# INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

APPLIED CHEMISTRY DIVISION

COMMISSION ON OILS, FATS AND DERIVATIVES\*

# STANDARD METHODS FOR THE ANALYSIS OF OILS, FATS AND DERIVATIVES

6th Edition

1st Supplement: Part 2 (1980)

# SECTION III. GLYCERINES

Prepared for publication by
A. HAUTFENNE
Université Catholique de Louvain
Louvain-la-Neuve, Belgium

\*Membership of the Commission during the period 1979-81 is as follows:

Chairman: C. PAQUOT (France); Secretary: A. HAUTFENNE (Belgium); Titular Members: H. BRÜSCHWEILER (Switzerland); D. FIRESTONE (USA): Ö. LEVIN (Sweden); M. NAUDET (France); J. POKORNY (Czechoslovakia); H. WESSELS (FRG): Associate Members: T. ASAHARA (Japan); J. L. BEARE-ROGERS (Canada); J. GRACIAN TOUS (Spain); E. KURUCZ (Hungary); P. R. E. LEWKOWITSCH (UK); A. T. MØLLER (Denmark); M. TEUPEL (FRG); J. C. VAN DER WEEL (Netherlands); National Representatives: A. R. JOHNSON (Australia); G. ZWERENZ (Austria); B. JACOBSBERG (Belgium); R. C. DE ARAUJO LAGO (Brazil); D. G. CHOBANOV (Bulgaria); A. TULLOCH (Canada); U. ENGELRUD (Denmark); A. VISAPAA (Finland); J. P. WOLFF (France); V. M. KAPOULAS (Greece); M. M. CHAKRABARTY (India); B. McGWYNNE (Ireland); E. FEDELI (Italy); T. HASHIMOTO (Japan); P. W. HENDRIKSE (Netherlands); F. B. SHORLAND (New Zealand); W. ZWIERZYKOWSKI (Poland); M. GASSIOT MATAS (Spain); R. OHLSON (Sweden); A. DIEFFENBACHER (Switzerland); A. A. KARABATUR (Turkey); K. A. WILLIAMS (UK): A. E. WALTKING (USA): B. OŠTRIČ-MATIJAŠEVIĆ (Yugoslavia).

# 3.111 DETERMINATION OF ALKALINITY OR ACIDITY. (Fifth Edition: Method III.A.5)

## 1. SCOPE AND FIELD OF APPLICATION.

This Standard describes a titrimetric method for the determination of the alkalinity or acidity of industrial glycerines, crude or purified (Note 1)

#### 2. DEFINITION.

The alkalinity or acidity of glycerines is defined as the number of milliequivalents of alkali or acid contained in 100 g of glycerine.

#### 3. PRINCIPLE.

Titration of a test portion with a standard solution of hydrochloric acid or sodium hydroxide in the presence of phenolphtalein as indicator.

# 4. APPARATUS.

- 4.1. 250-ml conical flask.
- 4.2. Microburette, graduated in 0.01 ml.

## 5. REAGENTS.

- 5.1. Ethanol 95 per cent (v/v).
- 5.2. Water free from carbon dioxide.
- 5.3. Hydrochloric acid, 0.1 N aqueous solution, accurately standardized.
- 5.4. Sodium hydroxide, 0.1 N aqueous solution, accurately standardized.
- 5.5. Phenolphtalein, 10 g/1 ethanolic solution (5.1).

# 6. PROCEDURE.

Weigh  $10 \stackrel{+}{=} 0.01$  g, to the nearest 0.001 g, of the test sample into the conical flask (4.1). Dilute the test portion with about 100 ml of water (5.2).

Add 3 drops of the phenolphtalein solution (5.5), mix and note the colour obtained.

Depending on the particular case, titrate the alkalinity with the standard hydrochloric acid solution (5.3) until the indicator is decolorized, or titrate the acidity with the standard sodium hydroxide solution (5.4) until the first persistent pink colour appears.

Use an ordinary burette, or, if the requisite volume of the aqueous standard solution is less than  $2 \, \text{ml}$ , a microburette (4.2).

# 7. EXPRESSION OF RESULTS.

The alkalinity or acidity is given by the formula:

- where V is the volume, in ml, of the aqueous standard solution (5.3) or (5.4), used for the titration;
  - T is the exact normality of the aqueous standard solution (5.3) or (5.4) used;
  - m is the mass, in g, of the test portion.

# 8. NOTE.

1. With coloured test samples a suitable potentiometric method may be used.

III. GLYCERINES 1980

3.131 DETERMINATION OF WATER CONTENT (KARL FISCHER METHOD).

(Fifth Edition: Method III.A.3)

# 1 SCOPE AND FIELD OF APPLICATION.

This Standard describes a method for determination of the water content of industrial glyce-rines.

# 2. DEFINITION.

The water content is the water quantity determined by the method described below and expressed as a percentage by mass (m/m) taking into account the possible alkalinity of the test sample measured as indicated in 3.111.

#### 3. PRINCIPLE.

Reaction between water, iodine and sulfur dioxide in the presence of alcoholic hydroxyl and pyridine:

$$H_2O + I_2 + SO_2 + 3 C_5H_5N + ROH \longrightarrow 3 C_5H_6N^+ + 2 I^- + RSO_4^-$$

# 4. APPARATUS.

Each item of apparatus must be absolutely dry at the time of use.

- 4.1. 150-ml conical flasks.
- 4.2. 1-1 brown glass flasks with ground stoppers.
- 4.3. 100-ml measuring cylinders with ground stoppers.
- 4.4. 25-ml graduated automatic burette suitably protected against atmospheric humidity by a desiccant.
- 4.5. 25-ml pipette.
- 4.6. Rubber stoppers without holes to fit flasks (4.1).
- 4.7. Two-holed rubber stoppers to fit flasks (4.1).

One hole is such that the delivery-tip of the burette can be inserted so as to reach to below the stopper; a tube of desiccant is fitted into the other hole.

4.8. Rubber bulb to fit the pipette (4.5).

#### REAGENTS.

- 5.1. Methanol, anhydrous.
- 5.2. Chloroform, pure.
- 5.3. Pyridine, anhydrous.
- 5.4. Sulfur dioxide, liquid, anhydrous.
- 5.5. Iodine re-sublimed.
- 5.6. Solvent mixture:

A mixture of chloroform (5.2) and methanol (5.1), 3:1 (v/v). Store in a brown glass bottle (4.2).

5.7. Solution A:

Dissolve 90 g iodine (5.5) in methanol (5.1)
Make up to 1 1 with methanol (5.1).
Store the solution in a brown glass bottle (4.2) at a temperature equal to or below 4°C

#### 5.8. Solution B (note 1):

Mix 450 ml pyridine (5.3) and 450 ml methanol (5.1) in a bottle (4.2).

Cool to 4°C.

Gradually add 90 g of sulfur dioxide by means of a glass tube delivering below the surface of the solution.

Protect against atmospheric humidity and as far as possible maintain the solution at a temperature below 4°C, stopping the gas flow and cooling whenever the temperature begins to rise.

As soon as the addition of sulfur dioxide is complete, remove the gas tube, stopper the bottle and shake to mix.

Keep in a refrigerator at about 4°C.

# 5.9. Solution C (reagent):

At the time of use, pour the required volume of solution A into a measuring cylinder (4.3).

Fit the stopper.

Pour the same volume of solution B into another measuring cylinder (4.3).

Fit the stopper.

Place the two measuring cylinders in melting ice for about 15 min.

Wipe the stoppers and the upper parts of the measuring cylinders with filter-paper in order to remove trace amounts of water which may have condensed during cooling and pour the whole of the solution A into the solution B at one go.

Stopper the measuring cylinder B. Shake the cylinder to mix the contents.

Allow the solution to warm up to room temperature.

#### 6. PROCEDURE.

Throughout the analysis take precautions to avoid contact with atmospheric humidity.

# 6.1. Standardization of the reagent (5.9).

Using a pipette (4.5) with a rubber bulb (4.8) transfer exactly 25 ml of the solvent mixture (5.6) into a conical flask (4.1).

Fit a two-holed rubber stopper (4.7).

Titrate with shaking (a magnetic stirrer is recommended) with solution C (5.9) until the colour of the solution changes from yellow to red-orange.

Tare another conical flask (4.1) to the nearest 0.0001 g.

Add a drop of water (note 2).

Record the increase in weight to the nearest 0.0001 g.

Add immediately and exactly 25 ml of the solvent mixture (5.6).

Shake and titrate immediately with agitation as for the solvent alone.

A, the water equivalent of the reagent C, in grams of water per ml, is given by the formula:

$$A = \frac{m_o}{v_1 - v_o}$$

where: V<sub>O</sub> is the number of ml of solution C used to titrate the solvent alone;
V<sub>1</sub> is the number of ml of solution C used to titrate the solvent containing the added water:

m is the mass, in g, of the added water.

# 6.2. Determination of the water content in glycerines.

Tare a conical flask (4.1) fitted with a stopper (4.6) to the nearest 0.001 g. Transfer into it a test portion corresponding to a maximum consumption of about 20 to 25 ml of the reagent C (5.9).

Stopper immediately.

Weigh to the nearest 0.001 g to determine the mass of the test portion.

Remove the stopper.

Using the pipette (4.5) and bulb (4.8), pipette in exactly 25 ml of the solvent mixture (5.6).

Replace the stopper in the flask.

Dissolve with shaking.

Titrate, with shaking, with solution C (5.9) as in (6.1).

# EXPRESSION OF RESULTS.

The water content expressed as a percentage (m/m) is given by the formula:

$$\frac{100 - (V - V_0) \times A}{m} - 0.018 \times A1k \qquad (note 3)$$

where V is the number of ml of solution C used in (6.2);

- V is the number of ml of solution C used in (6.1) for 25 ml of the solvent mixture alone;
- m is the mass, in g, of the test portion;

Alk is the alkalinity of the test portion, in milliequivalents per 100 g, determined according to 3.111.

# 8. NOTES.

- As the preparation of solution B, involving the use of sulfur dioxide gas is troublesome, it may well be preferred to utilize commercial ready-made solutions A and B.
  Store these in a refrigerator at about 4°C.
  As the time for use, prepare solution C as indicated above.
  Other commercial solutions comprising a single reagent of a different composition are
  also available: they may likewise be used.
- A hydrated crystalline salt (sodium tartrate, sodium acetate) may be used in place of a drop of water to standardize the reagent.
- 3. In most cases, the correcting factor 0.018xAlk is negligible.

III. GLYCERINES.

1980

# 3.135 DETERMINATION OF ASH CONTENT. (Fifth Edition: Method III.A.4)

# 1. SCOPE AND FIELD OF APPLICATION.

This Standard describes a gravimetric method for the determination of the ash content in industrial glycerines.

This method is an empirical one.

#### 2. DEFINITION.

The ash content is defined as the quantity of ash determined by the present method and expressed as a percentage by mass (m/m).

# 3. PRINCIPLE.

Combustion of the test portion, ignition of the organic matter and weighing of the organic residue.

# 4. APPARATUS.

- 4.1. Platinum dish, 70 to 90 mm diameter and 40 to 50 mm deep.
- 4.2. Desiccator.
- 4.3. Muffle furnace, regulated at 750 10°C.

# 5. PROCEDURE.

# 5.1. Test portion:

Heat the dish (4.1) for a few minutes in the furnace (4.3) regulated at 750 - 10°C.

Cool it to room temperature in the desiccator (4.2).

Weight the dish (4.1) to the nearest 0.001 g.

Then weigh into the tared dish, to the nearest 0.01 g, 5 to 100 g of the test sample, depending on the expected amount of ash (from over 1 per cent to under 0.01 per cent).

#### 5.2. Determination.

Gently heat the dish (4.1) containing the test portion (5.1) over a small flame until the vapours are ignited.

Turn off the heat and allow the glycerine to burn until a carbonaceous mass is obtained.

Allow to cool for 1 or 2 min.

Place the dish (4.1) for 10 min in the furnace (4.3) regulated at 750 - 10°C.

Allow to cool the dish (4.1) and contents in the desiccator (4.2).

Weigh to the nearest 0.001 g.

# 6. EXPRESSION OF RESULTS.

The ash content of the test portion, expressed as a percentage by mass (m/m), is given by the formula:

$$\frac{m_2 - m_1}{m_3 - m_1} \times 100$$

- where  $m_1$  is the mass, in g, of the empty dish (4.1);
  - m, is the mass, in g, of the dish containing the ash;
  - $m_{\chi}$  is the mass, in g, of the dish containing the test portion.

# Express the result to

- 3 decimal places for ash less than 0.01 per cent;
- 2 decimal places for ash between 0.01 and 1 per cent;
- 1 decimal place for ash greater than 1 per cent.

III. GLYCERINES 1980

# 3.136 DETERMINATION OF ARSENIC CONTENT. (PHOTOMETRIC METHOD EMPLOYING SILVER DIETHYLDITHIOCARBAMATE.)

(Fifth Edition : Method III.A.2)

# 1. SCOPE.

This Standard describes a photometric method employing silver diethyldithiocarbamate for the determination of arsenic content of industrial glycerines.

# 2. FIELD OF APPLICATION.

The method is applicable for the analysis of glycerines whose arsenic content is higher than 0.5~mg/Kg.

# 3. DEFINITION.

The arsenic content is the quantity of arsenic, expressed as milligrams of arsenic per kilogram of glycerine (ppm), determined by the present method.

#### 4. PRINCIPLE.

Production of arsenic by zinc in hydrochloric acid solution forming arsine, AsH2.

Absorption of the arsine in a solution of silver diethyldithiocarbamate (Ag(DDTC)) in pyridine.

Photometric measurement of the purplish-red coloration produced by the dispersed colloidal silver, at the maximum of the absorption curve (wave-length about 540 nm), colloidal silver being formed according to the equation:

$$AsH_3 + 6 Ag(DDTC) \longrightarrow 6 Ag + 3 H(DDTC) + As(DDTC)_3$$

#### General case.

Application of the method successively to a test portion "as is" and to another identical test portion containing a known amount of added arsenic.

The determination is considered to be valid if the amount added is recovered.

#### Special case.

If the added arsenic is not recovered, or is recovered partially, the determination is to be made after first igniting the test portion in an oxidizing medium, in order to destroy organic substances capable of inhibiting arsine formation without loss of arsenic; a control test is made similarly on an identical sample enriched by a known amount of arsenic, which should be recovered (note 1).

#### 5. APPARATUS.

All the glassware used in the determination of arsenic must first be washed with hot concentrated sulphuric acid (food analysis grade), rinsed thoroughly with distilled water and finally well dried.

- 5.1. All-glass apparatus with ground-glass joints suitable for the generation and complete absorption of arsine (see Fig. 1) (notes 2 and 3), consisting of:
- 5.1.1. 100-ml conical flasks for the generation of arsine.
- 5.1.2. Connecting tube for the absorption of hydrogen sulfide.
- 5.1.3. Absorption tube with 15 bulbs.
- 5.2. 1000-m1 volumetric flasks.
- 5.3. Graduated pipettes 2, 10 and 25-m1.
- 5.4. Spectrophotometer, or
- 5.5. Photocolorimeter fitted with a suitable filter for the wave-length range 520 560 nm.

Glycerines 1949

- 5.6. Cells, path-length 1 cm. And for the special case:
- 5.7. Porcelain evaporating dishes, 40 mm deep, 100 mm top diameter and 50 mm bottom diameter.
- 5.8. Electric oven, regulated at 150 + 10°C.
- 5.9. Muffle furnace, regulated between 560 and 600°C.

# 6. REAGENTS.

- 6.1. Pyridine pure, arsenic-free ( = 0.980 g/ml approx.).
- 6.2 Zinc granules, 0.5 1 mm in diameter, or other forms provided that experience indicates that they give equivalent results when the present method is used (note 4).
- / 6.3. Hydrochloric acid (p= 1.19 mg/ml approx.) 38 per cent solution (m/m) (approx. 12 N solution).
  - 6.4. Sodium hydroxide, 50 g/l aqueous solution.
- 6.5. Potassium iodide, 150 g/l aqueous solution. Dissolve 15 g of potassium iodide in distilled water, dilute to 100 ml and mix.
- 6.6. Tin (II) chloride, in hydrochloric acid solution. Dissolve 40 g tin (II) chloride dihydrate (SnCl<sub>2</sub>· 2 H<sub>2</sub>O) in a mixture of 25 ml of water and 75 ml of the hydrochloric acid solution (6.3.).
- 6.7. Silver diethyldithiocarbamate (AgSSCN(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub> or Ag(DDTC)), in solution in pyridine 5 g/l. Dissolve 1 g of Ag(DDTC) in pyridine (6.1.) and dilute to 200 ml with the same solvent. Store the solution in a dark-glass bottle tightly stoppered and kept in the dark. This solution will be stable for about 2 weeks.
- 6.8. Arsenic, standard aqueous solution containing 0.100 g/1.

  Weigh, to the nearest 0.0001 g, 0.1320 g of arsenic (III) oxide (As 203).

  Transfer into a 100 ml beaker.

  Dissolve with about 2 ml of the sodium hydroxide solution (6.4.)

  Transfer the solution quantitatively into a volumetric flask (5.2.).

  Wash the beaker several times with distilled water, transferring the washings into the volumetric flask.

  Dilute to 1000 ml with distilled water and mix.

  1 ml of this standard solution contains 100 g of arsenic.
- 6.9. Arsenic, standard aqueous solution containing 2.50 mg/l.

Prepare this solution immediately prior to use.

Pipette 25 ml of the standard solution (6.8.) into a volumetric flask (5.2.).

Dilute to 1000 ml with distilled water and mix.

1 ml of this standard solution contains 2.5 µg of arsenic.

6.10. Lead acetate wool.

Dissolve 50 g of lead acetate trihydrate  $(Pb(CH_3COO)_2$ . 3  $H_2O)$  in 250 ml of distilled water. Saturate some cotton wool with this solution, drain off excess solution and dry under vacuum at ambient temperature.

Store in an air-tight container.

And for the special case :

- 6.11. Glycerol, redistilled ( $\rho = 1.23 \text{ g/ml}$ ).
- 6.12; Magnesium oxide (MgO).
- 6.13. Magnesium nitrate, hexahydrate  $(Mg(NO_3)_2, 6 H_2O)$ , 100 g/1 aqueous solution.

#### 7. GENERAL CASE. PROCEDURE.

## Warning:

Because of the toxicity and unpleasant odour of pyridine, it is recommended that it should be handled with care and in a well-ventilated fume-cupboard.

# 7.1. TEST PORTIONS.

Weigh, to the nearest 0.01 g, two identical 10 g portions of the test sample directly into each of the two conical flasks (5.1.1.).

#### 7.2. Blank test.

Carry out a blank test at the same time as the determination and following the same procedure, using the same quantities of all the reagents.

# 7.3. Preparation of the calibration curve.

The calibration curve has to be constructed each time that a new bottle of zinc is used and each time that a new solution of Ag(DDTC) is prepared.

Into a series of 5 conical flasks (5.1.1.), introduce the volumes of the standard arsenic solution (6.9.) indicated in the following table.

Volume of standard arsenic solution (6.9.)	Corresponding mass of As.
m1 o <sup>(1)</sup>	Mg O
2	. 5
4	10
6	, i 15
8	20

#### (1) Blank solution.

Add to each flask 10 ml of the hydrochloric acid solution (6.3.) and enough distilled water to make approximatively 40 ml. Add 2 ml of the potassium iodide solution (6.5.) and then 0.5 ml of the tin (II) chloride solution (6.6.).

Shake and leave to stand for 15 min.

Place a little of the lead acetate cotton wool in the connecting tube (5.1.2.) to remove from the arsine any hydrogen sulphide that may be produced.

Grease the groud-glass joints with a grease insoluble in pyridine.

Transfer 5 ml, accurately measured from the 10 ml pipette (5.3.), of the Ag(DDTC) solution (6.7.) into the absorption tube (5.1.3.) and reconnect the tube (5.1.2.) to the absorption tube (5.1.3.) by means of the clip.

After the specified 15 min period, place 5 g of zinc granules in the conical flask (5.1.1.) and rapidly connect the apparatus as indicated in figure 1.

Leave for 45 min.

Remove the absorption tube (5.1.3.), shake to dissolve the red precipitate which has been formed in the lower part and mix the solution thoroughly.

The colour of the solution is stable for about 2 hours in the dark and measurements must be carried out within this period.

Measure the absorbance against the blank solution in a 1 cm cell, using the spectrophotometer (5.4.) at the maximum of the absorption curve (wavelength about 540 nm) or with the photocolorimeter (5.5.) fitted with suitable filters.

In each case adjust the instrument to show zero absorbance for the blank solution.

Plot the calibration curve with the concentrations of arsenic expressed in  $\mu g$  per 5 ml of the measured solution as abscissae and the corresponding optical density values as ordinates.

# 7.4. Control test.

To the second test portion, add  $5 \, \mu$ g of arsenic in the form of 2.00 ml of the standard arsenic solution (6.9.), accurately measured from a 2 ml pipette (5.3.).

#### 7.5. Determination.

Add 10 ml water and 10 ml of the hydrochloric acid solution (6.3.) to each of the two conical flasks containing respectively the control test (7.4.) and the first test portion (7.1.).

To each conical flask add distilled water to make up to 40 ml, 2 ml of the potassium iodide solution (6.5.) and 0.5 ml of the tin (II) chloride solution (6.6.).

Shake and allow to stand for 15 min.

Complete the determination as indicated in (7.3.): "Place a little of the lead acetate cotton wool in the connecting tube (5.1.2.)..."

Glycerines

1951

Measure the optical density of the blank test solution and test portion solution in a 1 cm cell, according to the conditions described in (7.3.) having adjusted the instrument to show zero absorbance for distilled water.

#### 8. EXPRESSION OF RESULTS.

By means of the standard calibration graph (7.3.), determine the quantities of arsenic corresponding to the test portion "as is", the control test, test portion enriched with 5 g of arsenic and the blank test; i.e.:

the mass  $m_1$ , in  $\mu g$ , of arsenic found in the test portion, "as is";

the mass m2, in Mg, of arsenic found in the control test portion;

the mass  $m_2$ , in  $\mu g$ , of arsenic found in the blank test (this figure must not exceed 1  $\mu g$ ).

If the difference m<sub>2</sub> - m<sub>1</sub> corresponds to the amount of arsenic added to the second test portion (i.e. 5 µg of arsenic) the assay may be considered to be valid.

In this case, if m is the mass, in g, of the first test portion, the arsenic content of the test sample, expressed in milligrams of arsenic per kilogram of glycerine (ppm), is given by the formula:

$$(m_1 - m_3) \times \frac{1}{1000} \times \frac{1000}{m_0} = \frac{m_1 - m_3}{m_0}$$

Express the result to 1 place of decimals.

#### 9. SPECIAL CASE.

#### 9.1. Foreword.

If the added arsenic is not recovered to the extent of at least 80 per cent, it is necessary to start again, and subject the sample to a preliminary oxidation step in order to destroy the organic matter which inhibits the formation of arsine, without losing any arsenic.

Similarly, the parallel sample, enriched with arsenic, must also be ashed before assay.

Furthermore, in order to prepare the standard calibration graph, the standard solutions must be made up with redistilled glycerol and subjected to the ashing procedure, as otherwise the results will be slightly low.

# 9.2. Principle.

Combustion of the glycerine in the presence of magnesium oxide.

Ignition at 600°C of the residue to which magnesium nitrate has been added.

Dissolution in hydrochloric acid.

Application of the principle given in section 4.

#### 9.3. Procedure.

# 9.3.1. Test portion.

Weigh to the nearest 0.01 g, 2 identical test portions of 10 g each in 2 porcelain evaporating dishes (5.7.).

#### 9.3.2. Blank test.

Carry out a blank test simultaneously with the assay, following the same sequence of operations and using the same quantities of all reagents.

## 9.3.3. Preparation of the standard calibration curve.

Into a series of 5 porcelain evaporating dishes (5.7.) introduce respectively the volumes of standard arsenic solution (6.9.) indicated in the table given in the clause (7.3.).

To each dish add 10 ml of glycerol (6.11) and 2 g of magnesium oxide (6.12), mix by means of a glass stirring-rod.

Warm gently, for example, on a hot plate, until an apparently dry residue is obtained.

Then increase the heating and burn off the glycerol trapped by the magnesium oxide. After the combustion, leave to cool.

Add 10 ml of the magnesium nitrate solution (6.13.).

Evaporate to dryness in the oven (5.8.) regulated at  $150 \pm 10^{\circ}$ C.

Then place the evaporating dishes in a muffle furnace at a temperature between 560 and 600°C for 1 hour.

After cooling, dissolve each residue in a mixture of 20 ml of distilled water and 20 ml of the hydrochloric acid solution (6.3.), stirring with a glass rod.

Transfer the solutions quantitatively to two of the conical flasks (5.1.1.).

Add 2 ml of the potassium iodide solution (6.5.). followed by 0.5 ml of the tin (II) chloride solution (6.6.) to each flask.

Shake and leave to stand for 15 min.

Follow the procedure described in (7.3.) from paragraph:

"Place a little of the lead acetate cotton wool in the connecting tube (5.1.2.)..."

Carry out the photometric measurements as indicated in (7.3.).

Plot the calibration curve as indicated in (7.3.).

9.3.4. Control test.

To the second test portion (9.3.1), add  $5 \mu g$  of arsenic in the form of 2.00 ml of the standard arsenic solution (6.9.), accurately measured from a 2 ml pipette (5.3.).

9.3.5. Determination.

Add 2 g of magnesium oxide (6.12) to each of the two porcelain dishes containing respectively, the first test portion (9.3.1) and the control test (9.3.4.).

Follow the procedure specified in (9.3.3.) starting from the paragraph: "Warm gently ..."

Carry out the photometric measurements according to the procedure specified in (7.3.).

9.3.6. Expression of results.

Calculate the results as indicated in section 8.

#### 10. SENSITIVITY AND ACCURACY.

Qualitatively, the method permits the detection of  $5\,\mathrm{Mg}$  of arsenic in the final solution examined in the spectrophotometer.

Quantitatively, an amount of 15  $\mu g$  of arsenic can be determined to within  $^{+}$  10 per cent (13.5 - 16.5  $\mu g$ ) in the final solution.

Using a test sample of 10 g of glycerol, the method enables the detection of 0.5 ppm and the determination of 1.5 ppm of arsenic.

# 11. NOTES.

- Ignition should also be applied to the samples prepared from redistilled glycerol, in order to construct the calibration curve, otherwise the results obtained will be slightly below the correct values.
- The apparatus illustrated in the figure is only one example of a suitable apparatus for this determination.
- 3. It is advisable to have three sets of the apparatus illustrated so that the determination described in sections 7 or 9 can be carried out simultaneously.
- 4. When for one reason or another zinc filings are used instead of granules, the following modifications should be made in the method:
  - (6.2.) Read: "Zinc, filings 2 3 mm in diameter;
  - (7.3.) Replace the first paragraph under the table by: "Add to each flask 10 ml of the hydrochloric acid solution (6.3.) and enough distilled water to make approximatively 30 ml; replace the eighth paragraph under the table by:

"Leave for about 1 hour", (instead of 45 min.).

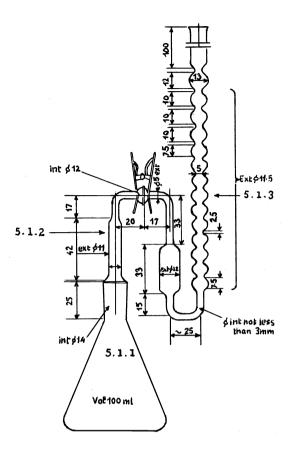


Fig. 1

# III. GLYCERINES

1980

3.137 CALCULATION OF MATTER (ORGANIC) NON-GLYCEROL. - MONG.
(Fifth Edition: Method III.A.6)

#### 1. SCOPE AND FIELD OF APPLICATION.

This Standard gives the definition and the method of calculation of the content of Matter (Organic) Non-Glycerol (MONG) in industrial glycerines.

This determination is prefered to that of the non-volatile organic residue, which is lengthy and leads to less reproducible results.

# 2. DEFINITION.

The Matter (Organic) Non-Glycerol (MONG) represents, by convention, the difference obtained by subtracting from 100 the sum of the contents, expressed as percentages of glycerol, ash and water.

#### 3. CALCULATION.

The content of Matter (Organic) Non-Glycerol (MONG) is given as a percentage (m/m) by the formula:

$$100 - (A + B + C)$$

- where: A is the glycerol content, expressed as a percentage (m/m), found by following the procedure described in 3.121;
  - B is the ash content, expressed as a percentage (m/m), found by following the procedure described in 3.135;
  - C is the water content, expressed as a percentage (m/m), found by following the procedure described in 3.131.

Express the result to the first decimal place.