BRITISH STANDARD 1594 : 1950

DIACETIN

(GLYCERYL DIACETATE)

REVISED PRICE 2/6

Price 2/– net, post free

BRITISH STANDARDS INSTITUTION

Incorporated by Royal Charter

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THIS BRITISH STANDARD, having been approved by the Fine Chemicals Industry Standards Committee and endorsed by the chairman of the Chemical Divisional Council, was published under the authority of the General Council on 24th March, 1950.

The Institution desires to call attention to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

In order to keep abreast of progress in the industries concerned, British Standards are subject to periodical review. Suggestions for improvements will be recorded and in due course brought to the notice of the committees charged with the revision of the standards to which they refer.

A complete list of British Standards, numbering over 1600, indexed and cross-indexed for reference, together with an abstract of each standard, will be found in the Institution's Yearbook, price 5s. post free.

British Standards are revised, when necessary, by the issue either of amendment slips or of revised editions. It is important that users of British Standards should ascertain that they are in possession of the latest amendments or edition.

Users wishing to be kept informed of any alteration to this standard should notify Sales and Distribution Department of the Institution, giving the number and title of the standard.

FCC/4

CONTENTS

PD. 3838

CONFIRMATION OF B.S. 1594 : 1950

Diacetin (glyceryl diacetate)

This British Standard has recently been reviewed in accordance with B.S.I. procedure and has been confirmed as satisfying present requirements.

June 1960

CO-OPERATING ORGANIZATIONS

The Fine Chemicals Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives of the following :—

Association of British Chemical Manufacturers
Ministry of Supply

The above, together with the following Government departments and scientific and industrial organizations, were directly represented on the committee entrusted with the preparation of this British Standard :—

Admiralty
Association of Cellulose Lacquer Manufacturers
British Plastics Federation
Department of the Government Chemist
National Paint Federation
Oil and Colour Chemists' Association
Society of Chemical Industry
Individual manufacturers

BRITISH STANDARD SPECIFICATION FOR

DIACETIN (GLYCERYL DIACETATE)

FOREWORD

This standard forms one of a series of British Standards for solvents and allied products, the preparation of which was authorized by the Fine Chemicals Industry Standards Committee.

Other standards in the series are :

B.S. 506. Methyl alcohol (Methanol).
B.S. 507. Ethyl alcohol (Ethanol).
B.S. 508. Normal butyl alcohol (Butanol).
B.S. 509. Acetone.
B.S. 549. Diacetone alcohol.
B.S. 551. Normal butyl acetate.
B.S. 552. Amyl acetate.
B.S. 553. Ethyl acetate.
B.S. 573. Dibutyl phthalate.
B.S. 574. Diethyl phthalate.
B.S. 575. Carbon tetrachloride.
B.S. 576. Acetic acid.
B.S. 577. Hexachlorethane.
B.S. 579. Technical ether.
B.S. 580. Trichlorethylene (Types A, B and C).
B.S. 662. Carbon disulphide.
B.S. 663. Ethyl lactate.
B.S. 1593. Perchlorethylene.
B.S. 1595. Isopropyl alcohol (Isopropanol).

This specification was originally one of a series issued by the Ministry of Aircraft Production to meet a requirement not then covered by a British Standard. It was issued under the title ' Material specification—Diacetin. No. D.T.D.661, March, 1945.' Owing to the increased demand for this material in industry, it has now been decided to include the specification in the series of British Standards for solvents.

SPECIFICATION

DESCRIPTION

1. British Standard diacetin shall be clear, colourless and free from matter in suspension, and shall consist essentially of the diacetic esters of glycerol, $C_3H_5(OH)(O.COCH_3)_2$.

NOTE. The material conforming to this specification is unlikely to be miscible with water and should not be used where this property is required.

SPECIFIC GRAVITY

2. The specific gravity of the material at 15·5°C/15·5°C. shall be not lower than 1·180 and not higher than 1·195, or at 20°C/20°C. shall be not lower than 1·175 and not higher than 1·190.

ASH

3. The material shall not leave more than 0·02 per cent by weight of ash when tested in the manner described in Appendix A.

ACIDITY

4. The material shall not contain more than 0·4 per cent by weight of acidity, calculated as acetic acid, CH_3COOH, and determined in the manner described in Appendix B.

ESTER CONTENT

5. The material shall show an ester content of not less than 94 per cent and not more than 101 per cent by weight, calculated as glyceryl diacetate, $C_3H_5(OH)(O.COCH_3)_2$, and determined in the manner described in Appendix C.

WATER AND GLYCEROL

6. The material shall not show any opalescence when tested by the method described in Appendix D.

SULPHATES

7. The material shall not contain more than 0·05 per cent by weight of sulphates, calculated as SO_4, and determined in the manner described in Appendix E.

CHLORIDES

8. The material shall not contain more than 0·05 per cent by weight of chlorides, calculated as chlorine, Cl, and determined in the manner described in Appendix F.

SAMPLING AND SIZE OF SAMPLE

9. A representative sample of the material, taken from the bulk and measuring not less than half a litre, is necessary for the purpose of examination under this specification. The sample shall be placed in a clean, dry and air-tight glass-stoppered bottle of such a size that it is nearly filled by the sample.

When it is necessary to seal the container, care shall be taken to avoid the risk of contaminating the sample in any way.

APPENDIX A

METHOD FOR THE DETERMINATION OF ASH

Slowly burn 50 ml. of the diacetin, in several portions, in a weighed platinum or silica basin, and gently ignite until all carbonaceous matter has disappeared. Cool in a desiccator and weigh.

Calculate the ash as per cent by weight of sample.

APPENDIX B

METHOD FOR THE DETERMINATION OF ACIDITY

Thoroughly shake 25 ml. of the diacetin with 50 ml. of recently boiled and cooled distilled water. Titrate with 0·1N. sodium hydroxide, using 3 drops (approx. 0·15 ml.) of bromothymol blue indicator. Calculate the acidity thus found as acetic acid, CH_3COOH, per cent by weight of sample.

1 ml. 0·1N-NaOH = 0·006 g. CH_3COOH.

NOTE. The indicator is prepared by warming 0·1g. of bromothymol blue with 1·6 ml. of 0·1 N. sodium hydroxide and 50 ml. of ethyl alcohol (95 per cent), and diluting to 250 ml. with water.

APPENDIX C

METHOD FOR THE DETERMINATION OF ESTER CONTENT

In each of two dry 250 ml. conical flasks with ground glass joints place 50 ml. of approximately N. alcoholic (ethyl) potassium hydroxide. Close the flasks with their glass stoppers. Transfer by means of a Lunge-Rey pipette an accurately weighed amount (1·5–2·2 g.) of the diacetin into one of the flasks.

Attach the flasks to water-cooled reflux condensers with ground glass joints, and heat for one hour in boiling water. Withdraw the flasks, still carrying their condensers, and immerse them in cold water. When cool, wash down the inside of each condenser with two 20 ml. portions of distilled water, well boiled and cooled. Disconnect the flasks and wash each joint with a further 20 ml. of the distilled water Add 0·5 ml. of phenolphthalein indicator and titrate immediately with N. hydrochloric acid until the pink colour is just discharged. From the difference between the titrations calculate the ester content expressed as glyceryl diacetate, using the following formula :—

$$\frac{8{\cdot}8\ (B-A)}{\text{wt. of sample}} - 1{\cdot}46C = \text{per cent by weight glyceryl diacetate,}$$

where A = ml. N-HCl required by test,

B = ml. N-HCl required by blank,

and C = per cent by weight of acetic acid determined as described in Appendix B.

NOTE 1. The correction for acidity is necessary in this case owing to the high acidity limit allowable.

NOTE 2. The indicator is prepared by dissolving 0·5 g. of phenolphthalein in 100 ml. of ethyl alcohol (95 per cent) and is made faintly pink by the addition of dilute sodium hydroxide solution.

APPENDIX D

METHOD FOR THE DETECTION OF WATER AND GLYCEROL

Mix 50 ml. of benzene (analytical reagent quality) with 10 ml. of distilled water and shake vigorously from time to time for 30 minutes. Allow the mixture to separate into two layers. Remove 25 ml. of the clear benzene and add it to 10 ml. of the diacetin in a dry glass-stoppered cylinder of about 100 ml. capacity. Allow to stand for 30 minutes, shake vigorously and examine for opalescence, which indicates the presence of water or glycerol or both. Carry out the test at a temperature of 20 $\pm$ 1°C.

APPENDIX E

METHOD FOR THE DETERMINATION OF SULPHATES

Dilute 50 ml. of the diacetin to 100 ml. with distilled water and heat to boiling point. To the hot liquid add 5 ml. of a boiling solution of barium chloride (10 per cent) drop by drop ; continue boiling for a few minutes and set aside on a hot plate for at least 4 hours. Wash the precipitate with hot water by decantation, pouring the supernatant liquid and washings through a prepared Gooch crucible fitted with an asbestos pad. Transfer the precipitate to the filter and wash it thoroughly with hot water. Finally dry the crucible and ignite it to constant weight. (Alternatively a filter paper may be used. In this case after ignition, cool, add one drop of concentrated sulphuric acid and one drop of fuming nitric acid, heat gently to evaporate the acids and ignite to constant weight.)

From the increase in weight of the crucible calculate the sulphates, expressed as SO_4, as per cent by weight of sample.

$$BaSO_4 \times 0{\cdot}4115 = SO_4.$$

APPENDIX F

METHOD FOR THE DETERMINATION OF CHLORIDES

Dilute 25 ml. of the diacetin to 100 ml. with distilled water. Add 2 ml. of glacial acetic acid (analytical reagent quality) and 1 ml. of potassium chromate solution (5 per cent) and titrate with 0·1 N. silver nitrate solution until the red colouration is permanent.

Calculate the chlorides thus found as chlorine, Cl, per cent by weight of sample.

$$1 \text{ ml. } 0{\cdot}1\text{N-}AgNO_3 = 0{\cdot}00355 \text{ g. Cl.}$$

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24/28 VICTORIA STREET, LONDON, S.W.1

BRITISH STANDARDS INSTITUTION

The British Standards Institution was founded in 1901 and incorporated by Royal Charter in 1929.

The principal objects of the Institution are to co-ordinate the efforts of producers and users for the improvement, standardization and simplification of engineering and industrial materials ; to simplify production and distribution ; to eliminate the waste of time and material involved in the production of an unnecessary variety of patterns and sizes of articles for one and the same purpose ; to set up standards of quality and dimensions, and promote the general adoption of British Standards.

In all its endeavours the Institution maintains the community of interest of purchaser and producer. It does not embark on any work without first ascertaining that there is a consensus of opinion in favour of its being proceeded with, and that it is to fulfil a recognised want.

This national work is carried on largely by means of grants received from the Government, professional institutions and industrial and trade organizations, as well as by the sale of its publications. The amount derived from these sources is, however, not sufficient, and the Institution has to look to industry as a whole for the further funds necessary to enable it to meet the increasing demands made upon it.

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