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# International Standard



# 3830

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## Petroleum products — Gasoline — Determination of lead content — Iodine monochloride method

*Produits pétroliers — Essence — Détermination de la teneur en plomb — Méthode au monochlorure d'iode*

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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 3830 was developed by Technical Committee ISO/TC 28, *Petroleum products*.

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 3830-1977), which had been approved by the member bodies of the following countries:

Australia	Ghana	Netherlands
Austria	Hungary	Portugal
Belgium	India	Romania
Brazil	Iran	South Africa, Rep. of
Bulgaria	Ireland	Sweden
Canada	Israel	Turkey
Czechoslovakia	Italy	United Kingdom
France	Japan	USA
Germany, F. R.	Mexico	USSR

No member body had expressed disapproval of the document.

# Petroleum products — Gasoline — Determination of lead content — Iodine monochloride method

## 1 Scope and field of application

This International Standard specifies a method for the determination of total lead in gasolines containing lead alkyls at concentrations between 0,03 and 1,0 g of lead per litre.

NOTE — The method is not applicable to gasoline containing manganese anti-knock additives.

## 2 Principle

A known volume of the sample is diluted with heavy distillate and shaken with aqueous iodine monochloride reagent. Any tetraalkyl lead compounds present react with the iodine monochloride and are extracted into the aqueous phase as the dialkyl lead compounds. The aqueous extract is separated from the gasoline and evaporated to low bulk to decompose free iodine monochloride. Any organic matter present is removed by oxidation with nitric acid, which also serves to convert the dialkyl lead compounds into inorganic lead compounds. The residue is dissolved in water and buffered to pH 5 with sodium acetate-acetic acid buffer. The lead content of the buffered solution is determined by titration with EDTA with xylenol orange as indicator.

## 3 Reagents

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

**3.1 Nitric acid**, concentrated ( $\rho_{20}$  1,42 g/ml).

**3.2 Ammonia**, solution (1 + 1).

Mix 1 volume of concentrated ammonia solution ( $\rho_{20}$  0,880 g/ml) with 1 volume of water.

**3.3 Heavy distillate**. A straight-run petroleum distillate having a maximum bromine number of 1,5 with approximately 10 % distilling at 205 °C and 90 % at 240 °C. It should also be lead free, having been, if necessary, previously extracted with the iodine monochloride solution (3.5).

**3.4 Sodium acetate-acetic acid buffer solution**.

Dissolve 23,0 g of anhydrous sodium acetate in about 500 ml of water. Using a burette, add 7,2 ml of glacial acetic acid. Dilute to the mark with water in a 1 000 ml one-mark volumetric flask and shake to mix.

**3.5 Iodine monochloride reagent**, 1,0 mol/l solution.

Dissolve 111,0 g of potassium iodine (KI) in approximately 400 ml of water. Add 445 ml of concentrated hydrochloric acid ( $\rho_{20}$  1,18 g/ml) and allow to cool to ambient temperature. Add 75,0 g of potassium iodate (KIO<sub>3</sub>) slowly and with stirring, until all free iodine initially formed has just redissolved to give a clear orange-red solution (the amounts of KI and KIO<sub>3</sub> are calculated to give a slight excess of iodate; if a greater excess is present, this will lead to precipitation of lead and indifferent end-points in the EDTA titration). Allow to cool to ambient temperature and dilute to 1 000 ml with water. Store in a glass-stoppered bottle.

### NOTES

1 Rubber bungs must never be used to stopper vessels containing iodine monochloride solutions.

2 Iodine monochloride will react with ammonium ions under certain conditions to yield explosive nitrogen tri-iodide. Care must be taken, therefore, that this reagent does not come into contact with ammonia or ammonium salts.

**3.6 Lead nitrate**, 0,005 mol/l standard solution.

Weigh, to the nearest  $\pm 0,001$  g, about 1,7 g of lead nitrate [Pb(NO<sub>3</sub>)<sub>2</sub>] that has been dried at 105 °C and allowed to cool in a desiccator. Dissolve it in water and add 10 ml of the concentrated nitric acid (3.1). Dilute to the mark with water in a 1 000 ml one-mark volumetric flask and shake thoroughly to mix.

Calculate the concentration  $c_0$  of the lead nitrate solution, in moles per litre, according to the equation

$$c_0 = \frac{m}{331,23}$$

where  $m$  is the mass, in grams, of lead nitrate dissolved.

**3.7 Disodiummethylenedinitrilotetraacetate dihydrate** ( $\text{Na}_2\text{EDTA}$ ), 0,005 mol/l standard volumetric solution.

### 3.7.1 Preparation

Dissolve approximately 3,75 g of the  $\text{Na}_2\text{EDTA}$  in 2 000 ml of water.

### 3.7.2 Standardization

Using a pipette, transfer 25,0 ml of the standard lead nitrate solution (3.6) to a 250 ml conical flask. Dilute to about 75 ml with water and add several drops of the bromothymol blue indicator solution (3.9). Titrate with the ammonia solution (3.2) until the colour of the solution just changes from yellow to blue, then add 10 ml of the sodium acetate-acetic acid buffer solution (3.4) and 5 drops of the xylenol orange indicator solution (3.8). In the presence of lead the solution will have a plum-red colour. Titrate with the  $\text{Na}_2\text{EDTA}$  solution (3.7.1). The colour changes to orange near the end-point, which is indicated by a sharp change from orange to a permanent bright lemon-yellow. Record the volume used and calculate the concentration of the  $\text{Na}_2\text{EDTA}$  solution. The addition of excess  $\text{Na}_2\text{EDTA}$  solution produces no further colour change at the end-point.

### 3.7.3 Calculation

Calculate the concentration  $c_1$  of the  $\text{Na}_2\text{EDTA}$  solution to the nearest 0,000 01 mol/l according to the equation

$$c_1 = \frac{25 c_0}{V}$$

where

$c_0$  is the concentration, in moles per litre, of the standard lead nitrate solution (3.6);

$V$  is the volume, in millilitres, of the  $\text{Na}_2\text{EDTA}$  solution used in the standardization.

### 3.8 Xylenol orange indicator solution.

Dissolve 0,2 g of xylenol orange, sodium salt, in 100 ml of water and add 1 drop of 1 + 1 hydrochloric acid ( $\rho_{20}$  1,18 g/ml).

Prepare freshly each week.

### 3.9 Bromothymol blue indicator solution.

Dissolve 0,1 g of bromothymol blue in 80 ml of 95 % (V/V) ethanol and dilute to 100 ml with water.

## 4 Apparatus

Ordinary laboratory apparatus including

**4.1 Separating funnel**, of borosilicate glass, glass stoppered, of capacity 250 ml.

**4.2 Conical flask**, borosilicate glass, wide mouthed, capacity 500 ml.

**4.3 Watch glass**, borosilicate glass, ribbed, of a size sufficient to cover the mouth of the conical flask (4.2).

NOTE — Although ribbed watch glasses may not be readily available in all countries, they have been found to significantly reduce the time required for evaporation of aqueous phases containing the extracted lead.

## 5 Procedure

**5.1** Transfer 50 ml of the iodine monochloride reagent (3.5) and 25 ml of the heavy distillate (3.3) to the separating funnel (4.1). Measure the temperature of the sample to the nearest 0,5 °C (see note 1). Using a pipette (see note 2), transfer  $25 \pm 0,05$  ml of the sample of the gasoline to the separating funnel. Immediately stopper the funnel and shake the contents for 1 min. Allow the funnel to stand for several minutes, until the two phases have separated, and run the lower aqueous phase into the conical flask (4.2). Wash the gasoline phase by shaking with three separate 20 ml portions of water and add the washings to the conical flask.

### NOTES

1 For gasoline having a Reid vapour pressure above 0,5 bar (50 kPa), the sealed sample container should be cooled to approximately 15 °C before removing the test sample for analysis.

2 **Leaded gasoline or corrosive liquids should not be sucked into a pipette by the mouth.**

**5.2** Add a few glass beads, cover the conical flask with the watch glass (4.3) and bring the aqueous solution to a low-boiling condition on a hot-plate. When the volume of solution has been reduced to 15 to 20 ml, slowly add, without removing the flask from the hot-plate, 5 ml of the nitric acid (3.1) down the side of the flask and evaporate the contents almost to dryness to oxidize any organic material present. Repeat the nitric acid treatment until a white residue remains. Finally, remove the watch glass and evaporate the solution to dryness. Remove the flask from the hot-plate and allow the contents to cool.

**5.3** Add about 200 ml of water to the flask and swirl to dissolve the residue. The residue may be quickly dissolved by heating the solution, but this must be cooled before titrating. Add several drops of the bromothymol blue indicator solution (3.9) and titrate with the ammonia solution (3.2) until the colour just changes from straw yellow to blue, then add 10 ml of the sodium acetate-acetic acid buffer solution (3.4) and 5 drops of the xylenol orange indicator solution (3.8). In the presence of lead, the solution will now have a plum-red colour.

**5.4** Titrate with the standard volumetric  $\text{Na}_2\text{EDTA}$  solution (3.7). The colour of the solution changes to orange near the end-point, which is indicated by a sharp change from orange to a permanent bright lemon-yellow. Record the volume used. The addition of excess standard volumetric  $\text{Na}_2\text{EDTA}$  solution produces no further colour change at the end-point.

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**5.5** Carry out a blank determination on the reagents, omitting the extraction stage and record the volume used.

## 6 Expression of results

### 6.1 Calculation

Calculate the concentration of lead, in grams per litre at 15 °C, by means of the following equation (see the note) :

$$8,288 (V_1 - V_0) c_1 [1 + 0,001 2 (t - 15)]$$

where

$V_0$  is the volume, in millilitres, of standard volumetric  $\text{Na}_2\text{EDTA}$  solution used for the blank test (5.5);

$V_1$  is the volume, in millilitres, of standard volumetric  $\text{Na}_2\text{EDTA}$  solution used to titrate the test portion;

$c_1$  is the concentration, in moles per litre, of the standard volumetric  $\text{Na}_2\text{EDTA}$  solution;

$t$  is the temperature, in degrees Celsius, of the gasoline when pipetting the sample.

Record the result to the nearest 0,002 g of lead per litre

NOTE — For gasoline containing only tetraethyl lead (TEL) or tetramethyl lead (TML) the grams of lead per unit volume can be converted to millilitres per unit volume by multiplying by the following factors :

For TEL = 0,946

For TML = 0,648

### 6.2 Precision<sup>1)</sup>

The precision of the method, as obtained by statistical examination of inter-laboratory test results, is as follows :

### 6.2.1 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty :

$$0,003 65 + 0,007 3 A$$

where  $A$  is the average of the results, in grams of lead per litre at 15 °C.

### 6.2.2 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty :

$$0,013 5 + 0,027 A$$

where  $A$  is the average of the results, in grams of lead per litre at 15 °C.

## 7 Test report

The test report shall contain at least the following information :

- the type and identification of the product tested;
- a reference to this International Standard or to a corresponding national standard;
- the result of the test (see 6.1);
- any deviation, by agreement or otherwise, from the procedure specified;
- the date of the test.

1) The precision of this International Standard was obtained in an ISO co-operative test program on samples covering a range of 0,3 to 1,0 g of lead per litre. In a subsequent ASTM testing program the range was extended down to 0,03 g of lead per litre with equal or better precision. The precision was established using dimethyl yellow indicator solution in place of bromothymol blue. Limited testing has shown that the change of indicator solution does not affect the precision.

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