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# Light olefins for industrial use — Determination of traces of chlorine — Wickbold combustion method

*Oléfines légères à usage industriel — Dosage des traces de chlore — Méthode de combustion Wickbold*

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## Foreword

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8915 was prepared by Technical Committee ISO/TC 47, *Chemistry*.

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# Light olefins for industrial use — Determination of traces of chlorine — Wickbold combustion method

**WARNING — The procedure specified in this International Standard includes the combustion of hydrogen in stainless steel apparatus, which is potentially hazardous, and all necessary precautions must therefore be carefully observed.**

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## 1 Scope and field of application

This International Standard specifies a method for the determination of the organic chlorine present as traces in light olefins for industrial use, in the compressed, liquefied or liquid state.

The method is applicable to olefins having chlorine contents greater than 0,5 mg/kg.

## 2 References

ISO 4260, *Petroleum products and hydrocarbons — Determination of sulfur content — Wickbold combustion method.*

ISO 6227, *Chemical products for industrial use — General method for determination of chloride ions — Potentiometric method.*

ISO 7382, *Ethylene for industrial use — Sampling in the liquid phase and in the gaseous phase.*

ISO 8563, *Propylene and butadiene for industrial use — Sampling in the liquid phase.*

## 3 Principle

Aspiration of the gaseous or liquid test portion into the oxyhydrogen flame of a burner where it is burnt with considerable excess of oxygen.

Absorption of the liberated chlorine and hydrochloric acid in hydrogen peroxide solution.

Conductimetric or potentiometric titration of the chloride ions with a silver nitrate solution.

## 4 Reagents and materials

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade.

**4.1 Water, triple distilled,** with the third distillation over potassium hydroxide.

This water shall be used for the preparation of solutions and to wash the apparatus.

**4.2 Oxygen,** compressed gas, commercial grade, free from chlorine.

**4.3 Hydrogen,** compressed gas, commercial grade, free from chlorine.

**4.4 Acetone.**

**4.5 2,2,4-Trimethylpentane (Isooctane),** spectrometric U.V. grade.

**4.6 Hydrogen peroxide,** 3 % ($m/m$) solution, free from chlorine.

**4.7 Silver nitrate,** standard volumetric solution, $c(AgNO_3)$ = 0,002 mol/l.

Introduce into a 500 ml one-mark volumetric flask 100 ml of standard volumetric silver nitrate solution, $c(AgNO_3)$ = 0,01 mol/l, standardized by potentiometry (see ISO 6227) and adjusted, and dilute to the mark with the water (4.1).

1 ml of this solution corresponds to 71 µg of Cl.

**4.8 Inorganic chlorine,** standard solution corresponding to 50 mg of Cl per litre.

Introduce into a 1 000 ml one-mark volumetric flask, by means of a burette, 14,1 ml of standard volumetric hydrochloric acid solution, $c(HCl)$ = 0,1 mol/l, and dilute to the mark with the water (4.1).

1 ml of this standard solution contains 50 µg of Cl.

**4.9 Organic chlorine,** standard solution corresponding to approximately 1 g of Cl per litre.

Introduce into a 100 ml one-mark volumetric flask 140 mg of 1,2-dichloroethane, weighed to the nearest 0,1 mg, and dilute to the mark with the isooctane (4.5).

1 ml of this standard solution contains approximately 1 mg of Cl.

**4.10 Organic chlorine**, standard solution corresponding to approximately 10 mg of Cl per litre.

Introduce into a 100 ml one-mark volumetric flask 1 ml of the organic chloride standard solution (4.9) and dilute to the mark with the isooctane (4.5).

1 ml of this standard solution contains approximately 10 µg of Cl.

## 5 Apparatus

Ordinary laboratory apparatus and

**5.1 Combustion apparatus** (see sub-clauses 5.1 and 13.1 and figures 1 and 3 of ISO 4260). Use a stainless steel burner (see figure 3 of ISO 4260) terminating in a male conical ground assembling element containing a neck in which an O-ring is located. The burner is equipped with a valve (24) for admission of gaseous test portions and an aspiration tube equipped with a valve (25) for liquid test portions.

**5.2 Safety appliances.**

See sub-clause 5.2 of ISO 4260.

**5.3 Conductimeter, direct reading**, permitting correction for the original conductivity.

**5.4 Recorder**, coupled to the conductimeter.

**5.5 Automatic burette**, coupled to the unwinding of the chart paper.

**5.6 Electrode assembly**, with a constant of about 0,5 $cm^{-1}$. A suitable cell has two platinum electrodes (covered with black platinum) each 10 mm × 10 mm, spaced 10 mm apart.

**5.7 Titration vessel**, about 300 ml capacity, jacketted to permit thermostatic control.

**5.8 Thermostat** and **circulation pump**, to maintain the titration vessel at a temperature of 25 ± 0,1 °C.

**5.9 Magnetic stirrer.**

## 6 Sampling

The test sample shall be taken in accordance with ISO 7382 or ISO 8563.

## 7 Procedure

### 7.1 Safety measures

See sub-clause 13.2 of ISO 4260.

### 7.2 Cleaning of apparatus

See sub-clause 8.1 of ISO 4260.

### 7.3 Assembly and preparation of apparatus

See sub-clauses 8.2 and 8.3 of ISO 4260.

Ensure that the absorption solution reservoir is filled with the hydrogen peroxide solution (4.6).

### 7.4 Combustion and absorption

See sub-clauses 8.4, 8.5 and 8.6 of ISO 4260.

Carry out the ignition of a sufficient volume of the test portion as given in table 1.

**Table 1 — Relation between expected chlorine content, mass of test portion and duration of combustion**

| Expected chlorine content | Mass of test portion [1] | Maximum duration of combustion-absorption |
|---|---|---|
| mg/kg | g | min |
| 0 to 2,5 | 30 | 30 |
| 2,5 to 5 | 20 | 30 |
| 5 to 8 | 15 | 30 |
| 8 to 16 | 10 | 15 |
| 16 to 30 | 5 | 10 |
| 30 to 50 | 3 | 10 |

1) The volume of gas test sample required may be calculated with sufficient accuracy from the mass and density of the gas.

### 7.5 Combustion apparatus, check test

Determine the chlorine content of the standard organic chlorine solution (4.10), by taking a test portion of about 10 g and with a duration of the combustion-absorption process of 15 min maximum.

Carry out a blank test, proceeding as specified in 7.7 but substituting isooctane (4.5) for the standard organic chlorine solution (4.10). Then calculate the chlorine content using the formula given in 8.1.

The chlorine content obtained shall correspond, within ±1 mg/kg, to the content of the standard solution.

### 7.6 Determination

#### 7.6.1 Conductimetric titration

##### 7.6.1.1 Conductimeter check

Into a 50 ml graduated glass cylinder, introduce 2 ml of the inorganic chlorine standard solution (4.8) and 10 ml of the hydrogen peroxide solution (4.6) and dilute to 50 ml with the water (4.1). Carry out the measurement (7.6.1.2) on the resultant solution. The corresponding content shall be 100 ± 2 µg of Cl per kilogram.

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#### 7.6.1.2 Titration

Transfer the hydrogen peroxide solution in the one-mark volumetric flask (13) to the 50 ml graduated glass cylinder* and rinse the one-mark volumetric flask (sub-clause 5.1.1.1 of ISO 4260) with the water (4.1) to obtain 50 ml of solution in the graduated glass cylinder.

Transfer the solution in the graduated glass cylinder to the titration vessel (5.7). Rinse the graduated glass cylinder with 50 ml of the acetone (4.4), transfer the contents of the cylinder to the titration vessel and add a further 150 ml of the acetone. Connect the titration vessel to the thermostat and the circulation pump (5.8) and allow the solution to reach temperature equilibrium at 25 ± 0,1 °C. Stir with the magnetic stirrer (5.9).

Choose a suitable range on the conductimeter (5.3) and adjust to correct for the initial conductivity of the solution to be titrated.

Introduce the electrode assembly (5.6) into the solution. Start the automatic burette (5.5) and recorder (5.4) together and titrate with the silver nitrate solution (4.7).

The titration curve obtained has two nearly linear parts, the point of intersection of which gives the equivalence point, the abscissa giving the volume, in millilitres, of the silver nitrate solution (4.7) used.

### 7.6.2 Potentiometric titration

Titrate the 50 ml of solution according to the procedure specified in sub-clause 6.2.3 of ISO 6227.

## 7.7 Blank test

Carry out a blank test, burning the oxygen and hydrogen for the same time as taken to burn the test portion, and determine the chlorine content of the absorption solution by the procedure specified in 7.6.

The blank test is considered satisfactory if two consecutive determinations give values of less than 0,02 mg of Cl per kilogram.

If a greater blank value is obtained, rinse the combustion apparatus and repeat the blank determination with another set of reagents.

# 8 Expression of results

## 8.1 Calculation

The chlorine content (Cl), expressed in milligrams per kilogram, is given by the formula

$$\frac{(V_1 - V_0) \times m_1}{m_0}$$

where

$V_0$ is the volume, in millilitres, of the silver nitrate solution (4.7) used for the blank test (7.7);

$V_1$ is the volume, in millilitres, of the silver nitrate solution (4.7) used for the determination (7.6);

$m_0$ is the mass, in grams, of the test portion (see table 1);

$m_1$ is the mass, in micrograms, of chlorine corresponding to 1 ml of the silver nitrate solution (4.7).

## 8.2 Precision

### 8.2.1 Repeatability

The difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value $r$ (see table 2) on average not more than once in 20 cases in the normal and correct operation of the method.

**Table 2 — Repeatability conditions**

Values in milligrams per kilogram

| Chlorine content | $r$ |
|---|---|
| 10 | 0,8 |
| 1 | 0,2 |

### 8.2.2 Reproducibility

No data are currently available.

# 9 Test report

The test report shall include the following information:

a) all information necessary for the complete identification of the sample (lot, date, time and duration of each sampling, etc.);

b) a reference to this International Standard and to ISO 4260;

c) the results, with the reference to the method used (conductimetric or potentiometric) and the form in which they are expressed;

d) the results of the periodical check test carried out before the determination;

e) details of any unusual features noted during the determination;

f) details of any operations not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

* If the volume of hydrogen peroxide solution is greater than 50 ml, use a graduated glass cylinder of greater capacity and adjust the volume of acetone to obtain a water/acetone ratio of 1/4.

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