



**International
Standard**

ISO 7431-1

**Thiourea for industrial use —
Part 1:
Test methods**

**First edition
2024-01**

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Published in Switzerland

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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 47, *Chemistry*.

A list of all parts in the ISO 7431 series can be found on the ISO website.

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

Thiourea (CAS¹⁾ No. 62-56-6), as a kind of chemical raw material, is widely used in the manufacture of drugs, dyes, resins, compression plastic powder, etc., and can also be used as vulcanization accelerator for rubber and flotation agent for metal minerals, especially in the field of high-folding optical resin materials.

Thiourea molecular weight: 76,12 (according to international relative atomic mass of 2019)

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1) Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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Thiourea for industrial use —

Part 1: Test methods

WARNING — Thiourea is environmental hazard and toxic by ingestion and skin absorption to humans. It is the responsibility of the users of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

The document specifies test methods for industrial thiourea properties, such as thiourea content, water insoluble substance, ignition residue, initial melting point, turbidity, thiocyanate content, calcium element and heavy metals.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3165, *Sampling of chemical products for industrial use — Safety in sampling*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6353-1:1982, *Reagents for chemical analysis — Part 1: General test methods*

ISO 7027-1, *Water quality — Determination of turbidity — Part 1: Quantitative methods*

ISO 7980:1986, *Water quality — Determination of calcium and magnesium — Atomic absorption spectrometric method*

ISO 8213, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps*

ISO 10304-3:1997, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 3: Determination of chromate, iodide, sulfite, thiocyanate and thiosulfate*

ISO 11885:2007, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

ISO 14911:1998, *Water quality — Determination of dissolved Li^+ , Na^+ , NH_4^+ , K^+ , Mn^{2+} , Ca^{2+} , Mg^{2+} , Sr^{2+} and Ba^{2+} using ion chromatography — Method for water and waste water*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology 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/>

4 Test methods

4.1 General

4.1.1 Without other specified requirements, the reagents used in test methods shall be analytical grade, and the water is the tertiary water specified in ISO 3696.

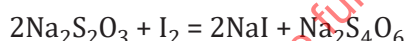
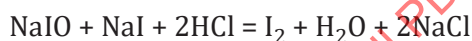
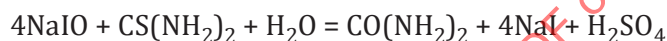
4.1.2 The standard volumetric solutions, impurity standard solutions, preparations and products used in the test methods are prepared in accordance with ISO 6353-1 when no other requirements are specified.

4.1.3 Sampling shall be in accordance with ISO 8213 and ISO 3165.

4.2 Determination of thiourea content

4.2.1 Principle

Under alkaline conditions, thiourea is quantitatively reacted with iodine, and the excess iodine is titrated with sodium thiosulfate standard volumetric solution.



4.2.2 Reagents

4.2.2.1 Hydrochloric acid solution: 1+2(V+V).

4.2.2.2 Sodium hydroxide solution: 40 g/L.

4.2.2.3 Iodine standard volumetric solution: $c(1/2 \text{I}_2) = 0,1 \text{ mol/L}$.

4.2.2.4 Standard sodium thiosulfate standard volumetric solution: $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/L}$.

4.2.2.5 Starch indicator solution: 10 g/L.

4.2.3 Apparatus

Ordinary laboratory apparatus and, the following shall be used.

4.2.3.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

4.2.3.2 Burette, of capacity 50 mL, graduated in 0,02 mL.

4.2.3.3 One-mark pipettes, of capacity 20 mL and 50 mL.

4.2.3.4 Graduated measuring cylinder, of capacity 100 mL.

4.2.3.5 Graduated pipettes, of capacity 10 mL, 20 mL, graduated in 0,1 mL.

4.2.3.6 Volumetric flask, of capacity 500 mL, fitted with ground glass stopper.

4.2.4 Procedure

4.2.4.1 Accurately weigh 0,50 g sample to the nearest 0,000 1 g and place in a 500 mL volumetric flask. Dissolve and dilute to the mark with water.

4.2.4.2 Pipette 20,00 mL thiourea solution into the iodine volumetric flask. Add 50,00 mL iodine standard volumetric solution and 20 mL sodium hydroxide solution, then place in the dark for 10 min prior to the addition of 100 mL water and 10 mL hydrochloric acid solution.

4.2.4.3 Titrate with the sodium thiosulfate standard volumetric solution. Near the titration endpoint, add 3 mL of starch indicator solution and continue titration until blue colour disappears.

4.2.4.4 A blank test shall be carried out in parallel by replacing the sample with water.

4.2.5 Calculation

The mass fraction of thiourea content w_1 , is calculated by using [Formula \(1\)](#):

$$w_1 = \frac{(V_0 - V) \times c \times M \times 25}{1\,000 \times m} \times 100 \% \quad (1)$$

where

V_0 is the volume of sodium thiosulfate standard volumetric solution consumed by blank test, mL;

V is the volume of sodium thiosulfate standard volumetric solution consumed by sample, mL;

c is the exact concentration of sodium thiosulfate standard volumetric solution, mol/L;

M is 1/8 of molar mass of thiourea, g/mol ($M=9,515$);

m is the mass value of sample, g;

25 is the sample dilution factor;

1 000 is the unit conversion factor.

4.2.6 Expression of results

The reported result is the arithmetic mean of two duplicate test results. The absolute differences between two duplicated tests shall not be greater than 0,2 %.

4.3 Determination of water insoluble substance

4.3.1 Principle

Dissolve the sample in water and filter. Then, dry and weigh the residue by mass difference.

4.3.2 Apparatus

4.3.2.1 Membrane filter, pore size diameter 0,45 μm .

4.3.2.2 Oven, capable of being maintained at 105 °C $\pm$ 2 °C.

4.3.3 Procedure

4.3.3.1 Weigh 100 g of sample to the nearest 0,000 1 g, and place in a 500 mL beaker. Add 200 mL water, to prepare an aqueous solution.

4.3.3.2 Filter through the membrane filter ([4.3.2.1](#)), previously dried for 1 h at 105 ± 2 °C, cooled in a desiccator and weighed to the nearest 0,1 mg.

4.3.3.3 Wash the residue with 60 °C 250 mL water (washing five times for 50 mL each), and operate in accordance with ISO 6353-1: 1982, 5.1

4.3.4 Calculation

The mass fraction of insoluble substances w_2 , is calculated by using [Formula \(2\)](#):

$$w_2 = \frac{m_1 - m_0}{m} \times 100 \% \quad (2)$$

where

m_1 is the total mass of the filter residue and the membrane filter, g;

m_0 is the membrane filter mass, g;

m is the sample mass, g.

4.3.5 Expression of results

The reported result is the arithmetic mean of two duplicated test results. The relative differences between the two duplicated tests shall not be greater than 20 %.

4.4 Determination of ignition residue

4.4.1 Principle

The sample of thiourea is heated until only ash carbon remains. After cooling, the residue is treated with sulfuric acid and heated at a low-temperature until oxygenated substances are completely volatilized and carbonized. Then heated at 850 °C to constant mass. The mass percentage of sulphated ash obtained is then calculated.

4.4.2 Apparatus

4.4.2.1 Quartz crucible, of capacity approximately 100 mL, flatbottomed.

4.4.2.2 Electric furnace, capable to provide condition at $850 \text{ °C} \pm 25 \text{ °C}$.

4.4.3 Procedure

Weigh 20 g of sample to the nearest 0,000 1 g and operate in accordance with ISO 6353-1:1982, 5.16. The ignition temperature is $850 \text{ °C} \pm 25 \text{ °C}$.

4.4.4 Calculation

The mass fraction of ignition residue w_3 , is calculated by using [Formula \(3\)](#):

$$w_3 = \frac{m_1 - m_0}{m} \times 100 \% \quad (3)$$

where

m_1 is the total mass of ignition residue and the crucible, g;

m_0 is the crucible mass, g;

m is the sample mass, g.

4.4.5 Expression of results

The reported result is the arithmetic mean of two duplicate test results. The relative differences between two duplicate tests shall not be greater than 20 %.

4.5 Determination of initial melting point

4.5.1 Principle

Determined by heating the sample in the capillary and observing the change of transmittance during the phase transformation.

4.5.2 Apparatus

4.5.2.1 Automatic melting point meter, with an accuracy of no less than 0,1 °C.

4.5.3 Procedure

Take the sample that dried at 105 °C to 110 °C and operate in accordance with ISO 6353-1:1982, 5.25.2. The initial temperature is 165 °C with a heating rate of 2 °C/min. Take the initial melting temperature as a result, accurate to the nearest 0,1 °C.

4.5.4 Calculation

The initial melting temperature is taken to report the results.

4.5.5 Expression of results

The reported result is the arithmetic mean of two duplicate test results. The absolute differences between two duplicated tests shall not be greater than 0,3 °C.

4.6 Determination of turbidity

Dissolve a sample in water to prepare a test portion with a concentration of 10 % (mass fraction) and operate in accordance with ISO 7027-1.

4.7 Determination of thiocyanate content

4.7.1 Principle

Separation of ions is carried out by liquid chromatography using a separating column due to the different relative affinity of various ions to the ion exchange resin. The sample flows through the conductivity cell, detected by conductivity (CD), ultraviolet (UV) or amperometric (AD) detectors, and draw the chromatogram

of each ion. The concentration of the respective ions is determined by calibration of the overall procedure. Particular cases may require calibration by means of standard addition (spiking).

4.7.2 Reagents

Laboratory reagents specified in ISO 10304-3:1997, 4.1.

4.7.3 Apparatus

Specified in ISO 10304-3:1997, 4.2.

4.7.4 Procedure

Weigh an appropriate amount of the sample to the nearest 0,000 1 g and place in a 100 mL volumetric flask. Dissolve and dilute to the mark with water. The procedure is carried out in accordance with ISO 10304-3:1997, 4.5.

4.7.5 Calculation

The mass fraction of thiocyanate w_4 , is calculated, by using [Formula \(4\)](#):

$$w_4 = \frac{pV \times 10^{-6}}{m} \times 100 \% \quad (4)$$

where

p is the concentration of thiocyanate in the test solution measured by ion chromatography, mg/L;

V is the dilution factor;

m is the sample mass, g.

4.7.6 Expression of results

The reported result is the arithmetic mean of two duplicate test results. The relative differences between the two duplicated tests shall not be greater than 20 %.

4.8 Determination of calcium element

4.8.1 General

One method in [4.8.2](#), [4.8.3](#) or [4.8.4](#) can be chosen for determination. In case of dispute, atomic absorption spectrometry in [4.8.4](#) shall be the referee method.

4.8.2 Ion chromatography

4.8.2.1 Reagents

Usual laboratory reagents and a stock solution with cation Ca^{2+} of concentration 1 000 mg/L (depending on the cation Ca^{2+} concentration expected, to prepare 5 to 10 standard solutions distributed over the expected working range as equally as possible).

4.8.2.2 Apparatus

Ion chromatography system and usual apparatus in accordance with ISO 14911:1998, Clause 7.