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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

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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.

International Standard ISO 3830 was drawn up by Technical Committee ISO/TC 28, *Petroleum products*, and was circulated to the Member Bodies in June 1975.

It has 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	U.S.A.
Germany	Mexico	U.S.S.R.

No Member Body 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 to 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 1 l with water in a 1 l one-mark volumetric flask and shake to mix.

**3.5 Iodine monochloride reagent 1,0 M solution**.

Dissolve 111,0 g of potassium iodide (KI) in approximately 400 ml of water. Add 445 ml of concentrated hydrochloric acid ( $\rho_{20}$  1,18 g/ml) and cool to room temperature. Add 75,0 g of potassium iodate ( $\text{KIO}_3$ ) 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  $\text{KIO}_3$  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). Cool to room temperature and dilute to 1 l 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 M standard solution**.

Weigh, to the nearest  $\pm 0,001$  g, about 1,7 g of lead nitrate [ $\text{Pb}(\text{NO}_3)_2$ ] that has been dried at 105 °C and cooled in a desiccator. Dissolve it in water and add 10 ml of the concentrated nitric acid (4.1). Dilute to 1 l with water in a 1 l one-mark volumetric flask and shake thoroughly to mix.

Calculate the molarity  $T_0$  of the lead nitrate solution according to the equation

$$T_0 = \frac{m}{331,23}$$

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

**3.7 EDTA (disodium salt), 0,005 M standard volumetric solution.**

### 3.7.1 Preparation

Dissolve approximately 3,75 g of (ethylenedinitrilo)-tetraacetic acid, disodium salt, dihydrate (EDTA disodium salt) in 2 l 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 dimethyl yellow indicator solution (3.9). Titrate with the ammonia solution (3.2) until the colour of the solution just changes from red to yellow, 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 disodium 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 molarity of the disodium EDTA solution. The addition of excess disodium EDTA solution produces no further colour change at the end-point.

### 3.7.3 Calculation

Calculate the molarity  $T_1$  of the disodium EDTA solution to the nearest 0,000 01 M according to the equation

$$T_1 = \frac{25 T_0}{V}$$

where

$T_0$  is the molarity of the standard lead nitrate solution (3.6);

$V$  is the volume, in millilitres, of the disodium 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 Dimethyl yellow indicator solution.

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

## 4 APPARATUS

Ordinary laboratory apparatus and

**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 shall 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 dimethyl yellow indicator solution (3.9) and titrate with the ammonia solution (3.2) until the colour just changes from red to yellow, 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 disodium 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 disodium EDTA solution produces no further colour change at the end-point.

5.5 Carry out a blank determination on the reagents, omitting the extraction stage and, if necessary, correct the volume used for the test portion with the volume used for the blank titration.

## 6 EXPRESSION OF RESULTS

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

$$8,288 (V_1 - V_0) T_1 [1 + 0,001 2 (t - 15)]$$

where

$V_0$  is the volume, in millilitres, of standard volumetric disodium EDTA solution used for the blank test (5.5);

$V_1$  is the volume, in millilitres, of standard volumetric disodium EDTA solution used to titrate the test portion;

$T_1$  is the molarity of the standard volumetric disodium EDTA solution;

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

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

## 7 PRECISION<sup>1)</sup>

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

### 7.1 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.

### 7.2 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.

## 8 TEST REPORT

Report the result to the nearest 0,002 g of lead per litre at 15 °C and make reference to this International Standard.

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.

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