
**Rubber compounding ingredients —
Magnesium oxide — Methods of test**

*Ingrédients de mélange du caoutchouc — Oxyde de magnésium —
Méthodes d'essai*

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Foreword

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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 rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 21869 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

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Introduction

Magnesium oxide is used in the rubber industry as a stabilizer, as an agent for modifying the vulcanization process and to enhance the heat resistance of rubber articles. The performance of magnesium oxide in these roles is dependent on its particle size, surface properties and purity. This International Standard specifies the methods used to determine these properties. It is based on NF T 45-006 (France).

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Rubber compounding ingredients — Magnesium oxide — Methods of test

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies the test methods to be used for magnesium oxide intended for use in the rubber industry as a stabilizer and vulcanizing agent.

The choice of the properties to be determined and the values required shall be agreed between the interested parties.

2 Normative references

The following referenced documents are indispensable for the application 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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 3819, *Laboratory glassware — Beakers*

ISO 4652-1, *Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Part 1: Single-point procedures*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 18852, *Rubber compounding ingredients — Determination of multipoint nitrogen surface area (NSA) and statistical thickness surface area (STSA)*

3 Sampling

Sampling shall be carried out in accordance with ISO 15528.

4 Moisture, magnesium hydroxide and magnesium carbonate content

Two methods are included: thermogravimetry and oven heating.

4.1 Thermogravimetry

4.1.1 Procedure

The tests are performed on a thermogravimetric analyser capable of controlling temperature at $105\text{ °C} \pm 10\text{ °C}$, $390\text{ °C} \pm 20\text{ °C}$ and $750\text{ °C} \pm 50\text{ °C}$.

The tests are performed in either an air or a nitrogen flow of $100\text{ cm}^3/\text{min} \pm 20\text{ cm}^3/\text{min}$. The temperature increase rate should be between $20\text{ °C}/\text{min}$ and $40\text{ °C}/\text{min}$ while the temperature sweep shall go from ambient to 800 °C .

4.1.2 Expression of results

4.1.2.1 Moisture content (mass loss from ambient to 105 °C)

$$\text{Moisture content (\%)} = 100 \times (m_1 - m_2)/m_1$$

where

m_1 is the mass of the test portion, in grams;

m_2 is the mass after heating to within the 95 °C to 115 °C temperature range, in grams.

4.1.2.2 Magnesium hydroxide content (mass loss from 105 °C to 390 °C)

$$\text{Mg(OH)}_2 \text{ content (\%)} = 100 \times 3,2(m_2 - m_3)/m_1$$

where

m_1 and m_2 are as defined in 4.1.2.1;

m_3 is the mass after heating to within the 370 °C to 410 °C temperature range, in grams;

3,2 is the ratio between 58, the molecular mass of magnesium hydroxide, and 18, the molecular mass of water, calculated on the basis of the following reaction:



4.1.2.3 Magnesium carbonate content (mass loss from 390 °C to 750 °C)

$$\text{MgCO}_3 \text{ content (\%)} = 100 \times 1,9(m_3 - m_4)/m_1$$

where

m_1 is as defined in 4.1.2.1;

m_3 is as defined in 4.1.2.2;

m_4 is the mass after heating to above 700 °C , in grams;

1,9 is the ratio between 84, the molecular mass of magnesium carbonate, and 44, the molecular mass of carbon dioxide, calculated on the basis of the following reaction:



4.1.2.4 Precision

The repeatability of the mass measurements is $\pm 2\%$.

4.2 Loss in mass on heating

4.2.1 Moisture content

4.2.1.1 Apparatus

4.2.1.1.1 Weighing dish, low form, approx. 70 mm diameter and 30 mm high (tared).

4.2.1.1.2 Oven, controlled at $115\text{ °C} \pm 10\text{ °C}$.

4.2.1.1.3 Analytical balance, accurate to 0,1 mg.

4.2.1.2 Procedure

Weigh into the tared weighing dish 5 g of magnesium oxide to the nearest 1 mg.

Spread the test portion to form an even layer in the bottom of the weighing dish. Place the dish, without its cover, in the oven with the temperature previously set at $115\text{ °C} \pm 10\text{ °C}$ and dry to constant mass (to the nearest 1 mg).

On removal from the oven, always place the cover on the weighing dish. Allow to cool in a desiccator. Weigh.

The mass loss represents the moisture content.

4.2.1.3 Expression of results

The moisture content is given by the equation:

$$\text{Moisture content (\%)} = \omega_m = 100(\Delta m_1/m_{01})$$

where

Δm_1 is the mass loss after heating, in grams;

m_{01} is the original mass of the test portion, in grams.

4.2.2 Magnesium hydroxide content

4.2.2.1 Apparatus

4.2.2.1.1 Crucible (tared), platinum or porcelain. (If a porcelain crucible is used, it shall be heated to $390\text{ °C} \pm 20\text{ °C}$ and cooled in a desiccator before the test.)

4.2.2.1.2 Furnace, capable of reaching $450\text{ °C} \pm 20\text{ °C}$.

4.2.2.1.3 Analytical balance, accurate to 0,1 mg.

4.2.2.2 Procedure

Weigh into the tared crucible 2 g of magnesium oxide to the nearest 1 mg.

Place the crucible containing the magnesium oxide in the furnace set at $390\text{ °C} \pm 20\text{ °C}$.

If a porcelain crucible is used, raise the temperature gradually. When 390 °C is reached, maintain it for 2 h in an oxidative atmosphere. Remove the crucible from the furnace, allow to cool in a desiccator and weigh.

Repeat the calcination to verify that a constant mass was reached.

It is preferable to allow a porcelain crucible to cool slowly in the furnace before placing it in the desiccator.

The mass loss represents the moisture plus magnesium hydroxide content.

4.2.2.3 Expression of results

The magnesium hydroxide content is given by the equation:

$$\text{Mg(OH)}_2 (\%) = 3,2 \times [100(\Delta m_2/m_{02}) - \omega_m] = \omega_h$$

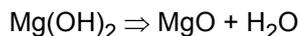
where

Δm_2 is the loss in mass after heating to 390 °C, in grams;

m_{02} is the original mass of the test portion, in grams;

ω_m is the moisture content (determined as specified in 4.2.1), in grams;

3,2 is the ratio between 58, the molecular mass of magnesium hydroxide, and 18, the molecular mass of water, calculated on the basis of the following reaction:



4.2.3 Magnesium carbonate content

4.2.3.1 Apparatus

4.2.3.1.1 Crucible (tared), platinum or porcelain. (If a porcelain crucible is used, it shall be heated to over 700 °C and cooled in a desiccator before the test.)

4.2.3.1.2 Furnace, capable of reaching over 700 °C.

4.2.3.1.3 Analytical balance, accurate to 0,1 mg.

4.2.3.2 Procedure

Weigh into the tared crucible 2 g of magnesium oxide to the nearest 1 mg.

Place the crucible containing the magnesium oxide in a furnace set at over 700 °C.

If a porcelain crucible is used, raise the temperature gradually. When 700 °C is reached maintain it for 2 h in an oxidative atmosphere. Remove the crucible from the furnace, allow to cool in a desiccator and weigh.

Repeat the calcination to verify that a constant mass was reached.

It is preferable to allow a porcelain crucible to cool slowly in the furnace before placing it in the desiccator.

The mass loss represents the moisture plus magnesium hydroxide plus magnesium carbonate content.

4.2.3.3 Expression of results

The magnesium carbonate content is given by the equation:

$$\text{MgCO}_3 (\%) = 1,9 \times [100(\Delta m_3/m_{03}) - \omega_m - \omega_h]$$

where

Δm_3 is the loss in mass after heating to over 700 °C, in grams;

m_{03} is the original mass of the test portion, in grams;

ω_m is the moisture content (determined as specified in 4.2.1), in grams;

ω_h is the magnesium hydroxide content (determined as specified in 4.2.2), in grams;

1,9 is the ratio between 84, the molecular mass of magnesium carbonate, and 44, the molecular mass of carbon dioxide, calculated on the basis of the following reaction:



4.2.4 Precision

With an analytical balance accurate to 0,1 mg for 5 g, the repeatability of the mass measurements can be considered as ± 1 %.

5 Determination of the specific surface area

The determination of the specific surface area shall be by the method described in ISO 4652-1 or ISO 18852.

6 Determination of the copper and manganese content

6.1 Principle

A test portion is dissolved in hydrochloric acid and the resulting solution is analysed using atomic absorption or atomic emission spectrometry. Any silicates that may be present are removed using hydrofluoric and sulfuric acids.

6.2 Reagents

During the analysis, unless stated otherwise, use only analytical-quality reagents and distilled water or water of equivalent purity.

6.2.1 Hydrochloric acid, $\rho_{20} = 1,19 \text{ Mg/m}^3$.

6.2.2 Hydrochloric acid, diluted 1+2.

Dilute 1 volume of hydrochloric acid (6.2.1) with 2 volumes of water.

6.2.3 Sulfuric acid, $\rho_{20} = 1,84 \text{ Mg/m}^3$.

6.2.4 Sulfuric acid, diluted 1+3.

Pour carefully 1 volume of concentrated sulfuric acid (6.2.3) into 3 volumes of water.

6.2.5 Hydrofluoric acid, $\rho_{20} = 1,13 \text{ Mg/m}^3$.

6.2.6 Hydrogen peroxide, 30 % (by mass) solution.

6.2.7 Copper, reference solution corresponding to 1 g of copper per cubic decimetre.

A commercially available copper reference solution may be used, or the solution may be prepared in the following manner:

Weigh, to the nearest 0,1 mg, 1 g of electrolytic copper (minimum purity: 99,9 %) and dissolve it in 50 cm³ of concentrated hydrochloric acid (6.2.1). Add 15 cm³ of hydrogen peroxide solution (6.2.6). After the copper has dissolved completely, decompose the excess hydrogen peroxide by boiling. Allow to cool and transfer to a one-mark 1 000 cm³ volumetric flask (6.3.5). Make up to the mark and mix well.

1 cm³ of this solution contains 1 000 µg of copper.

6.2.8 Copper, reference solution corresponding to 10 mg of copper per cubic decimetre.

Introduce precisely, using a pipette (6.3.6), 10 cm³ of copper reference solution (6.2.7) into a one-mark 1 000 cm³ volumetric flask (6.3.5), make up to the mark with diluted hydrochloric acid (1+2) (6.2.2) and mix well.

Prepare this reference solution on the same day as it will be used.

1 cm³ of this solution contains 10 µg of copper.

6.2.9 Manganese, reference solution corresponding to 1 g of manganese per cubic decimetre.

A commercially available manganese reference solution may be used, or the solution may be prepared in the following manner:

Use electrolytic manganese (minimum purity: 99,9 %), first cleaning its surface of oxides of manganese which may be present, as follows. Place a few grams of the metal in a beaker containing 60 cm³ to 80 cm³ of diluted (1+3) sulfuric acid (6.2.4) and about 100 cm³ of water. Shake and, after a few minutes, pour off the acid solution and pour water into the beaker. Wash repeatedly with water. Place the cleaned manganese metal in acetone and shake. Pour off the acetone and dry the metal in a hot-air oven (6.3.13) controlled at 100 °C ± 5 °C for about 2 min and allow to cool in a desiccator.

Weigh into a tall-form beaker, to the nearest 0,1 mg, 1 g of the cleaned manganese metal and dissolve it in 40 cm³ of diluted (1+3) sulfuric acid (6.2.4) and about 80 cm³ of water. Boil the solution for a few minutes. Allow to cool and quantitatively transfer to a one-mark 1 000 cm³ volumetric flask (6.3.5). Dilute to the mark and mix well.

1 cm³ of this solution contains 1 000 µg of manganese.

6.2.10 Manganese, reference solution, corresponding to 10 mg of manganese per cubic decimetre.

Introduce precisely, using a pipette (6.3.6), 10 cm³ of manganese reference solution (6.2.9) into a one-mark 1 000 cm³ volumetric flask (6.3.5), make up to the mark with diluted hydrochloric acid (1+2) (6.2.2) and mix well.

Prepare this reference solution on the same day as it will be used.

1 cm³ of this solution contains 10 µg of manganese.

6.3 Apparatus

Conventional laboratory equipment and the following:

6.3.1 Atomic absorption spectrometer, equipped with a burner using acetylene and air, pressurized to at least 60 kPa and 300 kPa, respectively; it shall also have a hollow copper cathode as a source of copper emission and a hollow manganese cathode as a source of manganese emission. The instrument shall be used in accordance with the manufacturer's instructions to ensure optimum performance.

As alternative equipment, an electrothermic atomizer (graphite oven) may be used. It shall be operated in accordance with the manufacturer's instructions to ensure optimum performance.

6.3.2 Analytical balance, accurate to 0,1 mg.

6.3.3 Muffle furnace, controllable at $550\text{ °C} \pm 25\text{ °C}$.

6.3.4 Sintered-glass filter, of porosity grade P40 (pore dimensions: 16 μm to 40 μm).

6.3.5 Volumetric flasks, one-mark, with ground-glass stoppers and with capacities of 50 cm^3 , 100 cm^3 , 200 cm^3 , 500 cm^3 and 1 000 cm^3 .

6.3.6 Pipettes, with capacities of 5 cm^3 , 10 cm^3 , 20 cm^3 and 50 cm^3 .

6.3.7 Electric hotplate or heated sand bath.

6.3.8 Steam bath.

6.3.9 Platinum wire, held in a borosilicate glass rod, for stirring.

6.3.10 Crucible, platinum, 50 cm^3 to 150 cm^3 capacity, depending on the size of the test portion.

6.3.11 Crucible, quartz or borosilicate glass, 50 cm^3 to 150 cm^3 capacity, depending on the size of the test portion.

6.3.12 Filter paper, ashless.

6.3.13 Oven, controllable at $100\text{ °C} \pm 5\text{ °C}$.

6.4 Sampling

Perform the sampling to obtain a sample representative of the whole lot.

6.5 Procedure

6.5.1 Test portion

Weigh, to the nearest 0,1 mg, approximately 10 g of sample into a crucible of suitable size (6.3.10 or 6.3.11). The size of the test portion shall be chosen as a function of the known or expected approximate copper and manganese content.

6.5.2 Preparation of the test solution

Add 20 cm^3 of concentrated hydrochloric acid (6.2.1) and heat the mixture over a steam bath for at least 10 min. Do not allow the reaction mixture to boil. Allow to cool to room temperature and quantitatively transfer the solution to a 50 cm^3 volumetric flask (6.3.5), using water to rinse. If dissolution of the material is not complete, proceed as follows:

Transfer the solution and the undissolved residue quantitatively to a platinum crucible (6.3.10), using water to rinse it. Add a few drops of concentrated sulfuric acid (6.2.3) and 5 cm³ of hydrofluoric acid (6.2.5). Heat on an electric hotplate or on a sand bath (6.3.7) under a fume hood; evaporate until dry when stirred with a platinum wire (6.3.9). Repeat this digestion twice more using the same quantities of sulfuric acid and hydrofluoric acid.

Allow to cool to room temperature, add 20 cm³ of concentrated hydrochloric acid (6.2.1), heat for 10 min and transfer quantitatively to a volumetric flask (6.3.5), using water to rinse.

Make up to the mark with water and mix well. If insoluble materials settle out, filter the solution through a sintered-glass filter (6.3.4) immediately before proceeding to 6.5.4.

6.5.3 Establishment of the reference curve

6.5.3.1 Preparation of the reference solutions

6.5.3.1.1 Using pipettes (6.3.6), introduce into a series of five 100 cm³ volumetric flasks (6.3.5) the amounts of copper reference solution (6.2.8) shown in Table 1. Dilute to the mark with diluted (1+2) hydrochloric acid (6.2.2) and mix well. Prepare manganese reference solutions in the same way using the amounts of manganese reference solution (6.2.10) shown in Table 2.

6.5.3.1.2 The reference solutions (6.5.3.1.1) shall be prepared daily.

Table 1 — Range of copper reference solutions

Volume of the copper solution (6.2.8) cm ³	Amount of copper in 1 cm ³ µg
50	5,0
20	2,0
10	1,0
5	0,5
0	0,0

Table 2 — Range of manganese reference solutions

Volume of the manganese solution (6.2.10) cm ³	Amount of manganese in 1 cm ³ µg
25	2,5
10	1,0
5	0,5
2	0,2
0	0,0

6.5.3.2 Spectrometric measurements

Turn on the spectrometer (6.3.1) at least 30 min before making any measurements to ensure that it is stable. The hollow copper or manganese cathode lamp shall be correctly positioned, the wavelength set at 324,7 nm for copper and at 279,5 nm for manganese and the sensitivity (slit opening) in conformity with the instrument's characteristics.

Set the air and acetylene pressures and flow rates in accordance with the manufacturer's instructions to obtain a clear blue non-bright oxidizing flame, suitable for the properties of the particular spectrometer used.

Aspirate the series of reference solutions (6.5.3.1.1) one after the other into the flame, reading the absorbance of each solution twice and determining the average of the readings. Ensure that the rate of aspiration remains constant during the course of this operation. The concentration of at least one of the reference solutions shall correspond to the concentration of the sample submitted to the test or be lower.

Aspirate water into the burner after each measurement.

6.5.3.3 Plotting the reference curve

Plot a curve showing, for example, the mass of copper, in micrograms, in 1 cm³ of the reference solution on the abscissa and the corresponding corrected absorbance values on the ordinate. [The absorbance values shall be corrected using the absorbance of the zero-level reference solution (see Table 1)]. Draw the best-fit curve through the points by visual judgement or calculate the curve using the least-squares method.

Plot the curve for manganese in the same way.

6.5.4 Copper and manganese levels

6.5.4.1 Spectrometric measurements

Perform the spectrometric measurements in duplicate at a wavelength of 324,7 nm for copper and 279,5 nm for manganese, using the test solution (6.5.2) and following the procedure described in 6.5.3.2.

6.5.4.2 Blank test

Perform a blank test in parallel with the determination of the concentration, using diluted (1+2) hydrochloric acid (6.2.2) but omitting the test portion.

If the preparation of the test solution requires the use of sulfuric acid and hydrofluoric acid, prepare the blank solution by repeating the whole procedure but omitting the test portion.

6.6 Expression of results

6.6.1 Read the copper or manganese content of the test solution directly from the reference curve (6.5.3.3). The copper or manganese content of the test portion, expressed as a percentage by mass, is then given by the following equation:

$$\omega(M) = [\omega(M)_t - \omega(M)_b]/200m$$

where

$\omega(M)_t$ is the copper or manganese content of the test solution (6.5.2), read from the reference curve, in micrograms per cubic centimetre;

$\omega(M)_b$ is the copper or manganese content of the blank solution (6.5.4.2), read from the reference curve, in micrograms per cubic centimetre;

m is the mass, in grams, of the test portion (see 6.5.1).

6.6.2 As a variation, if extrapolation of the linear portion of the reference curve intersects the axes at the origin and if the absorbance of the test solution falls within that linear portion, the copper or manganese content, expressed as a percentage by mass, is given by the following equation:

$$\omega(M) = [\omega(M)_t - \omega(M)_b]/200m$$

where

$\omega(M)_t$ is the copper or manganese content of the test solution, in micrograms per cubic centimetre, given by:

$$\omega(M)_t = \omega(M)_n(A_t/A_n)$$

$\omega(M)_b$ is the copper or manganese content of the blank solution, in micrograms per cubic centimetre, given by:

$$\omega(M)_b = \omega(M)_n(A_b/A_n)$$

where

A_t is the absorbance of the test solution;

A_b is the absorbance of the blank solution;

A_n is the absorbance of the reference solution having a copper or manganese content nearest to that of the test solution;

$\omega(M)_n$ is the copper or manganese content of the reference solution having an absorbance nearest to that of the test solution, in micrograms per cubic centimetre;

m is the mass, in grams, of the test portion (see 6.5.1).

6.6.3 Perform the test twice, using test portions taken separately from the same homogenized sample. The test result is the mean of the two values, rounded to two places of decimals if the concentration is expressed as a percentage and to the nearest whole number if the concentrations are expressed in milligrams per kilogram.

6.6.4 Report the copper or manganese content as a percentage if it is greater than or equal to 0,1 % or in milligrams per kilogram if it is below 0,1 %.

7 Determination of the sieve residue

7.1 Reagents

7.1.1 Sodium alkyl-aryl sulfonate, 0,5 % solution in water.

NOTE If it allows the determination to be carried out satisfactorily, water alone may be used.

7.2 Apparatus

7.2.1 Beaker, 400 cm³, tall form (in accordance with ISO 3819).

7.2.2 Sieve, with openings of 45 µm (in accordance with ISO 565).

7.2.3 Stirrer, with glass blades.

7.2.4 Glass rod, with a rounded end 6 mm in diameter.

7.2.5 Desiccator.

7.2.6 Analytical balance, accurate to 0,1 mg.

7.2.7 Oven, controlled at 105 °C ± 2 °C.

7.3 Procedure

Test portion: Weigh, to the nearest 1 mg, 10 g of magnesium oxide into a 400 cm³ beaker.

Testing: Wash and dry the sieve in an oven at 105 °C ± 2 °C. Tare it. Pour 300 cm³ of sodium alkyl-aryl sulfonate solution (7.1.1) or water into a beaker containing the test portion. Mix for 5 min with a stirrer turning at 260 rad/s (2 500 r/min). Pour the mixture onto the sieve. Rinse the beaker and pour the rinsings onto the sieve. Spray the sieve with a stream of water at a rate of 2,5 dm³/min. Using the rounded end of the glass rod, lightly scrape the bottom of the sieve until no more magnesium oxide is seen in the water flowing through the sieve. Dry the sieve in an oven at 105 °C ± 2 °C. Cool in a desiccator. Weigh to the nearest 1 mg.

Continue the drying operation until constant mass is reached.

7.4 Expression of the results

The sieve residue $\omega(\text{sr})$, expressed as a percentage, is calculated from the following equation:

$$\omega(\text{sr}) = 100(\omega_{t2} - \omega_{t1})/\omega_t$$

where

ω_{t1} is the mass, in grams, of the sieve;

ω_{t2} is the mass, in grams, of the sieve and the residue;

ω_t is the mass, in grams, of the test portion.

8 Determination of the chloride and sulfate contents

8.1 Determination of the chloride content

8.1.1 Principle

The chloride ions in a test portion are replaced by sulfate ions from magnesium sulfate. They are then precipitated from solution using silver nitrate. The end of the reaction is indicated by the precipitation of silver chromate caused by the presence of potassium chromate (argentometric titration).

8.1.2 Reagents

8.1.2.1 Hydrated magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$).

8.1.2.2 Silver nitrate solution (AgNO_3), 0,02 mol/l.

8.1.2.3 Potassium chromate (K_2CrO_4).

8.1.3 Apparatus

Conventional laboratory equipment and the following:

8.1.3.1 Analytical balance, accurate to 0,1 mg.

8.1.4 Procedure

Weigh, to the nearest 1 mg, 2 g of magnesium oxide into a 200 cm³ beaker. Add 15 cm³ to 30 cm³ of distilled water and 6 drops of potassium chromate (K_2CrO_4) solution. Then add 200 mg of hydrated magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$).