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ANALYTICAL METHODS - PLANT B

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Material: Trichlorbenzene
Pyranol Mixes
Aroclors
Test: Water Traces

Traces of water may be determined with Karl Fischer reagent.

The preparation and standardization of Karl Fischer reagent is described in the directions for General Method. Water Traces, FAL:MJW, 5/8/40, Plant B Analytical Methods.

Solvents are necessary to dissolve TCB, Pyranol and Aroclors to allow rapid and accurate titration. Direct titration with Fischer reagent when no solvent is present results in precipitation of salts which obscure the endpoint. With TCB, methanol is the only solvent necessary. However, with Pyranol mixes and Aroclors a mixture of benzol-methanol is necessary to obtain a clear solution with high fluidity in which the reagent is readily soluble.

TCB

To a dry 500 ml. Erlenmeyer flask is added 50 ml. of dry methanol which is titrated to a light brownish endpoint with Fischer reagent. 100 g. of TCB is weighed into the same flask and titrated to the same endpoint color to determine the moisture content of the TCB.

Examples

1) 50 ml. methanol - 0.9 ml. of 0.0028 g. H_2O/ml. reagent
100 g. TCB - 1.6 ml. " " " additional

$$\frac{1.6 \times .0028}{100} = 45 \text{ p.p.m., } 0.0045\%$$

2) Check against standards:
TCB, 799 g. containing 14 p.p.m. H_2O, was mixed with 5 ml. of standard methanol thus adding 0.025 g. or 36 p.p.m. of water.
Analysis: 100 g. requires 1.57 ml. of 0.0034 g. H_2O/ml. reagent, 52 p.p.m., an increase of 38 p.p.m.

This TCB, 494 g., was then mixed with 5 ml. of standard methanol adding 0.025 g. or 50.6 p.p.m. of water.
Analysis: 100 g. requires 3.0, 3.1 ml. of 0.0034 reagent, representing 102, 105 p.p.m., an average increase of 51.5 p.p.m.

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Pyranol Mixes and Aroclors

To a dry 500 ml. Erlenmeyer flask there is added 50 ml. of dry benzol and 50 ml. of dry methanol and the mixture is titrated to an endpoint with Fischer reagent. 100 g. of a Pyranol mix is weighed into the same flask, shaken to dissolve and titrated to the same endpoint color to determine the moisture content of the Pyranol.

A similar method is employed for Aroclors except 80 ml. of benzol is used along with 50 ml. of dry methanol. Both Aroclor and methanol-benzol may be warmed slightly, no more than 40°C to speed solution.

Examples:

1) Titre: 15.8 ml. of 0.00414 g. H_2O/ml. reagent for 50 ml. methanol and 50 ml. benzol
0.75 ml. of 0.00414 g. H_2O/ml. reagent for 100 g. Pyranol 1488.

$$\frac{0.75 \times .00414}{100} = 31 \text{ p.p.m. or } 0.0031\%$$

2) Check against standards:
5 ml. of standard methanol, 0.025 g. H_2O, was added to 100 g. Pyranol 1488 containing 31 p.p.m. of water. The increase in water content was 250 p.p.m. Reagent was .00368 g. H_2O/ml.

Titre: 5.7, 5.8 ml. for check runs for 50 ml. methanol and 50 ml. benzol.
7.7,7.8 ml. for 100 g. of Pyranol

$$\frac{7.75 \times .00368}{100} = 285 \text{ p.p.m. or } 0.0285\%$$

The increase shown by titration was 254 p.p.m, 102% of theory.

Summary

It is possible by this procedure to check standards to an accuracy of about 4%. Different operators can probably check each other to 8 to 10% without trouble.

If desired, the precision may be increased in the lower ranges, such as below 20 p.p.m., by titrating 50 ml. methanol and 100 g. trichlorbenzene to a brownish endpoint and using this solution as a color standard. By then titrating methanol or methanol-benzene to this color, adding TCB or Pyranol and titrating again to the same color, there should be no discrepancy between the endpoint colors. Check runs will agree within 2 p.p.m. when made in this manner. If these operations are carried out rapidly, the fading of endpoint color in the standard is negligible. At higher moisture contents the percentage error is smaller if either methanol or TCB endpoint is not run to exactly the same color and a color standard is not necessary.

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