
British Standard Methods of

Sampling and test for glycerol

Part 10. Determination of arsenic content: silver diethyldithiocarbamate method

[ISO title: Glycerols for industrial use — Determination of arsenic content —
Silver diethyldithiocarbamate photometric method]

Méthodes d'échantillonnage et d'essais pour le glycérol

Partie 10. Dosage de l'arsenic, méthode au diéthylthiocarbamate d'argent

Verfahren für die Probenahme und Prüfung von Glycerin

Teil 4. Bestimmung des Arsengehaltes, Verfahren mit Silberdiethyldithiokarbamate

NOTE. It is recommended that this Part of BS 5711 be read in conjunction with the general information given in BS 5711 : Part 0 'General introduction', issued separately.

National foreword

This Part of BS 5711 is identical with ISO 2465 'Glycerols for industrial use — Determination of arsenic content — Silver diethyldithiocarbamate method'.

For each of the references in the text to ISO 2590 'General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method' a corresponding reference to BS 4404 : 1968 'Method for the determination of arsenic-silver diethyldithiocarbamate procedure' applies, as follows.

ISO 2590	BS 4404	ISO 2590	BS 4404
Clause number	Clause number	Clause number	Clause number
3	2	5.1.1	4.1(1)
4	5	6	6
4.1	5.2	6.2	6.2.3
4.4	5.6	6.3	6.1
4.5	5.8	6.3.1	6.1.1
4.6	5.3	6.3.2	6.1.2
4.7	5.4	6.3.3	6.1.3
5	4	6.4.1	6.1.2
5.1	4.1		

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a silver diethyldithiocarbamate photometric method for the determination of the arsenic content of glycerols for industrial use.

1.1 General case

The method is applicable to the analysis of glycerols having an arsenic content greater than 0,1 mg/kg. It is applied successively to a test portion as it is, and to another identical portion with a known quantity of arsenic added. The determination is considered to be valid if the amount added is recovered.

1.2 Special case

If the arsenic added is not recovered or is recovered only partially, it is advisable to recommence the determination, after first igniting the test portion in an oxidizing medium in order to destroy the organic substances capable of inhibiting the arsine formation, without loss of arsenic.

A control test is also carried out in this case, after ignition, on an identical sample enriched by a known amount of arsenic, which should be recovered.

NOTE — Ignition should also be applied to the standard matching samples prepared with the aid of double-distilled glycerol in order to prepare the calibration curve, otherwise the results obtained will be slightly below the correct values.

2 REFERENCES

ISO 2096, *Glycerols for industrial use — Methods of sampling*.

ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method*.

3 PRINCIPLE

See ISO 2590, clause 3.

4 REAGENTS

See ISO 2590, clause 4.

5 APPARATUS

See ISO 2590, clause 5.

6 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.

6.1 Test portions

Weigh, to the nearest 0,1 g, two identical portions of 10 g of the test sample (see ISO 2096), directly into each of two of the conical flasks (5.1.1).

6.2 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.

6.3 Preparation of the calibration curve

See ISO 2590, sub-clause 6.3

6.3.1 Preparation of standard matching solutions

See ISO 2590, sub-clause 6.3.1, modifying the text as follows :

- Delete the note.
- Amend the third paragraph after the note to read :
"After allowing to stand for 15 min, dilute the test solution to 90 ml with water, add to the conical flask (5.1.1) ... "

6.3.2 Photometric measurements

See ISO 2590, sub-clause 6.3.2.

6.3.3 Plotting the calibration curve

See ISO 2590, sub-clause 6.3.3.