MONSANTO CHEMICAL COMPANY

Anniston, Alabama
Plant Laboratory

STANDARD ANALYTICAL METHOD

Method No. 014

Product AROCLORS AND PYRANOLS		DATE ISSUED 2/3/60	PAGE 1 OF 2
Test Initial and Corrosion Chlorides - Potentiometric		WRITTEN BY W.W.K.	APPROVED BY W.W.K.

This Method ☐ Supersedes ☐ Revises Instructions Issued On New

SCOPE:

This method quantitatively determines the amount of inorganic chlorides present in Aroclors and Pyranols.

EQUIPMENT:

1. 200-ml tall form beakers (Berzelius type).
2. Magnetic stirrer (Fisher Scientific Company, Cat. No. 14-511-1).
3. Teflon magnetic stirring bar. This bar should be cylindrical and of one piece molded construction, one inch long (Fisher Scientific Company, Cat. No. 9-511-9).
4. Microburet, 1 ml capacity, graduated in 0.01 ml divisions.
5. Silver electrode (Beckman Cat. No. 39261).
6. Glass electrode (L&N Cat. No. 1199-30).
7. L&N pH assembly (L&N Cat. No. 7664-A1).
8. Usual laboratory glassware.

MONS 074536

REAGENTS:

1. 1:1 Sulfuric Acid - prepared by a 50:50 volumetric dilution of ACS grade H_2SO_4 with chloride free distilled water. Always pour the acid into the water with constant stirring.
2. 0.0025N $AgNO_3$ - Dissolve 0.4247g. of ACS Grade $AgNO_3$ in 1 liter of chloride free distilled water containing 3.0 ml of concentrated HNO_3.
3. Acetone (chloride free) - 5 liters of acetone are refluxed with 0.5g. of ACS Grade $AgNO_3$ for 1/2 hour. The acetone is then distilled from the $AgNO_3$ discarding the first 100 ml. to flush the apparatus. 90% of the charge is distilled from the flask and the contents of the flask discarded.

SAFETY:

Aroclors and Pyranols are irritating to the skin. The vapors of hot materials are irritating to the eyes and upper respiratory system. If accidental contact is made with the eyes, flush immediately with large quantities of water for at least 15 minutes and get immediate medical attention.

DETERMINATION:

1. Tare a 200-ml beaker on a beam balance to the nearest 0.1g.
2. Mix the sample well and pour a small amount out to rinse the lip. (Note: Be sure the temperature of the sample is below 50°C.).
3. Add 100 ± 1.0g. of sample to the beaker. Reweigh to obtain the weight of sample.
4. Using clean, dry tongs place a magnetic stirring bar into the beaker.
5. Add 100 ml of chloride free acetone.
6. Add 2 drops of 1:1 H_2SO_4.
7. Stir for 5 minutes with a magnetic stirrer. Longer stirring may be necessary for the higher Aroclors.
8. The sample is titrated to the biggest potential break with 0.0025 N $AgNO_3$, using the silver-glass electrode system.

   Note: Normal samples require extremely small amounts of $AgNO_3$. For this reason the titration is run using 0.01 ml additions and allowing sufficient time for equilibrium to be established before recording the emf change. If a change of 1 mv or less is observed per 0.01 ml increment for 3 or 4 additions, increase the additions to about 0.05 ml until a larger change is observed. Then proceed with 0.01 ml additions. After corrosion chlorides on Pyranol 1470 and Interteen PPO will require larger titrations.

9. Run a reagent blank exactly as above, omitting the sample.

CALCULATION:

$$\text{Free Chlorides as ppm Cl} = \frac{(\text{sple titer} - \text{blk titer}) \times \text{N } AgNO_3 \times 35.46 \times 10^3}{\text{sample wt.}}$$

Report to the nearest 0.01 ppm.

REFERENCE:

General Electric's method No. CLTM-1 issued 1-10/59.

MONS 074537