

CONFIRMED
DECEMBER 2007

Specification for

Ammonia distillation apparatus (Markham) —

Microchemical apparatus —

Group B: apparatus for the
determination of elements by other
than combustion methods

Co-operating organizations

The Scientific Glassware and Related Laboratory Apparatus Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Admiralty*
 Air Ministry
 Association of British Chemical Manufacturers*
 Association of Scientific Workers
 Board of Trade
 British Association for the Advancement of Science
 British Chemical Ware Manufacturers' Association*
 British Laboratory Ware Association*
 British Lamplown Scientific Glassware Manufacturers' Association*
 British Pharmacopoeia Commission
 British Scientific Instrument Research Association*
 Chemical Society*
 D.S.I.R.—Laboratory of the Government Chemist*
 D.S.I.R.—National Chemical Laboratory*
 Glass Manufacturers' Federation*
 Institute of Petroleum
 Ministry of Aviation*
 Ministry of Health
 National Physical Laboratory (D.S.I.R.)*
 Oil Companies Materials Association
 Pharmaceutical Society of Great Britain*
 Royal Institute of Chemistry*
 Science Masters' Association
 Society for Analytical Chemistry*
 Society of Chemical Industry*
 Society of Glass Technology*
 Standardization of Tar Products Tests Committee

The Government departments and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this standard:

Association of Clinical Pathologists
 British Rubber Producers' Research Association
 Medical Research Council
 Physiological Society
 War Office
 Individual Manufacturers

This British Standard, having been approved by the Scientific Glassware and Related Laboratory Apparatus Industry Standards Committee and endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the General Council on 29 February 1960

© BSI 12-1999

First published December 1953
 First revision February 1960

The following BSI references relate to the work on this standard:
 Committee references LBC/11 and LBC/11/1
 Draft for comment CZ (LBC)5632

ISBN 0 580 34147 X

Amendments issued since publication

Amd. No.	Date	Comments

Contents

	Page
Co-operating organizations	Inside front cover
Foreword	ii
<hr/>	
1 Scope	1
2 Material	1
3 Design and dimensions	1
4 Alternative apparatus with ground joint	1
<hr/>	
Appendix A Notes on method of use	2
Appendix B	4
<hr/>	
Figure 1 — Markham ammonia distillation apparatus	3
<hr/>	

Foreword

This standard makes reference to the following British Standards:

BS 1428-A2, *Nitrogen combustion train (micro-Dumas)*.

BS 1428-B1, *Nitrogen determination apparatus (micro-Kjeldahl)*.

BS 1848, *Glass condensers*.

BS 2761, *Spherical ground glass joints*.

This standard, which was first published in 1953, is one of a series of Parts of BS 1428 "*Microchemical Apparatus*", (see list in Appendix B). The only substantial changes in the present revision are that the design of the condenser is now that specified in BS 1848¹⁾ and that the alternative, jointed apparatus now employs only the spherical joint introduced by amendment in 1956, the conical joint previously permitted having been deleted.

Part B2 specifies an ammonia distillation apparatus of the type described by Markham²⁾, which has been found especially useful for biochemical work. It has disadvantages if the quantity of distillant used exceeds 25 ml, and in this case more satisfactory results may be obtained by the addition of a splash-head, of the type specified in BS 1428- B1³⁾, between the distillation vessel and the condenser.

While only the apparatus is specified in this and other parts of BS 1428, it must be made clear that satisfactory results will not be obtained without careful attention to the procedure in carrying out the determination, and that this requires both training and experience in microchemical work. Notes on the method of use of the apparatus are given for guidance in Appendix A, but these are not intended to be either mandatory or comprehensive.

Apparatus for the micro-determination of nitrogen is specified also in BS 1428-A2⁴⁾ and BS 1428-B1³⁾

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

¹⁾ BS 1848, "*Glass condensers*".

²⁾ R. MARKHAM, *Biochem. J.*, 36, 1942, 790.

³⁾ BS 1428-B1, "*Nitrogen determination apparatus (micro-Kjeldahl)*".

⁴⁾ BS 1428-A2, "*Nitrogen combustion train (micro-Dumas)*".

1 Scope

This British Standard specifies steam-jacketed distillation apparatus of the Markham type for the micro-determination of nitrogen by the Kjeldahl method.

2 Material

The apparatus shall be constructed throughout of borosilicate glass, as free as possible from visible defects, and shall be well annealed.

3 Design and dimensions

The apparatus shall conform to the general design shown in Figure 1 and shall comply with the mandatory dimensions, which are underlined in the figure. Dimensions not underlined in the figure are recommended. The connection between the filling funnel and the main body, and also the glass tubing between the distillation vessel and the condenser, shall be robust.

4 Alternative apparatus with ground joint

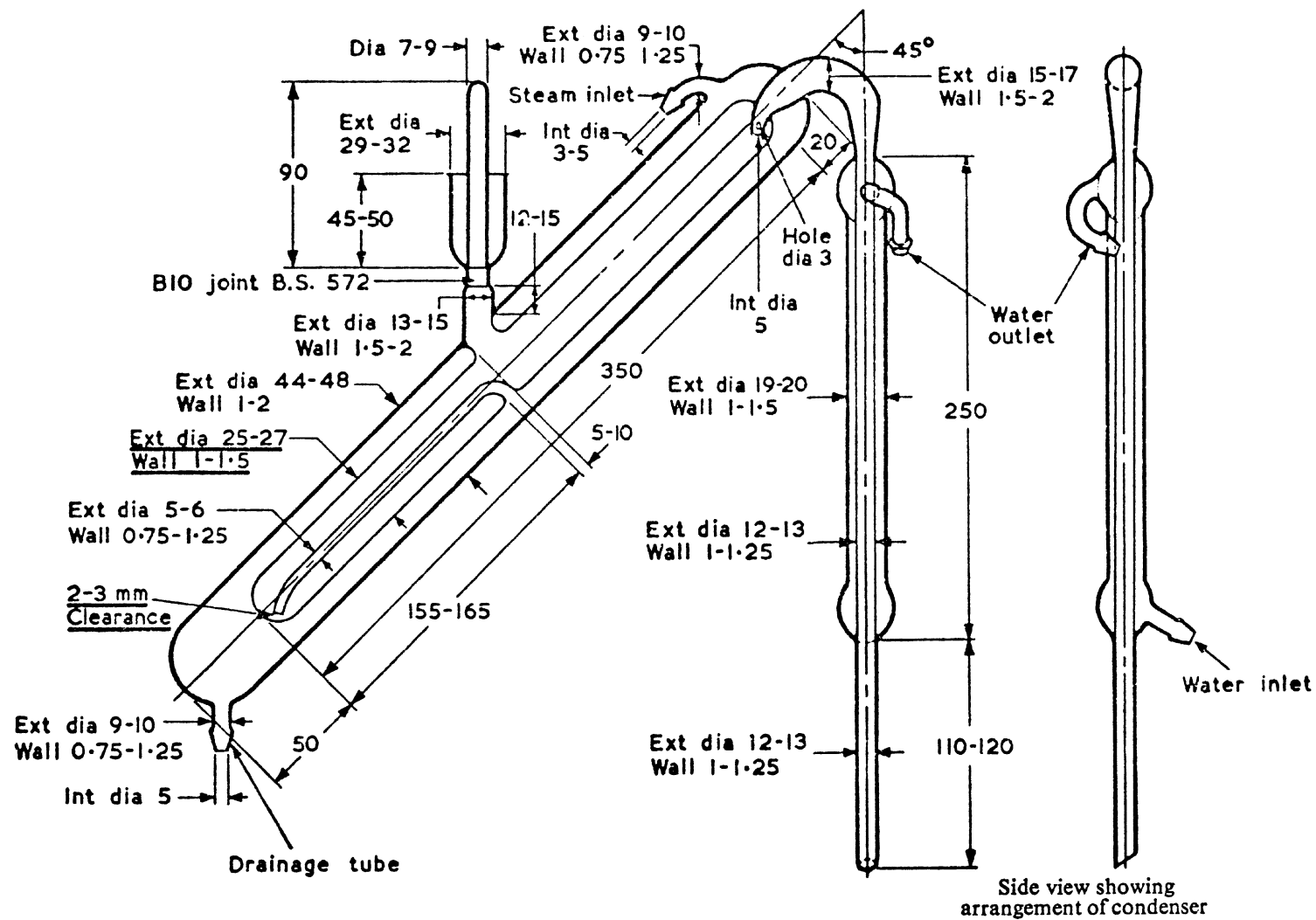
As an alternative to the one-piece apparatus illustrated in Figure 1, an interchangeable spherical ground glass joint, size 18/9 BS, complying with BS 2761⁵⁾ may be inserted between the distillation vessel and the condenser. The ball is fitted to the vertical portion of the outlet tube of the distillation vessel and the cup to the top of the condenser.

⁵⁾ BS 2761, "Spherical ground glass joints".

Appendix A Notes on method of use

Transfer the digested sample to the distillation vessel through the funnel and pass steam through the jacket, leaving the drainage tube open, until the apparatus is sufficiently warm for condensation not to occur. Add excess alkali (and a reagent to decompose mercury-ammonia complexes if mercury has been used in the digestion) and close the rubber tubing on the drainage tube with a spring clip so that steam passes into the distillation vessel, thus sweeping ammonia through the condenser. Continue the steam distillation under carefully controlled conditions until all the ammonia has been absorbed in a suitable solution in a conical flask and determine it by any suitable method.

After the distillation is complete, cut off the supply of steam so that the contents of the distillation vessel are sucked into the main body of the apparatus. Wash out and empty the distillation vessel by the same method ready for the next distillation.



All dimensions are in millimetres. Dimensions underlined are mandatory, others are recommended.

Figure 1 — Markham ammonia distillation apparatus

Appendix B

Parts of BS 1428, "Microchemical apparatus"

Group A. *Combustion trains for the determination of elements.*

- Part A1: Carbon and hydrogen combustion train (Pregl type);
- Part A2: Nitrogen combustion train (micro-Dumas);
- Part A3: Halogens and sulphur combustion train (Pregl);
- Part A4: Halogens and sulphur combustion train (micro-Grote);
- Part A5: Rapid method combustion tubes (Belcher and Ingram type).

Group B. *Apparatus for the determination of elements by other than combustion methods.*

- Part B1: Nitrogen determination apparatus (micro-Kjeldahl);
- Part B2: Ammonia distillation apparatus (Markham).

Group C. *Apparatus for the determination of organic groups.*

- Part C1: Alkoxy and alkylimino group determination apparatus;
- Part C2: Acetyl group determination apparatus (Wiesenberger).

Group D. *Volumetric apparatus.*

- Part D1: Burettes with pressure-filling device and automatic zero;
- Part D2: Washout pipettes;
- Part D3: Micro-nitrometer (Pregl type);
- Part D4: Capillary pipettes;
- Part D5: Syringe pattern micro-pipette;
- Part D6: Micrometer-operated burette.

Group E. *General accessory apparatus.*

- Part E1: Crucibles for microchemical analysis;
- Part E2: Micro-beakers;
- Part E3: Micro-centrifuge accessories.

Group F. *Filtration accessories.*

- Part F1: Filtration apparatus for microchemical analysis.

Group G. *Heating, cooling and drying accessories.*

- Part G1: Heating and cooling blocks for microchemical purposes;
- Part G2: Vacuum drying ovens for microchemical purposes.

Group H. *Weighing accessories.*

- Part H1: Weighing vessels for microchemical analysis.

Group I. *Combustion accessories.*

- Part I1: Combustion boats and sheath for microchemical analysis.

Group J. *Electrolytic accessories.*

- Part J1: Micro-electrolytic apparatus.

Group K. *Apparatus for physical methods.*

- Part K1: Vaporimetric molecular weight apparatus.

Group L. *Extraction accessories.*

(No Parts yet published).

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: 020 8996 9000. Fax: 020 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: 020 8996 9001. Fax: 020 8996 7001.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: 020 8996 7111. Fax: 020 8996 7048.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: 020 8996 7002. Fax: 020 8996 7001.

Copyright

Copyright subsists in all BSI publications. BSI also holds the copyright, in the UK, of the publications of the international standardization bodies. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI.

This does not preclude the free use, in the course of implementing the standard, of necessary details such as symbols, and size, type or grade designations. If these details are to be used for any other purpose than implementation then the prior written permission of BSI must be obtained.

If permission is granted, the terms may include royalty payments or a licensing agreement. Details and advice can be obtained from the Copyright Manager. Tel: 020 8996 7070.