
**Fertilizers and soil conditioners —
Determination of humic and
hydrophobic fulvic acids
concentrations in fertilizer materials**

*Engrais et amendements minéraux basique - Détermination des
acides humique et fulvique*





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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Humic substances are present in all ecosystems: oceans, rivers, lakes, and top soils. Quantifying the amount of humic material present in these systems is essential for academic research and commercial applications, specifically agricultural soil and plant management.

The increased use of humic substances in agriculture has generated intense interest among producers, consumers, and regulators for a reliable method for quantification of the active ingredients in raw humic ores and commercial fertilizer products; specifically humic and fulvic acids. As both commercial trade and regulation of humic products are based on percentage (%) of the humic and fulvic acids in commercial humic products, use of % units instead of SI units is warranted, therefore incorporated into this standard.

This document establishes a method for the determination of humic acids (HA) and acidic hydrophobic fulvic acids (HFA). The method is based on an existing preparative procedure use by the International Humic Substances Society (IHSS) for extracting high purity HA and HFA from soil samples^[1], which is a modified form of the “classical” technique described in detail by Stevenson^[2]. The “classical” methods and the IHSS method were developed as preparative methods for the fractionation of soil organic matter; they were not intended to be used as quantitative analytical methods. The classical method of extracting humic acids and *fulvic acids* from soil humus utilize a “strong base” to extract the alkaline soluble materials, and then the alkaline extract solution is acidified to flocculate the humic acids, which appear to precipitate out of solution. The remaining substances in solution after alkaline and acid treatment were called *fulvic acids*.

This method modifies the “classical” technique in a number of ways:

- it determines the quantity of humic substances on an “ash free” basis (mineral salts excluded);
- the alkali extraction is done under anoxic conditions to reduce oxidation of the analytical sample during extraction;
- it defines the materials that are soluble in both alkali and acid as the Fulvic Fraction;
- it can differentiate products containing certain non-humic materials that some manufactures claim to contain humic substances;
- it further defines HFA as materials of low sulfur content^[3] that bind to a hydrophobic resin at pH 1^[4]^[5]^[6], instead the classical, and perhaps more common definition, for *fulvic acids* as materials that are defined as soluble in both acid and alkali solution. This stricter definition is necessary to distinguish HFA from mineral salts, polysaccharides, amino sugars, amino acids, proteins, acids, and carbohydrates that are extracted along with humic substances when using the “classical” method^[1]^[4].

See [Annex B](#) for information on ISO/CD 19822 interlaboratory study.

Fertilizers and soil conditioners — Determination of humic and hydrophobic fulvic acids concentrations in fertilizer materials

1 Scope

This document specifies the procedure for the analysis of humic acids and hydrophobic fulvic acids which is applicable to dry and liquid materials used as ingredients in commercial fertilizers, soil amendments, and geological deposits.

2 Normative References

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

hydrophobic fulvic acids

HFA

materials composed of less than 0,75 % elemental sulfur (S) that are soluble in aqueous alkaline and acid solution and are adsorbed at pH 1 onto a polymeric adsorbent resin of moderate polarity. The resin is of a type designed for adsorption of amphiphilic compounds having molecular weights typical of fulvic acids

3.2

fulvic fraction

alkali extracted portions of humic substances that are soluble in both alkali and acid aqueous solutions

3.3

humic acids

HA

alkali extracted humic substances that are insoluble in strongly acidic solution and will precipitate from the alkali extract in acid solutions of pH 1

3.4

humic substance

major organic constituent of natural organic matter consisting of complex heterogeneous mixtures of carbon-based substances formed by biochemical reactions during the decay and transformation of plant and microbial remains

3.5

lignosulfonates

amorphous light to dark brown powder or liquid derived from the sulfite pulping of softwoods. The lignin framework is a sulfonated random polymer of three aromatic alcohols: coniferyl alcohol, p-coumaryl alcohol, and sinapyl alcohol, of which coniferyl alcohol is the principle unit

4 Principles

4.1 This method determines ash-free quantities of HA and HFA gravimetrically after separation from their matrix.

4.2 The method of extracting HA and HFA utilizes a strong base to extract the alkaline-soluble materials, and then, after removal of non-soluble components, the alkaline solution is acidified to flocculate the HA.

4.3 The liquid supernatant remaining after the removal of the HA is called the Fulvic Fraction. The Fulvic Fraction, which can contain Hydrophobic Fulvic Acids (HFA), is treated to determine the quantity of HFA in the Fulvic Fraction by selective adsorption onto a methacrylic-ester resin designed to separate the HFA from non-humic compounds. The material retained by the hydrophobic resin is referred to in the literature as the hydrophobic acid fraction of soluble organic matter[3].

5 Warnings

5.1 Requirements

5.1.1 Good laboratory practices

Related standards (e.g. ISO/IEC 17025) should be followed at all times in regards to personal protective equipment (safety glasses, handling strong acids, hydrochloric acid) and alkali (sodium hydroxide).

5.1.2 Moisture control

Humic and fulvic acids are hygroscopic materials; it is critical to prevent absorption of moisture during the handling of dried materials.

5.3 Lignosulfonates

Lignosulfonates will damage the hydrophobic resin. This analytical method cannot differentiate between hydrophobic fulvic acids and lignosulfonates, therefore pre-screening for the presence of lignosulfonates is recommended for liquid products of unknown origin.

See [Annex A](#).

5.4 Temperature control

Do not exceed 65 °C when drying the humic and fulvic analytes. The analytes are subject to decomposition at higher temperatures.

6 Reagents

6.1 Sodium hydroxide solution, 0,1 M, dilute 3,99 g of 99,99 % purity NaOH in 1 l of deionized water.

6.2 Sodium hydroxide solution, 0,5 M, dilute 19,99 g of 99,99 % purity NaOH in 1 l of deionized water.

6.3 Hydrochloric acid solution, 6 M, dilute 12 M HCl with an equal part of deionized water.

6.4 Hydrochloric acid solution, 1 M, dilute 83,3 ml of 12 M HCl with 1 l of deionized water.

6.5 Hydrochloric acid solution, 0,1 M, perform a 1:10 dilution using 1 M HCl prepared in [6.4](#) with final volume of 1 l using deionized water.