

Humic Acid Analysis

1. Scope:

This method may be used for solid samples containing at least 0.5% humic acid, and for liquids containing at least 0.05% humic acid.

2. Principle:

The humic acid(s) are dissolved by treatment with 0.5N sodium hydroxide and are then precipitated with hydrochloric acid.

3. Safety

Read all SDS before proceeding with analysis.

4. Apparatus and Equipment:

- 4.1 Centrifuge (9/5" radius)
- 4.2 100ml centrifuge bottles with screw caps
- 4.3 100°C drying oven
- 4.4 Mechanical shaker

5. Reagents and Supplies:

- 5.1 0.5N NaOH (20g/L)
- 5.2 1% NaOH (10g/L)
- 5.3 HCl, concentrated

6. Analysis:

- 6.1 Weigh an appropriate amount of finely ground sample into a 100ml wide mouth screw top centrifuge bottle to give approximately 500mg of dry humic acid precipitate.
- 6.2 Add 50ml 0.5N NaOH; seal tightly.
- 6.3 Shake on mechanical shaker for 1.5 hours for solid samples; 30 minutes for liquid samples.

- 6.4 Rinse the cap with 5-10ml 1% NaOH and centrifuge for 20 minutes at ~2000 rpm.
- 6.5 Decant supernatant liquid into a second, previously weighed centrifuge bottle.
- 6.6 Add 5-10ml 1% NaOH to first bottle, shake vigorously, and centrifuge.
- 6.7 Again, decant the supernatant into the second bottle. Discard the first bottle with the precipitate.
- 6.8 To the combined extracts in the second bottle, add enough HCl (~ 10ml) to lower the pH to ≤ 1 .
- 6.9 Centrifuge the sample for 20 minutes at ~2000 rpm.
- 6.10 Carefully decant and discard the liquid.
- 6.11 Add 25ml of distilled water previously adjusted to $\text{pH} \leq 1$ with HCl to the bottle, cap and shake vigorously to free all precipitate from the bottom, and centrifuge again.
- 6.12 Once more, carefully decant and discard the liquid.
- 6.13 Repeat steps 11-12 one final time.
- 6.14 Dry the bottle with humic acid overnight at 100-110°C.
- 6.15 Cool in a desiccator (2-3 hours) and weigh.

7. Calculations:

$$\% \text{ Humic Acid} = \frac{\text{Weight of Dried Residue}}{\text{Sample Weight}} \times 100$$

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