# LIGHT PETROLEUM

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**SYNONYMS** Petroleum spirits, petroleum ether

**DEFINITION** Light petroleum fractions boiling between 25 and 105°. Mixed paraffinic (normal and iso) and cycloparaffinic hydrocarbons

**DESCRIPTION** Clear, colourless, mobile, highly flammable liquid with a characteristic petroleum-like odour

**FUNCTIONAL USES** Extraction solvent

**CHARACTERISTICS**

IDENTIFICATION

Solubility (Vol. 4) Insoluble in water, soluble in ethanol

PURITY

Non-volatile residue (Vol. 4) Not more than 2 mg/100 ml

Sulfur (Vol. 4) Not more than 10 mg/kg

Benzene (Vol. 4) Not more than 0.05% v/v
Proceed as directed under the *Aromatic Hydrocarbons Determination* (see Volume 4)

Aromatic hydrocarbons (Vol. 4) Not more than 0.3% v/v (including benzene)

Polycyclic aromatic hydrocarbons (Vol. 4) The sample shall meet the following absorbance limits:

| nm | max. absorbance /cm path length |
|---|---|
| 280 - 289 | 0.15 |
| 290 - 299 | 0.12 |
| 300 - 359 | 0.08 |
| 360 - 400 | 0.02 |

Bromine index Not more than 200
See description under TESTS

Lead (Vol. 4) Not more than 2 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, “Instrumental Methods.”

## TESTS

PURITY TESTS

Bromine index

Principle

A known mass of the sample dissolved in a specified solvent is titrated with standard bromide-bromate solution. The end point is indicated by a dead stop electrometric titration apparatus, when the presence of free bromine causes a sudden change in the electrical conductivity of the system. The bromine index is the number of mg of bromine that will react with 100 g of the sample under the conditions of the test.

Apparatus

- Dead-Stop Electrometric Titration Apparatus: Any dead-stop apparatus may be used incorporating a high-resistance polarizing current supply capable of maintaining approximately 0.8 V across two platinum electrodes and with a sensitivity such that a voltage change of approximately 50 mV at these electrodes is sufficient to indicate the end point.
- Titration Vessel: A jacketed glass vessel of approximately 150-ml capacity of such a form that can be conveniently maintained at 1° to 5°. A pair of platinum electrodes spaced not more than 5 mm apart, shall be mounted to extend well below the liquid level. Stirring shall be by a mechanical or electromagnetic stirrer and shall be rapid, but not so vigorous as to draw air bubbles down to the electrodes.
- Burettes: 10 and 50-ml capacity.
- Iodine Number Flasks: Glass-stoppered, 500 ml capacity

Reagents

- Bromide-Bromate Solution (0.05 N): Dissolve 5.1 g of potassium bromide and 1.4 g of potassium bromate in water and dilute to 1,000 ml. Standardize to four significant figures as follows: Place 50 ml of glacial acetic acid and 1 ml of hydrochloric acid TS in a 500 ml iodine number flask. Chill the solution in an ice bath for approximately 10 min and with constant swirling of the flask, add from a 50 ml burette 40 to 45 ml of bromide-bromate solution, estimated to the nearest 0.01 ml, at a rate such that the addition takes between 90 and 120 sec. Stopper the flask immediately, shake the contents, place it again in the ice bath, and add 5 ml of 15% potassium iodide solution in the lip of the flask.
After 5 min remove the flask from the ice bath and allow the 15% potassium iodide solution to flow into the flask by slowly removing the stopper. Shake vigorously, add 100 ml of water in such a manner as to rinse the stopper, lip, and walls of the flask, and titrate promptly with 0.05 N sodium thiosulfate. Near the end of the titration add starch TS and titrate slowly to the disappearance of the blue colour.

Calculate the normality of the bromide-bromate solution as follows:

$$N_1 = \frac{A_2 N_2}{A_1}$$

where
$N_1$ = normality of the bromide-bromate solution;
$A_1$ = ml of the bromide-bromate solution;
$N_2$ = normality of the $Na_2S_2O_3$ solution; and
$A_2$ = ml of the $Na_2S_2O_3$ solution required for titration of the bromide-bromate
- Titration Solvent: prepare 1,000 ml of titration solvent by mixing the following volumes of materials: glacial acetic acid (714 ml), carbon tetrachloride (134 ml), methanol (134 ml), and sulfuric acid (18 ml of 1 + 5).

Procedure
Switch on the titrimeter and allow the electrical circuit to stabilise according to the manufacturer's instructions. Cool the titration vessel to $0^o$ - $5^o$ by circulating a suitable coolant through the jacketed titration vessel. Add 110 ml of the titration solvent and 8 to 10 g of the sample.
Switch on the stirrer and adjust to a rapid stirring rate, but not so rapid as to draw air bubbles into the solution. Allow the contents to cool to $0^o$ - $5^o$ and maintain this temperature throughout the titration. Add the bromide-bromate solution in small increments from a 10 ml burette until the end point detector (magic eye or potentiometric) indicates that the end point has nearly been reached. Continue adding 0.1 ml of the reagent at a time until the detector indicates a stable end point has been reached (end point lasting more than 30 sec). Repeat the determination but without the addition of the sample; less than 0.1 ml of the bromide-bromate solution should be required.

Calculation
Calculate the bromine index from

$$Bromine\ index = \frac{(T_1 - T_2) \times N \times 7{,}990}{W}$$

where
$T_1$ = number of ml of the bromide-bromate solution required for the titration of the sample;
$T_2$ = number of ml of the bromide-bromate solution required for the blank titration;
N = normality of the bromide-bromate solution; and
W = Weight of the sample in g