

Volume 08

08-S-04-14

Section 04

Revision 2

Date: 2-2-82

CHEMISTRY INSTRUCTIONSAMPLE PREPARATION FOR COUNTINGSAFETY RELATED

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## 1.0 PURPOSE

The purpose of this instruction is to provide detailed instructions for preparing samples for counting so that sample geometries will be reproducible.

## 2.0 REFERENCES

2.1 Standard Methods, 14 Edition, 560

2.2 Chemistry Procedure 08-S-03-1, Qualification of Chemistry Program

## 3.0 DEFINITIONS

None

## 4.0 PREREQUISITES

### 4.1 Apparatus Required

4.1.1 1-Liter Marinelli beaker

4.1.2 4-Liter Marinelli beaker

4.1.3 1-Liter poly bottle

4.1.4 14 ml off-gas vial

4.1.5 1 dram vial

4.1.6 Whatman filter paper, No. 542

4.1.7 Planchet

4.1.8 Liquid scintillation vial

4.1.9 Charcoal Cartridge, 81-705C-727 or equivalent

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4.1.10 Filter paper, 47mm

4.1.11 Resin paper, 47mm

4.1.12 Saran Wrap

4.1.13 Millipore Petri Dish, PD 10-047-00

4.1.14 Millipore Petri-Slide, PD 15-047-00

4.1.15 Mounting card, 3 x 2 1/2 inches

4.1.16 Tie wrap labels

4.1.17 Mylar film

4.1.18 Electrical tape or equivalent

4.1.19 Two-sided tape

4.1.20 Bag sealer

#### 4.2 Reagents Required

4.2.1 Insulin

4.2.2 Sodium Hydroxide, 1N

4.2.3 Sodium Sulfate, 100mg/ml

4.2.4 Gel-Guard or equivalent

4.2.5 Silicone grease

4.2.6 Scintillation solution

4.2.7 Acetone, reagent grade

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## 5.0 PRECAUTIONS

- 5.1 Extreme care should be exercised to keep the external surfaces of samples free of radioactive contamination.
- 5.2 Handle radioactive samples in accordance with radiological safety procedures.

## 6.0 INSTRUCTIONS

### 6.1 1-Liter Marinelli Beaker

- 6.1.1 Transfer 1.0 liter of sample to a clean Marinelli beaker. If the volume of sample is less than 1 liter, record the volume of sample and dilute to 1.0 liter with demineralized water.
- 6.1.2 If visible particles are present, the sample should be gelled with Gel-Guard or equivalent. Do not gel calibration standards or regulatory samples.
- 6.1.3 Label the Marinelli beaker lid appropriately.
- 6.1.4 Place cover on Marinelli beaker and secure with electrical tape or equivalent.
- 6.1.5 Clean the outside of the beaker to remove any contamination.
- 6.1.6 The sample is in the correct geometry for counting on a Germanium detector system.

### 6.2 4-Liter Marinelli Beaker

- 6.2.1 Transfer 3.0 liters of sample to a clean Marinelli beaker. If the volume of the sample is less than 3 liters, record the volume of the sample and dilute to 3.0 liters with demineralized water.
- 6.2.2 If visible particles are present, the sample should be gelled with Gel-Guard or equivalent. Do not gel calibration standards or regulatory samples.
- 6.2.3 Label the Marinelli beaker lid appropriately.

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- 6.2.4 Place cover on Marinelli beaker and secure with electrical tape or equivalent.
- 6.2.5 Clean the outside of the beaker to remove any contamination.
- 6.2.6 The sample is in the correct geometry for counting on a Germanium detector system.
- 6.3 1-Liter Poly Bottle
  - 6.3.1 Transfer 1.0 liter of sample to a clean 1-liter poly bottle. If the volume of the sample is less than 1 liter, record the volume of the sample and dilute to 1.0 liter with demineralized water.
  - 6.3.2 If visible particles are present, the sample should be filtered with Gel-Guard or equivalent. Do not gel calibration standards or regulatory samples.
  - 6.3.3 Cap the bottle securely and label appropriately.
  - 6.3.4 Clean the outside of the bottle to remove any contamination.
  - 6.3.5 Place the bottle in a plastic bag and seal with bag sealer or tape.
  - 6.3.6 The sample is in the correct geometry for counting on a Germanium detector system.
- 6.4 14 ml Off-Gas Vial
  - 6.4.1 The volume of individual off-gas vials are measured prior to use. The off-gas sample panel normally allows the vials to be completely filled, but when not completely filled with off-gas, the effective volume can be calculated from the following formula.

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$$V_2 = V_1 P$$

$$\frac{760}{\text{mm Hg}}$$

Where:

$V_2$  = The effective volume, ml

$V_1$  = Volume of the off-gas vial, ml

P = Pressure in filled off-gas vial, mm Hg

- 6.4.2 Use an adhesive label to label the off-gas vial with appropriate data.
- 6.4.3 Wrap the vial in Saran Wrap, twist and secure with tape. Trim the excess wrap.
- 6.4.4 The vial may also be labeled with a tie wrap label.
- 6.4.5 The sample is in the correct geometry for counting on a Germanium detector system or well counter.

#### 6.5 Charcoal Cartridge

- 6.5.1 Fission gases are removed from the charcoal cartridge by blowing clean dry air through the cartridge for 20 minutes in an operating hood.
- 6.5.2 Wrap the charcoal cartridge in Saran Wrap such that the excess wrap is on the side of the cartridge so that the cartridge will lay flat on both the inlet and outlet sides.
- 6.5.3 Secure the Saran Wrap with tape and trim the excess wrap.
- 6.5.4 The cartridge may be labeled with a tie wrap label.
- 6.5.5 The sample is in the correct geometry for counting on a Germanium detector system or well counter.

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#### 6.6 1-Dram Vial

- 6.6.1 Transfer 2 ml of sample to a clean 1 dram vial, secure the cap and clean the outside of the vial.
- 6.6.2 Use an adhesive label to label the vial with the appropriate data.
- 6.6.3 Wrap the vial in Saran Wrap, twist and secure with tape. Trim the excess wrap.
- 6.6.4 The vial may also be labeled with a tie wrap label.
- 6.6.5 The sample is in the correct geometry for counting on a well counter or Germanium detector system.

#### 6.7 Planchet

##### 6.7.1 Beta Counting

- a. Clean the planchet with reagent grade acetone. After cleaning, handle the planchet with forceps or tongs.
- b. Sparingly apply silicone grease to the edge of the planchet to prevent migration of the sample during evaporation.
- c. Transfer approximately 2 ml of sample to the planchet.
- d. Add 3 to 4 drops of insulin or NaOH (1N) to reduce the surface tension.
- e. Place the planchet in an infrared drying oven.
- f. As the sample evaporates, additional sample may be added to the planchet until all of the required sample has been transferred to the planchet. Record the total volume of liquid that is evaporated as the sample volume.



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- g. Add the appropriate volume of  $\text{Na}_2\text{SO}_4$  (100mg/ml) to the planchet to bring the total solid on the planchet to approximately 100 mg. The  $\text{Na}_2\text{SO}_4$  is added to give each sample a uniform self absorption.
- h. Evaporate to complete dryness exercising care not to overheat and splatter the sample.
- i. Remove the sample from the infrared oven and allow to cool to room temperature.
- j. The planchet should be stored in a labeled plastic Petri Dish.
- k. For counting in a beta detector, the planchet should be removed from the Petri Dish with forceps or tongs.
- l. For counting on a Germanium detector system or well counter, the planchet should be left in the plastic Petri Dish.

#### 6.7.2 Alpha Counting

- a. Clean the planchet with reagent grade acetone. After cleaning, handle the planchet with forceps or tongs.
- b. Transfer approximately 2 ml of clear sample (e.g., reactor water, demineralized water) to the planchet.
- c. Place the planchet in an infrared drying oven.
- d. As the sample evaporates, additional sample may be added to the planchet until all of the required sample has been transferred to the planchet. Record the total volume of liquid that is evaporated as the sample volume.
- e. Evaporate to complete dryness, exercising care not to overheat the sample.



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- f. Remove the sample from the infrared oven and allow to cool to room temperature.
- g. The planchet should be stored in a plastic Petri Dish.
- h. For counting on an alpha detector, the planchet should be removed from the Petri Dish with forceps or tongs.

#### 6.8 Filter Papers - 47 mm Diameter

- 6.8.1 Using forceps, transfer the 47mm filter paper (e.g. Millipore, anion paper, cation paper, particulate filter) to a plastic Petri-Slide.
- 6.8.2 Use an adhesive label to label the Petri-Slide with the appropriate information.
- 6.8.3 The sample is in the correct geometry for counting on a Germanium detector system or well counter.
- 6.8.4 For counting in a beta detector, the filter must be removed from the Petri-Slide and placed on a clean filter to prevent the beta counter from becoming contaminated.

#### 6.9 Whatman 542 Filter

##### 6.9.1 Gamma Counting

- a. Draw diagonal lines on the mounting card to facilitate centering of sample.
- b. Label the mounting card with the appropriate information.
- c. Place the Whatman 542 filter at the intersection of the diagonal lines.
- d. Cover the filter with 2 inch transparent tape.

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- e. The sample is in the correct geometry for counting on a Germanium detector system.
- f. If counting is required on the well counter, the sample card must be trimmed, rolled and placed in a 1 dram vial. Exercise care to keep the sample sealed while trimming the sample card.

#### 6.9.2 Beta Counting

- a. Label the underside of the mounting card with the appropriate information.
- b. Affix a piece of two-sided tape to the mounting card.
- c. If the sample is to undergo further chemical treatment at a later time, (i.e., SR-90 analysis) place a piece of paper on the two-sided tape.
- d. Place the Whatman 542 paper on the center of the mounting card with the precipitate side up.
- e. Cover the sample with Mylar film and seal to the two-sided tape. See Figure 1 for an illustration.

#### NOTE

Back the Mylar film with a damp tissue to eliminate static charge on the film.

- f. Trim the mounting card to fit into a plastic Petri Dish. Do not cut into the optional paper backing if used.
- g. Label the Petri Dish with the appropriate information.
- h. The sample is in the correct geometry for counting on the Germanium detector system or well counter.

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- i. For counting in a beta detector, the sample must be removed from the Petri Dish and placed in the detector, sample side up.

#### 6.10 Liquid Scintillation Vial

- 6.10.1 Add 10 ml of scintillation solution to a clean vial.
- 6.10.2 Transfer 1.0 ml of the sample for all isotopes, other than Fe 55, to the vial containing the scintillation solution. For Fe 55, transfer the organic layer from the sample ( 5 ml) to the vial.
- 6.10.3 Secure the cap and clean the outside of the vial.
- 6.10.4 Label the cap with the appropriate information.
- 6.10.5 The sample is in the correct geometry for counting in the liquid scintillation counter.

#### 6.11 Gas Marinelli Beakers

- 6.11.1 Label the Marinelli beaker lid appropriately.
- 6.11.2 The sample is in the correct geometry for counting on a Germanium detector system.

#### 7.0 DOCUMENTATION/CORRECTIVE ACTION

Document results on proper reporting forms as per Chemistry Procedure 08-S-03-3, Document Control.

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FILTER PAPER - MOUNTING ILLUSTRATIONS

Figure 1  
TOP VIEW

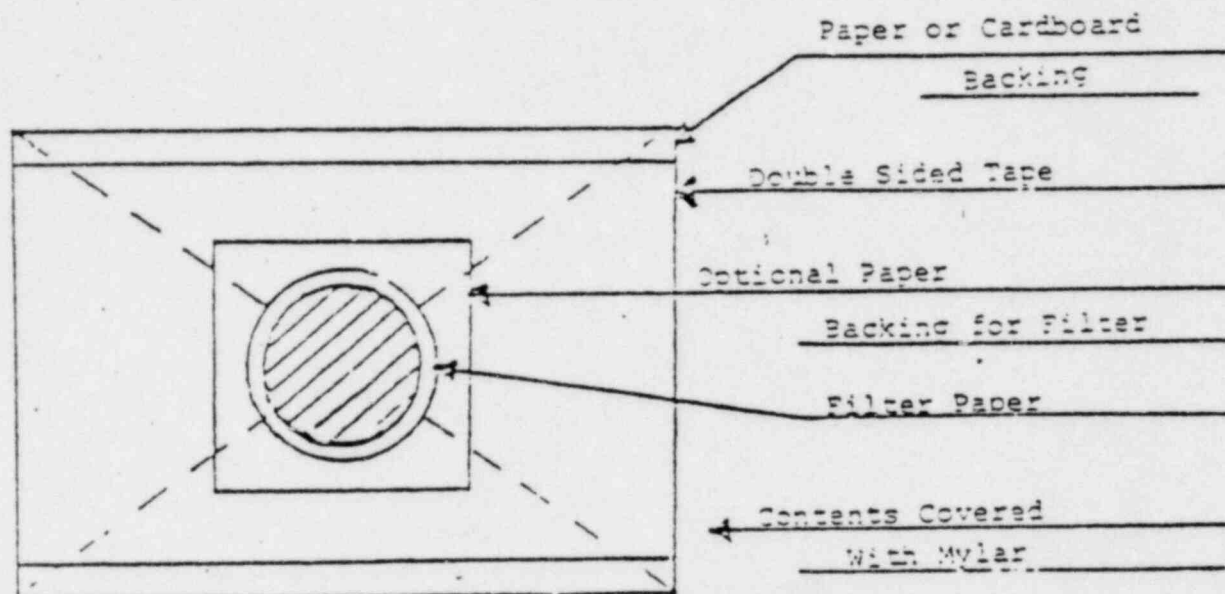


Figure 2  
SIDE VIEW

