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WASC 899

Conditio Acceptance

Tests

SC and Ingredients

WASC 899

ACCEPTANCE TESTS OF CORDITE, S.C.

AND ITS INGREDIENTS

SCHEDULE OF METHODS TO BE EMPLOYED

C.I.N.C.
21 NOV 1935
ADMIRALTY

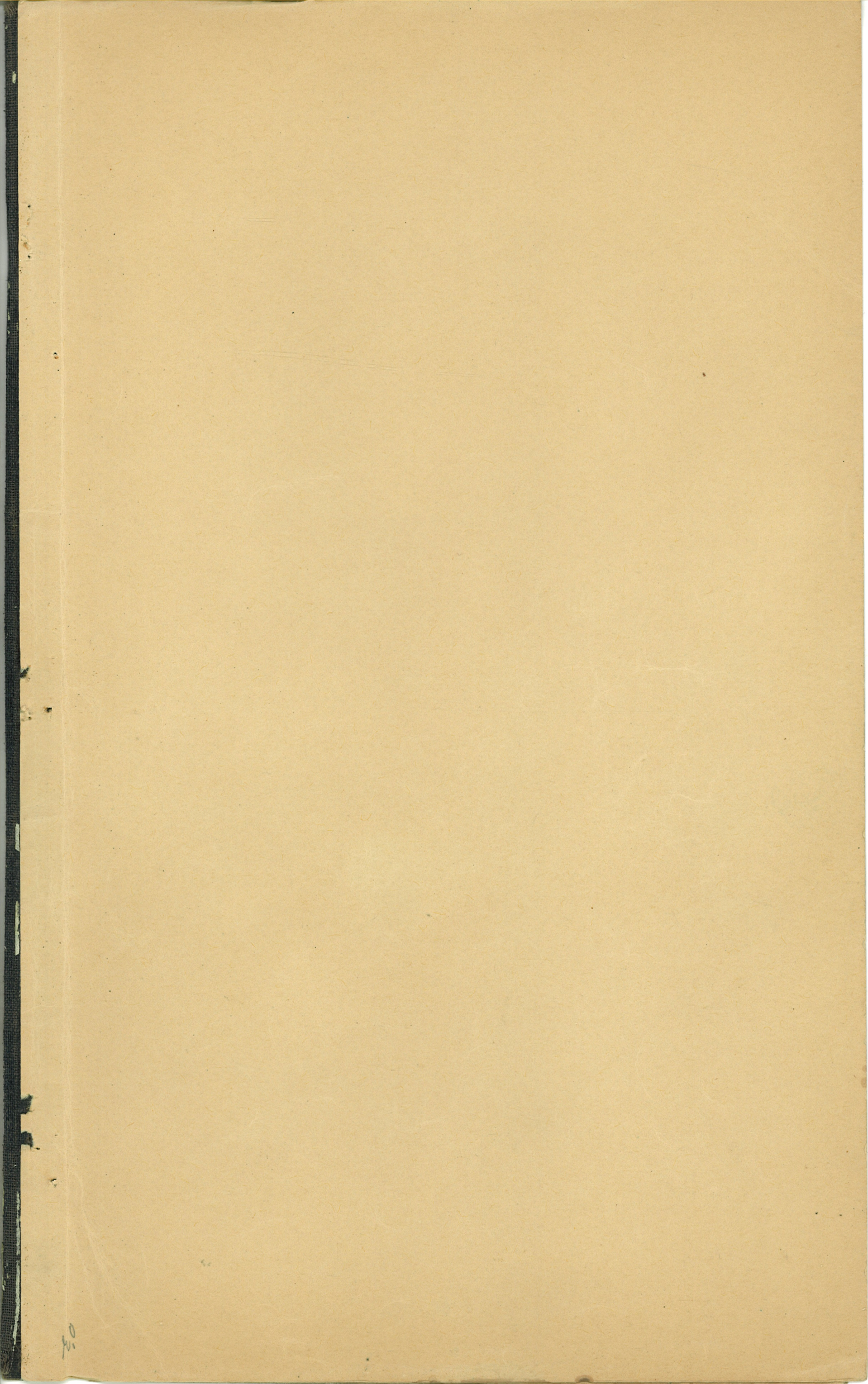
Amended in accordance
with - Circulars Amendment No 777.

R.D.

S. 569/42.
C. 1868/42

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S. W. M.



03



CORDITE, S.C. (Specification A.99):

Heat Test.....	1
Water Extract Test.....	1
Colour Test.....	1
Estimation of Volatile Matter.....	1
" " Nitrocellulose.....	2
" " Carbamite.....	2
" " Total Iron.....	2

Nitroglycerine (Specification A.99 Appendix A) -

Estimation of Volatile Matter.....	3
" " Alkalinity.....	3
Heat Test.....	3

Nitrocellulose (Specification A.99 Appendix B), --

Heat Test.....	4
Estimation of Magnetic Iron.....	7
" " Mineral Matter.....	9
" " Alkalinity.....	10
" " Sulphates.....	10
" " Organic Matter insoluble in Acetone.....	10
" " N.C. soluble in Ether-Alcohol.....	10
Stability Test at 132°C.....	11
Estimation of Nitrogen.....	12
Settling Test.....	13

Precipitated Chalk (Specification A.99 Appendix D) -

Settling Test.....	14
--------------------	----

Nitric Acid (Specification A.99 Appendix E) -

Test for Nitrous Acid.....	15
----------------------------	----

GLYCERINE (Specification A.95):

Estimation of Nitrogen.....	16
Test for Sugars etc.....	16
Estimation of Saponification Equivalent.....	16

CARBAMITE (Specification A.154):

Determination of Setting Point.....	17
-------------------------------------	----

PAPER WOOD CELLULOSE (Specification A.165):

Estimation of Lignin.....	18
" " Copper Value.....	18

CANDELILLA WAX (Specification A.97):

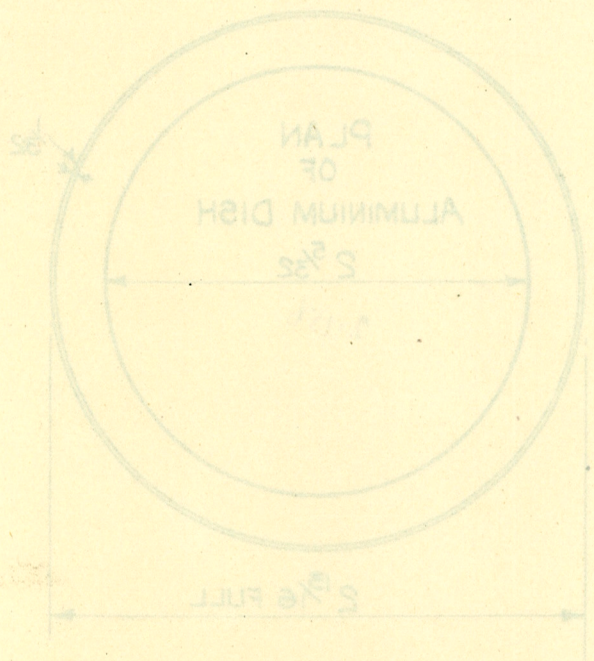
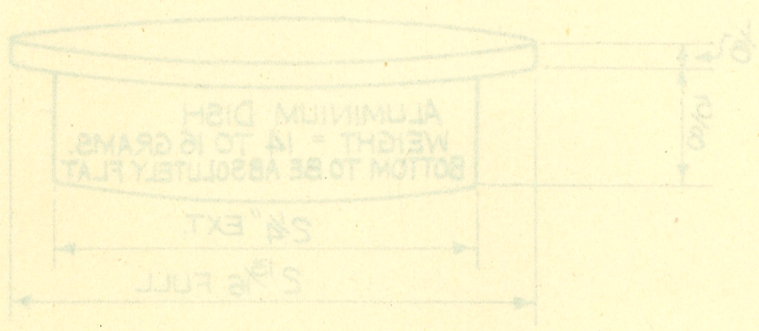
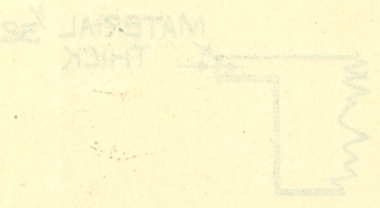
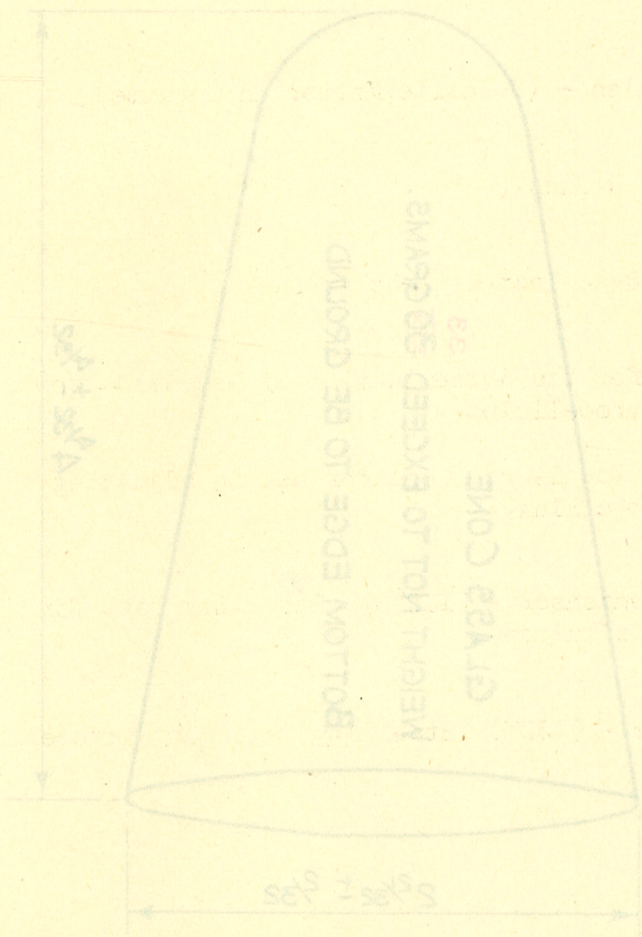
Determination of Setting Point.....	20
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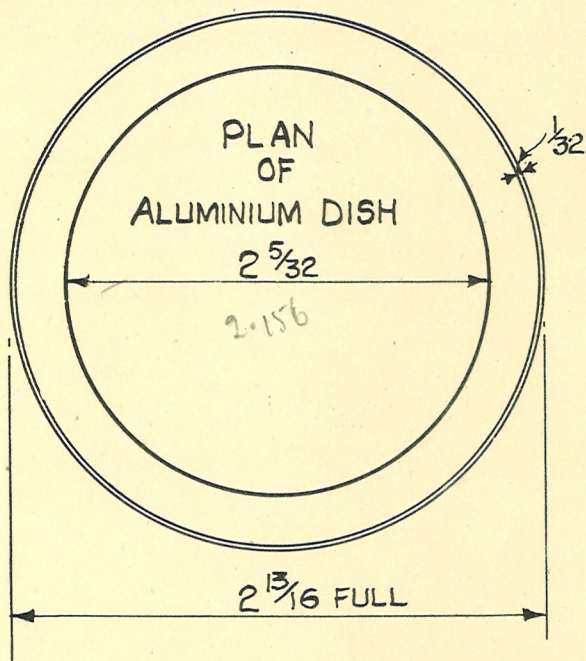
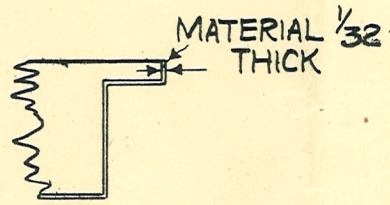
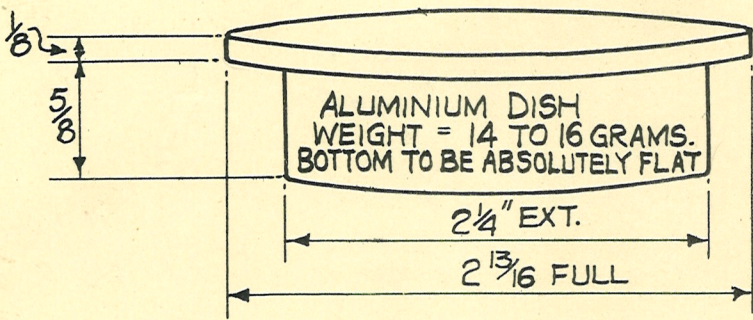
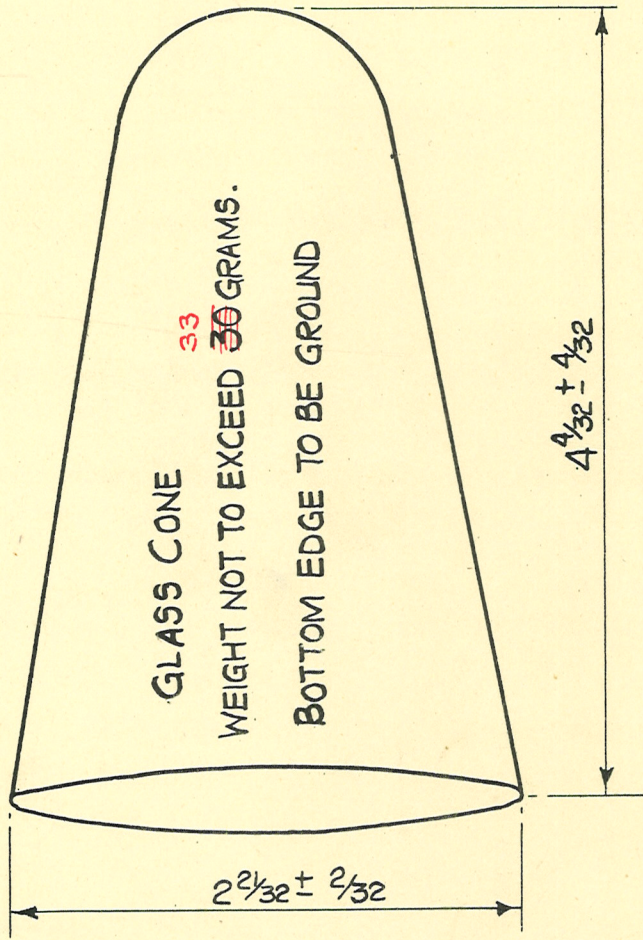
APPENDICES:

Specification for Ethyl Ether.....	21
" " Ethyl Alcohol.....	22
" " Acetone.....	23

The drawings referred to in these Methods of Test are as follows:-

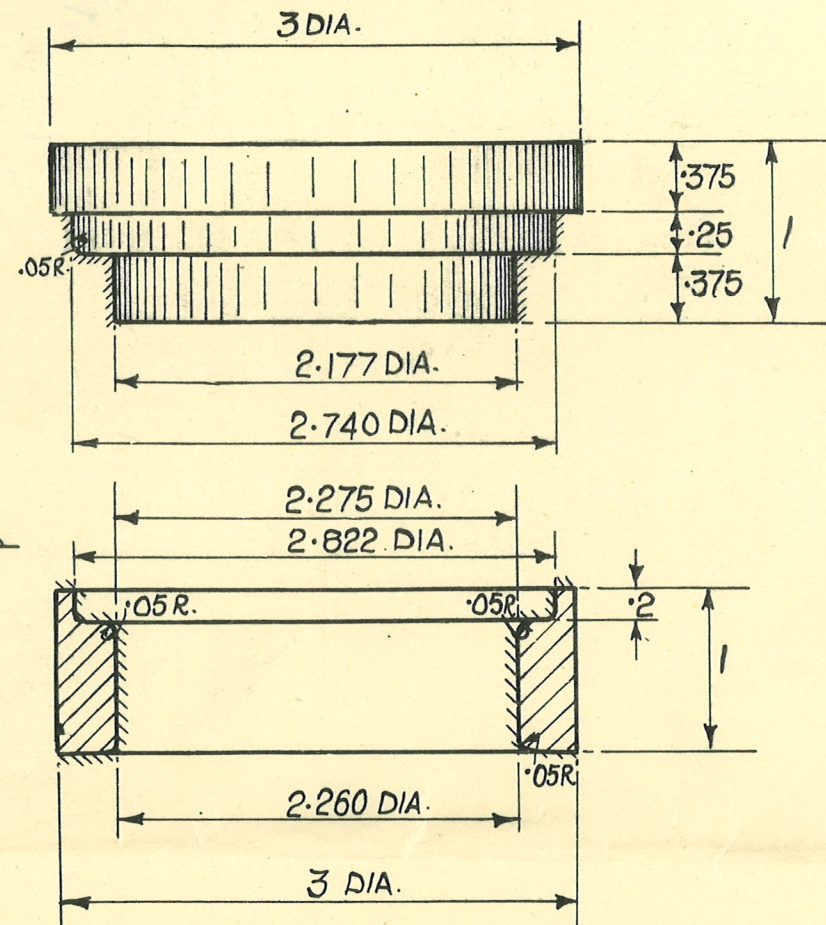
	Facing page
No. 1. Cone and dish - (Volatile Matter in Cordite).....	1
No. 2. Former for Dishes. (" " " ").....	1
No. 3. Apparatus Heat Test. Nitrocellulose.....	5
No. 4 & 4A. Apparatus for the determination of Magnetic Iron in Nitrocellulose.....	7
No. 5. Diagram of the Search Coil for use in adjusting the Magnetic Flux.....	9
No. 6. Bath and Condenser - (132°C. Stability Test for Nitrocellulose).....	11
No. 7. Glass Tubes - (132°C Stability Test for Nitrocellulose).....	11

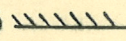




FORMER FOR DISHES

FOR FINISHING THE DISHES SHOWN ON DRG. No 1



BOTH PIECES HARDENED STEEL
& SURFACES MARKED THUS  TO BE GROUND.

C O R D I T E, S.C.

(Specification A.99)

HEAT TEST: See Pamphlet, "Schedule of Tests".

WATER EXTRACT TEST: " " " " "

COLOUR TEST: " " " " "

ANALYSIS:

(i) Estimation of Volatile Matter

Preparation of Sample

A representative portion of the sample to be tested is taken, 1/8 inch cut off each end of the sticks and discarded. The cordite is then cut up into small pieces about 1/8 inch long and ground in the cordite mill. The first grindings from the mill - 1 to 2 grams - are allowed to fall on to an aluminium tray and are discarded. The nest of sieves (Apparatus, Heat Test, Cordite) is then placed close under the outlet from the mill, so that the rim of the top sieve just overlaps the base of the cone-sheath. The ground cordite is collected on the top sieve, and when grinding is completed, sieves are removed and the cordite sieved. Time of sieving 2 minutes. The portion retained by the bottom sieve is placed in a small well-stoppered clean dry glass receptacle for use in the test.

Preparation of Cone and Dish (Drawing No. 1)

The edge of the cone should be free from flaws, giving complete contact when placed on a flat surface. To ensure this it may be necessary to grind down the edge with carborundum paste. The aluminium dish should be free from any pitting, bright and well polished, and should be corrected to the required shape by means of a former (Drawing No.2.) The bottom of the dish should be quite flat. During cleaning the dish may become slightly distorted; it is therefore necessary before commencing another test to reform the dish in the "former". Under these conditions the cone and dish will be found to fit evenly, and the dish will lie flat on the bath. ~~The cone and dish must not together weigh more than 45 grams.~~

Method of Test

The cone and dish are heated on the boiling water hot plate for 1 hour, removed, allowed to cool 30 minutes in a H₂SO₄ desiccator. It is then weighed and 5 grams of cordite introduced into the aluminium dish, direct from the receptacle. The cone and dish + cordite is then accurately weighed and the apparatus transferred to the hot plate. During heating care should be taken that the room is well ventilated but direct draught on the cone is to be avoided. After 2 hours' heating the dish and cone are removed, cooled 30 minutes in a H₂SO₄ desiccator and weighed. Occasionally it has been found that the dried cordite gains rapidly in weight during weighing; it is essential therefore that weighing should be completed in 1-1/2 minutes after removal of dish and cone from desiccator. "V.M" is reported as the percentage loss in weight of the cordite during the heating.

(ii) Estimation of Nitrocellulose

2 grams of the ground cordite which passes the bottom sieve during the preparation of the "V.M." sample are weighed into a previously tared alundum thimble (size 19 m.m. outside diameter, 90 m.m. in height, porosity R.A. 98), which has been ignited in a muffle, and allowed to cool in a desiccator. The cordite in the thimble is extracted (without previous soaking) in a soxhlet extractor for 5 hours with ether of the specified quality, after which the thimble is allowed to drain, placed for about 30 minutes in a beaker on top of a boiling water bath to remove excess ether. It is then steamed for 1 hour and dried for 4 hours at $105 \pm 1^{\circ}\text{C}$., cooled in a desiccator and weighed.

The drying is continued for further periods of 2 hours, as necessary, until successive weighings do not differ by more than 1 m.g.

Correction must be made as necessary for the solubility of the N.C. in ether. This is found to amount, in general, to about 0.4 per cent calc. on the N.C.

(wood N.C.)

(iii) Estimation of Carbamate

3 grams of cordite ground "V.M." size are placed in a 500 c.c. flask and 50 c.c. of 20 per cent Caustic Soda added. The flask is closed with a rubber stopper through which passes a steam inlet tube extending below the surface of the liquid in the flask, and a shorter outlet tube. The outlet-tube is connected to a well cooled condenser to which is attached an adapter. This adapter dips into about 20 c.c. water contained in a 700 c.c. separating funnel. The inlet tube is attached to a steam supply. The contents of the flask are gradually raised to a temperature of 105°C .- 110°C . by heating in a glycerine bath or heating direct with an Argand burner; during this stage only sufficient head of steam is maintained to prevent a suck back into the steam generator; 500 c.c. distillate is collected. The distillation is then stopped and condenser and adapter washed free from any solidified carbamate adhering to them by means of about 30 c.c. of warm alcohol (95 per cent) the washings being collected in a 350 c.c. conical flask. The aqueous distillate is then acidified with a few drops of hydrochloric acid, and extracted with three successive quantities of petroleum ether (B.Pt. $40-50^{\circ}\text{C}$.) 200 c.c. in all. The petroleum ether extracts are added to the alcohol washings.

The mixture of petroleum ether and alcohol is then distilled until practically all the petroleum ether is removed. During distillation it will not be found necessary to exceed a temperature of 55°C . Any traces of petroleum ether left behind after distillation are removed by a short gentle blowing. The Carbamate is thus left in solution in about 10 c.c. alcohol. To the cooled solution of carbamate in alcohol 0.15 c.c. bromine is added drop by drop, the liquid being agitated after each addition of bromine. The solution which should be orange red in colour, is allowed to stand for 15 minutes, and SO_2 is then passed over the liquid to discharge the colour of the bromine. Water is then added very gradually with continual agitation so that the product is precipitated as fine white crystals. After about 50 c.c. water has been added, further additions of water to give a total amount of 250 c.c. can be added more quickly. The precipitate which consists of dibromdiethyldiphenylurea is allowed to stand for about 1 hour, or until solution is clear, and then filtered through a Jena glass filtering crucible washed with water and dried for 1 hour at 100°C . The percentage carbamate is calculated on the V.M. free sample using the following factor:-

Carbamite = $0.629 \times$ dibromdiethyldiphenylurea.

NITROGLYCERINE
(Specification A.99, Appendix A)

Moisture

About 5 grammes of the nitro-glycerine, weighed accurately in a flat-bottomed, cylindrical, aluminium dish, 1 inch X 1 inch are placed in a desiccator over dry calcium chloride and dried to constant weight.

Alkalinity

100 cc. of distilled water are coloured with methyl orange and just acidified with N/20 H₂SO₄. The liquid is divided into two equal portions to one of which is added approximately 20 grams of the nitroglycerine with gentle hand shaking. The colour of the decanted water is matched to the other 50 cc. by titration with N/20 H₂SO₄.

Heat Test

The test is carried out with the apparatus and materials described on page 4.

Three filter papers made from a pure paper, free from chemical impurities, are folded as one and placed in a 1½ inch funnel. The funnel is placed with its end free in a heat test tube, supported in a test tube stand. The sample of nitroglycerine is well shaken up and poured into the filter until the latter is full to within 1/8 inch of the top. When 2 cc. of clear nitro-glycerine have collected in the test tube, the funnel is removed. Should the nitro-glycerine, filtered as above, show any signs of cloudiness, the filtration is continued until all the nitro-glycerine in the funnel has passed through the filter. It is then filtered again through a single paper and this is repeated, if necessary, until at least 2 cc. of clear nitro-glycerine have been obtained.

2 cc. of the filtered nitro-glycerine are used for the test, which is carried out in duplicate as described in the method for Heat Test Cordite.

NITROCELLULOSE
(Specification A.99, Appendix B.)

Nitrocellulose taken from the blending tank before the addition of Chalk is tested for Heat Test, Mineral Matter, N.C. Soluble in Ether-Alcohol, Nitrogen and Settling Test.

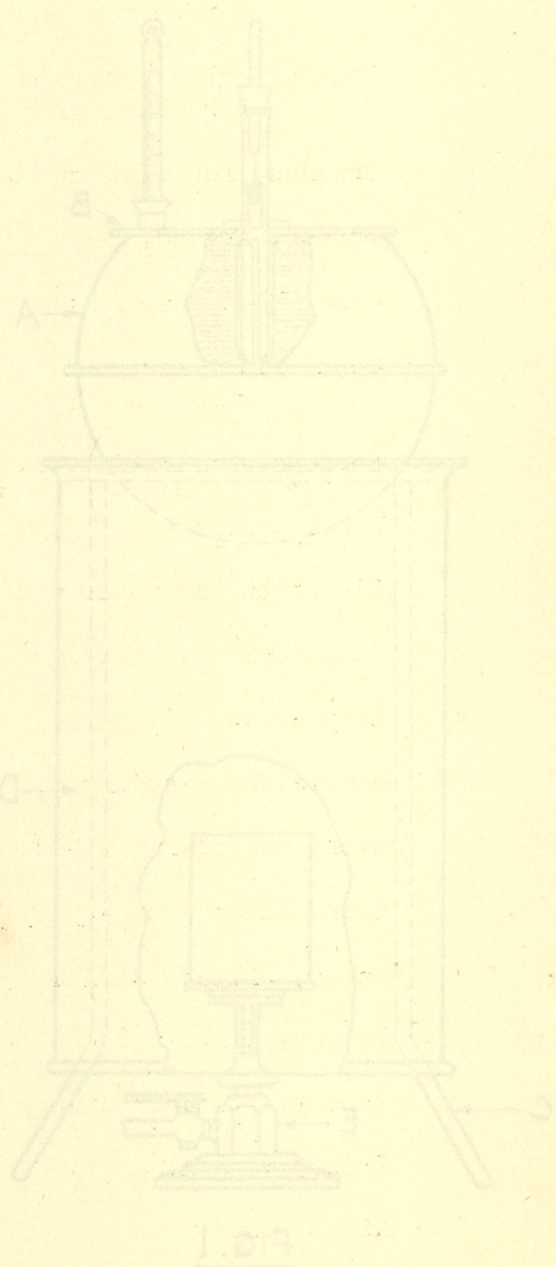
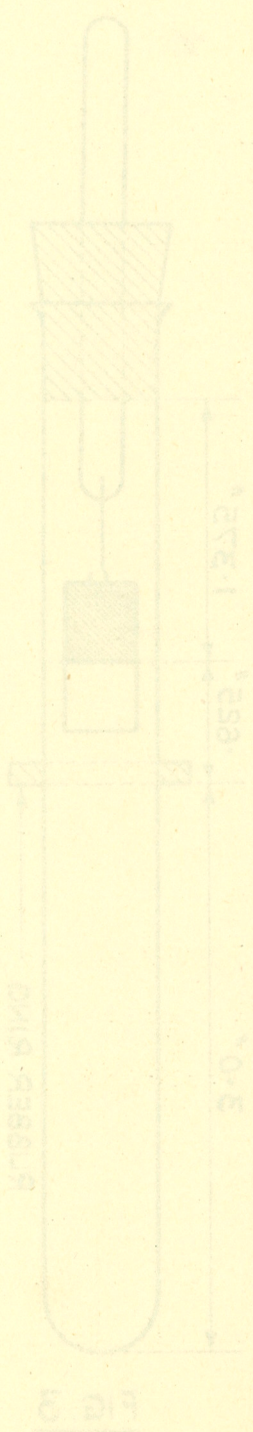
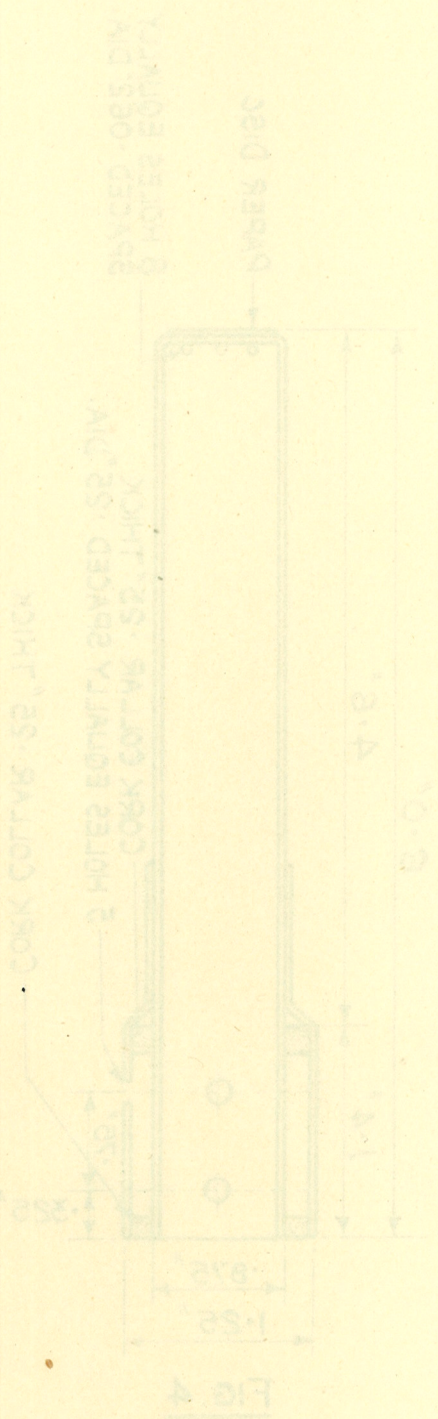
Nitrocellulose taken from the blending tank after the addition of Chalk is tested for Mineral Matter, Alkalinity, Sulphates, Organic Matter Insoluble in Acetone, Magnetic Iron and 132°C. Stability Test.

Determination of Heat Test:

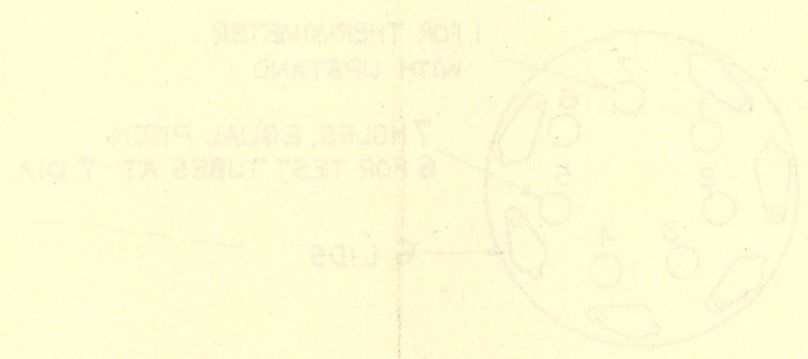
Apparatus and Materials required

Balance	1
Bath	1
Bottle, Dropping, for glycerine and water mixture.	1
Bottle ditto spare	1
Bottles, glass stoppered, brown glass, 6 oz. for test papers.	2
Bottles, glass stoppered, brown glass, 4 oz. for standard tint paper.	1
Brushes, for cleaning test tubes	4
+ Burner, Argand, gas	1
Caps, black paper, for test tubes	6
Cork, flat, for resting Heat Test paper on when piercing.	1
Forceps, for lifting Heat Test papers.	1
Funnel, Aluminium	1
Glycerine and water mixture	4 ozs.
* Lamp, Spirit, Berzelius	1
* Lamp Wicks	12
Needle, for piercing Heat Test papers.	1
Oven, for drying tubes, bottles and trays	1
Oven, Water, for drying Nitro-cellulose	1
Paper, Filter, 2' X 2'	A supply
Papers, Heat Test	A supply
Papers, standard tint	A supply
Press, with Aluminium lined plates	1
Rings, Test Tube	A supply

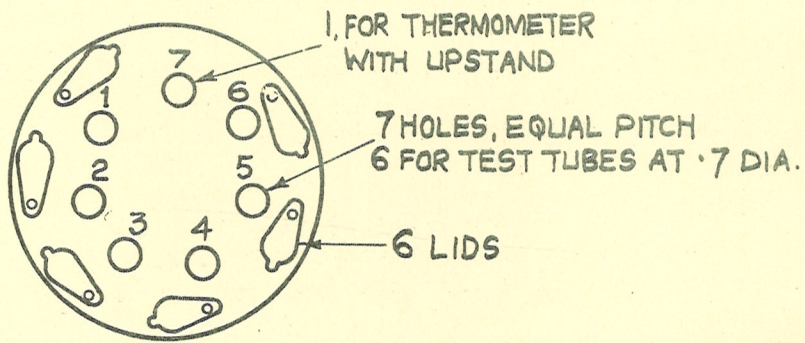
APPARATUS HEAT TEST NITROCELLULOSE



PLAN OF LID FIG. 2



APPARATUS, HEAT TEST, NITROCELLULOSE.



PLAN OF LID. FIG. 2.

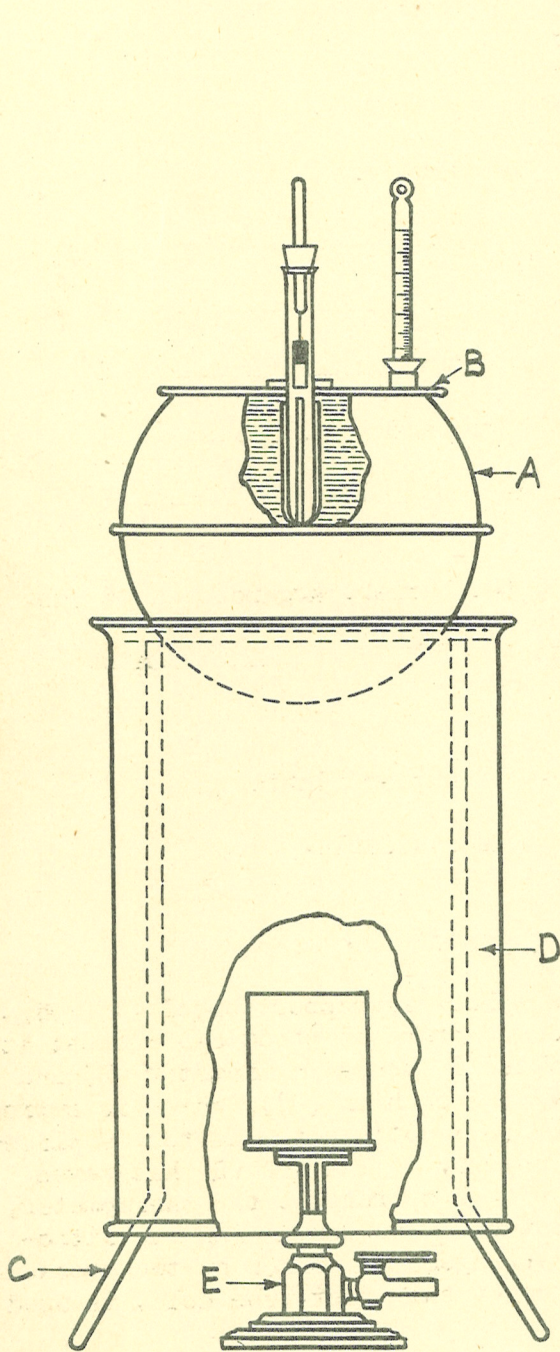


FIG. 1

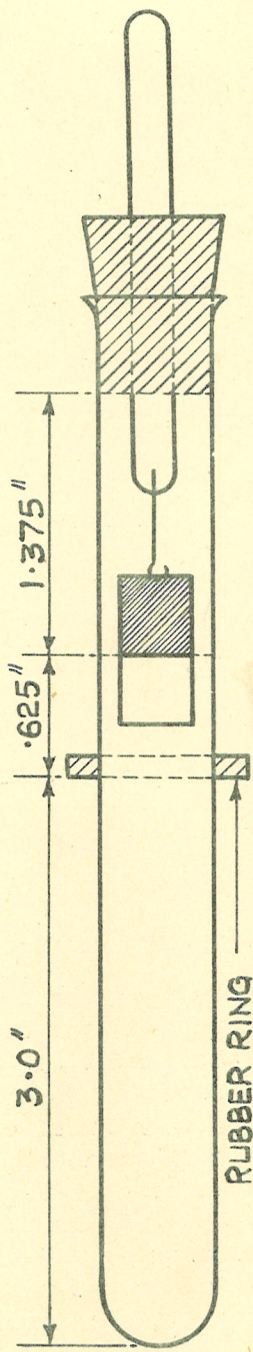


FIG. 3

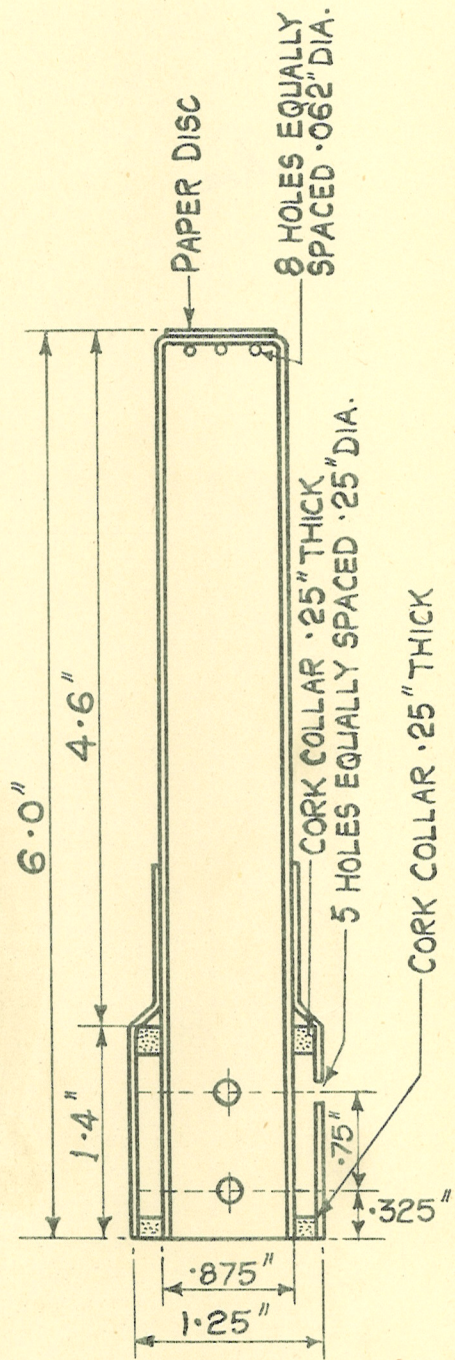


FIG. 4

	Rod, glass, flat-headed.....	1
	Rods, glass, platinum wire hook.....	6
	Scoops, for use in weighing nitro- cellulose.....	2
	Screen, burner, for water bath.....	1
	Spatula, bone.....	1
*	Spirit, Methylated, industrial (bottle of).....	1
	Stands, Test Tube, with holes, to hold test tubes when preparing nitro- cellulose for testing.....	2
	Stands, Test Tube, with pegs, for draining test tubes after cleaning.....	2
	Stand, tripod, water bath.....	1
	Stoppers, rubber, for glass rods.....	6
	Stoppers " " thermometers.....	6
	Test Tubes.....	12
	Thermometers, Heat Test.....	2
	Thermometers, for oven, water.....	2
	Thermometers, chemical standard.....	1
	Trays, Aluminium, for drying nitro- cellulose.....	6
+	Tubing, rubber $\frac{1}{4}$ inch, for burner, Argand.....	6 yds.
	Weights and forceps in box.....	A set
*	Only required where gas is not available.	
+	" " " " is available.	

Description of Apparatus. (Drawing No.3)

The water bath consists of a spherical copper vessel (A) (Fig. I) of about 8 inches diameter with an aperture of about 5.5 inches; it has a loose lid of sheet copper about 6 inches in diameter (B) and rests on a tripod stand about 15.5 inches high (C); which is surrounded with a screen of thin sheet copper (D); within the latter is placed an Argand burner (E) with copper chimney. The lid (B) has seven holes arranged as seen in Fig. 2, No. 7 to receive the thermometer, and Nos. 1, 2, 3, 4, 5 and 6, the test tubes containing the nitro-cellulose to be tested. A cage arrangement, formed by two crossed copper wires fixed to the underside of each test tube hole, is used to hold the test tubes in position.

No bath other than this is to be used without special authority as any change in its size and the distribution of the holes for test tubes affects the test and so renders the result unreliable.

When gas is available an Argand burner of the correct pattern only is used, and the Berzelius spirit lamp which has an arrangement for regulating the flame by raising or lowering the wick is for use where gas is not available.

Wherever electrical heating is used, the installation and maintenance of the system must be carried out by the Dockyard Authorities to ensure compliance with relevant regulations.

The test tubes (fig.3) are from $5\frac{1}{4}$ - $5\frac{1}{2}$ inches long, and of such diameter that they will hold from 20 to 22 cubic centimetres of water when filled to a height of 5 inches.

The ventilated light tight cap (Fig.4) is to shield the portion of the test tubes above the cover of the bath from the light, and also to prevent condensation on the outside of the test tube. It consists of a black paper tube, 6 inches long, closed at one end and having five ventilating holes 0.325 inch from the open end, and with smaller holes round the closed end. A strip of black paper is fixed over two cork collars covering the ventilating holes in the tube, and has five ventilating holes 0.75 inch above those in the tube. These caps will be made up locally. Absolute accuracy in making them is not essential. Rubber rings of about 1.2 inches diameter and 0.15 inch thick, with a hole 0.6 inch in diameter will be placed round the test tubes to prevent the escape of steam from the bath.

The glass rod, terminating in a platinum wire hook is for suspending the test paper, in the test tube, and is fitted into the latter by means of a perforated rubber stopper (Fig.3). The thermometer, ranging from 100° to 212°F . is fitted into the orifice in the lid of the water bath by means of a perforated cork or stopper. A minute clock or watch is also required for timing the duration of the test.

Preparation for testing

(a) The water bath is fitted up as shown in Fig. 1 and placed in the light of a window facing North or away from direct sunlight. It is filled with water to within a $\frac{1}{4}$ inch of the edge and provided with a thermometer which is fitted by means of a rubber stopper into the hole No. 7 of the lid and immersed in the water to a depth of $2\frac{3}{4}$ inches. The water is maintained at a constant temperature of $170^{\circ} \pm 0.50\text{F}$.

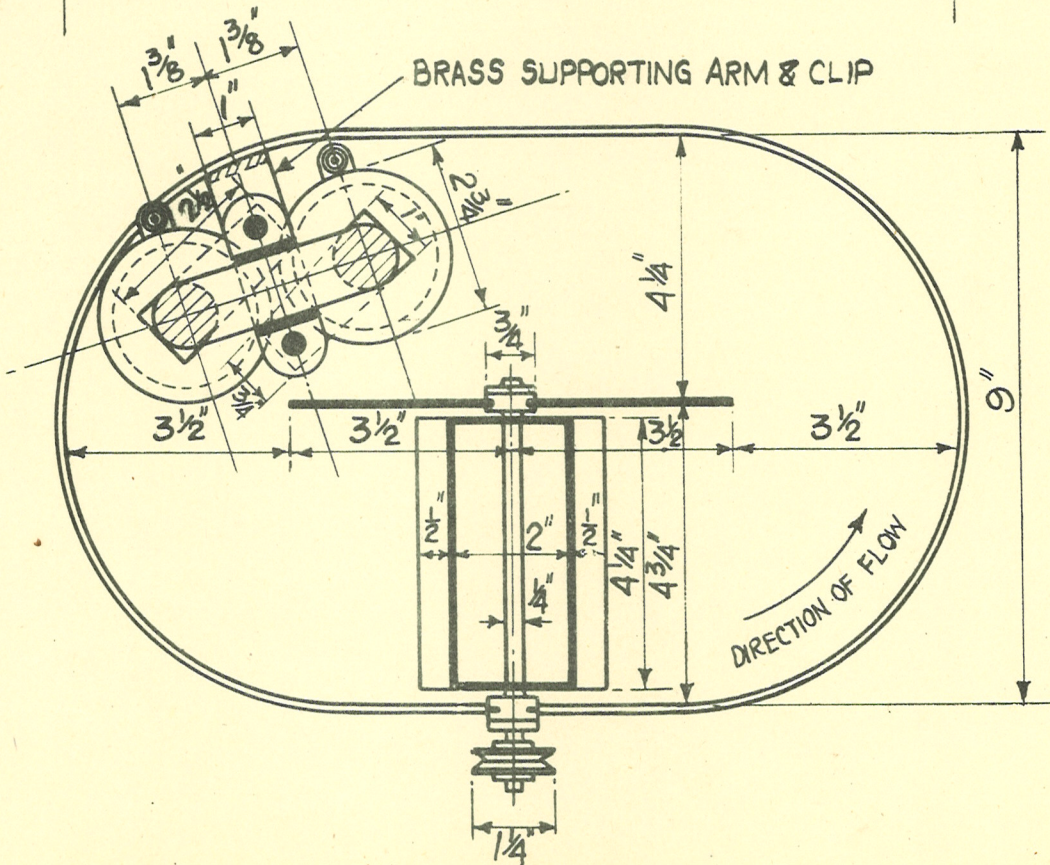
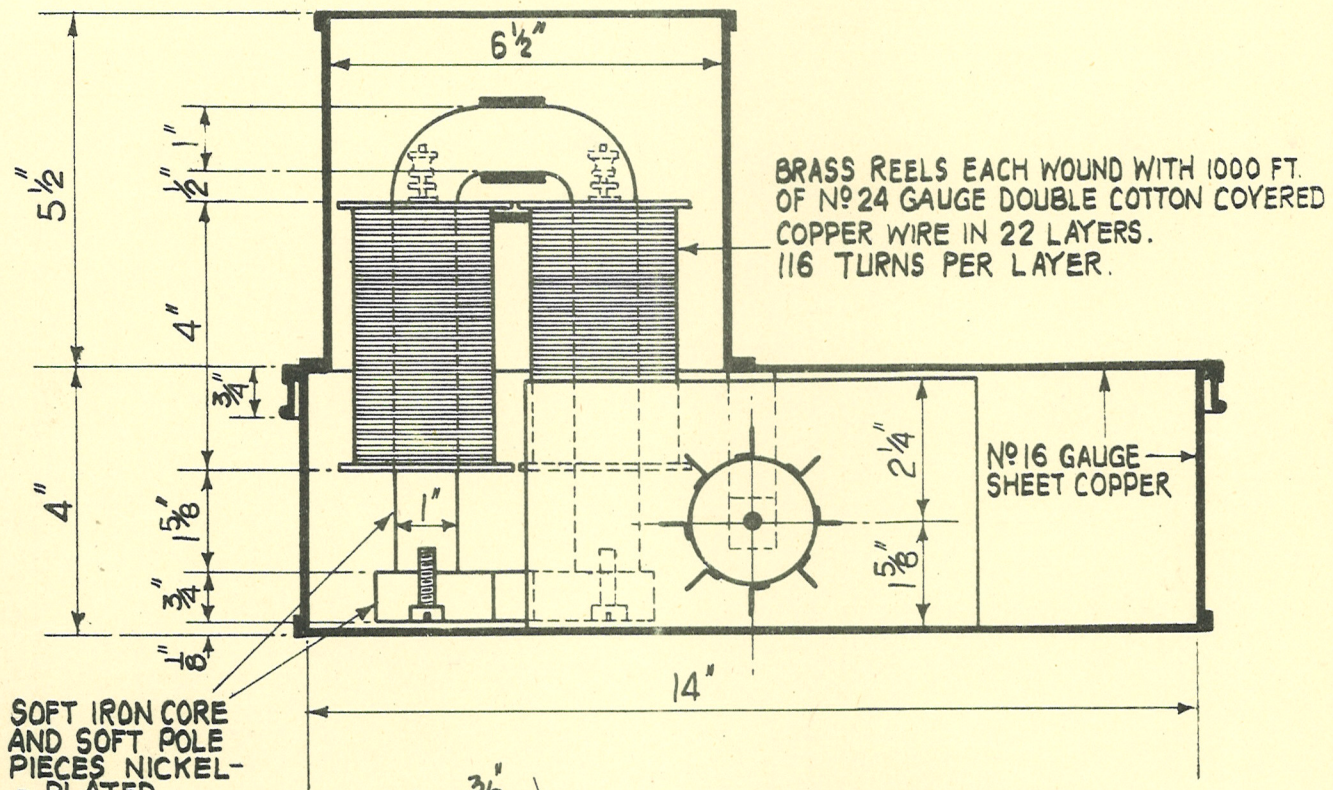
(b) About 20 grams of the sample are placed between the layers of coarse filter paper and put under a hand screw press, where it is subjected to a tolerably severe pressure for about 3 minutes. The filter paper must be a pure paper, free from chemical impurities. On removal from the press the sample is rubbed up very finely with the hands and spread out in a thin layer upon a paper tray, about 6 inches by $4\frac{1}{2}$ inches which is placed for 15 minutes in a well ventilated water oven at 120°F . The tray is then removed and exposed to the air of the room for 4 hours, the sample being, at some point within that time, rubbed upon the tray with the hand in order to reduce it to a fine and uniform state of division.

The object of this exposure is to ensure that the sample shall contain the amount of moisture which air-dried nitrocellulose will take up in the normal state of the atmosphere. It is important that the air of the room in which the sample is exposed should be quite free from acid fumes, and that the hands of the operator should be scrupulously clean.

MAGNETIC SEPARATOR FOR THE ESTIMATION OF IRON IN NITROCELLULOSE

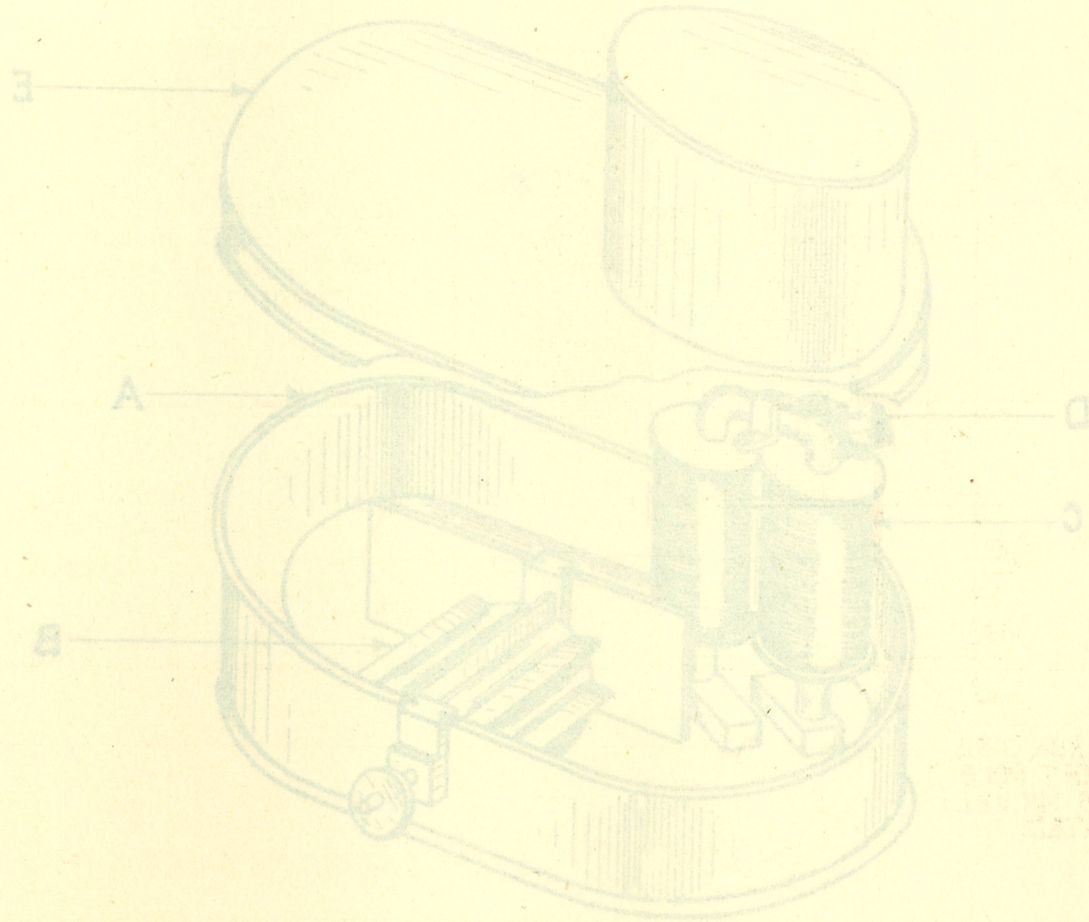
DRAWING N^o 4.

Cancelled -
addendum I.



MAGNETIC SEPARATOR FOR
THE ESTIMATION OF IRON IN
NITROCELLULOSE

DRAWING No 4A



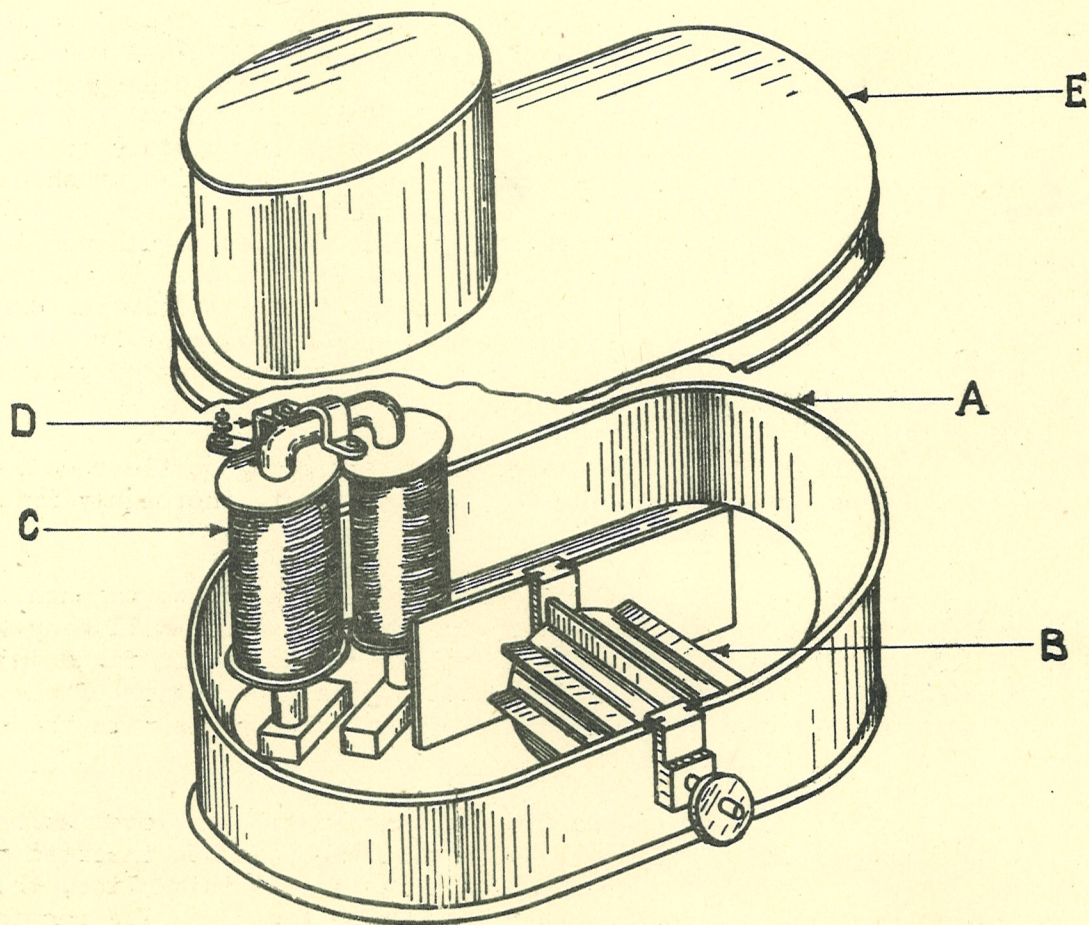
- A - BATH
- B - PADDLE WHEEL
- C - ELECTRO MAGNET WITH CORE & POLES NICKEL-PLATED
- D - SUSPENSION BRACKET
- E - COVER

NOTE - With the exception of magnet core and poles
 all metal parts of copper or brass.

MAGNETIC SEPARATOR FOR
THE ESTIMATION OF IRON IN
NITROCELLULOSE

DRAWING NO 4A.

Cancelled
assum I.



A = BATH.

B = PADDLE WHEEL.

C = ELECTRO MAGNET WITH
CORE & POLES NICKEL-PLATED.

D = SUSPENSION BRACKET.

E = COVER.

NOTE :- WITH THE EXCEPTION OF MAGNET CORE AND POLES
ALL METAL PARTS OF COPPER OR BRASS.

Application of Test

The thermometer is to be carefully watched and the temperature kept at the proper point by regulating the supply of heat; it should not be allowed to fluctuate more than $\frac{1}{2}^{\circ}$ on either side 170°F . during the test.

When this temperature is reached, 1.3 grams of the sample are weighed out on the aluminium scoop, transferred by means of the aluminium funnel, to one of the test tubes and gently pressed down until it occupies a space as nearly as possible $\frac{1}{4}$ inches in height.

The test paper is lifted from the supply by means of the forceps, placed on the flat cork and pierced with the clean needle in the centre, near the upper edge. Papers must not be touched with the fingers at any stage of the test.

The test paper is then held at the lower end by the forceps and the glycerine and water mixture as obtained by direct application from D.I.N.O., Holton Heath, is applied by means of the glass rod in the dropping bottle to the upper edge of the paper, and drawn rapidly and evenly down the surface of the paper to a distance about 0.4 cm. from the top edge. The amount of glycerine and water mixture to be applied is such that the wetted area at the completion of the test should cover completely the top portion of the paper to a distance of 0.9 to 1.2 cms. from the top edge of the paper. The wetted paper is allowed to stand for about $\frac{1}{2}$ minutes before use, by which time it will be possible to judge whether or not the wetting has been satisfactorily performed. Papers not used within two minutes of wetting are, however, to be discarded and all results obtained with under or over-wetted papers are to be disregarded.

When the glycerine and water in the dropping bottle becomes turbid it should be emptied out, the bottle rinsed out, thoroughly dried, and some fresh mixture added.

The platinum wire hook of the glass rod is passed through the hole at the top of the test paper, so that the paper will hang vertically. The stopper carrying the rod and paper is now fixed into the test tube, and the positions adjusted so that the bottom of the stopper and the testing line on the paper corresponds with the first and second lines on the tube.

A rubber ring is placed over the tube until its lower surface corresponds with the third line, and the tube is then inserted through one of the perforations of the lid as far as the third line, the rubber ring to rest on the lid of bath (see Fig.3). The portion of the tube above the cover of the bath is then shielded from the light by a ventilated cap of black paper, the cap being raised from time to time to observe the test paper.

The test is completed when the faint brown line, which after a time makes its appearance at the margin between the wet and dry portions of the test paper, equals in depth of tint the brown line drawn on the standard tint paper. The operator is to stand between the bath and the source of light, so that the test is read by reflected light.

~~Determination of Magnetic Iron~~

~~The test is to be carried out by means of apparatus constructed to the details shown in drawings Nos. 4 and 4A "Apparatus for the determination of Magnetic Iron in Nitrocellulose"; and the "Diagram of the Search Coil for use in adjusting the Magnetic Flux", (drawing No. 5.)~~

Adjustment of the Apparatus.

The pole-pieces of the electro-magnet of the Apparatus are adjusted so that the distance apart of their parallel faces is .375 inch determined with the approximate current on the solenoids. A plug gauge should be used for this adjustment.

The magnet is then lowered to its position and the current in the solenoids is adjusted so that the total flux across the gap between the pole pieces is 25,000 Maxwells, the method of testing this factor being as follows:-

A search coil of the shape and dimensions specified in Drawing No.5 is to be connected to a standard Grassot Fluxmeter, and placed symmetrically between the pole pieces of the apparatus. A throw is obtained by withdrawing the coil and this is to be equivalent to the figure given above.

Extraction of Ferruginous Matter

A wet weight of the nitrocellulose is taken, which, by experience, or by rough volatile matter determination, contains approximately 50 grams of the dry material. This is mixed with 1,300 c.cs. of water and is then put into the bath of the magnetic separator apparatus, and the paddle of the apparatus started, the rotation being at a constant speed of approximately 200 revs. per minute causing the stream to flow in the direction indicated on Drawing No. 4. After the extraction has proceeded for one hour the paddle wheel is stopped, and, without breaking the circuit, the magnet is raised out of the bath. The magnet poles are washed gently with a minimum quantity of distilled water and the current is then switched off. The extracted particles of magnetic matter are then wiped off with a moist ash-free filter paper, the poles being then replaced in position and the stirring continued.

The operation of wiping the magnets is carried out again after a further one hour stirring, and finally at the end of a further hour's stirring, making the total time of test 5 hours.

The filter paper containing all the ferruginous matter obtained during 5 hours extraction is incinerated and the ash treated with 5 cc. of conc. Hydrochloric Acid and a few drops of concentrated nitric acid with gentle heating. The solution is made up to a convenient volume with water and its iron content estimated colorimetrically, with ammonium thiocyanate. A blank determination for iron should be carried out on the apparatus after each series of approximately 100 tests, or more frequently if considered necessary.

Standard Iron Solution

The stock iron solution contains 0.05 gram of ferric iron and 2.5 c.cs. of concentrated Hydrochloric Acid per 100 c.cs. and is prepared by dissolving pure iron wire in Hydrochloric Acid. From the solution the iron, after oxidation, is precipitated with ammonia, the precipitate of Ferric Hydroxide is washed, and redissolved in Hydrochloric Acid. One volume of this solution is diluted with nine volumes of 10 per cent Hydrochloric Acid immediately before carrying out the determination. 1 cc. of this diluted solution contains 0.00005 gram iron.

Determination of Iron

The amount of iron in the test solution is first determined approximately by means of thiocyanate. An aliquot portion containing about 0.00007 to 0.0001 gram iron is then measured into a 100 cc. Nessler tube, made up to 50 cc. with water and well mixed. In a

~~second similar Nessler tube are placed 50 cc. of water. To each tube is then added 10 cc. of 10 per cent. nitric acid, followed by 10 cc. of 20 per cent. ammonium thiocyanate solution. The contents of each tube are then made up to 100 cc. with water and the blank is titrated with the standard iron solution to equivalent colour.~~

Acceptance test for new plating

The plated parts are to be immersed in the following reagent warmed to approximately 70°C. -

Gelatine.....	100	parts
Sodium Chloride.....	5	"
Potassium Ferricyanide.....	10	"
Water.....	885	"

The jelly is allowed to set and remain for 12 hours, at the end of which time no blue spots should have appeared.

Coil for use with Grassot Fluxmeter in determining flux across pole-pieces of magnetic iron test apparatus. (Drawing No.5).

1. Coil of 30 turns No. 36 S.W.G. enamelled copper wire, wound on former $2\frac{1}{4}$ inches x $1\frac{1}{4}$ inches with rounded corners. Coil to be shellaced, removed from former, taped with silk tape, and varnished with shellac varnish and baked until rigid.
2. Handle of thin walled brass tube, 3 inches long, $\frac{5}{16}$ inch diameter.
3. Brass lug soldered to (2), taped to (1), and varnished when assembly is complete.
4. Soldered joints from ends of coil to ends of twin flexible copper lead - length about 3 feet.
5. Insulating sleeves over joints.
6. Flexible leads.
- ~~7. Ebonite bush screwed into (2).~~

Method of preparation of sample for the following determinations:-

Mineral matter, Alkalinity, Sulphates, Organic matter Insoluble in Acetone, N.C. Soluble in ether-alcohol, 132°C. Stability Test, Nitrogen, and Settling Test.

Place the sample of Nitrocellulose (equal to about 50 grams of dry N.C.) between layers of thick filter paper and subject under a handscrew press to a tolerably severe pressure for about 3 minutes. The filter paper must be a pure paper free from chemical impurities. Alternatively the Nitrocellulose may be wrung in a centrifuge. Tease out the pressed sample loosely with the fingers, spread out in a thin layer on a paper or aluminium tray; dry in an oven for at least 3 hours at about 60°C., allow to cool in a desiccator, place in a clean dry bottle and close with a rubber bung.

Determination of Mineral Matter

Cover 2 grams of the prepared sample with B.P. paraffin, ignite, allow to burn away and remove the carbonaceous residue by gentle ignition. Add about 0.5 gram of solid Ammonium Carbonate, moisten with water, dry, and heat at 105-110°C. to constant weight.

Determination of Alkalinity

Transfer 10 grams of the prepared sample to a stoppered cylinder, add 100 cc. of N/10 Hydrochloric Acid, then 100 cc. of water, and shake for one hour. Stand to allow the nitrocellulose to settle, remove 100 c.c. of the clear liquid by means of a pipette, and titrate the residual acid contained in it with N/10 sodium carbonate using methyl orange as indicator. Carry out a blank experiment, omitting the nitrocellulose.

Report Alkalinity as CaCO_3 .

Determination of Sulphates

Heat 5 grams of the prepared sample gently on water bath with 50 cc. of concentrated nitric acid, until the nitrocellulose is dissolved and evolution of nitrous fumes ceases, cool, add 1 gram of sodium chlorate and evaporate solution to dryness. Treat the residue with 20 cc. of concentrated Hydrochloric Acid, add about 0.5 gram of sodium chlorate, and evaporate the mixture again to dryness. Repeat the treatment with Hydrochloric Acid and Sodium Chlorate. Estimate the Sulphate in the residue gravimetrically by precipitation with Barium Chloride. Carry out a blank experiment and apply a correction for the amount of Sulphate present in the reagents used.

Determination of Organic Matter Insoluble in Acetone

Transfer 1 gram of the prepared sample to a glass tube (45 cms. by 2.5 cms.) containing 150 cc. acetone (of the quality specified in the appendix). Shake to dissolve the nitrocellulose, and stand overnight to allow the insoluble material to settle out. Siphon off the clear supernatant liquid, and add a further quantity of 150 cc. acetone, then add two or three drops of hydrochloric acid to destroy the calcium carbonate.

The volume of settled residue is noted and compared with the volume occupied by the residue from 1 gram of standard nitrocellulose, the residue from which has been previously determined gravimetrically.

If on comparison the amount of insoluble material appears greater than the standard, wash the residue on to a tared ~~glass~~ filtering crucible and wash with acetone until washings leave no residue on evaporation. Dry residue in boiling water oven for 2 hours or until constant, ~~then gain in weight~~ calculate percentage organic insoluble material. ~~ignite, reweigh and.~~

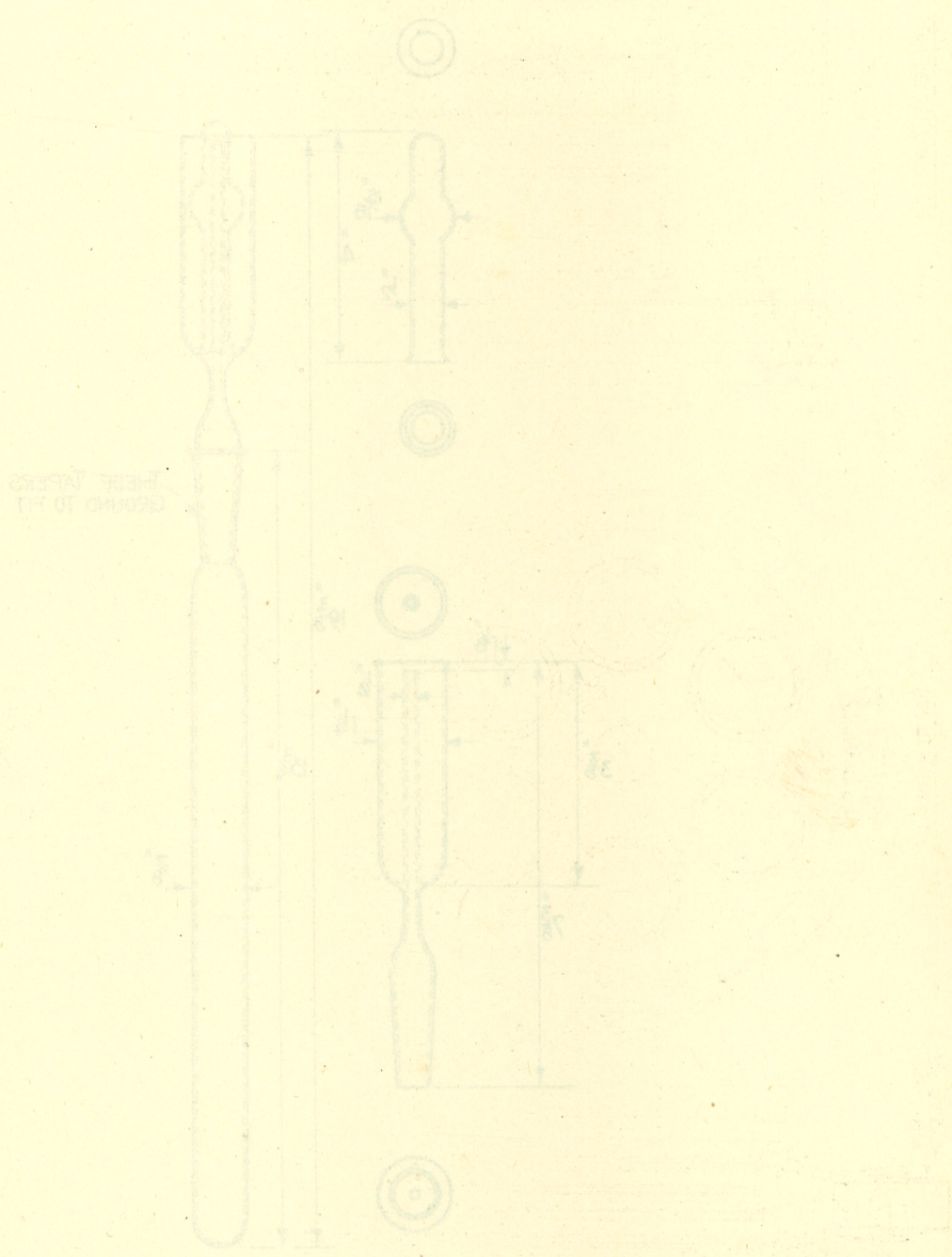
Specification for Acetone (see Appendix III)

Determination of N.C. Soluble in Ether-Alcohol

Transfer 1 gram of the prepared sample to a 100 cc. cylinder, add 25 cc. alcohol, shake thoroughly to soak the N.C., then add 25 cc. ethyl ether (alcohol and ether to be of the quality specified in the appendix). Shake to dissolve the N.C. and stand overnight to allow insoluble material to settle out.

The volume of settled residue is noted and compared with the volume occupied by the residue from 1 gram of standard nitrocellulose the residue from which has been previously determined gravimetrically. If insoluble is less than or equal to the standard report the solubility as greater than or equal to $100 - (X + Y)$ where X is the percentage of undissolved fibrous material in the standard and Y is the percentage ash in the sample.

TEST TUBE
DRAWING No 7



SECTION JK60 C.D

If greater than the standard, siphon off the clear supernatant liquid, add fresh ether-alcohol up to the original volume. Shake, allow to settle completely and repeat siphoning operation. Wash residue into a tared Jena glass filtering crucible, and wash with ether-alcohol until washings leave no deposit on evaporation. Dry the crucible in a boiling water oven for 2 hours or until constant; the increase in weight of crucible gives the amount of insoluble material; from this figure calculate and report the percentage soluble material in the N.C.

Specification for Ethyl, Ether and Alcohol (see Appendices I and II)

132°C. Stability Test

Description of Apparatus (Drawing No. 6)

- (a) Bath. The bath consists of a cylindrical copper vessel fitted with a Habermann condenser. Fixed flush in the top of the bath are six copper tubes (for holding test tubes) which are closed at the bottom. Alternatively, the tubes and the top of the bath may consist of one brass casting. The bath is covered on the sides and top with a layer of asbestos lagging about $\frac{1}{2}$ inch in thickness. The top of the bath is fitted with a horizontal collar of asbestos board projecting about 6 inches from the side of the bath in order to prevent undue heating of the atmosphere above the bath. The bath should be surrounded by a roomy screen for safety and to prevent access of draughts.
- (b) Filling for the bath. The bath is filled with a mixture of glycerine and water containing approximately 86.4 parts by volume of glycerine adjusted to have a boiling point of 132°C. (270°F.). The copper tubes contain paraffin B.P.
- (c) Preparation for testing. The bath is heated by a Teclu burner placed centrally. The volume of boiling liquid must be adjusted to be exactly at the height of the side outlet during the test. When the bath is boiling a stability test tube, (Drawing No.7,) is inserted into each of the copper tubes in turn to a depth of 6 inches from the metal top of the bath and the quantity of paraffin B.P. in the copper tubes is adjusted to be 2 inches from the metal top of the bath with the test tube in this position. The temperature of the bath is taken by means of six thermometers fixed by corks in the copper tubes so that the bottom of each thermometer is at a depth of six inches below the level of the metal top of the bath. The proportions of the glycerine mixture, and the position of the burner, are so adjusted that the temperature in all the copper tubes will remain at 132°C. \pm 0.2°C. during the test.

The temperature in the copper tubes is verified over a period of one hour before the test is commenced and two of the thermometers on opposite sides of the bath are left in the copper tubes during the test. The temperature of the air above the bath in the neighbourhood of the absorption cups must not exceed 40°C.

Care should be taken to free the Stability Test tubes from alkali before use.

Application of the Test

Sieve 10 grams of the prepared sample through the ^{upper} ~~lower~~ Heat Test sieve, or alternatively through a screen of ~~aperture~~ ^{aperture} approximately of similar ~~aperture.~~ ^{aperture.} 0.03 inch. Weigh out three portions each of 2 grams and transfer to Stability Test tubes settling the Nitrocellulose by gentle tapping; put one tube aside as a blank test, and fit absorption cups containing

8 to 10 cc. of water, in order to seal the apparatus, to the other two tubes, lubricating the joints with non-reactive grease; fix each of the two tubes vertically to a depth of 6 inches in one of the copper tubes of the bath by means of a cork with a central hole: leave in the bath for exactly two hours, remove, add a further quantity of 20 to 30 cc. to the cups and allow to cool, for not more than one hour, to room temperature: rinse each cup into a conical flask and add 20 cc. N/10 Hydrochloric Acid to each test-tube and also to the blank: cork the tubes and shake for 15 minutes: filter the contents of each tube without delay into the appropriate flasks and wash thoroughly: add 25 cc. N/10 caustic soda to each solution, adjust to same bulk, which should be about 300 cc. and titrate with N/10 Hydrochloric Acid adding a few drops of methyl orange immediately before titration.

Then if A = titre of the heated sample
B = " " " blank

the amount of Nitrogen liberated per gram of nitrocellulose

$$= 0.7 (B - A) \text{ m.g.}$$

Determination of Nitrogen

Take about 5 grams of the prepared sample and sieve completely through the ^{upper} ~~lower~~ Heat Test sieve, or alternatively through a screen of aperture ~~approximately 0.03 inch~~. Transfer about 0.52 gram to a tared weighing bottle, dry for 1 hour at 100°C., cool in desiccator and weigh.

Add 5 cc. of pure Sulphuric Acid (94 per cent by weight), thoroughly mix the Nitrocellulose by minimum amount of stirring with a glass rod, and transfer the mixture without delay to the cup of a 150 cc. Lunge Nitrometer (the time of this operation should be about 1 to 2 minutes).

Wash the residue in the weighing bottle and cup into the nitrometer tube with a further 10 cc. of acid, added in several portions each of about 2 cc., each portion of the acid being at once transferred into the nitrometer tube. Allow to stand for 20 minutes: * shake vigorously for 2 minutes; support at rest at roughly atmospheric pressure for 3 minutes; shake vigorously for $\frac{1}{2}$ minute, then (care being taken to leave the inside of the nitrometer clean) allow to stand for 30 minutes at roughly atmospheric pressure, with a thermometer in contact with the bulb.

Finally, read the temperature and rapidly adjust gas to atmospheric pressure, using as indicator the bubble in the nitrometer cock (half way down capillary). Allow to stand 3 to 5 minutes, read the volume and correct to N.T.P.

* At this stage, if mechanical shaking is adopted, the measuring arm of the nitrometer is laid in the wooden case of the mechanical shaker, the covering lid of which is firmly secured before shaking is commenced. When at rest the box of the shaker is inclined at an angle of approximately $26\frac{1}{2}^{\circ}$ to the surface of the supporting table. This table also carries the necessary vertical support for supporting the levelling arm of the nitrometer.

The shaking procedure to be adopted involves two stages, viz., the preliminary shaking and the final shaking. The preliminary shaking comprises one or more series of swings of short duration, the number of swings on each occasion being four to six. The motor, driving the shaker, is shut off between each series of swings and the shaking recommenced after a pause of a few seconds. The duration of the preliminary shaking is governed by the time required to disengage the major portion of the gas. When the greater portion of the nitric oxide has been evolved, as shewn by the rise of the mercury column in

the upright levelling arm of the nitrometer, the final stage of shaking is commenced (usually after a preliminary shaking involving three series of swings). At this stage shaking is continued for 3 minutes, without interruption, after which the nitrometer is removed and mounted vertically with the gas at a pressure slightly below atmospheric. A thermometer rests in actual contact with the 100 cc. gas bulb of the nitrometer, being secured in position by means of a spring. After 30 minutes' standing the temperature is read and an accurate adjustment of the gas to atmospheric pressure is made by raising or lowering the levelling tube of the nitrometer by means of a pulley device. The use of such a device obviates the necessity for handling the nitrometer.

Settling Test

8 grams of the prepared nitrocellulose are placed in a 250 cc. stoppered cylinder graduated every 2 cc. About 30 c.cs. of distilled water are added and the nitrocellulose mixed well, using a glass rod. After stirring, sufficient additional distilled water is added to bring the level up to the 200 cc. mark. The cylinder is then stoppered and shaken by hand for 10 minutes, being afterwards placed on a flat surface free from vibration and allowed to stand for one hour. The level of the settled nitrocellulose is then read off.

The cylinder used is to be of such dimensions that the 200 cc. mark is 20 to 22 cms. from the zero mark.

PRECIPITATED CHALK
(Specification A.99, Appendix D.)

Settling Test

5 grams of chalk are introduced into a calibrated tube, 55 c.m. long and 1.6 c.m. internal diameter, and water is added up to the 100 cc. mark. The mixture is thoroughly shaken and allowed to settle in a vertical position on a bench free from vibration. At the end of one hour the reading is taken of the sharp line between the deposit of settled chalk and the turbid mixture above.

Settling Test

5 grams of the prepared nitrocellulose are placed in a 250 cc. stoppered cylinder graduated every 20 cc. About 30 cc. of distilled water are added and the nitrocellulose mixed well, using a glass rod. After stirring, sufficient additional distilled water is added to bring the level up to the 200 cc. mark. The cylinder is then stoppered and shaken by hand for 10 minutes, being afterwards placed on a flat surface free from vibration and allowed to stand for one hour. The level of the settled nitrocellulose is then read off.

The cylinder used is to be of such dimensions that the 200 cc. mark is 30 to 32 cms. from the zero mark.

NITRIC ACID
(Specification A.99, Appendix E.)

Test for Nitrous Acid:

5 grams of the acid, diluted with 1 litre of water, are treated with 32 cc. of N/10 Potassium Permanganate (1 cc. = 0.00235 gram of nitrous acid).

GLYCERINE 203
(Specification A. 95)

Nitrogen

2 grams of the glycerine are treated with 20 cc. of Sulphuric Acid and 10 grams of Potassium Sulphate by the Kjeldahl method, the final figure being obtained colorimetrically by means of Nessler tubes.

Sugar, Molasses and Gums

5 grams of the glycerine are agitated with an equal volume of concentrated sulphuric acid. Any darkening so produced is attributed to the presence of these impurities.

Saponification Equivalent

Weigh 25 grams of the sample into a flask, add 5 cc. of distilled water free from CO_2 , and 50 cc. N/10 NaOH. Boil under reflux condenser for 5 minutes, cool, and titrate uncombined alkali with N/10 H_2SO_4 using phenolphthalein as indicator.

(For determinations see the specification)

CARBAMITE 206
(Specification A. 154)

~~Determination of Setting Point~~

~~Melt about 50 grams of the sample in a 100 cc. beaker in the boiling water oven.~~

~~10 minutes after melting is complete, pour the molten carbamite into a test-tube (6 inches x 1 inch) and fit a cork carrying a standardised thermometer and stirrer. The standard thermometer has a range 67-77°C. and is graduated in 0.1°C.~~

~~The test-tube and stirrer are heated alongside the sample.~~

~~Fit the test-tube into an empty boiling tube 7 inches x 1½ inches and stir until the temperature falls to 77°C., then support the apparatus in an empty vessel of about 1 litre capacity, and continue vigorous stirring so long as the temperature readings fall. When the temperature begins to rise, withdraw the stirrer, and record the subsequent thermometer readings.~~

~~The highest reading obtained after super-cooling is taken as the Setting Point.~~

(For ~~other~~ determinations see the Specification)

PAPER WOOD CELLULOSE
(Specification A.165.)

Estimation of Lignin

0.2 gramme of air-dry paper previously cut into thin strips is accurately weighed, and stirred with a glass rod into 25 c.c. of colourless concentrated sulphuric acid at room temperature until completely dissolved. One hour after adding the paper to the sulphuric acid the colour developed in the acid is compared, by means of the Duboscq colorimeter, with the colour similarly developed by the same weight of a standard paper of known lignin value.

Estimation of Copper Value

The following solutions are required:-

- | | | |
|-----|---|--------------|
| (A) | Pure Copper Sulphate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ | 100 gms.) |
| | Water..... | to 1 litre) |
| (B) | Sodium Bicarbonate..... | 50 gms.) |
| | Sodium Carbonate (crystals)..... | 350 gms.) |
| | Water..... | to 1 litre) |
| (C) | Iron Alum..... | 100 gms.) |
| | Sulphuric Acid (S.G.1.84)..... | 140 cc.) |
| | Water..... | to 1 litre) |

Approximately 2.2 grams of air-dry paper is accurately weighed and transferred to a litre round-bottom flask, 10 cc. of Solution A. (Copper Sulphate) and 190 cc. of Solution B. (Sodium Carbonate + bicarbonate) are mixed, brought to the boil and immediately poured on to the paper. A few pieces of porous pot are added and the flask is fitted with a reflux condenser, preferably connected with glass joints or alternatively passing through a cork completely covered with tin foil. The contents of the flask are gently boiled for $1\frac{1}{2}$ hours, then immediately filtered hot on a suction filter, through hardened filter paper or muslin previously extracted with boiling water and the paper (which now contains cuprous oxide) washed with 200 cc. of a cold 5 per cent aqueous sodium carbonate solution followed by an equal volume of distilled water. The filter flask is emptied, cleaned with distilled water and replaced. 25 cc. of iron alum solution (C) is added to the paper in the funnel from a burette in two quantities of 15 cc. and 10 cc. respectively. The paper is well stirred with a glass rod into this solution, suction being applied after each addition and mixing. It is usually found that this 25 cc. of iron alum solution suffices to dissolve all the cuprous oxide from the paper, as seen by the disappearance of its characteristic red colour but in the case of highly reducing celluloses, a further 10 cc. of solution (C) may be required. The paper is now washed three times with 25 cc. of approximately 2 N sulphuric acid. The combined filtrate and acid washings is titrated with N/25 potassium permanganate solution until a faint pink colouration is produced. At least two determinations are to be made.

It is necessary to carry through a "blank" experiment on the solutions as used. The same volume of solution as used above is boiled alone under a reflux condenser for $1\frac{1}{2}$ hours, filtered through the same type of filter paper or muslin as used in the actual determination and this is washed with sodium carbonate solution and hot distilled water. After discarding filtrate and washings the filter

paper or muslin is treated with 25 cc. of iron alum solution and washed with sulphuric acid, the combined filtrate and washings being titrated with N/25 potassium permanganate. The blank titration (usually 0.30 to 0.35 cc. KMnO_4) is subtracted from the number of cc. required in the determinations.

Calculation

1 cc. N/25 $\text{KMnO}_4 = 0.00284$ gramme Cu_2O .

The Copper value is expressed as gramme Cu_2O per 100 grammes calculated on the bone dry paper.

For other determinations see Specification ~~A.165 E.~~^{the}

CANDELILLA WAX
(Specification A.97)

Setting Point

A test-tube, 6 inches by 1 inch, is filled within $\frac{1}{2}$ inch of the top with the melted wax, and is supported by means of a cork inside a glass bottle. The latter is of such a size that $4\frac{1}{2}$ inches of the test-tube are within the bottle and there is a space of not less than $\frac{3}{4}$ inch all round the test-tube. A thermometer is fitted through a cork in the test-tube so that the bottom of its bulb is 1 inch from the bottom of the test tube.

The wax is allowed to cool without stirring and temperature readings are taken at intervals of 1 minute.

The first such reading which is immediately followed by one not more than 0.1°C . lower is taken as the Setting Point.

(For the other determinations
see the Specification.)

APPENDIX I

Specification for Ethyl Ether

(Used in analysis of Cordite, and in the determination of the solubility of Nitrocellulose in ether-alcohol)

1. Specific Gravity:

The Specific Gravity at 15.5°C . is to be 0.720 ± 0.001 .
 15.5°C .

2. Boiling Point:

500 cc. of the ether distilled through a Glinsky tube must distil over completely between 34°C . and 35°C . at normal pressure.

3. Residue:

The residue remaining after spontaneous evaporation of 50 cc. in a glass dish till no ether remains must be clear, colourless, odourless, and neutral to litmus, After drying on the water bath at 80°C ., No residue must remain.

4. Acidity:

50 cc. of the ether are transferred to a stoppered separating funnel containing 50 cc. of CO_2 - free water which has been neutralised to phenolphthalein as indicator. The mixture is shaken and the aqueous extract is run off and titrated with N/100 sodium hydroxide. Limit . 0.002 per cent. calculated as H_2SO_4 .

5. Peroxides:

10 cc. of the ether are shaken with 10 cc. of water containing 1 cc. of 0.1% solution of titanous acid. A yellow colouration indicates the presence of peroxides. No yellow colour should be produced.

6. Sulphur Compounds:

One drop of pure mercury must show no discolouration when shaken in the presence of air with 10 cc. of ether which has been acidified with a few drops of acetic acid.

7. Iodoform Yielding Substances:

Not more than 0.2 gram iodoform should be obtained from 100 cc. ether when tested for iodoform yielding substances by the following method:-

50 cc. ether, 50 cc. distilled water and 10 cc. N/1 NaOH are shaken together, 20 cc. N/10 Iodine are slowly added at frequent intervals with shaking 11 cc. N/1 H_2SO_4 are then added and just sufficient Sodium Thiosulphate solution to discolourise the solution on vigorously shaking. The ethereal layer is then separated, washed three times with small quantities of distilled water and then evaporated quickly at ordinary temperature in a current of dry air. The residue is dried for 1 hour in a vacuum desiccator in the presence of a small amount of iodoform. The residue should not exceed 0.1 gram.

APPENDIX II

Specification for Alcohol

(Used in the determination of the solubility of nitrocellulose in ether-alcohol)

1. The alcohol is to be undenatured distilled spirit.
2. The alcohol must be colourless, and free from suspended matter or deposit.

3. Acidity:

50 cc. of water are boiled for 5 minutes in a flask of "resistance" glass. 50 cc. of the alcohol are then added, and the boiling is continued for 5 minutes. The contents of the flask are rapidly cooled and titrated with N/100 Sodium Hydroxide solution using phenolphthalein as indicator.

4. Residue:

The alcohol must leave no residue on evaporation.

5. Specific Gravity:

The specific gravity at $\frac{15.5^{\circ}\text{C.}}{15.5^{\circ}\text{C.}}$ is to be 0.830 ± 0.001 .

6. Aldehydes:

The alcohol must comply with the following test for the presence of aldehydes:-

A solution is made up in which 100 cc. contain 0.75 gram silver nitrate, 0.75 gram caustic soda and 5 cc. of ammonium hydrate (Sp.Gr.0.880).

5 cc. of the alcohol are diluted with 10 cc. of distilled water and 1 cc. of the above silver nitrate solution, together with 3 drops of 2 per cent. solution of ferric alum are added and mixed. After standing for one hour in the dark, the liquid is filtered and the filtrate acidified with nitric acid. A few drops of hydrochloric acid are added. A turbidity must be obtained indicating excess of unreduced silver salt.

APPENDIX III

Specification for Acetone

(Used in the Colour Test of Cordite, S.C. and in the determination of organic matter in nitrocellulose insoluble in acetone)

1. The acetone is to contain no ingredients other than small quantities of substances which are normal by-products of its manufacture. It is to be colourless and transparent.
2. Turbidity with water:
When mixed with distilled water in any proportion it is not to show any turbidity.
3. Residue:
It is not to leave any residue when evaporated to dryness over a water bath.
4. Specific Gravity:
The specific gravity is not to be greater than 0.8000 at 15.5°C.
15.5°C.
5. Permanganate Test:
1 cc. of an 0.1% solution of potassium permanganate is added to 100 cc. of the acetone, the mixture being maintained at 15.5°C. and protected from light during the test. The colour should remain the same for not less than 30 minutes.
6. Acidity, including carbon dioxide:
50 cc. of the acetone is added to an equal volume of cold neutralized CO₂- free water containing a small quantity of phenolphthalein and the mixture titrated with N/20 caustic soda. *no more than 10 cc should be required to produce a pink tint.*
7. Acidity after removal of carbon dioxide:
50 cc. of the acetone similarly diluted with water are boiled until the bulk of the acetone has been removed, and the remaining liquid similarly titrated. *the acidity must not exceed 0.002 calc. as acetic acid*
8. Alkalinity:
50 cc. of the acetone are added to 50 cc. of cold CO₂ free water neutralised to paranitrophenol. ~~The mixture is titrated with N/20 sulphuric acid.~~ *The solution must not turn yellow.*
9. Alcoholic Impurities:
1 cc. of the acetone is mixed with 3 cc. of Agulhon's reagent in a test-tube and allowed to stand in the cold for 5 minutes. No blue or violet colouration should be obtained.

NOTE: Agulhon's reagent shall be prepared by dissolving 0.5 gram of A.R. potassium dichromate in 100 cc. of Nitric Acid of Specific Gravity 1.332.

Specification for Acetone

filled in the color test of Corbis, S.C., and in the determination of organic matter is nitrocellulose insoluble in acetone)

1. The acetone is to contain no ingredients other than small quantities of substances which are normal by-products of the manufacturer. It is to be colorless and transparent.

2. It is to be free from turbidity when mixed with distilled water in any proportion it is not to show any turbidity.

3. Residue. It is not to leave any residue when evaporated to dryness over a water bath.

4. Specific Gravity: The specific gravity is not to be greater than 0.8000 at 15.5°C. 15.5°C. 15.5°C.

5. Permanganate Test: 1 cc. of an 0.1% solution of potassium permanganate is added to 100 cc. of the acetone, the mixture being maintained at 15.5°C. and protected from light during the test. The color should remain the same for not less than 30 minutes.

6. Alkalinity, including carbon dioxide: 50 cc. of the acetone is added to an equal volume of cold neutralized 2% free water containing a small quantity of phenolphthalein and the mixture titrated with N/20 caustic soda.

7. Alkalinity after removal of carbon dioxide: 50 cc. of the acetone similarly diluted with water are boiled until the bulk of the acetone has been removed, and the remaining liquid similarly titrated.

8. Alkalinity: 50 cc. of the acetone are added to 50 cc. of cold 0.5% free water neutralized to phenolphthalein. The mixture is titrated with N/20 caustic soda.

9. Alcoholic Impurities: 1 cc. of the acetone is mixed with 5 cc. of Anilone's reagent in a test-tube and allowed to stand in the cold for 5 minutes. No blue or violet coloration should be obtained.

NOTE: Anilone's reagent shall be prepared by dissolving 0.5 gram of A.R. potassium dichromate in 100 cc. of Nitric Acid of Specific Gravity 1.532.

In Samman

6th May 1936
(G2647/36)

Addendum (I)

Acceptance Tests of Cordite S.C
and its Ingredients
Schedule of Methods to be employed

Approved. 21.11.1935

INDEX

Cordite S.C (Specification A99)

Add

Estimation of Total Iron

Page
2

Nitrocellulose (Specification A99.
Appendix B)

Delete

Estimation of Magnetic Iron

7

Page 2. (ii) Estimation of Nitrocellulose

1.

paragraph 1. line 4

after "98" insert "or 360".

IV Estimation of Total Iron

Place 10 grams of Cordite ground "VM" size in a crucible about 3 inches in diameter; add about 20 cc. nitric acid Sp.G. 1.5 and warm carefully. After the evolution of red fumes has subsided, evaporate the solution to dryness and heat the residue until all carbonaceous matter is burnt off.

Dissolve the ash in 5 cc. concentrated Hydrochloric Acid, add a few drops of strong nitric acid and boil, cool, and make up the volume to 100 cc. with water. To a measured portion of this solution (chosen so as to give a suitable tint in the subsequent estimation), add 10 cc. of a 10 per cent solution of nitric acid and 10 cc. of a 4 per cent Ammonium Thiocyanate solution. Transfer solution to a Nessler tube and make up the volume to 100 cc. with water.

In a second Nessler tube make up a blank solution containing similar amounts of nitric acid and Ammonium Thiocyanate and add standard iron solution from a burette until equal intensity of colour in the two solutions, each of 100 cc., is obtained.

Standard Iron Solution

(a) Stock Solution: Dissolve a weighed amount of pure iron wire in Hydrochloric acid, oxidise with a few drops of nitric acid, and precipitate with Ammonium Hydroxide. Filter, wash and redissolve in Hydrochloric acid. Adjust the solution to contain 0.05 gram iron and 2.5 cc. of Hydrochloric acid in 100 cc.

(b) Test Solution: Dilute one volume of the stock solution with 9 volumes of 10 per cent Hydrochloric Acid immediately before making a determination. 1 cc. of this diluted solution contains 0.00005 gram of iron.

3. Pages 7-8-9-Diagrams 4.4A & 5 "Determination of Magnetic Iron" - cancelled -

4. Page 10. Determination of Organic matter.
Insoluble in Acetone.

paragraph 3 line 2

delete "glass"

line 5.

delete "constant. From gain in weight"
insert "constant, ignite, reweigh, and"

5. Page 11. Application of the Test

paragraph 1. line 1

delete "lower"

insert "upper"

lines 2 & 3

delete "of aperture approximately 0.03 inch"
insert "of similar aperture".

6. Page 12. Determination of Nitrogen

paragraph 1. line 2

delete - "lower"

insert - "upper"

line 3

delete "of aperture approximately 0.03 inch"
insert "of similar aperture".

7. Page 19. last line.

delete "For other determinations see
Specification A165E"

insert (For other determinations see the
Specification).

8. Page 20. Candelilla Wax

insert at end

"(for other determinations see the Specification)

9. Page 21. Appendix I

Clause 3. line 3

delete "litmus, after"

insert "litmus. After"

line 4

delete "80°C. No."

insert "80°C, no."

Clause 4. line 5.

after "hydroxide"
add "Limit - 0.002 per cent calculated as H₂SO₄"

Clause 5. line 3

after "peroxides"
add "No yellow colour should be produced"

10. Page 22. Appendix II

Clause 3. add at end of clause.

"The acidity must not exceed 0.005 per cent
"calculated as H₂SO₄

insert new clause 7.

"The alcohol must shew no turbidity
"when one volume is mixed with 5
"volumes of distilled water at 15°C."

11. Page 23. Appendix III

Clause 6 add at end of clause

"Not more than 1.0cc should be required
"to produce a pink tint".

Clause 7 add at end of clause

"The acidity must not exceed 0.002 per
"cent calculated as acetic acid."

Clause 8. lines 2 & 3

delete "The mixture.....acid"
insert "The solution must not turn yellow."