

MATERIALS LICENSE

Pursuant to the Atomic Energy Act of 1954, as amended, the Energy Reorganization Act of 1974 (Public Law 93-438), and Title 10, Code of Federal Regulations, Chapter I, Parts 30, 31, 32, 33, 34, 35, 36, 39, 40, and 70, and in reliance on statements and representations heretofore made by the licensee, a license is hereby issued authorizing the licensee to receive, acquire, possess, and transfer byproduct, source, and special nuclear material designated below; to use such material for the purpose(s) and at the place(s) designated below; to deliver or transfer such material to persons authorized to receive it in accordance with the regulations of the applicable Part(s). This license shall be deemed to contain the conditions specified in Section 183 of the Atomic Energy Act of 1954, as amended, and is subject to all applicable rules, regulations, and orders of the Nuclear Regulatory Commission now or hereafter in effect and to any conditions specified below.

302368

Licensee		In accordance with letter dated February 26, 1997	
1. CERAC, Inc.		3. License Number SMB-1402 is amended in its entirety to read as follows:	
2. P. O. Box 1178 Milwaukee, WI 53201		4. Expiration Date May 31, 2005	
6. Byproduct, Source, and/or Special Nuclear Material		5. Docket or Reference No. 040-08805	
7. Chemical and/or Physical Form		8. Maximum Amount that Licensee May Possess at Any One Time Under This License	
A. Natural uranium	A. Oxides and salts	A. 500 kilograms	
B. Depleted uranium	B. Metals	B. 500 kilograms	
C. Natural thorium	C. Metals, oxide and salts	C. 7,000 kilograms	

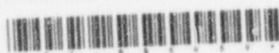
9. Authorized Use:

- A., B. and C. The licensee is authorized to manufacture, process, package, and distribute thorium and uranium compounds, as described in letter dated July 19, 1994, to persons generally licensed pursuant to Section 40.22 of 10 CFR Part 40 and to persons authorized to receive licensed material pursuant to terms and conditions of a specific license issued by the Nuclear Regulatory Commission or an Agreement State.

CONDITIONS

10. Licensed material shall be used only at the licensee's facilities located at 407 N. 13th Street, Milwaukee, Wisconsin.
11. Licensed material shall be used by, or under the supervision of, Mitchell C. Colton, Ph.D.
12. The Radiation Safety Officer for this license is Mitchell C. Colton, Ph.D.
13. The licensee is authorized to transport licensed material only in accordance with the provisions of 10 CFR Part 71, "Packaging and Transportation of Radioactive Material."

280039

9703280275 970314
PDR ADOCK 04008805
C PDR

COPY

**MATERIALS LICENSE
SUPPLEMENTARY SHEET**

License Number

SMB-1402

Docket or Reference Number

040-08805

Amendment No. 05

14. Licensed material shall not be used in or on human beings.
15. Except as specifically provided otherwise in this license, the licensee shall conduct its program in accordance with the statements, representations, and procedures contained in the documents, including any enclosures, listed below. The U.S. Nuclear Regulatory Commission's regulations shall govern unless the statements, representations, and procedures in the licensee's application and correspondence are more restrictive than the regulations.
- A. Application dated August 25, 1993; and
- B. Letters dated March 15, 1994, July 19, 1994, February 6, 1995, April 28, 1995, May 10, 1995, and February 1997.

FOR THE U.S. NUCLEAR REGULATORY COMMISSION

Date

March 14, 1997

By

Charles E. Trojer
Nuclear Materials Licensing Branch, Region III

COPY

BETWEEN:

License Fee Management Branch, ARM
and
Regional Licensing Sections

(FOR LFMS USE)
INFORMATION FROM LTS

Program Code: 11300
Status Code: 0
Fee Category: 2C
Exp. Date: 20050531
Fee Comments:
Decom Fin Assur Req'd: Y

1997 MAR -3 AM 11:42

LICENSE FEE TRANSMITTAL

A. REGION

1. APPLICATION ATTACHED

Applicant/Licensee: CERAC, INC.
Received Date: 970226
Docket No: 4008805
Control No.: 302368
License No.: SMB-1402
Action Type: Amendment

2. FEE ATTACHED

Amount: 420
Check No.: 64429

3. COMMENTS

Signed
Date

D. Hersey
2-27-97

B. LICENSE FEE MANAGEMENT BRANCH (Check when milestone 03 is entered / ☒)

1. Fee Category and Amount: 2C #420

2. Correct Fee Paid. Application may be processed for:

Amendment
Renewal
License

3. OTHER

Signed
Date

SC
3/3/97

MAR 07 1997

Log	<i>Mar 1 III</i>
Remitter	
Check No.	<i>64429</i>
Amount	<i>#420</i>
Fee Category	<i>2C</i>
Type of Fee	<i>and</i>
Date Check Rec'd	<i>3/3/97</i>
Date Completed	<i>3/3/97</i>
By:	<i>SC</i>

CERACTM Incorporated
Quality Manufacturing Since 1964

P.O. BOX 1178 · MILWAUKEE, WI 53201-1178 · U.S.A. · PHONE: 414-289-9800 · FAX: 414-289-9805

Reference: Amendment to License No. SMB-1402, part 1, February 26, 1997

Re. Effluent Releases to the Sewer

This is an interim procedure as a follow up to an enforcement action. *We request an expedited review for this part of the amendment.* Other topics, including our bioassays program, detailed water effluent sampling, and stack effluent evaluations, will be covered at a future time to be included with this amendment.

The following is a proposed change to our original license application. We propose, for the near term, releasing our liquid effluent as discussed below. This is for both our chemical process and our fume scrubber liquids. Longer term solutions will be discussed as we develop our plans.

Qualifications for Release:

1. It is sufficient to demonstrate compliance with 10 CFR 20.2003(a)(1) regarding solubility of material released to the sanitary sewer by passing all liquid effluent in question through a 0.45 μ or smaller filter.
2. Total activity levels of material released to the sewer will be within release limits. In the short term, we will sample and test every release until we have an agreed-upon sampling and testing protocol.
3. For the short term, filter integrity will be maintained by the following procedure:
 - a) Change the 0.45 μ filter at least once per month (represents about 600-1000 gallons)
 - b) or more often if determined necessary (e.g. after we process the drums we have collected)
 - c) or as we change process streams. For example, process water is typically pH of 8 or higher and scrubber water is about pH of 1-2. We would change filters before filtering one after the other.
 - d) As an additional safety measure, we will put a 0.20 μ filter after the 0.45 μ filter and pass all liquids through it as well. This 0.20 μ filter will be changed whenever the 0.45 μ filter is changed

Sincerely,



Dr. Mitchell C. Colton
Radiation Safety Officer

RECEIVED

FEB 26 1997

REGION III

302368

MAR 17 1997

Mitchell C. Colton, Ph.D.
CERAC, Inc.
P. O. Box 1178
Milwaukee, WI 53201

Dear Dr. Colton:

Enclosed is Amendment No. 05 to your NRC Material License No. SMB-1402 in accordance with your request.

Please note that in accordance with 10 CFR 30.36 of the regulations, your license expiration date has been extended by five years.

Please review the enclosed document carefully and be sure that you understand all conditions. If there are any errors or questions, please notify the U.S. Nuclear Regulatory Commission, Region III office at (630) 829-9887 so that we can provide appropriate corrections and answers.

Please be advised that your license expires at the end of the day, in the month, and year stated in the license. Unless your license has been terminated, you must conduct your program involving byproduct materials in accordance with the conditions of your NRC license, representations made in your license application, and NRC regulations. In particular, note that you must:

1. Operate in accordance with NRC regulations 10 CFR Part 19, "Notices, Instructions and Reports to Workers; Inspections," 10 CFR Part 20, "Standards for Protection Against Radiation," and other applicable regulations.
2. Notify NRC, in writing, within 30 days:
 - a. When the Radiation Safety Officer permanently discontinues performance of duties under the license or has a name change; or
 - b. When the licensee's mailing address changes (no fee is required if the location of byproduct material remains the same).

302368

3. In accordance with 10 CFR 30.36(b) and/or license condition, notify NRC, promptly, in writing, and request termination of the license when you decide to terminate all activities involving materials authorized under the license.
4. Request and obtain a license amendment before you:
 - a. Change Radiation Safety Officers;
 - b. Order byproduct material in excess of the amount, or radionuclide, or form different than authorized on the license;
 - c. Add or change the areas of use or address or addresses of use identified in the license application or on the license; or
 - d. Change ownership of your organization.
5. Submit a complete renewal application with proper fee or termination request at least 30 days before the expiration date of your license. You will receive a reminder notice approximately 90 days before the expiration date. Possession of byproduct material after your license expires is a violation of NRC regulations. A license will not normally be renewed, except on a case-by-case basis, in instances where licensed material has never been possessed or used.

In addition, please note that NRC Form 313 requires the applicant, by his/her signature, to verify that the applicant understands that all statements contained in the application are true and correct to the best of the applicant's knowledge. The signatory for the application should be the licensee or certifying official rather than a consultant.

You will be periodically inspected by NRC. Failure to conduct your program in accordance with NRC regulations, license conditions, and representations made in your license application and supplemental correspondence with NRC will result in enforcement action against you. This could include issuance of a notice of violation, or imposition of a civil penalty, or an order suspending, modifying or revoking your license as specified in the General Policy and Procedures for NRC Enforcement Actions. Since serious consequences to employees and the public can result from failure to comply with NRC requirements,

M. Colton

-3-

prompt and vigorous enforcement action will be taken when dealing with licensees who do not achieve the necessary meticulous attention to detail and the high standard of compliance which NRC expects of its licensees.

Sincerely,

Original Signed By
Cassandra Frazier
Nuclear Materials Licensing Branch

License No. SMB-1402
Docket No. 040-08805

Enclosure: Amendment No. 05

DOCUMENT NAME: M:\04008805.CL7

To receive a copy of this document, indicate in the box: "C" = Copy without attachment/enclosure "E" = Copy with attachment/enclosure "N" = No copy

OFFICE	DNMS/RIII								
NAME	CFFrazier:brt								
DATE	03/14/97								

OFFICIAL RECORD COPY

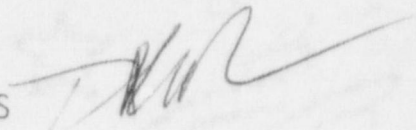


UNITED STATES
NUCLEAR REGULATORY COMMISSION

WASHINGTON, D.C. 20555-0001

March 11, 1997

MEMORANDUM TO: John Madera, Chief
Nuclear Materials Inspection Branch 1
Region III

FROM: Donald A. Cool, Director
Division of Industrial and
Medical Nuclear Safety, NMSS 

SUBJECT: TECHNICAL ASSISTANCE REQUEST CONCERNING CERAC, INC., AND
EFFLUENT RELEASES TO THE SANITARY SEWER

I am responding to your technical assistance request dated February 20, 1997, (attached) requesting a determination if passing radioactive material liquid effluents through a 0.45 micron filter is an adequate methodology to insure compliance with 10 CFR 20.2003(a)(1). In a letter from Cerac, Inc., dated February 11, 1997, the licensee proposes to use a 0.45 micron filter, or smaller, and pass all liquid effluent in question through it prior to discharge to the sanitary sewer.

The filtration and radiometric analysis of suspended solids is described as an acceptable method for determining solubility of an effluent in Information Notice 94-07, "Solubility Criteria for Liquid Effluent Releases to Sanitary Sewerage Under the Revised 10 CFR Part 20." The Notice cited as acceptable procedures using a 0.45 micron filter to determine the suspended solids in the effluent. The IN indicates "... activity in the suspended solids portion of the effluent greater than that found in similarly processed background water would indicate the presence of insoluble radioactive material." Conversely, if the material passes through the filter, the portion that passes through should be considered soluble. Therefore, the proposal by the licensee in Cerac's February 11, 1997, letter is deemed adequate to show compliance with 10 CFR 20.2003(a)(1). It should be noted that a 0.2 micron filter, mentioned in their letter, is neither used in the standard analysis, nor is it required to show compliance.

Attachment: As stated

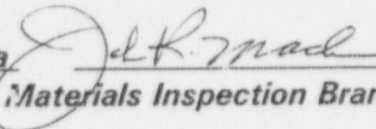
CONTACT: Joe DeCicco, NMSS/IMNS
(301) 415-7833
e-mail (jxd1)

MAR 14 1997

REGIONAL TECHNICAL ASSISTANCE REQUEST FORM

Date: February 20, 1997

Mail or E-Mail to: Donald A. Cool (DAC), Mail Stop: 6H3-OWFN, If E-mail, cc: CLE
Division of Industrial and Medical Nuclear Safety, NRCSS

From: John Madera  Region III
Chief, Nuclear Materials Inspection Branch I

Licensee: CERAC, Inc. License No. SMB-1402

- ☐ Control No. _____ (if applicable)
- ☐ Letter dated: _____ (if applicable)
- ☐ Suggested change in licensing procedure (enclosed):
- ☐ Problem/Issue:

The licensee possesses an NRC license to process natural thorium which results in radioactive liquid and air effluent releases to the environment. On December 18, 1996, the NRC identified the licensee had not determined the activity or solubility of liquid effluent discharges into the sanitary sewer system. The licensee's immediate corrective action was to hold all liquid effluent discharges generated from licensed activities in 55 gallon drums until the liquid could be analyzed for activity and solubility. Prior to placing the liquid effluent into 55 gallon containers, the licensee filtered the water with a 0.45 micron filter to remove suspended solids in the liquid. In addition, the licensee also possesses a water scrubber which removes licensed material from air effluent discharges prior to release to the environment.

The licensee sent a faxed letter to the NRC dated February 19, 1997 which contains analysis data of water samples taken from 13 55 gallon (2.09E5 milliliter) containers (One sample from each container) and the scrubber holding reservoir. According to the licensee, each sample was analyzed for dissolved (soluble) and suspended (insoluble) licensed material. The licensee stated that all gross alpha activity was assumed to be thorium-232 decay. According to the licensee, licensed activities generate approximately 14 55 gallon drums (2.93E6 milliliters) per month.

In a faxed letter to the NRC dated February 11, 1997, the licensee stated that approximately 5.44E5 gallons (2.07E9 milliliters) of water per month was used by the licensee and sent through the sanitary sewer system. According to the licensee's calculations in the above letter, one 55 gallon container containing 3E5 microcuries per milliliter of licensed material would be approximately 1% of the monthly sanitary sewer release limit (see 10 CFR 20, Appendix B, table 3: thorium-232). In addition assuming the licensee generates approximately 14 55 gallon of radioactive liquid effluent per month into the sanitary sewer system, the licensee's water dilution factor is approximately 3 orders of magnitude.

The licensee is requesting that radioactive liquid effluent filtered through a 0.45 micron filter be placed directly into the sanitary sewer system without further testing. The licensee stated that NRC Information Notice 94-07 referencing ASTM D 1888-78 "Standard Test Methods for Particulate and Dissolved Matter, Solids, or Residue in Water" infers that all licensed material passing through a 0.45 micron filter is considered soluble for the purposes of releasing licensed material into the sanitary sewer system and that the mentioned filtering system can be used to demonstrate solubility of radioactive liquid effluent discharges into the sanitary sewer system.

☐ Action Required:

Determine if passing radioactive liquid effluents through a 0.45 micron filter is an adequate methodology to insure compliance with 10 CFR 20.2003(a)(1), licensed material is readily soluble in water.

☐ Recommended Action (with revisions): ☐ Approve or ☐ Reject

The region believes that passing radioactive liquid effluents through a 0.45 micron filter is an adequate methodology to insure compliance with 10 CFR 20.2003(a)(1). As supporting evidence, NRC Information Notice 94-07 referencing ASTM D 1888-78 "Standard Test Methods for Particulate and Dissolved Matter, Solids, or Residue in Water" does appear to infer water passed through a 0.45 micron filter does not contain significant quantities of suspended solids. In addition, the region believes the small quantity of "suspended" licensed material identified by the licensee in the 55 gallon containers and scrubber holding reservoir combined with the 544,000 gallon dilution water from the licensee's facility into the sanitary sewer system does not pose a significant health risk to members of the general public.

Remarks:

Enclosed are the documents, in whole or in part, as stated above.

If you have any questions, please contact Michael M. LaFranzo by phone, 630-829-9865 or E-mail, address MML.

Attachment A:	Licensee letter dated February 19, 1997
Attachment B:	Licensee letter dated February 11, 1997
Attachment C:	NRC Information Notice 94-07
Attachment D:	ASTM D 1888-78 "Standard Test Methods for Particulate and Dissolved Matter, Solids, or Residue in Water"

Headquarters Reviewer: Joseph DeCicco

Regional Reviewer: Michael LaFranzo

Reviewer Code: M7L

Reviewer Phone No.: (630) 829-9865 Fax No.: (708) 515-1259

Request Needed by: 3/3/97 (date)

cc w/o att: *C. Pederson, RIII*
Inspector, RIII

Form TAR-1010/96



Attachment A

Quality Manufacturing Since 1964

P.O. BOX 1178 • MILWAUKEE, WI 53201-1178 • U.S.A. • PHONE: 414-289-9800 • FAX: 414-289-9805

February 19, 1997

page 1 of 2

Mr. John Madera
U.S. Nuclear Regulatory Commission
Region III
801 Warrenville Road
Lisle, IL 60532-4351

fax: 630 515-1259
voice: 630-829-9834

Re. Proposed Effluent Releases to the Sewer

Dear Mr. Madera:

As requested, following are the results of our sampling and testing of process and scrubber water. They were performed similar to the method discussed in APHA method 7110. Sample 14 represent a water sample taken from the scrubber holding reservoir. All number are in units of $\mu\text{Ci/ml}$.

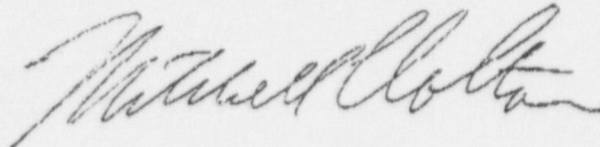
Sample	Dissolved Gross Alpha	Suspended Gross Alpha
1.	3.8E-06	2.4E-08
2.	1.5E-06	8.6E-07
3.	3.9E-06	1.2E-07
4.	5.6E-06	6.2E-07
5.	2.3E-06	7.9E-08
6.	6.3E-06	2.3E-07
7.	3.7E-06	2.3E-07
8.	4.0E-07	2.4E-07
9.	4.2E-06	3.5E-07
10.	7.4E-06	7.2E-07
11.	6.4E-06	3.0E-08
12.	6.3E-06	7.9E-08
13.	1.0E-06	3.1E-07
14.	1.4E-06	2.3E-08
MDA	3.4E-07 $\mu\text{Ci/ml}$	3.1E-09 $\mu\text{Ci/ml}$

CERAC, Incorporated

page 2

Let me know what additional information you need to resolve this matter. I look forward to your quick reply.

Sincerely,
Dr. Mitchell C. Colton
Radiation Safety Officer

A handwritten signature in cursive script, reading "Mitchell C. Colton". The signature is written in dark ink and is positioned below the typed name and title.



Attachment B

Quality Manufacturing Since 1964

P.O. BOX 1178 · MILWAUKEE, WI 53201-1178 · U.S.A. · PHONE: 414-289-9800 · FAX: 414-289-9805

February 11, 1997

page 1 of 3

Mr Michael LaFronza, Radiation Specialist
Nuclear Materials Inspection, Section
U.S. Nuclear Regulatory Commission
Region III
801 Warrenville Road
Lisle, IL 60532-4351

fax: 630-515-1259
voice: 630-829-9865

Re. Effluent Releases to the Sewer

Dear Mr. LaFronza:

Based on our recent telephone conversations, I request your approval to release our collected liquid effluent and our near term continuation as discussed below. This is for both our chemical process and our fume scrubber liquids. Longer term solutions will be discussed as we develop our plans.

The following qualifications apply:

Qualifications for Release:

1. It is sufficient to demonstrate compliance with 10 CFR 20.2003(a)(1) regarding solubility of material released to the sanitary sewer by passing all liquid effluent in question through a 0.45µ or smaller filter.
2. Total activity levels of material released to the sewer will be within release limits. In the short term, we will sample every drum until we have an agreed-upon sampling and testing protocol.

(As an example, the attached spreadsheet output represents typical activity and percent of limit for a 55-gallon drum of process water. If a sample of a 55-gallon drum has on the order of 3×10^{-5} µCi/ml or less of total activity, its release represents about 1% of our monthly limit assuming all activity is due to Th-232, which is a conservative assumption. If you assume the activity is half from each of Th-232 and Th-228, the number becomes approximately 0.6%. Including activity from any other isotopes reduces this number further.)

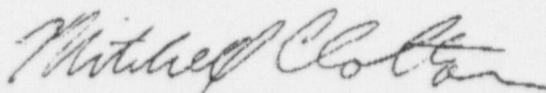
CERAC, incorporated

page 2

3. For short term verification of filter integrity, we will:
- a) Change the 0.45 μ filter at least once per month (represents about 600-1000 gallons)
 - b) or more often if determined necessary (e.g. after we process the drums we have collected)
 - c) or as we change process streams. For example, process water is typically pH of 8 or higher and scrubber water is about pH of 1-2. We would change filters before filtering one after the other.
 - d) As an additional safety measure, we will put a 0.20 μ filter after the 0.45 μ filter and pass all liquids through it as well. This 0.20 μ filter will be changed whenever the 0.45 μ filter is changed

I look forward to your quick reply on this matter.

Sincerely,



Dr. Mitchell C. Colton
Radiation Safety Officer

Calculations for RAM water disposal

3E-07 uCi/ml Th-232 monthly average release to sewer
10 CFR, pg 447, 1/1/93 (most restrictive)

6E-07 uCi/ml U-232 monthly average release to sewer
10 CFR, pg 447, 1/1/93 (most restrictive)
3E-06 is for U-nat

6E-07 uCi/ml Ra 226 monthly average release to sewer
10 CFR, pg 447, 1/1/93

1.110E-01 uCi/g Th-nat activity
6.77E-01 uCi/g U-nat activity, depleted is less
3.60E-01 uCi/g, specific activity for depleted U

water results:

pCi/l x 1E-9 = uCi/ml

each drum = 55 gal 216,216
or 208,191.81 ml

water from: 2190 ccf
4/93-7/93 1,638,120 gal
6.20E+09 ml
low estimate 2.07E+09 ml/month-avg
recent months have been higher

g/month allowable	
Th	U
5586.293	1831.842

208191.808 ml process water/barrel (=55 gal)
2.0669E+09 ml sewer water/month

Conc/Activity	Units	uCi/ml/mont	% of monthly limit
3E-05	uCi/ml	3.0218E-09	1.01%
		per 55 gal barrel	

3E-05 x 1.638 x 10⁶
2.07 x 10⁹

UNITED STATES
NUCLEAR REGULATORY COMMISSION
OFFICE OF NUCLEAR MATERIAL SAFETY AND SAFEGUARDS
WASHINGTON, D.C. 20555

Attachment C

January 28, 1994

NRC INFORMATION NOTICE 94-07: SOLUBILITY CRITERIA FOR LIQUID EFFLUENT
RELEASES TO SANITARY SEWERAGE UNDER THE
REVISED 10 CFR PART 20

Addressees

All byproduct material and fuel cycle licensees with the exception of licensees authorized solely for sealed sources.

Purpose

The U.S. Nuclear Regulatory Commission is issuing this information notice to emphasize the changes in 10 CFR Part 20 with respect to liquid effluent releases to sanitary sewerage and to encourage you to prepare for these revisions. It is expected that licensees will review this information for applicability to their operations, distribute it to appropriate staff, and consider actions to prepare for, and incorporate, these changes. Suggestions contained in this information notice are only recommendations; therefore, no specific action nor written response is required.

Background

On December 21, 1984, NRC released an information notice documenting several instances of reconcentration of radionuclides released to sanitary sewerage (IN No. 84-94, "Reconcentration of Radionuclides Involving Discharges into Sanitary Sewage Systems Permitted under 10 CFR 20.303"). Several other instances have since occurred in Portland, Oregon; Ann Arbor, Michigan; Erwin, Tennessee; and Cleveland, Ohio. The primary contributors, in some of these cases, appear to have been insoluble materials released as dispersible particulates or flakes. This issue was addressed again on May 21, 1991, by NRC, when it published its revision of Part 20 in the Federal Register (56 FR 23360), which removed insoluble non-biological material from the types of material that may be released to sanitary sewerage. Relative to this issue, the NRC Office of Nuclear Regulatory Research is conducting a study to clarify the mechanisms underlying reconcentration in sanitary sewerage and sewage treatment facilities.

9401240059

* Sanitary sewerage is defined by 10 CFR 20.1003 as "a system of public sewers for carrying off waste water and refuse, but excluding sewage treatment facilities, septic tanks, and leach fields owned or operated by the licensee [emphasis added]."

IN 94-07

January 28, 1994

Page 2 of 5

Description of Circumstances

To help prevent further reconcentration incidents at public sewage treatment facilities, 10 CFR 20.2003(d)(1), effective January 1, 1994, was written as follows:

§20.2003 Disposal by release into sanitary sewerage

(a) A licensee may discharge licensed material into sanitary sewerage if each of the following conditions is satisfied:

(1) The material is readily soluble (or is readily dispersible biological material) in water; and...

However, this revision to Part 20 did not contain an operational definition of solubility, and this precipitated many questions, from licensees, concerning how the solubility of a material may be demonstrated. Without the ability to demonstrate compliance, these licensees were unable to determine whether new procedures should be developed, new treatment systems installed, or whether they should apply for an exemption, based on the principle of maintaining all doses as low as is reasonably achievable (ALARA).

Discussion

In some of the known reconcentration incidents, the greatest reconcentrations appear to have been due to compounds released to sanitary sewerage that were not soluble. There are many approaches that may be used to determine a chemical compound's solubility in water. The following discusses two of the more common approaches:

1. Direct Determination of Compound Solubility Class. Formal Solubility, or Solubility Product (K_{sp}).

This approach would be applicable whenever there is sufficient knowledge of the chemical form of all materials contained in the liquid effluent at the point of release. With this knowledge, it would be possible to use one (or more) of the following methods:

(a) Solubility Class Determination:

The solubility class of the compound to be released could be determined directly from common literature data (e.g., *Handbook of Chemistry and Physics* - CRC Press, and *Lange's Handbook of Chemistry* - McGraw-Hill Book Company). If a compound is classified as "v s" (very soluble) or "s" (soluble), this would indicate the compound is "readily soluble." On the other hand, if it is classified as "i" (insoluble), "sl s" (slightly soluble), or "v sl s" (very slightly soluble), this would indicate materials that are "not readily soluble." Certain compounds are designated as class "d" (decompose). If the decomposed species of these compounds are classified as either "v s" or "s," this would indicate that the parent compound is "readily soluble." If these decomposed species are simple ions, such compounds (class "d") should be considered "readily soluble."

IN 94-07

January 28, 1994

Page 3 of 5

(b) Solubility Product (K_{sp}) Determination:

The solubility product constant of the compound could also be used to determine if a compound is readily soluble in water. The solubility product constant, K_{sp} , for a strong electrolyte $M_m A_a$, is expressed as:

$$K_{sp} = [M]^m [A]^a$$

where $[M]$ and " m " are the ionic concentration (mole/liter) and the number of moles, respectively, of the dissolved cation; and $[A]$ and " a " are the ionic concentration and the number of moles, respectively, of the dissolved anion.

For a simple electrolytic compound, with one mole of a dissolved cation species and one mole of a dissolved anion species, a K_{sp} greater than $1.00 \text{ E-05 mole}^2/\text{liter}^2$ would indicate that a compound is "readily soluble." For other compounds with more complex dissolution reactions (i.e., more than one mole dissolved for each species and/or more anionic or cationic species present in the dissolved products), the K_{sp} constant would increase exponentially, based on the number of moles and/or the number of dissociated species. For example, if three moles are present (two for the anion and one for the cation), the unit of K_{sp} would be $\text{mole}^3/\text{liter}^3$, and the corresponding K_{sp} would be $(1 \text{ E-05})^{3/2}$ or $3.2 \text{ E-08 mole}^3/\text{liter}^3$; the same principle could be applied for more complex dissolution reactions.

(c) Formal Solubility Determination:

Compound solubilities (g/100 ml or mole fraction per 100 ml) are also listed in the chemical literature. From a review of general scientific literature, "formal solubilities" greater than 0.003 mole/liter would indicate that a compound is "readily soluble."

** The general relation between the formal solubility, S_f , and the solubility product, K_{sp} , of a strong electrolyte $M_m A_a$ in water is given by:

$$S_f = \sqrt[m+a]{\frac{K_{sp}}{m^m a^a}}$$

where K_{sp} is the solubility product, $[M]$ is the molar concentration of the metal ion (cation), $[A]$ is the molar concentration of the anion, " m " is the number of moles of dissolved cation per mole of dissolved substance, and " a " is the number of moles of the dissolved anion per mole of dissolved substance.

For further discussion on the determination of solubility products and formal solubility, refer to Chapter 6, "Precipitation and Dilution," from Water Chemistry, by Vernon L. Snoeyink and David Jenkins (John Wiley and Sons: 1983) or texts relating to physical and/or analytical chemistry.

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Formal solubilities less than 0.003 mole/liter would indicate compounds that are "not readily soluble."

It should be pointed out that all values mentioned above (e.g., solubility class, formal solubility, and solubility product) correspond to measurements taken under standard conditions (e.g., 25°C, 101.3 kPa, pH of 7, and E_h of 0).

2. Filtration and Radiometric Analysis of Suspended Solids

This approach may be used if knowledge of the chemical form of all materials contained in the liquid effluent at the point of release is incomplete. It is most applicable when releases are made in a batch mode. This approach involves the use of standard laboratory procedures to test representative samples of the waste stream for the presence of suspended radioactive material.

The following two laboratory procedures were developed specifically to determine the suspended solids content of water: ASTM Method D 1888-78, "Standard Test Methods for Particulate and Dissolved Matter, Solids, or Residue in Water," and the American Public Health Association's Method 7110, "Gross Alpha and Gross Beta Radioactivity (Total, Suspended, and Dissolved)" from Standard Methods for the Examination of Water and Wastewater. It should be noted that ASTM Method D 1888-78 was developed to measure the total suspended solids content of water, not just the radioactive portion. In either case, activity in the suspended solids portion of effluent greater than that found in similarly processed background water samples would indicate the presence of insoluble radioactive material.

Whether one of the above approaches or a self-developed alternative is used, it is a good health physics practice to document this approach in the form of a procedure. Procedures such as these usually include provisions for the documentation of any models, calculations, analytical measurements, and/or quality control measures used. This information is usually maintained with the applicable release records, to demonstrate that the developed procedure will ensure compliance with the regulations.

If material to be released would not qualify as being "readily soluble," 10 CFR 20.2003(a)(1) would prohibit release to sanitary sewerage unless an exemption has been granted. Exemptions will be judged on a case-by-case basis, when it is demonstrated that release to sanitary sewerage is in accordance with the ALARA principle, consistent with applicable regulations, and in the public interest.

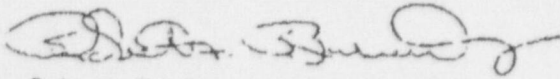
It is expected that licensees will review this information for applicability to their operations, and consider actions, as appropriate to their licensed activities. However, suggestions contained in this information notice are not NRC requirements; therefore, no specific action nor written response is required.

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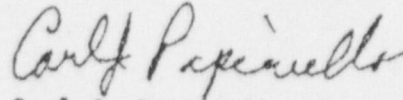
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If you have any questions about the information in this information notice, please contact one of the technical contacts listed below or the appropriate regional office.



Robert F. Burnett, Director
Division of Fuel Cycle Safety
and Safeguards
Office of Nuclear Material
Safety and Safeguards



Carl J. Paperiello, Director
Division of Industrial and
Medical Nuclear Safety
Office of Nuclear Material
Safety and Safeguards

Technical contacts: Rateb (Boby) Abu-Eid, NMSS
(301) 504-3446

Cynthia G. Jones, NMSS
(301) 504-2629

Attachments:

1. List of References
2. List of Recently Issued NMSS Information Notices
3. List of Recently Issued NRC Information Notices

Attachment 1
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REFERENCES

Annual Book of ASTM Standards. Volume 11.01, "Water (I)." American Society for Testing and Materials, Easton, MD, 1989.

CRC Handbook of Chemistry and Physics. CRC Press, Inc., Boca Raton, FL, 65th ed, 1984.

Lange's Handbook of Chemistry. McGraw-Hill, Inc., New York, NY, 13th ed, 1985.

Snoeyink, Vernon L. and David Jenkins, Water Chemistry. John Wiley & Sons, Inc., New York, NY, 1980.

Standard Methods for the Examination of Water and Wastewater. American Public Health Association, Washington, DC, 17th ed, 1989.

Designation: D 1888 - 78¹

Standard Test Methods for PARTICULATE AND DISSOLVED MATTER, SOLIDS, OR RESIDUE IN WATER¹

This standard is issued under the fixed designation D 1888; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

¹NOTE—Section 2 was added editorially and subsequent sections renumbered in January 1985.

1. Scope

1.1 These test methods cover the determination of particulate, dissolved, and total matter, sometimes referred to as the suspended, dissolved, and total solids, in water. Two procedures consistent with the total matter content, are provided as follows:

	Sections
Method A—Particulate and Dissolved Matter in Water with More Than 25 ppm of Total Matter	7 to 13
Method B—Particulate and Dissolved Matter in Water with 25 ppm or Less of Total Matter (Automatic Evaporation)	14 to 20

1.2 The test methods actually cover the determination of (1) the constituents of water that can be removed by filtration, and (2) the residue on evaporation to dryness of either filtered or unfiltered samples; as a result, they do not always measure water components as defined. Separation of particulate matter by filtration requires precise definition of the filtering medium since some materials that are in no sense dissolved, for example, certain colloids, may not be removed by the filter used. Secondly, an evaporation residue will usually differ in composition from the particulate and dissolved matter present in the water.

1.3 When particulate matter is determined separately (the sample is filtered and the residue quantitatively assessed), provision is made for the use of either a membrane filter that will remove all particles over 0.45 μ m in size or an asbestos fiber medium in a Gooch crucible. However, unless otherwise specified when results are reported, use of the membrane filter shall be

assumed. It is further provided that all buoyant floating particles or large particulate agglomerations that cannot be dispersed throughout the sample by vigorous shaking need not be considered as fundamental constituents of the water under examination and may be excluded, therefore, from the test portion.

1.4 The test methods include steps for the determination of volatile matter in the dry residue from either filtration or evaporation. They do not, however, cover water constituents that are (1) volatile at the boiling temperature, or (2) normally classified as "oily matter," which is extractable with organic solvents or volatile at the drying temperature of filtration residues. For the determination of the latter, refer to Test Method D 2778.

2. Applicable Documents

2.1 ASTM Standards:

- D 1066 Practice for Sampling Steam²
- D 1129 Definitions of Terms Relating to Water²
- D 1192 Specification for Equipment for Sampling Water and Steam²
- D 1193 Specification for Reagent Water²
- D 2778 Test Method for Solvent Extraction of Organic Matter from Water³
- D 3370 Practices for Sampling Water²

¹ These test methods are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved April 28, 1978. Published August 1978. Originally published as D 1888 - 61 T. Last previous edition D 1888 - 67 (1974).

² Annual Book of ASTM Standards, Vol 11.01.

³ Discontinued; see 1983 Annual Book of ASTM Standards, Vol 11.02.

Definitions

3.1 The terms *particulate matter*, *dissolved matter*, *total matter*, and others related to water constituents determined in these test methods, are defined in accordance with Definitions D 1129 as follows:

3.1.1 *particulate matter*—that matter, exclusive of gases, existing in the nonliquid state which is dispersed in water to give a heterogeneous mixture.

3.1.2 *dissolved matter*—that matter, exclusive of gases, which is dispersed in water to give a single phase of homogeneous liquid.

3.1.3 *total matter*—the sum of the particulate and dissolved matter.

3.1.4 *volatile matter*—that matter that is changed under conditions of the test from a solid to a liquid state to the gaseous state.

3.1.5 *fixed matter*—residues remaining after ignition of particulate or dissolved matter, or both.

3.2 For definitions of other terms used in these test methods, refer to Definitions D 1129.

Interferences

4.1 Some evaporation residues readily absorb moisture; rapid weighing is essential to this method. Some residues contain materials, such as ammonium carbonate, that decompose at low 103°C; others contain liquids, such as glycol and sulfuric acid, that will remain as a liquid residue at 103°C with or without solution of salts at might also be present.

4.2 Rapid weighing of ignited residues, also, is important because of possible moisture absorption. Furthermore, there is likelihood of interference from carbonates, organic matter, nitrite and nitrate nitrogen, water of hydration, chlorides, and sulfates which may be decomposed either completely or in part when ignited at 600°C. No single temperature is known that will eliminate these interferences. Reasonably reproducible results should be obtained, however, at the prescribed 600°C.

4.3 Because the water being sampled is of necessity in contact with the sample container and tubing, it is important, especially in the case of glass, that the possible precipitation of cations and the absorption of substances originally present in the water, on these surfaces, be recognized.

Purity of Reagents

5.1 Reagent grade chemicals shall be used in

all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193. Referee grade reagent water shall be used for Method A and the nonreferee grade for Method B.

5.3 Except for concentrated hydrochloric acid (HCl, sp gr 1.19), reagents including reagent water should be membrane-filtered prior to use.

6. Sampling

6.1 Collect the sample in accordance with the applicable ASTM standard, as follows: Practice D 1066, Specification D 1192, and Practices D 3370.

6.2 Because of the low concentration of total matter in some waters and the possible effects of aeration on others, sampling shall be carried out in a manner which reduces atmospheric exposure to a minimum. The type and size of the container shall be consistent with the nature of the water being sampled (see 16.1 and 19.1).

6.3 Samples containing 25 ppm or less of total matter on which only the total matter content is to be determined shall be immediately acidified with 0.2 mL of concentrated HCl (sp gr 1.19)/L of water to prevent iron deposition on the walls of the container. If particulate matter is to be separately determined, the sample, regardless of total matter content, shall be filtered as soon as possible (see 18.3) and then acidified.

METHOD A—PARTICULATE AND DISSOLVED MATTER IN INDUSTRIAL WATER WITH MORE THAN 25 PPM OF TOTAL MATTER

7. Scope

7.1 This method is primarily applicable to water that will yield a residue on evaporation at 103°C of at least 25 mg/L.

⁴—Reagent Chemicals, American Chemical Society Specifications, Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

8. Summary of Method

8.1 Total matter is determined by evaporation of an appropriate aliquot, or the particulate and dissolved matter are separated by filtration and individually assessed. The particulate matter is dried and weighed. The dissolved matter is determined by weighing the residue obtained after evaporating the filtered sample. Volatile matter and fixed matter under any of the above classifications are determined by weighing the residues remaining after ignition at a temperature of 600°C.

9. Apparatus

9.1 *Sample Reservoir*—A chemical-resistant container of 1 to 4-L capacity.

9.2 *Membrane Filter Assembly*—See 16.4.

9.3 *Glass Petri Dish*, 150-mm diameter.

9.4 *Filter Crucible*—See 16.6.

9.5 *Evaporating Dish*—A straight-wall or round-bottom platinum dish of 80 to 100-mm diameter and approximately 200-mL capacity. A porcelain dish may be substituted for the platinum dish if the residue is not to be analyzed.

9.6 *Heater*—A controlled electric hot plate, infrared lamp, or steam bath for maintaining the temperature of the evaporating sample near the boiling point.

10. Reagents

10.1 *Chloroform or Benzene*, purified or USP grade.

10.2 *Ethyl Alcohol* (95 %).

NOTE 1—Specially denatured ethyl alcohol conforming to Formula 3A or 30 of the U.S. Bureau of Internal Revenue may be substituted for ethyl alcohol (95 %).

10.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

11. Procedure

11.1 Weigh a quantity of sample sufficient to yield on evaporation approximately 25 mg of residue if only the amount is to be determined, or at least 100 mg if this residue is to be analyzed. The sample shall be well shaken before removing the aliquot and inclusion of floating material or agglomerates that cannot be dispersed shall be avoided. If only total matter is to be determined, without classification, proceed in accordance with 11.3. If both particulate and dissolved matter are to be determined, proceed in accordance with 11.2.

11.2 *Particulate Matter*—This determination shall preferably be made using a membrane filter following the procedure given in 18.3.1, except that a 0.2-mg residue from the solvent washings shall be permissible. The less desirable alternative use of an asbestos fiber medium is described in 18.3.2. In either case the filtrate shall be immediately acidified with 0.2 mL of HCl (sp gr 1.19)/L of water unless the sample contains significant amounts of alkaline chemicals, for example, sodium hydroxide (NaOH), whose composition would be affected by the acid; acidification will prevent deposition of iron on the sample container.

11.3 *Total Matter and Dissolved Matter*—Transfer the sample aliquot provided for total matter determination (11.1) or the filtrate obtained from the particulate matter separation (11.2) to a sample reservoir having a valve-controlled outlet. Fill an evaporating dish that previously has been ignited at $600 \pm 25^\circ\text{C}$ for 1 h, cooled in a desiccator, and weighed, to within approximately $\frac{1}{4}$ in. (6.3 mm) of the top with the sample from the reservoir. Heat the dish to evaporate the sample, but do not allow the sample to boil. Periodically add sample from the reservoir to the dish to prevent drying until the reservoir is empty. Rinse the reservoir several times with water, adding the rinsings to the contents of the evaporating dish. Then evaporate the remainder of the material in the dish to near dryness. Transfer to a 103°C oven and complete the evaporation. Dry the dish and its contents for 1 h (see 4.1), cool in a desiccator, and weigh. Repeat the cycle of drying (1-h periods), cooling and weighing until loss in weight is no more than 4 % of the previous weight. Record the weight of residue as "weight of total matter" (or, if the sample had been filtered, "dissolved matter"). Ignite the dish contents for 30 min at $600 \pm 25^\circ\text{C}$, cool in a desiccator, and reweigh (see 4.2). Record the loss in weight as "weight of volatile matter" (or "volatile dissolved matter") and the weight of the ignited residue as "weight of fixed matter" (or "fixed dissolved matter").

12. Calculation

12.1 Calculate the result of each specific determination in parts per million, as follows:

$$\text{Matter, ppm} = (W_x/V) \times 1000$$

where:

W_x = W_1 = grams of total matter found,

W_2 = grams of particulate matter found,

W_3 = grams of dissolved matter found,
 W_4 = grams of volatile matter found,
 W_5 = grams of volatile particulate matter found,
 W_6 = grams of volatile dissolved matter found,
 W_7 = grams of fixed matter found,
 W_8 = grams of fixed particulate matter found, or
 W_9 = grams of fixed dissolved matter found, and
 = litres of sample used.

12.2 When particulate and dissolved matter have been separately determined, total matter, volatile matter, and fixed matter can be calculated by adding the two appropriate values.

12.3 If asbestos fiber filtration was used for the removal of particulate matter, it is mandatory that this be stated when reporting either particulate or dissolved matter. Otherwise, use of a membrane medium shall be assumed.

13. Precision

13.1 No statement can be made concerning the precision of this method. The precision is influenced by both the nature and the amount of entrained matter and by the effects of drying and ignition on its actual composition.

METHOD B—PARTICULATE AND DISSOLVED MATTER IN INDUSTRIAL WATER WITH 25 PPM OR LESS OF TOTAL MATTER (AUTOMATIC EVAPORATION)

14. Scope

14.1 This method is intended primarily for steam condensate and distilled or demineralized water that contains 5 ppm or less of total matter. Because of the automatic evaporation feature, the method is desirable for use, however, on all waters containing up to 25 ppm of total matter, particularly if a large residue is desired for chemical analysis.

15. Summary of Method¹

15.1 Total matter is determined by evaporation, or the particulate and dissolved matter are separated by filtration and individually evaluated. The particulate matter is dried, freed of oily matter by extraction, dried again, and weighed. The solution of dissolved matter is evaporated to dryness using a dish provided with a constant-level control. Sufficient sample is evaporated to give the desired accuracy for the measurement and provide ample material for other analytical

requirements. The residue is dried and weighed. Volatile matter in any of the three classifications is subsequently removed by ignition. The total, particulate, dissolved, volatile, or fixed matter are then calculated from the various weights obtained.

16. Apparatus

16.1 *Sample Reservoir*—A covered, 20-L (5-gal) container of corrosion-resistant metal, suitable plastic, or chemical-resistant glass with necessary tubular connections. Most waters with very low total matter exhibit a pH in the range from 6 to 9. For samples of such waters, containers of TFE-fluorocarbon, block tin, polyethylene, or chemical-resistant glass shall be selected with that order of preference, depending upon the purity.

16.2 *Automatic Evaporation Assembly*—A dust shield, constant-level device, heater, and evaporation dish. Typical assemblies are described in 16.2.1 and 16.2.2.

16.2.1 Evaporation Assembly A (Fig. 1):

16.2.1.1 *Dust Shield*—A heat-resistant cover glass enclosing the Monel-sheathed ring heater, platinum evaporating dish, antenna, and electrical terminal posts, with provision for introducing the water sample through the base. Minimum practicable enclosed space is necessary to prevent condensation on the cover. The top of the dust shield is covered with a "dunce cap" to prevent foreign material from dropping into the dish while permitting free passage for the moisture-laden air. An open-bottom aluminum platform supporting two filter cylinders and having an opening under the glass cover is provided to supply filtered inlet air. Either a seal must be provided or filter material used between glass cover and the platform as well as between chassis and platform.

16.2.1.2 *Evaporator Assembly*, as shown under the glass cover—The Monel-sheathed ring heater is suspended over the platinum evaporating dish by two stainless steel arms which are connected through the electronic control system to a power circuit containing a timer. The platinum dish is supported by an aluminum plate provided with leveling screws so that the distance from the dish to the heater can be adjusted. A stainless steel inlet tube is used for addition of sample at the pouring spout of the platinum vessel.

16.2.1.3 Electronic Control Circuit—Control

of the water level in the platinum dish is effected by a capacitance-type electrode or antenna which can be made conveniently of a flat coil of platinum wire (16 to 20-gage). The antenna is suspended from a stainless steel arm which makes contact with the electric control circuit³ through a terminal post. A change of the water level activates the shut-off valve⁶; if the water level in the platinum dish does not return to the upper level control within 45 s after reaching this lower level of capacitance control, the current to the ring heater is broken by means of a time interlock. The purpose of this interlock is to prevent the drying of the dish at a temperature above the specified 103°C (217°F) level, should additional sample fail to reach the dish. Since the 45-s timer automatically turns off the heater when sample flow is interrupted, an additional timer is incorporated which may be used upon completion of evaporation to keep the heater on for a specified time period to lower the water level in the dish and thus facilitate its removal from the test assembly. An overflow device is incorporated in the assembly, also. A platinum wire electrode is positioned so that its tip is suspended slightly above the normal water level in the platinum dish. This electrode serves as an additional upper-water level control should a failure occur in the capacitance system.

16.2.2 Evaporation Assembly B (Figs. 2, 3 and 4):

16.2.2.1 *Dust Shield*—The dust shield compartment consists of a heat-resistant glass bell jar equivalent to that used on assembly "A" and is contained in an enclosed dust-shielded compartment. Air is provided through an external filter source into this shielded sample compartment.

16.2.2.2 *Evaporator Assembly*—The evaporator assembly as shown schematically in Fig. 4 consists of a balance,⁷ one arm of which extends into the dust shield compartment. The balance arm extending into the dust shield holds a platinum sample dish. Also extending into this compartment from the balance base and mounted in the dust shield compartment is a heater connection consisting of the necessary wiring connections and a Monel-sheathed ring heater similar to that used in evaporator assembly A. In addition, a solenoid water sample valve⁸ is provided with a 1/4-in. (3.2-mm) outside diameter stainless steel tubing connection feeding into the shielded sample compartment; and then to the platinum sample dish. Automatic sample addition is ac-

complished by a level switch on the counter balance arm and this actuates the water sample valve. Control effected by counter balance arm can be dampened by a dash pot. If desired, a timer mechanism can be installed to record the volume of water evaporated. Calibration of this assembly is accomplished by using a calibrated sample reservoir and timing the addition and evaporation rate. This calibration will have to be carried out under atmospheric conditions similar to those pertaining at the actual sampling location.

16.2.2.3 *Wiring Diagram*—The wiring diagram for this assembly is also shown in Fig. 3.

16.3 *Sampling Device* (see Fig. 5)—A cooling coil with overflow pipe and solenoid valve suitable for sampling from a water source to a continuous sample evaporator. (The cooling coil, of course, necessary only when sample is at room temperature.)

16.4 *Membrane Filter Assembly*—A borosilicate glass or stainless steel funnel with a flat, fritted base of the same material, and membrane filters (0.45- μ m pore size) to fit.⁸

16.5 *Glass Petri Dish*—150-mm diameter.

16.6 *Filter Crucible*—A Gooch crucible containing an evenly distributed filter mat, approximately 5-mm thick and composed of finely divided asbestos fiber, produced by pouring a slurry of acid-washed asbestos into the crucible under slight suction.

16.7 *Evaporating Dish*—A straight-walled or round-bottom platinum dish of 80 to 100-mm diameter and approximately 200-mL capacity.

17. Reagents

17.1 See Section 10.

¹ The RCA Thermocap Relay Unit manufactured by the Niagara Electron Laboratories, Niagara Falls, NY, or equivalent, has been found satisfactory for this purpose.

² The electrically operated valve (No. 5004141312) sold by Diamond Power Specialty Corp., Lancaster, OH, or an air-operated valve (No. 1000A 2-way Demi G 303 with No. 5049 stainless diaphragm) manufactured by the G. W. Dahl Co., Inc., Bristol, RI, have been found satisfactory for this purpose. The Dahl valve must be coupled with a solenoid air valve such as the Skinner Electric Valve, 3-way, vented, No. VSD4200 manufactured by Skinner Electric Valve Div., The Skinner Chuck Co., 100 Edgewood Ave., New Britain, CT, or its equivalent. It is imperative that new valves be tested to determine that contamination does not occur from mechanical wear on materials of construction.

³ The balance manufactured by the Fisher Scientific Co., Catalog No. 2-035, or its equivalent, has been found satisfactory for this purpose.

⁸ Suitable membrane filter holder and filters. HAW-PO4700, are available from Millipore Co., Bedford, MA.

18. Procedure

18.1 Select a volume of sample sufficient to yield an evaporation residue of approximately 25 mg if only the matter content is to be determined, or approximately 100 mg if the evaporation residue is to be analyzed.

18.2 If both particulate and dissolved matter are to be determined, proceed in accordance with 18.3; if only total matter is desired, follow the procedure starting with 18.4.

18.3 *Particulate Matter*—This water component is preferably separated by filtration using a membrane having a pore size of 0.45 μm (see 18.3.1); an alternative procedure using an asbestos fiber medium, generally considered to have a 5- μm pore size, is described in 18.3.2.

18.3.1 *Membrane Filtration*—Place $n + 1$ plain, white filter disk of the prescribed pore size in a 150-mm petri dish, where n equals the number of tests to be run. Place the dish and filters in a drying oven at 103°C for 15 min or in a vacuum desiccator for 30 min. If oven-dried, allow the filters to cool to room temperature while exposed to the air. Weigh each filter to the nearest 0.1 mg. With most balances it is desirable to have a polonium alpha emitter source to dispel effects of static electricity. Label filters with ballpoint pen and mark the extra filter C for "control." Proceed with the filtration in accordance with 18.3.1.1 through 18.3.1.5.

18.3.1.1 Place a weighed filter on the fritted base of the filter holder, and clamp the funnel portion of the apparatus in place on top of the filter. Place the filtration assembly on a filter flask of appropriate size and with the aid of a vacuum from a vacuum-pressure pump or water aspirator, pour the sample into the funnel and draw through the filter into the filter flask. Where sample bottles are employed for collection of the sample, the entire contents of a sample bottle should be filtered. Wash the bottle with an appropriate quantity of filtered water (may be obtained from the filter flask) and pour this also into the filter funnel. Transfer sample and washings to sample reservoir. Dry the membrane by drawing air through the filter and wash with chloroform or benzene until 10 mL of the washings leave not more than 0.1 mg of residue on evaporation at 103°C. Air-dry the sediment for several minutes. Discard the washings. Release the vacuum and with flat-bladed forceps, remove the filter from the fritted base and place in the petri dish.

18.3.1.2 Wet the control filter (C) with the sample water from the filter flask, and place it also in the petri dish.

18.3.1.3 Place the petri dish in the drying oven at 103°C for 30 min; allow the filters to cool to room temperature and equilibrate to ambient humidity after removing from the oven, and reweigh.

18.3.1.4 Record the weight of particulate matter adjusted for the difference between final and initial weight of the test filter as "weight of particulate matter." Make a positive or negative adjustment in the event of any weight change occurring in the "control" filter.

18.3.1.5 Place the filter used in the particulate matter determination in a clean, ignited, small, porcelain crucible, which has been weighed, after ignition and cooling, to the nearest 0.1 mg. Add approximately 1 mL of ethyl alcohol and ignite with a match when the filter is fully wetted. After the alcohol has burned off, place the lid on the crucible and ignite it in the furnace at $600 \pm 25^\circ\text{C}$ for at least 30 min. Remove the crucible from the furnace and allow it to cool to room temperature in a desiccator. Remove the crucible cover and weigh the crucible to the nearest 0.1 mg (see 4.2). Record the loss in weight as "weight of volatile particulate matter" and the weight of the ignited residue as "weight of fixed particulate matter."

18.3.2 *Asbestos Fiber Filtration* (Note 2)—Filter the selected volume of sample through a filter crucible (see 16.6) that previously has been dried for 1 h at 103°C, cooled in a desiccator, and weighed. After filtration, wash the filter crucible contents twice with water, transferring the filtrate and washings to the sample reservoir for subsequent determination of dissolved matter as described in 18.4. Dry the crucible contents by drawing air through the crucible for several minutes; then wash the crucible contents with chloroform or benzene until 10 mL of the washings leave not more than 0.1 mg of residue on evaporation at 103°C. Discard the washings. Air-dry the sediment for several minutes; then place the crucible in an oven at 103°C for 1 h, cool in a desiccator, and weigh. Record the weight of the residue as "weight of particulate matter." Ignite the crucible contents for 30 min at $600 \pm 25^\circ\text{C}$, cool in a desiccator, and reweigh (see 4.2). Record the loss in weight as "weight of volatile particulate matter" and the weight of the ignited residue as "weight of fixed particulate matter."

NOTE 2—Since asbestos fiber filters are generally considered to have a pore size of 5 μm , no process of coagulation shall be employed that will alter the content of either dissolved or particulate matter.

18.3.3 Immediately acidify the filtrate and washings with 0.2 mL of HCl (sp gr 1.19)/L of water.

18.4 *Total Matter and Dissolved Matter*—Weigh a platinum dish that has been dried for 1 h at 103°C and cooled in a desiccator. Using Evaporation Assembly A or B, start the evaporation of the selected volume of sample for total matter only or the filtrate and washings from the particulate matter removal (see 18.3), as follows:

18.4.1 *Evaporation Assembly A*—With the current off, insert the clean, weighed platinum dish (previously ignited at $600 \pm 25^\circ\text{C}$) in the evaporator assembly and adjust the dish height by use of the leveling screws in the aluminum base. Antenna adjustments, if necessary, may also be made at this time, using the set screw provided at the end of the antenna arm. Turn on the circuit and heater switches and set the control knob on the relay to allow water to rise to the proper level with respect to the antenna and the desired water level in the dish. It is advisable to operate the relay with the water level as close to the antenna as possible. Observe the evaporator for a period of time to ascertain the satisfactory operation of the relay and assure the absence of boiling in the dish.

18.4.2 *Evaporation Assembly B*—Adjust the apparatus to the most rapid rate feasible without boiling. The adjustment is accomplished by the

addition of 7 to 10-g weights to the tare weight of the evaporating dish on the counter weight arm of the balance. With the top of the shielded compartment removed, manually trip the level switch on the counter balance arm several times to actuate the water sample valve and flush out the sampling line. Then place a carefully cleaned and weighed platinum dish on the balance pan in the shielded compartment. Place a clean bell jar carefully on the seal mounted on the support plate in the dust shielded compartment (Fig. 4). Close the shield compartment leaving the vent open. Turn on the heater current and observe the operation long enough to assure satisfactory performance.

18.4.3 When evaporation is almost complete, remove the dish from the assembly, transfer to a 103°C oven, and heat to dryness. Continue heating for 1 h, cool in a desiccator, and weigh (see 4.1). Record the weight of the residue as "weight of total matter" (or, if sample had been filtered, "dissolved matter"). Ignite the dish and contents for 1 h at $600 \pm 25^\circ\text{C}$, cool in a desiccator, and reweigh (see 4.2). Record the loss in weight as "weight of volatile matter" (or "volatile dissolved matter"), and the weight of the ignited residue as "weight of fixed matter" (or "fixed dissolved matter").

19. Calculation

19.1 See Section 12.

20. Precision

20.1 See Section 13.

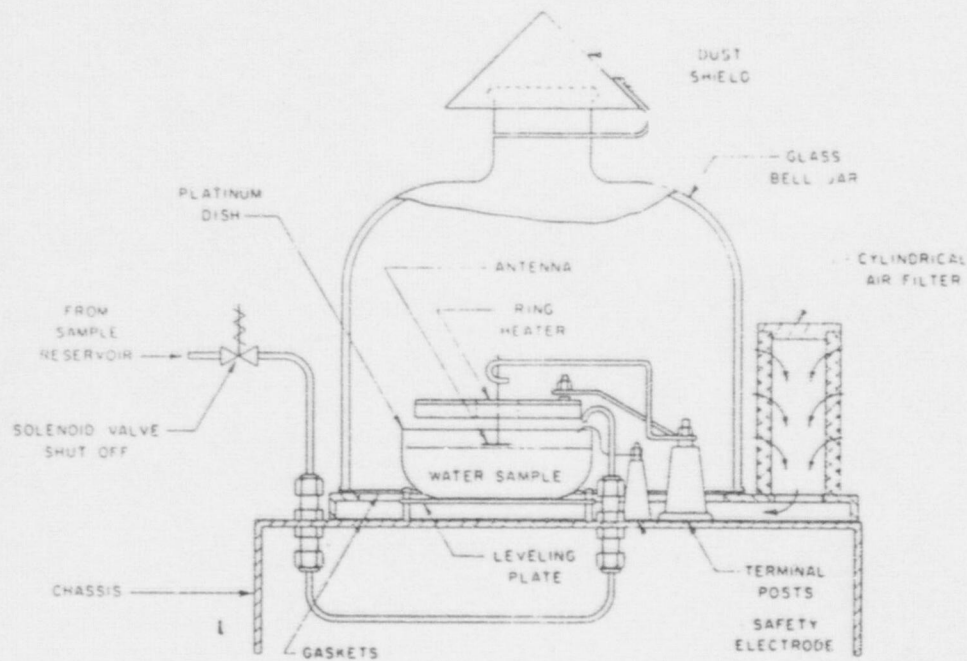
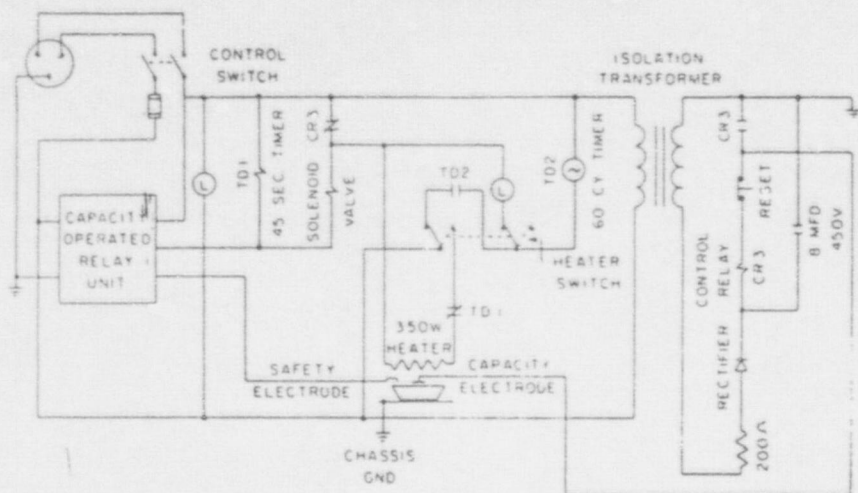


FIG. 1 Evaporation Assembly A

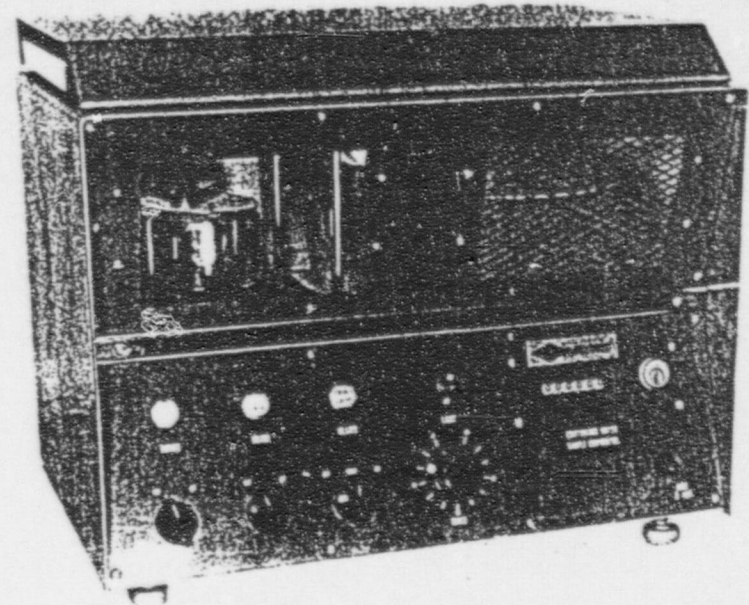


FIG. 2 Evaporation Assembly B

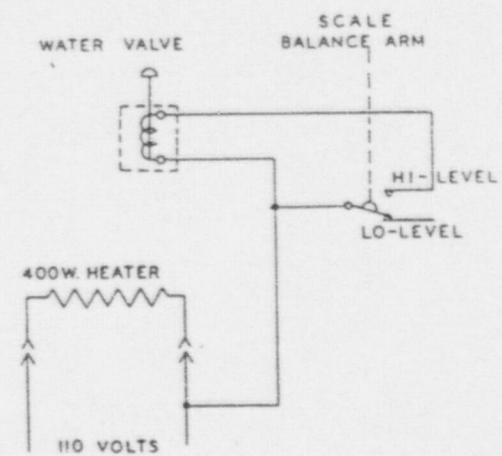


FIG. 3 Automatic Evaporation Circuit

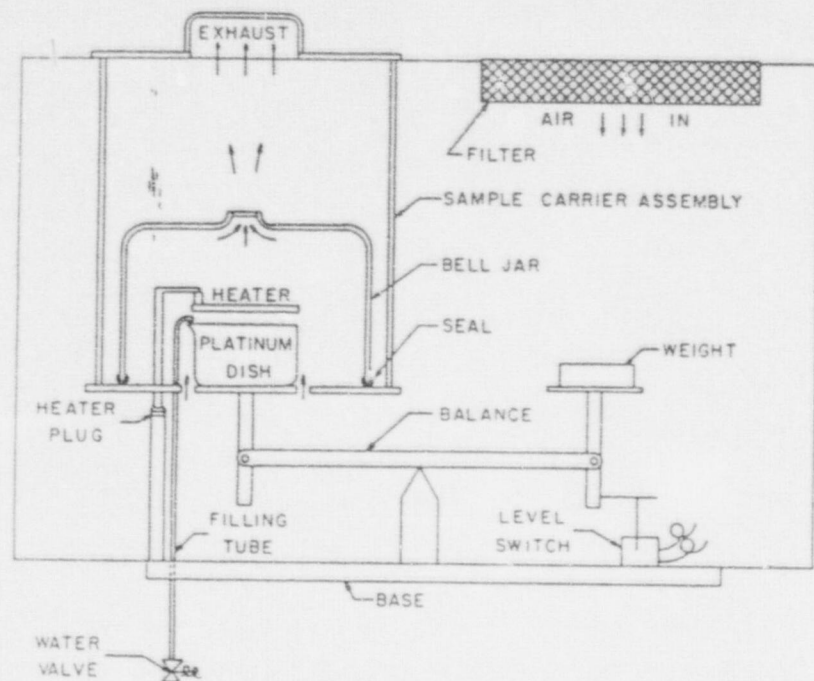


FIG. 4 Automatic Evaporation Assembly

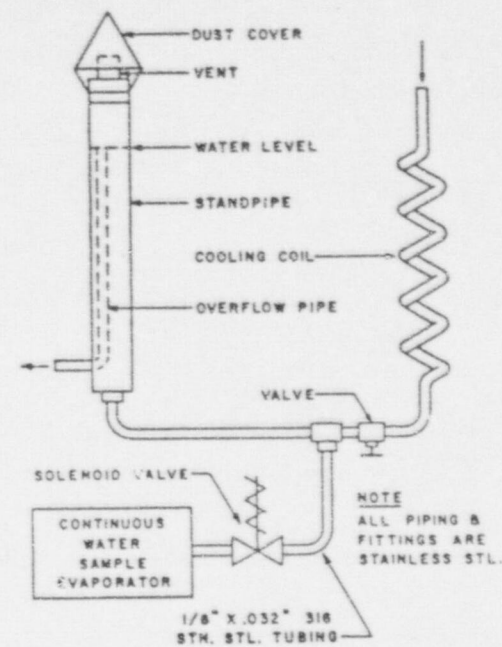


FIG. 5 Automatic Evaporator Sampling Equipment

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March 3, 1997

Mitchell C. Colton, Ph.D.
Radiation Safety Officer
Cerac, Inc.
P. O. Box 1178
Milwaukee, WI 53201

SUBJECT: ACKNOWLEDGEMENT OF CORRESPONDENCE
(Letter Dated 02/26/97)

Dear Licensee:

In response to your request, we have completed the initial processing, which is an administrative review of your application for a(n):

☐ New License ☒ Amendment ☐ Renewal
☐ Termination ☐ Auth User (Amendment not required)
☐ Other _____

No administrative deficiencies were identified during this initial review. However, it should be noted that a technical review may identify omissions in the submitted information.

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