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International Standard ISO 1387

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Methanol for industrial use — Methods of test

Méthanol à usage industriel — Méthodes d'essai

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 1387 was developed by Technical Committee ISO/TC 47, *Chemistry*. It results form the combination into one single document of parts 1 to 7 of draft International Standard ISO/DIS 1387, which were circulated to the member bodies in December 1980.

It has been approved by the member bodies of the following countries:

Austria
Belgium
China
Czechoslovakia
Egypt, Arab Rep. of
France
Germany, F.R.
Hungary
India
Ireland*
Italy
Korea, Dem. P. Rep. of
Korea, Rep. of
Mexico
Netherlands**
Philippines
Poland
Portugal
Romania
South Africa, Rep. of
Switzerland
Thailand
United Kingdom***
USSR

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (UICPA).

It cancels and replaces ISO Recommendation R 1387-1970, of which it constitutes a technical revision.

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* Ireland did not vote on clause 10 (formerly part 2)

** The Netherlands disapproved clause 10 (formerly part 2)

*** The United Kingdom disapproved clauses 14 and 15 (formerly parts 6 and 7)

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Methanol for industrial use — Methods of test

1 Scope and field of application

This International Standard gives general instructions and specifies methods of test for the analysis of methanol for industrial use.

The methods of test relating to methanol for industrial use are the following:

- Determination of density at 20 °C
- Determination of dry residue after evaporation on a water bath
- Measurement of colour
- Determination of distillation yield
- Determination of water content
- Detection of the alkalinity or determination of the acidity to phenolphthalein
- Estimation of content of carbonyl compounds present in small amounts — Spectrometric method
- Estimation of content of carbonyl compounds present in moderate amounts — Titrimetric method
- Test for miscibility with water
- Determination of permanganate time
- Determination of methyl ketones — Iodometric method

NOTE — 1,10-Phenanthroline spectrometric methods for the determination of the total iron and non-volatile iron contents will be added later.

2 References

ISO 758, *Liquid chemical products for industrial use — Determination of density at 20 °C.*

ISO 759, *Volatile organic liquids for industrial use — Determination of dry residue after evaporation on a water bath — General method.*

ISO 760, *Determination of water — Karl Fischer method (General method).*

ISO 918, *Volatile organic liquids for industrial use — Determination of distillation yield — General method.*[1]

ISO 2211, *Liquid chemical products — Measurement of colour in Hazen units (platinum cobalt scale).*

General instructions

3 Sampling[2]

Place the laboratory sample in the dark, in a clean, dry and air-tight, ground glass stoppered bottle or a screw-capped bottle fitted with a polyethylene cone insert of such capacity that it is almost entirely filled by the sample. If it is necessary to seal the bottle, care shall be taken to avoid contaminating the contents in any way.

NOTE — A sample of not less than 1 000 ml is necessary for performing all the tests specified for the product.

4 Test report

The test report, for each determination, shall contain the following particulars :

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

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[1] At present at the stage of draft. (Revision of ISO/R 918.)

[2] The sampling of liquid chemical products for industrial use will form the subject of a future International Standard.

Methods of test

5 Determination of density at 20 °C

Use the method specified in ISO 758.

6 Determination of dry residue after evaporation on a water bath

Use the method specified in ISO 759.

7 Measurement of colour

Use the method specified in ISO 2211.

8 Determination of the distillation range

Use the method specified in ISO 918, with the following modifications appropriate to methanol.

8.1 Thermometer (see ISO 918, sub-clause 5.1.2)

Use a thermometer complying with the requirements of ISO 918 suitable for measuring temperatures in the range 24 to 78 °C.

8.2 Corrections to be applied to temperatures

If the corrected barometric pressure deviates from 1 013 mbar[1], apply a correction to the observed temperature by subtracting 0,025 °C for every millibar above, or adding 0,025 °C for every millibar below, 1 013 mbar (see ISO 918 clause 9).

9 Determination of water content

Use one of the methods specified in ISO 760.

10 Detection of alkalinity or determination of acidity to phenolphthalein

10.1 Applicability

The method is applicable to products having acidities, expressed as formic acid (HCOOH), greater than or equal to 0,000 8 % (*m*/*m*).

10.2 Principle

Dilution of a test portion with carbon dioxide-free water.

Checking whether the test solution is alkaline or acid to phenolphthalein, and, if appropriate, determination of the acidity by titration with standard volumetric sodium hydroxide solution.

10.3 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity, carbon dioxide-free, recently prepared.

10.3.1 Water, carbon dioxide-free.

Boil distilled water and allow it to cool in a flask fitted with a stopper carrying a soda-lime guard-tube.

10.3.2 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0{,}1$ mol/l.

10.3.3 Phenolphthalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of 95 % (*V*/*V*) ethanol and add the sodium hydroxide solution (10.3.2) until a pale pink coloration is obtained.

10.4 Apparatus

Ordinary laboratory apparatus, and

10.4.1 Conical flask, of borosilicate glass, of capacity 500 ml, fitted with a ground glass stopper carrying a soda-lime guard-tube.

10.4.2 Burette, of capacity 10 ml, graduated in 0,02 ml.

10.5 Procedure

10.5.1 Test portion

Take 100 ± 0,1 ml of the laboratory sample (see clause 3).

10.5.2 Determination

Place 100 ml of the water (10.3.1) in the conical flask (10.4.1), add 0,5 ml of the phenolphthalein solution (10.3.3) and restore the pale pink coloration by the addition of 1 or 2 drops of the sodium hydroxide solution (10.3.2). Add the test portion (10.5.1) and a further 0,5 ml of the phenolphthalein solution (10.3.3) and note whether the solution is alkaline; if acid, titrate the test solution with the sodium hydroxide solution (10.3.2), stoppering the flask and swirling its contents after each addition, until a pink coloration, persisting for about 15 s, is obtained. Shake the contents of the flask, with the stopper in position, after each addition of sodium hydroxide solution.

10.6 Expression of results

10.6.1 Alkaline products

Indicate whether the product is alkaline to phenolphthalein.

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[1] 1 bar = 10^5 Pa

10.6.2 Acidic products

The acidity, expressed as a percentage by mass of formic acid (HCOOH), is given by the formula

$$\frac{0{,}004\,6 \times V}{\varrho}$$

where

V is the volume, in millilitres, of the sodium hydroxide solution (10.3.2) used for the determination;

ϱ is the density, in grams per millilitre, of the sample at 20 °C (see clause 5);

0,004 6 is the mass, in grams, of formic acid corresponding to 1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 0{,}100$ mol/l.

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

11 Estimation of content of carbonyl compounds present in small amounts — Spectrometric method

11.1 Applicability

The method is applicable to products having carbonyl compounds contents, expressed as formaldehyde, between 0,000 2 and 0,01 % (*m*/*m*).

NOTE — This method, which is used commercially, allows determination of only those carbonyl compounds which react under the specified conditions.

11.2 Principle

Reaction in acid medium of the carbonyl compounds in a test portion with 2,4-dinitrophenylhydrazine. Formation of the corresponding 2,4-dinitrophenylhydrazones, which, after making the solution alkaline, take on a yellowish-red coloration.

Spectrometric measurement of this red coloration at a wavelength of about 445 nm.

11.3 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

11.3.1 Methanol, free from carbonyl compounds, purified as follows.

Boil under reflux 1 000 ml of methanol with 10 g of 2,4-dinitrophenylhydrazine and 10 drops of the hydrochloric acid solution (11.3.3), for 2 to 3 h. Distil the methanol slowly using a Widmer distillation column, about 300 mm long and about 25 mm in diameter, or any other suitable column. Reject the first 75 ml of distillate and collect the next 850 ml, rejecting the remainder. If the distillate is coloured, redistil it.

11.3.2 2,4-Dinitrophenylhydrazine, saturated solution in the methanol (11.3.1) at ambient temperature [about 0,06 % (*m*/*m*) solution].

11.3.3 Hydrochloric acid, ϱ approximately 1,19 g/ml, about 38 % (*m*/*m*) solution.

11.3.4 Potassium hydroxide, 100 g/l solution in a 70 % (*V*/*V*) solution of the methanol (11.3.1).

11.3.5 Carbonyl compounds, standard solution corresponding to 0,030 g of carbonyl compounds, expressed as formaldehyde, per litre.

Weigh, to the nearest 0,000 1 g, 1,200 g of acetophenone, and dissolve it in a little of the methanol (11.3.1). Transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark with methanol of the same quality and mix. Take 5,0 ml of this solution, transfer it to a 500 ml one-mark volumetric flask, dilute to the mark with the methanol (11.3.1) and mix.

1 ml of this standard solution contains 30 µg of carbonyl compounds, expressed as formaldehyde.

11.4 Apparatus

Ordinary laboratory apparatus, and

11.4.1 Water bath, capable of being controlled at 50 ± 2 °C.

11.4.2 Test tubes, fitted with ground glass stoppers.

11.4.3 Spectrometer with a radiation selector for continuous variation, fitted with cells of optical path length 1 cm, or

11.4.4 Spectrometer with a radiation selector for discontinuous variation, fitted with filters providing maximum transmission at a wavelength of about 445 nm.

11.5 Procedure

11.5.1 Test portion

Take 1,0 ml of the laboratory sample (see clause 3) and place it in one of the test tubes (11.4.2).

11.5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents used for the determination, but replacing the test portion by 1,0 ml of the methanol (11.3.1).

11.5.3 Preparation of the calibration graph

11.5.3.1 Preparation of dilute standard solutions, with a view to preparation of standard colorimetric solutions

Into a series of seven 25 ml one-mark volumetric flasks, introduce the volumes of the standard carbonyl compounds solution (11.3.5) indicated in the following table and dilute to the mark with the methanol (11.3.1).

Standard carbonyl compounds solution (11.3.5)	Corresponding mass of carbonyl compounds, expressed as HCHO	Mass of carbonyl compounds in 1 ml of dilute standard solution
ml	µg	µg
0*	0	0
1,5	45,0	1,8
2,5	75,0	3,0
5,0	150,0	6,0
7,5	225,0	9,0
10,0	300,0	12,0
12,5	375,0	15,0

* Compensation solution.

11.5.3.2 Preparation of standard colorimetric solutions, for spectrometric measurements carried out in cells of optical path length 1 cm

Into a series of seven of the test tubes (11.4.2), place 1,0 ml of each of the dilute standard solutions (11.5.3.1) and treat the contents of each tube according to the procedure specified in 11.5.3.3.

11.5.3.3 Colour development

Add 1,0 ml of the 2,4-dinitrophenylhydrazine solution (11.3.2) and one drop of the hydrochloric acid solution (11.3.3). Stopper the tubes and heat for 30 min on the water bath (11.4.1), controlled at 50 ± 2 °C. Allow to cool, add 5,0 ml of the potassium hydroxide solution (11.3.4), mix, and allow to stand for 5 min.

11.5.3.4 Spectrometric measurements

Immediately carry out the spectrometric measurements on each of the standard colorimetric solutions, using either the spectrometer (11.4.3), set at a wavelength of about 445 nm, or the spectrometer (11.4.4) fitted with appropriate filters, after having first adjusted the instrument to zero absorbance against the methanol (11.3.1).

11.5.3.5 Plotting the graph

Deduct the absorbance of the compensation solution from those of the standard colorimetric solutions (11.5.3.3). Plot a graph having, for example, the masses, in micrograms, of carbonyl compounds, expressed as HCHO, in 1 ml of each dilute standard solution (11.5.3.1) as abscissae, and the corresponding corrected values of absorbance as ordinates.

11.5.4 Determination

11.5.4.1 Colour development

Treat the test portion (11.5.1) in the test tube, following the procedure specified in 11.5.3.3.

11.5.4.2 Spectrometric measurements

Immediately carry out the spectrometric measurements on the coloured test solution and the blank test solution following the procedures specified in 11.5.3.4, after having adjusted the instrument to zero absorbance against the methanol (11.3.1).

NOTE — If the absorbance exceeds the maximum of the calibration graph, repeat the determination (11.5.4) using as the test portion 1,0 ml of a test solution prepared by diluting 1,0 ml of the laboratory sample with an appropriate volume (not more than 4,0 ml) of the methanol (11.3.1).

11.6 Expression of results

By means of the calibration graph (11.5.3.5), determine the masses of carbonyl compounds corresponding to the values of the spectrometric measurements.

The carbonyl compounds content, expressed as a percentage by mass of formaldehyde (HCHO), is given by the formula

$$\frac{(m_1 - m_0) \times 100}{1{,}0 \times \varrho \times 10^6} \times r_D$$

$$= \frac{m_1 - m_0}{\varrho \times 10^4} \times r_D$$

where

m_0 is the mass, in micrograms, of carbonyl compounds found in the blank test solution (11.5.2);

m_1 is the mass, in micrograms, of carbonyl compounds found in the test solution;

ϱ is the density, in grams per millilitre, of the sample at 20 °C (see clause 5);

r_D is the ratio of the volume of the diluted test solution (see the note to 11.5.4.2) to the volume of the aliquot portion taken for the determination (if the test portion was not diluted, r_D is equal to 1);

1,0 is the volume, in millilitres, of the test portion (11.5.1).

12 Estimation of content of carbonyl compounds present in moderate amounts — Titrimetric method

12.1 Applicability

The method is applicable to products having carbonyl compounds contents, expressed as formaldehyde, greater than or equal to 0,01 % (m/m).

NOTE — This method, which is used commercially, allows determination of only those carbonyl compounds which react under the specified conditions.

12.2 Principle

Reaction of hydroxylammonium chloride with the carbonyl compounds present in a test portion and titration of the hydrochloric acid formed with a standard volumetric sodium hydroxide solution in the presence of bromophenol blue as indicator.

12.3 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

12.3.1 Hydroxylammonium chloride reagent.

WARNING — Corrosive and irritating. Avoid contact with eyes and skin.

Dissolve 4 g of hydroxylammonium chloride in 20 ml of water and dilute to 200 ml with 95 % (*V*/*V*) ethanol. Heat under reflux for 30 min on a boiling water bath, cool to ambient temperature, add 5 ml of the bromophenol blue solution (12.3.4) and just sufficient of the sodium hydroxide solution (12.3.2) to produce a dichroic green coloration.

12.3.2 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0{,}1$ mol/l.

12.3.3 Hydrochloric acid, approximately 4 g/l solution.

12.3.4 Bromophenol blue, 2 g/l ethanolic solution.

Dissolve 0,2 g of bromophenol blue in 1,5 ml of the sodium hydroxide solution (12.3.2) and dilute to 100 ml with 95 % (*V*/*V*) ethanol.

12.4 Apparatus

Ordinary laboratory apparatus and

12.4.1 Conical flasks, of capacity 250 ml, fitted with ground glass stoppers.

12.5 Procedure

12.5.1 Test portion

Take 50 ± 0,1 ml of the laboratory sample (see clause 3) by means of a safety pipette and place it into one of the conical flasks (12.4.1).

12.5.2 Determination

Place 50 ml of the hydroxylammonium chloride reagent (12.3.1) in a second conical flask (12.4.1), to be used as the colour standard.

Add 1,25 ml of the bromophenol blue solution (12.3.4) to the flask containing the test portion (12.5.1) and add, drop by drop, either the sodium hydroxide solution (12.3.2) or the hydrochloric acid solution (12.3.3) until the colour matches that of the colour standard. Then add, to each of the flasks, 25 ml of the hydroxylammonium chloride reagent (12.3.1) and stopper the flask containing the colour standard.

Loosely stopper the flask containing the test solution and heat it for 10 min on a boiling water bath. Remove the flask from the water bath, cool to ambient temperature and titrate the solution with the sodium hydroxide solution (12.3.2) until the colour matches as closely as possible that of the colour standard.

12.6 Expression of results

The carbonyl compound content, expressed as a percentage by mass of formaldehyde (HCHO), is given by the formula

$$\frac{0{,}003\,0 \times V \times 100}{50 \times \varrho}$$

$$= \frac{0{,}006\,0\,V}{\varrho}$$

where

V is the volume, in millilitres, of the sodium hydroxide solution (3.2) used for the determination;

ϱ is the density, in grams per millilitre, of the sample at 20 °C (see clause 5);

0,003 0 is the mass, in grams, of carbonyl compounds, expressed as formaldehyde, corresponding to 1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 0{,}100$ mol/l sodium hydroxide solution;

50 is the volume, in millilitres, of the test portion (12.5.1).

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

13 Test for miscibility with water

13.1 Principle

Addition of water to a test portion, under specified conditions, and examination for opalescence.

13.2 Reagent

During the analysis, use only distilled water or water of equivalent purity.

13.3 Apparatus

Ordinary laboratory apparatus, and

13.3.1 Two matched Nessler cylinders, of capacity 100 ml.

13.4 Procedure

13.4.1 Test portion

Take 5 ml of the laboratory sample (see clause 3) or a different volume agreed between the interested parties.

13.4.2 Test

Place the test portion (13.4.1) in one of the Nessler cylinders (13.3.1), and dilute with water to the 100 ml mark. Mix and adjust the temperature to about 20 °C. Place 100 ml of water in the other Nessler cylinder.

Examine the cylinder containing the test solution vertically for opalescence, against a black background with side illumination, using the second Nessler cylinder containing water as the standard.

13.5 Expression of results

Report the dilution-ratio of the test portion and the presence or absence of opalescence.

14 Determination of permanganate time

14.1 Definition

permanganate time : The number of minutes required, after adding 2 ml of 0,2 g/l potassium permanganate solution to 50 ml of the sample, for the colour to match that of a colour standard.

14.2 Principle

Addition to a test portion, under specified conditions, of potassium permanganate solution. Determination of the time taken for the colour of this test solution to match that of a cobalt(II) chloride and uranyl nitrate colour standard.

14.3 Reagents

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

14.3.1 Potassium permanganate, 0,2 g/l solution.

Use water previously boiled for 30 min with sufficient dilute potassium permanganate solution to give a stable faint pink coloration. Cool the water to ambient temperature before preparation of the solution.

Prepare this solution immediately before use and protect it from light.

14.3.2 Cobalt(II) chloride and uranyl nitrate, colour standard.

To 5 ml of a 50 g/l solution of cobalt(II) chloride hexahydrate ($CoCl_2 \cdot 6H_2O$), add 7 ml of a 40 g/l solution of uranyl nitrate hexahydrate [$UO_2(NO_3)_2 \cdot 6H_2O$], and dilute with water to 50 ml.

Prepare this solution on the day of use.

14.4 Apparatus

NOTE — Clean the glassware used so as to avoid any risk of contamination.

Ordinary laboratory apparatus, and

14.4.1 Water bath, capable of being controlled at 15 ± 0,2 °C.

14.4.2 Two matched cylinders, of capacity 100 ml, of transparent and colourless glass, graduated at 50 ml and fitted with ground glass stoppers.

14.4.3 Burette, of capacity 10 ml, graduated in 0,05 ml divisions.

14.5 Procedure

14.5.1 Test portion

Carry out the test as soon as possible after receipt of the sample. (Instructions for the storage of the sample are specified in clause 3.)

Rinse one of the cylinders (14.4.2), first with 15 to 20 ml of hydrochloric acid, ϱ approximately 1,19 g/ml, about 38 % (*m*/*m*) solution, then six times with tap water, twice with distilled water and finally with some of the laboratory sample.

Immediately fill the cylinder to the mark with more of the laboratory sample at a temperature of about 15 °C.

14.5.2 Determination

Fill the second cylinder (14.4.2) to the mark with the colour standard (14.3.2).

Place the cylinder containing the test portion (14.5.1) in the water bath (14.4.1), controlled at 15 ± 0,2 °C, so that the water level in the bath is approximately 25 mm below the neck of the cylinder. After 15 min, remove the cylinder from the water bath and, using the burette (14.4.3), add 2,0 ml of the potassium permanganate solution (14.3.1). Note the time. Immediately stopper the cylinder, shake, and replace it in the water bath.

Remove the cylinder from the water bath, at intervals of 1 min and compare the colour, viewing vertically downwards against a white background, with the colour of the colour standard. Avoid exposing the test solution to strong daylight.

Note the time at which the colour of the test solution matches that of the colour standard.

14.6 Expression of results

Report the time, in minutes, from the addition of the potassium permanganate solution, for the colour of the test solution to match that of the colour standard.

15 Determination of methyl ketones — Iodometric method

15.1 Applicability

The method is applicable to products having methyl ketone contents, expressed as acetone, between 0,02 and 0,5 % (*m*/*m*).

15.2 Principle

Reaction of any methyl ketones present in a test portion with an excess of iodine in the presence of sodium hydroxide solution. Titration of the unreacted iodine with standard volumetric sodium thiosulphate solution.

NOTE — With this method, impurities other than acetone (such as $CH_3-\overset{|}{\underset{|}{C}}-OH$ compounds) are also partially or fully oxidized.

15.3 Reaction

$$(CH_3)_2CO + 3I_2 + 4NaOH \rightarrow$$
$$CHI_3 + CH_3COONa + 3NaI + 3H_2O$$

15.4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

15.4.1 Sodium hydroxide, 80 g/l solution.

15.4.2 Iodine, 12,7 g/l solution.

Place 12,7 g of iodine and 60 g of potassium iodide to an 800 ml beaker, add 30 ml of water, and stir until dissolution is complete. Dilute with water to about 500 ml and filter through a sintered-glass filter. Wash the filter with about 15 ml of water, transfer the combined filtrate and washings to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix. Store the solution in a glass-stoppered, amber-glass bottle in a cool place.

15.4.3 Sulphuric acid, 98 g/l solution.

15.4.4 Sodium thiosulphate, standard volumetric solution, $c(Na_2S_2O_3) = 0,1$ mol/l.

15.4.5 Starch, solution.

Triturate 1 g of soluble starch with 5 ml of water and, while stirring, pour the mixture into 100 ml of boiling water. Boil for a few minutes and cool.

Discard the solution after 2 weeks.

15.5 Apparatus

Ordinary laboratory apparatus, and

15.5.1 Conical flask, of borosilicate glass, capacity 500 ml, fitted with a ground glass stopper.

15.5.2 Burettes, of capacity 25 ml, graduated in 0,05 ml.

15.6 Procedure

15.6.1 Test portion

Take 50,0 ml of the laboratory sample (see clause 3) by means of a safety pipette and transfer it into a 1 000 ml one-mark volumetric flask containing about 500 ml of water. Shake, dilute to the mark and mix well.

Take as test portion, 100,0 ml of the solution corresponding to 5,0 ml of the laboratory sample.

15.6.2 Blank test

Carry out a blank test at the same time as the determination, using the same quantities of all the reagents [except for the sodium thiosulphate solution (15.4.4)] as used for the determination and replacing the test portion by 100 ml of water.

15.6.3 Determination

Transfer the test portion (15.6.1) into the 500 ml conical flask (15.5.1), containing 10 ml of the sodium hydroxide solution (15.4.1), and cool the mixture to about 15 °C. Add little by little from a burette, (15.5.2), 25,0 ml of the iodine solution (15.4.2), shaking continuously and keeping the temperature of the mixture at about 15 °C. Stopper the flask and keep it at about 15 °C for 1 h in the dark, shaking it occasionally. Then add 11 ml of the sulphuric acid solution (15.4.3) and titrate the excess iodine with the sodium thiosulphate solution (15.4.4) until the colour turns pale yellow. Then add 0,5 ml of the starch (15.4.5) and continue the titration until the blue colour is discharged.

15.7 Expression of results

The methyl ketone content, expressed as a percentage by mass of acetone $[(CH_3)_2CO]$, is given by the formula

$$\frac{(V_0 - V_1) \times 0{,}000\,967 \times 100}{5 \times \varrho}$$

$$= \frac{0{,}019\,34\,(V_0 - V_1)}{\varrho}$$

where

V_0 is the volume, in millilitres, of the sodium thiosulphate, solution (15.4.4) used to titrate the excess iodine solution (15.4.2) in the blank test solution (15.6.2);

V_1 is the volume, in millilitres, of the sodium thiosulphate solution (15.4.4) used for the determination (15.6.3);

ϱ is the density, in grams per millilitre, of the sample at 20 °C (see clause 5);

0,000 967 is the mass, in grams, of acetone corresponding to 1,00 ml of sodium thiosulphate solution, $c(Na_2S_2O_3) = 0{,}100$ mol/l.

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.