### INTERNATIONAL STANDARD

ISO 3839

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# Petroleum products — Determination of bromine number of distillates and aliphatic olefins — Electrometric method

Produits pétroliers — Détermination de l'indice de brome de distillats et d'oléfines aliphatiques — Méthode électrométrique



#### **Foreword**

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting

International Standard ISO 3839 was prepared by Technical Committee ISO/TC 28, Petroleum products and lubricants.

This second edition cancels and replaces the first edition (ISO 3839:1978), which has been technically revised.

Annex A of this International Standard is for information only.

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## Petroleum products — Determination of bromine number of distillates and aliphatic olefins — Electrometric method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

#### 1 Scope

This International Standard specifies a method for the determination of the bromine number of the following materials:

a) petroleum distillates that are substantially free of material lighter than 2-methylpropane, and that have 90 %(V/V) (i.e. volume fraction 90 %) distillation recovery temperatures under 327 °C. The method is generally applicable to gasolines (including leaded, unleaded and oxygenated fuels), kerosines and distillates in the gas oil range that fall within the following limits:

90 %(V/V) recovery distillation	Bromine number, max.
temperature (ISO 3405)	(see note 1)
Under 205 °C	175
205 °C to 327 °C	10

b) commercial olefins that are essentially mixtures of aliphatic monoolefins and that fall within the range of 95 to 165 bromine number (see note 1).

The method has been found suitable for such materials as commercial propene trimer and tetramer, butene dimer, and mixed nonenes, octenes and heptenes. The method is not suitable for normal alpha-olefins.

#### NOTES

- 1 These limits are imposed since the precision of the method has been determined only up to or within the range of these bromine numbers.
- 2 The value of the bromine number is an indication of the quantity of bromine-reactive constituents, not an identification of constituents. Annex A and table A.1 give information related to the use of this International Standard as a measure of olefinic unsaturation.

#### 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the

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possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3405:1988, Petroleum products — Determination of distillation characteristics.

ISO 3696:1987, Water for analytical laboratory use — Specification and test methods.

#### 3 Definition

For the purposes of this International Standard, the following definition applies:

**3.1 bromine number:** Mass, in grams, of bromine which will combine with 100 g of the sample under standardized conditions.

#### 4 Principle

A known mass of the test portion dissolved in a specified solvent maintained at 0 °C to 5 °C is titrated with standard volumetric bromide/bromate solution. The end-point is indicated by a sudden change in potential on an electrometric end-point titration apparatus due to the presence of free bromine.

#### 5 Reagents and materials

During the analysis, use only reagents of recognized analytical grade, and water equivalent to grade 3 of ISO 3696.

#### 5.1 1,1,1-Trichloroethane (CH<sub>3</sub>CCl<sub>3</sub>).

 ${\sf CAUTION-1,1,1-trichloroethane}$  is hazardous to the environment. A substitute is under active investigation.

#### 5.2 Methanol (CH<sub>3</sub>OH).

#### 5.3 Potassium iodide solution, 150 g/l.

Dissolve 150 g of potassium iodide (KI) in water and dilute to 1 l.

#### **5.4** Sulfuric acid, dilute solution (1:5).

Carefully mix 1 volume of concentrated sulfuric acid  $[H_2SO_4, 98\%(m/m)]$  (i.e. mass fraction 98%) min.] with 5 volumes of water.

#### 5.5 Titration solvent.

Prepare 1 I of titration solvent by mixing the following volumes of materials: 714 ml of acetic acid (5.9), 134 ml of 1,1,1-trichloroethane (5.1), 134 ml of methanol (5.2) and 18 ml of sulfuric acid solution (5.4).

#### **5.6 Bromide/bromate solution,** $[c(Br_2) = 0.250 \text{ mol/l}].$

Dissolve 51,0 g  $\pm$  0,1 g of potassium bromide (KBr) and 13,92 g  $\pm$  0,01 g of potassium bromate (KBrO<sub>3</sub>), both dried at 105 °C for 30 min, in water and dilute to 1 l.

NOTE — If the bromine numbers of the reference olefins specified in clause 7 and determined using this solution do not conform to the limits specified, or if there is some uncertainty as to the quality of primary reagents, it is recommended that the concentration (mol/l) be determined (and used in subsequent calculations) by standardizing the solution. The standardization procedure shall be carried out as follows:

Place 50 ml of acetic acid (5.9) and 1 ml of concentrated hydrochloric acid (5.10) in a 500-ml iodine-number flask. Chill the solution in an ice bath for approximately 10 min, and with constant swirling of the contents of the flask, add from a 10-ml