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Petroleum products and lubricants — Neutralization number — Potentiometric titration method

Produits pétroliers et lubrifiants — Indice de neutralisation — Méthode par titrage potentiométrique

Reference number
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Foreword

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Annex A of this International Standard is for information only.

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Petroleum products and lubricants — Neutralization number — Potentiometric titration method

1 Scope

1.1 This International Standard specifies a method for the determination of acidic constituents in petroleum products and lubricants soluble or nearly soluble in mixtures of toluene and propan-2-ol. It is applicable for the determination of acids whose dissociation constants in water are larger than 10^{-9}; extremely weak acids whose dissociation constants are smaller than 10^{-9} do not interfere. Salts react if their hydrolysis constants are larger than 10^{-9}.

NOTE — In new and used oils, the constituents that may be considered to have acidic characteristics include organic and inorganic acids, esters, phenolic compounds, lactones, resins, salts of heavy metals, salts of ammonia and other weak base constituents, acid salts of polybasic acids, and additives such as inhibitors and detergents.

1.2 This method may be used to indicate relative changes that occur in an oil during use under oxidizing conditions regardless of the colour or other properties of the resulting oil. Although the titration is made under definite equilibrium conditions, the method does not measure an absolute acidic or basic property that can be used to predict performance of an oil under service conditions. No general relationship between bearing corrosion and acid or base number is known.

NOTE — A colour-indicator titration method is also available in ISO 6618 (see bibliography in annex A). The acid numbers obtained by the potentiometric method may or may not be numerically the same as those obtained by ISO 6618, but they are generally of the same order of magnitude.

1.3 New and used petroleum products may contain acidic constituents that are present as additives or as degradation products formed during service, such as oxidation products. The relative amounts of these materials can be determined by titrating with bases. The acid number is a measure of this amount of acidic substances in the oil under the conditions of the test. The acid number is used as a guide in the quality control of lubricating oil formulations. It is also sometimes used as a measure of lubricant degradation in service. Any condemning limits must be empirically established.

As a variety of oxidation products contribute to the acid number and the organic acids vary widely in corrosion properties, the test cannot be used to predict corrosiveness of an oil under service conditions. No general correlation is known between acid number and the corrosive tendency of oils toward metals. Compounded engine oils can, and usually do, have an acid number in this test.

2 Definitions

For the purposes of this International Standard, the following definitions apply.

2.1 acid number: The quantity of base, expressed in milligrams of potassium hydroxide per gram of sample, required to titrate potentiometrically a test portion in a specified solvent from its initial meter reading to a meter reading corresponding to that obtained with a freshly prepared basic non-aqueous buffer solution or a well defined inflection point as specified in the standard.

2.2 strong acid number: The quantity of base, expressed in milligrams of potassium hydroxide per gram of sample, required to titrate potentiometrically a test portion in a specified solvent from its initial meter reading to a meter reading corresponding to that obtained with a freshly prepared acidic non-aqueous buffer solution or a well defined inflection point as specified in the standard.

3 Principle

The test portion is dissolved in a mixture of toluene and propan-2-ol containing a small amount of water and titrated potentiometrically with alcoholic potassium hydroxide using a glass indicating electrode and a calomel reference electrode. The meter readings are plotted manually or automatically against the respective volumes of titrating solution and the end points are taken only at well defined inflections in the resulting curve. When no definite inflections are obtained, end points are taken at meter readings corresponding to those found for non-aqueous acidic and basic buffer solutions.

4 Reagents

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

4.1 Non-aqueous acidic buffer solution

Add 10 ml of buffer stock solution A (4.5) to 100 ml of titration solvent. Use within 1 h.

4.2 Non-aqueous basic buffer solution

Add 10 ml of buffer stock solution B (4.6) to 100 ml of titration solvent. Use within 1 h.

4.3 2,4,6-Trimethylpyridine (γ-Collidine) (M_r = 121,18), conforming to the following requirements:

Boiling range	168 to 170 °C
Refractive index, n_D^{20}	1,498 2 ± 0,000 5
Colour	colourless

Store the reagent over activated alumina and keep in a brown glass bottle.

4.4 *m*-Nitrophenol (M_r = 139,11), conforming to the following requirements:

Melting point	96 to 97 °C
Colour	pale yellow

Store the reagent in a brown glass bottle.

4.5 Buffer stock solution A

Accurately weigh 24,2 ± 0,1 g of 2,4,6-trimethylpyridine (γ-collidine) and transfer to a 1-litre volumetric flask containing 100 ml of anhydrous propan-2-ol (4.9). Using a 1-litre graduated cylinder, add to the flask, while continuously stirring its contents, $(150/c_1)$ ml ± 5 ml of 0,2 mol/l alcoholic HCl solution (4.8) (c_1 being the actual concentration of the HCl solution as found by standardization). Dilute to the 1 000 ml mark with propan-2-ol and mix thoroughly. Use within one month.

4.6 Buffer stock solution B

Accurately weigh 27,8 ± 0,1 g of *m*-nitrophenol and transfer to a 1-litre volumetric flask containing 100 ml of anhydrous propan-2-ol (4.9). Using a 500 ml graduated cylinder, add to the flask, while continuously stirring its contents, $(50/c_2)$ ml ± 1 ml of 0,2 mol/l alcoholic KOH solution (4.12) (c_2 being the actual concentration of the KOH solution as found by standardization). Dilute to the 1 000 ml mark with propan-2-ol and mix thoroughly. Use within one month.

4.7 Hydrochloric acid, standard volumetric alcoholic solution, $c(\text{HCl})$ = 0,1 mol/l.

Mix 9 ml of hydrochloric acid (HCl) (ϱ_{20} = 1,19 g/ml) with 1 litre of anhydrous propan-2-ol (4.9). Standardize frequently enough to detect concentration changes of 0,000 5 mol/l by potentiometric titration of approximately 8 ml (accurately measured) of the 0,1 mol/l alcoholic KOH solution (4.11) diluted with 125 ml of carbon-dioxide-free water.

4.8 Hydrochloric acid, standard volumetric alcoholic solution, $c(\text{HCl})$ = 0,2 mol/l.

Prepare and standardize as directed in 4.7, but use 18 ml of HCl (ϱ_{20} = 1,19 g/ml).

4.9 Propanol-2-ol, anhydrous [less than 0,1 % (m/m) H_2O].

If adequately dry reagent cannot be procured, it can be prepared by distillation through a multiple-plate column, discarding the first 10 % of material distilling overhead and the last 10 % remaining. Drying may also be accomplished using molecular sieves such as Linde type 4A[1], passing the solvent upward through a molecular sieve column using one part of molecular sieve per ten parts of solvent.

4.10 Potassium chloride electrolyte

Prepare a saturated solution of potassium chloride (KCl) in water.

4.11 Potassium hydroxide, standard volumetric alcoholic solution, $c(\text{KOH})$ = 0,1 mol/l.

Add 6 g of potassium hydroxide (KOH) to approximately 1 litre of anhydrous propan-2-ol (4.9) in a 2-litre flask. Boil gently under reflux for 10 min and then shake to ensure it dissolves completely. Add at least 2 g of barium hydroxide [$Ba(OH)_2$] and again boil gently for 5 to 10 min.

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1) Linde type 4A is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

Allow the solution to stand for 2 days and then filter the supernatant liquid through a funnel fitted with a fine sintered-glass filter. Store the solution in a chemically resistant bottle.

Dispense in a manner such that the solution is protected from atmospheric carbon dioxide (CO_2), by means of a guard tube containing soda lime or soda non-fibrous silicate absorbant, and such that the solution does not come into contact with cork, rubber or saponifiable stopcock grease.

Standardize frequently enough to detect concentration changes of 0,000 5 mol/l by potentiometric titration against 0,1 to 0,15 g of potassium hydrogen phthalate (dried for 2 hours at 105 °C) weighed to 0,000 2 g accuracy and dissolved in about 100 ml of carbon-dioxide-free water.

4.12 Potassium hydroxide, standard volumetric alcoholic solution, $c(\text{KOH}) = 0{,}2$ mol/l.

Prepare, store and standardize as directed in 4.11, but use 12 to 13 g of KOH to approximately 1 litre of anhydrous propan-2-ol (4.9). For standardization use 0,2 to 0,3 g of potassium hydrogen phthalate, weighed to 0,000 2 g accuracy, dissolved in about 100 ml of carbon-dioxide-free water.

4.13 Titration solvent

Add 500 ml of toluene and 5 ml of water to 495 ml of anhydrous propan-2-ol (4.9). The titration solvent should be made up in large quantities, and its blank value determined daily by titration prior to use.

NOTE — A titration solvent in which chloroform is used in place of toluene may be required to completely dissolve certain heavy residues of asphaltic materials.

5 Apparatus

The cell assembly used for the potentiometric titration is shown in figure 1.

5.1 Manual titration apparatus

5.1.1 Meter, either a voltmeter or a potentiometer that will operate with an accuracy of ± 0,005 V and a sensitivity of ± 0,002 V over a range of at least ± 0,5 V when the meter is used with the electrodes specified in 5.1.2 and 5.1.3 and when the resistance between the electrodes falls within the range of 0,2 to 20 MΩ. The meter shall be protected from stray electrostatic fields so that no permanent change in the meter readings over the entire operating range is produced by touching, with a grounded lead, any part of the exposed surface of the glass electrode, the glass electrode lead, the titration stand or the meter.

NOTE — A suitable apparatus could consist of a continuous-reading electronic voltmeter designed to operate on an input of less than 5×10^{-12} A when an electrode system having 1 000 MΩ resistance is connected across the meter terminals and provided with a metal shield connected to the ground, as well as a satisfactory terminal to connect the shielded connection wire from the glass electrode to the meter without interference from any external electrostatic field.

5.1.2 Glass electrode (C in figure 1), pencil type, 125 to 180 mm in length and 8 to 14 mm in diameter.

The body of the electrode shall be made of a chemically resistant glass tube with a wall thickness of 1 to 3 mm.

The end dipping into the solution shall be closed with a hemisphere of glass sealed on to the electrode tube and the radius of this hemisphere shall be about 7 mm. The thickness

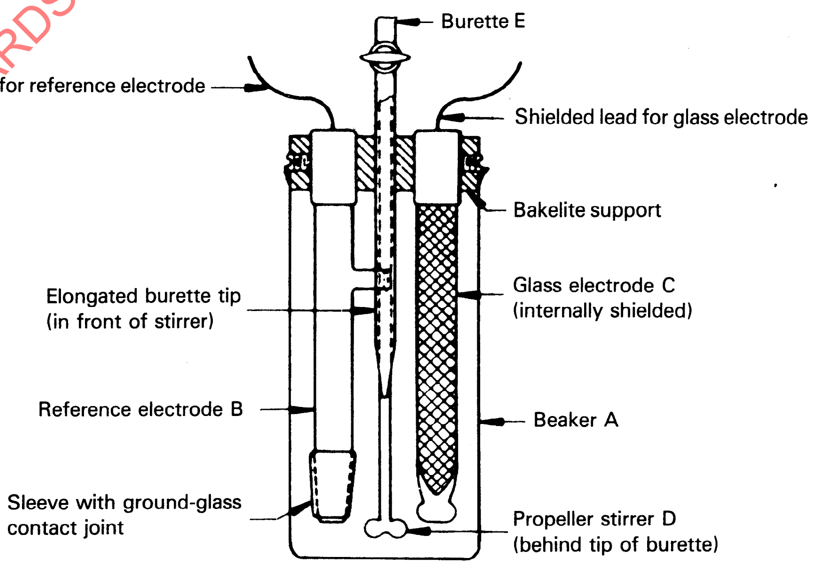


Figure 1 — Cell for potentiometric titration

of the glass in the hemisphere shall be great enough so that the resistance of the hemisphere is 100 to 1 000 MΩ at 25 °C.

The electrode shall contain a reproducible, permanently sealed liquid cell for making electrical connection with the inner surface of the hemisphere.

The entire electrical connection from the sealed contact cell to the meter terminal shall be surrounded by an electrical shield that will prevent electrostatic interference when the shield is grounded.

The shield shall be insulated from the electrical connection by insulating material of the highest quality, such as rubber and glass, so that the resistance between the shield and the entire length of the electrical connection is greater than 50 000 MΩ.

5.1.3 Calomel reference electrode (B in figure 1), pencil type, 125 to 180 mm in length and 8 to 14 mm in diameter.

This electrode shall be made of glass and shall be provided with an external, removable glass sleeve on the sealed end that is dipped into the titration solution.

The glass sleeve shall be 8 to 25 mm in length, shall be slightly tapered, and shall be ground to fit the electrode so that the sealed end of the electrode protrudes 2 to 20 mm beyond the sleeve. The ground surface shall be continuous and free of smooth spots.

At a point midway between the extremities of the ground surface, the electrode tube shall be pierced by a hole or holes 1 mm in diameter. The electrode shall contain the necessary mercury, calomel and electrical connection to the mercury, all arranged in a permanent manner.

The electrode shall be filled almost to capacity with saturated KCl electrolyte (4.10) and shall be equipped with a stoppered port through which the electrolyte may be replenished.

When suspended in the air and with the sleeve in place, the electrode shall not leak electrolyte at a rate greater than one drop in 10 min.

NOTE — Combination electrodes and silver/silver chloride electrodes, because of their slow response, have been found unsuitable for this method.

5.1.4 Variable-speed mechanical stirrer, of a suitable type, equipped with a glass, propeller-type stirring paddle (D in figure 1). A propeller with blades 6 mm in radius and set at a pitch of 30 to 45° is satisfactory. A magnetic stirrer is also satisfactory.

If electrical stirring apparatus is used, it shall be electrically correct and grounded so that connecting or disconnecting the power to the motor will not produce a permanent change in meter reading during the course of the titration.

5.1.5 Burette (E in figure 1), 10 ml capacity, graduated in 0,05 ml divisions, and calibrated with an accuracy of ± 0,02 ml. The burette shall have a glass stopcock and shall have a tip that extends 100 to 130 mm beyond the stopcock. The burette for KOH shall have a guard tube containing soda lime or other CO_2-absorbing substance.

5.1.6 Titration beaker (A in figure 1), 250 ml capacity, made of borosilicate glass.

5.1.7 Titration stand, suitable for supporting the electrodes, stirrer and burette in the position shown in figure 1.

NOTE — An arrangement that allows the removal of the beaker without disturbing the electrodes, burette and stirrer is desirable.

5.2 Automatic titration apparatus

Automatic titration systems shall be generally in accordance with 5.1 and provide the following technical performance characteristics or features:

5.2.1 Automatic adaptation of the titration speed in the continuous titrant delivery mode to the slope of the titration curve with the capability of complying with the potential equilibrium specified and providing titration rates of less than 0,2 ml/min during titration and preferably 0,05 ml/min at inflections and at non-aqueous acid and basic end points.

5.2.2 Interchangeable precision motor-driven burettes with a volume dispensing accuracy of ± 0,01 ml.

5.2.3 A record of the complete course of titration by continuously printing out the relative potential versus volume of titrant added.

6 Preparation of a test sample of used oil

6.1 Strict observance of the sampling procedure is necessary, since the sediment itself is acidic and may also have absorbed acidic material from the sample. Failure to obtain a representative test sample will cause serious errors.

As used oil may change appreciably in storage, samples shall be tested as soon as possible after removal from the lubricating system; the dates of sampling and testing shall be noted.

6.2 Heat the laboratory sample of used oil to 60 ± 5 °C in the original container with agitation until all of the sediment is homogeneously suspended in the oil. If the original container is a can, or if it is glass and more than three-fourths full, transfer the entire sample to a clear glass bottle having a capacity at least one-third greater than the volume of the sample. Transfer all traces of sediment from the original container to the bottle by violent agitation of portions of the sample in the original container.

NOTE — When laboratory samples are visibly free from sediment, the heating procedures described may be omitted.

6.3 After complete suspension of all sediment, strain the test sample or a convenient aliquot through a 100-mesh screen to remove large contaminating particles.

7 Preparation of electrode system

7.1 Maintenance of electrodes

7.1.1 Clean the glass electrode (5.1.2) at frequent intervals (not less than once every week during continual use) by immersing in cold chromic acid cleaning solution. A suitable detergent instead of the chromic acid cleaning solution can also be used.

NOTE — In order to prevent damage to the electrode, do not leave it immersed in the chromic acid for more than a few minutes.

7.1.2 Drain the calomel reference electrode (5.1.3) at least once each week and refill with fresh KCl electrolyte (4.10). Ascertain that crystallized KCl is present. Maintain the electrolyte level in the calomel electrode above that of the liquid in the titration beaker at all times.

7.1.3 When the electrodes are not in use, immerse the lower halves in water. Do not allow them to remain immersed in titration solvent for any appreciable period of time between titrations. Although the electrodes are not particularly fragile, handle them carefully at all times.

7.2 Preparation of electrodes

7.2.1 Before and after use, wipe the glass electrode thoroughly with a clean cloth or a soft absorbent tissue, and rinse with water. Wipe the calomel reference electrode with a cloth or tissue, carefully remove the ground-glass sleeve and thoroughly wipe both ground surfaces.

7.2.2 Replace the sleeve loosely and allow a few drops of electrolyte to drain through to flush the ground-glass joint. Wet the ground surfaces thoroughly with electrolyte, set the sleeve firmly in place, and rinse the electrode with water.

7.2.3 Prior to each titration, soak the prepared electrodes in water for at least 5 min immediately before use, and touch the tips of the electrodes with a dry cloth or tissue to remove the excess water.

7.3 Testing of electrodes

7.3.1 Test the meter/electrode combination when first put into use, or when new electrodes are installed, and retest at intervals thereafter by dipping the electrodes into a well stirred mixture of 100 ml of the titration solvent and 1,0 to 1,5 ml of 0,1 mol/l alcoholic KOH solution (4.11).

7.3.2 The meter/electrode combination is suitable for use when the potential changes more than 480 mV from the potential between the same electrodes when dipped into the acidic non-aqueous buffer solution (4.1).

NOTE — Cleaning the electrodes thoroughly, keeping the ground-glass joint free of foreign materials, and regular testing of the electrodes are very important in obtaining reproducible potentials, since contamination may introduce uncertain, erratic and undetectable liquid-contact potentials. While this is of secondary importance when end points are chosen from inflection points in the titration curve, it may be quite serious when end points are chosen at arbitrarily fixed cell potentials.

8 Standardization of apparatus

8.1 Determination of meter readings for the non-aqueous buffer solutions corresponding to acid end points

To ensure comparable selection of end points when definite inflection points are not obtained in the titration curve, determine daily, for each electrode pair, the meter readings obtained with the acidic (4.1) and basic (4.2) non-aqueous buffer solutions.

8.2 Prepare the electrodes as described in 7.2, immerse them in the non-aqueous buffer solution and stir for 5 min, maintaining the buffer solution at a temperature within 2 °C of that at which the titrations are to be made. Read the cell voltage. The readings so obtained are taken as the end points in titration curves having no inflection points.

9 Procedure for total acid number and strong acid number

9.1 Into the 250 ml titration beaker (5.1.6), introduce a weighed quantity of test sample, as prescribed in table 1, and 125 ml of titration solvent (4.13). Prepare the electrodes as directed in 7.2.

Place the beaker on the titration stand (5.1.7) and adjust its position so that the electrodes are about half-immersed. Start the stirrer (5.1.4), and stir throughout the determination at a rate sufficient to produce vigorous agitation without spattering and without stirring air into the solution.

Table 1 — Size of test portion

Acid number	Mass of test portion, g	Accuracy of weighing, g
0,05 to 1,0	20,0 ± 2,0	0,10
1,0 to 5,0	5,0 ± 0,5	0,02
5 to 20	1,0 ± 0,1	0,005
20 to 100	0,25 ± 0,02	0,001
100 to 250	0,1 ± 0,01	0,000 5

9.2 Fill the burette with the 0,1 mol/l alcoholic KOH solution (4.10) and place the burette in position in the cell assembly, taking care that the tip is immersed about 25 mm in the liquid in the beaker. Record the intitial burette and meter (cell potential) readings.

9.3 Manual titration method

9.3.1 Add suitable small portions of 0,1 mol/l alcoholic KOH solution and, after waiting until a constant potential has been established, record the burette and meter readings.

NOTE — Consider the cell potential to be constant when it changes less than 5 mV per minute. This may mean waiting approximately 1 to

2 min per 100 mV change in potential when adding 0,05 ml increments; 0,1 ml increments may require 3 to 5 min.

9.3.2 At the start of the titration and in any subsequent regions (inflections) where 0,1 ml of 0,1 mol/l KOH solution consistently produces a total change of more than 30 mV in the cell potential, add 0,05 ml portions.

9.3.3 In the intermediate regions (plateaux) where 0,1 ml of 0,1 mol/l alcoholic KOH solution changes the cell potential less than 30 mV, add larger portions sufficient to produce a total potential change approximately equal to, but not greater than, 30 mV.

9.3.4 Titrate in this manner until the potential changes less than 5 mV per 0,1 ml of KOH solution added and the cell potential indicates that the solution is more basic than the non-aqueous basic buffer.

9.3.5 Remove the titrated solution and rinse the electrodes and burette tip with the titration solvent, then with propan-2-ol and finally with distilled water. Immerse the electrodes in distilled water for at least 5 minutes before another titration to restore the aqueous gel layer of the glass electrode. Store electrodes in distilled water when not in use. If electrodes are found to be dirty and contaminated, proceed as in clause 7.

9.4 Automatic titration method

9.4.1 Adjust the apparatus in accordance with the manufacturer's instructions to comply with the potential equilibrium mode requirements established for the manual titration as explained in the note to 9.3.1 and/or to operate in a variable/continuous delivery rate mode of titration with a delivery rate of less than 0,2 ml/min during the titration and preferably 0,05 ml/min through the region of an inflection or at the end point corresponding to that found for the standard non-aqueous basic buffer solution.

9.4.2 Proceed with the automatic titration and record potentiometric curves or derivative curves as the case may be.

9.4.3 Titrate in this manner with the 0,1 mol/l alcoholic KOH solution until the potential becomes constant [for example, it changes less than 5 mV per 0,1 ml of KOH solution (automatic end point)] or until the potential reading indicates that the solution is more basic than the freshly prepared non-aqueous basic buffer solution (pre-selected end point).

9.4.4 On completion of the titration, rinse the electrodes and burette tip with the titration solvent, then with propan-2-ol and finally with distilled water. Keep the electrodes immersed in water for at least 5 min before another titration to restore the aqueous gel layer of the glass electrode. Store the electrodes in distilled water when not in use. If electrodes are found to be dirty and contaminated, proceed as in clause 7.

9.5 Blank test

Make a blank titration of 125 ml of titration solvent for each set of samples. For a manual titration, add 0,1 mol/l alcoholic KOH solution in 0,05 ml increments and record meter reading and burette reading when the former becomes constant. For automatic titrations, proceed as in 9.4.

10 Expression of results

The results shall be expressed as acid number or strong acid number, as appropriate.

10.1 Manual titration method

Plot the volumes of 0,1 mol/l alcoholic KOH solution added against the corresponding meter readings (see figure 2). Mark as an end point only a well defined inflection point that is closest to the cell voltage corresponding to that obtained with the freshly prepared non-aqueous acidic or basic buffer. If inflections are ill defined or no inflection appears (see figure 2, curve B), mark the end point at the meter reading corresponding to that obtained with the appropriate non-aqueous buffer.

For all titrations on used oils, mark as an end point the point on the curve that corresponds to the meter reading obtained with the freshly prepared non-aqueous acidic buffer.

NOTES

1 An inflection point is generally recognizable by inspection as the point at which several successive 0,05 ml increments of KOH solution each produce a cell potential change greater than 15 mV and at least 30 % greater than those produced by previous or subsequent increments of the same size. Generally, definite inflection points may be discerned only in regions where increments of the same size are used.

2 Cooperative work done on acid number determinations on fresh oils, additive concentrates and used oils has indicated well defined inflection points for fresh oils and additive concentrates, and generally ill defined inflections or no inflection points at all for used oils.

10.2 Automatic titration method

Mark the end points on the curves obtained in 9.4, in the same way as for the manual titration method (see 10.1).

10.3 Method of calculation (applicable to manual and automatic methods)

Calculate the total acid number and strong acid number, expressed as milligrams of KOH per gram of sample, using the following equations:

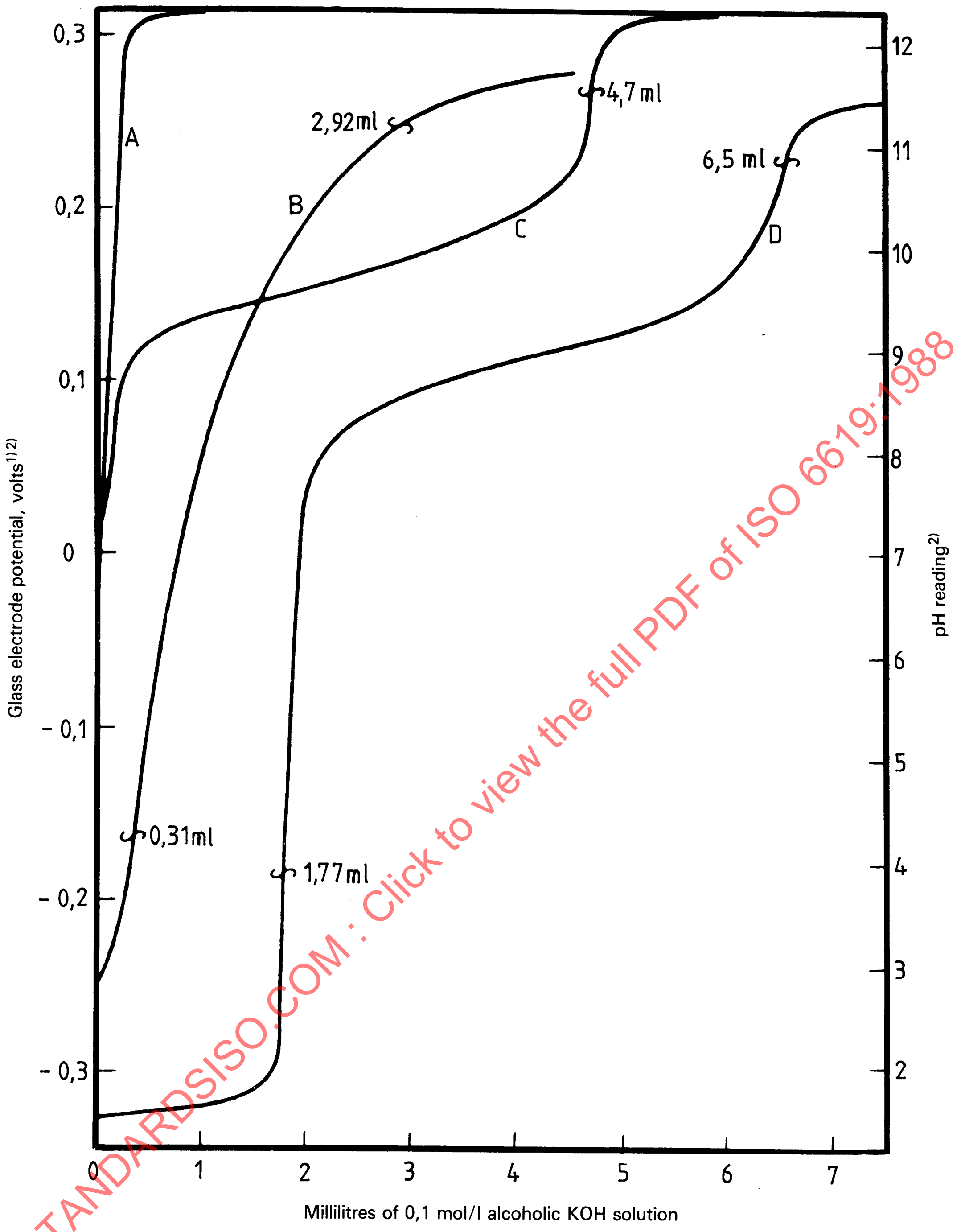
$$\text{Total acid number} = \frac{(V_1 - V_0) \times c_3 \times 56{,}1}{m}$$

$$\text{Strong acid number} = \frac{(c_3 V_2 + c_4 V_3) \times 56{,}1}{m}$$

where

V_0 is the volume, in millilitres, of the alcoholic KOH solution (4.11) used for the blank titration;

V_1 is the volume, in millilitres, of the alcoholic KOH solution (4.11) used to titrate the test portion to an end point (see 10.1 or 10.2);



Key

Curve A — Blank on 125 ml of titration solvent.

Curve B — 10,00 g of used crankcase oil plus 125 ml of titration solvent. Since no sharp inflections are apparent, the end points are chosen at the meter readings obtained with the two non-aqueous buffer solutions.

Curve C — 10,00 g of oil containing a weak acid plus 125 ml of titration solvent. The end point is chosen as the point at which the curve is most nearly vertical.

Curve D — 10,00 g of oil containing weak and strong acids plus 125 ml of titration solvent. The end points are chosen as the points at which the curve is most nearly vertical.

Figure 2 — Illustrative titration curves

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1) On some meters, the voltage sign is reversed.

2) In some instruments, the relation between glass electrode potential and pH reading is only approximately as shown.