COPY

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Dr. R. L. Jenkins, Anniston

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cc: Mr. Gerber, Anniston

I am sending you, herewith, copies of our method for Moisture in Pyranols and Aroclors, and our General Method for Traces of Moisture. The first gives specific directions for the determination of moisture in the Pyronals and Aroclors, whereas the latter gives detailed instructions as to the preparation and standardization of the Karl Fischer Reagent.

We do not take any specific precautions in the sampling, nor for removing adsorbed moisture from the titration flasks. The flasks are dried in the usual type of drying oven and the solvent is titrated to the end point before the sample is added. This should preclude contamination of the sample with adsorbed moisture.

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Material: Aroclors and Pyranols

Test: H_2O

Moisture in Aroclors and Pyranols is determined with Karl Fischer reagent (Method No. 10, 100-40, (G.H. 7)). Because of the varying solubility of the products tested, different proportions of a mixed solvent are required. The analysis itself, however, is the same for all Aroclors and Pyranols.

The analysis is made as follows:

1) Add the solvents as listed below to a 500 ml. Erlenmeyer flask.

Material	Anhyd. Benzene	Anhyd. Methanol
Pyranol 1478	0 ml.	50 ml.
Pyranol 1488	50 ml.	50 ml.
Pyranol 1495	80 ml.	50 ml.
All Aroclors	80 ml.	50 ml.

2) Titrate the solvent to the characteristic brown end point with Fischer reagent.

3) Refill the buret to the zero mark.

4) Weigh a 100.0 g. sample by difference into the solvent. Mix well.

5) Titrate the solution to the same endpoint as before.

Calculation: $\% H_2O = \frac{\text{ml. Fischer reagent} \times H_2O \text{ factor} \times 100}{\text{Sample Weight}}$

The method is accurate to ± 0.0005%

Report the results to the nearest 0.0005%.

Material: General Method Method No. 10,100-40

Test: Water, Traces

Traces of water may be determined by means of a titration with Karl Fischer Reagent.

The reagent is prepared as follows: Place 667 ml. of dry methanol and 269 ml. of dry 2° pyridine in a dry 1000 ml. Florence flask. Add 84.7 g. of iodine and shake to dissolve. Stopper and cool to 0° to 2°C in a slurry of ice and water. When cooled to proper temperature, add 64 g. of SO_2 at such rate as to require about 90 minutes for complete addition. Keep cold during the procedure, and shake frequently. The SO_2 is best dispensed from a graduated cylinder, from which the lip has been removed, and is provided with a one hold rubber stopper through which a bent glass tube is passed. This bent tube should be connected to another tube leading into the Florence flask the outlet of which is below the surface of the liquid. Prepare the reagent in one liter quantities only. This reagent should be put into use between three and seven days after preparation. Two bottles should always be onhand. When not in use, store the reagent in a 32 ounce glass stoppered bottle.

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Standardize the reagent as follows: Prepare a solution of dry methanol containing a known amount of water - about 6.0000 g. per liter. Titrate 10.0 ml. of this solution with the reagent. The endpoint is a color change from yellow to brown. Call this titration B. Also titrate 10.0 ml. of the methanol used in the preparation of the water standard. Call this titration A. Titration A will account for 10% of the water present in the methanol used for the standard. This correction is negligible. Calc'd: the water factor as follws:

(1) Ml. factor = $\frac{\text{g. of water per 10.0 ml.}}{\text{B-A}}$

Calc'd: the amount of water originally present in the liter of methanol.

Titration A x Factor x 100

It will be found that one liter of methanol contains about 1.000 g. of water. This amount plus the amount of water added is the total amount of water in the standard. After the original calculation, the total water in the standard is known, and the following calculation for daily determinations of the factor can be employed:

(2) Ml. factor = $\frac{\text{Total g. water per 10.0 ml.}}{\text{ml. Titration}}$

Formula (1) is used only in determining total water in the standard. Formula (2) is used for all routine standardizations. The total g. of water per 10.0 ml. of standard will be posted on the bottle containing the standard solution.

The standardization is carried out in a clean, perfectly dry 250 ml. Erlenmeyer flask. The factor will be approx. 0.00330 g. H_2O per Ml. Standardize the solution every day, immediately before first use in the morning.

The reagent is dispensed from an automatic burette set-up provided with a liter reservoir and a drying bottle provided with alternate layers of glass wool and P_2O_5. The drying agent must be renewed whenever examination shows that it is necessary. The air used in moving the reagent is drawn from the drying bottle through a pressure bulb and forced into the reservoir. All air vents are connected to the drying bottle.

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DIRECTIONS FOR TEST NO. 14-39-41

SUBJECT: Water Content of Liquid Aroclors

Method: Titration with Karl Fischer Reagent

Outline

The Aroclor is dissolved in a mixture of benzene and methanol which has been titrated with Fischer reagent to the characteristic endpoint. Fischer reagent is a solution of iodine, sulfur dioxide, and pryidine. Iodine is consumed as long as any water is present. The solution is again titrated to obtain the water content introduced by the Aroclor.

Solutions and Apparatus

Karl Fischer Reagent: Weigh into a flask 264 grams of pyridine, Barrett 2-A or Eastman 214-H, and add 61 grams of liquid sulfur dioxide. Add the sulfur dioxide directly from the inverted cylinder by attaching to the outlet valve a glass tube extending into the pyridine. Store in a glass stoppered bottle.

Transfer 65.6 grams of the pyridine-SO_2 mixture and 134 cc of absolute methanol to a 1 liter Florence flask. Cool thoroughly in an ice water bath. Add 16.9 grams of iodine, stopper, cool again before shaking. Alternately cool and swirl until the iodine is in solution. Makes 200 cc of reagent. Store in glass stoppered bottle. Let stand 24 hours before using. When fresh, one cc of reagent is equivalent to about 0.0033 gms. of water.

Aroclor Solvent: Mix 2 parts dry benzol with 1 part absolute methanol. The dry benzol is prepared by shaking the commercial grade with anhydrous calcium chloride, decanting into a flask and distilling, rejecting the first 10% to come over. Keep in glass stoppered bottles with minimum exposure to air.

Micro Buret: 10 cc x 0.05 cc. Koch automatic with glass stoppered reservoir. Eck and Krebs #2460. Equip reservoir with a moisture guard tube.

Procedure

Heat a clean 500 cc narrow mouth erlenmeyer in an oven or on hot plate until thoroughly dry. Cool in desiccator. Sweep out for 10 minutes with air which has been thoroughly dried by passing through tubes containing 8 mesh drierite. Disengage the flask from the aspirating train and immediately close with a paper cap held by a rubber band.

Without delay, transfer 90 cc of the benzene-methanol solvent to the flask through a small hole punctured into the paper cap. Titrate at once with Karl Fischer reagent to a distinct red-brown end point which does not fade after swirling several times. The Fischer reagent is dispensed from the microburet the tip of which projects into the flask through the puncture in the paper cap. At the end point any water content of the solvent has been reacted. Superimpose a fresh paper cap.

Obtain weight of the solvent and well-covered flask on a Torsion balance or equivalent. Quickly introduce about 100 grams of the slightly warmed Aroclor to be tested. Cover at once and weigh again to obtain the weight of Aroclor to nearest gram. Shake until the Aroclor is dissolved.

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