DIRECTIONS FOR TEST NO. 14-38-41

SUBJECT: Free Chlorides in Liquid Aroclors

METHOD: Turbidimetric Silver Chloride

Solutions Required

Standard Chloride Solution: Dissolve 0.660 grams of pure sodium chloride in sufficient water to measure exactly 500 cc.; 1 cc. then contains 800 micrograms Cl. Pipette 25 cc. of this solution to a 500 cc. flask and make up to the mark to give a 0.00113 M NaCl solution, 1 cc 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 cc. of water and add 10 cc. of concentrated nitric acid.

Preparation of Water Extract

Transfer 100 cc. of distilled water from a graduate to a clean 250 cc. erlenmeyer flask. Stopper and shake thoroughly. Draw off 50 cc. of the water into a 50 cc. centrifuge tube. Test this water for chloride by the Tyndall Beam method; a negative result must be obtained before proceding.

To the 50 cc. of water remaining in the flask add 200 grams of Aroclor which has been heated to 100°C. Stopper and shake vigorously for at least 20 seconds. Allow to settle. Decant off most of the water extract into the 50 cc. centrifuge tube. Centrifuge until the extract is perfectly clear.

The water extract may alternately be clarified by filtration using suction. In this step the extract is decanted thru a fritted glass filter funnel of medium porosity which has been coated with a little Johns Manville filter-aid and washed with acetone, dilute nitric acid, and distilled water until shown by test to be free of chloride.

Tyndall Beam Test

Transfer 10 cc. of the water extract, clarified by centrifuge or filtration, to each of two test tubes (LaMotte 10 cc. color tubes). To one of these add 0.5 cc. of the 10% silver nitrate solution and mix with a plunger-type stirring rod which must be absolutely clean. The solution in the second tube is used as a blank.

Compare against other tubes, prepared at the same time, containing known amounts of chloride. The knowns are prepared as follows: Dilute exactly 5 cc. of the 0.00133 M NaCl solution to 100 cc. in the volumetric flask; 1 cc. then contains 2 micrograms of Cl. Transfer 1, 2, and 3 cc. portions to similar 10 cc. tubes. Make up to the 10 cc. mark and add 0.5 cc. of the silver nitrate to each as well as to a fourth tube containing only distilled water such as used in the preparation of the knowns.

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Compare the 6 tubes in a dark room where illumination is afforded only by a pencil of bright light to provide the Tyndall Beam effect. Holding the tubes so that the light enters the bottom of the tube at an angle of about 45° is sometimes helpful. Note the known solution which nearest matches the water extract, estimating to 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 Aroclor represented by the extract to obtain chloride content of the Aroclor in p.p.m.

In the procedure described, the 10 cc. of 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 p.p.m.

Precautions

Because of the sensitivity of this test, serious errors may result thru very slight contamination or inattention to details. Ammonium chloride deposits are common to laboratory apparatus. These must be avoided, all containers must be scrupulously cleaned, the distilled water must be absolutely chloride-free, and blank tests applied unremittingly.

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