Date: 12-15-48 Material: Aroclors and Pyranols Method No. 10,195-48
By: RAC:HOH Test: Inorganic Chlorides Plant: B No. 233-405

This test is run on Aroclors 1262, 1260, 1254, 1248, 1242, and Pyranols 1495, 1488, 1467, 1481, and 1478 (TCB).

1. Thoroughly rinse two separatory funnels with chloride-free water three or four times. Then take an aliquot from each funnel in a test tube which has been rinsed with chloride-free water. Test these qliquots for Tyndall beams by adding 3-5 drops of $AgNO_3$ and allowing 45 sec. for full beam to evolve. Absolutely no dust or chloride beam should be present. (If beam is present rinse all equipment with 1:1 HNO_3 and repeat Step 1.)

2. When funnels are beam-free, drain out all the water except 50 ml. in one and 25 ml. in the other. Heat the water in both funnels to boiling. (Hold stopper while heating as steam may cause stopper to fall.)

3. Transfer 50 ml. of the sample from the sample bottle at a temperature of 95-100°C into the separatory funnel containing the 50 ml. of boiling water. (As a precautionary measure pour some of the sample from the sample bottle into a waste beaker before adding the 50 ml. to the funnel.)

4. Stopper the funnel and shake vigorously for at least 1 minute, venting frequently through the stopcock. (Care must be exercised at all times to touch neither the lower part of the funnel stem nor the ground part of the stopcock.)

5. Allow the layers to separate and drain off the sample into the second funnel containing the 25 ml. of boiling water. (As before, drain off a few ml. of the sample into a waste beaker before draining the sample into the second separatory funnel.) It may be necessary to heat the sample when transferring the sample to the second funnel; e.g. Aroclor 1260.

6. Repeat step 4 and allow the layers to separate. Then drain off the sample into a waste beaker.

7. Combine both water extracts in one funnel and shake thoroughly.

8. Take approximately a 10 ml. aliquot of the water extract out through the bottom of the funnel into a 3/4" x 6" test tube. Again, first allow a few ml. to drain out before taking the aliquot. (The test tube used should be rinsed with chloride-free water several times before using.)

9. Add approx. an equal portion of chloride-free ether. (The ether is tested by shaking a portion of it with chloride-free water and testing for Tyndall beam at the end of 45 sec. If beam is present, wash ether several times with chloride-free water until washings show no beam after adding 3-5 drops $AgNO_3$).

10. Shake the ether-water mixture until the emulsion in the sample disappears and the water layer is completely beam free before adding $AgNO_3$. If emulsion is difficult to break, add sample dropwise through the ether and then shake.

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11. Add 3-5 drops of 10% $AgNO_3$ solution and test for chloride beam for 45 sec. exactly. If no beam is present at the end of 45 seconds, report as <0.1 ppm. The very faintest of beams is considered 0.1 ppm. If 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 sec.

The method is precise to the nearest 0.1 ppm.

Report results to the nearest 0.1 ppm.

Note 1. The following standards are made up by the Service Dept.:

Make a primary standard of 100.0 ppm by weighing 0.1648 g. C.P. NaCl into a chloride-free 1-liter volumetric flask. Dilute to the mark and mix throughly. Make a 10.0 ppm standard by diluting 100 ml. of the primary standard to 1 liter, and mixing well. For every 0.1 ppm standard, dilute 10 ml. of the 10 ppm standard to one liter and mix well. A 0.1 ppm beam is considered the very faintest beam preceptible to the eye between 15-45 seconds after adding the $AgNO_3$ solution. If the beam intensity is not visible at all, or if easily visible (too strong), discard the solutions and make new standards.

Note 2. Weigh 20.0 g. C. P. $AgNO_3$ into a chloride-free dark bottle. Add 20 ml. C. P. HNO_3 (chloride-free). Dilute to 200 ml. with water.

Note 3. All solutions should be freshly prepared every two weeks and stored in glass-stoppered Pyrex bottles.

Note 4. Employ the 2-battery Penlite flashlight, having a 3-4 mm. light aperture. New batteries must be used frequently in order to perceive beams properly.

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