FORM 168

MONSANTO CHEMICAL COMPANY
W. G. KRUMMRICH PLANT LABORATORY

STANDARD ANALYTICAL METHOD

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ATTACHMENTS:		

PRODUCT: Aroclors, Pyranols, Tri-Chlorobenzene and Tri-Tetrachlorobenzene Blends

TEST: Inorganic Chlorides

THIS FORM: ☐ SUPERSEDES METHOD _____ ISSUED ON 1/30/58
☒ REVISES INSTRUCTIONS

SCOPE

This method determines traces of inorganic chlorides in transformer and capacitor fluids and their components. The chlorides are extracted with hot water and determined turbidimetrically by comparison with standard solutions.

EQUIPMENT

a. Penlight flashlight, Ray-O-Vac Co., Madison, Wisc., Model Z-21-P, or equivalent.

The light bulb is coated with black paint. After drying, the paint is rubbed off the end with a flat file to expose approximately 3 - 4 mm of the bulb surface.

b. Funnels, separatory, 250 ml., Fisher Scientific Co., Cat. No. 10-436, or equivalent. Cut the stems to 10 mm length.

Calibrate two funnels at 25 and 50 ml. respectively by measuring the desired amount of H_2O in a graduate and then pouring the water into the funnels. Mark the funnels at the water level by etching with a vibratool or by scratching with a file.

c. Test tubes, 16 x 150 mm, Pyrex, Fisher Scientific Co., Cat. No. 14-955, or equivalent.

d. Usual laboratory apparatus.

REAGENTS

a. Silver nitrate, A.R. (Mallinckrodt Chemical Co., Cat. No. 2169, or equivalent), 10% solution.

Prepare a 10% solution by dissolving 10 g. $AgNO_3$ in 80 ml. of chloride free distilled water and 10 ml. conc. HNO_3. Store in a black-painted bottle.

REAGENTS (Cont'd.)

b. Nitric acid, A.R. (Mallinckrodt Chemical Co., Cat. No. 2704, or equivalent), 1:1 solution.

Mix equal volumes of HNO_3 and distilled water.

c. Ether, anhydrous, A.R. (Mallinckrodt Chemical Co., Cat. No. 0848, or equivalent), chloride free.

Wash with chloride-free distilled water until the water extract is beam free when checked by Tyndall beam in a dark room as directed in step 4, below.

d. Sodium chloride, A.R. (Mallinckrodt Chemical Co., Cat. No. 7581, or equivalent), standard solutions.

Prepare the standard solution by dissolving 0.1648 g. NaCl in 100 ml. distilled water and diluting to 1 liter. Dilute 100 ml. of the above solution to 1 liter to prepare a 10.0 ppm standard. For every 0.1 ppm standard, dilute 10 ml. of the 10.0 ppm standard to 1 liter. Use chloride-free water for all dilutions and mix each thoroughly. These working solutions should be prepared fresh before each use.

SAFETY

Aroclors, Pyranols, Tri-Chlorobenzene and Tri-Tetrachlorobenzene blends are irritating to the skin. The vapors of the 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. Exercise caution to avoid possible thermal burns through contact with hot equipment.

Ether is highly flammable. Use and store in a well ventilated hood. The vapors of ether are heavier than air and can travel great distances along the floow. Do not expose to heat, open flames, or lighted cigarettes. No bunsen burners can be lit and no smoking should be allowed while working with ether.

DETERMINATION

IMPORTANT: The quantity of chlorides in these materials is extremely small, so exercise great care not to contaminate the sample or equipment. Wear canvas gloves and do not touch the equipment at any point which will be exposed to sample contact.

1. Thoroughly rinse two separatory funnels with chloride-free distilled water three or four times. Fill both funnels to approximately 100 ml. with distilled water.

2. Drain a small amount of water from both funnels into a waste beaker and then drain approximately 10 ml. of H_2O from each funnel into each of two 16 - 150 mm test tubes which have been similarly rinsed with chloride-free distilled water.

DETERMINATION (Cont'd.)

3. Add an equal volume of chloride-free ether and mix thoroughly.

NOTE: Perform the next step in a darkroom.

4. Add 3 - 5 drops of 10% $AgNO_3$ and allow 45 seconds, measured by stopwatch, for full beam to evolve. Examine the sample with the light from a penlight for any visible beam. Absolutely no dust or chloride beam should be present.

NOTE: If beam is present, rinse all equipment with 1:1 HNO_3 and repeat steps 1 through 4.

5. Drain the funnels until they contain 50 ml. and 25 ml. of water respectively. Lay the stoppers atop the funnels and heat the water in funnel containing 50 ml. to boiling.

6. Pour a small amount of the sample over the lip of the sample bottle into a waste beaker, then transfer 50 ml. of sample by pouring directly into the funnel containing 50 ml. of H_2O.

CAUTION: Wear canvas gloves while handling hot equipment.

7. Stopper the funnel and shake vigorously for at least 1 minute, venting frequently through the stopcock.

8. Allow the two layers to separate and drain approximately 2 ml. of the sample into a waste beaker. Heat the water in a second funnel to boiling and transfer the sample into it.

NOTE: It may be necessary to heat the first funnel when transferring the sample, especially in the case of the more viscous Aroclors.

9. Stopper funnel and shake thoroughly, as in step 7, allow the layers to separate, and drain the sample into a waste beaker.

10. Combine both water extracts in the first funnel and shake thoroughly.

11. Drain approximately 2 ml. of the water from the funnel into the waste beaker and then drain approximately 10 ml. into each of two 16 x 150 mm test tubes which have been rinsed thoroughly with chloride-free distilled H_2O.

12. Add an equal volume of chloride-free ether to each and shake the mixture until any emulsion is broken and the water layer is beam free.

NOTE: Perform the next step in a dark room.

DETERMINATION (Cont'd.)

13. Add 3 - 5 drops of 10% $AgNO_3$ and mix the solution by shaking. Examine the sample with the light from a penlite for exactly 45 seconds (by stopwatch).

14. If no beam is detectable in the water layer at the end of 45 seconds, report the chlorides as "no detectable amount (NDA)". The very faintest of beams is considered 0.1 ppm. If the beam is stronger, it will be necessary to compare with standards of 0.15, 0.20, up to 1.0 ppm, adding the 3-5 drops of $AgNO_3$ and comparing at the end of 45 sesonds.

CALCULATION

Report results as NDA, less than 0.1 ppm or to the nearest 0.1 ppm.

DISCUSSION

No precision data has been compiled on this method but experience shos the results can be duplicated within $\pm$ 0.05 ppm.

While we use a darkroom for the comparison of beams in this laboratory, a light-tight box, which would permit handling and viewing of the samples, should serve adequately.

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