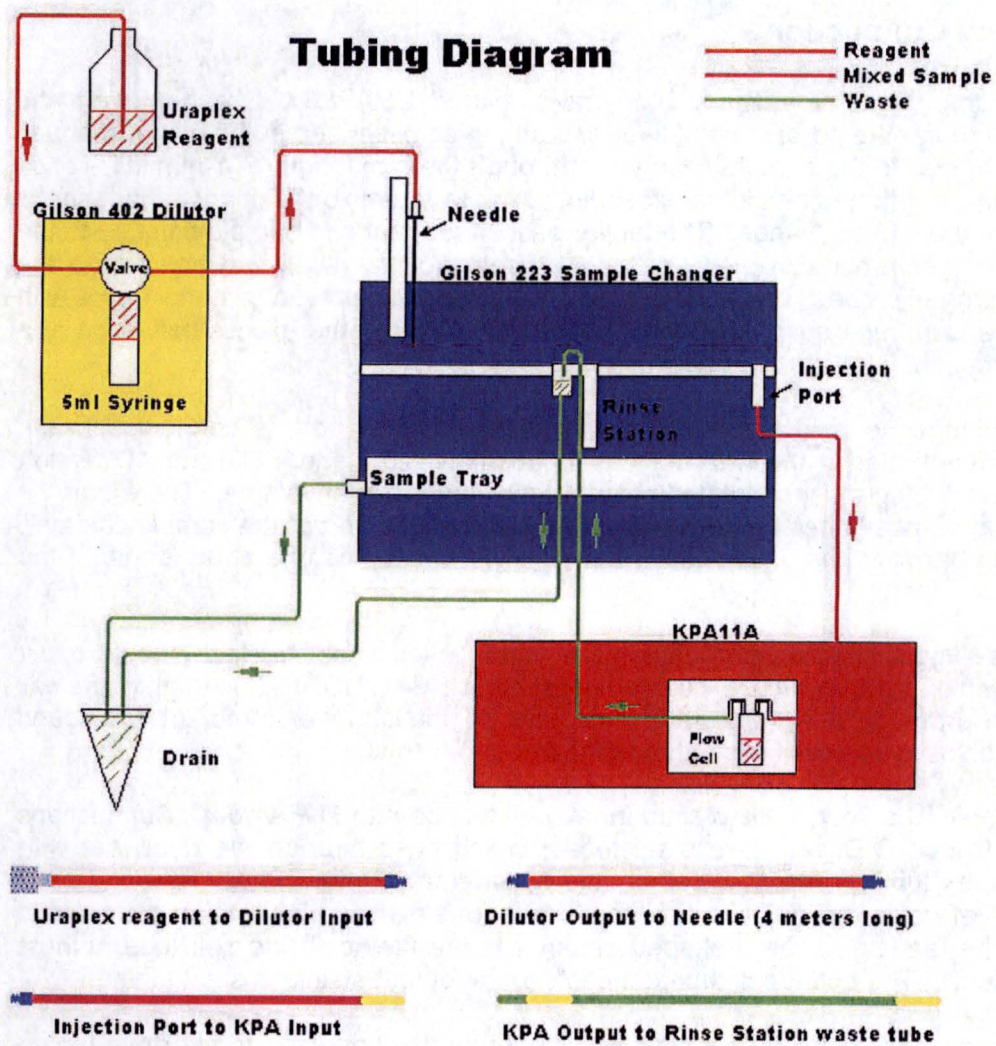


Figure 7. Tubing Diagram – Automatic Operation



## 9.4 Automatic Operation Overview

Automation accomplishes these steps.

1. Zeroes the dilutor syringe (it should already be empty).
2. Draw 3.0 mL of enhancing reagent/diluent (URAPLEX™ for uranium) into dilutor syringe.
3. Pick up (aspirate) 1 mL of sample from sample vial.
4. Dispense aliquot into the corresponding mixing tube.
5. Dispense 1 mL of enhancing reagent into the tube.
6. Aspirate 1.9 mL of mixed solution and inject into KPA flow cell.
7. Stop-flow for KPA measurement.
8. Flush sample solution out and rinse the flow cell.
9. Flush out the rinse station, emptying the syringe.

Either 42 or 108 samples (depending on choice of rack configuration) can be automatically analyzed per run. These may be any combination of samples, standards, blanks, and standard-addition sample pairs. Results are stored, printed, and accessed through *Reports → Selection*. The same automatic advantages are available for Calibration measurements.

## 9.5 Automatic Sample Analysis

PLEASE NOTE: The 402 dilutor must be turned on prior to the 223 sample changer.

To analyze samples, follow the steps listed.

1. Proceed to the Analysis Input Form (*Analyze → Sample Input*).
2. Input all needed sample parameters to correspond with rack loading.
3. Load the racks to correspond to the Analysis Input Form.
4. Load the 4<sup>th</sup> rack with new mixing tubes to correspond to the loading of the sample vial racks.
5. Make sure there is enough enhancing reagent (URAPLEX™ for uranium). It takes 3.0 mL per sample and there should always be enough remaining to partially cover the porous metal filter. This means more than 200 mL in a small diameter reservoir (for 42 samples) or perhaps 500 mL in a larger one (for 108 samples). Running out of reagent will mean invalid results. In addition, the probe and flow cell will need to be primed. Use the rinse function (*Tools → Auto Changer → Rinse*) for priming.
6. Click *Analyze → Analyze All*.
7. Results can be reviewed and/or printed out later through *Reports → Selection*. See KPAWin© User's Guide for details.

NOTE: During automatic analysis, the range will switch to:

HIGH – The result is > 120% of the highest Low Range calibration standard.

LOW – The result is < 80% of the lowest High Range calibration standard.

These values may be changed in the KPA Unit Settings tab.

## 9.6 Interrupts

Aborting – Sometimes analytical operations are started with an error in sampling, identification, loading of sample trays, etc, and you will wish to INTERRUPT the operation. The stated method in all cases is to press the ESCape key.

Be sure to rinse the flow cell with the rinse function (*Tools → Auto Changer → Rinse*) before starting to analyze other samples.

Sample changer – Occasionally in a run, the sample changer may experience "improper head movement" from encountering an obstruction or get out of step and complain with a buzz. Quickly reset by keying ESCape. Then either turning it OFF, then ON again, or use the reset function (*Tools → Auto Changer → Reset*).

## 9.7 Flow Cell and Syringe Pump Care

### Flow Cell Care

The flow cell is a critical component of the automatic KPA system. One advantage is that it is NOT routinely handled, so exterior contamination is seldom a problem. On the other hand, the cell interior is of concern, because it cannot easily be cleaned. The following are some useful guidelines.

#### Stand-By Mode

Never leave significant concentrations of analyte solution in the cell. Actually, the sample changer does a partial flush after each KPA measurement. However, you should always flush the cell by one of the following methods.

1. Finish with a blank.
2. Use a post rinse on the final sample.
3. Flush the cell with the rinse function (*Tools → Auto Changer → Rinse*).

NOTE: These three methods leave the cell and tubing filled with the reagent/diluent.

4. Place the tip of a squirt bottle in the injection port and force several mL of reagent grade water through the flow cell. Water is preferred because the reagent/diluent may either decompose or corrode tubes. This is the recommended method if the instrument will be unused for periods longer than 2 weeks. Otherwise, one of the first three methods should be adequate.

#### Solids

The presence of particulate material in the sample aliquots can be a serious matter. Reason ONE is disruption of the spectrometric analysis. Reason TWO is that some particles may accumulate within the flow cell where they may be difficult to remove. The sample changer probe is programmed to not come closer than 3 mm to the vial bottoms in order to avoid picking up solid material. In order to remove suspended solids in your samples, you should:

1. Settle them over time,
2. Centrifuge, or
3. Filter

#### Cleaning

A cell chamber dirty from grease or oil may cause air bubbles to adhere to walls or cause direct spectral interference. Clean by flushing with 10% nitric acid followed by water at the cell connections. Particulates may require agitation, forward or back flushing, hot acids (fill the cell and place in a water bath), and/or ultrasonic treatment. Any detergent used must not contain lanolin, oils, or insoluble matter.

**CAUTION:** If the flow cell is handled, it may need its windows cleaned before a low background can be obtained.

## ORIENTATION

Flow cell orientation is important. Position the cell to maximize its length. The side with the U-shaped channel (there should be writing at the top ex. QS 10.00mm) should face the reference cell. Do not confuse the inlet and outlet connections. The inlet connection to the cell delivers solution to the bottom of the sample chamber via a U-shaped channel and clears the cell from the top of the sample chamber. A cell that is installed incorrectly will trap bubbles and will display extremely high and variable backgrounds.

## Syringe Pump Care

The Uraplex™ reagent is a concentrated salt solution. Rinse the valve of the syringe pump with deionized water before prolonged storage of the device to avoid a salt accumulation that will freeze the valve. An easy way to do this would be to perform four or five rinses (*Tools → Auto Changer → Rinse*) with DI water.

## 9.8 Conversion to Manual Operation and Analysis

To convert from automatic to manual operation and analysis please do the following.

1. Change the hardware configuration (*Settings* → *General Configuration*) by clicking on the Hardware Settings tab. Once there change from the automatic setting (1000 Release for new KPAs using comports 1 and 2) to the manual setting (999 Eprom 3 Man for KPAs using comports 1 and 2) and press "current".
2. Remove the flow cell with connecting tubes attached and wrap in lens tissue for storage.
3. Use the low background phosphorescence cuvettes for manual operation of the KPA as described in the "Operation Manual" for the KPA 11.

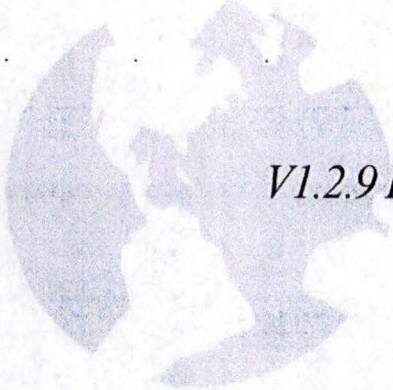
**Attachment 6**  
**KPAWin Software User's Guide (Chemchek)**



1845 Terminal Dr.  
Richland, WA 99352  
Tel: 509-943-5000  
Fax: 509-946-3368  
Email: [service@chemchek.com](mailto:service@chemchek.com)

Chemchek Instruments Inc.

# KPAWin© Software User's Guide



*V1.2.9 Release – 21 May 2009*



## KPAWin© Software User's Guide

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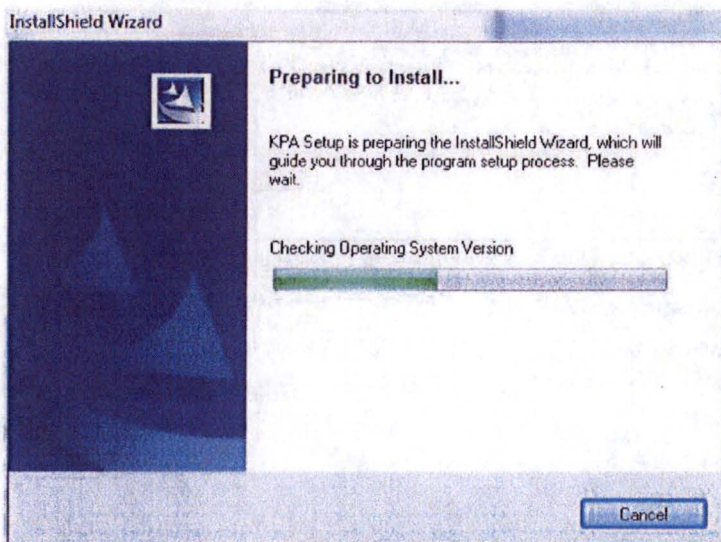


## **KPAWin© - Version 1.2.9 Installation Instructions**

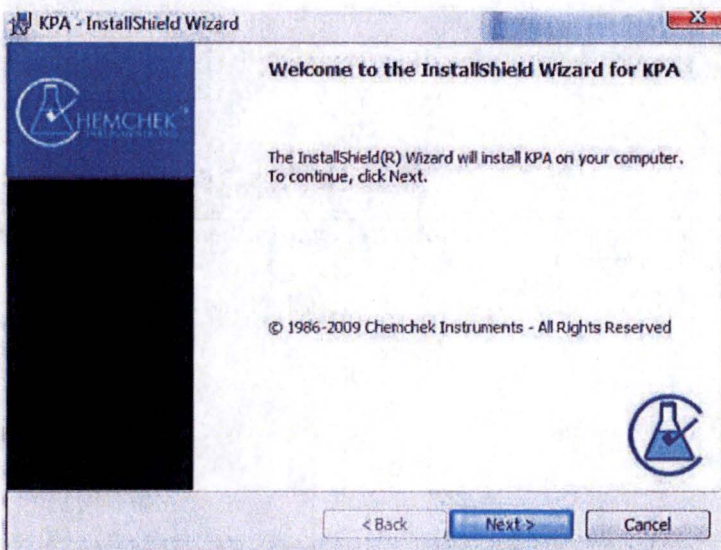
The following instructions install the KPAWin© software, the associated Databases, and all Documentation on your system. The software can be installed on an IBM Compatible Pentium class PC. Users can exercise the Demo Scripts to familiarize themselves with the software on any PC. A connection to a KPA Unit is NOT required to exercise the Demo Scripts. A connection to a KPA Unit is only required if a user desires to actually analyze Samples.

### **KPAWin© Software Installation**

Insert the CD into any available CD drive. The Installation software will automatically begin.



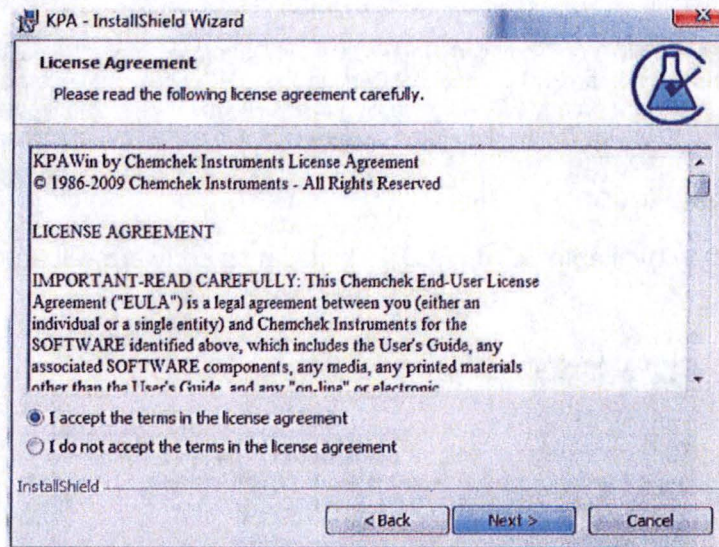
Preparing to install. The installation program is verifying that the OS is sufficient, there is enough disk space, and there are no previous versions installed.



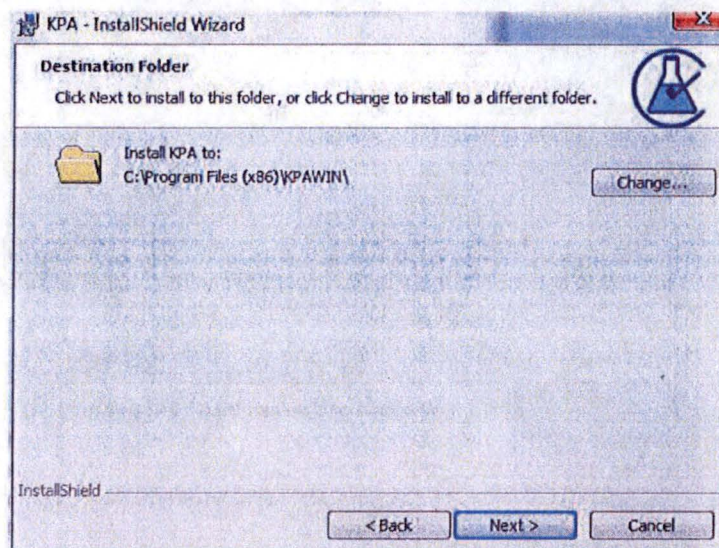
If everything is OK the installation program will stop at the Welcome Screen. Select Next to



continue.

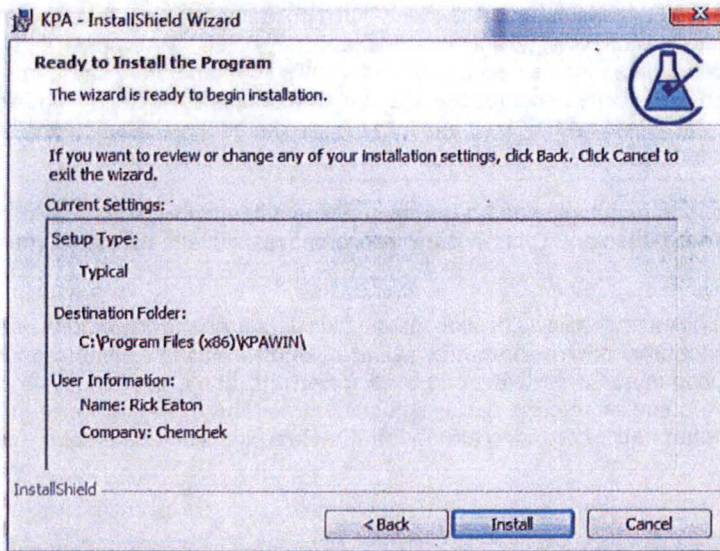


Read and accept the License Agreement. Click Next to continue.

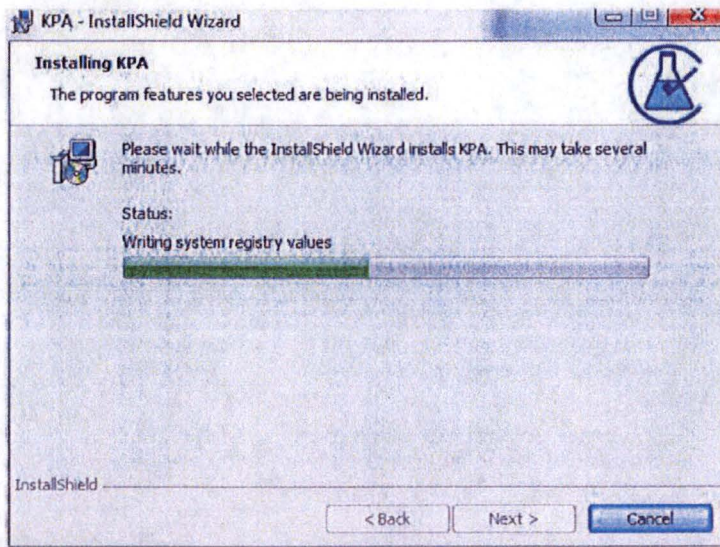


Accept or change the database location. Normally, there is no reason to change this path or the program location path.





The installation program pauses to review the choices. Press Back to change anything or Install to continue.



The program installs.

After installation is successful, the KPAWin© software will start automatically.

A desktop icon is installed on your desktop to launch KPAWin©.





Located in the KPAWin folder is a Database subfolder. The KPAEmpty database file is a template and should not be used directly to store the data from the KPAWin© software. Make a copy of the KPAEmpty file in the KPAWin\Database directory and give the copied file a name that is descriptive and follows your laboratory standards (e.g. C:\Programs\KPAWin\Database\MyLab.mdb). This copy becomes the site-standard template. Any copy made must have .mdb as an extension or KPAWin© will not be able to use it. Be sure to leave an unaltered copy of the original KPAEmpty and of your site standard template file in the directory.

If you use Windows Explorer to copy the database file between different directories, hold the Ctrl Key down as you drag the KPAEmpty file to the Database directory. This will copy but not move the file to the new directory.

The Configuration Installation section that follows describes how the site-standard copy of KPAEmpty can be modified to contain all local preferences for configuration settings. For example, DOSKPA can be simulated with KPAWin© by using only four of the eight configuration tabs: General, Hardware, KPA Settings, and Calibration. The Alarms and Warnings tab is disabled simply by not selecting any alarms items. The other three tabs for Performance Limits, Quality Control Limits and Maintenance are deselected by pressing the "Do Not Use" radio button at the top of each of the three tabs.

## Installation Test

1. Initialize KPAWin© from the icon on the Desktop.
2. Select "Database" and "Open" from the command line.
3. Select C:\Programs\KPAWin\Database\KPADemo.
4. Select the "Login Menu" button on the left-end of the button bar.
5. Select the user "Supervisor".
6. Enter the Supervisor password of "Supervisor".
7. Select "Configuration Tabs" from the button bar (fifth button from the left). The form will open.
8. Select the Hardware Settings tab. Step through the seven alternative configurations with the "List Text Box" in the lower right of the form. The index of the release version is 1000 for a KPA-11A. All other six configurations have the "Inactive Box" checked. Configurations are preloaded by KPA model (KPA-11/KPA-10). KPA EPROMs are (KPA-11 (3), KPA-10 Auto shutter (2), or KPA-10 Manual shutter (1)). Autochanger (A) models and Manual (M) models are also specified. Select the appropriate index. Select "Current" and "Apply".

<u>Index</u>	<u>Model</u>	<u>EPROM</u>	<u>Auto/Manual</u>
994	KPA-10	1	Auto
995	KPA-10	2	Auto
996	KPA-11	3	Auto
997	KPA-10	1	Manual
998	KPA-10	2	Manual
999	KPA-11	3	Manual
1000	KPA-11	3	Auto (Release default)

9. No action is required for an automatic KPA-11. For all other configurations, mark configuration 1000 as inactive and the correct configuration as active by deselecting the "Inactive Box".
10. Select the KPA Unit Settings tab. No action is required for a KPA-11 since index 1000 is the release configuration. For a KPA-10, deselect index 1000 and select index 999. Select "Current" and "OK".
11. Select "Analysis Input Form" from the button bar (third button from the left). The form should open. "Close" the form.
12. Select "Sample Reports" from the button bar (fourth button from the left). The form should open. "Close" the form.
13. Select "Initialize KPA" from the button bar (sixth button from the left). All subsystems and functions of the KPA and autochanger are tested. Communications are established and tested. Maintenance status is evaluated against release standards. (See Configuration Tabs below.)
14. Responses to any failures observed in this test are discussed below under Possible Start-up Problems.

## **Possible Startup Problems**

### **General KPAWin® Failure**

Installation is successful but one of the following three problems are found during testing.

1. "Error loading from file" while attempting to load and execute the KPAWin®.
2. "An error occurred while loading or registering the file 'C:\Windows\System\Comctl32.ocx' while attempting to load and execute the KPAWin
3. Devices on the button bar or the command line do not respond.

Oleaut32.dll should be loaded and registered when Windows is installed. If the file is missing, obsolete, or unregistered with the operating system the Object Linking and Embedding functions and devices fail. The Regsvr32.exe and Oleaut32.dll files, included on the Auxiliary File diskette of the release, correct these problems. If your system responds correctly, disregard the following operations.

1. From Explorer select "View" from the command line and "Show all files" from "Options".
2. Open C:\Winnt\System32 for Windows NT or C:\Windows\System for Windows 98/95 and verify that Oleaut32.dll is present. Verify that its version number is 2.20 or higher. The version is available from Properties that can be accessed from the right button of your mouse.
3. If necessary, copy the current Oleaut32 from the Auxiliary File diskette to the system folder. Reset the "Options" selection in the "View" menu to "Hide files of these types".
4. Register the Oleaut32 file with the Regsvr32.exe program that is also included on the Auxiliary File diskette.
  - a. Put in the Auxiliary File diskette.
  - b. Select the "Start" button.
  - c. "Run".
  - d. In the "Open:" space, key in "A:\Regsvr32 Oleaut32.dll". (A space is required between Regsvr32 and Oleaut32.dll.
  - e. Select the "OK" button.
  - f. The system should respond that the file was registered successfully.
  - g. Then try to start (execute) the KPAWin® program again. It should load successfully.

### **Initialization Failure**

Initialization failure can result from hardware, software, or administrative failure. Administrative failures are corrected later during configuration. Disregard failure of Maintenance tests until the hardware and software systems are stable.

### **Hardware Failure**

Installation of the KPAWin® program in a previously operable system is not likely to create a hardware failure. The results of the Initialization test specify the component that has failed and if the cause is due to communications. Check for power and laser indicator lights, laser cycling, low-range solenoid operation, and autochanger and syringe pump operation.

Components that can cause the KPA to fail initialization include:

1. Power switch is off,
2. Power cable,
3. Communication cable to computer,

4. Interlocks,
5. Nitrogen laser,
6. Photomultipliers, and
7. EPROM.

Components that can cause the autochanger and syringe pump to fail initialization include:

1. Power switch is off,
2. Power cables,
3. Communication cable to computer and included modem,
4. Y-cable between communication cable, autochanger (bottom center) and syringe pump,
5. DIP switch settings on Model 402 syringe pump (From the rear, #1 @ bottom and #8 switched to the right for 401 emulation mode and #1, #6, and #8 switched to the right for 402 emulation mode),
6. EPROM.

Components that can cause the computer to cause communication failure include:

1. Power switch is off,
2. Power cable,
3. Serial COM ports improperly identified and cabled,
4. Serial COM ports not enabled in BIOS,
5. Modem with hard-wired serial COM port setting of 1 or 2,
6. Serial mouse with an automatic system.

In general, if initialization fails, check for the power indicator lights and cabling. Rerun the EPROMs by cycling the power switches on the KPA and the autochanger or by repeating the initialization test.

### **Software Failure**

Software failure can result from missing, defective or unregistered system files, errors in KPAWin© configuration settings, or a damaged database. Assignment of serial COM ports in the configuration is easily checked by changing cables or settings on the tab. Damaged databases can be identified by the successful load of a database that is known to be good. The computer identifies missing system files when the program attempts to call them.



## Configuration Settings

KPAWin© has eight Configuration Tabs that can be accessed from the third button on the button bar or from the General Configuration option under Settings on the command line. Refer to the KPAWin© Configuration Documentation in the Documentation folder for descriptions of the labels and devices that are used on these tabs. These descriptions are also available on line from Help on the command line.

It is highly recommended that you create a site-standard template of the "KPAEmpty" database. Follow the instructions below to enter the configuration preferences in single or multiple copies of each tab. Name and save the site-standard template. Working copies of the template should be made, named, and used to conduct laboratory operations. This strategy eliminates the need to reconfigure the distribution database every time that you need a new local copy. See the Configuration instruction section of this manual for details.

Basic KPA operation requires completion of four tabs: General Items, Hardware Settings, KPA Unit Settings, and Calibration Settings. If basic KPA operation is desired, the "Do Not Use" radio button must be selected on three tabs: Maintenance, Performance Limits, and Quality Control Limits. In addition for basic KPA operation, do not select any flags from the Alarms/Warnings configuration tab.

Advanced KPA operation utilizes the four basic tabs, selection of either the "Use Defaults" or the "Use Site Custom" radio button on the Maintenance, Performance Limits, and/or the Quality Control Limits configuration tabs, and selection of flags of your choice from the Alarms/Warnings configuration tab.

All configuration tabs are managed by common devices located at the right and at the bottom of each tab. Each alternative copy of each configuration tab is unique and may qualify specific data in your database. Taken together, these properties require that existing copies of any one configuration tab can not be deleted but additional copies can be created. Obsolete copies can be hidden from the Sample Input form by selecting Inactive in the box at the bottom of the form when the index for the copy is listed in the List Text Box.

The buttons on the right of all configuration tabs operate consistently. "Help" calls on-line help. "Cancel" terminates a tab edit session. "Select" provides a drop-down list of the names of all copies of the selected tab. You can cursor to any label in the list, highlight the selection, and select the entry. "Add" makes an editable copy of the displayed copy of the tab. After your edit is complete you will be prompted to assign a unique index and label to the copy. "Current" sets the displayed copy of the tab as the default for Sample Input. It is essential that your preferences for the General Items, Hardware Settings, and Maintenance tabs be marked as "Current" because they cannot be changed on the Sample Input form. "Apply" places a created, new copy of a tab into memory and returns to the Configuration Tabs form. "OK" places any newly created copy of a tab into memory and exits the Configuration Tabs form.

### General Items

The General Items tab contains two frames: options for reporting immediately after analysis of a sample, and options for login and passwords. Display of results in scientific notation is recommended. User IDs are recommended to permit KPAWin© to "remember" user preferences that are reloaded when the individual logs back onto the system.

### Hardware Settings

Hardware Settings are critical to the operation of the system. Select the preloaded copy of the tab according to the model of your KPA, the EPROM version, and the presence or absence of an autochanger. The index of the release version is 1000 for a KPA-11A. All other six configurations have the Inactive Box checked. Configurations are preloaded by KPA model (KPA-11/KPA-10). KPA EPROMs are (KPA-11 (3), KPA-10 Auto shutter (2), or KPA-10 Manual shutter (1)). Autochanger (A) models and Manual (M) models are also specified.

<u>Index</u>	<u>Model</u>	<u>EPROM</u>	<u>Auto/Manual</u>
994	KPA-10	1	Auto
995	KPA-10	2	Auto
996	KPA-11	3	Auto
997	KPA-10	1	Manual

998	KPA-10	2	Manual	
999	KPA-11	3	Manual	
1000	KPA-11	3	Auto	(Release default)

### **KPA Unit Settings**

The KPA Unit Settings tab contains the parameters that control the KPA and qualify the raw intensity data that it generates. Only the Pulses Per Analysis is likely to be changed in the Laser frame. The parameter has the greatest impact on the signal to noise ratio of any KPA setting. As such, it dominates the detection limit with increasing pulses providing lower analytical detection limits.

The Detection frame provides three parameters that are used in the interpretation of intensity data. PMT Dead Time (ns) is used to correct the raw intensity counts in a data channel that was acquired at high photon flux levels. At low photon acquisition rates, the logarithm of intensity is directly proportional to the analyte concentration. As the photon flux increases, the photomultiplier switches on and off to limit saturation of the detector. This behavior is normal but creates curvature in the logarithm of intensity versus concentration data at higher concentrations. The photomultiplier manufacturer recommends the dead time correction factor. KPAWin® applies the factor to the raw data to create coincidence-corrected data. A dead time of 10 ns is used for all KPA-11 instruments. The dead time for KPA-10 instruments is photomultiplier specific but typically ranges from 120 to 140 ns.

The Maximum Intensity Factor/Pulse is used to limit the influence of the coincidence error. During data analysis by KPAWin®, low-order time gates are ignored if the intensity per laser pulse exceeds the configuration tab setting. Gate Shifting is the term that is used to describe this phenomenon. Settings for a KPA-10 are typically from 50 to 60. For a KPA-11, values from 180 to 220 can be selected based on user preference.

The Minimum Intensity Factor is used to disregard high-order time gates that contain an insignificant number of intensity counts accumulated for all laser pulses in the analysis. The minimum acceptable value for this parameter is "1" to preclude the program from taking logarithms of negative numbers. Gate Dropping is the term this is used to describe this procedure.

The Time Gates frame contains default specification. Total Time Gates is standard at 128. Time Gate Width (μs) is 13 μs for uranium analysis and shorter for the lanthanides as described in the KPA instrument manual. The Start LSR Gate and Number of LSR Gates are preferred values that can be overridden by either Gate Shifting for the Start Gate or Gate Dropping for the Number of Gates prior to the Least Squares Regression of the intensity and decay time data. The Minimum Number LSR Gates does not interfere with the data interpretation or reporting. This minimum specifies a user tolerance that is reported as an alarm or warning with the data.

The Range Selection frame specifies the parameters that are used to allow auto range selection during analysis and to qualify sample data based on the relevance of calibration standard concentrations to sample concentrations. The Auto Efficiency Factor is an estimate of the intensity intercept of a sample analyzed in the low range to the intercept of the same sample analyzed in the high range. The factor is best evaluated in the middle of the crossover between the two ranges since the number decreases with increasing sample concentration. The factor is used to evaluate a precount test of a sample that is analyzed in the high range. If the high-order time gates do not Gate Drop then the sample can be analyzed in the high range. If the low-order time gate multiplied by the factor does not Gate Shift, then the sample can be analyzed in the low range. If both ranges are qualified, the range of the most recently analyzed sample is used.

The High Intercept Tolerance and the Low Intercept Tolerance compare concentrations of standards at the extremes of the calibration to the concentrations of analytical samples. If the concentration of a sample exceeds the concentration of the most concentrated standard in the range by the factor of the High Intercept Tolerance, the data results are alarmed. If the concentration of a high-range sample is less than the least concentrated standard for the high range by the factor of the Low Intercept Tolerance, the data results are alarmed.

### **Calibration Settings**

The Calibration Settings specifies calibration, units, and intensity vector arithmetic preferences. The default database units of μg/l can be changed to an alternative user global default for data entry and reporting. The units can subsequently be changed at the time of sample input on a sample-by-sample basis. Specific gravity, specific activity and atomic mass

specifications on this tab provide a global capability to use per mass units, radiometric units, and molar units. These can also be changed on a sample-by-sample basis at the time of sample input.

Calibration range can be Auto, High, Low, or Both. If calibrations are performed in the high and low ranges during sample analysis, the global default selected on this tab can be changed on a sample-by-sample basis at the time of sample input. Auto uses precounts to qualify analysis in the high and/or low range with the High Intercept Tolerance and Low Intercept Tolerance used only for reporting flagged alarms. The High, Low, and Both options use these tolerances to "switch" to another calibrated range if an analysis fails to meet a tolerance. These three manual calibration options also flag alarms when a tolerance test fails.

Dilution data can be entered as Total Volume or Add Volume. These apply to both dilution at the time of sample preparation and aliquot dilution at the time of analysis. In both cases, the initial volume is the actual initial volume. For Total Volume, the final volume is the total volume of the dilution. For Add Volume, the final volume is the volume that is added to the initial volume to make the final dilution.

Four corrections are made to the intensity data by time gate before the data are regressed to provide an intensity intercept: coincidence correction, data smoothing, reference correction, and background subtraction. Coincidence correction is a required vector arithmetic operation since it accounts for the non-linearity of the photomultiplier output with concentration. Any of the other three can be removed from the intensity vector interpretation at the discretion of the operator. See the KPA operator's manual for a description of these corrections.

Select the form of the calibration for the high and low range based on the order of the equation, allowance for non-zero intercept, and the minimum number of calibration standards.

The above configuration provides an installation that is equivalent to the DOS software for the KPA. The remaining four configuration tabs can be included in your configuration to provide control and quality reporting capabilities.

## KPAWin© Demo Analyst/Operator Script

This script familiarizes a new KPAWin© user possessing Standard privileges with some of the functions and features of the KPAWin© software. **A KPA unit is not required to complete the actions in this script.** The script provides the necessary steps to exercise most functions required by a standard User (Analyst/Operator) running the KPAWin© software. To start the KPAWin© software use either the Windows Start button or double click on the desktop shortcut (if created). KPAWin© begins to initialize and stops at the Login Sequence.

**Note:** KPAWin© Options that are displayed in "**Black**" are Available to the user while those that are displayed in Light Grey, "**Grayed Out**" are NOT Available.

### **I. Administration Demonstration**

#### Database Operations

1. Use the Database Dropdown menu to attach and open the KPADemo Database.
2. At the Top of the KPAWin© display, all dropdown menu's are listed. These include **File**, **Security**, **Database**, **Reports**, **Analyze**, **Settings**, **Tools**, **Window**, and **Help**. Click on "**Database**" and then select (highlight) and click the "**Open**" item.
3. The "Looking for KPA Database File" menu is displayed. Select (highlight) the **KPADemo** file and click the "**Open**" button. The Demo database contains information to demonstrate the software features and functions. It is only used for demonstration purposes.

#### Login Procedures

1. Click the first Toolbar (**Login**) button.
2. There are four UserIDs registered in the Demo Database (**Analyst**, **MXF**, **Supervisor**, & **WRM**). No Password is required for the Demo User ID "**Analyst**". The default UserID is Analyst. Click the **OK** button to complete the Login sequence.

#### Help Demo

1. Click the "**Help**" dropdown menu and then click the "**Contents**" item. This displays the KPAWin© Help **Table of Contents** listing all sections with Online Help information available. Click on any item of interest and browse as completely as desired.
2. With the Help menu displayed, click on the "**Index**" Toolbar button to display the list of available topics sorted alphabetically for perusal. Select one or more items and click the "**Display**" button at the bottom of the menu. Close the Help display when finished.
3. KPAWin© utilizes "**Context Sensitive Help**" on all displays. Context sensitive help provides immediate help with a screen element of interest (menu command, dialog box, etc.). The program takes you directly to the help information you seek without the need to traverse the help indexing system.
4. To get context sensitive help on a display **Label** or **Input Text** item, select the label or text box in question with the mouse pointer and then press F1 while the box is active. This action displays the Help information for that item.
5. To get context sensitive help on a specific **Menu Command** place the mouse pointer over the command button (**DO NOT** click the command button as the command will be executed) and press F1.



## II. MainDisplays (Toolbar/Misc. Dropdown Menu's)

### Toolbar Buttons

The KPAWin© main Toolbar contains several functions used extensively throughout the analysis process. Prior to Logging onto the KPAWin© software, only the Login, Stop/Exit, and Help buttons are available. Once a user has successfully Logged in, Logout, Stop/Exit, Analyze, Reports, Configuration, Initialization, Tickler, and Help buttons are on display. When the mouse pointer is placed over a button, text indicating the function of that button appears for approximately 5 seconds.

<b>Logout</b>	Logs the current user out but KPAWin© continues to run.
<b>Exit</b>	Exits KPAWin© and returns to the operating system.
<b>Analysis Input -</b>	Displays the Input Analysis display..
<b>Sample Reports</b>	Displays the Sample Selection display.
<b>Calibration Tabs</b>	Displays the Calibration Tabs display.
<b>Initiallize KPA -</b>	Displays and Executes the Initialization process.
<b>Tickler Menu</b>	Displays the Maintenance Configuration Tab.
<b>Help Information</b>	Displays the Help Contents.

### Dropdown Menu's

Several of the items on the Dropdown menus are demonstrated earlier in this script. They are not addressed again.

### File

**Open** - Enabled only when the Sample Input screen is on Display. Retrieves Sample Input information previously saved template.

**SaveAs** - Saves the Sample Input information (template data) to a filename of your choice.

**Exit** - Exits the KPAWin© software

### Database

**Open/Repair/Pack** are all database functions designed to manipulate the KPAWin© database.

**Archive** removes data from the current database and stores it on other media (e.g., another Hard drive, Tape, CDROM, Floppy disks).

**Administration Functions** are discussed later.

### Reports

**Selection** - Displays the Sample Selection Report form. These functions are demonstrated later in the script.

**Trend** - Displays the Trend report.

**Analyze** is discussed in the section on Sample Input.

**Settings** displays the Configuration Tabs and is discussed in the Configuration section.

### Tools

**Auto Changer** has two submenu items, Reset and Rinse. Use Reset and Rinse only with an Autochanger

**Diagnostics** has a Probe KPA Unit submenu. It attempts to communicate with the KPA hardware and KPAWin© database. If not successful, an Error message is displayed indicating the probable cause and offering an opportunity to retry the operation. Click the "Ignore" button if a KPA unit is not connected to your PC.

**Calculator** invokes the system calculator function.

### III. Sample Input Display

Click on either the **Analyze** dropdown menu followed by clicking the **Sample Input** item or use the Toolbar button **Analysis Input Form** (Third toolbar button from the Left). This displays and loads all **Entered and Processed but Unaccepted Samples** from the database. The Demo Database has a total of 36 Processed Samples with at least one example of all 19 Sample types. The Demo script allows the user to create more Sample Input entries. Note the display is divided into two sections, an upper portion with Labels and Input Text areas and a lower Grid display portion. Data may be entered in either section.

#### Loading Sample Data from Database

1. Click on the far left Grid Column labeled **"Analysis"** and highlight Analysis # 1 (SampleID = 1002, Sample Type = BckGnd.) Note that several labels in the upper portion of the display have changed color. Red Indicates the item is **Required to be filled in**. Blue indicates an **Optional item**. Black indicates the data is taken from a **Basis Sample** and Grayed Out items are **Not Available** and therefore not applicable to this sample type. Each sample type requires a unique set of Required, Optional, Basis, and Not Applicable Sample Input items. The color code guides your input. All Required items must be entered before attempting to enter or change another Sample. The software displays an Error message describing any missing information.
2. Now click Analysis # 9 (SampleID = 1010, Sample Type = IchkStd) and again notice the color code changes to a number of fields in the upper portion of the display. The fields Standard ID and Standard Concentration are now colored Red while Calibration Config and Concentration Units are Grayed Out.
3. Change the **"Standard ID"** from **S-1** to **S-2** in either the upper portion of the display or on the Grid (Data may be entered in either section.)
4. To collapse any of the fields in the grid section of the form, place your mouse pointer on the Label Text in the upper portion of the display and double click. Try the **Concentration Units** field in the by double clicking on the label "Concentration Units". The Sample Units field in the grid completely collapses. To expand the field again double click the Label again and the Grid Header expands. All collapsed Grid fields are saved for each user in the operating systems Register file. Each time the user logs onto KPAWin®, all saved display preferences are reinstated. These preferences remain until the user changes them. Each user customizes these fields to their own preference.

#### Sample Input Toolbar

##### Saving / Restoring Sample Entries

1. Users enter Sample data into the Sample input form and Save it to a System file before analyzing the samples. This is very useful if a Laboratory runs the same or similar set of samples on a reoccurring basis.
2. Enter the set of Sample data in the Sample Input form and Highlight the samples to be saved in the database.
3. Click the **"Save As Initial Samples"** Toolbar button (the picture of a floppy disk) and the Save menu is displayed. Key in the name for the selected Sample Input Data and click **"Save"** to save selected Samples. Click the **"SaveAll"** button to save all Samples on the Input Screen.
4. Click the **"Open"** Toolbar Button (the picture of an open file folder) and the **"Select Sample to Load"** menu is displayed. Highlight the Sample name and click the **"Load"** button. These Samples are loaded into the Grid from the database.

### Adding a New Sample

1. Click the **"Add New Sample"** Toolbar button (picture of plus sign "+"). Either the Highlighted Sample or if nothing is highlighted, the last Sample in the Grid is duplicated. However the Sample ID is concatenated with a number or the last number in the ID is incremented. The same rules apply to the Sample Description.
2. The user can change any of the displayed data items that are NOT Grayed out.
3. Users can also add a selected Sample using the **"Insert Before"** or **"Insert After"** feature. Highlight any sample by clicking the number in the **Analysis Column** to select the sample and then click the **"Right Mouse"** button to display the **"Input Pop-Up Menu"**. Select either the **"Insert Before"** or **"Insert After"** command and the highlighted sample is duplicated and placed in the desired order.
4. The **"Input Pop-Up Menu"** contains a considerable number of easy to use commands that you can exercise as desired. Some of these commands such as **Clear**, **Delete**, and **Analyze** are also executed by using the buttons on the Input Toolbar.
5. Several commands such as **"Accept"**, **"Reject"**, **"Reprocess"**, **"Calibrate"**, **"Insert Before"**, **"Insert After"**, and **"Move"** are only executed by using the Pop-Up menu.

### Calibration Display

1. Placing the cursor over the first column "Analysis, right click and the Pop-Up menu will appear. Highlight the the **"Calibrate"** command and Left click. This will force a calibration using the processed samples on the Sample Input Form and displays all the calibration data (both range data is displayed if there is data for both ranges).
2. Users may elect to disable the automatic calibration process by Checking the **"User Cal"** checkbox in either range. When checked, the user provides a name for the calibration and selects or deselects any specific calibration samples by **"Double Clicking"** on the specific sample. The "+" sign disappears when the sample is deselected and reappears if selected again. This method allows the user to select specific samples to be used in the calibration.
3. After selecting the samples you desire to be included in the calibration, click the Calibrate command button located on the right of the display to re-calibrate.
4. Plots are also available for display by clicking the **"Plot"** command button.
5. Printing the Calibration details or Plots is accomplished using the **"Print"** command button.
6. The **"Stop"** Toolbar button exits the Input display and returns to the previous display.

#### IV. Displays and Reports

Click on the dropdown **Reports**, then the **Selection** item or click on the **Sample Reports** Toolbar button to display the **Sample Report Selection** form. This display is divided into two sections. The upper portion contains all the selection options while the lower portion lists the available samples from the database after the "**Retrieve**" button is clicked. All user selected items are color-coded **Blue** and the KPAWin© defaults are selected when the form is initially displayed. The display design allows users a variety of options in selecting Samples from the database (e.g., selection by **Sample ID**, **Sample Type**, **Analyst**, **Customer**, **Procedure**, or **Date Processed**, etc.) as well as the type of display used to report that selection (e.g., **Single** or **Multiple Sample Display** reports, **Single** or **Multiple Print** reports, etc.) This script only uses a few of the available selection criteria. However you can employ as many as you desire.

Selecting the Fields options that will be displayed in the lower portion of the report **MUST** be completed prior to Retrieving the actual Sample from the database.

1. Operator/Analysts cannot change the fields that have been selected by a Supervisor. If you desire different fields in this display, contact a user with Supervisor privileges.

Selecting Samples from the database is accomplished by using the various selection options on the top portion of the display.

1. In the "**Sample Type**" frame, use the dropdown display to select the "**CalStd**" sample type. Notice that the Radio (round) Button in the frame changes from **ALL** to **AND**. Now click the **Retrieve** button to load the data from the database. All the CalStd type samples meeting the other selection criteria are extracted from the database and displayed according to your selection order in the lower portion of the screen. You can try other selection criteria at your discretion.

##### On Screen Display Options - Single Sample Displays

1. In the "**Sample Type**" selection frame, click the "**All**" button and then the "**Retrieve**" button again. This displays ALL of the samples in the database.
2. To view one of the Samples, highlight the desired sample by clicking in the First column, in the Demo Database example the first column is labeled "**Sample ID**".
3. Using the scroll bar on the right of the display, scroll to the Sample ID "**1027**" and highlight this sample. Using the KPAWin© default display options **Display Reports** and **Single Display**, click the "**Display Reports**" command button. This displays the results of the sample. Click the lower "**Stop Sign**" toolbar button or the X in the upper right hand corner of the display when finished viewing the report. (Note: there are two sets of window buttons in the upper right hand corner of the display, the lower set is for the Single Sample report you are viewing while the top set controls the KPAWin program. Clicking the X in the lower set will close the Single Sample Report while clicking the X in the upper set will close the KPAWin© program)
4. Now highlight another sample, then click the "**Display**" command to view these results. This displays the single sample display for the selected sample. Both the **Sample Intensities** and **Plots** are also available from this display.
5. To view the **Sample Plots**, click the first button on the Sample Display Toolbar (lower toolbar - picture of a graph). A frame is displayed depicting the Plot options with the default of "**Net Data**" (change the display selection to any option desired); then click the Display Plots button. When through viewing the Plots, click the "**X**" in the extreme upper right corner of the Plot display and then the **Cancel** command on the Plot frame.
6. To view the **Sample Intensities**, click the second button on the Sample Display Toolbar (lower toolbar - picture of a report). A frame is displayed depicting the Intensity options with the default of "**Net Sample Data**" (change the display selection to any option desired); then click the Display



Intensities button. When through viewing the Intensities, click the "X" in the extreme upper right corner of the Intensity display and then the **Cancel** command on the Intensity frame.

7. Resize the Single Sample display, if required, and click the **"Stop"** Toolbar button to return to the Select Sample display when through viewing the report.

#### **On Screen Display Options - Multiple Sample Displays**

1. To select Sample for the Multi Display, select all the samples on display by highlighting the first one, then the last sample while holding down the **Shift** key. Now click the **"Multi Display"** radio (round) button, (note that all the Multi Display options are now enabled), select the **"Sample Analysis"** option, and click the **"Display"** command. This displays the **Multi Sample Analysis** report containing one line of data for each sample. Use the Scroll Bar at the bottom of the report to view all the data. When through viewing the report, click the "X" in the extreme upper right corner of the **Multiple Sample Analysis Report** (Located approximately Mid Screen on the right edge) to return to the previous display.
2. Using all the above selection criteria, click another report radio button (e.g., **"Quality Control"**) and click the **"Display"** command to view other multiple sample displays.

#### **Print Report Options - Single Sample Reports**

1. In the **"Print Report Option"** frame, click the **"Single Sample"** radio button. All the MultiReport checkboxes are disabled. The Calibration and Configuration selections remain active. The Details option is also available when printing Single Sample reports.
2. Highlighting one sample at a time, select various sample types (e.g., BckGnd, Sample, MtdStd, SASamp, SampSpk, etc.). Then click the **"Print"** command button. Although all Single Sample Print Reports have many items in common, each sample type produces a slightly different report.
3. Check as many report types as desired to become familiar with the various reports.

#### **Print Report Options - Multiple Sample Reports**

1. In the **"Report Options"** frame, click the **"Print Reports"** radio (round) button. This changes the **Monitor Display Options** to **Print Report Options**. Click the **MultiReport Option**, if not already selected, and there are four enabled checkbox reports and two disabled items. The disabled items (Calibration and Configuration) are available when one of the MultiReport options is selected.
2. Select two or more of the samples on display by highlighting them. Then check one of the report options, use **"Analytical Results"** and the two additional checkboxes are available. If checked, these additional checkboxes produce additional **Calibration** or **Configuration** reports. Detailed reports are obtained by checking the **"Details"** checkbox to the right of the Calibration and Configuration report options.
3. Check as many report options as desired to become familiar with the various reports.

This concludes the familiarization script. If additional users wish to exercise this script, the original KPADemo should be reloaded to remove any changes made while performing the various exercises in the script.

## **"KPA Sample Types"**

### **Summary Description**

The KPAWin® software recognizes 19 unique sample types. Collectively they provide the analyst with a system to accumulate and evaluate interactively all of the luminescence-specific and general-laboratory evaluations associated with sample analysis and analytical performance. Sample Input is designed to guide the analyst by identifying the minimum input data set for each unique sample type. Required data fields are labeled in red and on-screen warnings are displayed when critical information is not provided. The display and reporting for each Sample Type is designed to minimize secondary data handling by the analyst.

#### **1. Background - "BckGnd"**

"BckGnd" is the designator for the background sample. Raw intensity vectors are corrected for coincidence and smoothed. The smoothed background intensity vector is used to correct the intensity vectors of most sample types for background contributions. Only Standard Addition Samples and Standard Addition Spikes, Confidence Tests, and Quench-Corrected Sample vectors are not corrected for background if the background correction is set to true. If it is set to false then no samples are background corrected. The intensity intercept for the reference that is recorded during the analysis of the background sample is defined as the "Reference Mark" ( $R_M$ ). For all succeeding samples in the analytical batch of the background, a reference ratio is computed from the intercept intensity of the reference ( $R_I$ ) and  $R_M$  as the ratio  $R_I / R_M$ . Unless the option "Reference Corrected Data" is deselected on the Calibration Configuration Tab, accumulated intensity in the smoothed intensity vector of the sample is corrected for reference drift by division by the reference ratio.

#### **2. Calibration Standard - "CalStd "**

"CalStd" is the designator for a calibration standard. Data are entered by the analyst and reported by KPAWin® to indicate the laboratory control Identifier for specific standards as well as their concentrations and the volumes in ml or masses in g. Concentrations are based on the laboratory's preference indicated in the Calibration Configuration Tab which can be amended in the Sample Input grid. The definition of amount on a /volume or /mass basis is consistent with the selected concentration units.

#### **3. Continuing Check Blank- "CChkBk"**

"CChkBk" is a deionized water or analyte-free laboratory blank and is the designator for the continuing check blank. The blank is used to verify the calibration subsequent to the initial verification. The acceptable performance of the blank is specified in the Quality Control Limits Configuration Tab as a multiple of the instrument detection limit (IDL).

#### **4. Continuing Check Standard - "CChkStd "**

"CChkStd" is an analyte standard in deionized water or analyte-free laboratory sample and is the designator for the continuing check standard. The standard is used to verify the calibration subsequent to the initial verification and should be prepared from a traceable standard independent of the calibration standards. The acceptable performance of the blank is specified in the Quality Control Limits Configuration Tab as a standard recovery error.

#### **5. Confidence Tests - "ConfTst"**

"ConfTst" is the designator for a confidence test. The test provides the coefficient of variation of a series of analyses on the same sample under identical analytical conditions. These results demonstrate the stability of the analytical system.

#### **6. Initial Check Blank - "IChkBk"**

"IChkBk" is a deionized water or analyte-free laboratory blank and is the designator for the initial check blank. The blank is used to verify the calibration immediately following the calibration. The acceptable performance of the blank is specified in the Quality Control Limits Configuration Tab as a multiple of the instrument detection limit (IDL).

#### **7. Initial Check Standard - "IChkStd"**

"IChkStd" is an analyte standard in deionized water or analyte-free laboratory sample and is the designator for the initial check standard. The standard is used to verify the calibration immediately following the calibration and should be prepared from a traceable standard independent of the calibration standards. The acceptable performance of the blank is specified in the Quality Control Limits Configuration Tab as a standard recovery error.

**8. Instrument Detection Limit - "IDL"**

"IDL" is the designator for the instrument detection limit sample that consists of an analyte standard in deionized water or an analyte-free laboratory blank. The Instrument Detection Limit is an estimate of the minimum sample concentration that can be detected with a 99% confidence. The calculation is based on a Student's t-test with all input parameters selected in the detection limit frame of the Quality Control Limits Configuration Tab. The parameters include number of standards, detection limit type, maximum duration in weeks, maximum concentration factor, and limit in  $\mu\text{g/l}$ . Detection limit type is a KPAWin® term to distinguish between detection limits based on the standard deviation of a series of individual analyses (Type 1) and the average of a series of standard deviations of a series of analyses made in triplicate (Type 2).

In operation, an IDL is analyzed according to a frequency established by laboratory protocol. KPAWin® pools all IDL results and estimates the detection limit for every day of KPA operation based on the measurements made within the previous number of weeks specified in the maximum duration specification. This detection limit is reported with the data recorded for the day of operation and is used to evaluate the performance of IChkBlk and CChkBlk samples. Errors are reported if the number of standards (or standard deviations for Type 2 daily standards) is insufficient, if the true average concentration of the standards (makeup concentrations) is greater than the specified multiple of the detection limit, or if the value of the detection limit exceeds the user-defined limit.

**9. Method Detection Limit - "MDL"**

"MDL" is the designator for the method detection limit sample consisting of an analyte standard in deionized water or an analyte-free laboratory blank processed by the procedure employed for samples. The Method Detection Limit is an estimate of the minimum sample concentration that can be detected with a 99% confidence. The calculation is based on a Student's t-test with all input parameters selected in the detection limit frame of the Quality Control Limits Configuration Tab. The parameters include number of standards, detection limit type, maximum duration in weeks, maximum concentration factor, and limit in  $\mu\text{g/l}$ . Detection limit type is a KPAWin® term to distinguish between detection limits based on the standard deviation of a series of individual analyses (Type 1) and the average of a series of standard deviations of a series of analyses made in triplicate (Type 2).

In operation, an MDL is analyzed according to a frequency established by laboratory protocol. KPAWin® pools all MDL results and estimates the detection limit for every day of KPA operation based on the measurements made within the previous number of weeks specified in the maximum duration specification. This detection limit is reported with the data recorded for the day of operation and is used to evaluate the performance of the current MtdBlk sample. Errors are reported if the number of standards (or standard deviations for Type 2 daily standards) is insufficient, if the true average concentration of the standards (makeup concentrations) is greater than the specified multiple of the detection limit, or if the value of the detection limit exceeds the user-defined limit.

**10. Sample Duplicate - "SampDup"**

"SampDup" is the designator of a duplicate sample. The sample is prepared for analysis and analyzed precisely analogous to the basis sample. Acceptable performance is specified in the Sample Control frame of the Quality Control Configuration tab as the relative percent deviation (%RPD).

**11. Sample Spike - "SampSpk"**

"SampSpk" is the designator of a spiked sample. The sample is prepared for analysis and analyzed precisely analogous to the basis sample. The volume of the spike addition replaces a portion of the deionized water used to prepare the final analytical sample. Acceptable performance is specified in the Sample Control frame of the Quality Control Configuration tab as the spike recovery.

**12. Quenched Sample - "QchSamp"**

"QchSamp" is the designator of a quenched sample. The sample is an aliquot of the basis sample that was prepared for analysis and diluted and analyzed precisely analogous to the basis sample. A volume of quenchant, typically 0.1 ml of methanol, replaces a portion of the deionized water used to prepare the final analytical sample. The methanol provides a non-spectroscopic, thermal pathway for relaxation of the excited uranium complex and the resulting intensity vector reports the non-uranium component present in the basis sample. The smoothed intensity vector of the quenched sample replaces the smoothed background vector in the estimate of the net intensity vector. A user-request for a report of the basis sample alone provides the standard report with sample intensities corrected for the batch background. Alternatively, a user-request for a quenched sample report provides a report with sample intensities corrected for the quenched intensities.

**13. Sample - "Sample"**

"Sample" is the designator of a matrix sample. The Sample Id of this unknown can be the Basis Id for other sample types that are evaluated and reported with this sample by KPAWin®. The sample types linked with this sample type are SampDup, SampSpk, QchSamp, SerDil, and MtdSA.

**14. Method Blank - "MtdBIK"**

"MtdBIK" is the designator for the method blank that consists of deionized water or analyte-free laboratory matrix that has been processed by the procedure employed for samples. The acceptable performance of the blank is specified in the Method Control frame of the Quality Control Limits Configuration Tab. It is expressed as a multiple of the method detection limit (MDL), the percentage of the contract reporting detection limit (CRDL), the percentage of the sample concentration, or the largest of the aforementioned three conditions.

**15. Method Standard Addition - "MtdSA"**

"MtdSA" is the designator of analyte-spiked samples quantitating the analyte concentration in the basis sample and the interferences to that quantitation. Spiked samples are prepared for analysis and analyzed precisely analogous to the basis sample. The volume of the spike addition replaces a portion of the deionized water used to prepare the final analytical sample. The parameters that control the interpretation of MtdSA data are contained in the Sample Control frame of the Quality Control Limits Configuration Tab under the Method of Standard Additions. These parameters are Number of Spikes, Recovery Error (%), Slope Error (%), and  $R^2$  (correlation coefficient). The value for Number of Spikes can be either one (1) or three (3).

A single MtdSA sample provides quantitation and Recovery Error (%) from the external batch calibration. Triplicate MtdSA samples with the basis sample provides an internal calibration by a linear least-squares regression of the spike concentrations versus the increment of the intensity intercept that is attributed to the spike (Intercept of a spiked sample minus intercept of the basis sample). The negative value of the concentration intercept is the reported concentration of the analysis.  $R^2$  is the correlation coefficient of the internal calibration least-squares regression. Recovery Error (%) is the difference between the analyte concentrations for the basis sample for the internal-MtdSA calibration and the external calibration divided by the result from the internal calibration when expressed as a percent. Slope Error (%) is the difference between slopes for the internal-MtdSA calibration and the external calibration divided by the slope from the internal calibration when expressed as a percent.

**16. Method Standard - "MtdStd"**

"MtdStd" is the designator for the method standard that consist of an analyte standard in deionized water or analyte-free laboratory sample that has been processed by the procedure employed for samples. The acceptable performance of the blank is specified in the Method Control frame of the Quality Control Limits Configuration Tab as a standard recovery error.

**17. Standard Addition Sample - "SASamp"**

"SASamp" is the designator for the sample analyzed by standard addition. Standard addition quantitates the analyte in a basis sample by internal calibration. The process compares the incremental increase of intercept intensity from this basis sample to a spiked sample to the incremental analyte concentration of the spiked sample. The quantum efficiency factor is used to estimate the analyte concentration in the basis sample. Neither the basis sample nor its spike requires a background or an external batch calibration.

**18. Standard Addition Spike - "SASpk"**

"SASpk" is the designator for the analyte-spiked sample analyzed by standard addition. Standard addition quantitates the analyte in a basis sample by internal calibration. The process compares the incremental increase of intercept intensity from this basis sample to a spiked sample to the incremental analyte concentration of the spiked sample. The quantum efficiency factor is used to estimate the analyte concentration in the basis sample. Neither the basis sample nor its spike requires a background or an external batch calibration.

**19. Serial Dilution - "SerDil"**

"SerDil" is the designator of a serial dilution of a sample. The sample is prepared for analysis and analyzed precisely analogous to the basis sample. Additional deionized water prepares the final analytical sample so that a five-fold dilution of the basis sample is created. Acceptable performance is specified in the Sample Control frame of the Quality Control Configuration tab as the serial dilution error.



## KPAWin© Demo Supervisor Script

This script familiarizes a new KPAWin© user possessing Supervisor privileges with the functions and features of the KPAWin© software. **A KPA unit is not required to complete the actions in this script.** The script provides the necessary steps to exercise most standard functions required by a supervisor maintaining the KPAWin© software configurations. To start the KPAWin© software use either the Windows Start Taskbar button or double click on the desktop shortcut (if created). KPAWin© begins to initialize and stops at the Login Sequence. If additional users wish to exercise this script, the original KPADemo should be reloaded to remove any changes made while performing the various exercises in the script.

**Note:** KPAWin© Options that are displayed in "Black" are Available to the user while those that are displayed in Light Grey, "Grayed Out" are NOT Available.

### **I. Administration Demonstration**

#### Database Operations

1. Use the Database Dropdown menu to attach and open the KPADemo Database.
2. At the Top of the KPAWin© display, all dropdown menu's are listed. These include **File, Security, Database, Reports, Analyze, Settings, Tools, Window, and Help**. Click on "Database" and then select (highlight) and click the "Open" item.
3. The "Looking for KPA Database File" menu is displayed. Select (highlight) the **KPADemo** file and click the "Open" button. The Demo database contains information to demonstrate the software features and functions. It is only used for demonstration purposes.

#### Login Procedures

1. Click the first Toolbar (Login) button.
2. There are four UserIDs registered in the Demo Database (**Analyst, MXF, Supervisor, & WRM**) - The Password for the Demo Supervisor UserID is "Supervisor". Once you add additional Users to the KPAWin© database, you can select any UserID. Only those listed with Supervisor privileges can perform the Administration functions within KPAWin©. It is recommended you remain Logged in as "Supervisor".
3. After a site is through exercising the Supervisor Script and adding their supervisors to the KPAWin© Database, change the status of the UserID "Supervisor" to **Inactive**.

#### User Administration

1. **Adding KPAWin© Users** to the database.
2. Click on the "Security" dropdown menu item and then click "User Administration". This displays the User Administration Menu.
3. Click the "Add" button. Fill in all fields with fictitious information. Remember this is the Demo Database and none of the information entered is used. Passwords are **NOT** required by KPAWin©. They are entered only if the site requires their use.
4. Click the "Apply" button and repeat Steps 3 and 4 to add additional Users. Enter as many new Users as desired for practice.
5. Click the "Select" button and the User List is displayed with a dropdown list of all users in the database. Highlight any one of the Users. You can now Edit the displayed information (e.g., make the User Active/Inactive, change the Password, etc.) and then click the OK or Cancel button. A Supervisor is the only administrator that can enter or change a User Password. If more stringent password security is required, consider using the Microsoft NT operating system. The OK button exits the User Administration menu and returns to the KPAWin© Main display.

### **Customer Administration**

1. Click the **"Database"** dropdown menu and then click the **"Customer Administration"** item. This displays the Customer Administration Menu.
2. Repeat Steps 3 through 5 from the **User Administration** section to exercise these entries and add one or more Customers to the Demo database.

### **Procedure Administration**

1. Click the **"Database"** dropdown menu and then click the **"Procedure Administration"** item. This displays the Procedure Administration Menu.
2. Repeat Steps 3 through 5 from the **User Administration** section to exercise these entries and add one or more Procedures to the Demo database.

### **Laboratory Administration**

1. Click the **"Database"** dropdown menu and then click the **"Laboratory Administration"** item. This displays the Laboratory Administration Menu.
2. Repeat Steps 3 through 5 from the **User Administration** section to exercise these entries and add your Laboratory to the Demo database.

### **Units of Measure Administration**

1. Click the **"Database"** dropdown menu and then click the **"Units Administration"** item. This displays the Units of Measure Administration Menu.
2. Click the **Select** button and Browse the Units Database. Most available Units are already entered into the database. You change the report default Units using the **"Apply"** button.

### **Help Demonstration**

1. Click the **"Help"** dropdown menu and then click the **"Contents"** item. This displays the KPAWin© **Help Table of Contents** listing all sections with Online Help information available. Click on any item of interest and browse as completely as desired.
2. With the Help menu displayed, click on the **"Index"** Toolbar button to display the list of available topics sorted alphabetically for perusal. Select one or more items and click the **"Display"** button at the bottom of the menu. Close the Help display when finished.
3. KPAWin© utilizes **"Context Sensitive Help"** on all displays. Context sensitive help provides immediate help with a screen element of interest (menu command, dialog box, label, or text item, etc.). The program takes you directly to the help information you seek without the need to traverse the Help Indexing system.
4. To get context sensitive help on a display **Label** or **Input Text** item, select the label or text box in question with the mouse pointer and press F1 while the box is active. This action displays the Help information for that item.
5. To get context sensitive help on a specific **Menu Command** place the mouse pointer over the command button (**DO NOT** click the command button as the command is executed) and press F1.

## II. Main Displays (Toolbar/Miscellaneous Dropdown Menu's)

### Toolbar Buttons

The KPAWin© main Toolbar contains several functions used extensively throughout the analysis process. Prior to Logging onto the KPAWin© software, only the Login, Stop/Exit, and Help buttons are available. Once a user has successfully Logged on, Logout, Stop/Exit, Analyze, Reports, Configuration, Initialization, Tickler, and Help buttons are on display. When the mouse pointer is placed over one of the buttons, text indicating the function of the button appears for approximately 5 seconds.

<b>Logout</b>	Logs the current user out but KPAWin© continues to run.
<b>Exit</b>	Exits KPAWin© and returns to the operating system.
<b>Analysis Input -</b>	Displays the Input Analysis display.
<b>Sample Reports</b>	Displays the Sample Selection display.
<b>Configuration Tabs</b>	Displays the Configuration Tabs display.
<b>Initialize KPA -</b>	Displays and Executes the Initialization process.
<b>Tickler Menu</b>	Displays the Maintenance Configuration Tab.
<b>Help Information</b>	Displays the Help Contents.

### Dropdown Menu's

Many items on the Dropdown menus are demonstrated earlier in this script and are not addressed again.

### File

**Open** - Enabled only when the Sample Input screen is on Display. Retrieves Sample Input information previously saved.

**SaveAs** - Saves the Sample Input information to a file of your choice.

**Exit** - Exits the KPAWin© software.

### Database

**Open/Repair/Pack** are all database functions designed to manipulate the KPAWin© database.

**Archive** removes data from the current database and stores it on other media (e.g., another Hard drive, Tape, CDROM, Floppy disks).

**Administration Functions** are discussed above.

### Reports

**Selection** - Displays the Sample Selection Report form. This is demonstrated later in the script.

**Trend** - Displays the Trend report.

**Analyze** is discussed in the section on Sample Input.

**Settings** displays the Configuration Tabs and is discussed in that section.

### Tools

**Auto Changer** has two submenu items, Reset and Rinse.

**Diagnostics** has a Probe KPA Unit submenu. It attempts to communicate with the KPA hardware and KPAWin© database. If not successful, an Error message displays indicating the probable cause and offering an opportunity to retry the operation. Click the "Ignore" button if a KPA unit is not connected to your PC.

**Calculator** invokes the system calculator function.

### III. KPAWin© Configuration

Click on **Settings**, then the **General Configuration** item in the dropdown menu or use the Toolbar button, **Configuration Tabs**. This displays the eight Configuration Tabs. The Demo Database uses the KPAWin© default configuration settings for each of the eight tabs except for the General Items Tab. It contains the "1001 Demo Config" which uses the defaults but also requires the use of Passwords. Only the UserID of "Supervisor" actually has a password assigned. All others are blank. The Demo script allows the supervisor to create one or more configuration sets for each tab.

**Command Buttons** - All command buttons are enabled for users with Supervisory Privileges, however only the **Help**, **Cancel**, **Select**, and **OK** buttons are available for Non-Privileged Users.

**Add Button** - Enables all entries on the currently displayed Tab and allows privileged users to make changes to the displayed values and create a new configuration set.

**Cancel Button** - Cancels the creation of a new configuration and discards any entered values or changes.

**Apply Button** - Applies and saves the newly created values to the KPA Configuration and Database.

**Current Button** - Applies the currently displayed values to the KPA Configuration.

**Select Button** - Allows the user to select another previously defined set of configuration parameter for the Tab on display.

**OK Button** - Exits the Configuration Tabs and returns to the previous display.

**General Settings Tab** - This tab contains the KPAWin© general items. Click on the **Add** button and all items on the tab are enabled.

1. In the "Auto Reporting" frame, check "Print Single Sample Reports".
2. In the "Required Login Input" frame, check "Require Passwords" and press function key F1. (This displays the Context Sensitive Help for this item.) Close the Help display when finished.
3. Click the "Apply" button and the General Items menu requesting you to "Enter Configuration Name" is displayed with a default name of "Config". Enter a name of your choice and click the "OK" button.

**Hardware Settings** - Click on the "Hardware Settings" tab to bring it forward. Click on the "Add" button and all items on this tab are enabled.

1. Uncheck the "Include Auto Changer" checkbox in the "KPA Hardware Setup" frame. All data in the Auto Changer Setup frame disappears.
2. Click the "Apply" button and the Hardware Settings menu requesting you to "Enter Configuration Name" is displayed with a default name of "Config". Enter a name of your choice and click the "OK" button.

**Performance Settings** - Click on the "Performance Settings" tab to bring it forward. Click on the "Add" button and all items on this tab are enabled.

1. Change the "Calibration Limits" frame item "Percent Discrepancy" from 10 to 20.
2. Click the "Apply" button and the Performance Settings menu requesting you to "Enter Configuration Name" is displayed with a default name of "Config". Enter a name of your choice and click the "OK" button.



**KPA Unit Settings** - Click on the "KPA Unit Settings" tab to bring it forward. Click on the "Add" button. All items on this tab are enabled.

1. Change the "Laser" frame item "Pulses Per Analysis" from 1000 to 2000.
2. Click the "Apply" button and KPA Unit Settings menu requesting you to "Enter Configuration Name" is displayed with a default name of "Config". Enter a name of your choice and click the "OK" button.

**Maintenance Settings** - Click on the "Maintenance Settings" tab to bring it forward. Click on the "Add" button and all items on this tab are enabled.

1. Change the item "Dye Change (months)" from 2 to 4.
2. Click the "Apply" button and Hardware Settings menu requesting you to "Enter Configuration Name" is displayed with a default name of "Config". Enter a name of your choice and click the "OK" button.

**Calibration Settings** - Click on the "Calibration Settings" tab to bring it forward. Click on the "Add" button and all items on this tab are enabled.

1. Change the "Calibration Settings" frame item "Calibration Ranges" from Both to Low.
2. Click the "Apply" button and Calibration Settings menu requesting you to "Enter Configuration Name" is displayed with a default name of "Config". Enter a name of your choice and click the "OK" button.

**QC Limit Settings** - Click on the "Quality Control Settings" tab to bring it forward. Click on the "Add" button and all items on this tab are enabled.

1. Change the "Method Control" frame item "MDL Multiple" from 3 to 7.
2. A message display appears informing you the value you selected is **OUTSIDE** the Acceptable Range and provides the acceptable values to choose. All Configuration items have a "Gross Error Check" and allows only values within a predefined range. Click the "OK" button.
3. Change the value to 5 and then Click the "Apply" button. The Quality Control Settings menu requesting you to "Enter Configuration Name" is displayed with a default name of "Config". Enter a name of your choice and click the "OK" button.

**Alarms/Warnings** - Click on the "Alarms/Warning" tab to bring it forward. Click on the "Add" button and all items on this tab are enabled.

1. Select several items and click the **Alarms Checkbox** and several other items and click the **Warnings Checkbox**. The Alarms checked turn **RED** while the Warnings checked turn **YELLOW**.
2. Click the "Apply" button and the Hardware Settings menu requesting you to "Enter Configuration Name" are displayed with a default name of "Config". Enter a name of your choice and click the "OK" button.

This concludes the Configuration Changes Demo. Click the "OK" button to Exit the configuration tabs and return to the previous display.

#### IV. Sample Input Display

Click on either the **Analyze** dropdown menu followed by clicking the **Sample Input** item or use the Toolbar button **Analysis Input Form**. This displays and loads all **Entered and Processed but Unaccepted Samples** from the database. The Demo Database has a total of 36 Processed Samples with at least one example of all 19 Sample types. The Demo script allows the user to create more Sample Input entries. Note that the display is divided into two sections, an upper portion with Labels and Input Text areas and a lower Grid display portion. Data is entered into either section.

##### Loading Sample Data from Database

1. Click on the far left Grid Column labeled "**Analysis**" and highlight Analysis # 1 (SampleID = 1002, Sample Type = BckGnd.) Note that several labels in the upper portion of the display have changed color. Red indicates the item is **Required to be filled in**. Blue indicates an **Optional item**. Black indicates the data is taken from a **Basis Sample** and Grayed Out items are **Not Available** and therefore not applicable to this sample type. Each sample type requires a unique set of Required, Optional, Basis, and Not Applicable Sample Input items. The color code guides your input. All Required items must be entered before attempting to enter or change another Sample. The software displays an Error message describing any missing information.
2. Now click Analysis # 9 (SampleID = 1010, Sample Type = IChkStd) and again notice the color code changes to a number of fields in the upper portion of the display. The fields Standard ID and Standard Concentration are now colored Red while Calibration Config and Concentration Units are Grayed Out.
3. Change the "**Standard ID**" from **S-1** to **S-2** in either the upper portion of the display or on the Grid (Data may be entered in either section.)
4. To collapse any of the fields in the grid section of the form, place your mouse pointer on the Label Text in the upper portion of the display and double click. Try the **Concentration Units** field in the by double clicking on the label "Concentration Units". The Sample Units field in the grid completely collapses. To expand the field again double click the Label again and the Grid Header expands. All collapsed Grid fields are saved for each user in the operating systems Register file. Each time the user logs onto KPAWin©, all saved display preferences are reinstated. These preferences remain until the user changes them. Each user customizes these fields to their own preference.

##### Sample Input Toolbar

##### Saving / Restoring Sample Entries

1. Users enter Sample data into the Sample input form and Save it to a System file before analyzing the samples. This is very useful if a Laboratory runs the same or similar sets of samples on a reoccurring basis.
2. Enter the set of Sample data in the Sample Input form and Highlight the samples to be saved in the database.
3. Click the "**Save As Initial Samples**" (the picture of a floppy disk) Toolbar button and the Save menu is displayed. Key in the name for the selected Sample Input Data and click "**Save**" to save selected Samples. Click the "**SaveAll**" button to save all Samples on the Input Screen.
4. Click the "**Open**" Toolbar Button (the picture of an open file folder) and the "**Select Sample to Load**" menu is displayed. Highlight the Sample name and click the "**Load**" button. These Samples are loaded into the Grid from the database.

### Adding a New Sample

1. Click the **"Add New Sample"** Toolbar button (picture of plus sign "+"). Either the Highlighted Sample or if nothing is highlighted, the last Sample in the Grid is duplicated. However the Sample ID is concatenated with a number or the last number in the ID is incremented. The same rules apply to the Sample Description.
2. The user can change any of the displayed data items that are NOT Grayed out.
3. Users can also add a selected Sample using the **"Insert Before"** or **"Insert After"** feature. Highlight any sample by clicking the number in the **Analysis Column** to select the sample and then click the **"Right Mouse"** button to display the **"Input Pop-Up Menu"**. Select either the **"Insert Before"** or **"Insert After"** command and the highlighted sample is duplicated and placed in the desired order.
4. The **"Input Pop-Up Menu"** contains a considerable number of easy to use commands that you can exercise as desired. Some of these commands such as **Clear**, **Delete**, and **Analyze** are also executed by using the buttons on the Input Toolbar.
5. Several commands such as **"Accept"**, **"Reject"**, **"Reprocess"**, **"Calibrate"**, **"Insert Before"**, **"Insert After"**, and **"Move"** are only executed by using the Pop-Up menu.

### Calibration Display

1. Placing the cursor over the first column "Analysis, right click and the Pop-Up menu will appear. Highlight the the **"Calibrate"** command and Left click. This will force a calibration using the processed samples on the Sample Input Form and displays all the calibration data (both range data is displayed if there is data for both ranges).
2. Users may elect to disable the automatic calibration process by Checking the **"User Cal"** checkbox in either range. When checked, the user provides a name for the calibration and selects or deselects any specific calibration samples by **"Double Clicking"** on the specific sample. The "+" sign disappears when the sample is deselected and reappears if selected again. This method allows the user to select specific samples to be used in the calibration.
3. After selecting the samples you desire to be included in the calibration, click the Calibrate command button located on the right of the display to re-calibrate.
4. Plots are also available for display by clicking the **"Plot"** command button.
5. Printing the Calibration details or Plots is accomplished using the **"Print"** command button.
6. The **"Stop"** Toolbar button exits the Input display and returns to the previous display.

## V. Displays and Reports

Click on the dropdown **Reports**, then the **Selection** item or click on the **Sample Reports** Toolbar button to display the **Sample Report Selection** form. This display is divided into two sections. The upper portion contains all the selection options while the lower portion lists the available samples from the database after the "**Retrieve**" button is clicked. All **user selected items** are color-coded **Blue** and the KPAWin® defaults are selected when the form is initially displayed. The display design allows users a variety of options in selecting Samples from the database (e.g., selection by **Sample ID**, **Sample Type**, **Analyst**, **Customer**, **Procedure**, or **Date Processed**, etc.) as well as the type of display used to report that selection (e.g., **Single** or **Multiple Sample Display** reports, **Single** or **Multiple Print** reports, etc.) This script only uses a few of the available selection criteria. However you can employ as many as you desire.

**Selecting the Fields** options that will be displayed in the lower portion of the report **MUST** be completed prior to Retrieving the actual Sample from the database.

1. Selecting the Order of the Displayed Fields is only available to user with Supervisor privileges and only applicable to the Select Report Sample Display. By default, all Sample fields on this display are in Alphabetical Order.
2. Using the "Select Fields" function on the Select Report Sample display, Users can customize the viewable Grid Input data fields. The user can toggle between Select Fields and Move Fields by Double-Clicking the left mouse button on any selected field. While in the Move Fields mode, fields can be selected by dragging-and-dropping selections to the Top of the list. This list will define the order of the fields of the display Grid. Once the order of the Fields have been selected, Double Click the right mouse button again, which will change the display back to the Select Fields mode. Now Highlight all contiguous fields in the listing with a single click of the left mouse button and dragging the mouse pointer down to select those fields to be displayed. You may select only a few fields or all of them depending on your desires. We recommend that the Sample ID and Sample Type be selected and placed near the Top of the List because only the first column in the Select Reports Sample Grid will be used to select Samples from the Grid to be analyzed and displayed. The user-selected, custom view is also retained in the system Registry File and is only applicable the this computer. The Selected View will be reloaded each time that user signs into KPAWin on that computer.

**Selecting Samples** from the database is accomplished by using the various selection options on the top portion of the display.

1. In the "**Sample Type**" frame, use the dropdown display to select the "**CalStd**" sample type. Notice that the Radio (round) Button in the frame changes from **ALL** to **AND**. Now click the **Retrieve** button to load the data from the database. All CalStd type samples meeting the other selection criteria are extracted from the database and displayed according to your selection order in the lower portion of the screen. You can try other selection criteria at your discretion.

### **On Screen Display Options - Single Sample Displays**

1. In the "**Sample Type**" selection frame, click the "**All**" button and then the "**Retrieve**" button again. This displays ALL of the samples in the database.
2. To view one of the Samples, highlight the desired sample by clicking in the First column, in the Demo Database example the first column is labeled "**Sample ID**".
3. Using the scroll bar on the right of the display, scroll to the Sample ID "**1027**" and highlight this sample. Using the KPAWin® default display options **Display Reports** and **Single Display**, click the "**Display Reports**" command button. This displays the results of the sample. Click the lower "**Stop Sign**" toolbar button or the X in the upper right hand corner of the display when finished viewing the report. (Note: there are two sets of window buttons in the upper right hand corner of the display, the lower set is for the Single Sample report you are viewing while the top set controls the KPAWin program. Clicking the X in the lower set will close the Single Sample Report while clicking the X in the upper set will close the KPAWin® program)

4. Now highlight another sample, then click the **"Display"** command to view these results. This displays the single sample display for the selected sample. Both the **Sample Intensities** and **Plots** are also available from this display.
5. To view the **Sample Plots**, click the first button on the Sample Display Toolbar (lower toolbar - picture of a graph). A frame is displayed depicting the Plot options with the default of **"Net Data"** (change the display selection to any option desired); then click the Display Plots button. When through viewing the Plots, click the **"X"** in the extreme upper right corner of the Plot display and then the **Cancel** command on the Plot frame.
6. To view the **Sample Intensities**, click the second button on the Sample Display Toolbar (lower toolbar - picture of a report). A frame is displayed depicting the Intensity options with the default of **"Net Sample Data"** (change the display selection to any option desired); then click the Display Intensities button. When through viewing the Intensities, click the **"X"** in the extreme upper right corner of the Intensity display and then the **Cancel** command on the Intensity frame.
7. Resize the Single Sample display, if required, and click the **"Stop"** Toolbar button to return to the Select Sample display when through viewing the report.

#### On Screen Display Options - Multiple Sample Displays

1. To select Sample for the Multi Display, select all the samples on display by highlighting the first one, then the last sample while holding down the **Shift** key. Now click the **"Multi Display"** radio (round) button, (note that all the Multi Display options are now enabled), select the **"Sample Analysis"** option, and click the **"Display"** command. This displays the **Multi Sample Analysis** report containing one line of data for each sample. Use the Scroll Bar at the bottom of the report to view all the data. When through viewing the report, click the **"X"** in the extreme upper right corner of the **Multiple Sample Analysis Report** (Located approximately Mid Screen on the right edge) to return to the previous display.
2. Using all the above selection criteria, click another report radio button (e.g., **"Quality Control"**) and click the **"Display"** command to view other multiple sample displays.

#### Print Report Options - Single Sample Reports

1. In the **"Print Report Option"** frame, click the **"Single Sample"** radio button. All the MultiReport checkboxes are disabled. The Calibration and Configuration selections remain active. The Details option is also available when printing Single Sample reports.
2. Highlighting one sample at a time, select various sample types (e.g., BckGnd, Sample, MtdStd, SASamp, SampSpk, etc.). Then click the **"Print"** command button. Although all Single Sample Print Reports have many items in common, each sample type produces a slightly different report.
3. Check as many report types as desired to become familiar with the various reports.

#### Print Report Options - Multiple Sample Reports

1. In the **"Report Options"** frame, click the **"Print Reports"** radio (round) button. This changes the **Monitor Display Options to Print Report Options**. Click the **MultiReport Option**, if not already selected, and there are four enabled checkbox reports and two disabled items. The disabled items (Calibration and Configuration) are available when one of the MultiReport options is selected.
2. Select two or more of the samples on display by highlighting them. Then check one of the report options, use **"Analytical Results"** and the two additional checkboxes will be available. If checked, these additional checkboxes produce additional **Calibration** or **Configuration** reports. Detailed reports are obtained by checking the **"Details"** checkbox to the right of the Calibration and Configuration report options.
3. Check as many report options as desired to become familiar with the various reports.

This concludes the familiarization script for supervisors.



## **KPAWin© Error Codes**

### **KPAWin© Program Errors**

- E1 KPAWin© Error: Unexpected
- E2 KPAWin© Error: Bad Routine Call Parameter
- E3 KPAWin© Error: Incorrect call to Move Samples
- E4 KPAWin© Error: Message Format is Bad
- E5 KPAWin© Error: Undefined Alarm and Warning Message
- E6 KPAWin© Error: Report Record not found
- E7 KPAWin© Error: Sample Record does not exist
- E8 KPAWin© Error: Intensity Record does not exist
- E9 KPAWin© Error: Configuration
- E10 KPAWin© Error: ToolBar Menu

### **KPAWin© Database Errors**

- E51 KPAWin© Database Error
- E52 KPAWin© Database Error: Record NOT Found

### **User Control Messages**

- E91 User Escape
- E92 User Abort
- E93 User Cancel
- E94 User Ignore
- E95 User Retry

### **Sample Changer Error Messages (100 Series)**

- E101 Sample Changer Movement Data is Missing
- E102 Sample Changer Error
- E103 RackNb: & VialNb: NOT Valid
- E104 Sample Changer Error
- E105 ERROR - Number of Sample Vials Exceed number of Mix Vials
- E106 Reference Lifetime Too Short
- E107 Reference Intensity Is Excessive
- E108 Auto Changer Error

### **Calculations Error Messages (200 Series)**

- E201 Unable To Calculate Reference Intercept
- E202 Unable To Calculate Sample Intercept
- E203 Equals Water Blank
- E204 Not enough Samples available for &1& calculation
- E205 Basis Sample Loop
- E206 Auto Calibration Completed
- E207 All Samples must contain same number of Measurements
- E208 Spike and Sample have same Integrated Intensities
- E209 Initial Analysis Volume is Zero
- E210 Sample Must have Standard Concentration Specified
- E211 There are NO Valid Calibration Points
- E212 Calibration Curve Fit Error
- E213 KPA NOT Calibrated
- E214 Precounts High For High Range
- E215 Confidence Test Aborted
- E216 Basis Sample Must Be Assigned before Measurement
- E217 Basis Sample Must Be Measured First
- E218 There are NO Calibration Points

### **CurveFit Error Messages (400 Series)**

- E401 Inverse Bad

**DBRoutines Error Messages (500 Series)**

- E501 Database File: & FileName & could NOT be Closed  
E502 Database File: & FileName & could NOT be Opened"

**KPA Error Messages (600 Series)**

- E601 Check KPA & Msg  
E602 Auto Changer Error  
E603 Put Sample & SampleId Msg(SampleData)  
E604 Put Sample Into KPA Cancel/Error  
E605 Gilson Device Not Responding  
E630 KPA Analyze Error

**KPAIO Error Messages (700 Series)**

- E701 KPA Not Responding  
E702 This KPA Cannot Use this Command  
E703 Sample Intensity KPA Data Read Error  
E704 Reference Intensity KPA Data Read Error  
E705 Max Nb of Intensities & Data values must be received  
E706 Place RANGE selector switch in the & SwitchMsg & position"  
E707 This KPA Cannot Use the OUTALL Command  
E708 There are no Standards for this sample  
E709 Reference Intensity Is Excessive  
E710 KPA NOT Ready  
E711 Waiting for KPA Data  
E712 Waiting for KPA Laser  
E713 KPA Checksum Error  
E714 KPA Background Measurement Required

**MDIControl Error Messages (800 Series)**

- E801 Cannot find FindString

**SelectCalData Error Messages (900 Series)**

- E901 No & Title & Found

**ShowResults Error Messages (1000 Series)**

- E1001 Reference  $R^2 < .99$

**Utilities Error Messages (1100 Series)**

- E1101 KPAWin© Configuration  
E1102 The Value &1& Is NOT WITHIN the Acceptable Range  
E1103 Range Must be Between &Value1& and &Value2&  
E1104 Resetting to Previous Value  
E1105 & DataText & Validation Rule is NOT valid & Validation Rule

**MDIForm Error Messages (1200 Series)**

- E1201 Only ONE instance of + KPAWin + is allowed

**General Config Error Messages (1300 Series)**

- E1301 Invalid Configuration Name provided - Default: & DefaultName & Used

**Configuration Error Messages (1400 Series)**

- E1402 Polling & Format(DeviceNb)

**GilsonSerial and KPASerial Error Messages (1500 Series)**

- E1501 &Msg& Equipment Connected  
E1502 Check &1& Power and Cables

**Login Error Messages (1800 Series)**

E1801 Invalid Password Try Again ,, Invalid Login Attempt"

**MultiReport Error Messages (1900 Series)**

E1901 KPAWin© Error: Report Record not found

**ProcedureAdmin Error Messages (2100 Series)**

E2101 KPAWin© Error

**SampleInput Error Messages (2200 Series)**

E2201 Analyze SELECTED Samples?  
E2202 Add Sample?  
E2203 Insert After Sample: + SampleIdMsg + ?  
E2204 Insert Before Sample: + SampleIdMsg + ?  
E2205 There are no Samples saved with this name  
E2206 Delete InitialSample: &(InitialSampleName)  
E2207 There are no Samples to Analyze  
E2208 There are no unprocessed samples  
E2209 There are no samples  
E2210 Clear Sample: + SampleIdMsg + ?  
E2211 Delete ALL Samples?  
E2212 Delete Sample: + SampleIdMsg + Delete SELECTED Samples?  
E2213 Approve Processed Sample: + SampleIdMsg + Approve Processed and SELECTED Samples?  
E2214 Reject Processed Sample: + SampleIdMsg + Reject Processed and SELECTED Samples?  
E2215 Reprocess Processed Sample: + SampleIdMsg + Reprocess Processed and SELECTED Samples?  
E2216 Input data is Invalid  
E2217 Move SELECTED Samples?  
E2218 All Samples have NOT been Analyzed & Continue Analysis?  
E2219 Can not delete a Sample which has & Saved Intensities.  
E2220 Record is currently locked. Try again later.  
E2221 Data has been changed by another user.  
E2222 There are no Samples to Save  
E2223 Initial Samples cannot be Saved without a Valid Name  
E2224 There is already a saved sample with this name" & "Replace Current Initial Sample?"  
E2225 Initial Sample(s) &Msg& have been Saved"  
E2226 Database Errors: All Samples may NOT have been saved  
E2227 Batch Identification is NOT the first sample  
E2228 Samples being used as a Basis can NOT be deleted  
E2229 There can only be one background Per Batch  
E2230 Basis Sample has been deleted

**SelectRptSample Error Messages (2300 Series)**

E2301 You Must SELECT a Sample to Display with the Single Sample Report  
E2302 You Must SELECT a Display Intensity Option  
E2303 You Must SELECT a Display Plot Option  
E2304 You Must SELECT a Display Type when using the MultiReport Option

**Admin Error Messages (2400 Series)**

E2401 &1& DataBase Information has been Updated  
E2402 Discarding Changes  
E2403 Exiting &1& Administration Menu  
E2404 Updating &1& Administration DataBase  
E2405 Password Entries Do Not Match - Reenter Confirmation  
E2406 Please Reenter Confirmation Password

**Serial I/O Error Messages**

E9000 Serial I/O: Base Number  
E9001 Serial I/O: Received Bad Character

### **"Alarms / Warnings Message List"**

Alarm / Warning #1	Background High - High Range
Alarm / Warning #2	Background High - Low Range
Alarm / Warning #3	Calibration Correlation (R Squared) Low - High Range
Alarm / Warning #4	Calibration Correlation (R Squared) Low - Low Range
Alarm / Warning #5	Calibration Standard Discrepancy High - High Range
Alarm / Warning #6	Calibration Standard Discrepancy High - Low Range
Alarm / Warning #7	Maximum Calibration Age Exceeded
Alarm / Warning #8	Sample Correlation (R Squared) Low
Alarm / Warning #9	Sample Lifetime Low
Alarm / Warning #10	Sample Lifetime High
Alarm / Warning #11	Reference Lifetime Low
Alarm / Warning #12	Reference Ratio High
Alarm / Warning #13	Reference Ratio Low
Alarm / Warning #14	Gate Shifting Excessive
Alarm / Warning #15	Gate Dropping Excessive
Alarm / Warning #16	Sample Concentration Out of Calibration Range
Alarm / Warning #17	Too Few Valid Time Gates For Evaluation
Alarm / Warning #18	Sample Concentration High For Low Range
Alarm / Warning #19	Sample Concentration Low For High Range
Alarm / Warning #20	Dye Output Low
Alarm / Warning #21	Laser Plasma Cartridge Output Low
Alarm / Warning #22	Laser Plasma Cartridge Age Exceeds Service Life
Alarm / Warning #23	Dye Age Exceeds Service Life
Alarm / Warning #24	Reference Age Exceeds Service Life
Alarm / Warning #25	Schedule Preventive Maintenance
Alarm / Warning #26	Too Few Calibration Points - High Range
Alarm / Warning #27	Too Few Calibration Points - Low Range
Alarm / Warning #28	Initial Calibration Check Blank High - High Range
Alarm / Warning #29	Initial Calibration Check Blank High - Low Range
Alarm / Warning #30	Initial Calibration Check Standard Invalid - High Range
Alarm / Warning #31	Initial Calibration Check Standard Invalid - Low Range
Alarm / Warning #32	Continuing Calibration Check Blank High - High Range
Alarm / Warning #33	Continuing Calibration Check Blank High - Low Range
Alarm / Warning #34	Continuing Calibration Check Standard Invalid - High Range
Alarm / Warning #35	Continuing Calibration Check Standard Invalid - Low Range
Alarm / Warning #36	Too Few IDL Standards - Type 1 IDL
Alarm / Warning #37	Too Few IDL Averages - Type 2 IDL
Alarm / Warning #38	Average Concentration of IDL's High
Alarm / Warning #39	IDL Above Maximum Acceptable Value
Alarm / Warning #40	Too Few MDL Standards - Type 1 MDL
Alarm / Warning #41	Too Few MDL Averages - Type 2 MDL
Alarm / Warning #42	Average Concentration of MDL's High
Alarm / Warning #43	MDL Above Maximum Acceptable Value
Alarm / Warning #44	Relative Percent Deviation High
Alarm / Warning #45	Sample Spike Recovery High
Alarm / Warning #46	Sample Spike Recovery Low
Alarm / Warning #47	Serial Dilution Recovery High
Alarm / Warning #48	Serial Dilution Recovery Low
Alarm / Warning #49	Multiple MSA Spike Recovery High
Alarm / Warning #50	Multiple MSA Spike Recovery Low
Alarm / Warning #51	MSA Spike Recovery High
Alarm / Warning #52	MSA Spike Recovery Low
Alarm / Warning #53	Multiple MSA Slope Error High
Alarm / Warning #54	Multiple MSA Calibration Correlation (R Square) Low
Alarm / Warning #55	MDL Multiple Exceeds Min Sample Result
Alarm / Warning #56	CRDL% Exceeds Min Sample Result
Alarm / Warning #57	Sample % Exceeds Min Sample Result

Alarm / Warning #58	MDL Multiple, CRDL%, or Sample % Exceeds Min Sample Result
Alarm / Warning #59	Method Standard Invalid - High Range
Alarm / Warning #60	Method Standard Invalid - Low Range
Alarm / Warning #61	Precounts High For High Range
Alarm / Warning #62	Precounts Low For High Range
Alarm / Warning #63	Precounts High For Low Range
Alarm / Warning #64	No Concentration Unit Conversion Available
Alarm / Warning #65	Units Conversion / Check Specific Gravity
Alarm / Warning #66	Units Conversion / Check Specific Activity
Alarm / Warning #67	Units Conversion / Check Atomic Mass
Alarm / Warning #68	Invalid Reference Intensities
Alarm / Warning #69	No Background - High Range
Alarm / Warning #70	No Background - Low Range
Alarm / Warning #71	Gate Shifting
Alarm / Warning #72	Gate Dropping
Alarm / Warning #73	Sample Intensity Intercept Below Background
Alarm / Warning #74	Sample Intensity Intercept Below Quenched Background
Alarm / Warning #75	No Calibration - High Range
Alarm / Warning #76	No Calibration - Low Range
Alarm / Warning #77	No Basis Sample For Sample Duplicate
Alarm / Warning #78	No Basis Sample For Sample Spike
Alarm / Warning #79	No Basis Sample For Quenched Background
Alarm / Warning #80	No Basis Sample For Method of Standard Addition
Alarm / Warning #81	No Basis Sample For Standard Addition Spike
Alarm / Warning #82	No Basis Sample For Serial Dilution
Alarm / Warning #83	Reference Correlation (R Squared) Low
Alarm / Warning #84	Reference Intensity Low
Alarm / Warning #85	Reference Intensity High
Alarm / Warning #86	Maximum of 7 Calibration Standards Exceeded Oldest Ignored - High Range
Alarm / Warning #87	Maximum of 7 Calibration Standards Exceeded Oldest Ignored - Low Range
Alarm / Warning #88	Sample Concentration High For High Range



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## **KPAWin© Configuration Selection**

The purpose of this file is to simplify the creation and use of KPAWin configuration tabs. The KPAWin program is delivered with a KPAEmpty.mdb database. This database is an integral part of the program. The copy of the database that resides with the KPAWin executable in the KPAWin\Exe folder on your computer is the exact form of the database that is used during execution of the program; KPA databases that are loaded from any other source are checked for conformity to this database and converted to the form of this database, if necessary.

During installation, you placed a copy of the KPAEmpty database into the KPAWin/Database folder. When you prepare a user database, you will make a copy of KPAEmpty and rename it as you require. The user database is run with KPAWin to specify, collect, and report analytical data. The procedure below greatly simplifies the process of creating a new user database.

The simplification results from a one-time creation of an intermediate database that becomes the user's template. The template is a copy of the KPAEmpty data base that contains all of the user configuration preferences and is assigned a local laboratory template name.

The remainder of this utility contains two sections. Both contain all of the configuration fields, by configuration tab. The first section contains three column: typical setting for a field, a blank space for the user to enter an alternative local preference, and label of the field. Typical settings are for a KPA-11 with a Gilson model 223 auto changer and model 402 syringe pump, and "Site Custom" tabs with full quality control. As your requirements vary from these, make the appropriate selections. The second section contains a short textual description of each field with comments on selection of settings.

While the program contains eight unique configuration tabs, satisfactory operation of a KPA only requires the use of four tabs: General Items, Hardware Settings, KPA Unit Settings, and Calibration Settings. Collectively, these required tabs provide the same level of operation as the DOSKPA program that preceded KPAWin. The use of the other four tabs is optional: Maintenance, Performance Limits, Quality Control Limits, and Alarms/Warnings. The four optional tabs work together to provide real-time quality control that is either active or passive. If "STOP KPA Processing on Alarms" is selected on the Alarms/Warnings tab, any selected alarm or warning will halt KPA operation when an action item is violated. User intervention is required to resume operation. Whether the check box is selected or not, "A" for alarm or "W" for warning are reported with the action number for each analytical violation at the time sample results are displayed or printed. These violations can also be exported with the data at the user's discretion. Some action items are qualitative such as "No Calibration - High Range". Most are quantitative with user-specified control values taken from one of the other three optional tabs. For example, "Background High - Low Range" violates a Maintenance tab specification.

We recommend that you negotiate Data Quality Objectives with your customers by simply completing the following form for the optional tabs. The customer-specific tabs that you create are used to run that customer's samples. Analytical failures are detected at run time if the appropriate Alarm/Warning action items are selected. Corrective action can be taken immediately and reanalysis can be performed with fresh samples.

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## Configuration Tabs

### Configuration Tab Buttons

These six buttons are common to all eight configuration tabs.

Help	On-line help
Select	Display drop-down listing of all alternative selections of current tab
Add	Make copy of existing tab to edit and rename
Current	Set current tab as default
Apply	Accept and Continue in configuration tabs
OK	Accept and Exit

### General Items Tab

Blank	<input type="checkbox"/> InActive
"Local"*	<input type="checkbox"/> List of Available Configurations

\* Make a copy of KPAEmpty.mdb and save as your "Local" Template. Name each "Local" tab according to your laboratory preference. For each of the eight configuration tabs, check all configurations except your "Local" Template as InActive. In the future, when you need a new KPAWin database, use a copy of your "Local" Template. Alternative versions of each configuration can be created and left Active as your laboratory requires. InActive tabs remain in the database to qualify the configuration of historical samples in the database that used these tabs. Inactive tabs are not available, and cannot be selected during sample input. Only Active (unchecked) tabs are available during sample input.

### Auto Reporting / Miscellaneous Options

Check	<input type="checkbox"/> Display Single Sample Reports (Check to select.)
Blank	<input type="checkbox"/> Print Single Sample Reports (Check to select.)
Blank	<input type="checkbox"/> Display Multiple Sample Reports (Check to select.)
Blank	<input type="checkbox"/> Print Intensity Plots (Check to select.)
Blank	<input type="checkbox"/> Print Calculated Intensities (Check to select.)
Blank	<input type="checkbox"/> Print Measured Intensities (Check to select.)
Check	<input type="checkbox"/> Display Results in Scientific Notation (Check to select.)
Check	<input type="checkbox"/> Required Login Input (Check to select.)
Check	<input type="checkbox"/> Require User ID's (Check to select.)
Blank	<input type="checkbox"/> Require Passwords (Check to select.)

---

## **Hardware Settings Tab**

Blank ☐ InActive

"Local" ☐ List of Available Configurations

### **KPA Hardware Setup**

1  Serial Port (KPA) (Range: 1 to 99)

9600,N,8,1  Serial Port Settings (KPA) (KPA-10 is 4800,N,8,1)

3  Eprom Version (KPA-10 /w auto shutter is 2. KPA-10 w/ manual shutter is 1.)

Check ☐ Include Auto Changer (Check to select.)

### **Auto Changer Setup**

2  Serial Port (AutoChanger) (Range: 1 to 99)

9600,N,8,1  Serial Port Settings (AutoChanger)

### **Syringe Pump**

1  Device Number (Syringe Pump) (Range: 0 to 62)

402  Model Number (Syringe Pump) (Alternative is 401)

SP40250  Flow Data Name (Syringe pump 401 is SP40150.)

### **Sample Changer**

0  Device Number (Sample Changer) (Range: 0 to 62)

223  Model Number (Sample Changer) (Alternative is 222)

24242429  Tray Data Name (Sample changer 222 takes five racks.)

### **Position Offsets**

0  X

0  Y

0  Z

---

## KPA Unit Settings Tab

Blank ☐ InActive

"Local" ☐ List of Available Configurations

### **Laser**

20 ☐ Pulses Per Second (Range: 1 to 20)

1000 ☐ Pulses Per Analysis (Range 1 to 10,000)

15 ☐ Warm-Up Time (min) (Range: 0 to 1,000)

200 ☐ Warm-Up Pulses (Range: 1 to 9,999)

### **Detection**

10 ☐ PMT Dead Time (ns) Range: 0 to 200 (KPA-11 is 10. KPA-11 is ~80 to 130.)

1 ☐ Minimum Intensity Factor (Range: 1 to 100)

200 ☐ Maximum Intensity Factor/Pulse (Range 1 to 255)

### **Time Gates**

127 ☐ Total Time Gates (Range 2 to 127)

5 ☐ Start LSR Gate (KPA-10: use 4 for uranium analysis.)

45 ☐ Number of LSR Gates (Range: 1 to 127) (For urine, use 35.)

15 ☐ Minimum Number LSR Gates (Range: 5 to 127)

13 ☐ Time Gate Width ( $\mu$ s) (Range: 1 to 40) (For lanthanides, see KPA manual.)

### **Range Selection**

85 ☐ Auto Efficiency Factor (Range: No rule)

120 ☐ High Intercept Tolerance (%) (Range: 1 to 2,000)

80 ☐ Low Intercept Tolerance (%) (Range: 1 to 100)

---

### Calibration Settings Tab

Blank ☐ InActive

"Local" ☐ List of Available Configurations

#### Untitled Frame

$\mu\text{g/l}$  ☐ Concentration Units (Default for database)

1 ☐ Specific Gravity (g/ml) (Range: 0.1 to 10))

25000 ☐ Specific Activity (Bq/g) (Range: 1 to 80,000)

Uranium ☐ Select Analyte

238.0289 ☐ Atomic Mass (g/mole)

Auto ☐ Calibration Ranges

Total Vol. ☐ Dilution Method (Alternative is "Add To" for addition volume for dilution.)

#### High-Range

Quadratic ☐ Equation Type (Alternative is "Linear".)

Blank ☐ Include Zero Point

3 ☐ Minimum Number of Standards (Range: 1 to 7)

#### Low-Range

Linear ☐ Equation Type (Alternative is "Quadratic".)

Blank ☐ Include Zero Point

3 ☐ Minimum Number of Standards (Range: 1 to 7)

#### Analysis Settings

Select ☐ Smooth Analysis Results Data (Check to deselect correction.)

Select ☐ Reference Correct Data (Check to deselect correction.)

Select ☐ Background Correct Data (Check to deselect correction.)

Blank ☐ Proceed After Auto Calibration (Check to proceed without review.)



---

## **Maintenance Tab**

Blank ☐ InActive

"Local" ☐ List of Available Configurations

### **Maintenance Options**

Blank ☐ Use Defaults

Select ☐ Use Site Custom

Blank ☐ Do Not Use Limits

1 ☐ Excitation Test Standard ( $\mu\text{g/l}$ ) (Range: 0.1 to 5,000)

10 ☐ Dye Strength (Intercept counts/pulse) (Range 1 to 300)

10 ☐ Laser Power (Intercept counts/pulse) (Range: 1 to 300)

3 ☐ Number of Confidence Test Repeat Analyses

Low ☐ Confidence Test Analysis Range

18\* ☐ Laser Plasma Cartridge Change (months) (Range: 12 to 24)

3\* ☐ Dye Change (months) (Range: 1 to 6)

7\* ☐ Reference Change (days) (Range: 1 to 31)

12\* ☐ Scheduled Preventive Maintenance (months) (Range: 6 to 12)

\* Enter dates as appropriate. A new reference requires a new Maintenance Tab.

---

## **Performance Limits Tab**

Blank ☐ InActive

"Local" ☐ List of Available Configurations

### **Performance Limits Options**

Blank ☐ Use Defaults

Select ☐ Use Site Custom

Blank ☐ Do Not Use Limits

### **Calibration Limits**

25 ☐ Maximum Background High Range (Range: 1 to 1,000)

1500 ☐ Maximum Background Low Range (Range: 1 to 10,000)

0.90 ☐ Calibration R Squared (Range: 0.5 to 1.0)

20 ☐ Percent Discrepancy (Range: 1 to 1,000)

100 ☐ Maximum Calibration Duration (days) (Range: 1 to 1,000)

### **Analysis Limits**

0.90 ☐ Sample R Squared (Range 0.5 to 1.0)

150 ☐ Minimum Sample Lifetime ( $\mu$ s) (Range: 1 to 10,000)

325 ☐ Maximum Sample Lifetime ( $\mu$ s) (Range: 1 to 10,000)

150 ☐ Minimum Reference Lifetime ( $\mu$ s) (Range: No rule)

0.20 ☐ Reference Ratio (+/-) (Range: -0.5 to 0.5)

10 ☐ Gate Shifting (Range: 1 to 125)

10 ☐ Gate Dropping (Range: 1 to 127)

---

### **Quality Control Limits Tab**

Blank ☐ InActive  
"Local" ☐ List of Available Configurations

#### **QC Limit Options**

Blank ☐ Use Defaults  
Select ☐ Use Site Custom  
Blank ☐ Do Not Use Limits

#### **Detection Limits\***

7 ☐ Number of Standards (Range: 0 to 13)  
1 ☐ Type (Type 1 uses the standard deviation of individual daily measurements. Type 2 uses the average of daily standard deviations of multiple measurements.)  
13 ☐ Maximum Duration (weeks) (Range: 1 to 13)  
5 ☐ Maximum Concentration Factor (Range is 1 to 10)  
Blank ☐ Limit  $\mu\text{g/l}$  (Range: 0 to 1)

\* All specifications for IDL and MDL. CRDL specifies only regulatory limit or customer reporting detection limit.

#### **Calibration Control \***

3/3 ☐ Calibration Blank (IDLMultiple) (Range: 1 to 5)  
10/20 ☐ Calibration Control Standard Error (%) (Range: 1 to 100)

\* First value is for Initial (immediately after calibration) and second specification is for Continuing (any time after initial).

#### **Method Control \***

3 ☐ MDL Multiple (Range: 1 to 5)  
Blank ☐ CRDL Percentage (Range: 0 to 100)  
Blank ☐ Sample Percentage (Range: 1 to 100)  
Blank ☐ Use Highest of the Above (MDL Multiple, CRDL % or Sample %)  
25 ☐ Method Control Standard Error (+/-%) (Range: 1 to 100)

\* Select only one of first four check boxes if Method Blanks are used.

#### **Sample Control**

20 ☐ Precision % (Relative Percent Difference of two measurements) (Range: 1 to 100)  
15 ☐ Spike Recovery (%) (Range: 1 to 100)  
10 ☐ Serial Dilution Error (%) (Range: 1 to 100)

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**Method of Standard Addition \***

3        \_\_\_\_\_ Number of Spikes (Range: 0 to 3) (<3 spikes don't give Slope Error or R Squared.)

Blank    \_\_\_\_\_ Recovery Error (%) (Range: 0 to 100)

Blank    \_\_\_\_\_ Slope Error (%) (Range: 0 to 100)

Blank    \_\_\_\_\_ R Squared (Range: 0 to 1)

\* Method provides comparison of batch and internal calibration.

---

## Alarms/ Warnings Tab

Blank       InActive

"Local"      List of Available Configurations

Blank       STOP KPA Processing on Alarms

### Alarms / Warnings Optional Action Items

Alarm	Warning
_____	A/W #1 Background High - High Range
_____	A/W #2 Background High - Low Range
_____	A/W #3 Calibration Correlation (R Squared) Low - High Range
_____	A/W #4 Calibration Correlation (R Squared) Low - Low Range
_____	A/W #5 Calibration Standard Discrepancy High - High Range
_____	A/W #6 Calibration Standard Discrepancy High - Low Range
_____	A/W #7 Maximum Calibration Age Exceeded
_____	A/W #8 Sample Correlation (R Squared) Low
_____	A/W #9 Sample Lifetime Low
_____	A/W #10 Sample Lifetime High
_____	A/W #11 Reference Lifetime Low
_____	A/W #12 Reference Ratio High
_____	A/W #13 Reference Ratio Low
_____	A/W #14 Gate Shifting Excessive
_____	A/W #15 Gate Dropping Excessive
_____	A/W #16 Sample Concentration Out of Calibration Range
_____	A/W #17 Too Few Valid Time Gates For Evaluation
_____	A/W #18 Sample Concentration High For Low Range
_____	A/W #19 Sample Concentration Low For High Range
_____	A/W #20 Dye Output Low
_____	A/W #21 Laser Plasma Cartridge Output Low
_____	A/W #22 Laser Plasma Cartridge Age Exceeds Service Life
_____	A/W #23 Dye Age Exceeds Service Life
_____	A/W #24 Reference Age Exceeds Service Life
_____	A/W #25 Schedule Preventive Maintenance
_____	A/W #26 Too Few Calibration Points - High Range
_____	A/W #27 Too Few Calibration Points - Low Range
_____	A/W #28 Initial Calibration Check Blank High - High Range
_____	A/W #29 Initial Calibration Check Blank High - Low Range
_____	A/W #30 Initial Calibration Check Standard Invalid - High Range
_____	A/W #31 Initial Calibration Check Standard Invalid - Low Range
_____	A/W #32 Continuing Calibration Check Blank High - High Range
_____	A/W #33 Continuing Calibration Check Blank High - Low Range
_____	A/W #34 Continuing Calibration Check Standard Invalid - High Range
_____	A/W #35 Continuing Calibration Check Standard Invalid - Low Range
_____	A/W #36 Too Few IDL Standards - Type 1 IDL
_____	A/W #37 Too Few IDL Averages - Type 2 IDL
_____	A/W #38 Average Concentration of IDL's High
_____	A/W #39 IDL Above Maximum Acceptable Value
_____	A/W #40 Too Few MDL Standards - Type 1 MDL
_____	A/W #41 Too Few MDL Averages - Type 2 MDL
_____	A/W #42 Average Concentration of MDL's High
_____	A/W #43 MDL Above Maximum Acceptable Value
_____	A/W #44 Relative Percent Deviation High
_____	A/W #45 Sample Spike Recovery High
_____	A/W #46 Sample Spike Recovery Low
_____	A/W #47 Serial Dilution Recovery High



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_____	A/W #48	Serial Dilution Recovery Low
_____	A/W #49	Multiple MSA Spike Recovery High
_____	A/W #50	Multiple MSA Spike Recovery Low
_____	A/W #51	MSA Spike Recovery High
_____	A/W #52	MSA Spike Recovery Low
_____	A/W #53	Multiple MSA Slope Error High
_____	A/W #54	Multiple MSA Calibration Correlation (R Square) Low
_____	A/W #55	MDL Multiple Exceeds Min Sample Result
_____	A/W #56	CRDL% Exceeds Min Sample Result
_____	A/W #57	Sample % Exceeds Min Sample Result
_____	A/W #58	MDL Multiple, CRDL%, or Sample % Exceeds Min Sample Result
_____	A/W #59	Method Standard Invalid - High Range
_____	A/W #60	Method Standard Invalid - Low Range
_____	A/W #61	Precounts High For High Range
_____	A/W #62	Precounts Low For High Range
_____	A/W #63	Precounts High For Low Range
_____	A/W #64	No Concentration Unit Conversion Available
_____	A/W #65	Units Conversion / Check Specific Gravity
_____	A/W #66	Units Conversion / Check Specific Activity
_____	A/W #67	Units Conversion / Check Atomic Mass
_____	A/W #68	Invalid Reference Intensities
_____	A/W #69	No Background - High Range
_____	A/W #70	No Background - Low Range
_____	A/W #71	Gate Shifting
_____	A/W #72	Gate Dropping
_____	A/W #73	Sample Intensity Intercept Below Background
_____	A/W #74	Sample Intensity Intercept Below Quenched Background
_____	A/W #75	No Calibration - High Range
_____	A/W #76	No Calibration - Low Range
_____	A/W #77	No Basis Sample For Sample Duplicate
_____	A/W #78	No Basis Sample For Sample Spike
_____	A/W #79	No Basis Sample For Quenched Background
_____	A/W #80	No Basis Sample For Method of Standard Addition
_____	A/W #81	No Basis Sample For Standard Addition Spike
_____	A/W #82	No Basis Sample For Serial Dilution
_____	A/W #83	Reference Correlation (R Squared) Low
_____	A/W #84	Reference Intensity Low
_____	A/W #85	Reference Intensity High
_____	A/W #86	Maximum of 7 Calibration Standards Exceeded Oldest Ignored – High Range
_____	A/W #87	Maximum of 7 Calibration Standards Exceeded Oldest Ignored – Low Range
_____	A/W #88	Sample Concentration High For High Range

**General Items**

The General Items form contains two frames, an Active/Inactive checkbox, and a Database ID list box. The first frame contains options for automatic reporting. The second frame contains options for user and supervisor logins. Supervisor login with password reserves changes of this tab to the supervisor. Otherwise, it is modified by any valid user.

**InActive**

The Inactive checkbox indicates the displayed configuration is NOT currently active. Activate by clicking the Select command button. Select the Configuration ID to display and then clear the check mark.

**List of Available Configurations**

The List Test Box displays all available configurations for this configuration tab. Any active tab is selected from the list for the background and retained throughout the batch.

**Auto Reporting Options**

The Auto Reporting Options frame enables a Supervisor to select the screen displays and reports queued to the printer immediately after a sample is analyzed.

**Display Single Sample Reports**

The Display Single Sample Reports checkbox indicates the use of the Single Sample Report mechanisms within KPAWin® to produce monitor display reports. The system default is True. Click the checkbox to deselect this item.

**Print Single Sample Reports**

The Print Single Sample Reports checkbox enables automatic printing of Single Sample Reports for each selected sample. The system default is True. Click the checkbox to deselect this item and to suppress the display of single sample reports after each analysis.

**Display Multiple Sample Reports**

The Display Multiple Sample Reports checkbox displays the Multiple Sample Reports on screen. The system default is False. Click the checkbox to select this item and to suppress the printing of single sample reports after each analysis.

**Print Intensity Plots**

The Print Intensity Plots checkbox enables the automatic printing of sample and reference intensity plots. The system default is False. Click the checkbox to select this item.

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### **Print Calculated Intensities**

The Print Calculated Intensities checkbox enables automatic printing of net sample and net reference intensities by time gate. The system default is False. Click the checkbox to select this item.

### **Print Measured Intensities**

The Print Measured Intensities checkbox enables automatic printing of sample and reference intensities by time gate for raw data. The system default is False. Click the checkbox to select this item.

### **Display Results in Scientific Notation**

The Display Results in Scientific Notation checkbox enables the display and printing of the sample results using Scientific Notation format. The system default is False. Click the checkbox to select this item.

### **Required Login Input**

The Required Login Input frame contains two check box options to control the login process to the KPAWin® software. The software supports both the use of individual Login IDs along with or without associated passwords. Login User IDs is the Default and password protection is optional. Two classes of users are recognized: Supervisors and Analysts. Supervisors have complete access to the system. Analysts cannot add configuration tabs but can change the Active/Inactive checkbox for a configuration and can mark an alternative of any tab as "Current". The current configuration of any tab is the default of that configuration that is used at the time of sample input. The "General", "Hardware", and "Maintenance" tabs are fixed to the alternative that is selected as current when the background is analyzed for an analytical batch. All alternatives of the other five configuration tabs that are marked "Active" by deselecting the Active/Inactive checkbox on their tab are available at the time of sample input and selections can be changed on a sample-by-sample basis.

### **Require User ID's**

The Require User IDs checkbox requires the use of KPAWin® system User IDs. When User IDs are selected, the KPAWin® settings that are active when an individual logs out will be restored when that individual logs in again. The system default is True. Click the checkbox to deselect this item and login without User ID.

### **Require Passwords**

The Require Passwords checkbox requires the use of KPAWin® system Passwords by Supervisors and by Analysts. The system default is False. Click the checkbox to select this item.

### **Hardware Settings**

The Hardware Settings form contains two frames, an Active/Inactive checkbox, and a Database ID list box. The first frame manages the KPA hardware. The second frame manages optional automatic sampling equipment. Supervisor login with password reserves changes of this tab to the supervisor. Otherwise, it is modified by any valid user.

#### **InActive**

The Inactive checkbox indicates the displayed configuration is NOT currently active. Activate by clicking the Select command button. Select the Configuration ID to display and then clear the check mark.

#### **List of Available Configurations**

The List Test Box displays all available configurations for this configuration tab. Any active tab is selected from the list for the background and retained throughout the batch.

#### **KPA Hardware Setup**

The KPA Hardware Setup frame specifies the assignment of the KPA Serial Port, Serial Port Settings, KPA Eprom Version, and includes an Auto Changer selection check box.

#### **Serial Port (KPA)**

The Serial Port of the KPA is the computer port for communications with the KPA. The Default setting is 1.

#### **Serial Port Settings (KPA)**

The Serial Port Settings for the KPA Unit consist of baud, bits, parity, and stop bits delimited by commas. The default values for KPA-11 are 9600 BAUD, 8 Bits, No Parity, and 1 Stop Bit. For a KPA-10, settings of "4800 BAUD, 8 Bits, No Parity, and 1 Stop Bit" should be selected.

#### **Eprom Version**

The Eprom Version is a number from 1 to 3 that identifies the Eprom Version of the KPA. Version 3 is for a KPA-11. Version 2 is for a KPA-10 with automatic shutter control. Version 1 is for a KPA-10 with manual shutter control. The default is 3.

#### **Include Auto Changer**

The Include Auto Changer checkbox selects or deselects the Auto Changer as a component of the hardware configuration. The default is checked.

#### **Auto Changer Setup**

The Auto Changer Setup frame contains two elements and three subframes. The elements are Serial Port and Serial Port Settings for the changer. The subframes are Sample Changer, Syringe Pump, and Position Offsets.

#### **Serial Port (AutoChanger)**

The Serial Port AutoChanger is the computer port for communications with the Auto Changer. The Default setting is 2.

#### **Serial Port Settings (AutoChanger)**

The Serial Port Settings AutoChanger for the Auto Changer Unit consist of baud, bits, parity, and stop bits delimited by commas. The default values for Gilson equipment are 9600 BAUD, 8 Bits, No Parity, and 1 Stop Bit.

#### **Syringe Pump**

The Syringe Pump subframes contain three items to specify the Device Number, Model Number, and Flow Data Name and a Poll Button to acquire the three data items associated with the pump.

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### **Device Number (Syringe Pump)**

The Device Number selects the Syringe Pump Device Number. If unknown and Auto Changer is connected, select the Poll Button, and an attempt is made by the KPAWin© software to determine the correct value. The default is 1.

### **Model Number (Syringe Pump)**

The Model Number is the Gilson dilutor number. If unknown and an Auto Changer is connected, select the Poll Button and an attempt is made by the KPAWin© software to determine the correct value. The default is 402. The model number for syringe pumps delivered before the year 1999 is the 401.

### **Flow Data Name**

The Flow Data Name selects the appropriate Syringe Pump Flow Data Name. If unknown and Auto Changer is connected, select the Poll Button and an attempt is made by the KPAWin© software to determine the correct value. The default is SP402500 for pump model 402. The flow data name for pump model 401 is SP401500.

### **Syringe Pump Poll**

The Poll button activates the Syringe Pump and acquires the three data items associated with the pump: Device Number, Model Number, and Tray Data Name.

### **Sample Changer**

The Sample Changer subframes contain three items to specify the Device Number, Model Number, and Tray Data Name and a Poll Button to acquire the three data items associated with the changer.

### **Device Number (Sample Changer)**

The Device Number selects the Sample Changer Device Number. If unknown and Auto Changer is connected, select the Poll Button and an attempt is made by the KPAWin© software to determine the correct value. The default is 0.

### **Model Number (Sample Changer)**

The Model Number is the Gilson changer number. If unknown and Auto Changer is connected, select the Poll Button and an attempt is made by the KPAWin© software to determine the correct value. The default is 223. The model number for sample changers delivered before the year 2000 is the 222.

### **Tray Data Name**

The Tray Data Name is a concatenation of the racks in the sample tray as identified by their Gilson identification numbers. For example, the Tray Data Name of 24242429, the default, indicates the Gilson tray contains, from left to right, three model 24 racks and one model 29 rack. If unknown, select the Poll Button and an attempt is made by the KPAWin© software to determine the correct value.

### **Sample Changer Poll**

The Poll button activates the Sample Changer and acquires the three data items associated with the unit: Device Number, Model Number, and Tray Data Name.

### **Position Offsets**

The Position Offsets subframe provides offsets for the X, Y, and Z vial coordinates to compensate for differences between Sample Changer adjustments.

**X**

The X Position Offset in units of 1/10 mm from the position with no offset to the center of vial 1 of rack 1. The Default is 0.

**Y**

The Y Position Offset in units of 1/10 mm from the position with no offset to the center of vial 1 of rack 1. The Default is 0.

**Z**

The Z Position Offset in units of 1/10 mm from the position with no offset to the bottom of the sample container of vial 1 of rack 1. The Default is 0.



**KPA Unit Settings**

KPA Unit Settings form contains three frames, an Active/Inactive checkbox, and a Database ID list box. The first frame contains laser options. The second frame contains detection options. And the third frame contains time-gate options. Supervisor login with password reserves changes of this tab to the supervisor. Otherwise, it is modified by any valid user.

**InActive**

The Inactive checkbox indicates the displayed configuration is NOT currently active. Activate by clicking the Select command button. Select the Configuration ID to display and then clear the check mark.

**List of Available Configurations**

The List Test Box displays all available configurations for this configuration tab. Any active tab is selected from the list for the background and retained throughout the batch.

**Laser**

The Laser frame controls firing of the nitrogen laser and includes: Pulses Per Second, Pulses Per Analysis, Warm-Up Time, and Warm-Up Pulses.

**Pulses Per Second**

The Pulses Per Second setting specifies the number of pulses fired each second during an analysis. The default is 20.

**Pulses Per Analysis**

Pulses Per Analysis specifies the total number of laser pulses fired during each analysis. The default is 1000.

**Warm-Up Time (min)**

The Warm-Up Time specifies the time in minutes that elapse between analyses before the high-voltage transformer of the nitrogen laser is preformed with a series of laser pulses equal to the Warm-Up Pulses specification of the KPA Unit Settings configuration tab. The default is 15.

**Warm-Up Pulses**

The Warm-Up Pulses specifies the number of nitrogen laser pulse cycles preceding an analysis if the Warm-Up Time specified in the KPA Unit Settings configuration tab is exceeded. The default is 200.

**Detection**

The Detection frame provides correction and control for photomultiplier operation and includes PMT Dead Time, Minimum Intensity Factor, and Maximum Intensity Factor/Pulse.

**PMT Dead Time (ns)**

The PMT Dead Time is a factor correcting a nonlinear relationship between the logarithm of the intensity versus analyte concentration to a linear relationship. The correction, also known as the coincidence correction, compensates for saturation of the counting circuitry resulting from cycling the PMT on and off during periods of high photon flux. The dead time for PMT's used in KPA-11 instruments is 10 and is the default. The dead time for PMT's used in KPA-10s is uniquely determined for each PMT and its photon counting unit. The KPA-10 dead time has a default value of 120 and ranges from 110 to 150. The correction, applied to all raw intensity data, is  $(\text{RawIntensity} * \text{Pulses} * \text{GateWidth}) / (\text{Pulses} * \text{GateWidth} + \text{RawIntensity} * \text{DeadTime})$ .

**Minimum Intensity Factor**

The Minimum Intensity Factor sets a threshold from the total net intensity for sum of all laser cycles in each time gate of an analysis. The first time gate in an analysis with a total intensity below the Minimum Intensity Factor and the intensity data in all succeeding time gates is ignored in the least squares regression of the net intensity data. The default value is 1.

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## **Maximum Intensity Factor/Pulse**

The Maximum Intensity Factor/Pulse sets a maximum raw intensity for each laser pulse to identify data accumulated under saturation conditions. Time gates with intensities that exceed the Maximum Intensity Factor/Pulse specification are ignored in the least squares regression of the net intensity data. The default value for KPA-11 instruments is 180 with 200 as a maximum. The default value for KPA-10 instruments is 55 with a maximum of 60. Values that exceed this limit produce an Alarm or Warning issued to the operator. The default is 200.

## **Time Gates**

The Time Gates frame controls the multi-channel analyzer and the default least-squares analysis and includes Total Time Gates, Start LSR Gate, Number of LSR Gates, Minimum Number LSR Gates, Time Gate Width, High Intercept Tolerance, and Low Intercept Tolerance.

## **Total Time Gates**

The Total Time Gates is the number of time gates used to collect intensity data during an analysis. The default and maximum number is 127.

## **Start LSR Gate**

The Start LSR Gate defines the user-specified low-order time gate for the least square regression of the intensity data. The Start LSR Gate value shifts to a higher-order time gate if the raw intensity data in the user-specified gate exceeds the Maximum Intensity Factor/Pulse specification in the KPA Unit Settings configuration tab. The default value for uranium is 5 for the KPA-11 instrument and 4 for the KPA-10 instrument. The default value for lanthanides varies from 2 for samarium and thulium to 4 for dysprosium, europium, and terbium. See the KPA Operator's Manual or call Chemchek Instruments, Inc. for lanthanide settings.

## **Number of LSR Gates**

The Number of LSR Gates setting specifies the default number of time gates used in the analysis. The default number of time gates is truncated before the least-squares regression for the first corrected intensity less than the Minimum Intensity Factor of this configuration tab. The default is 45 for uranium. Bioassay for uranium in urine is improved with a selection of 35 LSR gates. See the KPA Operator's Manual or call Chemchek Instruments, Inc. for lanthanide settings.

## **Minimum Number LSR Gates**

The Minimum Number LSR Gates specifies the minimum number of time gates that must contain valid data to produce an analysis. The default is 15.

## **Time Gate Width ( $\mu$ s)**

The Time Gate Width specifies the width (Length of Time in  $\mu$ sec.) of each time gate used in the analysis. The default is 13 for uranium analysis. See the KPA Operator's Manual or call Chemchek Instruments, Inc. for lanthanide settings.

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## Range Selection

The Range Selection frame controls the automatic and manual selection of the KPA analytical ranges.

Auto range selection is based on sample precounts in the high range with range selection based on actual performance in the high range and inferred performance in the low range. Actual performance in the high range during automatic range selection uses the Minimum Intensity Factor specification in this tab to determine if enough counts can be detected during an analysis to assure that a successful least-squares regression of the data can be performed (no attempt will be made to take a logarithm of a negative number of counts after background subtraction). High-range precounts are used with the Maximum Intensity per Pulse specification to determine if the acquisition rate of counts in the fifth time gate would be excessive in the low range. The user-specified setting for the "Auto Efficiency Factor" in this frame predicts the ratio of counts in the low range to counts in the high range for a sample. Experience indicates that this factor is about 100 for samples with 1 µg/l of uranium and about 80-85 for samples with uranium concentrations where detection in both ranges is possible. multi-channel analyzer and the default least-squares analysis and includes Total Time Gates, Start LSR Gate, Number of LSR Gates, Minimum Number LSR Gates, Time Gate Width, High Intercept Tolerance, and Low Intercept Tolerance.

Manual range selection is based on evaluation of analytical data after an analysis based on the maximum concentration and minimum concentration of calibration samples for each KPA range. Analytical results that exceed the maximum concentration of calibration samples in the range of the analysis multiplied by the "High Intercept Tolerance" are flagged, if requested in the Alarms/Warnings Tab by the user. If the specification is violated in the low range and a high-range calibration exists, the analysis will be reattempted in the high range. Analogously, if the analytical result is less than the minimum concentration of the calibration samples in the high range (the low range is assumed to extend to zero concentration) multiplied by the Low Intercept Tolerance, the result is flagged and reattempted in the low range if a low-range calibration exists. If the range between the highest low-range calibration standard and the lowest high-range calibration standard is large and the intercept tolerances are close to 100%, range switching stops after the analysis in the second range, range errors are reported for both ranges, and the results for the second attempted analysis is reported.

### Auto Efficiency Factor

The Auto Efficiency Factor estimates what the Counts-Per-Pulse would be in the Low Range using the Precount value obtained during the Precount operation in the High Range. The default is 85.

### High Intercept Tolerance (%)

The High Intercept Tolerance (%) is used to extend the limits of the high and low calibration ranges by the percent indicated. The default is 120.

The High Intensity Tolerance (HIT) is the percentage of the highest concentration standard in either calibration range that can be used to quantify a sample result in that range. The default is 120 (%). The KPA Settings configuration tab will accept values for the Low Intensity Tolerance (%) of  $1 * HIT * 2000$ .

If the highest concentration standard for a low-range calibration is 10 ppb, an error will be reported and no analysis will be performed for a sample with a concentration that is greater than 12 ppb for a HIT of 120%. If a high-range calibration exists, the KPA will automatically reattempt the analysis in the high range. See Low Intensity Tolerance (%) in the Configuration - KPA Settings Help file for qualification of the reanalysis. If the reanalysis is also disqualified, then an error will be reported and no analysis will result for the existing calibration with the existing LIT and HIT values.

### Low Intercept Tolerance (%)

The Low Intercept Tolerance (%) is used to extend the limits of the high and low calibration ranges by the percent indicated. The default is 80.

The Low Intensity Tolerance (LIT) is the percentage of the lowest concentration standard in the high calibration range that can be used to quantify a sample result in that range. The default is 80 (%). The KPA Settings configuration tab will accept values for the Low Intensity Tolerance (%) of  $1 * HIT * 100$ .

If the lowest concentration standard for a high-range calibration is 10 ppb, an error will be reported and no analysis will be performed for a sample with a concentration that is less than 8 ppb for a LIT of 80%. If a low-range calibration exists, the KPA will automatically reattempt the analysis in the high range. See High Intensity Tolerance (%) in the Configuration - KPA Settings Help file for qualification of the reanalysis. If the reanalysis is also disqualified, then an error will be reported and no analysis will result for the existing calibration with the existing LIT and HIT values.

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## Configuration Tabs

## Calibration Settings

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### Calibration Settings

The Calibration Settings form contains four frames, an Active/Inactive checkbox, and a Database ID list box. The first frame contains general options. The second frame contains options for a high-range calibration. The third frame contains options for a low-range calibration. The fourth frame contains analysis settings. Supervisor login with password reserves changes of this tab to the supervisor. Otherwise, it is modified by any valid user.

### InActive

The Inactive checkbox indicates the displayed configuration is NOT currently active. Activate by clicking the Select command button. Select the Configuration ID to display and then clear the check mark.

### List of Available Configurations

The List Test Box displays all available configurations for this configuration tab. Any active tab is selected from the list for the background and retained throughout the batch.

### "Untitled"

The first frame includes Concentration Units, Specific Gravity, Specific Activity, Select Analyte, Atomic Mass, Calibration Ranges, and Dilution Method.

### Concentration Units

The Concentration Units selection is the dimensions for input and output values of concentration. All concentration data are recorded in the KPAWin® database in units of ug/l. The user is prompted to provide the Specific Gravity for concentrations on a per mass basis, Specific Activity for concentrations in radiometric units, and Atomic Mass for concentrations on a molar basis in the Calibration Settings configuration tab. Default values for these conversions are 1 g/ml for dilute aqueous solutions and 25,000 Bq/g (0.677 uCi/g) for natural uranium. The default for concentration units is ug/l. Select the appropriate Concentration Units from the dropdown list provided.

### Specific Gravity (g/ml)

Specific Gravity (g/ml) is a factor used to convert KPAWin® database concentrations in units of ug/l from input units to output units that are expressed on a per mass basis. The default value is 1 g/ml for dilute aqueous solutions.

### Specific Activity (Bq/g)

Specific Activity (Bq/g) is a factor used to convert KPAWin® database concentrations in units of ug/l from input units to output units that are expressed in radiometric units. The default value is 25,000 Bq/g for natural uranium.

### Select Analyte

The Select Analyte setting provides a dropdown list of the KPA analytes. A new selection here also sets a new default Atomic Mass. The default is Uranium.

### Atomic Mass

Atomic Mass (g/mole) is a factor used to convert KPAWin® database concentrations in units of ug/l from input units to output units expressed on a molar basis. The default value is 238.0289 g/mole for natural uranium.

### Calibration Ranges

The Calibration Ranges include low (only), high (only), both (high and low) or auto (high and/or low based on precount rates). Backgrounds are analyzed in the single range requested or in both ranges if both or auto is requested. Successive calibration samples are analyzed in the ranges specified in the sample input with analysis with precounts determining which range or ranges are analyzed for samples marked auto. The default is Auto.

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## **Dilution Method**

The Dilution Method dropdown includes Add Volume (used when the initial sample volume is added to prepare a final volume) or Total Volume (used when lab procedure dilutes an initial sample volume to a final total volume) methods to adjust sample volumes during preparation and before analysis. If Add Volume is selected, a five-fold dilution of 1 ml is expressed as an initial volume of 1 ml and a final volume of 4 ml. If Total Volume is selected, a five-fold dilution of 1 ml is expressed as an initial volume of 1 ml and a final volume of 5 ml. The default is Total Volume.

### **High-Range**

The High-Range calibration frame includes Equation Type, the Include Zero Point check box, and the Minimum Number of Standards.

### **Low-Range**

The Low-Range calibration frame includes Equation Type, the Include Zero Point check box, and the Minimum Number of Standards.

### **Equation Type**

The Calibration Equation Type dropdown list specifies the form of the calibration equation for the least square regression of the intensity intercepts versus true concentrations of calibration standards and backgrounds for both the low range and the high range. The available regressions are linear or quadratic and either force the fit through zero concentration (background) or do not force the fit through zero. The default is quadratic for the high range and linear for the low range.

### **Include Zero Point**

The Include Zero Point checkbox results in forcing the calibration equation through zero when selected. The default is unselected.

### **Minimum Number of Standards**

The Minimum Number of Standards specifies the minimum number of standards that MUST be analyzed for each specified range. The default for the high range and for the low range is 3.

### **Analysis Settings**

The Analysis Settings frame includes Smooth Analysis Results Data, Reference Correct Data, and the Proceed After Auto Calibration checkbox.

#### **Smooth Analysis Results Data**

The Smooth Analysis Results Data checkbox selects smoothing of the coincidence (dead time) corrected raw data for samples and references. Smoothing averages the intensities over five time gates for the central time gate for all data in the least square regression. E.g. the smoothed sample intensity for time gate 10 is the average of the coincidence-corrected sample intensities for time gates 8 through 12. Smoothed intensities for the first two time gates in the regression are not corrected but are set equal to their corresponding coincidence-corrected values. The system default sets this to TRUE. Click the checkbox to deselect this item.

#### **Reference Correct Data**

The Reference Correct Data checkbox indicates that smoothed intensities are divided by the reference ratio to create reference corrected intensities for each time gate in the least square regression. The system default sets this to TRUE. Click the checkbox to deselect this item. If smoothing is deselected, coincidence corrected intensities are used.

#### **Background Correct Data**

The Background Correct Data checkbox indicates that smoothed background intensities are subtracted from reference-corrected intensities to create net for each time gate for evaluation by a least square regression. The system default sets this to TRUE. Click the checkbox to deselect this item. If reference correction is deselected, smoothed intensities are used. If reference correction and smoothing are deselected, coincidence corrected background intensities are subtracted from coincidence corrected sample intensities. If all corrections are deselected, coincidence corrected sample intensities are evaluated by least square regression.

#### **Proceed After Auto Calibration**



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The Proceed After Auto Calibration checkbox specifies automatic analysis of samples and standards proceeds without pausing for user review after the calibration. The system default sets this to FALSE. Click the checkbox to select this item.

**Maintenance**

The Maintenance form contains three frames, an Active/Inactive checkbox, and a Database ID list box. The first frame contains general options. The second frame contains options for excitation and confidence testing and includes Excitation Test Standard, Dye Strength, and Laser Power. The third frame contains instrument maintenance and service information. It includes Laser Plasma Cartridge Change, Dye Change, Reference Change, and Scheduled Preventive Maintenance. Supervisor login with password reserves changes of this tab to the supervisor. Otherwise, it is modified by any valid user.

**InActive**

The Inactive checkbox indicates the displayed configuration is NOT currently active. Activate by clicking the Select command button. Select the Configuration ID to display and then clear the check mark.

**List of Available Configurations**

The List Test Box displays all available configurations for this configuration tab. Any active tab is selected from the list for the background and retained throughout the batch.

**Maintenance Options**

The Maintenance Options frame provides a choice among three alternative uses of the Maintenance tab: Use Defaults, Use Site Custom, or Do Not Use Limits.

**Use Defaults**

The Use Defaults option button selects the 1000 Release configuration for Maintenance Options. These limits are parameters supplied by Chemchek Instruments Inc. as defaults for the software.

**Use Site Custom**

The Use Site Custom option button opens the Maintenance Options configuration tab for user-specified entries and is the default.

**Do Not Use Limits**

The Do Not Use Limits option button selects the 999 No Limits configuration for the Maintenance Options. This allows a site to DISABLE the use of all of the KPAWin© software performance limits.

**Excitation Test Standard (ug/l)**

The Excitation Test Standard (ug/l) specifies the nominal concentration of a standard that supports Dye Strength and Laser Power in the Maintenance configuration tab. The intensity intercept for each analysis of the standard after a Dye change is normalized to the value of the excitation test standard and stored in the database for the trend analysis of Dye Strength. Similarly, each intensity intercept for each analysis of the standard with fresh dye is normalized to the value of the excitation test standard and stored in the database for the trend analysis of Laser Power. Alarms are activated when either normalized intercept is less than the specifications for Dye Strength or Laser Power in the Maintenance Options configuration tab. Select a trend line for Dye Strength or Laser Power as a function of date. Select from the dropdown Tools on the main menu. The default is 1.

**Dye Strength (Intercept counts/pulse)**

The Dye Strength (intercept counts/pulse) entry specifies the minimum intensity intercept counts per laser pulse for the excitation test standard. The default is 10.

**Laser Power (Intercept counts/pulse)**

The Laser Power (intercept counts/pulse) entry specifies the minimum intensity intercept counts per laser pulse for the excitation test standard. The default is 10.

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### **Number of Confidence Test Repeat Analyses**

The Number of Confidence Test Repeat Analyses is the number of replicate analyses performed on a confidence test sample to estimate the system reproducibility. The default is 3.

### **Confidence Test Analysis Range**

The Confidence Test Analysis Range is the range used to analyze confidence test samples. The default is Low.

### **Laser Plasma Cartridge Change (months)**

The Laser Plasma Cartridge Change (months) is the number of months from the installation of a new laser plasma cartridge. Last Occurrence is the date of the last cartridge change in the mm/dd/yyyy format. The Last Occurrence date defines the beginning of the Laser Power trend analysis. The default is 18.

### **Dye Change (months)**

The Dye Change (months) is the number of months from the replenishment of the excitation dye in the dye laser. Last Occurrence is the date the excitation dye was replaced in the dye laser in the mm/dd/yyyy format. The Last Occurrence date defines the beginning of the Dye Strength trend analysis. The default is 3.

### **Reference Change (days)**

The Reference Change (days) is the number of days from the replenishment of the standard in the reference cuvette. Last Occurrence is the date the standard was changed in the reference cuvette. The default is 7.

### **Scheduled Preventive Maintenance (months)**

The Scheduled Preventive Maintenance (months) is the number of months from the last preventive maintenance service of the KPA. The default is 12.

**Performance Limits**

The Performance Limits form contains three frames, an Active/Inactive checkbox, and a Database ID list box. The frames provide control over KPA-specific data quality metrics. The first frame contains general options. The second frame contains options for Calibration Limits. The third frame contains options for Analysis Limits. Supervisor login with password reserves changes of this tab to the supervisor. Otherwise, it is modified by any valid user.

**InActive**

The Inactive checkbox indicates the displayed configuration is NOT currently active. Activate by clicking the Select command button. Select the Configuration ID to display and then clear the check mark.

**List of Available Configurations**

The List Test Box displays all available configurations for this configuration tab. Any active tab is selected from the list for the background and retained throughout the batch.

**Performance Limits Options**

The Performance Limits Option frame provides a choice among three alternative uses of the Performance Limits tab: Use Defaults, Use Site Custom, or Do Not Use Limits.

**Use Defaults**

The Use Defaults option button selects the 1000 Release configuration for Performance Limits. These limits are parameters supplied by Chemchek Instruments Inc. as defaults for the software.

**Use Site Custom**

The Use Site Custom option button opens the Performance Limits Option configuration tab for user-specified entries.

**Do Not Use Limits**

The Do Not Use Limits option button selects the 999 No Limits configuration for Performance Limits. This allows a site to DISABLE the use of all the KPAWin© software performance limits.

**Calibration Limits**

The Calibration Limits frame contains five metrics that relate to Maximum Background High Range, Maximum Background Low Range, Calibration R Squared, Percent Discrepancy of Calibration Points, and Maximum Calibration Duration.

**Maximum Background High Range**

The Maximum Background High Range is the upper intensity limit of the Background used in the HIGH Range. If this limit is exceeded, an Alarm is issued to the operator and the calibration halted until the situation is corrected. The default value is 25.

**Maximum Background Low Range**

The Maximum Background Low Range is a control that alarms when the intensity intercept for the Background exceeds the specification in the low Range. The default value is 1500.

**Calibration R Squared**

The Calibration R Squared is a control that alarms when the correlation coefficient of the least-squares regression of intensity intercepts of calibration standards is less than the specification. The default value is 0.90.

**Percent Discrepancy**

The Percent Discrepancy is a control that alarms when the difference between the intensity intercept of a calibration standard and

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the value calculated from the calibration equation exceeds the specification. The default value is 10.

#### **Maximum Calibration Duration (days)**

The Maximum Calibration Duration is a control that alarms when the difference between the analysis time/date for a sample and the time/date for the calibration exceed the specification. The default value is 100 days.

#### **Analysis Limits**

The Analysis Limits frame contains seven analysis metrics that relate to minimum Sample R Squared, Minimum Sample Lifetime, Maximum Sample Lifetime, Minimum Reference Lifetime, Maximum Reference Ratio Deviation, Maximum Gate Shifting, and Maximum Gate Dropping.

#### **Sample R Squared**

The Sample R Squared is a control that alarms when the correlation coefficient of the least-squares regression of the corrected sample intensities is less than the specification. The default value is 0.90.

#### **Minimum Sample Lifetime ( $\mu$ s)**

The Minimum Sample Lifetime is a control that alarms when the lifetime of a sample is less than the specification. The default value is 150.

#### **Maximum Sample Lifetime ( $\mu$ s)**

The Maximum Sample Lifetime is a control that alarms when the lifetime of a sample exceeds the specification. The default value is 350.

#### **Minimum Reference Lifetime ( $\mu$ s)**

The Minimum Reference Lifetime is a control that alarms when the lifetime of the reference is less than the specification. The default value is 150.

#### **Reference Ratio (+/-)**

The Reference Ratio is the reference intensity intercept at the time the sample is analyzed divided by the reference intensity intercept at the time the background sample was analyzed. Sample intensities corrected for coincidence and smoothed (if smoothing is selected in the Calibration Settings configuration tab) are divided by the Reference Ratio (if reference correction is selected in the Calibration Settings configuration tab) to provide reference corrected sample intensities. To preclude division by zero, the default ratio is 0.2.

#### **Gate Shifting**

The Gate Shifting setting is the maximum difference between the Start LSR Gate specification in the KPA Unit Settings configuration tab and the actual start gate of the least-squares regression of the corrected sample intensities. Gate Shifting from the configuration specification occurs when the raw sample intensity of any time gate exceeds the Maximum Intensity Factor/Pulse specification in the KPA Unit Settings configuration tab. The default is 20 gates.

#### **Gate Dropping**

The Gate Dropping setting is the maximum difference between the Number of LSR Gates specification in the KPA Unit Settings configuration tab and the actual number of gates in the least-squares regression of the corrected sample intensities. Gate Dropping truncates the intensity vector at the first time gate within the specification for the number of gates with a corrected intensity less than the Minimum Intensity Factor specified in the KPA Unit Settings configuration tab. The default is 5 gates.



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## **Configuration Tabs**

## **Quality Control Limits Settings**

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### **InActive**

The Inactive checkbox indicates the displayed configuration is NOT currently active. Activate by clicking the Select command button. Select the Configuration ID to display and then clear the check mark.

### **List of Available Configurations**

The List Test Box displays all available configurations for this configuration tab. Any active tab is selected from the list for the background and retained throughout the batch.

### **Quality Control Limits**

The Quality Control Limits contains five frames, an Active/Inactive checkbox, and a Database ID list box. The first frame contains quality control options. The second frame contains detection limit options. The third frame contains calibration control options. The fourth frame contains method control options. And the fifth frame contains sample interference control options. Supervisor login with password reserves changes of this tab to the supervisor. Otherwise, it is modified by any valid user.

### **QC Limit Options**

The QC Limits Options frame provides a choice among three alternative uses of the QC Limits tab: Use Defaults, Use Site Custom, or Do Not Use Limits.

#### **Use Defaults**

The Use Defaults option button selects the 1000 Release configuration for QC Limit Options. These limits are parameters supplied by Chemchek Inc. as defaults for the software.

#### **Use Site Custom**

The Use Site Custom option button opens the QC Limit Options configuration tab for user-specified entries.

#### **Do Not Use Limits**

The Do Not Use Limits option button selects the 999 No Limits for the QC Limits Options. This allows a site to DISABLE the use of all the KPAWin© software Quality Control Checks.

### **Detection Limits**

The Detection Limits frame specifies parameters for the IDL, MDL, and CRDL. The IDL and MDL parameters include Number of Standards, Type of Statistic, Maximum Duration, Maximum Concentration Factor, and Limit. The CRDL list only pertains to Limit.

#### **IDL / MDL / CRDL**

The IDL & MDL & CRDL entries list specifications for the alternative detection limits, the number of standards required to estimate a limit, the type of calculation used to estimate the limit, the maximum age of the data used in the estimation, the proximity of the average true concentrations of detection standards to the estimated detection limit, and the maximum value of the estimated detection limit.

#### **Number of Standards**

The Number of Standards specifies the number of Type 1 measurements or Type 2 daily detection limits required for the estimation of a detection limit. The default for IDL and for MDL is 7.

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## Type

The Type detection limit is a KPAWin® term specifying the data collection and analysis protocol used to estimate a detection limit. A Type 1 detection limit is the sample standard deviation of the most recently measured concentrations of a detection limit standard. Each measurement consists of a single observation. The total number of measurements in the estimate must be equal to the specified number of standards and the observations must be made within the number of weeks specified in the maximum duration. A Type 2 detection limit is the average of a series of sample standard deviations. Each sample standard deviation is estimated from a series of analyses of a single detection standard on a single day. Different standards with different true analyte concentrations can be used on different days. The total number of standard deviations must be equal to the specified number of standards and the observations must be made within the number of weeks specified in the maximum duration. The default for IDL and for MDL is 1.

## Maximum Duration

The Maximum Duration is the maximum period in weeks that the detection limit data is collected. The default for IDL and for MDL is 13.

## Maximum Concentration Factor

The Maximum Concentration Factor is the maximum observed average concentration of the detection limit standard(s) expressed as a multiple of the detection limit. The default for IDL and for MDL is 5.

## Limit ug/l

The Limit ug/l is the maximum acceptable value of a detection limit. The default for IDL and for MDL is 0.1 and for CRDL is 1.

## Calibration Control

The Calibration Control frame specifies parameters for Initial and Continuing Calibration Control. Parameters include Blank and Standard Error for both the Initial and Continuing Calibration Control.

## Initial / Continuing

The Initial & Continuing entries list alternative control limits for analyte in a check blank and recovery of analyte from a check standard. Initial limits confirm the performance of check samples immediately after calibration. Continuing limits confirm the performance of check samples throughout and at the end of an analytical batch.

## Blank (\*IDL)

The Blank (\*IDL) is the maximum acceptable concentration of a calibration check blank expressed as a multiple of the IDL. Unique values are entered for the initial calibration check blank analyzed immediately after calibration or the continuing calibration check blank analyzed subsequently. The default for Initial and for Continuing is 3.

## Calibration Control Standard Error (%)

The Standard Error is the maximum acceptable percentage difference between the true concentration of the calibration control standard and its detected concentration. The error is the absolute difference between the true concentration and the detected concentration as a percentage of the true concentration. Unique recovery limits are entered for the initial calibration control standard analyzed immediately after calibration or the continuing control standard analyzed subsequently. The defaults are 10 for Initial and 20 for Continuing.

## Method Control

The Method Control frame includes four alternative options for specifying the maximum concentration of the Method Blank Control and the Standard Error of the Method Standard. Alternatives for the maximum acceptable Method Blank Control are MDL Multiple, CRDL Percentage, Sample Percentage, or Use Highest of the Above (previous three values).

## MDL Multiple

The MDL Multiple is the maximum acceptable concentration of a method blank if the MDL Multiple or Use Highest of the Above checkboxes are checked. The default is 3.

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### **CRDL Percentage**

The CRDL Percentage is the maximum acceptable concentration of a method blank if the CRDL Percentage or Use Highest of the Above checkboxes is checked. The default is 5.

### **Sample Percentage**

The Sample Percentage is the maximum acceptable concentration of a method blank if the Sample Percentage or Use Highest of the Above checkboxes is checked. The default is 5.

### **Use Highest of the Above**

The Use Highest of the Above specifies the concentration of the method blank cannot exceed the largest of three values: the MDL Multiple, the CRDL Percentage, or the Sample Percentage. CRDL is the acronym for contract reporting detection limit. The default is unselected.

### **Method Control Standard Error (+/-%)**

The Method Control Standard Error +/- % is the maximum acceptable percentage difference between the true concentration of the Laboratory Control Standard and its detected concentration. The error is the absolute difference between the true concentration and the detected concentration as a percentage of the true concentration. The default is 25.

### **Sample Control**

The Sample Control frame provides parameters to evaluate interference to analyte detection in samples. Control parameters include Relative Percent Deviation (RPD), Spike Recovery, Serial Dilution Error and four values for the Method of Standard Additions with a valid external calibration. The Method of Standard Additions values are Number of Spikes, Recovery Error, Slope Error and R Squared.

### **Precision (%)**

Precision is the Relative Percent Difference of a sample and a sample duplicate. The specification is the maximum acceptable relative percent deviation of a sample and its duplicate. The deviation is the absolute difference of the observations as a percentage of their average. The default is 20.

### **Spike Recovery (%)**

The Spike Recovery is the maximum acceptable percent recovery of a known spike from a sample. The recovery is the difference between true concentration of the spike in the sample and its incremental increase in concentration from the sample as a percentage of the true concentration. The default is 15.

### **Serial Dilution Error (%)**

The Serial Dilution Error is the maximum acceptable percentage difference between observations of a sample and a dilution of the sample corrected to their original concentrations. The error is the absolute difference between the serial dilution result and the undiluted sample as a percentage of the serial dilution result. The default is 10.

### **Method of Standard Addition**

The Method of Standard Addition entries list the number of sample spikes for comparison of the external batch calibration with the internal spike calibration and the performance criteria for a protocol with multiple sample spikes.

### **Number of Spikes**

The Number of Spikes specifies the number of unique spikes in a series of Method of Standard Addition samples. The Method of Standard Additions uses the external batch calibration to estimate sample interference with the recovery of a known spike. Analysis of the sample and three step-wise spike increases in analyte concentration also return estimates for the comparison of the external and internal calibration slopes and the curvature of the internal calibration. The default is 1.

### **Recovery Error (%)**

The MSA Recovery Error is the maximum acceptable percentage error in the recovery of a known spike(s) from a sample by the Method of Standard Additions. The error is the absolute difference between the true analyte concentration of the spike and the incremental increase in the concentration of the spiked sample as a percentage of the true concentration. The default is 25.

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**Slope Error (%)**

The MSA Slope Error is the maximum acceptable percentage difference between the slope of the external calibration and the slope, from the Method of Standard Additions for analyte recovery from the analysis of an unspiked sample and three spikes of the sample with step-wise increases of the analyte. The error is the absolute difference of the slopes of the external and internal calibrations as a percentage of the slope of the external calibration. The default is 20.

**R Squared**

The MSA R Squared is the correlation coefficient of the internal Method of Standard Additions calibration. The default is 0.96.

**Alarms/Warnings**

The Alarms/Warnings form contains a listing of off-standard conditions with Alarm or Warning check boxes, a Stop Processing on Alarm check box, an Active/Inactive checkbox, and a Database Id list box. Supervisor login with password reserves changes of this tab to the supervisor. Otherwise, it is modified by any valid user. Default selections are deselect.

**InActive**

The Inactive checkbox indicates the displayed configuration is NOT currently active. Activate by clicking the Select command button. Select the Configuration ID to display and then clear the check mark.

**List of Available Configurations**

The List Test Box displays all available configurations for this configuration tab. Any active tab is selected from the list for the background and retained throughout the batch.

**STOP KPA Processing on Alarms**

The Check Alarms will STOP KPA Processing checkbox indicates that analysis is interrupted if a selected alarm tolerance is exceeded. User response is required to resume normal analysis.

**QC Alarms/Warnings**

This display lists the KPA and KPAWin® failure modes that alarm or warn of off-standard analytical conditions. Failure modes of concern and responses are selected by the site supervisor. The tolerance level of alarms is set to specifications in the configuration tabs. The Alarms / Warnings Messages are listed next.

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## **Alarms / Warnings Message List**

Alarm / Warning #1	Background High - High Range
Alarm / Warning #2	Background High - Low Range
Alarm / Warning #3	Calibration Correlation (R Squared) Low - High Range
Alarm / Warning #4	Calibration Correlation (R Squared) Low - Low Range
Alarm / Warning #5	Calibration Standard Discrepancy High - High Range
Alarm / Warning #6	Calibration Standard Discrepancy High - Low Range
Alarm / Warning #7	Maximum Calibration Age Exceeded
Alarm / Warning #8	Sample Correlation (R Squared) Low
Alarm / Warning #9	Sample Lifetime Low
Alarm / Warning #10	Sample Lifetime High
Alarm / Warning #11	Reference Lifetime Low
Alarm / Warning #12	Reference Ratio High
Alarm / Warning #13	Reference Ratio Low
Alarm / Warning #14	Gate Shifting Excessive
Alarm / Warning #15	Gate Dropping Excessive
Alarm / Warning #16	Sample Concentration Out of Calibration Range
Alarm / Warning #17	Too Few Valid Time Gates For Evaluation
Alarm / Warning #18	Sample Concentration High For Low Range
Alarm / Warning #19	Sample Concentration Low For High Range
Alarm / Warning #20	Dye Output Low
Alarm / Warning #21	Laser Plasma Cartridge Output Low
Alarm / Warning #22	Laser Plasma Cartridge Age Exceeds Service Life
Alarm / Warning #23	Dye Age Exceeds Service Life
Alarm / Warning #24	Reference Age Exceeds Service Life
Alarm / Warning #25	Schedule Preventive Maintenance
Alarm / Warning #26	Too Few Calibration Points - High Range
Alarm / Warning #27	Too Few Calibration Points - Low Range
Alarm / Warning #28	Initial Calibration Check Blank High - High Range
Alarm / Warning #29	Initial Calibration Check Blank High - Low Range
Alarm / Warning #30	Initial Calibration Check Standard Invalid - High Range
Alarm / Warning #31	Initial Calibration Check Standard Invalid - Low Range
Alarm / Warning #32	Continuing Calibration Check Blank High - High Range
Alarm / Warning #33	Continuing Calibration Check Blank High - Low Range
Alarm / Warning #34	Continuing Calibration Check Standard Invalid - High Range
Alarm / Warning #35	Continuing Calibration Check Standard Invalid - Low Range
Alarm / Warning #36	Too Few IDL Standards - Type 1 IDL
Alarm / Warning #37	Too Few IDL Averages - Type 2 IDL
Alarm / Warning #38	Average Concentration of IDL's High
Alarm / Warning #39	IDL Above Maximum Acceptable Value
Alarm / Warning #40	Too Few MDL Standards - Type 1 MDL
Alarm / Warning #41	Too Few MDL Averages - Type 2 MDL
Alarm / Warning #42	Average Concentration of MDL's High
Alarm / Warning #43	MDL Above Maximum Acceptable Value
Alarm / Warning #44	Relative Percent Deviation High
Alarm / Warning #45	Sample Spike Recovery High
Alarm / Warning #46	Sample Spike Recovery Low
Alarm / Warning #47	Serial Dilution Recovery High
Alarm / Warning #48	Serial Dilution Recovery Low
Alarm / Warning #49	Multiple MSA Spike Recovery High
Alarm / Warning #50	Multiple MSA Spike Recovery Low
Alarm / Warning #51	MSA Spike Recovery High
Alarm / Warning #52	MSA Spike Recovery Low
Alarm / Warning #53	Multiple MSA Slope Error High
Alarm / Warning #54	Multiple MSA Calibration Correlation (R Square) Low
Alarm / Warning #55	MDL Multiple Exceeds Min Sample Result
Alarm / Warning #56	CRDL% Exceeds Min Sample Result



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Alarm / Warning #57	Sample % Exceeds Min Sample Result
Alarm / Warning #58	MDL Multiple, CRDL%, or Sample % Exceeds Min Sample Result
Alarm / Warning #59	Method Standard Invalid - High Range
Alarm / Warning #60	Method Standard Invalid - Low Range
Alarm / Warning #61	Precounts High For High Range
Alarm / Warning #62	Precounts Low For High Range
Alarm / Warning #63	Precounts High For Low Range
Alarm / Warning #64	No Concentration Unit Conversion Available
Alarm / Warning #65	Units Conversion / Check Specific Gravity
Alarm / Warning #66	Units Conversion / Check Specific Activity
Alarm / Warning #67	Units Conversion / Check Atomic Mass
Alarm / Warning #68	Invalid Reference Intensities
Alarm / Warning #69	No Background - High Range
Alarm / Warning #70	No Background - Low Range
Alarm / Warning #71	Gate Shifting
Alarm / Warning #72	Gate Dropping
Alarm / Warning #73	Sample Intensity Intercept Below Background
Alarm / Warning #74	Sample Intensity Intercept Below Quenched Background
Alarm / Warning #75	No Calibration - High Range
Alarm / Warning #76	No Calibration - Low Range
Alarm / Warning #77	No Basis Sample For Sample Duplicate
Alarm / Warning #78	No Basis Sample For Sample Spike
Alarm / Warning #79	No Basis Sample For Quenched Background
Alarm / Warning #80	No Basis Sample For Method of Standard Addition
Alarm / Warning #81	No Basis Sample For Standard Addition Spike
Alarm / Warning #82	No Basis Sample For Serial Dilution
Alarm / Warning #83	Reference Correlation (R Squared) Low
Alarm / Warning #84	Reference Intensity Low
Alarm / Warning #85	Reference Intensity High
Alarm / Warning #86	Maximum of 7 Calibration Standards Exceeded Oldest Ignored – High Range
Alarm / Warning #87	Maximum of 7 Calibration Standards Exceeded Oldest Ignored – Low Range
Alarm / Warning #88	Sample Concentration High For High Range

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## **KPAWin© General Display Documentation**

### **"Kinetic Phosphorescence Analyzer System"**

#### **"Contacts"**

The "Contacts" button displays the Chemchek contacts, street address, email address, phone number and fax number.

#### **"OK"**

The "OK" button exits the "About KPAWin" display and returns to the previous display.

#### **"Technical Info"**

The "Technical Info" button displays the KPAWin© hardware and software requirements to run the KPAWin© software package.

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## **"Customer Administration Menu"**

### **Summary Description**

The Customer Administration form has a number of text areas that contain customer information: name, address, contact person, telephone numbers, and if applicable, e-mail address. It also contains a checkbox indicating if the above listed customer is Active or Inactive. Setting or clearing the checkbox changes the Active/Inactive Status. If the Select button is depressed, a list of current customers from the KPAWin© Database is displayed. The user then selects one of these customers and the database information populates the customer text areas with the database entries.

### **"Customer Dropdown List"**

The "Customer" drop-down list displays all Customers currently stored in the KPAWin© Database. It is displayed when the Select button is depressed.

### **"Inactive Customer"**

The "Inactive" checkbox identifies a Customer that is not currently Active.

### **"Add"**

The "Add" button opens a new Customer Administration form and positions the cursor in the first entry of the form.

### **"Apply"**

The "Apply" button updates the KPAWin© Database and returns to the Customer Administration display.

### **"Cancel"**

The "Cancel" button restores the Customer List form and the KPAWin© Database to the original values, exits the current display and returns to the previous display.

### **"Clear Entries"**

The "Clear" button clears all existing data from the Customer Administration screen and returns to the current display.

### **"OK"**

The "OK" button updates any record in the Customer Administration form and the KPAWin© Database, exits the current display and returns to the previous display.

### **"Select"**

The "Select" button queries the KPAWin© Database and displays a drop-down list of all active Customers in the Database. The user then selects a Customer entry to edit.

### **"Customer List"**

The "Customer List" frame manages customer accounts. Accounts are created, edited, marked as active or inactive, and entered into the KPAWin© database. Each account contains the following locator information: name, address, FAX number, e-mail address, customer contact, and customer telephone number.

### **"Customer ID"**

The "Customer Id" identifies the Customer or Project that submitted the sample. The laboratory can identify calibration and control samples. This field must be completed for all samples. All active Customer Ids are viewed in the Customer Id drop-down list of the Sample Input form.

### **"Customer Contact"**

Position of Customer's contact person(s).

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**"Customer's Telephone Number"**

Telephone number of Customer's contact person(s).

**"Customer's Fax Number"**

Customer's Fax number. Delimit fields according to modem standards for direct dial capability.

**"Customer's Email Address"**

Customer's e-mail address.

**"Customer's Name"**

Customer's name.

**"Customer's Address"**

Customer's street address.

**"Customer's City"**

Customer's city.

**"Customer's State"**

Customer's state.

**"Customer's Zip Code"**

Customer's ZIP code.

**"Customer's Country"**

Country where the Customer is located.

**"Customer's First Name"**

First name of Customer's contact person(s).

**"Customer's Last Name"**

Last name of Customer's contact person(s).

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## **"Calibration Report"**

### **Summary Description**

The "Calibration Report" displays the results of the calibration. The report includes the results and evaluation of the calibration by range for the background and standards.

### **"User Cal"**

The "User Cal" checkbox disables the automatic calibration and permits the user to either exclude or include each of the calibration points. When this item is selected, the Calibration Id text item is also enabled and the user may enter a unique name for the calibration.

### **"Calibrate"**

The "Calibrate" button forces a recalibration of the KPA with the User's selection of equations, configurations, and calibration standards. When completed, the new results are updated on the same Calibration form.

### **"Plot"**

The "Plot" button displays calibration curves for intensity intercepts versus calibration standard concentrations for all calibrated ranges. The button toggles between Plot to display the curves and Grid to display the data and results.

### **"OK"**

The "OK" command button exits the calibration display and returns to the previous display.

### **"Print"**

The "Print" button queues both calibration grid information, if it is on display, as well as the plots, if they are on display, to the system-selected printer.

### **"Save As"**

The "Save As" command is only displayed when the plots are being displayed. It saves the calibration plots to a user-selected file for display or printing at a later time.

### **"Calibration Details"**

"Calibration Details" identifies the Laboratory, Customer, Analyst, Procedure, and Calibration Configuration Ids.

### **"High Range Details"**

"High Range Details" identifies the high-range Calibration Id, Minimum Number of Standards used in the Calibration, Alarms that were selected and exceeded, the Calibration  $R^2$ , and Calibration Variance.

### **"Low Range Details"**

"Low Range Details" identifies the low-range Calibration Id, Minimum Number of Standards used in the Calibration, Alarms that were selected and exceeded, the Calibration  $R^2$ , and Calibration Variance.

### **"Calibration Grid Information"**

The Calibration grid displays the Calibration Range, a Use Flag, the Sample Ids, Sample Types, Standard Concentrations, Standard Ids, Intercepts, Uncertainties, Percent Discrepancies, Descriptions, Date/Times, Time Gates, Reference Ratios, Lifetimes, Calibration  $R^2$  Intensities, and the Integrated Intensities. Use flags include "+", "-", and ". KPAWin© automatically assigns a "+" flag to qualified calibration points that are included in the default calibration or a "-" flag for invalid points. Selection of the User Cal checkbox permits the user to select by toggling between "+" for included and "-" for not included. Toggling the flag is accomplished by Double-Left Clicking with the mouse on the standard flag "+" or "-".

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### **"Lab ID"**

The "Lab Id" indicates the identity of the performing laboratory. Complete information for the laboratory is contained in the Laboratory Database accessed from the menu bar.

### **"Customer ID"**

The "Customer Id" indicates a specific source of samples to be quantitated by the calibration. Complete information for the customer is contained in the Customer Database accessed from the menu bar. The Lab Id can be used as default.

### **"Analyst"**

The "Analyst" establishes the identity of the laboratory employee performing the calibration. Complete information for the analyst is contained in the User Administration Database under Security accessed from the menu bar.

### **"Procedure ID"**

The "Procedure Id" indicates the formal laboratory method(s) that were used to analyze the background and calibration standards. Complete information for the procedure is contained in the Procedure Database accessed from the menu bar.

### **"Cal Config ID"**

The "Calibration Configuration Id" identifies the Calibration Settings Configuration tab used to collect the calibration data.

### **"High Calibration ID"**

The "Calibration Id" identifies a unique high-range calibration for a specific background analysis and specific calibration standard analyses by a linear or quadratic equation by least-squares regression that forces the fit through zero concentration for zero net intensity or does not force the fit through zero. If the User Cal checkbox is checked, the user may delete standards and change the order and intercept of the calibration equation.

### **"Min Nb Stds"**

The "Minimum Number of Standards" specifies the smallest number of calibration standards required for a given high-range calibration.

### **"Alarms"**

"Alarms" lists the user-specified performance measures in the Alarm/Warning Configuration tab violated by the high-range calibration. Alarms are listed with an "A" prefix. Warnings are listed with a "W" prefix. The specific off-standard condition is indicated by a unique two-digit number.

### **"High R Squared"**

"R Squared" is the correlation coefficient of the least-squares regression of the background and calibration standards for the high-range calibration.

### **"Variance"**

The "Variance" is estimated from the differences between the true concentrations of the background and calibration standards for the high-range calibration and the concentrations estimated for their analytical intercept intensities from the calibration equation.

### **"Cal Eq Type"**

The "Calibration Equation Type" for the high-calibration range is either linear for a first order relation of intercept intensity to concentration or quadratic for a second order relationship between intercept intensities and concentrations.

### **"Cal Eq"**

The "Calibration Equation" is the high-range calibration equation with concentration as the independent variable and intercept intensity as the dependent variable.



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### **"Low Calibration ID"**

The "Calibration Id" identifies a unique low-range calibration for a specific background analysis and specific calibration standard analyses by a linear or quadratic equation by least-squares regression that forces the fit through zero concentration for zero net intensity or does not force the fit through zero. If the User Cal checkbox is checked, the user may delete standards and change the order and intercept of the calibration equation.

### **"Min Nb Stds"**

The "Minimum Number of Standards" specifies the smallest number of calibration standards required for a given low-range calibration.

### **"Alarms"**

"Alarms" lists the user-specified performance measures in the Alarm/Warning Configuration tab violated by the low-range calibration. Alarms are listed with an "A" prefix. Warnings are listed with a "W" prefix. The specific off-standard condition is indicated by a unique two-digit number.

### **"Low R Squared"**

"R Squared" is the correlation coefficient of the least-squares regression of the background and calibration standards for the low-range calibration.

### **"Variance"**

The "Variance" is estimated from the differences between the true concentrations of the background and calibration standards for the low-range calibration and the concentrations estimated for their analytical intercept intensities from the calibration equation.

### **"Cal Eq Type"**

The "Calibration Equation Type" for the low-calibration range is either linear for a first order relation of intercept intensity to concentration or quadratic for a second order relationship between intercept intensities and concentrations.

### **"Cal Eq"**

The "Calibration Equation" is the low-range calibration equation with concentration as the independent variable and intercept intensity as the dependent variable.

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## **"KPA Confidence Testing"**

### **Summary Description**

The "Confidence Test" report displays the coefficient of variation for a series of analyses on the same sample that are run in uninterrupted order without changing cuvettes or any KPA parameter. This parameter measures the stability of the analytical system. The coefficient of variation is the standard deviation of the intercepts of the intensity data that have been corrected for coincidence and been smoothed. No prior background analysis is required. The coefficient of variation is the ratio of the standard deviation of the replicate measurements divided by their true (makeup) concentrations. Instrument inputs to the test are the Number of Confidence Test Repeat Analyses in the Maintenance Configuration Tab and the Number of Pulses in the KPA Unit Settings Configuration Tab.

### **"Exit"**

The "Exit" command button closes the Confidence Test display and returns to the previous display.

### **"Help"**

The "Help" command button displays the KPAWin® Help contents screen.

### **"Sample ID"**

The "Sample Id" displays the Sample Id of the Confidence Test.

### **"Standard ID"**

The "Standard Id" displays the laboratory identification of the standard that is used to conduct the Confidence Test.

### **"Coefficient of Variation"**

The "Coefficient of Variation" displays the dimensionless quotient of the standard deviation of the replicate intensity intercepts divided by their true (makeup) concentration.

### **"Description"**

The "Description" is the name of the Confidence Sample.

### **"Standard Concentration"**

The "Standard Concentration" displays the true (makeup) concentration of the Confidence Test sample.

### **"Confidence Test Grid"**

The "Confidence Test Grid" displays the Sample Range, Analysis Run Numbers, Integrated Intensities, Sample Intercepts, R Squares, Life Times, Life Time Errors, and Variances for each analysis run in the Confidence Test.

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## **"KPA Confidence Test Report"**

### **Summary Description**

The "Confidence Test" report displays the coefficient of variation of a series of analyses on the same sample under identical analytical conditions. These results demonstrate the stability of the analytical system.

#### **"Abort"**

The "Abort" command button terminates the Confidence Test. It does not interrupt a KPA analysis in progress but stops (Abort) when the current analysis on the KPA Unit is completed.

#### **"OK"**

The "OK" command button accepts a completed Confidence Test, exits the display and returns to the previous display.

#### **"ReRun"**

The "ReRun" command button repeats the specified Confidence Test.

#### **"Coefficient of Variation"**

The "Coefficient of Variation" displays the results of the Confidence Test. A series of confidence tests that produce monotonic decreasing results may indicate that a "cold" instrument or activated optics has not stabilized. Fluctuating results may indicate an excitation or emission detection problem exists.

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## **"KPA Win Main Display"**

### **Summary Description**

The KPAWin© Main automatically displays after a successful login. From this screen, the user selects various KPAWin© functions using either the Tool Bar or the drop-down menus. The major portion of this display is reserved as the display area for all subsequent KPAWin© displays.

### **"KPAMain Toolbar"**

**Login Menu** - This button displays the Login form to log into the KPAWin© software.

**Logout Menu** - This button logs the current user from the KPAWin© software.

**Exit KPAWin** - This button closes the KPAWin© software Analyze Menu.

**Input Form** - This button displays the Sample Input form.

**Sample Reports** - This button displays the Select Sample Report form, allowing a user to select not only which Samples to report but also the type of report to display or print.

**Configuration Tabs** - This button displays the Configuration Tab form to select an existing configuration or create a new configuration.

**Initialize KPA** - This button displays and executes the Power-On-Confidence tests (POC) and processes and reports the results of these tests on the initialization form.

**Tickler Menu** - This button displays the Tickler Menu to provide a view of the maintenance dates from the Maintenance Configuration Tab.

**Help Menu** - This displays the Help information about the KPAWin© software.

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## **"KPAMain Drop-down / Popup Menu Items"**

### **"Analyze"**

The "Analyze" menu displays a drop-down list: Sample Input, Analyze, Analyze All, Calibrate, and four dispositions for analyzed samples. Of these, only Sample Input can be accessed until a Sample Input form is displayed. The Sample Input form is used to input sample data, select samples to measure, and dispose of results according to Accept, Reject, or Reprocess. Sample dispositions are available from the Analyze drop-down list and a popup menu on the Sample Input form. These functions are available in a drop-down menu and cannot be accessed through the Sample Status window of the Sample Input form.

### **"Sample Input"**

The "Sample Input" item of the Analyze drop-down list displays the Sample Input form that contains the data for sample specification and analysis. The form consists of four portions: the Sample Input button bar, a pop-up command list, the detail of the current sample, and the input for all samples in the analytical batch. The button bar includes Open Initial Sample, Save As Initial Sample, Add New Sample, Cut, Copy, Paste, Clear Selection, Delete Selection, Cancel Input, Exit Input, and Help Input. The pop-up menu list is called with the right mouse button and contains all the items in the Analyze and Edit menu drop-down lists. The current sample detail contains the information for the currently highlighted sample. Data fields can be completed from the user's keyboard or from option lists. The analytical batch input contains all the sample information for the current analytical batch. Data fields are collapsed or restored by drag-and-drop mouse movements to create a customized view of the form stored as a preference of a specific user. The custom view is restored each time the user logs onto KPAWin®.

### **"Analyze"**

The "Analyze" item of the Analyze drop-down list initiates the analysis of samples selected in the first column of the Sample Input form if they are marked "Entered" or "Reprocess". This Item is active when the Sample Input form is Active.

### **"Analyze All"**

The "Analyze All" item of the Analyze drop-down list initiates the analysis of all samples in the Sample Input form if they are marked "Entered" or "Reprocess". This Item is active when the Sample Input form is Active.

### **"Calibrate"**

The "Calibrate" item of the Analyze drop-down list displays the input information to the calibration process and the results of the calibration. The input consists of the header information and the results from the analysis of the background and standards for a calibration.

### **"Database"**

The "Database" menu displays a drop-down list: Open, Repair, Pack, Archive, Customer Administration, Procedure Administration, Units Administration, and Laboratory Administration. Open, Repair, Pack, and Archive are utilities of Microsoft ACCESS that are used to manage the KPAWin® database. The four administration databases document the specifications that are referenced in the KPAWin® database and qualify the data.

### **"Open"**

The "Open" item of the Database drop-down list opens a database that is different from the default. Examples include a Demo database, an alternative operational database, or an Archive database to look at past history.

### **"Repair"**

The "Repair" item on the Database drop-down list attempts a repair of a corrupted database. If this fails, the recourse is to restore from a backup.

### **"Units Administration"**

The "Units Administration" item on the Database drop-down list displays the Units Administration form. The form is used to add, change, or inactivate a unit's entry. The KPAWin® database stores units in ug/l for concentrations and l for volumes. Beyond the unit conversions embedded in KPAWin®, alternative units must be registered with the Units Administration database. After registration, user preferred units can be specified in the Calibration Settings configuration tab for a sample batch and refined in the Sample Input form for an individual sample. Inputted and reported units are converted between

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those of the database and those preferred by the user.

### **"Laboratory Administration"**

The "Laboratory Administration" item on the Database drop-down list displays the Laboratory Administration form. The form adds, changes, or inactivates a laboratory entry.

### **"Pack"**

The "Pack" item on the Database drop-down list reduces the size of the database (i.e. Removes unused space). This normally would be done after an Archive and Delete.

### **"Archive"**

The "Archive" item on the Database drop-down list archives the database data between the selected dates. The KPAWin© database should be backed up before an Archive is done. The Archive copies the sample data and associated records for the selected dates to another or a new database and then deletes the Archived Sample data from the current database. A Pack will be done automatically at this time. When data is Archived, Customers, Procedures, and Configuration Entries are not deleted unless they are marked inactive and are not being used by any records remaining in the database after the archive.

### **"Customer Administration"**

The "Customer Administration" item on the Database drop-down list displays the Customer Administration form. The form adds, changes, or inactivates a customer entry.

### **"Procedure Administration"**

The "Procedure Administration" item on the Database drop-down list displays the Procedure Administration form. The form adds, changes, or inactivates a procedure entry.

### **"Probe KPA Unit"**

The "Probe KPA Unit" on the Tools drop-down list executes the KPA Power-On-Confidence (POC) Initialization tests. These tests are also accessed with real-time status reports from Initialization on the button bar.

### **"Edit"**

The "Edit" menu supports the Sample Input form and provides seven sample-edit options and three data field edit options. The sample options are Add, Insert After, Insert Before, Move Selected, Clear, Delete, and Delete All. The data field edit options are Copy, Cut, and Paste.

### **"Cut"**

The "Cut" option on the Edit drop-down list is active when the Sample Input form is active and data is being entered via the data text boxes. It deletes the selected data field and copies it to the clipboard.

### **"Copy"**

The "Copy" option on the Edit drop-down list is active when the Sample Input form is active. If a text box is active, the selected data field is copied to the clipboard. If grid columns are selected and the grid is active, the Selected Grid column in the first selected row is copied to a KPAWin© array.

### **"Delete"**

The "Delete" option on the Edit drop-down list is active when the Sample Input form is active. It deletes the selected Sample Input records. Analyzed samples are marked as rejected. Samples to be analyzed are removed from the database.

### **"Delete All"**

The "Delete All" option on the Edit drop-down list is active when the Sample Input form is active. This is the same as Delete, except that all records in the current Sample Input are removed.



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## **"Paste"**

The "Paste" option on the Edit drop-down list is active when the Sample Input form is active. If a text box is active, the selected data field is replaced with the clipboard contents. If grid columns are selected and the grid is active, columns from the first selected row are replaced with the KPAWin© array contents. The columns replaced are the ones from which the data was copied.

## **"Add"**

The "Add" option on the Edit drop-down list is active when the Sample Input form is active. The Add command inserts a copy of the currently selected sample record to the end of the Grid.

## **"Insert After"**

The "Insert After" option on the Edit drop-down list is active when the Sample Input form is active. This is the same as the Add command except that the new sample record is placed after the selected record.

## **"Insert Before"**

The "Insert Before" option on the Edit drop-down list is active when the Sample Input form is active. This is the same as the Add command except that the new sample record is placed before the selected record.

## **"Move Selected"**

The "Move Selected" option on the Edit drop-down list is active when the Sample Input form is active. The selected sample records are moved to a new location in the Grid. The user is prompted for the new row number where the selected rows are to begin.

## **"Clear"**

The "Clear" option on the Edit drop-down list is active when the Sample Input form is active. The command resets all data fields for a sample to the default values for the sample type.

## **"File"**

The "File" menu manages the Sample Input form and provides an exit from KPAWin© and printing of KPAWin© reports or figures. Alternative Sample Input forms can be called or saved.

## **"Open"**

The "Open" option on the File drop-down list is active when the Sample Input form is active. This opens the Initial Sample dialog box and allows the user to load previously saved sample input data.

## **"Save As"**

The "Save As" option on the File drop-down list is active when the Sample Input form is active. This opens the Initial Sample dialog box and allows the user to save sample-input data for later loading by the Open command.

## **"Exit"**

The "Exit" option on the File drop-down list closes the KPAWin© program.

## **"Help"**

The "Help menu" displays a drop-down list: Contents, Search, Technical Contacts, and About KPAWin.

## **"Contents"**

The "Contents" option on the Help drop-down list displays the list of topics available in the KPAWin© Help file.

## **"Technical Contacts"**

The "Technical Contacts" option on the Help drop-down list displays the alternative methods that users can use to contact Chemchek Instruments, Inc.

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## **"About KPAWin"**

The "About KPAWin" option on the Help drop-down list displays the About KPAWin© form. The form provides copyright information and further information on Contacts and Technical Information. Contacts is the same as the "Technical Contacts" option with a return to the About KPAWin© form. Technical Information lists the hardware requirements for KPAWin© with a return to the About KPAWin© form through the Contacts screen.

## **"Reports"**

"Reports" provides the Selection item that displays the Select Sample Report form.

## **"Selection"**

"Selection" displays the Select Sample Report form allowing a user to select the samples to report and the type of report to display or print.

## **"Trend"**

"Trend" displays the Trend Report form that graphs the performance of the Laser Plasma Cartridge and Dye Laser.

## **"Security"**

"Security" displays the three Security functions of Login, Logout, and User Administration.

## **"Login"**

"Login" allows a user or a supervisor to Log Into the KPAWin© software.

## **"Logout"**

"Logout" allows a user or supervisor to Log Off the current KPAWin© session.

## **"User Administration"**

"User Administration" displays the User Administration form that allows supervisors to Add, Inactivate, or Change user entries.

## **"Settings"**

The "Settings" menu item displays the General Configuration selection. Once selected, all the Configuration Tabs are available to the user.

## **"Analyze"**

The "Analyze" item on the pop-up menu initiates the analysis of samples selected in the first column of the Sample Input form if marked "Entered" or "Reprocess". This Item is active when the Sample Input form is Active.

## **"Analyze All"**

The "Analyze All" item on the pop-up menu initiates the analysis of all samples in the Sample Input form if marked "Entered" or "Reprocess". This Item is active when the Sample Input form is Active.

## **"Calibrate"**

The "Calibrate" menu item on the pop-up menu initiates the interpretation of the calibration standards and attempts to calibrate both the High and Low ranges. It produces and displays the calibration report when completed.

## **"Accept"**

"Accept", from the pop-up menu, enables the user to mark the measured sample as OK.

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## **"Reject"**

"Reject", from the pop-up menu, enables the user to mark the measured sample as discarded. The sample is not selected when sample data are retrieved for reports.

## **"Reprocess"**

"Reprocess", from the pop-up menu, enables the user to mark a sample for reanalysis. The previous measured data is deleted at this time.

## **"Add"**

The "Add" option on the pop-up menu is active when the Sample Input form is active. The Add command inserts a copy of the currently selected sample record to the end of the grid.

## **"Insert Before"**

The "Insert Before" option on the pop-up menu is active when the Sample Input form is active. This is the same as the Add command except that the new sample record is placed before the selected record.

## **"Insert After"**

The "Insert After" option on the pop-up menu is active when the Sample Input form is active. This is the same as the Add command except that the new sample record is placed after the selected record.

## **"Move Selected"**

The "Move Selected" option on the pop-up menu is active when the Sample Input form is active. The selected sample records are moved to a new location in the grid. The user is prompted for the new row number where the selected rows are to begin.

## **"Clear Sample"**

The "Clear" option on the pop-up menu is active when the Sample Input form is active. The command resets all data fields for a sample to the default values for the sample type.

## **"Delete"**

The "Delete" option on the pop-up menu is active when the Sample Input form is active. It deletes the selected Sample Input records. Analyzed samples are marked as rejected. Samples to be analyzed are removed from the database.

## **"Delete All"**

The "Delete All" option on the pop-up menu is active when the Sample Input form is active. This is the same as Delete, except that all records in the current Sample Input are removed.

## **"Tools"**

The "Tools" menu item displays the available items of AutoChanger (Reset or Rinse), Diagnostics (Probing KPA Unit), and Calculator functions.

## **"Calculator"**

This item invokes the system Calculator from within the KPAWin© software and makes it available to the users.

## **"Window"**

The "Window" menu displays the four Window items available: Tile Horizontal, Tile Vertical, Cascade, and Close All.

## **"Tile Horizontal"**

The "Tile Horizontal" option on the Window menu tiles the open KPAWin© display windows horizontally.

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**"Tile Vertical"**

The "Tile Vertical" option on the Window menu tiles the open KPAWin© display windows vertically.

**"Cascade"**

The "Cascade" option on the Window menu cascades the open windows within the KPAWin© display area.

**"Close All"**

The "Close All" option on the Window menu closes all open windows within the KPAWin© display area except Sample Input.

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## **"Laboratory Administration Menu"**

### **Summary Description**

The Laboratory Administration form has a number of text areas that contain laboratory information: name, address, contact person, telephone numbers, and if applicable, email address. It also contains a checkbox indicating if the above listed laboratory is Active or Inactive. Setting or clearing the checkbox changes the Active/Inactive Status. If the select button is pushed, a list of current Laboratories from the KPAWin© Database is displayed. The user then selects one of these Laboratories and the database information populates the laboratory text areas with the database entries.

### **"Laboratory Dropdown List"**

This "Laboratory" drop-down list displays all Laboratories currently stored in the KPAWin© Database. It displays when the Select button is depressed.

### **"Inactive Laboratory "**

The "Inactive" checkbox identifies a laboratory that is not currently Active.

### **"Add "**

The "Add" button opens a new Laboratory Administration form and positions the cursor in the first entry of the form.

### **"Apply"**

The "Apply" button updates the KPAWin© Database and returns to the Laboratory Administration display.

### **"Cancel"**

The "Cancel" button restores the Laboratory Administration form and the KPAWin© Database to the original values, exits the current display and returns to the previous display.

### **"Clear Entries"**

The "Clear" button clears all existing data from the Laboratory Administration screen and returns to the current display.

### **"OK"**

The "OK" button updates any record in the Laboratory Administration form and the KPAWin© Database, exits the current display and returns to the previous display.

### **"Select"**

The "Select" button queries the KPAWin© Database and displays a drop-down list of all active Laboratories in the Database. The user then selects a laboratory entry to edit.

### **"Laboratory Administration List"**

The "Laboratory Administration" list manages information about the servicing laboratory or laboratories. Laboratory accounts are created, edited, marked as active or inactive, and entered into the KPAWin© database. Each account contains the following descriptors: Id, name, address, Fax number, e-mail address, laboratory contact, and contact telephone number.

### **"Laboratory Contact Person Information"**

The "Laboratory Contact Person Information" identifies the laboratory's contact person(s) and how to communicate with them.

### **"Laboratory ID"**

The "Laboratory Id" identifies the laboratory that performs the sample analysis. This field must be completed for all samples.

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**"Laboratory Fax"**

The "Laboratory's Fax Number" identifies the telephone number that the laboratory uses to send and receive facsimiles. Fields can be delimited according to modem standards for direct dial capability.

**"Laboratory Email Address"**

The "Laboratory Email Address" is the internet account of the laboratory.

**"Laboratory Name"**

The "Laboratory Name" is the registered name of the laboratory.

**"Laboratory Address"**

The "Laboratory Address" is normally the street address that is used to accept freight.

**"Laboratory City"**

The "Laboratory City" is the city where the laboratory is located.

**"Laboratory State"**

The "Laboratory State" is the state where the laboratory is located.

**"Laboratory Zip Code"**

The "Zip Code" is the Zip code where the laboratory is located.

**"Laboratory Contact First Name"**

The "Laboratory Contact First Name" is the first name of the laboratory contact.

**"Laboratory Contact Last Name"**

The "Laboratory Contact Last Name" is the last name of the laboratory contact.

**"Laboratory Contact Telephone Number"**

The "Laboratory Contact Telephone Number" identifies the telephone number of the Laboratory's contact person(s).



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## **"KPAWin© Login Screen"**

### **Summary Description**

The Login form is displayed whenever anyone attempts to login to the KPAWin© software. It solicits the User Id and Password during the login sequence for all users attempting to gain access to the KPAWin© system. Customer sites have the option of using either or both of these security measures. If no User Id or Passwords are entered into the system by a system supervisor, anyone can start the KPAWin© software and browse the contents of the KPA database to view previously analyzed samples and analyze new samples.

The screen contains two text entries, the User Id and the Password. When a user requires the use of the KPAWin© software, the system supervisor must create an entry (using User Administration) by entering a new User Id and optionally, a password for the new user. Each time this or any user attempts to gain access to the system, they are required to enter these data during the login sequence. The use of passwords is optional.

The form requires one of two possible entries. All user information is stored in the KPAWin© database. If user sites elect to use this security option, supervisors enter a User Id and Password for each new user to the system.

The selection box contains all current active User Ids. Anyone attempting to access the software can list these valid users and select the appropriate entry. If the customer site elects to also use passwords, every user is required to enter the correct password for the user name specified.

### **"User Dropdown List"**

The "User" drop-down list contains all current, valid User Ids. To logon, a user must select his/her User Id. During a successful login, KPAWin© restores system defaults to those selected by the specific user during his/her last session.

### **"Cancel"**

The "Cancel" button terminates the login sequence, exits the display and returns to the main Idle display.

### **"OK"**

The "OK" button initiates a verification of the Login entries. If the entries are valid, the login is accepted, default values for KPAWin© are set to those specified by the user during the user's last KPAWin© session, the login display is exited and the main KPAWin© display is opened. If the entries are invalid, the user is advised and the login display is retained.

### **"Passwords"**

The "Password" is a site-selected option that is entered at Login. Users are required to enter the correct, user-specific password during each login sequence.

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## **"Procedure Administration"**

### **Summary Description**

The "Procedure Administration" form has a number of text areas that contain procedure information, Procedure Id, Title, Revision, and Protocol. It also has a checkbox to indicate if the above listed procedure is Active or Inactive. Setting or clearing the checkbox changes the Active/Inactive Status. In addition, if the select button is pushed, a list of current procedures from the KPAWin© Database is displayed. The user then selects one of these procedures and the database information populates the text areas with the database entries.

### **"Procedure List"**

The "Procedure List" drop-down list displays all Procedures currently entered in the KPAWin© Database. It displays whenever the Select button is depressed.

### **"Add"**

The "Add" button opens a new Procedure form and positions the cursor at the first entry of the form.

### **"Apply"**

The "Apply" button updates the KPAWin© Database and returns to the Procedure Administration display.

### **"Cancel"**

The "Cancel" button restores the Procedure Administration form and the KPAWin© Database to the original values, exits the current display and returns to the previous display.

### **"Clear Entries"**

The "Clear" button clears all existing data from the Procedure Administration form and returns to the current display.

### **"OK"**

The "OK" button updates any record in the Procedure Administration form and the KPAWin© Database, exits the current display and returns to the previous display.

### **"Select"**

The "Select" button queries the KPAWin© Database and displays a drop-down list of all active Procedures in the Database. The user then selects one Procedure to edit.

### **"Procedure ID"**

The "Procedure Id" uniquely identifies the referenced laboratory procedure for the preparation and/or analysis of samples.

### **"Procedure Revision"**

The "Procedure Revision" uniquely identifies the referenced laboratory procedure revision for the preparation and/or analysis of samples.

### **"Procedure Title"**

The "Procedure Title" uniquely identifies the referenced laboratory procedure title for the preparation and/or analysis of samples.

### **"Procedure Protocol"**

The "Procedure Protocol" uniquely identifies the referenced laboratory procedure protocol for the preparation and/or analysis of samples.

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## **"Sample Input Form"**

### **Summary Description**

The "Sample Input" form accepts sample information required to analyze and evaluate sample results. The form contains a Toolbar, an input area (Text and Drop-down Text areas), and a Display Grid at the bottom of the form. Sample Input data in the input area is mirrored in the display grid sample identified by an asterisk (\*) in the first column, the Sample Analysis Number. Data is entered into either location and is automatically duplicated in the other area. Drop-down lists in the input area provide convenient data entry with assurance that formats are consistent and data-handling errors are reduced. The user customizes the Display Grid to include or hide specific fields in the display. This current user preference is maintained by KPAWin© and reloaded whenever the user logs onto the system.

### **"Preferred Range List"**

The "Range Drop-down List" is a drop-down box containing four selections: Auto, Both, Low, and High. The selected range is the first range attempted for an analysis. The selection overrides any alternative preferred range specified in the current Calibration Configuration Tab. The Auto option selects the optimal range based on sample precounts in the high range and defaults to the range for the last analysis if both ranges are qualified. Precounts in the high range are scaled to anticipate counts in the low range by the Auto Efficiency Factor in the KPA Unit Settings Configuration Tab. The Both option results in an analysis in the most recently used range. This is followed by an analysis in the other range if the first analysis fails the high-intensity tolerance or the low-intensity tolerance specified in the KPA Unit Settings Configuration Tab. Selection of either the High or Low option limits analysis to the requested range with results qualified by the high-intensity tolerance or the low-intensity tolerance. Intensity tolerance factors for both the high and low ranges are specified in the KPA Unit Settings Configuration Tab.

### **"Concentration Units List"**

The "Units" drop-down list provides options for sample units. Selection of units on a per mass basis requires the specification of the Specific Gravity. Selection of radiometric units requires the specification of the Specific Activity. Selection of units in molarity requires the specification of the atomic mass of the analyte. When Specific Gravity or Specific Activity are required at the time of sample input, the appropriate label(s) will be rendered in red. Selection of concentration units on a per volume basis require the use of amounts in ml units. Selection of concentration units on a per mass basis require the use of amounts in g units.

### **"Alarms/Warnings Drop-down List"**

The "Alarms/Warnings" drop-down list provides all alternative active configurations of Alarms and Warnings.

### **"Calibration Configuration Drop-down List"**

The "Calibration" drop-down list provides all alternative active configurations of Calibration.

### **"KPA Unit Settings Drop-down List"**

The "KPA Unit Settings" drop-down list provides all alternative active configurations of KPA Unit Settings.

### **"Performance Limits Drop-down List"**

The "Performance Limits" drop-down list provides all alternative active configurations of Performance Limits.

### **"QC Limits Drop-down List"**

The "QC Limits" drop-down list provides all alternative active configurations of QC Limits.

### **"Sample Status Drop-down List"**

The "Sample Status" drop-down list includes Entered, Processed, Accepted, Rejected and Reanalyze. The Entered option is automatically listed when Sample Input data is entered and before the sample is analyzed. Processed is also entered automatically at the completion of sample analysis. Accepted marks a processed sample as acceptable to the user. Rejected marks a processed sample as unacceptable to the user. Reanalyze marks a processed sample as requiring reanalysis. Samples identified as Entered or Reanalyze can be analyzed. Samples identified as Processed or Accepted can be retrieved for sample reporting. Rejected samples are retained in the database but cannot be retrieved by KPAWin©.

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### **"Sample Type Drop-down List"**

The "Sample Type" drop-down list contains the 19 unique sample types that are analyzed, interpreted, and reported by KPAWin®. The Help menu is consulted for details on individual Sample Types. Selection of a specific sample type from the drop-down list causes data-field labels to reset to Red for required, Blue for optional, Grey for defaults that cannot be edited, and Black for not appropriate for the sample type. Select the Sample Type for the sample from the drop-down list. It is automatically completed if a sample selection is highlighted on the grid.

### **"Procedure ID Drop-down List"**

The "Procedure Id" drop-down list contains all active laboratory procedures. Select the Procedure Id for the sample from the drop-down list. It is automatically completed if a sample selection is highlighted on the grid.

### **"Customer ID Drop-down List"**

The "Customer Id" drop-down list contains all active laboratory customers. Select the Customer Id for the sample from the drop-down list. It is automatically completed if a sample selection is highlighted on the grid.

### **"Basis ID Drop-down List"**

The "Basis Id" drop-down list includes all of the samples that can be related to quality control samples that they qualify. Select the Basis Id for the sample from the drop-down list. It is automatically completed if a sample selection is highlighted on the grid.

### **"PreRinse Option"**

The "Pre-Rinse Option" checkbox, if selected, rinses the flow cell before the sample is charged to the cell. The Pre-Rinse event is only available with the Gilson sample changer. It is automatically completed if a sample selection is highlighted on the grid.

### **"PostRinse Option"**

The "Post-Rinse Option" checkbox, if selected, rinses the flow cell after the sample is charged to the cell and analyzed. The Post-Rinse event is only available with the Gilson sample changer. It is automatically completed if a sample selection is highlighted on the grid.

### **"Sample Input Grid"**

The "Sample Input Grid" is located at the bottom of the Sample Input form. The display grid contains all information for each sample and is customized by the user by selecting or hiding specific fields. The current configuration of the display grid is saved by User Id and is reinstalled whenever that particular user Logs onto the KPAWin® software.

### **"Sample Input Toolbar"**

**Open Initial Sample** - This toolbar button displays a dialog box containing previously saved Initial samples and allows the user to load one of them.

**Save As Initial Sample** - This toolbar button saves samples from the Sample Input data. These Initial samples can then be reloaded as Sample Input Data.

**Add New Sample** - This toolbar button adds a new Sample Input to the grid.

**Clear Selection** - This toolbar button clears all entries from the selected samples or contiguous multiple samples.

**Delete Selection** - This toolbar button deletes the selected sample or contiguous multiple samples.

**Analyze Selection** - This toolbar button analyzes the selected sample or contiguous multiple samples.

**Exit** - This toolbar button exits the Sample Input screen.

**Help** - This toolbar button displays the Help information.

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**"Sample Analysis Number"**

The "Sample Analysis Number" is a KPA index that sequences samples in an analytical batch. It is automatically incremented as samples are entered into the grid. An asterisk (\*) in the field indicates the sample is the current sample and is the target for all changes and requested actions.

**"Initial Preparation (Prediluted) Volume"**

The "Initial Preparation (Prediluted) Volume" for a sample is the amount of authentic sample that is taken for sample preparation. Spikes that are to be subjected to sample preparation should be added to this volume and prior to its dilution to the Final Preparation volume. Units of ml or g are required consistent with the basis of the concentration units as per volume or per mass. It is automatically completed if a sample selection is highlighted on the grid.

**"Final Preparation (Diluted) Volume"**

The "Final Preparation (Diluted) Volume" for a sample is the final, total amount of sample at the end of sample preparation. Units of ml or g are required consistent with the basis of the concentration units as per volume or per mass. It is automatically completed if a sample selection is highlighted on the grid.

**"Initial Analysis (Sample) Volume"**

The "Initial Analysis (Sample) Volume" for a sample is the prediluted amount of prepared sample that is taken for dilution to the analytical sample. Units of ml or g are required consistent with the basis of the concentration units as per volume or per mass. It is automatically completed if a sample selection is highlighted on the grid.

**"Final Analysis Volume"**

The "Final Analysis Volume" for a sample is the amount of diluted, prepared sample that is taken for sample analysis. Spikes that are added after sample preparation should be added before this final dilution. Units of ml or g are required consistent with the basis of the concentration units as per volume or per mass. It is automatically completed if a sample selection is highlighted on the grid. The numerical amount should be identical to the final analysis amount for calibration samples, check samples, and basis samples.

**"Standard ID"**

The "Standard Id" for a sample uniquely relates the standard to its source through the laboratory's standards control program. It is automatically completed if a sample selection is highlighted on the grid.

**"Standard Concentration"**

The "Standard Concentration" units of a sample must be consistent with those specified by the user in the Sample Input for the standard. It is automatically completed if a sample selection is highlighted on the grid.

**"Standard Volume"**

The "Standard Volume" specifies the amount of a standard added to a sample. Amount units can be ml or g but must be consistent with the volume or mass basis specified by the user in the Sample Input for the standard. For example, concentrations in molarity require "Standard Volume" units in ml. Concentrations in pCi/g require "Standard Volume" units in grams. It is automatically completed if a sample selection is highlighted on the grid.

**"Specific Gravity"**

The "Specific Gravity" of a sample is only required for preferred units on a mass basis. It is automatically completed if a sample selection is highlighted on the grid.

**"Sample ID"**

The "Sample Id" for a sample is user-defined and establishes administrative laboratory control of the sample. It is automatically completed if a sample selection is highlighted on the grid.

**"Specific Activity"**

The "Specific Activity" of a sample is only required for preferred units in radiometric units. It is automatically completed if a sample selection is highlighted on the grid.

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**"Atomic Mass"**

The "Atomic Mass" for the analyte in a sample is only required for preferred units specified in molarity. It is automatically completed if a sample selection is highlighted on the grid.

**"Rack Number"**

The "Rack Number" for a sample is only required with the Gilson sample changer. It is automatically completed if a sample selection is highlighted on the grid.

**"Vial Position"**

The "Vial Position" for a sample is only required with the Gilson sample changer. It is automatically completed if a sample selection is highlighted on the grid.

**"Sample Description"**

The "Sample Description" of a sample is a brief label that identifies the purpose or source of the sample. It is automatically completed if a sample selection is highlighted on the grid.



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## **"Select Archive Data"**

### **Summary Description**

The "Archive" function appends the selected portion of the KPAWin© Database to the Archive file of KPAWin©.

#### **"Archive"**

The "Archive" button appends the selected portion of the KPAWin© Database to the Archive file of KPAWin©.

#### **"Cancel"**

The "Cancel" button restores the KPAWin Archive form and the KPAWin© Database to the original values, exits the current display and returns to the previous display.

#### **"Between"**

The "Between" option allows the user to Archive selected data from the KPAWin© Database that fall within the two dates (Beginning date and Ending date) provided.

#### **"During the Previous"**

The "During the Previous" option allows the user to Archive selected data from the KPAWin© Database that fall within the Previous number of days selected by the user.

#### **"All"**

The "All" option allows the user to Archive ALL data from the KPAWin© Database.

#### **"Select Spin Wheel"**

The "Select" spin wheel allows the user to Select the number of days to collect data to archive.

#### **"Beginning Date"**

The "Beginning Date" field accepts the Beginning date for the Archive Command to collect data from the KPAWin© Database.

#### **"Ending Date"**

The "Ending Date" field accepts the Ending date for the Archive Command to collect data from the KPAWin© Database.

#### **"Number of Days"**

The "Number of Days" text area accepts the number of days to collect data from the KPAWin© Database to Archive.

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## **"KPA Sample Types"**

### **Summary Description**

The KPAWin® software recognizes 19 unique sample types. Collectively they provide the analyst with a system to accumulate and evaluate interactively all of the luminescence-specific and general-laboratory evaluations associated with sample analysis and analytical performance. Sample Input is designed to guide the analyst by identifying the minimum input data set for each unique sample type. Required data fields are labeled in red and on-screen warnings are displayed when critical information is not provided. The display and reporting for each Sample Type is designed to minimize secondary data handling by the analyst.

#### **1. Background - "BckGnd"**

"BckGnd" is the designator for the background sample. Raw intensity vectors are corrected for coincidence and smoothed. The smoothed background intensity vector is used to correct the intensity vectors of most sample types for background contributions. Only Standard Addition Samples and Standard Addition Spikes, Confidence Tests, and Quench-Corrected Sample vectors are not corrected for background. The intensity intercept for the reference that is recorded during the analysis of the background sample is defined as the "Reference Mark" ( $R_M$ ). For all succeeding samples in the analytical batch of the background, a reference ratio is computed from the intercept intensity of the reference ( $R_I$ ) and  $R_M$  as the ratio  $R_I/R_M$ . Unless the option "Reference Corrected Data" is deselected on the Calibration Configuration Tab, accumulated intensity in the smoothed intensity vector of the sample will be corrected for reference drift by division by the reference ratio.

#### **2. Calibration Standard - "CalStd "**

"CalStd" is the designator for a calibration standard. Data are entered by the analyst and reported by KPAWin® to indicate the laboratory control identifier for specific standards as well as their concentrations and the volumes in ml or masses in g. Concentrations are based on the laboratory's preference indicated in the Calibration Configuration Tab which can be amended in the Sample Input grid. The definition of amount on a /volume or /mass basis is consistent with the selected concentration units.

#### **3. Continuing Check Blank- "CChkBik"**

"CChkBik" is a deionized water or analyte-free laboratory blank and is the designator for the continuing check blank. The blank is used to verify the calibration subsequent to the initial verification. The acceptable performance of the blank is specified in the Quality Control Limits Configuration Tab as a multiple of the instrument detection limit (IDL).

#### **4. Continuing Check Standard - "CChkStd "**

"CChkStd" is an analyte standard in deionized water or analyte-free laboratory sample and is the designator for the continuing check standard. The standard is used to verify the calibration subsequent to the initial verification and should be prepared from a traceable standard that is independent of the calibration standards. The acceptable performance of the blank is specified in the Quality Control Limits Configuration Tab as a standard recovery error.

#### **5. Confidence Tests - "ConfTst"**

"ConfTst" is the designator for a confidence test. The test provides the coefficient of variation of a series of analyses on the same sample under identical analytical conditions. These results demonstrate the stability of the analytical system.

#### **6. Initial Check Blank - "IChkBik"**

"IChkBik" is a deionized water or analyte-free laboratory blank and is the designator for the initial check blank. The blank is used to verify the calibration immediately following the calibration. The acceptable performance of the blank is specified in the Quality Control Limits Configuration Tab as a multiple of the instrument detection limit (IDL).

#### **7. Initial Check Standard - "IChkStd"**

"IChkStd" is an analyte standard in deionized water or analyte-free laboratory sample and is the designator for the initial check standard. The standard is used to verify the calibration immediately following the calibration and should be prepared from a traceable standard that is independent of the calibration standards. The acceptable performance of the blank is specified in the Quality Control Limits Configuration Tab as a standard recovery error.

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**8. Instrument Detection Limit - "IDL"**

"IDL" is the designator for the instrument detection limit sample that consists of an analyte standard in deionized water or an analyte-free laboratory blank. The Instrument Detection Limit is an estimate of the minimum sample concentration that can be detected with a 99% confidence. The calculation is based on a Student's t-test with all input parameters selected in the detection limit frame of the Quality Control Limits Configuration Tab. The parameters include number of standards, detection limit type, maximum duration in weeks, maximum concentration factor, and limit in  $\mu\text{g/l}$ . Detection limit type is a KPAWin® term to distinguish between detection limits based on the standard deviation of a series of individual analyses (Type 1) and the average of a series of standard deviation of a series of analyses made in triplicate (Type 2).

In operation, an IDL is analyzed according to a frequency established by laboratory protocol. KPAWin® pools all IDL results and estimates the detection limit for every day of KPA operation based on the measurements that were made within the previous number of weeks specified in the maximum duration specification. This detection limit is reported with the data recorded for the day of operation and is used to evaluate the performance of IChkBlk and CChkBlk samples. Errors are reported if the number of standards (or standard deviations for Type 2 daily standards) is insufficient, if the true average concentration of the standards (makeup concentrations) is greater than the specified multiple of the detection limit, or if the value of the detection limit exceeds the user-defined limit.

**9. Method Detection Limit - "MDL"**

"MDL" is the designator for the method detection limit sample consisting of an analyte standard in deionized water or an analyte-free laboratory blank processed by the procedure employed for samples. The Method Detection Limit is an estimate of the minimum sample concentration that can be detected with a 99% confidence. The calculation is based on a Student's t-test with all input parameters selected in the detection limit frame of the Quality Control Limits Configuration Tab. The parameters include number of standards, detection limit type, maximum duration in weeks, maximum concentration factor, and limit in  $\mu\text{g/l}$ . Detection limit type is a KPAWin® term to distinguish between detection limits based on the standard deviation of a series of individual analyses (Type 1) and the average of a series of standard deviations of a series of analyses made in triplicate (Type 2).

In operation, an MDL is analyzed according to a frequency established by laboratory protocol. KPAWin® pools all MDL results and estimates the detection limit for every day of KPA operation based on the measurements that have been made within the previous number of weeks specified in the maximum duration specification. This detection limit is reported with the data recorded for the day of operation and is used to evaluate the performance of the current MtdBlk sample. Errors are reported if the number of standards (or standard deviations for Type 2 daily standards) is insufficient, if the true average concentration of the standards (makeup concentrations) is greater than the specified multiple of the detection limit, or if the value of the detection limit exceeds the user-defined limit.

**10. Sample Duplicate - "SampDup"**

"SampDup" is the designator of a duplicate sample. The sample is prepared for analysis and analyzed precisely analogous to the basis sample. Acceptable performance is specified in the Sample Control frame of the Quality Control Configuration tab as the relative percent deviation (%RPD).

**11. Sample Spike - "SampSpk"**

"SampSpk" is the designator of a spiked sample. The sample is prepared for analysis and analyzed precisely analogous to the basis sample. The volume of the spike addition replaces a portion of the deionized water used to prepare the final analytical sample. Acceptable performance is specified in the Sample Control frame of the Quality Control Configuration tab as the spike recovery.

**12. Quenched Sample - "QchSamp"**

"QchSamp" is the designator of a quenched sample. The sample is an aliquot of the basis sample that was prepared for analysis and diluted and analyzed precisely analogous to the basis sample. A volume of quenchant, typically 0.1 ml of methanol, replaces a portion of the deionized water used to prepare the final analytical sample. The methanol provides a non-spectroscopic, thermal pathway for relaxation of the excited uranium complex and the resulting intensity vector reports the non-uranium component present in the basis sample. The smoothed intensity vector of the quenched sample replaces the smoothed background vector in the estimate of the net intensity vector. A user-request for a report of the basis sample alone provides the standard report with sample intensities corrected for the batch background. Alternatively, a user-request for a quenched sample report provides a report with sample intensities corrected for the quenched intensities.

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**13. Sample - "Sample"**

"Sample" is the designator of a matrix sample. The Sample Id of this unknown can be the Basis Id for other sample types that are evaluated and reported with this sample by KPAWin®. The sample types that can be so linked with this sample type are SampDup, SampSpk, QchSamp, SerDil, and MtdSA.

**14. Method Blank - "MtdBlk"**

"MtdBlk" is the designator for the method blank that consists of deionized water or analyte-free laboratory matrix that has been processed by the procedure employed for samples. The acceptable performance of the blank is specified in the Method Control frame of the Quality Control Limits Configuration Tab. It is expressed as a multiple of the method detection limit (MDL), the percentage of the contract reporting detection limit (CRDL), the percentage of the sample concentration, or the largest of the aforementioned three conditions.

**15. Method Standard Addition - "MtdSA"**

"MtdSA" is the designator of analyte-spiked samples quantitating the analyte concentration in the basis sample and the interferences to that quantitation. Spiked samples are prepared for analysis and analyzed precisely analogous to the basis sample. The volume of the spike addition replaces a portion of the deionized water used to prepare the final analytical sample. The parameters that control the interpretation of MtdSA data are contained in the Sample Control frame of the Quality Control Limits Configuration Tab under the Method of Standard Additions. These parameters are Number of Spikes, Recovery Error (%), Slope Error (%), and  $R^2$  (correlation coefficient). The value for Number of Spikes can be either one (1) or three (3).

A single MtdSA sample provides quantitation and Recovery Error (%) from the external batch calibration. Triplicate MtdSA samples with the basis sample provides an internal calibration by a linear least-squares regression of the spike concentrations versus the increment of the intensity intercept that is attributed to the spike (Intercept of a spiked sample minus intercept of the basis sample). The negative value of the concentration intercept is the reported concentration of the analysis.  $R^2$  is the correlation coefficient of the internal calibration least-squares regression. Recovery Error (%) is the difference between the analyte concentrations for the basis sample for the internal-MtdSA calibration and the external calibration divided by the result from the internal calibration when expressed as a percent. Slope Error (%) is the difference between slopes for the internal-MtdSA calibration and the external calibration divided by the slope from the internal calibration when expressed as a percent.

**16. Method Standard - "MtdStd"**

"MtdStd" is the designator for the method standard that consist of an analyte standard in deionized water or analyte-free laboratory sample that has been processed by the procedure employed for samples. The acceptable performance of the standard is specified in the Method Control frame of the Quality Control Limits Configuration Tab as a standard recovery error.

**17. Standard Addition Sample - "SASamp"**

"SASamp" is the designator for the sample analyzed by standard addition. Standard addition quantitates the analyte in a basis sample by internal calibration. The process compares the incremental increase of intercept intensity from this basis sample to a spiked sample to the incremental analyte concentration of the spiked sample. The quantum efficiency factor is used to estimate the analyte concentration in the basis sample. Neither the basis sample nor its spike requires a background or an external batch calibration.

**18. Standard Addition Spike - "SASpk"**

"SASpk" is the designator for the analyte-spiked sample analyzed by standard addition. Standard addition quantitates the analyte in a basis sample by internal calibration. The process compares the incremental increase of intercept intensity from this basis sample to a spiked sample to the incremental analyte concentration of the spiked sample. The quantum efficiency factor is used to estimate the analyte concentration in the basis sample. Neither the basis sample nor its spike requires a background or an external batch calibration.

**19. Serial Dilution - "SerDil"**

"SerDil" is the designator of a serial dilution of a sample. The sample is prepared for analysis and analyzed precisely analogous to the basis sample. Additional deionized water prepares the final analytical sample so that a five-fold dilution of the basis sample is created. Acceptable performance is specified in the Sample Control frame of the Quality Control Configuration tab as the serial dilution error.

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## **"Select Export Fields"**

### **Summary Description**

The Select Export Fields Menu displays the Sample fields available for Export to a file. The use of the selection buttons is explained at the top of the form.

### **"Data Delimiter"**

The "Data Delimiter" dropdown list allows the user to select the delimiter used when exporting the sample data to the file selected by the user.

### **"Export Format"**

The "Export Format" dropdown list displays the Export format(s) available. Currently, KPAWin© only supports a "TXT" file format. Additional formats may be available with future releases of the software.

### **"Heading Information"**

The "Heading Information" checkbox allows the user to select whether Heading Information is included with the Sample Information exported to the user named file.

### **"Sample Intensities - Sample"**

The "Sample Intensities Sample" checkbox allows the user to select the Sample Intensities to be Exported.

### **"Sample Intensities - Reference"**

The "Sample Intensities Reference" checkbox allows the user to select the Sample Reference Intensities to be Exported.

### **"Apply"**

The "Apply" command button applies the selected fields, updating the database to reflect these selections. This button **MUST** be used to apply the selections. The selected fields are used to export the sample information the next time the Export button is used.

### **"Cancel"**

The "Cancel" command button cancels the Export Fields selection process and returns to the Select Report Sample screen without affecting any changes.

### **"Export Data"**

The "Export Data" command button exports the selected fields to the file selected by the user and returns to the Select Report Sample screen.

### **"Include All"**

The "Include All" button selects All available report fields from the Available Report Fields listbox and moves them to the Included Report Fields listbox.

### **"Add Field(s)"**

The "Add Field(s)" button moves all selected (highlighted) report fields from the Available Report Fields listbox to the Included Report Fields listbox.

### **"Rem Field(s)"**

The "Rem Field(s)" button moves all selected (highlighted) report fields from the Included Report Fields listbox to the Available Report Fields listbox.

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### **"Remove All"**

The "Remove All" button selects All available report fields from the Included Report Fields listbox and moves them to the Available Report Fields listbox.

### **"Rename"**

The "Rename" button allows the user to Rename the selected field to a name of their choice.

### **"Heading Information"**

The "Heading Information" frame allows the user to select whether Heading Information is included with the Sample Information exported to the user named file.

### **"Intensity Selection"**

The "Intensity Selection" frame contains the six intensity options available for Export.

### **"Intensities Data"**

The "Sample Intensities" frame allows the user to select the intensities to be Exported. Select either the Sample or the Reference or Both.

### **"Available Report Fields"**

The "Available Report Fields" listbox displays all available fields that can be selected. The selected fields, after the calculations are performed, contain sample information to Export to a file selected by the user. These fields are selected (moved to the "Included Report Fields" listbox) and then rearranged into any sequence that the user desires. When a field is selected, it is deleted from the Available Fields list and inserted into the Included Fields box. Individual fields are highlighted and moved with the "Add Field(s)" command or removed using the "Rem Field(s)" command or by Double Clicking on the field.

### **"Included Report Fields"**

The "Included Report Fields" listbox displays all selected fields. The selected fields, after the calculations are performed, contain sample information to Export to a file selected by the user. These fields are selected (moved to the "Included Report Fields" listbox) and then rearranged into any sequence that the user desires. When a field is removed, it is deleted from the Included Fields list and inserted into the Available Fields box. Individual fields are highlighted and included with the "Add Field(s)" command or removed using the "Rem Field(s)" command or by Double Clicking on the field.

### **"Do Not Include Intensities"**

The "Do Not Include Intensities" option removes the possibility of exporting any Intensity data regardless of other Intensity settings.

### **"Measured Intensities"**

The "Measured Intensities" Option causes the selected Measured or Raw intensities to be exported to the named file.

### **"Coincidence Corrected"**

The "Coincidence Corrected" Option causes the selected Coincidence Corrected or Deadtime intensities to be exported to the named file.

### **"Smoothed"**

The "Smoothed" Option causes the selected Smoothed intensities to be exported to the named file.

### **"Referenced Corrected"**

The "Referenced Corrected" Option causes the selected Referenced Corrected intensities to be exported to the named file.



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### **"Net Sample"**

The "Net Sample" Option causes the selected Net Sample or final result intensities to be exported to the named file.

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## **Select Report Fields**

### **Summary Description**

The Select Report Fields Menu displays the fields available to be displayed in the grid in the lower portion of the form. The use of the selection buttons is explained on the form.

#### **"Apply"**

The "Apply" command button applies the selected fields, updating the database to reflect these selections. This button **MUST** be used to apply the selections. The selected fields are used to display the sample information the next time that the Retrieve button is used.

#### **"Cancel"**

The "Cancel" command button cancels the Report Fields selection process and returns to the Select Report Sample screen without affecting any changes.

#### **"Exit"**

The "Exit" button returns the user to the Select Report Sample screen.

#### **"Include All"**

The "Include All" button selects All available report fields from the Available Report Fields listbox and moves them to the Included Report Fields listbox.

#### **"Add Field(s)"**

The "Add Field(s)" button moves all selected (highlighted) report fields from the Available Report Fields listbox to the Included Report Fields listbox.

#### **"Rem Field(s)"**

The "Rem Field(s)" button moves all selected (highlighted) report fields from the Included Report Fields listbox to the Available Report Fields listbox.

#### **"Remove All"**

The "Remove All" button selects All available report fields from the Included Report Fields listbox and moves them to the Available Report Fields listbox.

#### **"Available Report Fields"**

The "Available Report Fields" listbox displays all available fields that can be selected. These fields display the retrieved sample information from the KPAWin® database in the Grid on the Select Report Sample form. These fields are selected (moved to the "Included Report Fields" listbox) and then rearranged into any sequence the user desires. When a field is selected, it is deleted from the Available Fields list and inserted into the Included Fields box. Individual fields are highlighted and included with the "Add Field(s)" command or removed using the "Rem Field(s)" command or by Double Clicking on the field.

#### **"Included Report Fields"**

The "Included Report Fields" listbox displays all selected fields. These fields display the retrieved sample information from the KPAWin® database in the Grid on the Select Report Sample form. These fields are selected (moved to the "Included Report Fields" listbox) and then rearranged into any sequence the user desires. When a field is removed, it is deleted from the Included Fields list and inserted into the Available Fields box. Individual fields are highlighted and included with the "Add Field(s)" command or removed using the "Rem Field(s)" command or by Double Clicking on the field.

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## **"Select Report Sample(s)"**

### **Summary Description**

The "Select Report Sample" form allows a KPAWin© user to select a variety of displays and print options. Report selections are based inclusively or exclusively on dates, sample types, customers, procedures, KPA analysts, sort order of the displays, and the type of data to be displayed and/or printed. The display grid is customized to include or hide particular fields and the order the fields appear in the display. This current updated display grid is saved by User ID and reloaded whenever that user Logs onto the KPAWin© software.

### **"Sort Order List"**

The "Sort Order" specifies the alphabetical order the fields are viewed and listed in the Select Fields tabulation. The user specifies either Ascending or Descending order. The default is Ascending order.

### **"Customer Drop-down List"**

The "Customer" drop-down lists all unique customers associated with samples in the KPAWin© database.

### **"Analyst Drop-down List"**

The "Analyst" drop-down lists all unique analysts associated with samples in the KPAWin© database.

### **"Procedure Drop-down List"**

The "Procedure" drop-down lists all unique procedures associated with samples in the KPAWin© database.

### **"Sample Type Drop-down List"**

The "Sample Type" drop-down lists all unique sample types associated with samples in the KPAWin© database.

### **"Calibration Details"**

The "Calibration Details" checkbox on the MultiReport Option prints the Calibration Details for the selected samples.

### **"Configuration Details"**

The "Configuration Details" checkbox on the MultiReport Option prints all Configuration Details for the Selected samples.

### **"Multiple Sample Report"**

The "Multiple Sample Report" checkbox selects multiple sample displays or print files.

### **"Sample Identification"**

The "Sample Identification" checkbox on the MultiReport Option prints the Sample Input Details for the selected samples.

### **"Sample Preparation"**

The "Sample Preparation" checkbox on the MultiReport Option prints the Sample Input Details for the selected samples.

### **"Analytical Results"**

The "Analytical Results" checkbox on the MultiReport option prints the Sample Results Details for the selected samples.

### **"Quality Control"**

The "Quality Control" checkbox on the MultiReport option prints the QC information for the selected samples.

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## **"Calibration"**

The "Calibration" checkbox on the MultiReport option prints the Calibration Information for the selected samples. It also displays and enables the Calibration Details checkbox.

## **"Configuration"**

The "Configuration" checkbox on the MultiReport option prints all Configuration Ids for the selected samples. It also displays and enables the Configuration Details checkbox.

## **"Retrieve"**

The "Retrieve" button displays a list of the selected samples stored in the KPAWin© database. Users must first make their option choices, then click Retrieve to acquire the selected samples from the KPAWin© Database prior to either displaying results on the monitor or printing reports.

## **"Display / Print"**

The "Display" button serves two functions. If the Display Reports radio button on the Report Options frame is selected, and after the selected samples are retrieved from the database, the Display button produces the various selected display options on the computer monitor. However, if the Print Reports radio button is selected on the Report Options frame, this command button changes to read "Print" and clicking it causes the selected samples to be routed to the system printer.

## **"Report Options"**

This frame provides the option of selecting a radio button to Display Reports or Print Reports. The default is Display Reports.

## **"Customer"**

The "Customer" frame provides the option of selecting All, And, Or logic to select samples by customer. As an alternative, a customer can be selected from the drop-down list.

## **"Analyst"**

The "Analyst" frame provides the option of selecting All, And, Or logic to select samples by analyst. As an alternative, an analyst can be selected from the drop-down list.

## **"Procedure"**

The "Procedure" frame provides the option of selecting All, And, Or logic to select samples by procedure. As an alternative, a procedure can be selected from a drop-down list.

## **"Sample Type"**

The "Sample Type" frame provides the option of selecting All, And, Or logic to select samples by sample type. As an alternative, a sample type can be selected from a drop-down list.

## **"Sample ID"**

The "Sample Id" frame provides the option of selecting All, And, Or logic to select samples by a specific sample Id. The use of wildcards is supported to locate and display all samples that meet the entered criteria. The following characters can be used:

- "?" - Will match any single character
- "\*" - Will match zero or more characters
- "#" - Will match any single digit (0-9)

## **"Date"**

The "Date" frame provides the option of selecting All Samples, just those analyzed during the past n-days, or just those analyzed between two bounding dates.

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## **"Select Fields"**

The Select Fields command button displays a menu allowing users to select from the various fields available to be displayed in the grid in the lower portion of the form. The use of the selection buttons is explained on the form. See Select Report Fields for additional information on selecting fields.

## **"Between"**

The "Between" option permits the user to specify the sample dates to display.

## **"Monitor Display Options"**

The "Monitor Display Options" frame provides the user with the two options for displaying sample results. The format option permits the selection of either a Single Display format (only one Sample Report per display or printed page) or a Multi Display format (many Sample Reports per display or printed page). The default is the Single Display format. The display option permits the selection of data for categories of the Multi Display format by function.

## **"Multi Display"**

The "Multi Display" radio button enables the selection of display types for both the multiple sample displays or printed reports.

## **"During the Previous"**

The "During the Previous" option is the number of days prior to the current date that contain the retrieved sample results.

## **"Print Reports"**

The "Print Reports" option selects printed reports for the selected samples.

## **"ALL"**

The "All" option enables an all-inclusive retrieval of sample data for all customers, all analysts, all procedures, all sample types, and all dates.

## **"AND"**

The "And" option enables an exclusive retrieval of sample data that meet the selection criterion of the specific category and the selection criteria of all other categories.

## **"OR"**

The "Or" option enables the retrieval of samples that meet the selection criterion of the specific category or the selection criteria of any other categories.

## **"Single Display"**

The "Single Display" format generates a single Sample Report per display or printed page. The default for Monitor Display Options is Single Display.

## **"Display Reports"**

The "Display Reports" option generates sample reports displayed on the system monitor.

## **"Sample ID"**

The "Sample Id" Multi Display option displays only the data items pertaining to the Sample Id for the selected samples.

## **"Sample Prep"**

The "Sample Prep" Multi Display option displays only the data items pertaining to the Sample Preparations for the selected samples.

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### **"Sample Analysis"**

The "Sample Analysis" Multi Display option displays only the data items pertaining to the Sample Analysis (Results) for the selected samples.

### **"Standard Add"**

The "Standard Add" Multi Display option displays only the data items pertaining to the Sample Standard Additions for the selected samples.

### **"Quality Control"**

The "Quality Control" Multi Display option displays only the data items pertaining to the Quality Control functions for the selected samples.

### **"Display All Data"**

The "Display All Data" Multi Display option displays all data items pertaining to the selected samples.

### **"Calibration"**

The "Calibration" Multi Display option displays all data items pertaining to the Calibration.

### **"Spin Day Button"**

The "Spin Day" device increments or decrements the numbers of days in the Previous Days selection text box.

### **"Starting Date"**

The "Starting Day" box defines the beginning of the period for sample results retrieved by period.

### **"Ending Date"**

The "Ending Day" box defines the end of the period for sample results retrieved by period.



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## **"Single Sample Report"**

### **Summary Description**

The basic form contains four main groups displaying the various significant sample data. The four data groups are Sample Details, Analysis Details, Sample Results, and Final Results. Each group frame contains the significant data items within that section. Each group frame is described in detail in its own section. A toolbar is provided to control the functions of the display.

### **"Analysis Details Section"**

The "Analysis Details" section on the Single Sample Display reports the data elements required to interpret the analytical data. These data elements include Total Dilution Factor, Calibration Configuration Id, Calibration Data Id, Sample Amount, Standard Amount, and Calibration Alarms. Data elements not required for a specific sample type are not reported with that sample type.

### **"Final Results Section"**

The "Final Results" section on the Single Sample Display reports the interpreted results for the analysis. The number of these data elements varies by data type. For example, a customer sample would include the Final Result (Analytical Concentration corrected for dilution) and Relative Percent Deviation for a duplicate or Spike Recovery for a spiked sample.

### **"Results Section"**

The "Results" section on the Single Sample Display reports the results of the sample analysis. These data elements include Range, LSR Time Gates, Reference Ratio, Analysis Concentration, Lifetime, LSR Correlation Coefficient, Intensity Intercept, Integrated LSR Intensities, and Sample Analysis Alarms.

### **"Sample Details Section "**

The "Sample Details" section on the Single Sample Display reports the characteristics that identify the sample. These data elements include Sample Id, Sample Type, Basis Id, Description, Date/Time of analysis, Customer Id, Procedure Id, and Analyst.

### **"Display Intensity Options"**

The "Display intensity Options" on the toolbar enables the user to access the numerical sample intensity vector data.

### **"Plot Options"**

The "Plot Options" on the toolbar enables the user to access the graphical sample intensity vector data.

### **"Sample Details - Sample ID"**

The "Sample Id" is a unique identification assigned by the analyst when the sample data was entered on the Sample Input form. It is the primary reference to the sample data and remains with the specific sample as long as the information is in the KPAWin® database.

### **"Sample Details - Customer ID"**

The "Customer Id" identifies the customer or project submitting the sample or the laboratory preparing a blank or standard. The Customer Id is entered during sample data input.

### **"Sample Details - Sample Type"**

The "Sample Type" identifies the purpose of the analysis and the logic used to evaluate and report the data. Sample Type is specified during sample data input.

### **"Sample Details - Procedure ID"**

The "Procedure Id" identifies the documented laboratory protocol(s) used to prepare and analyze the sample. The

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Procedure Id is entered during sample data input.

#### **"Sample Details - Basis ID"**

The "Basis Id" is the Sample Id of the sample that the current sample is related to when results are calculated from more than one analysis. For example, the Basis Id for a spiked sample is the Sample Id for the unspiked sample. The Basis Id is assigned during sample data input.

#### **"Sample Details - Analyst ID"**

The "Analyst" is the KPA user performing the analysis of the sample. The Analyst's name or Id is entered during sample data input.

#### **"Sample Details - Sample Description"**

The "Description" is a unique descriptive label for a sample. The Description is entered during sample data input.

#### **"Sample Details - Date/Time"**

The "Date/Time" is the time stamp of the analyzed sample on display. KPAWin© assigns the Date/Time at the beginning of the analysis.

#### **"Analysis Details - Sample Amount"**

The "Sample Amount" is the Initial Analytical Volume of a sample analyzed by Standard Additions and quantified by internal calibration.

#### **"Analysis Details - Total Dilution Factor"**

The "Total Dilution Factor" is the product of the preparation dilution factor and the analysis dilution factor. The product of the Total Dilution Factor and the KPA analytical result is the final result, which is the concentration of the analyte in the authentic sample.

#### **"Analysis Details - Standard ID"**

The "Standard Id" is the control code assigned by the laboratory uniquely identifying the source and make-up protocol of the standard. The Standard Id is assigned during sample data input.

#### **"Analysis Details - Standard Concentration"**

A "Standard Concentration" is the make-up concentration of the analyte documented by a purchased or prepared standard according to a documented protocol.

#### **"Analysis Details - Standard Amount"**

The "Standard Amount" is the volume of standard spiked into a Standard Additions sample prior to dilution to the Final Analytical Volume.

#### **"Analysis Details - Calibration Configuration ID"**

The "Calibration Configuration Id" is the unique statement of the configuration of KPAWin© permanently stored in the KPAWin© database. KPAWin© assigns the Calibration Configuration Id when a background is measured.

#### **"Analysis Details - Calibration Data ID"**

The "Calibration Data Id" uniquely identifies a calibration. KPAWin© assigns the Calibration Data Id when a calibration is defined. A calibration consists of a background analysis, calibration standard analyses for one or both ranges, and the associated Calibration Configuration Id. Multiple Calibration Data Ids are created for a single, multiple-calibration-standard batch by inclusion of different standards. Since raw data are reevaluated before the generation of every report, data are unambiguously related to a specific Calibration Data Id reported with the sample data and its corresponding calibration data. The Calibration Data Id and its included analyses are stored as a permanent part of the data in the KPAWin© database.

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## **"Analysis Details - Calibration Alarms/Warning"**

The "Alarms/Warnings" are codes of the alarms and warnings violated during the Calibration. The Alarms/Warnings configuration tab defines the conditions of concern. Alarm levels are incidental to the condition or are specified in other Configuration Tabs for the analysis.

### **"Final Result - Final Result"**

The "Final Result" is the Analytical Result corrected for sample preparation and analytical sample dilution factors. The sample preparation dilution factor is the total final concentration of the prepared sample divided by the amount of authentic sample that is used in the sample preparation. The analytical sample dilution factor is the final amount of the diluted analytical sample divided by the amount of the aliquot of prepared sample taken for analysis. The Final Result is the product of the Analytical Result and the dilution factors. For example if the sample preparation dilution factor for a 1 ml portion of authentic sample that is ultimately diluted to 10 ml by the end of sample preparation is 10 (10 ml/1 ml). The analytical sample dilution factor for 1 ml of the prepared sample diluted to 100 ml is 100 (100 ml/1 ml). The Final Result for an Analytical Result of 2 ppb is 2 ppm ( $2\text{ppb} \times 10 \times 100$ ).

### **"Final Result - % Recovery"**

The "% Recovery" is the difference between the measured and true concentrations of a spike divided by the true concentration expressed as a percent.

For a spiked sample or a single-sample Method of Standard Addition analysis, the measured concentration is found by interrogating the batch calibration by the difference between the intercept intensity of the spiked sample and the intercept of the unspiked sample. The true concentration is the product of the spike concentration and the spike volume.

For a three-sample Method of Standard Addition analysis the measured concentration is found by interrogating the batch calibration by the intensity intercept of the basis sample. The true concentration is found by interrogating the internal calibration of the basis sample and the Method of Standard Addition spiked samples by the intercept of the basis sample.

### **"Final Result - Calibration Error"**

The "Calibration Error" is the correlation coefficient of the least-squares regression of a three-sample Method of Standard Addition analysis.

### **"Final Result - Slope Error (%)"**

The "Slope Error (%)" is the absolute difference between the slope of the external batch calibration and the slope of the internal calibration for a three-sample Method of Standard Additions analysis divided by the slope of the internal calibration expressed as a percent.

### **"Result - Range"**

The "Range" can be Low or High and indicates the analytical range used for the analysis.

### **"Result - Time Gates"**

The "Time Gates" indicates the initial and final time gates used for the least-squares regression of the intensity data. The Maximum Intensity/ Pulse and the Minimum Intensity Factor settings in the KPA Unit Settings Configuration Tab may force a deviation from the user-specified values as a result of gate shifting or gate dropping.

### **"Result - Reference Ratio"**

The "Reference Ratio" is the ratio of the Reference Intercept for the sample to the Reference Intercept for the background (Reference Mark). The intercepts are the extrapolation of the smoothed reference intensities or, as default, the coincidence corrected reference intensities if smoothing is deselected.

### **"Result - Concentration"**

The "Concentration" in the Analytical Results frame reports the magnitude of the KPA measurement, uncorrected for dilution factors, in the specified units.

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**"Result - Lifetime"**

"Lifetime" is the time in microseconds required for the population of the excited state to decrease to  $1/e$  of its original value. The slope of the least-squares regression of KPA intensity data is the negative inverse of the sample lifetime expressed in milliseconds.

**"Result - R Squared"**

The "R Squared" value is the correlation coefficient of the linear least-squares regression of the corrected sample intensities versus time function (related to time gate.)

**"Result - Intercept"**

The "Intercept" is the corrected sample intensity at time zero estimated from the linear least-squares regression of the intensity data.

**"Result - Integrated Intensities"**

The "Integrated Intensities" is the sum of all corrected sample intensities in the range of time gates specified for the linear least-squares regression meeting the Maximum Intensity/Pulse and Minimum Intensity Factor specifications in the KPA Unit Settings Configuration Tab for the sample.

**"Result - Sample Alarms/Warnings"**

The "Sample Alarms/Warnings" are codes of the alarms and warnings violated during the analysis. The Alarms/Warnings Configuration Tab for the analysis defines the off-standard conditions. Alarms are reported by an "A" followed by two digits for the condition in the Alarm/Warning tab. Warnings are reported similarly with a "W" rather than an "A".

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## **"Kinetic Phosphorescence Analyzer System Contacts"**

### **Summary Description**

The "Kinetic Phosphorescence Analyzer System Contacts" screen displays the Technical information concerning the KPAWin© system. It provides users with The Technical Contacts including telephone numbers, Fax numbers, e-mail addresses and the address of Chemchek Instruments, Inc.

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## **"Kinetic Phosphorescence Analyzer Technical Information"**

### **Summary Description**

The "Kinetic Phosphorescence Analyzer Technical Information" screen displays technical information concerning the KPAWin Hardware system. It provides users with the minimum technical hardware requirements to operate the KPAWin© software.

### **"Contacts"**

The "Contacts" button displays the Technical Contacts at Chemchek Instruments, Inc.

### **"OK"**

The "OK" button exits the display and returns to the previous display.

### **"Hardware Requirements"**

This frame displays the Hardware System Requirements to run the KPAWin© software in a reasonably responsive mode. Extensive data computations and manipulations occur during the analysis process. Consequently, a faster processor, memory, and video display is needed to enhance the performance of the software.

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## **"Units Administration Menu"**

### **Summary Description**

The "Units Administration" form has text areas that contain unit information such as units, unit's name, conversion formulas, and conversion factors. A checkbox indicates if the above listed unit is Active or Inactive. Setting or clearing the checkbox changes the Status of each. If the Select button is depressed, a list of current units from the KPAWin© Database is displayed. The user then selects one of these units and the database information populates the text areas with the database entries.

### **"Units Drop-down List"**

The "Units" drop-down list displays all units currently entered in the KPAWin© Database. It displays whenever the Select button is depressed.

### **"Inactive Units"**

The "Inactive" checkbox identifies a unit that is not currently Active.

### **"Add"**

The "Add" button opens a new Units Administration form and positions the cursor in the first entry of the form.

### **"Apply"**

The "Apply" button updates the KPAWin© database and returns to the Units Administration display.

### **"Cancel"**

The "Cancel" button restores the Units Administration form and the KPAWin© database to the original values, exits the current display and returns to the previous display.

### **"Clear Entries"**

The "Clear" button clears all existing data from the Units Administration display and returns to the current display.

### **"OK"**

The "OK" button updates any record in the Units Administration form and the KPAWin© database, exits the current display and returns to the previous display.

### **"Select"**

The "Select" button queries the KPAWin© database and displays a drop-down list of all active units of measurement in the database. The user then selects one unit to edit.

### **"Units ID"**

The "Units Id" box identifies the concentration units of the input and output data for an individual sample.

### **"Concentration Name"**

The "Concentration Name" box identifies the concentration units of the input and output data for an individual sample.

### **"Concentration Multiplier"**

The "Concentration Multiplier" is the numerical value, exclusive of density and specific activity factors, that is divided into the value of the Concentration Name to convert the input units to KPAWin© database units of µg/l.



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**"Sample/Standard Quantity"**

The " Sample/Standard Quantity " identifies the units of sample/standard consistent with the input Concentration Name. The Sample/Standard Quantity is always ml for input Concentration Units expressed on a per volume basis. The Sample/Standard Quantity is always g for input Concentration Units expressed on a per mass basis.

**"Sample/Standard Quantity Multiplier"**

The "Sample/Standard Quantity Multiplier" is the numerical value, exclusive of density, multiplied by the identified Sample/Standard Quantity to convert the input units to KPAWin© database units of  $\mu\text{l}$ . The value is always .001 with per mass unit selection prompting the user for a density in g/ml. This conversion is applied to input and output units automatically to provide a consistent basis for the database. It does not require user action.

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## **"User Administration Menu"**

### **Summary Description**

The User Administration form has a number of text boxes that contain user information such as User Id and Name. It also has a checkbox indicating if the above listed user is Active or Inactive. You change the Active/Inactive Status simply by setting or clearing the checkbox. The form also provides the option to include user passwords. In addition, if the select button is depressed, a list of current users from the KPAWin© Database is displayed. The user then selects one of these users and the database information populates the text areas.

### **"Drop-down User List"**

The "User" drop-down list displays all users currently entered in the KPAWin© Database. It displays whenever the Select button is depressed.

### **"Inactive User"**

The "Inactive" checkbox identifies a user that is not Active. Only active users are displayed in the Login selection list.

### **"Standard User"**

The "Standard User" checkbox identifies a user with routine privileges. Only a supervisor can edit this screen.

### **"Supervisor"**

The "Supervisor" checkbox indicates a user with all privileges within the KPAWin© software.

### **"Add"**

The "Add" button opens a new User Administration form and positions the cursor in the first entry of the form.

### **"Apply"**

The "Apply" button updates the KPAWin© database and returns to the User Administration display.

### **"Cancel"**

The "Cancel" button restores the User Administration form and the KPAWin© database to the original values and clears the current display.

### **"Clear Entries"**

The "Clear" button clears all existing data from the User Administration display and returns to the display.

### **"OK"**

The "OK" button updates any record in the User Administration form and the KPAWin© database, exits the current display and returns to the previous display.

### **"Select"**

The "Select" button queries the KPAWin© database and displays a drop-down list of all users in the database. A supervisor then selects one user to edit.

### **"KPAWin© Login ID"**

The "KPAWin© Login Id" field contains the KPAWin© Login Id. A Supervisor enters it when the user information is entered.

### **"User First Name"**

The "First Name" field contains the User First Name. A Supervisor enters it when the user information is entered.

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**"User Last Name"**

The "Last Name" field contains the User Last Name. A Supervisor enters it when the user information is entered.

**"User Password"**

The "User Password" field contains the User Password, if required. A Supervisor enters it when the user information is entered.

**"User Password Conformation"**

The "User Password Confirmation" field is used to Confirm or Verify the User Password.

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## **KPAWin© Error Codes**

### **KPAWin© Program Errors**

- E1 KPAWin© Error: Unexpected
- E2 KPAWin© Error: Bad Routine Call Parameter
- E3 KPAWin© Error: Incorrect call to Move Samples
- E4 KPAWin© Error: Message Format is Bad
- E5 KPAWin© Error: Undefined Alarm and Warning Message
- E6 KPAWin© Error: Report Record not found
- E7 KPAWin© Error: Sample Record does not exist
- E8 KPAWin© Error: Intensity Record does not exist
- E9 KPAWin© Error: Configuration
- E10 KPAWin© Error: ToolBar Menu

### **KPAWin© Database Errors**

- E51 KPAWin© Database Error
- E52 KPAWin© Database Error: Record NOT Found

### **User Control Messages**

- E91 User Escape
- E92 User Abort
- E93 User Cancel
- E94 User Ignore
- E95 User Retry

### **Sample Changer Error Messages (100 Series)**

- E101 Sample Changer Movement Data is Missing
- E102 Sample Changer Error
- E103 RackNb: & VialNb: NOT Valid
- E104 Sample Changer Error
- E105 ERROR - Number of Sample Vials Exceed number of Mix Vials
- E106 Reference Lifetime Too Short
- E107 Reference Intensity Is Excessive
- E108 Auto Changer Error

### **Calculations Error Messages (200 Series)**

- E201 Unable To Calculate Reference Intercept
- E202 Unable To Calculate Sample Intercept
- E203 Equals Water Blank
- E204 Not enough Samples available for &1& calculation
- E205 Basis Sample Loop
- E206 Auto Calibration Completed
- E207 All Samples must contain same number of Measurements
- E208 Spike and Sample have same Integrated Intensities
- E209 Initial Analysis Volume is Zero
- E210 Sample Must have Standard Concentration Specified
- E211 There are NO Valid Calibration Points
- E212 Calibration Curve Fit Error
- E213 KPA NOT Calibrated
- E214 Precounts High For High Range
- E215 Confidence Test Aborted
- E216 Basis Sample Must Be Assigned before Measurement
- E217 Basis Sample Must Be Measured First
- E218 There are NO Calibration Points

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### **CurveFit Error Messages (400 Series)**

E401 Inverse Bad

### **DBRoutines Error Messages (500 Series)**

E501 Database File: & FileName & could NOT be Closed

E502 Database File: & FileName & could NOT be Opened"

### **KPA Error Messages (600 Series)**

E601 Check KPA & Msg

E602 Auto Changer Error

E603 Put Sample & SampleId Msg(SampleData)

E604 Put Sample Into KPA Cancel/Error

E605 Gilson Device Not Responding

E630 KPA Analyze Error

### **KPAIO Error Messages (700 Series)**

E701 KPA Not Responding

E702 This KPA Cannot Use this Command

E703 Sample Intensity KPA Data Read Error

E704 Reference Intensity KPA Data Read Error

E705 Max Nb of Intensities & Data values must be received

E706 Place RANGE selector switch in the & SwitchMsg & position"

E707 This KPA Cannot Use the OUTALL Command

E708 There are no Standards for this sample

E709 Reference Intensity Is Excessive

E710 KPA NOT Ready

E711 Waiting for KPA Data

E712 Waiting for KPA Laser

E713 KPA Checksum Error

E714 KPA Background Measurement Required

### **MDIControl Error Messages (800 Series)**

E801 Cannot find FindString

### **SelectCalData Error Messages (900 Series)**

E901 No & Title & Found

### **ShowResults Error Messages (1000 Series)**

E1001 Reference  $R^2 < .99$

### **Utilities Error Messages (1100 Series)**

E1101 KPAWin© Configuration

E1102 The Value &1& is NOT WITHIN the Acceptable Range

E1103 Range Must be Between &Value1& and &Value2&

E1104 Resetting to Previous Value

E1105 & DataText & Validation Rule is NOT valid & Validation Rule

### **MDIForm Error Messages (1200 Series)**

E1201 Only ONE instance of + KPAWin + is allowed

### **General Config Error Messages (1300 Series)**

E1301 Invalid Configuration Name provided - Default: & DefaultName & Used

## **EXHIBIT C**

### **Pilot Study Performance Data**

## **EXHIBIT C.1**

### **300 gpm Pilot System Performance Data**



## **EXHIBIT C.2**

### **1200 gpm Pilot System Performance Data**

## **EXHIBIT D**

### **Worker Dose Assessment**



Environmental Restoration Group, Inc.  
8809 Washington St NE, Suite 150  
Albuquerque, NM 87113  
ph: (505) 298-4224  
www.ERGoffice.com

### TECHNICAL MEMORANDUM

<b>To:</b> Tom Wohlford (HMC); Toby Wright (WES)	<b>Date:</b> August 8, 2017
<b>From:</b> Randy Whicker (ERG)	<b>Project:</b> HMC Grants Reclamation Project
<b>Direct:</b> 970-556-1174	<b>Task(s):</b> Radiation Protection Administrator
<b>Cc:</b> Chuck Farr (ERG)	
<b>Subject:</b> Occupational dose and toxicity assessment for potential uranium exposure at Zeolite facilities	

Dear Mr. Wohlford and Mr. Wright,

Per the request of Toby Wright (Wright Environmental Services, Inc.), this Technical Memorandum provides conservative occupational radiation dose and non-radiological (toxicity) assessments for workers that could potentially be occupationally exposed to uranium when working at the Zeolite water treatment facilities at the Homestake Mining Company of California (HMC) Grants Reclamation Project. The intent of this information is to support a license amendment application regarding operation of these facilities as part of HMC's continued efforts to remediate impacted groundwater at the HMC Grants Reclamation Project site.

Please let me know if you have questions or need more information.

Thanks,

***Randy Whicker, CHP***  
*RPA, HMC Grants Reclamation Project*



Environmental Restoration Group, Inc.  
8809 Washington St. NE, Suite 150  
Albuquerque, NM 87113  
Email: [RandyWhicker@ergoffice.com](mailto:RandyWhicker@ergoffice.com)

## Occupational Dose/Toxicity Assessment for Zeolite Facility Operations

### 1. Introduction

This Technical Memorandum provides conservative radiological dose and chemical toxicity assessments for workers at the Homestake Mining Company of California (HMC) Grants Reclamation Project whom could become occupationally exposed to uranium when working at the Zeolite water treatment facilities. The Zeolite facilities, designed to remove uranium from impacted groundwater, include two separate systems [300 and 1,200 gallons per minute (gpm) water treatment capacity] located on top of the Large Tailings Pile. Treatment pond series (trains) containing Zeolite media beds are filled with impacted groundwater during water treatment cycles. During occasional maintenance periods, the water is drained and media beds are exposed to the atmosphere. Each circumstance has the potential for occupational exposure to uranium as described below.

### 2. Potential Exposure Scenarios

Based on inspection of the Zeolite facilities and review of operational procedures conducted by HMC's Radiation Protection Administrator (RPA) on July 21, 2017, the following two potential pathways for occupational exposure to uranium from Zeolite water treatment operations have been identified:

- 1) Accidental ingestion of dissolved uranium in contaminated influent groundwater.
- 2) Incidental inhalation of airborne particulates from Zeolite beds when water treatment ponds are drained for maintenance and become exposed to the atmosphere (the media beds can dry out, and under this condition there is the potential for resuspension of Zeolite particulates to air).

For the ingestion pathway, it was conservatively assumed for this dose assessment that a worker could conceivably fall into one of the ponds and accidentally ingest as much as 1 liter of water containing elevated levels of dissolved uranium. For the inhalation pathway, it was conservatively assumed that a worker could be intermittently exposed to airborne particulate matter resuspended from dry zeolite beds containing elevated levels of uranium.

### 3. Source Terms

The source terms used for each occupational exposure scenario are based on the assumptions and calculations shown at right regarding uranium concentrations in influent groundwater and maximally loaded Zeolite media as developed by Hydro-Engineering, LLC. The key parameters for this assessment are the estimated concentrations of uranium in influent groundwater and

Assumptions	
Influent Concentration	0.4mg/l
Effluent Concentration	0.03mg/l
Flow Rate	300gpm
Time between Regeneration Cycles	60days
1 Cell Volume of Zeolite	15045cf
Zeolite Density	53.7lbs/cf
Calculated Results	
Volume of Water Treated	25,920,000gal
Mass of Uranium deposited	80.0lbs
Uranium in Zeolite Concentration	99.03mg/kg

in loaded Zeolite media. These estimates are believed to be conservative.

For the ingestion pathway, the concentration of uranium in the ingested water is assumed equivalent to an average influent concentration of 0.4 mg/L as indicated above.

For the inhalation pathway, mass-based and radioactivity-based concentrations of uranium in the inhaled air were calculated based on the concentration of uranium in loaded zeolite media as given above ( $\approx 100$  mg/kg), an assumed mass loading factor for inhalation of  $0.0001 \text{ g/m}^3$  [the default value for releases from soil to air as given in the RESRAD-OFFSITE computer code (ANL, 2013)], and a specific activity for natural uranium of  $0.691 \text{ } \mu\text{Ci/g}$  (ORISE, 2017).

Using applicable parameters noted above, the assumed mass-based concentration of uranium in air was calculated as follows:

$$100 \frac{\text{mg}}{\text{kg}} \left( \frac{\text{kg}}{1000\text{g}} \right) \left( \frac{0.0001\text{g}}{\text{m}^3} \right) = 1 \times 10^{-5} \frac{\text{mg}}{\text{m}^3}$$

Using applicable parameters noted above, the assumed activity-based concentration of uranium in air was calculated as follows:

$$100 \frac{\text{mg}}{\text{kg}} \left( \frac{\text{kg}}{1000\text{g}} \right) \left( \frac{0.0001\text{g}}{\text{m}^3} \right) \left( \frac{\text{g}}{1000 \text{ mg}} \right) \left( \frac{0.691\mu\text{Ci}}{\text{g}} \right) \left( \frac{\text{m}^3}{1 \times 10^6 \text{ mL}} \right) = 6.9 \times 10^{-15} \frac{\mu\text{Ci}}{\text{mL}}$$

#### 4. Radiological Dose Assessment

##### INGESTION PATHWAY

For the ingestion pathway, 10 CFR 20 Appendix B gives an Annual Limit on Intake (ALI) for biologically soluble and insoluble forms of natural uranium as follows:

- Soluble uranium ALI (non-stochastic) =  $10 \text{ } \mu\text{Ci}$  (critical organ = bone surface)
- Insoluble uranium ALI (stochastic) =  $20 \text{ } \mu\text{Ci}$

The amount of uranium activity ingested under the assumed scenario is calculated as follows:

$$\text{Ingestion Intake} = \left( \frac{0.4 \text{ mg}}{\text{L}} \right) \left( \frac{0.000691 \text{ } \mu\text{Ci}}{\text{mg}} \right) (1 \text{ L}) = 2.8 \times 10^{-4} \text{ } \mu\text{Ci}$$

Assuming that dissolved uranium in the influent groundwater is biologically soluble, the assumed intake of uranium via the ingestion pathway would result in a committed dose equivalent (CDE) to bone of:

$$\text{CDE} = (2.8 \times 10^{-4} \text{ } \mu\text{Ci}) \left( \frac{50 \text{ rem}}{10 \text{ } \mu\text{Ci}} \right) \left( \frac{1000 \text{ mrem}}{\text{rem}} \right) = 1.4 \text{ mrem}$$

Assuming that the dissolved uranium in the influent groundwater is biologically insoluble, the assumed ingestion intake of uranium would result in a committed effective dose equivalent (CEDE) to the whole body of:

$$CEDE = (2.8 \times 10^{-4} \mu\text{Ci}) \left( \frac{5 \text{ rem}}{20 \mu\text{Ci}} \right) \left( \frac{1000 \text{ mrem}}{\text{rem}} \right) = 0.07 \text{ mrem}$$

The biological solubility of dissolved uranium in treated groundwater has not been established, but regardless of solubility class, the conservatively calculated doses above are negligible relative to the annual stochastic dose limit (5 rem/yr) or non-stochastic dose limit (50 rem) for workers as specified in 10 CFR 20.1201.

#### INHALATION PATHWAY

For the inhalation pathway, 10 CFR 20 Appendix B gives a Derived Air Concentration (DAC) for soluble and insoluble forms of natural uranium as follows:

- Soluble uranium DAC =  $5 \times 10^{-10} \mu\text{Ci/mL}$
- Insoluble uranium DAC =  $2 \times 10^{-11} \mu\text{Ci/mL}$

It would be unrealistic to assume that any worker could be continuously exposed to the calculated airborne uranium activity concentration of  $6.9 \times 10^{-15} \mu\text{Ci/mL}$  (Section 3). It was instead conservatively assumed that on an annual basis, a worker is exposed to this airborne uranium concentration 10% of the time (200 hours out of a total of 2,000 work hours per year). Based on this assumption, the annual CEDE from inhalation of uranium for each solubility class are calculated as follows:

$$CEDE (\text{soluble } U) = \left( 6.9 \times 10^{-15} \frac{\mu\text{Ci}}{\text{mL}} \right) \left( \frac{1 \text{ DAC}}{5 \times 10^{-10} \frac{\mu\text{Ci}}{\text{mL}}} \right) (200 \text{ hrs}) \left( \frac{2.5 \text{ mrem}}{\text{DAC} - \text{hr}} \right) = 6.9 \times 10^{-3} \text{ mrem}$$

$$CEDE (\text{insoluble } U) = \left( 6.9 \times 10^{-15} \frac{\mu\text{Ci}}{\text{mL}} \right) \left( \frac{1 \text{ DAC}}{2 \times 10^{-11} \frac{\mu\text{Ci}}{\text{mL}}} \right) (200 \text{ hrs}) \left( \frac{2.5 \text{ mrem}}{\text{DAC} - \text{hr}} \right) = 0.2 \text{ mrem}$$

Regardless of the actual biological solubility of uranium loaded on Zeolite media, the expected CEDE to any worker is negligible relative to the 5 rem dose limit for radiation workers. This would also be true if a worker were continuously exposed for 2,000 hours/year to this concentration of insoluble uranium in air due to releases of particulate Zeolite media to air (the expected CEDE under this scenario would be about 2 mrem).

## 5. Non-radiological (Toxicity) Assessment

Although the biological solubility of dissolved uranium in treated groundwater, or uranium adsorbed to Zeolite media, has not been established, it is known that soluble forms present a toxicity concern for the kidneys. 10 CFR 20.1201(e) requires licensees to limit the intake of soluble uranium by workers to 10 mg in any 1-week period based on chemical toxicity. For the accidental ingestion scenario, the occupational intake of uranium from ingestion of 1 liter of influent groundwater at the Zeolite facilities would be 0.4 mg. To exceed the 10 mg/week regulatory limit, a worker would have to ingest 25 liters of influent groundwater in 1 week.

With respect to the 10 mg/week regulatory limit on intakes via the inhalation pathway, footnote 3 of Appendix B to 10 CFR 20 gives an average airborne concentration limit of 0.2 mg/m<sup>3</sup> for soluble uranium. Assuming a mass-based concentration of soluble uranium in air of  $1 \times 10^{-5}$  mg/m<sup>3</sup> as previously calculated (Section 3), a breathing rate of 20 L/minute, and a maximum plausible exposure time of 40 hours in any given week, the calculated weekly inhalation intake to a worker occupationally exposed to uranium in airborne particulates from the Zeolite beds was calculated as follows:

$$\text{Inhalation Intake} = 1 \times 10^{-5} \frac{\text{mg}}{\text{m}^3} \left( \frac{\text{m}^3}{1000 \text{ L}} \right) \left( \frac{20 \text{ L}}{\text{min}} \right) \left( \frac{60 \text{ min}}{\text{hour}} \right) (40 \text{ hours}) = 4.8 \times 10^{-4} \text{ mg}$$

The calculated maximum plausible intake of uranium via the inhalation pathway when working at the Zeolite facilities ( $4.8 \times 10^{-4}$  mg) is insignificant relative to the 10 mg/week regulatory limit on intakes based on toxicity concerns.

## 6. Conclusions

The results of this assessment indicate that potential occupational exposures to uranium from Zeolite facility operations will not result in significant radiological doses or intakes with respect to toxicity concerns. The conservatively calculated estimate for each occupational health endpoint evaluated is far below any regulatory limits.

## 7. References

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