Monsanto Chemical Company

Anniston Method No. 14-48-48

SUBJECT: The Testing of Aroclors for Inorganic Chlorides

METHOD:

Solutions Required

Standard Chloride Solution: Dissolve 0.660 grams of pure sodium chloride in sufficient water to measure exactly 500 ml.; 1 ml. then contains 800 micrograms Cl. Pipette 25 ml. of this solution to a 500 ml. flask and make to the mark to give a 0.00113 M NaCl solution, 1 ml. of which contains 40 micrograms of Cl. Preserve in a Pyrex bottle. Caution: The water used in preparation must be chloride free by the Tyndall Beam test; the 0.00113 M solution should not be kept over 1 month.

Silver Nitrate Solution 10%: Dissolve 5 grams of silver nitrate crystals in 40 ml. of water and add 10 ml. of concentrated nitric acid.

Chloride free distilled water.

Apparatus Required

LaMotte 10 ml. color tubes (13 mm. X 100 mm).

Test tube rack with a black surface at the base for uniform comparison (Note: we are using black gasket rubber over the entire board that supports the base of test tubes during comparison.)

Box. About 7" long and 4" wide. This box contains holes in top and notches in the bottom to hold Lamotte color tubes. It is just high enough to allow 1/4" of test tube to extend through the holes in the top. The box should be painted a flat black on the inside and on top and should contain enough holes to accomodate at least seven tubes.

Method

Clean all flasks, tubes, and pipettes to be used. Then rinse with dilute nitric acid then with chloride free distilled water.

Add 50 ml. chloride free water to a 250 ml. Erlenmeyer flask, heat to boiling, add 200 gram of the Aroclor to be tested, which has been heated to 100°C, shake vigorously for 1 minute, cool in cooling pan. Carefully decant some of the water extract to a separatory funnel. Wash with pure ethyl ether (chloride free).

Transfer 10 ml. of the ether washed extract to a 10 ml. LaMotte color tube after rinsing tube with portion of extract. Prepare standards by diluting exactly 5 ml. of the 0.00133 M NaCl solution to 100 ml. in a

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volumetric flask. One ml. then contains 2 micrograms of Cl. Transfer 1, 2, and 3 ml. portions to 10 ml. tubes. Make to the 10 ml. mark with Cl free H_2O. Place standards and sample in light free box from which only the top of tubes are exposed add 0.5 ml. of the silver nitrate to each tube compare in a specially prepared test tube rack.

The comparison is to be made quickly and from a constant source of light, preferably day light from a window away from the sunlight. Comparison is easier when other sources of light are shielded from the comparison rack. Look directly into the top of the tubes and compare. Note the known solution which most nearly matches the water extract estimating to the nearest whole microgram. The extract blank and the distilled water should show no turbidity.

Calculation

Divide the chloride content in micrograms of the known solution which matches the extract by the weight of the Aroclor represented by the extract to obtain the chloride content of the Aroclor in p.p.m.

In the procedure described, the 10 ml. extract represents 40 grams of Aroclor (1/5 of 200 g. taken). The three knowns contain 2, 4, and 6 micrograms of Cl, thus representing 0.05, 0.10, and 0.15 p.p.m. respectively. Values should be reported to the nearest 0.025 ppm.

Precautions

Because of the sensitivity of this test, serious errors may result through very slight contamination or inattention to details. All equipment must be scrupulously cleansed, the distilled water absolutely chloride free and testing done away from fumes of HCl or chlorine fumes.

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