Determination of HCl in Aroclor 1111

Scope

This method measures HCl concentrations from 0.01 meq/100g. to 6.0 meq/100g.

Principle

This method is intended for the determination of acidity attributable to HCl in the Aroclor 1111. It involves a total acid titration with standardized NaOH.

Reagents

1. Toluene, ASTM Grade
2. Sodium Hydroxide, 0.1N.
3. Hydrochloric Acid, 0.1N.
4. Nitrogen

Apparatus

1. 2-Burets, graduated in 0.1 ml divisions
2. Separatory funnels, 250 ml.
3. 150 ml, coarse, ASTM 40-60, Pyrex glass sintered funnels
4. Erlenmeyer Flasks, 250 ml.
5. No. 13-1/2 Rubber stopper with one hole
6. Pan Balance
7. Small glass funnels
8. Cotton

Procedure

1. Add by buret 10.0 ml. 0.1N NaOH to the separatory funnel.
2. Add 100 ml toluene to the separatory funnel.
3. Get total tare weight on funnel, toluene, and NaOH.
4. Pour or drain (not pipet) approx. 50 gm of Aroclor into the glass sintered funnel which is placed in the separatory funnel.
5. Place rubber stopper over glass sintered funnel and force the Aroclor thru the filter with nitrogen.
6. Reweigh separatory funnel to get the weight of the sample.
7. Extract sample with the NaOH layer.
8. Filter extract through cotton plug into the Erlenmeyer flask.
9. Wash organic layer with 50 ml. distilled H_2O.
10. Filter extract again and repeat H_2O wash and filter.
11. Add methyl red indicator.
12. If solution is red, titrate with 0.1N NaOH to methyl red end-point.
13. If solution is yellow, titrate with 0.1N HCl to methyl red end-point.

Calculations

if 0.1N HCl is used:

$$\text{meq/100g acid} = \frac{(10.0 - \text{ml } 0.1\text{N HCl used})\ (N)\ (100)}{\text{sample weight}}$$

if 0.1N NaOH is used:

$$\text{meq/100g acid} = \frac{(10.0 + \text{ml } 0.1\text{N NaOH used})\ (N)\ (100)}{\text{sample weight}}$$

Notes

The sample cannot be drawn with a pipet because the vacuum will draw off the HCl.

A qualitative test for HCl can be used with this procedure also. Alkacid test paper can be exposed to the fumes above the sample. Trace amounts of HCl can be detected by this method, down to 0.1 meq/100g.

Sample must be kept above 54°C. Otherwise, solids will form.