

HYDROGEN FLUORIDE IN HF ALKYLATION REGENERATOR BOTTOMS

UOP Method 379-81

SCOPE

This method is for determining hydrogen fluoride (HF) in HF alkylation regenerator bottoms. The bottoms material can be either a single or 2-phase system; the upper phase in a 2-phase system is rich in organic material and contains little HF, while the lower phase is rich in HF. The lower phase may contain large concentrations of water when there is appreciable water present in any of the feed streams. This method is applicable to both types of samples.

OUTLINE OF METHOD

The sample is weighed into a polyethylene bottle, diluted with water, extracted with isooctane, and an aliquot of the aqueous phase is titrated with standard sodium hydroxide, using phenolphthalein as the indicator.

APPARATUS

Balance, analytical, 2000 g capacity, capable of weighing to 0.01 g, Mettler PC 8000, available from Sargent-Welch Scientific Co., or equivalent

Beaker, polyethylene, 250-mL

Bottles, polyethylene, with screw caps, 500- and 1000-mL

Buret, 50-mL, with Teflon stopcock, 0.1-mL subdivisions

Pipet, polyethylene, 10-mL

Pipet, polypropylene, 100-mL, Bel-Art Products, Cat. No. H37906, or equivalent

Stirrer, magnetic, with Teflon-covered stirring bar

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).

REAGENTS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, when such specifications exist, unless otherwise specified. References to water mean deionized or distilled water. Unqualified references to solutions mean aqueous solutions.

Isooctane, ASTM grade

Phenolphthalein, 1% solution. Dissolve 0.5 g of phenolphthalein in 50 g of ethyl alcohol.

Sodium hydroxide, 1 *M*, standardized with potassium hydrogen phthalate to 4 significant figures. Store in a polyethylene bottle.

PROCEDURE

Before this analysis is started, put on rubber gloves, face shield and protective clothing appropriate for working with HF or HF-containing materials. Work in a well-ventilated hood; otherwise, provide means to avoid inhaling any HF fumes (see *PRECAUTIONS*).

Clean and dry a 500-mL polyethylene bottle and weigh it and a cap to the nearest 0.01 g. Pipet 100 mL of water into the bottle and cap it. Weigh the capped polyethylene bottle and contents to the nearest 0.01 g. If the sample is single-phase, add by pipet 10 mL of sample to the bottle. If the sample is 2-phase, shake the sample container first and then immediately pipet 10 mL of sample into the bottle. Cap the bottle and weigh it to the nearest 0.01 g. This increase in the weight of the bottle is the sample weight. Adjust the contents of the bottle to a final weight of about 400 g with water; weigh to the nearest 0.01 g. Shake the bottle for one minute and allow it to stand for 10 minutes. Add 25 mL of isooctane to the bottle, shake it for one minute and allow it to stand for 10 minutes. This allows the phases to separate.

Weigh to the nearest 0.01 g a clean and dry 250-mL polyethylene beaker containing a Teflon-covered magnetic stirring bar. Transfer by pipet, a 100-mL aliquot of the diluted acid lower phase (be careful not to include any of the hydrocarbon phase) into the polyethylene beaker. Reweigh the beaker to the nearest 0.01 g. Add 5 drops of the phenolphthalein indicator solution to the aliquot and turn on the magnetic stirrer. Titrate the aliquot with standard 1.0 *M* sodium hydroxide to a persistent red color. While titrating, wash down the sides of the beaker frequently with water to ensure that the entire solution is titrated.

CALCULATIONS

Obtain the weight-percent of HF as follows:

$$\text{Hydrogen fluoride, wt-\%} = \frac{2 \text{ VMA}}{\text{BW}}$$

where:

A = final weight to which the sample was diluted (sample + water), g

B = weight of 100-mL aqueous aliquot taken for titration, g

M = molarity of sodium hydroxide solution used for titration

V = volume of standard sodium hydroxide solution used for titration, mL

W = weight of sample taken for analysis, g

PRECAUTIONS

Even dilute HF solutions can cause painful and dangerous burns if not washed off the skin immediately. Wear rubber gloves, face shield and protective clothing at all times. If *any* of the sample liquids or the untitrated aqueous solutions come into contact with the skin, wash the area for 10-15 minutes with running water, and seek medical attention immediately.

PRECISION

The estimated standard deviation (esd) based on the indicated replicates is shown below. Duplicate results by the same operator should not be considered suspect unless they differ by more than the amounts shown in the “allowable difference” column (95% probability).

<u>Type of Sample</u>	<u>Number of Replicates</u>	<u>Level, wt-% HF</u>	<u>esd, wt-% HF</u>	<u>Allowable Difference, wt-% HF</u>
Regenerator Bottoms	6	5	0.04	0.15

TIME FOR ANALYSIS

The elapsed time for one analysis is one hour. The labor requirement is also one hour.

SUGGESTED SUPPLIERS

Bel-Art Products, Pequannock, NJ 07440

Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60076