WGK 145

MONSANTO CHEMICAL COMPANY
W. G. KRUMMRICH PLANT LABORATORY

STANDARD ANALYTICAL METHOD

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WRITTEN BY	APPROVED BY
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ATTACHMENTS: Method 10,873, Drawing 153,154,155,157

PRODUCT: Aroclors, Pyranols, and Electrical Grade Chlorobenzenes

TEST: Monsanto Stability

THIS FORM ☐ SUPERSEDES METHOD _____ ISSUED ON 9/23/52
THIS FORM ☒ REVISES INSTRUCTIONS

SCOPE

This method determines the stability of chlorinated benzenes and biphenyls used as dielectrics. Certain components in the dielectric may break down at elevated temperatures to form HCl. The volatile HCl is swept out of the sample with air, absorbed in water and titrated potentiometrically with silver nitrate.

EQUIPMENT

a. Heating bath - Drawing No. 155, assembled in our laboratory to maintain a set temperature to within ± 0.5°C. See attached drawing for particulars. Two 3 x 5" stainless steel plates not shown in the drawing are placed on top of the straightening vanes in the bottom of the bath.

1. Thermoregulator - American Instrument Co., Silver Springs, Maryland, Cat. No. 4-235.

2. Relay - Our relay is fabricated in our own laboratory, but any sensitive, reliable relay can be used.

3. Lo-Lag heater - 500 watt, 110 volt, American Instrument Co., Silver Springs, Maryland, Cat. No. 3-404.

4. Variac - 115 volt input, General Radio Corp., Cambridge 39, Mass., Type V-20M

5. Strip heater - 500 watt, 110 volt, General Electric Co., Cat. No. 2A-404.

6. Stirring motor - 1/5 H.P. Bodine motor, General Radio Co., Chicago, Ill., Type NSE-12.

7. Powerstat - The Superior Electrical Co., Bristol, Conn.

8. Stirrer - 4 bladed metal, 10-1/2" long, American Instrument Co., Cat. No. 4-640.

9. Bath fluid - Dow Corning 550, Dow Corning Corp. Midland, Mich.

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The heating bath is essentially a 29" x 7" x 8" bath made from #16 gauge 316 SS which has been insulated with 1" of Magnesia. The three 500 watt strip heaters are connected to the variac. The thermoregulator controls the relay for the Lo-Lag heater. The bath temperature is regulated with the strip heaters to 210°C. and then the thermoregulator is set to control the temperature within ± 0.5°C. The powerstat controls the stirring motor speed at a rate to maintain a uniform temperature throughout the bath.

2 b. Air supply - 40 psi air is reduced to 8 psi by a pressure regulator. The 8 psi air is purified by passing through an empty trap, a scrubber bottle containing 20% NaOH, a second empty trap, a scrubber bottle containing C.P. H_2SO_4, a third empty trap, and a vapor trap immersed in dry ice and acetone. The purified air line is connected to a capillary tube, cut to regulate the pressure at 24 - 26 mm Hg. on the manometer, and then to a glass T; one side going to a 6" mercury manometer, the other side going to a glass manifold having one connection for each sample. Capillaries are cut to a length that permits a flow of 45 ml. per minute of air and inserted between the manifold and the inlet down tube of the absorption flask.

1. Empty traps - 1000 ml. filtering flask, Fisher Scientific Co., Cat. No. 10-180.

2. Scrubbers - Fisher-Milligan Gas Washer, Fisher Scientific Co., Cat. No. 7-513.

3. Flow meter - Fisher Scientific Co., Cat. No. 11-163, fitted with flowrator tube No. 08-150/13.

4. Vapor trap - fabricated by our glassblower according to specifications given on Drawing No. 153.

5. Evacuated flask, 1 quart capacity - Fisher Scientific Co., Cat. No. 10-197. The flask is wrapped with plastic tape to help prevent injury by flying glass should the flask be accidentally broken.

6. Pressure regulator - outlet pressure range, actual 0 - 35 psi, Fisher Governor Co., Marshalltown, Iowa.

7. Capillaries, 0.25 mm I.D. - Fisher Scientific Co., Cat. No. 11-367.

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8. Flask, 300 ml. Erlenmeyer with 34/45 ♂ joint - Ace Glass Co., Vineland, N. J.

9. Absorption flasks - Fabricated in our laboratory from 125 ml. Volhard flask, E. H. Sargent and Co., Cat. No. S-63585. See Drawing No. 154.

c. Potentiometer - our potentiometer is fabricated in our laboratory, but any sensitive, reliable potentiometer can be used.

d. Silver wire electrode - B and S #12 gauge silver wire, Handy and Harman, 1800 W. Kenzie, Chicago 22, Illinois.

e. Mercurous sulfate reference electrode - modified L and N calomel reference electrode, L and N #7724-6-P2 (inner element) and #7724-6-P1 (outer element). Prepared as follows: Dismantle the internal element from the salt bridge tube of a standard L and N calomel reference electrode. Discard the saturated KCl solution from the tube and clean out the mercurous chloride and mercury from the internal element. Clean the parts thoroughly. Add sufficient new clean mercury to the internal element to make contact with the electrode wire and repack chamber of internal element with mercurous sulfate moistened with 0.5 M potassium sulfate. Seal the chamber with non-absorbant cotton. Fill the salt bridge tube with 0.5 M potassium sulfate and re-assemble units.

f. Magnetic stirrer - Labline Inc., Chicago, Ill., Cat. No. 1250 with plastic coated stirring bar.

g. Buret, 1.0 ml. microburet - Koch-Fisher Scientific Co., Cat. No. 20-110.

h. Other usual laboratory glassware.

REAGENTS

a. Acetone - Technical grade, Carbon and Carbide Chemical Co., or equivalent. A blank determination of the acetone must not exceed 0.03 ml. of 0.005 N $AgNO_3$.

b. Silver nitrate, 0.005 N solution, A.R. grade - Mallinckrodt Chemical Co., Cat. No. 2169. Make a 0.1 N solution by diluting 16.99 g. A.R. $AgNO_3$ to 1 liter. Take 50 ml. of this solution and dilute to 1000 ml, store in a dark bottle.

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5. Cover the oil bath with the transite boards and the exposed part of the flasks with aluminum foil.

6. Bubble air through the sample for 16 hours at the rate of 45 ml. per minute.

7. Disconnect the air supply.

8. Remove the absorption flask and transfer the contents to a 200 ml. beaker, rinsing the absorption flask into the beaker with two 5 ml. portions of distilled water.

9. Add 100 ml. of acetone and 2 drops of 1% HNO_3 to the beaker.

10. Titrate the sample potentiometrically following the procedure outlined in General Method No. 35, WGK Method No. 10,873 with the following modifications:

   a. Use a Ag-Hg·Hg_2SO_4, 0.5 M K_2SO_4 electrode system.

   b. Use 0.0025 N $AgNO_3$.

   c. Use a magnetic stirrer.

   d. Titrate to the maximum change in potential, usually between 75 and 100 mv.

CALCULATION

$$\text{Chlorides (ppm)} = \frac{\text{ml.} \times \text{N} \times .035457 \times 100 \times 10{,}000}{\text{Sample weight}}$$

$$= \text{ml. } 0.005 \text{ N } AgNO_3 \times 0.61$$

Report results to the nearest 0.1 ppm.

Duplicate results should check within ± 0.04 ppm.

PRECISION

Based on 20 duplicate determinations by different analysts using different apparatus. The standard deviation of this method is ± 0.04 ppm.

DISCUSSION

The precision of the method is based on material having a chloride content of approximately 0.5 ppm. Our material is normally in

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this range so no effort has been made to estimate precision at higher concentrations.

For directions on the setting and wiring of the thermoregulator, consult "Instructions for 'Quickset' Bimetallic Thermoregulator - Instructions No. 545" supplied by American Instrument Co., Silver Springs, Maryland.

The apparatus is cleaned with acetone to remove the dielectric and then rinsed thoroughly with distilled water. After the distilled water rinse, check the last water rinse from the flask for chlorides by the Tyndall beam method using 10% $AgNO_3$ solution. The water must be beam free at the end of 45 seconds. If a beam appears, wash the apparatus until the distilled water is beam free. Dry the apparatus in an oven before use.

In case the electrode system shows abnormal behavior, it may be checked by titrating a standard chloride solution. Prepare a 1.00 ppm standard by diluting 1.64 ml. of 0.5 N HCl to 1000 ml. in a volumetric flask with distilled water. Dilute a 10 ml. aliquot with 50 - 75 ml. methanol in a 100 ml. beaker and titrate with 0.005 N $AgNO_3$. Calculate the ppm chlorides indicated by the titration and compare with the standard value. Fresh standard should be prepared each time.

REFERENCE

a. Laboratory Notebook Reference: 148, 47.

b. "Instructions for 'Quickset' Bimetallic Thermoregulator" - Instructions No. 545, American Instrument Co. Inc., Silver Springs, Maryland.

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