Humic Acid Method

1. **Scope**:

   This procedure is to be used for humic acid analysis of solid samples containing a minimum of 0.5% humic acid and for liquid samples containing a minimum of 0.05% humic acid.

2. **Principle**:

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

3. **Safety**:

   Care must be taken when working with acids and bases. Follow the proper safety procedures outlined in the Injury and Illness Prevention Program Manual.

   Refer to the Instruction Manual for the proper and safe use of the centrifuge.

4. **Interferences**:

   None

5. **Apparatus and Equipment**:

   - Centrifuge (9.5 inch radius)
   - Mechanical Shaker
   - Drying Oven (100 – 110°C)
   - Centrifuge Bottles with Screw Caps (100 ml)
   - General Laboratory Glassware

6. **Reagents and Supplies**:

   - 0.5N Sodium Hydroxide (NaOH)
   - 1% Sodium Hydroxide (NaOH)
   - Hydrochloric Acid, Concentrated (HCl)

7. **Standards Preparation**:

- Obtain reference material from the International Humic Substances Society.
- Use the reference material as received. Keep covered.

8. **Sample Preservation and Storage**:

- Store solid fertilizer samples in glass jars.
- Store liquid fertilizer samples in plastic bottles.

9. **Test Sample Preparation**:

- No additional test sample preparation is needed.
- Tumble the glass jar to mix the solid fertilizer sample prior to weighing.
- Shake the plastic bottle to mix the liquid fertilizer sample prior to weighing.

10. **Instrument Calibration**:

None

11. **Analysis**:

11.1 Weigh an amount of finely ground solid or well-mixed liquid fertilizer sample into a centrifuge bottle to give approximately 500 mg of dry humic acid precipitate. Record the weight.

11.2 Add 50 ml 0.5N NaOH. Cap and seal tightly.

11.3 Shake on mechanical shaker for 1.5 hours for solid samples or 30 minutes for liquid samples.

11.4 After shaking, rinse the cap with 1% NaOH.

11.5 Centrifuge for 20 minutes at ~ 2000 rpm.

11.6 Decant the supernatant into a second, previously weighed centrifuge bottle.

11.7 Add 5 – 10 ml 1% NaOH to the first bottle, shake vigorously, and centrifuge.

11.8 Combine this second supernatant with the first by decanting into the second centrifuge bottle. Discard the first bottle with the precipitate.

11.9 To the combined extracts in the second bottle, add enough concentrated HCl (approximately 10 ml) to lower the pH to ≤ 1.

11.10 Centrifuge the sample for 20 minutes at ~ 2000 rpm.

11.11 Carefully decant and discard the liquid.

11.12 Add 25 ml distilled H_2O previously adjusted to pH ≤ 1 (with HCl) to the bottle, cap and shake vigorously to free all precipitate from the bottom, and centrifuge.

11.13 Carefully decant and discard the liquid.

11.14 Repeat Steps 11.12 – 11.13 one final time.

11.15 Dry the bottle with humic acid overnight at 100 – 110°C.

11.16 Cool in a desiccator (2 – 3 hours) and weigh.

12. **QA/QC**:

Run a reference material obtained from the International Humic Substances Society or a well-characterized humic acid sample as a check sample. For the International Humic Substances Society Reference Material, an acceptable recovery is ≥ 90%. For a well characterized humic acid sample, an acceptable recovery is ± 10% of the average value (based on a minimum of 10 results).

The minimum reporting limit for solid samples is 0.5%, and for liquid samples is 0.05%.

13. **Calculations**:

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

14. **Discussion and References**:

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A.L. Page, Methods of Soil Analysis Part 2, American Society of Agronomy, Inc., Madison, Wisconsin, 1982

R.S. Swift, Methods of Soil Analysis Part 3, American Society of Agronomy, Inc., Madison, Wisconsin, 1996