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## Fertilizers and soil conditioners — Determination of monosilicic acid concentrations in nonliquid fertilizer materials

*Détermination des concentrations en silicium soluble dans les matières fertilisantes non liquides*

ICS: 65.080

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ISO copyright office  
CP 401 • Ch. de Blandonnet 8  
CH-1214 Vernier, Geneva  
Phone: +41 22 749 01 11  
Fax: +41 22 749 09 47  
Email: [copyright@iso.org](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards

adopted by the technical committees are circulated to the member bodies for voting. Publication as an

International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent

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ISO 19747 was prepared by Technical Committee ISO/TC 134 for fertilizers and soil conditioners.

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## Introduction

Although Silicon is ubiquitous in nature, making up a quarter of the earth's crust (1), not all forms of silicon found in soils or fertilizer products are soluble and plant-available (2,3). The form of silicon in soils that is soluble and available for plant uptake is monosilicic acid. Worldwide, it has been estimated that annual removal of silicon from soils during crop production can amount to 239-255 mega tons annually based on FAO 1998 global crop production estimates and a conservative annual increase of 1 % through 2012. Although the first U.S. patent on a solid Si fertilizer was issued in 1881 (4), fertilizer manufacturers, governmental regulators and consumers had no means of evaluating nonliquid silicon fertilizer materials for their monosilicic acid supplying capacity to meet and replace plant uptake needs.

The first research into the use of silicon fertilizers was reported in 1840 (5). Additionally, increased plant silicon concentrations were first associated with reductions in rice (*Oryza sativa* L.) blast disease (*Magnaporthe grisea* M.E. Barr) over a century ago in Japan (6). Since then, research has extended to other grasses and grains (e.g. barley (*Hordeum vulgare* L.) (7), corn (*Zea mays* L.) (8), oats (*Avena sativa* L.) (9), wheat (*Triticum aestivum* L.) (10), sugarcane (*Saccharum officinarum* L.) (11), pasture (12), turf grasses (13), and to dicotyledonous crops (e.g. cucumber (*Cucumis sativus* L.) (14), grapes (*Vitis vinifera* L.) (15), pepper (*Capsicum* L.) (16), pumpkin (*Curcubita pepo* L.) (17), soybean (*Glycine max* (L.) Merr.) (18), tomato (*Solanum lycopersicum* L.) (19)). Beneficial effects from silicon fertility have included increased stress tolerance (disease, insect, drought, salt, nutrient imbalance, UV-rays, low and high temperature) and yield increases with or without stress (2). Other benefits from silicon supplements to soils have included CO<sub>2</sub> sequestration (20), reductions in metals toxicity (21), and reduced phosphorus run-off while increasing phosphorus use efficiency (22).

Considering the extensive research, a growing market, and the potential benefits from silicon fertility to global agriculture; it is important that a standard method exist to enable regulation of nonliquid silicon fertilizer materials based on their monosilicic acid supplying capacity. This is the first method developed which correlates well with plant silicon uptake while using commonly available laboratory equipment at a reasonable cost for the analysis.

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# Fertilizers and soil conditioners — Determination of monosilicic acid concentrations in nonliquid fertilizer materials

## 1 Scope

This document establishes a method for the determination of monosilicic acid concentrations in nonliquid fertilizer materials. Monosilicic acid is reported as % Si.

This extraction method is applicable to the detection of monosilicic acid in nonliquid fertilizer products, blended products, and beneficial substances at silicon (Si) concentrations of 0,2 to 8,4 %, with a Limit of Detection (LOD) of 0.06 % Si, and a Limit of Quantification (LOQ) of 0,20 % correlating well with plant uptake.

This method is not recommended for liquid silicon fertilizer sources due to an expected low bias of Si recovery and low correlation with plant uptake.

## 2 Normative References

The following referenced documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696:1995, *Water for Analytical Laboratory Use — Specification and Test Methods*

ISO/DIS 8157, *Fertilizers and Soil Conditioners — Vocabulary*  
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## 3 Terms and Definitions

### 3.1

#### monosilicic acid

monosilicic acid is an inorganic molecule that is soluble in soil solution and is the form of silicon available for plant uptake

## 4 Principle

Monosilicic acid (reported as % Si) from nonliquid Si fertilizer sources is extracted at ambient room temperature using a dilute  $\text{Na}_2\text{CO}_3\text{-NH}_4\text{NO}_3$  extractant. The extractant solution is analyzed by manual spectrophotometry at 660 nm using the heteropoly blue method.

## 5 Safety

**5.1 General Requirements:** A minimum of standard laboratory personal protective equipment including safety glasses, gloves, and lab coats should be worn always. Consult individual SDS for chemicals listed and follow safety and handling conditions per individual SDS instructions.

## 6 Reagents

**6.1 General Requirements:** Protect from sunlight. Store all reagents in a manner consistent with general laboratory practices. Store in temperatures between 20-24 °C unless otherwise noted.

**6.2** Sodium Carbonate solution: 0,094 M- Fill a 19 l plastic dispensing bottle with 18 l water. Add 180 g anhydrous  $\text{Na}_2\text{CO}_3$ . Stir to dissolve.

**6.3** Ammonium Nitrate solution: 0,20 M- Fill a 19 l plastic dispensing bottle with 18 l water. Add 288 g  $\text{NH}_4\text{NO}_3$ . Stir to dissolve.

NOTE Ammonium Nitrate ( $\text{NH}_4\text{NO}_3$ ): Is a strong oxidizing agent and should not be used near flames, heating or ignition sources, combustible materials, or reducing agents to avoid the potential for combustion or explosion hazards.  $\text{NH}_4\text{NO}_3$  must be separated from all organic materials present within the laboratory. Check with governmental agencies for any additional regulatory licensing requirements before obtaining or using  $\text{NH}_4\text{NO}_3$ .

**6.4** Sodium Carbonate-Ammonium Nitrate extractant solution: 9,4 pH - Add 50 ml of each solution (6.1 & 6.2) to a plastic beaker, stir, and verify a pH of 9.4 for the mixed solution using a pH meter.

NOTE Mixing of Sodium Carbonate and Ammonium Nitrate: Flexible vinyl gloves are to be worn when mixing  $\text{Na}_2\text{CO}_3$  and  $\text{NH}_4\text{NO}_3$ , due to the caustic nature of this extractant. Care must be taken to avoid eye or skin contact. If contact is made with eyes or skin, flush immediately with clean tap water and seek medical attention. Clean up any spills immediately.

**6.5** Silicon 1000 mg/l Stock Solution: Silicon Standard - This solution preferably 2 % Sodium Hydroxide is commercially available from numerous sources. Follow specific manufacturer's SDS for proper storage and shelf life.

**6.6** Silicon spike solution: 500 mg/l- Pipette 50 ml of a stock 1000 mg/l Silicon standard into a 100 ml volumetric flask. Dilute to 100 ml with water.

**6.7** Ammonium Molybdate solution: 0,42 M- Add 75 g ammonium molybdate [ $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ ] to a 1 l beaker. Add 500 mL water. Dissolve. Slowly add 100 mL concentrated (18.4 M) sulfuric acid ( $\text{H}_2\text{SO}_4$ ). Cool. Transfer to a 1 l volumetric flask. Dilute to 1 l with water.

NOTE Handling of Concentrated Sulfuric Acid: Gloves, safety goggles, face shields, and lab coats should always be worn when handling concentrated sulfuric acid. Sulfuric acid is extremely corrosive and dehydrating, causing severe burns. Care must be taken to avoid eye or skin contact. If contact is made with eyes or skin, flush immediately with clean tap water and seek immediate medical attention. To avoid the potential for fuming and spattering of concentrated sulfuric acid during dilution, always add sulfuric acid to water and **do not** add water to sulfuric acid.

**6.8** Tartaric Acid solution: 1,33 M-M- Add 200 g tartaric acid to a 1 l beaker. Add 700 ml water. Stir. Transfer to a 1 l volumetric flask. Dilute to 1 l with water.

**6.9** Ascorbic Acid solution: 0,017 M- Add 3 g ascorbic acid to 1 l volumetric flask. Dilute to 1 l with water. Stopper the flask and mix by inverting 10 times. Transfer to a plastic storage bottle. Cap tightly and refrigerate.

**6.10** Intermediate Silicon standard solution: 50 mg/l- Dilute 5 ml of a stock 1000 mg/l Silicon standard to 100 mL in a volumetric flask. Transfer immediately to a plastic storage bottle.

## 7 Calibration Standards

**7.1** Blank: 0 mg Si/l- Add 10 mL sodium carbonate–ammonium nitrate extractant solution (6.4) to a 1 l volumetric flask. Dilute to 1 l with water. Stopper the flask and mix by inverting 10 times.

**7.2** Standard 1: 0,25 mg Si/l- Add 10 ml sodium carbonate–ammonium nitrate extractant solution (6.4) to a 1 L volumetric flask. Add 5 ml intermediate silicon standard solution (6.9). Dilute to 1 l with water. Stopper the flask and mix by inverting 10 times.



**7.3** Standard 2: 0,50 mg Si/l- Add 10 ml sodium carbonate–ammonium nitrate extractant solution (6.4) to a 1 l volumetric flask. Add 10 ml intermediate silicon standard solution (6.9). Dilute to 1 l with water. Stopper the flask and mix well by inverting 10 times.

**7.4** Standard 3: 1,0 mg Si/l- Add 10 ml sodium carbonate–ammonium nitrate extractant solution (6.4) to a 1 l volumetric flask. Add 20 ml intermediate silicon standard solution (6.9). Dilute to 1 l with water. Stopper the flask and mix by inverting 10 times.

**7.5** Standard 4: 2.0 mg Si/l- Add 10 ml sodium carbonate–ammonium nitrate extractant solution (6.4) to a 1 l volumetric flask. Add 40 ml intermediate silicon standard solution (6.9). Dilute to 1 l with water. Stopper the flask and mix by inverting 10 times.

**7.6** Spike Sample: Prepare a spiked talc sample by drying (9.1.1) grinding (9.1.2) and weighing out (9.1.3) a 0,2 g talc  $[Mg_3Si_4O_{10}(OH)_2]$  test portion sample. Add 100 ml each of sodium carbonate (6.2) and ammonium nitrate (6.3) solutions using a plastic graduated cylinder. Before shaking (9.2) add 3 mL Silicon spike solution (6.5) to the talc test sample. This talc-spiked test sample is processed and used for the matrix spike recovery test verifying that soluble (spike) rather than insoluble silicon (talc) is extracted and reported using this method.

## 8 Apparatus

As silica ( $SiO_2$ ) is a constituent of commonly used laboratory glassware, and detection limits are at 0,06 % Si, it is important to adhere to strict glassware cleaning instructions.

**8.1** Glassware: Glassware is to be thoroughly acid cleaned prior to use in reagent and calibration standard preparation by soaking in a 4 %  $HNO_3$  solution for 30 min and rinsing three times with deionized water. Allow glassware to air dry prior to use. Glassware items needed include 100 ml to 1 l volumetric flasks, 1 l beakers, and assorted 5 to 50 ml calibrated pipets. All glassware (including flasks and pipets) must conform to Class A tolerances as recommended for routine laboratory applications.

**8.2** Plastic ware: Plastic ware should be cleaned well with warm clean tap water, followed by triple rinsing with deionized water. Specific plastic ware items needed include: 50 to 1 l graduated cylinders, 200 ml Nalgene volumetric flasks with screw closure; 250 ml Nalgene Erlenmeyer flasks with screw closure; 50 ml centrifuge or test tubes with screw closure; 250 ml to 1 l Nalgene or fluorinated ethylene propylene (FEP) narrow-mouth storage bottles with screw closure; and 20 l wide-mouth dispensing bottles with screw closure (also Nalgene or FEP).

**8.3** pH meter: Accumet™ AB15 Basic pH/mV benchtop meter (Fisher Scientific) or equivalent.

**8.4** Analytical balance: Mettler AG200, accuracy to 0.1 mg (Mettler Toledo, Columbus, OH), or equivalent.

**8.5** Shakers

**8.5.1** Touch agitator: Thermolyne Maxi Mix II 12v Vortex Mixer, Model M37615 (Barnstead Thermolyne Corp., Dubuque, IA), or equivalent.

**8.5.2** Orbital platform shaker or Wrist-action shaker

**8.5.2.1** Orbital platform shaker: LabLine Orbit (Melrose Park, IL) shaker, model 3590 equipped with timer and adjustable rpm control, or equivalent.

**8.5.2.2** Wrist-action shaker: Burrell (Pittsburgh, PA), VWR, or equivalent.