Analysis of Humic Substances

Lawrence Mayhew
“Classical” Extraction of Humic Acids

- Solid Media
- Extract with Alkali
- Treat Soluble Extract with Acid

Humin (insoluble)

Humic Acids (ppt)

Fulvic Acids (soluble)
CDFA Method

- Solid Media
  - Humin (insoluble)
  - Extract with Alkali
    - Treat Soluble Extract with Acid
      - Humic Acids (ppt)
      - Fulvic Acids (soluble)
CDFA Method and It’s Variants

Humic Acids (ppt) Also contain Ash Oxidized materials pH is not standardized Not intended to be an analytical method

Flaws with CDFA method were published in 2009

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Solid Media

Extract with Alkali

Acidification

Humic Acids (ppt)

"Fulvic Acids"

DAX 8 Resin

Hydrophobic Fulvic Acids


Fulvic Fraction

Hydrophilic – attracted to water, usually soluble
mineral salts, polysaccharides, amino sugars, amino acids, proteins, lignins, tannins, fatty acids, carbohydrates

Lignosulfonates

Hydrophobic – water seemingly repelled by water

Fulvic Acids
HPTA Protocol Highlights

Humic Acids are extracted NaOH under N₂ (anoxic conditions)

Humic Acids are flocculated (ppt) at pH 1

Results are adjusted by ash content (ash-free basis)
i.e. Si, Al, Fe, Mg, Ca, Na, K, Ti, Mn, Ba

Fulvic acids are adsorbed onto a hydrophobic exchange resin to separate them from non-humic substances

Detect adulterated or fake products
H⁺ Exchange Column

DAX-8 Column
A New Standardized Method for Quantification of Humic and Fulvic Acids in Humic Ores and Commercial Products

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Increased use of humic substances in agriculture has generated intense interest among producers, consumers, and regulators for an accurate and reliable method to quantify humic acid (HA) and fulvic acid (FA) in raw ores and products. Here we present a thoroughly validated method, the new standardized method for determination of HA and FA contents in raw humate ores and in solid and liquid products produced from them. The methods used for preparation of HA and FA were adapted according to the guidelines of the International Humic Substances Society involving alkaline extraction followed by acidification to separate HA from the fulvic fraction. This is followed by separation of FA from the fulvic fraction by adsorption on a nonionic macroporous acrylic ester resin at acid pH. It differs from previous methods in that it determines HA and FA concentrations gravimetrically on an ash-free basis. Critical steps in the method, e.g., initial test portion mass, test portion to extract volume ratio, extraction quantification of humic and fulvic acids in humic ores and commercial products. This single laboratory validation (SLV) study was conducted under the guidance of the Association of American Plant Food Control Officials (AAPFCO) to validate a quantitative analytical method for analysis of HA and FA in commercial humic products. Until this work, there has been no validated analytical method for determining the quantity of HA and FA in any material.

The proposed NSM is intended to quantify HA and FA in solid and liquid commercial humic products, peat, soil, and humate-containing geological deposits. This method is based on a procedure for extracting HA and FA from natural materials. Like the method of Swift (2), the proposed method is a modified form of the “classical” technique described in detail by Stevenson (3). The classical method of extracting HA and FA from soil humus utilizes a strong base to extract the alkaline-soluble materials, and then, after removal of nonsoluble components, the alkaline solution is acidified to precipitate the HA. Waksman (4) credits Oden, a German scientist who worked to determine the chemical nature and
ISO/TC 134 Fertilizers and soil conditioners

Secretariat: ISIRI
Secretary: Mrs Mojdeh R. Tabari
Chairperson: Mr William L. Hall until end 2014
ISO Central Secretariat contact: Mrs. Jenny Pellaux
Creation date: 1969

Scope:
Standardization in the field of fertilizers and soil conditioners, that is, materials whose addition is intended to ensure or improve the nourishment of cultivated plants and/or to improve the properties of soils.

Total number of published ISO standards related to the TC and its SCs (number includes updates): 30
Number of published ISO standards under the direct responsibility of ISO/TC 134 (number): 30

Quick links
Work programme
(drafts and new work items of ISO/TC 134)

Business plans
Working area on ISOTC and Public information folder
Thank You

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