



BS 757 : 1975

UDC 668.317.001.4:543

Methods for
Sampling and testing gelatine
(physical and chemical methods)

Gr 8
British Standards Institution

This British Standard, having been approved by the Adhesives Industry Standards Committee, was published under the authority of the Executive Board on 31 July 1975.

© British Standards Institution, 1975
 First published November 1937
 First revision June 1944
 Second revision March 1959
 Third revision 31 July 1975.

ISBN: 0 580 08037 4

Copyright

Users of British Standards are reminded that copyright subsists in all BSI publications. No part of this publication may be reproduced in any form without the prior permission in writing of 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. Enquiries should be addressed to the BSI Secretariat.

Contract requirements

Attention is drawn to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

Revision of British Standards

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.

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 editions.** Full information on all BSI publications, numbering over 6000, will be found in the *British Standards Yearbook*. This information is supplemented by details published each month in *BSI News* of new publications, revisions and amendments.

BSI News is available to Subscribing Members of the Institution. All other publications may be purchased direct from Sales Department. Enquiries concerning subscribing membership, which has considerable advantages, will be welcomed, and should be made to Subscriptions Department. Both departments are located at:

101 Pentonville Road, London N1 9ND (Telephone 01-837 8801 ; Telex 23218).

The following BSI references relate to the work on this standard:
 Committee reference ADC/19 Draft for comment 71/53176

Co-operating organizations

The Adhesive Industry Standards Committee, under whose supervision this British Standard was prepared consists of representations from the following Government departments and scientific and industrial organizations.

- | | |
|---|--|
| Association of British Plywood and Veneer Manufacturers | *Department of Trade and Industry |
| British Adhesive Manufacturers' Association | *Federation of Gelatine and Glue Manufacturers |
| *British Food Manufacturing Industries Research Association | Ministry of Defence |
| British Furniture Manufacturers' Federated Associations | PIRA (the Research Association for the Paper and Board |
| British Paper Bag Association | Printing and Packaging Industries) |
| British Plastics Federation | Royal Institute of British Architects |
| British Woodwork Manufacturers' Association | Wallpaper Manufacturers Limited |
| *Department of the Environment | |

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 British Standard.

- | | |
|---|--|
| British Paper and Board Makers' Association | Technical Section of the British Paper and Board Makers' |
| Coated Abrasives Manufacturers' Association | Association |
| Cocoa, Chocolate and Confectionery Alliance | Individual expert |
| Food Manufacturers' Federation Incorporated | |



BS 757 : 1975

UDC 668.317.001.4:543

Methods for
Sampling and testing gelatine
(physical and chemical methods)

Amendments issued since publication

Amd. No.	Date of issue	Text affected

British Standards Institution · 2 Park Street · London W1A 2BS

Telephone 01-629 9000
Telex 266933

BS 757 : 1975

Contents

	Page		Page
Co-operating organizations	Inside front cover		
Foreword	2	19. Methods for the determination of copper content	23
Methods		20. Method for the determination of zinc content	26
1. Scope	4	21. Method for the determination of lead content	28
2. References	4		
3. Sampling	4	Appendix	
4. Test preparation	5	A. Importance of moisture content	30
5. Method for the determination of moisture content	6		
6. Method for the comparison of jelly strength	6	Tables	
7. Methods for the measurement of jelly strength	7	1. Minimum number of items to be sampled	4
8. Method for the determination of viscosity	13	2. Test quantities	5
9. Method for the determination of melting point	13		
10. Method for the determination of the setting point	15	Figures	
11. Method for the determination of water absorption	15	1. Bloom type gelometer	8
12. Method for the determination of keeping quality	15	2. Bloom type gelometer: detail of relay and contact system	9
13. Method for the determination of pH	16	3. Detail of plunger for gelometer	10
14. Method for the determination of grease content	16	4. Dummy Bloom device	10
15. Method for the determination of ash	17	5. Boucher electronic jelly taster	11
16. Methods for the determination of sulphur dioxide content	17	6. Determination of melting point: positions of test tube	14
17. Method for the determination of chloride content	21	7. Apparatus for the determination of sulphur dioxide content (method 1)	18
18. Methods for the determination of arsenic content	21	8. Apparatus for the determination of sulphur dioxide content (method 2)	20
		9. Apparatus for the determination of arsenic content (method 2)	23

Foreword

Introduction. Gelatine is obtained by the hydrolysis of purified skin or bone material; its properties depend on the molar mass, and also the extent to which the purification of the materials has been carried out. The tests to be described, therefore, come under two heads:

- (1) physical tests, the results of which are mainly determined by the degree of hydrolysis, and
- (2) chemical tests, which determine freedom from associated substances.

It is not necessary for any one particular trade to use all the tests described. In practice, it is usual for the user to find by experience the most suitable gelatine for his purpose and check future deliveries in respect of those properties which are relevant.

Amongst these properties, jelly strength, viscosity and melting point or any combination of them are usually included as an index of the grade. The chemical tests are particularly necessary when testing edible products.

The keeping quality test is intended simply as an index of the behaviour of the gelatine, particularly technical gelatine, in practice, and it should not be taken as a substitute for any bacteriological examination. A standard giving methods for the bacteriological examination of gelatine is in course of preparation.

For certain uses there are also specialized tests, agreed between buyer and seller, which are not suitable for inclusion in the present standard. (For example, the colour of a gelatine or the colour and clarity of a jelly is of particular importance in many industries. At present, the usual method of assessing these properties is by visual comparison with a reference sample.)

It is very necessary, particularly with physical tests, to follow carefully the technique described, as only in this way can consistent results be obtained.

BS 757 : 1975

Sampling. The results of the analysis of gelatine carried out in accordance with the information given are limited in their practical use by the degree to which the 1 kg final laboratory sample represents the whole delivery. Rules have therefore been drawn up for guidance in bulk sampling.

Revision. In the present revision several changes from the previous edition have been made, as follows:

- (1) In accordance with the change from inch to metric units the standard units are metric.
- (2) A second method for the measurement of jelly strength using the Boucher electronic jelly tester has been introduced.
- (3) A method for the determination of the setting-point of gelatine has been provided.
- (4) An alternative method for the determination of the melting point has been introduced.
- (5) The method for determination of keeping quality has been revised.
- (6) New alternative methods for the determination of arsenic and copper contents have been introduced.

BS 757 : 1975

British Standard Methods for Sampling and testing gelatine (physical and chemical methods)

1. Scope

This British Standard specifies physical and chemical methods to be used for sampling and testing gelatine.

2. References

The titles of the British Standards referred to in this standard are listed on the inside back cover.

3. Sampling

3.1 Introduction. The successive sampling stages from the initial consignment or lot down to a suitable test sample are described with reference to the physical state of the gelatine involved.

NOTE. As soon as any sample is taken it should be placed in a clean, dry, air tight, non absorbent container.

3.2 Primary sample. The number of containers from which this is obtained should be by agreement between the vendor and the purchaser. As a guide, suggestions for the number of containers to be sampled are given in 3.2.1. From the containers chosen, take samples of the amount indicated in 3.2.2 by the method described in 3.2.3 or 3.2.4 according to the physical state of the gelatine.

3.2.1 Number of containers to be sampled from a lot. This should be the next higher integer to three times the cube root of the number of containers in the lot, except when the number of containers is small, as indicated in Table 1.

Table 1. Minimum number of items to be sampled

Number of items in the lot	Minimum number to be sampled
1-10	All the items
11-49	11
50-64	12
65-81	13
82-101	14
102-125	15
126-151	16
152-181	17
182-216	18
217-254	19
255-296	20
297-343	21
344-394	22
395-450	23
451-512	24

3.2.2 Amount to be taken from each container. This should be in proportion to the number and size of containers as follows:

One container only to be sampled	Not less than 1 kg for containers of 50 kg or more of gelatine
Two or more containers to be sampled	Not less than 500 g from each container of 50 kg or more of gelatine

For containers of less than 50 kg of gelatine, take a proportionate amount.

3.2.3 Sheet gelatine. Take the whole or equal proportions of not fewer than four sheets, representative of the content of the container.

BS 757 : 1975

3.2.4 Powdered, pearl, granulated, flake and kibbled gelatine. Sample by means of a sampling tube, scoop or similar tool to ensure gelatine being taken from the top, middle and bottom of the container.

3.3 Preparation of final lot samples. These should be not less than 1 kg, obtained as follows.

3.3.1 Sheet gelatine. Break or cut a representative piece from each sheet of the primary sample, taking the same proportion of each one. If it is necessary to reduce the amount of sample, break up the pieces as much as possible then mix and quarter until a sample of not less than 1 kg is obtained.

3.3.2 Powdered, pearl, granulated, flake and kibbled gelatine. Mix the material thoroughly and take the sample from the bulk.

3.4 Preparation of laboratory samples and test samples. If the final lot sample provided is greater than 1.5 kg, reduce it to about 1 kg by mixing and quartering before grinding, as described in 3.4.1, to produce the laboratory sample.

3.4.1 Laboratory sample. Prepare a separate test sample for moisture content (see note 1) and grind the remainder of the 1 kg sample in a mortar or alternatively in a laboratory disintegrator of a type agreed between purchaser and vendor to a grist of approximately 3 mm.

NOTE 1. In view of the loss of moisture in grinding, it is necessary to take a separate sample for the moisture content test from the quantity obtained by the preliminary breaking up, before putting through the disintegrator. This small separate sample can then be ground in a mortar, or cut with scissors, and is suitable for the moisture content test. Adjustment should be made for the moisture content of the mechanically ground sample by comparing it with the hand-ground sample, which should be taken as having the correct moisture content. By comparing the actual moisture content found on the hand-ground sample with the moisture content of the mechanically-ground sample, the adjustment that should be made to the mass of the gelatine to be used in the various tests can be found by calculation.

NOTE 2. It is important to put all samples immediately into air-tight containers because even a small change in the moisture content will affect the result of some of the tests; for example, an increase of 1 % in the moisture content figure would result in a decrease of about 2.5 % in jelly strength when determined on the Bloom type gelometer.

3.4.2 Test sample. Mix and quarter the approximately 1 kg of ground gelatine to bring the final mass to 500 g, taking care that a representative amount of all grists is included in the quartering. Use the 500 g obtained for all the tests except for the moisture content (see note 1 of 3.4.1 and clause 4).

4. Test preparation

4.1 Test portions. It is preferable to weigh out the specified quantity of the test sample given in table 2 separately for each test rather than to work from a large quantity of stock solution.

Table 2. Test quantities

Clause	Test	Mass of gelatine	Volume of water
		g	ml
6	Jelly strength comparison	2 to 10	50
7	Bloom strength	7.5*	105
8	Viscosity	7.5*	105
9	Melting point	7.5*	105
10	Setting point	10	90
12	Keeping quality	7.5*	105
13	pH	1 to produce	100 of solution

NOTE. If the Bloom strength of a gelatine is less than 100 g, the test may be repeated using 15 g of gelatine to 105 ml of water.

4.2 Method of dissolving. Weigh the powdered sample into the container specified in the test method, containing the requisite amount of cold water complying with BS 3978, stirring with a stainless steel or glass rod. Cover the container and allow to soak for 3 h at a temperature not above 22 °C. Then heat the container in a water bath for about 15 min, ensuring that the gelatine is completely dissolved. This may be ascertained by lifting up the container and inspecting through the bottom. When the sample has all dissolved, remove the container from the water bath. The final temperature of the solution when in the bath should reach, but not exceed, 60 °C. This is ensured by adjustment of the bath temperature, which should on no account be above 70 °C. During this heating period gently stir the solution with a rod.

*If the concentration is too low, use 15 g of gelatine and 105 ml of water and note this in the test report.

BS 757 : 1975

5. Method for the determination of moisture content

5.1 Principle. A weighed sample of the gelatine is maintained at 105 °C for 18 h and then is reweighed.

5.2 Definition. Moisture content is defined as the percentage loss in mass of the sample when a thin, evenly-distributed film of the gelatine is dried at 105 °C for 18 ± 1 h in the manner described below. See appendix A.

5.3 Apparatus. The following are required.

- (a) Stainless steel dish, flat bottomed, of 70 mm diameter and 15 mm high, weighing about 30 g, fitted with an aluminium cover for use when cooling and weighing;
- (b) Oven, complying with BS 2648, capable of being controlled at 105 °C.

5.4 Procedure. Weigh into the tared dish, to the nearest 0.01 g, about 1 g of the unground sample (see note of 4.1).

NOTE. Gelatine, especially in the finely divided state, rapidly alters in moisture content according to the relative humidity of the air to which it is exposed. The operations of crushing and sampling described in clause 3 should be carried out without delay and in surroundings which avoid excessive humidity or dryness. There should be no delay in transferring the sample to an air-tight container before weighing out.

Add 10 ml of water complying with BS 3978, and allow the gelatine to soak. Place the dish (5.3(a)) on a water bath until the gelatine is dissolved and a homogeneous solution obtained, and leave until most of the water has evaporated, giving a uniformly thin film.

Transfer the dish to the oven (5.3(b)), controlled at 105 °C, and allow to remain for 18 ± 1 h, during which time the oven door shall not be opened. Cool in a desiccator and weigh.

Take every precaution to prevent access of moisture to the dried film both before and during weighing.

5.5 Expression of results. Moisture content, expressed as a percentage by mass, is given by the formula

$$\frac{M_0 - M_1}{M_0} \times 100$$

where

M_0 is the mass of original sample (g)

M_1 is the mass of the sample after drying (g)

5.6 Test report. Report the result obtained and the method used, i.e. that given in BS 757, and mention any circumstances which may have affected the result.

6. Method for the comparison of jelly strength

6.1 Introduction. If there is a sample agreed between purchaser and vendor, it is necessary to compare the jelly strength of the sample for test with that of the agreed sample under the same conditions of test.

Any suitable instrument* may be used for this purpose, provided that there is previous agreement on procedure, concentration and temperature. See also appendix A.

6.2 Definition. The jelly strength is a measure in arbitrary units of the rigidity modulus of a gel prepared and matured under standard conditions.

6.3 Principle. A solution of gelatine is prepared and allowed to gel for 16 h before comparison of jelly strength, in a test instrument, with the agreed sample.

6.4 Procedure. Weigh 2 g to 10 g, according to grade and use (see note below), of the powdered test portion (see clause 4) into a 150 ml beaker, and add 50 ml of cold water, complying with BS 3978, from a pipette. Dissolve the sample in the manner specified in 4.2.

Pour the gelatine solution immediately into a suitable cylindrical container and, after 2 min, put on the lid. Leave the container for 16 h in a bath of running cold water, or preferably in a thermostatic bath maintained at an agreed temperature, then remove the lid and compare the jelly with that of the agreed sample, treated in the same way.

NOTE. The above method of determining jelly strength by reference to an agreed standard is of very general application, because the conditions of concentration and temperature may be drawn up to suit the proposed use of the gelatine. For example, if the gelatine is to be used for the preparation of certain confectionery products, 2 g of the agreed sample and of the sample under test may be taken and the jelly matured at 10 °C.

Provided that the procedure, concentration and temperature are agreed, an instrument can be used to give a numerical reading in cases where there is no reference sample.

*In addition to the equipment employed in clause 7 the following may be used for comparative testing:

- (a) Boucher Jelly Tester (Kuprowski, *Analyst* 1951, 76, 732).
- (b) The Gelatine and Glue Research Association Jelly Tester, (Saunders & Ward, *Proc. 2nd Rheological Congress* 1954, Butterworth Ltd. London) Supplied by C. Farley & Sons Ltd., Ponders End, Middlesex.
- (c) Food Industry Research Association Jelly Tester (Campbell *J.S.C.I.* 1938, 57, 413) Supplied by H. A. Cardon & Co., Croydon, Surrey.

6.5 Expression of results. Express the jelly strength as less than, equal to or greater than that of the agreed sample, as the case may be.

6.6 Test report. Report the result obtained and the method used, i.e. that given in BS 757, and mention any circumstances which may have affected the result.

7. Methods for the measurement of jelly strength

7.1 Introduction. If there is no agreed sample for comparison, it becomes necessary to measure the jelly strength of the sample for test under arbitrary conditions. It has been decided to adopt the Bloom jelly strength* as the British Standard. This measures the mass in grams necessary to produce the force to give a 4 mm depression in a jelly of 6.67 % concentration by mass, matured at 10 °C, using a plunger of 12.7 mm diameter (see figure 3). See also appendix A.

7.2 Principle. A 6.67 % solution of the sample is prepared in a wide-mouthed test bottle. The bottle is kept for 16 h to 18 h in a thermostatic bath at 10 °C and the resulting jelly is tested in a gelometer, either of the Bloom type or the Boucher electronic type.

7.3 Apparatus. The following are required.

- (a) Bottle of internal diameter 59 ± 1 mm, approximately 85 mm high, having a capacity of approximately 155 ml†. A stopper of rubber or cork approximately 43 mm in diameter should fit snugly into the neck of the bottle and should be pierced centrally with an air vent about 0.5 mm in diameter.
- (b) Thermostatic bath fitted with thermometer, totally enclosed, and capable of being controlled at 10.0 ± 0.1 °C. (Thermometer No. A20C/100 complying with BS 593 : 1974 and calibrated according to appendix C of that standard is suitable).
- (c) Bloom type gelometer (see figures 1 and 2) adjusted to give a 4 mm depression and to deliver lead shot at the rate of 200 g per 5 s when the hopper contains 800 g of polished No. 12 chilled shot. Shot passing a test sieve (complying with BS 410) of nominal aperture 1.20 mm and which is retained on a sieve of nominal aperture 1.00 mm is satisfactory.
- (d) Alternatively the Boucher electronic jelly tester‡ (see figure 5) which determines the gel strength automatically by depressing a standard plunger (see figure 3) 4 mm at a constant rate of loading of 29 counts per second.

7.4 Dissolving the sample. Dissolve 7.5 g of the sample in 105 ml of water in the wide neck bottle (7.3(a)) as described in clause 4, closing the bottle with the stopper instead of a watch glass. To prevent caking, swirl the bottle vigorously to wet the gelatine completely, and occasionally swirl during dissolving. At this stage, take care not to build up more froth, by excessive agitation, than will collapse before inserting the bottle in the thermostatic bath. Particular care is necessary to see that the gelatine is all dissolved and that the solution is homogenous. Striations in the solution after swirling the bottle indicate the need for more prolonged agitation. The striations are best seen in a bright light. Place a finger over the perforation in the stopper and invert the bottle several times to mix in the water that has condensed on the walls of the bottle and the underside of the stopper.

7.5 Chilling the solution. To prevent cracking, allow the bottle to cool for 15 min at room temperature, and then place in the thermostatic bath (7.3(b)), controlled at 10.0 ± 0.1 °C, for not less than 16 h and not more than 18 h. Ensure that the platform of the thermostatic bath is horizontal and that the bottle stands evenly on it.

7.6 Procedure for determining jelly strength

7.6.1 Procedure for determining jelly strength by Bloom type gelometer. (See figures 1 and 2.) Stand the gelometer perfectly level on a rigid support. Adjust the levelling screws R until the bottle platform O is horizontal. Check by using a spirit level on the platform in two directions at right angles. The spindle B should not then normally be rubbing against the guides K; any contact which does occur should be insufficient to restrict free movement. Do not change position of the guides K but keep them directly under the point of suspension of spring G.

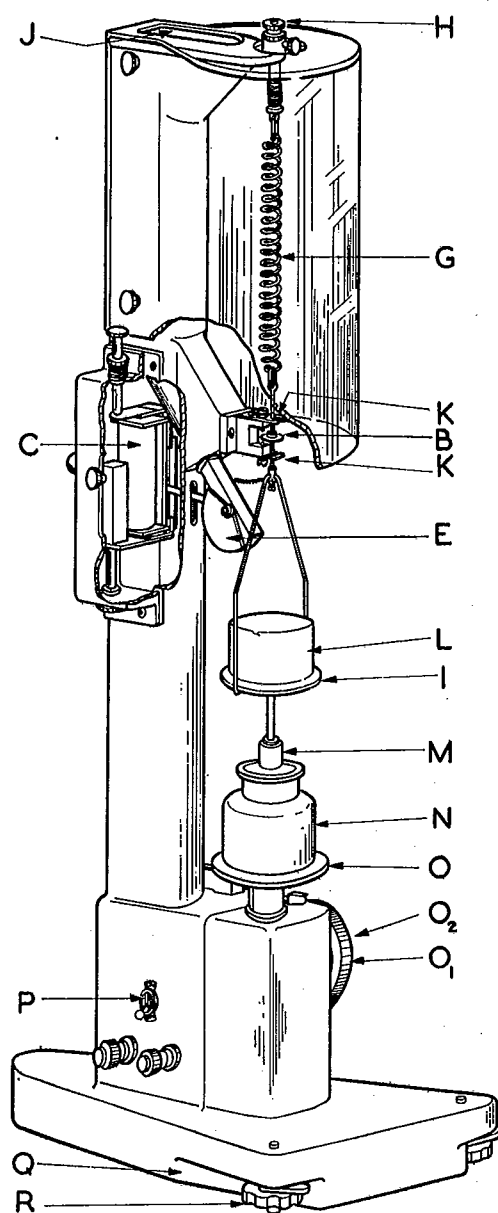
Ensure that the spring is of such stiffness that between 2 g and 3 g placed on the pan I will bring the disc B from top contact A₁ to lower contact A₂.

*Based on the method given in *Industrial and Engineering Chemistry*, Analytical Edition, 1930, 2, 348.

†Suitable bottles are supplied by C. Stevens & Sons Ltd., 287-289 Goswell Road, London, E.C.1.

‡Boucher electronic jelly tester. Supplied by C. Stevens & Sons Ltd., 287-289 Goswell Road, London, E.C.1.

BS 757 : 1975



- A₁ Upper contact
- A₂ Lower contact
- B Silver disc
- B₁ Disc spindle
- C Relay
- D Relay catch
- E Cut-off
- F Cut-off arm
- G Spring
- H Thumbscrew nut
- I Pan
- J Shot hopper
- K Guides
- L Shot receiver
- M Plunger
- N Test bottle
- O Platform
- O₁ Rack and pinion
- O₂ Fine adjustment
- P Switch
- Q Bush
- R Levelling screw
- S Shot rate adjustment
- T Locknut

Figure 1. Bloom type gelometer

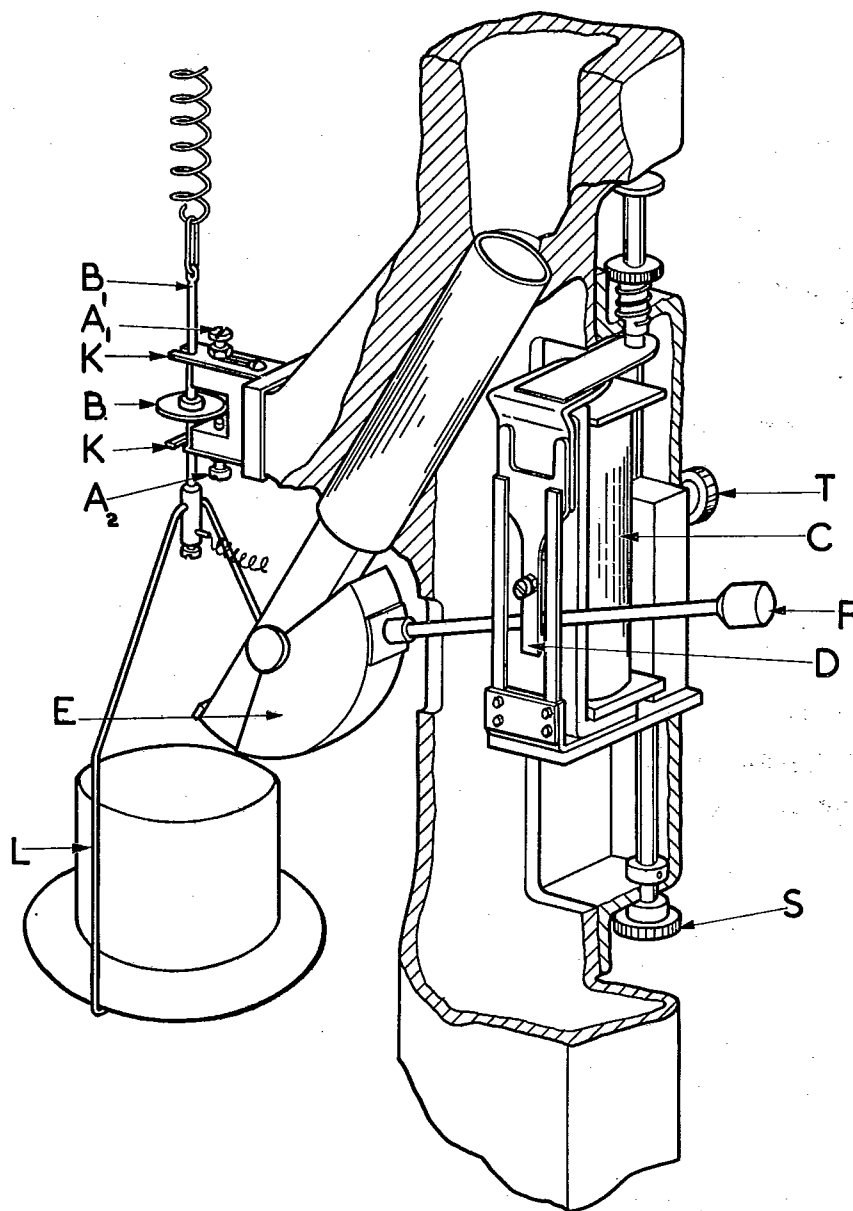


Figure 2. Bloom type gelometer: detail of relay and contact system