

UNCONTROLLED

WASHINGTON PUBLIC POWER SUPPLY SYSTEM

PLANT PROCEDURES MANUAL

WNP- 2

PROCEDURE NUMBER *12.10.5	APPROVED <i>J. Martin</i>	DATE 06/21/83
VOLUME NAME 12 CHEMISTRY PROCEDURES		
SECTION 12.10 POST ACCIDENT SAMPLING PROCEDURES		
TITLE *12.10.5 GAS SAMPLE ANALYSIS		

12.10.5.1 Purpose

This procedure determines gas concentrations and activity from a sample taken from the post accident sample station.

12.10.5.2 Precautions/Prerequisites

- A. Lab personnel shall be issued extremity dosimetry as per Health Physics policy.
- B. Lead shielding must be setup prior to sample handling.
- C. The gamma spectrometer system must be operable.
- D. The gas chromatograph must be calibrated (see 12.10.5.5) prior to analysis.
- E. The gas chromatograph must be operating 24 hours prior to analysis.
- F. Carrier gas flow must be approximately 20cc/min (20 psi).
- G. All sample handling must be carried out in a hood.
- H. Appropriate dose rate meter must be available and in calibration. Use continuously to assure exposure ALARA.

12.10.5.3 Equipment

- A. 1.0 ml and 0.1 ml gas tight syringes
- B. Oxygen, hydrogen and nitrogen gas standards
- C. 14 ml and 125 ml serum vials

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#### 12.10.5.4 Procedure

- A.. On HP3390 integrator enter scale setting:
  - 1. OP ( ) 6
  - 2. Scale setting
- B. Open valve on gas tight syringe.
- C. Insert 1.0 ml syringe needle into sample part and withdraw syringe plunger to 1.0 ml.
- D. Allow a few seconds for pressure to equalized across the needle.
- E. Close valve on syringe and release plunger.
- F. Note actual volume sampled and record on Attachment I.
- G. Withdraw syringe from sample.
- H. Insert syringe into septum on the gas chromatograph.
- I. Open valve on syringe and inject sample.
- J. If not already depressed, press the AUTO button then press clear.
- K. Read percent concentration from HP3390 integrator and calculate percent concentration in containment as per Attachment I.

#### 12.10.5.5 Gamma Analysis

- A. From sample dose rate determine what dilutions are needed to reduce sample dose rate to less than 5 mrem at 2 inches. Record dilutions on Data sheet, Attachment III.
- B. Place septums or appropriate number of serum vials and evacuate.
- C. Place vials behind lead shielding.
- D. With an appropriate syringe inject a volume of H<sub>2</sub>O equal to the desired aliquot then withdraw an aliquot and transfer to a 14 ml or 125 ml serum vial.
- E. Complete required dilutions behind the lead shield as planned. Final dilution is into a 14 ml serum vial for counting.

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- F. Bag or wrap in plastic the final dilution, smear and survey then when less than 1000 dpm/100cm<sup>2</sup> contamination, remove to counting room.
- G. Adjust sample counting time to reflect its activity as estimated by its dose rate.

#### 12.10.5.6 Calibration

- A. Using a gas tight syringe obtain a 100 micro liter sample of standard hydrogen gas.
- B. Inject into gas chromatograph.
- C. If not already depressed, press AUTO.
- D. Press CLEAR.
- E. When chromatogram is complete enter the data into the HP3390 integrator:
  - 1. Del CALIB
  - 2. CALIB ESTD
  - 3. Ref RTW = 5
  - 4. RTW = 5
  - 5. RT = (retention time for hydrogen)
  - 6. AMT = 10.0 (10.0 vol percent)
  - 7. ENTER
- F. Repeat Steps 12.10.5.5.A through 12.10.5.5.D for oxygen gas standard.
- G. Enter data into HP3390 integrator:
  - 1. EDIT CALIB
  - 2. REF RTW = 5
  - 3. RTW = 5
  - 4. CAL # = 2
  - 5. RT = (retention time for oxygen)

6.  $AMT = 10.0$  (volume percent)
  7.  $AMT/HT = (10.0 \text{ volume percent divided by peak height})$
  8. ENTER
- H. Repeat Steps 12.10.5.5.A through 12.10.5.5.D for nitrogen.
- I. Enter data into HP3390 integrator:
1. EDIT Calib
  2. REF RTW = 5
  3. RTW = 5
  4. CAL# = 3
  5. RT = (retention time for nitrogen)
  6.  $AMT = 10.0$  (volume percent nitrogen)
  7.  $AMT/HT = (10.0 \text{ volume percent divided by peak height})$
  8. ENTER
- J. Steps 12.10.5.5.A through 12.10.5.5.D can be repeated for any additional gases.
- K. If a calibration table already exists, the calibration table can be updated by injection of a standard as in Steps 12.10.5.5.A through 12.10.5.5.D then pressing RCAL. This process would have to be repeated for each gas unless a mixed gas standard is available.

#### 12.10.5.7 Documents

- A. Attachment I, Calculation of Containment Gas Concentration
- B. Attachment II, Conversion Factors
- C. Attachment III, Dilution Data
- D. Attachment IV, Final Concentration Calculation

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# Calculation of Containment Gas Concentration

Sample percent by volume concentration of each gas:

$$A_S = \frac{V_A + V_S}{V_A} A_T$$

Where:

$A_T$  = volume percent analyzed

$A_S$  = volume percent in vial before analysis

$V_A$  = volume of aliquot removed from standard

$V_S$  = volume of vial

Percent by volume of each gas in containment:

$$C_C = \frac{(A_S)(P_C)(T_V)}{(P_V)(T_C)}$$

$P_C$  = (Containment Pressure) - (Partial Pressure of Water)

$T_V$  = Vial Temperature = \_\_\_\_\_ °K

$P_V$  = Vial Pressure = \_\_\_\_\_ mm Hg

$T_C$  = Containment Temp = \_\_\_\_\_ °K

$C_C$  = Containment concentration in percent by volume = \_\_\_\_\_ %  $H_2$

\_\_\_\_\_ %  $O_2$

\_\_\_\_\_ %  $N_2$

Attachment I

## Conversion Factors

pounds/square inch to mm Hg

$$(\text{measured lb/in}^2) \frac{760 \text{ mmHg}}{14.696 \text{ lb/in}^2} = \text{mm Hg}$$

°F to °C

$$^{\circ}\text{F} = 9/5 (^{\circ}\text{C} + 32)$$

°C to °K

$$^{\circ}\text{K} = ^{\circ}\text{C} + 273$$

Attachment II

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Dilution 1

aliquot \_\_\_\_\_ cc (a)

final volume \_\_\_\_\_ cc (b)

Dilution 2

aliquot \_\_\_\_\_ cc (c)

final volume \_\_\_\_\_ cc (d)

Dilution 3

aliquot \_\_\_\_\_ cc (e)

final volume \_\_\_\_\_ cc (f)

$$\frac{b}{a} \times \frac{d}{c} \times \frac{f}{e} = \text{dilution factor (df)}$$

(measured uCi/cc)(df) =  $A_R$ , activity remaining in vial after gas chromatography analysis

$$A_i = \frac{V_A + V_S}{V_S} A_R$$

$A_i$  = initial uCi/cc in sample

$V_A$  = 1 cc

$V_S$  = volume of vial

Attachment III

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<u>Isotope</u>	<u>uCi/cc</u>	<u>df</u>	$\frac{V_A + V_S}{V_A}$	<u>Final Concentration</u> <u>uCi/cc</u>
kr85	_____	_____	_____	_____
kr88	_____	_____	_____	_____
Xe-133	_____	_____	_____	_____
Xe-135	_____	_____	_____	_____

Attachment IV

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