



A Honeywell Company

## **Analysis of Hydrogen Fluoride Alkylation Catalysts and Regenerator Bottoms**

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### **UOP Method 629-08**

#### **Scope**

This 4-part procedure is for the sampling of liquid hydrogen fluoride (HF) streams and the analysis of these streams for acidity, water content and acid-soluble oil. The method is applicable to fresh acid and alkylation process system acid. The acid-soluble oil determination is also applicable to regenerator bottoms samples. However, acidity and water determinations on regenerator bottoms samples must be conducted according to UOP Methods 379 and 382, respectively. The Appendix provides information about an online instrumental option that could significantly reduce the number of samples that are brought into the lab for analysis.

#### **Caution!**

HF is toxic and may be fatal if inhaled, absorbed through skin or swallowed. It is extremely corrosive and destructive to tissue, and specialized medical treatment is required for all exposures. All appropriate safety procedures, including those listed in the HF MSDS, must be read, understood, and followed. Appropriate protective equipment must be worn throughout the procedure and all laboratory work must be performed in an appropriate fume hood.

#### **References**

MSDS, Hydrofluoric Acid, available from the supplier of the chemical.

UOP Method 379, "Hydrogen Fluoride in HF Alkylation Regenerator Bottoms," [www.astm.org](http://www.astm.org)

UOP Method 382, "Water in Regenerator Bottoms from Hydrogen Fluoride Alkylation," [www.astm.org](http://www.astm.org)

#### **Outline of Method**

A cleaned and dried sample cylinder is used to take a plant sample of liquid HF. The cylinder containing the sample is taken to the laboratory, where 3 portions of the sample are transferred to respective polypropylene bottles and analyses for acidity, water, and acid-soluble oil are made.

To determine acidity, the sample is diluted with neutralized 2-propanol. An aliquot is titrated with standard 1-*M* sodium hydroxide.

<p><b>IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).</b></p>
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To determine water, the HF is allowed to volatilize from the sample and any remaining acid is neutralized with dried pyridine. The neutralized sample is transferred to a moisture analyzer and titrated coulometrically.

To determine oil in liquid HF, pentane is added to the oil residue after all the excess HF has been volatilized. The pentane-oil mixture is warmed until a constant mass is achieved. The mass of the oil residue is calculated. Oil in regenerator bottoms is determined in a similar fashion to the liquid HF.

## Part I — Plant and Laboratory Sampling

### Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

*Balance*, readability 0.01-g, 1800 g capacity

*Beaker*, 150-mL, Griffin, borosilicate glass, VWR, Cat. No. 89000-202

*Beaker*, polypropylene, 4-L, VWR, Cat. No. 13890-181 (3 required)

*Bottles*, polypropylene, narrow mouth, with screw caps, 125-mL (2 required) and 500-mL, VWR, Cat. Nos. 16067-066 and 16067-102, respectively

*Bottle*, polyethylene, 1000-mL, VWR, Cat. No. 16129-704 (used for waste)

*Bottles*, polyethylene, wash, 500-mL, VWR, Cat. No. 16651-493

*Box*, wood, with hinged cover and 2 hasp-type locks, large enough to accommodate several sampling cylinders

*Bucket*, polypropylene, 7.6-L, VWR, Cat. No. 73470-016

*Clamp*, 3-prong, utility, VWR, Cat. No. 21573-265

*Cylinder sampling tube*, U-tube, 316 stainless steel, form as shown in Figure 1, EG & G Chandler Engineering Drawing No. 60-167, AMETEK Chandler Instruments

*Flask*, filtering, with side arm, 1000-mL, VWR, Cat. No. 89000-386

*Gloves*, heat resistant, VWR, Cat. No. 32888-340

*Manifold*, acid sampling, EG & G Chandler Engineering Drawing No. 60-170, AMETEK Chandler Instruments

*Oven*, drying, capable of operation at 50°C and 65°C

*Pipet*, glass, volumetric, Class A, 100-mL, VWR, Cat. No. 89003-368

*Pipet filler*, VWR, Cat. No. 53497-115

*Regulator*, air, two-stage, high purity, delivery pressure range 15-200 kPa (2-30 psi), Matheson Tri-Gas, Model 3121-590, if needed to control air pressure

*Sample cylinder*, Monel, 150-mL, see Figure 2 for details and construction. A sample cylinder assembly equipped with valve caps is available from AMETEK Chandler Instruments by specifying the sample cylinder described by their Drawing No. 60-120; includes Valve Stem, “K” Monel; Valve Gland, “R” Monel; Valve Body, “R” Monel; pressure test to  $1.72 \times 10^4$  kPa (2500 psi) hydrostatic.

*Stirring rod*, fluoropolymer, 20-cm long, Fisher Scientific, Cat. No. 14-518-10C

*Support stand*, 61-cm rod length, 15- x 23-cm base, VWR, Cat. No. 60110-244

*Tubing*, FEP fluoropolymer, 5.0-mm ID, VWR Scientific, Cat. No. 63014-714

*Tubing*, rubber or flexible plastic

*Vacuum system*, house vacuum or vacuum pump

## Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used. Unless otherwise specified, references to water mean deionized or distilled water.

*Acetone* (2-propanone), 99.5% minimum purity, VWR, Cat. No. BJ010-1

*Air*, compressed

*Ice*, crushed

*Phenolphthalein*, 1% solution, VWR, Cat. No. VW3341-2

*Sodium hydroxide*, 50% solution, VWR, Cat. No. VW3246-1

*Tags*, with string, white and red

*Water*, deionized or distilled

*Water*, tap, cold and hot

## Procedure

**CAUTION:** HF is a highly hazardous material. All appropriate safety procedures, as well as those listed in the HF MSDS, must be read, understood, and followed. Appropriate protective equipment must be worn throughout the analysis and all laboratory work must be performed in an appropriate fume hood.

### Cylinder Preparation

1. Remove any previous sample by emptying the cylinder into a polypropylene bucket containing crushed ice. Open the valve slowly at first to prevent splashing.
  - If the cylinder is new, begin the cleaning procedure with Step 7.
2. Add several mL of phenolphthalein indicator solution to the crushed ice-HF mixture.
3. Neutralize the HF by slowly adding 50% sodium hydroxide solution, while stirring with a Teflon rod, until a persistent pink color is obtained.
4. Squirt approximately 25 to 30 mL of acetone from a wash bottle into the cylinder.
5. Close the valve on the cylinder and place the cylinder in hot (approximately 50°C) tap water.
6. Shake the cylinder, invert it and release the acetone into the bucket containing the previously neutralized sample.
  - This bucket is used to temporarily contain neutralized samples and acetone washes. Dispose of the waste material following local safety practices and in an environmentally safe manner.
7. Attach the cylinder sampling tube to the cylinder and evacuate the cylinder with a vacuum pump protected by a filtering flask.
8. Close the valve on the cylinder and disconnect it from the vacuum system.