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ANALYTICAL METHODS - PLANT B

Date: 2/18/41
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Ref: Karl Fischer, Angew.Chem 48, 394 (1935)

Material: Attapulgus Earth
Test: Water Content

Water may be determined in Fuller's or Attapulgus earth by titration with Karl Fischer reagent.

The preparation and standardization of Karl Fischer reagent is described in the directions for General Method, Water Traces, FAL:MJW, 3/8/40, Plant B Analytical Methods. This procedure is adapted from that described by Karl Fischer in the above reference.

To a dry 50 ml. Erlenmeyer there is added 10 ml. of dry methanol which is then titrated to a brown endpoint with Fischer reagent. 0.2-0.5 g. of Attapulgus earth is added and the suspension shaken vigorously while titrating slowly to a brown endpoint.

Example

1) Titre: 0.2 ml. of 0.0028 g. H_2O/ml.reagent for 10 ml. methanol
7.8,7.7 ml. of " " " for 0.5 g. earth

$$\frac{7.75 \times .0028}{0.5} \times 100 = 4.34\% \; H_2O$$

2) Titre: 0.2 ml. of 0.0028 g. H_2O/ml.reagent for 10 ml. methanol
1.5,1.7 ml1 of " " " for 0.5 g. earth

$$\frac{1.6 \times .0028}{0.5} \times 100 = 0.9\% \; H_2O$$

Check runs can be duplicated to 2% of the moisture content. The article by Karl Fischer lists the following items to demonstrate the adequacy of this method.

Material	Titrates, % H_2O	Loss in oven (120°C), % H_2O
Fuller's Earth	15.6	15.5
Earth, saturated	22.4	22.6
Earth, dried at 120°C	1.6	0.0

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2/19/41

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