Extra (2)

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The recent disagreements between Anniston and G. E. Laboratories as regards the moisture content of various Pyranols have been the source of concern of the Monsanto Aroclor personnel. The writer was therefore called in to carefully check the Anniston analytical procedures for moisture determinations.

After making up several fresh standards and standardizing the Karl Fischer reagent, the conclusion was reached that the source of error was in the water standard used at the Anniston plant during recent moisture determinations. The faulty water standard had been purchased in early September.

A water standard ceases to be a standard after its water content varies from the amount thought to be present. The bought water showed 1 mg. H_2O per ml. on the bottle, but it had increased to 2.6 mg when it was checked. Therefore it was no longer a standard. The calculation of moisture determinations by the Karl Fischer method is based on the water content of the water standard. Therefore the water content of the standard must be known.

The analytical procedure as written up and practised now is satisfactory for moisture determinations. Therefore this first recommendation will concern the water standard and its handling.

The recommendation is simply that the water standard be made up often enough to be a standard and that it be protected from the atmosphere. Once a week should be the maximum time between new standards. Always make up a new standard after a period of inactivity in moisture determinations. The standard can be kept in a 100 cc volumetric flask with a ground glass stopper, but for better protection the use of an automatic burette with a reservoir is preferred. A calcium chloride or Drierite tube should protect all openings on the burette from the atmosphere and a stopcock should be inserted between the drying bulb and the reservoir. This feature would prevent the loss of methanol from the standard since it would only be opened while dispensing a standard water sample.

All pipettes and flasks used in the water analysis must be dried by heating to 120°C and then cooling by passing dry air through them.

The second recommendation concerns the preparation of sample bottles to eliminate any possible contamination of the Pyranol sample by the bottle or its closure. An approved G. E. method of cleaning and using bottles should be concurred with in order to decrease the possibility of any rejections on advance samples to General Electric Co.

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The following outline is taken from the available information on the manner in which samples should be prepared and shipped to G. E.

1. Select a 1/2 gal. wide-mouthed, screw-cap bottle for the reserve sample and an amber 5-pt., narrow-mouthed, Bakelite screw-cap bottle for the advance sample to General Electric Co. if called for. Select a 1-gal., wide-mouthed, screw-cap bottle for the laboratory working sample.

2. If the bottle has been used before, wash all the Pyranol from the inside and outside with warm (50°C) trichlorobenzene.

3. Rinse the TCB from the bottle with hot tap water.

4. Wash the bottle inside and outside with trisodium phosphate and a brush that is used exclusively for this purpose. (If a new bottle is used, omit step 2-3.)

5. Rinse out the TSP with hot tap water and drain the bottle.

6. Rinse the bottle next with about 500 cc. chromic acid cleaning solution (50°C), being certain that the solution comes in contact with the entire inner surface of the bottle.

7. Rinse all traces of cleaning solution from the bottle with tap water, using at least four washes and allowing each to drain well before adding the next.

8. Finally rinse inside and outside three times with distilled water, again draining well between each rinse.

9. Observe the water film as the bottle drains. If it remains unbroken, the bottle is chemically clean. If it breaks or separates into droplets, it must be rewashed (Steps 4-8).

10. Wipe the screw caps carefully with a clean, dry cloth inside and outside. These caps must be new and must be fitted with aluminum foil liners.

11. Place the bottles in the oven at 120°C with their caps and dry for 3 hours.

12. Blow the bottles for 5 minutes with dry air that has passed through a Drierite filter and a cotton filter or with oil compressed nitrogen.

13. Again place the bottles in the oven and dry for an additional 1 hour or more.

14. Remove the bottles and caps from the oven and screw the caps on firmly.

15. Rinse the bottles twice with the material to be sampled and fill to within 1/2 inch of the top. Screw the cap on firmly.

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16. Make all determinations on the 1 gallon bottle. After eight hours or longer analyze the 5-pt. amber bottle for moisture.

17. When all analyses are completed and the moistures check on the two bottles analyzed for moisture, put the 1/2 gal. sample in reserve and ship the 5-pt. sample to General Electric Co.

References: "Preparation of Sample Bottle"

Plant B Method 246-029, 8/9/43

"Preparation of Glass Containers for Sampling Pyranol" D. W. Caird, General Electric Co. -

Sketch No. H-6871316, 6/25/40

As a third recommendation, the drums which are to be filled with Pyranol should be blown out with dry air before filling in order to eliminate the drums as a source of moisture pick-up. The inner surface of the drums should of course be dry before blowing with dry air. This practice should be followed in the filling of all drums used for Aroclor or Dykanol as well as Pyranol.

A fourth recommendation concerns the Pittsburgh Lectrodryer. In its present position it has a limited use and requires time for someone to attend it occasionally. By installing this unit in the Aroclor building, it could be watched and checked easily and it would be near the point of use. The dry air could be used to dry out drums and to reduce the moisture content of all the liquid Aroclors before shipment.

Whether or not the unit is relocated, it would be a wise move to replace the old adsorbent with the new activated alumina which is on hand. The lowest dew points reached on the existing set-up has been from 0-5°F, whereas it is desirable to go as low as -40°F for the most efficient drying by aeration. The present inability to get a low dew point is probably due to the fact that the absorbent was clogged or poisoned in its former use as a benzene remover. New alumina would give us an Lectrodryer unit which should produce dry air having a very low dew point.

The above recommendations sum up in my opinion the steps necessary to insure the correctness of our moisture determinations and the plant procedures which could be used to produce an Aroclor ready for shipment with a low moisture content.

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