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MONSANTO CHEMICAL COMPANY

Anniston, Alabama
Plant Laboratory

STANDARD ANALYTICAL METHOD

Method No. 010

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Product	AROCLORS (ELECTRICAL GRADE)	7/26/61	1 OF
Test	HYDROLYSIS STABILITY TEST	WRITTEN BY: W. W. K.	APPROVE: W. W.

This Method ☐ Supersedes ☒ Revises Instructions Issued On 10/15/59

SCOPE:

This method quantitatively determines the presence of unstable Chlorine compounds in Aroclors (chlorinated Biphenyl).

EQUIPMENT:

1. 200-ml tall form beakers (Berzelius type).
2. Magnetic stirrer (Fisher Scientific Co., 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 Co., Cat. No. 9-311-9)
4. Microburet graduated in 0.01 ml. divisions (Scientific Glass Apparatus Company, Cat. No. JM-570).
5. Silver electrode (Beckman Cat. No. 39261).
6. Glass electrode (L&N Cat. No. 1199-30).
7. L&N pH assembly (Cat. No. 7664-A1).
8. Water bath, 150 mm dia. x 75 mm high (Fisher Scientific Co., Cat. No. 8-741).
9. Usual laboratory glassware.

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REAGENTS:

1. Methanol (chloride free) - 5 liters of methanol are refluxed with 0.5g Analytical Reagent Grade $AgNO_3$ for 1/2 hour. The methanol 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 are discarded. The methanol should be checked to assure purity by titration. The chloride ion concentration should be less than 0.02 ml. of 0.0025N $AgNO_3$ per 100 ml. of methanol.

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REAGENTS: continued

2. Sodium Hydroxide Reagent - Analytical Reagent Grade NaOH is used to prepare this reagent. A 0.1N solution is prepared by dissolving 4.0 grams of analytical reagent grade NaOH in 1 liter of chloride free methanol. (For Anniston, 8.0 g. of 50% plant NaOH may be used).

3. Sulfuric acid - Prepared by a 50:50 volumetric dilution of Analytical Reagent Grade concentrated sulfuric acid with chloride free distilled water. The acid is always poured into the water with constant stirring to prevent any dangerous build-up of heat.

4. 0.0025N $AgNO_3$ - Dissolve 0.4247g. of Analytical Reagent Grade $AgNO_3$ crystals in one liter of chloride free water containing 3.0 ml. of concentrated nitric acid. This solution should be standardized against a pure chloride standard. A sodium chloride crystal such as used in infrared spectrometer cells is a good source of pure NaCl. The $AgNO_3$ solutions should be checked (at least monthly) to assure a consistent reagent.

5. Acetone (chloride free) - Prepared by distillation from $AgNO_3$ as described above, for methanol and should also be checked by potentiometric titration to assure optimum purity. Normally a chloride content of less than 0.02 ml. of 0.0025N $AgNO_3$ per 100 ml. is derived by this method.

6. Benzene - Analytical Reagent Grade benzene should be used. This material is normally chloride free but should be checked by potentiometric titration to be certain.

SAFETY:

Aroclors 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:

I. Aroclor 1242:

1. Tare a 200-ml. beaker on a beam balance to the nearest 0.01 g.

2. Mix the sample well and pour a small amount over the lip of the sample bottle to clean it.

3. Add 25.0± 1.0 g. of sample to the beaker. Reweigh to obtain the weight of sample.

4. Using clean, dry tongs place the magnetic stirring bar into the beaker.

5. Add, by pipet, 25 ml. of 0.1N methanolic NaOH. Cover the beaker with a watch glass.

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6. To the water bath add 1-1/4 inches of water at 40± 1°C.

7. Place the beaker and sample in the water bath and then on the magnetic stirrer. Clamp the water bath to a ringstand so it won't slide off the stirrer.

DETERMINATION: continued

8. Stir the sample at as fast a speed as possible, without pronounced splashing, for 1 hour.

   Note: During this hour the water bath will cool down to approximately room temperature.

9. Remove the beaker from the water bath.

10. By pipet add 0.5 ml. of 1:1 H_2SO_4.

11. Now add, by graduate, 125 ml. of chloride free acetone.

12. The sample is titrated to the biggest potentiometric 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, then increase the additions to about 0.05 ml. until a larger change is observed. Then proceed with 0.01 ml. additions.

13. Run a reagent blank exactly as above omitting the sample.

Calculation

$$\text{Reactive chlorine (ppm)} = \frac{(\text{sple titer-blk titer}) \times N_{AgNO_3} \times 35.46 \times 10^3}{\text{sample wt.}}$$

II. Aroclor 1254 and Aroclor 1260:

The procedure is the same as in Part I above except as follows:

1. After Step I-4, add by pipet 5 ml. of chloride free benzene.

2. Heat the sample to dissolve the Aroclor in the benzene.

3. Cool to room temperature.

4. Proceed with Step I-5.

Calculation

Same as in Part I.

REFERENCE:

G. E. Dechlorination Test Method for Unstable Chlorine Compounds in Askarels.

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