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Certified Correct Copy of
Sealed/Unsealed Drawing/s &
Specification
At this date..... 24/2/98

for C. I. M. E. Kirkee

Certified Correct Copy of
Sealed/Unsealed Drawing/s &
Specification
At this date 22-2-94

for C. I. M. E. Kirkee

IND/ME/856 ~~PROV~~

Based on MIL C 20470 A)

CALCIUM RESINATE

Type I FUSED

Type II PRECIPITATED

QC(TC)

Issued by

CONTROLLERATE OF INSPECTION (MILITARY EXPLOSIVES)
KIRKEE, PUNE -411 003.

DEPARTMENT OF DEFENCE PRODUCTION

MINISTRY OF DEFENCE.

IND/ME/856 (PROV.)

AMENDMENT RECORD

DC(I) NO. Date	Brief Particulars of alteration	Numeral to which specification advanced	Remarks and Initials

CALCIUM RESINATE

CONTENTS

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0. FOREWORD

0.1 This specification has been prepared by the CONTROLLERATE OF INSPECTION (MILITARY EXPLOSIVES) KIRKEE, PUNE-411 003.

0.2 For additional copies or any other enquiry regarding this specification, reference should be made to CI(ME) Kirkee who is Authority Holding Sealed Particulars and Inspection Authority for this specification.

1. SCOPE

1.1 This specification is meant to govern supply and inspection of calcium resinate type I fused and type II precipitated.

1.2 The material is suitable for use in the manufacture of composition Igniter ME 408 or similar pyrotechnic compositions.

2. RELATED DOCUMENTS

2.1 The related documents mentioned at clause 2.2 are those applicable at the date of publication of this specification. It is contractor's responsibility to confirm their current applicability and to obtain from the Authority Holding Sealed Particulars (i.e. CI(ME) KIRKEE) information concerning any change that may be necessary due to cancellation, replacement or supersession of any of the document.

2.2 Copies of the related document are obtainable from

I.S. 460 -1962 — Indian Standards Institution
MANAK Bhavan,
9, Bahadur Shah Zafar Marg,
NEW DELHI -110 002.

.....5.

3. DESCRIPTION

3.1 The calcium resinate type I shall be yellow or brown lumps free from foreign matter and visible impurities. and type II shall be yellowish white amorphous powder free . from lumps, cakes and agglomerates, foreign matter and visible impurities.

4. TENDER SAMPLE

4.1 The contractor shall submit two tender samples each of 250 g, essentially from the same batch/lot, free of charge and conforming to this specification.

5. PRE-INSPECTION

5.1 Before tendering the store to the Inspector the contractor shall carry out a thorough inspection of each delivery to satisfy himself that the store fully conforms to this specification and shall render a certificate to that effect to the Inspecting Officer.

6. INSPECTION

6.1 The calcium resinate type I and II and the packages in which it is contained shall be subject to inspection by and to the final approval of the Inspecting Officer/Authority.

6.2 Samples of the material and of the packages may be taken from any portion of a consignment.

6.3 If, on examination, any sample be found not to conform to this specification, the whole consigbment may be rejected.

6.4 The foregoing provisions shall apply equally to prime contractors and to sub-contractors, if any.

7. SAMPLING

7.1 Normally two representative samples, each of 250 g shall be drawn from each batch/lot . However, the number of samples to be drawn from each batch/lot will be at the discretion of the Inspecting Officer.

8. TEST REQUIREMENTS

8.1 Samples taken from any portion of the supply shall comply with clause 3 above and shall conform to the following requirements :-

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Sl. No.	Characteristics	Passing Standard Type I	Passing Standard Type II	Test Method
1	2	3	4	5
1.	Calcium resinate content, percent, Min.	56	77	Appendix C
2.	Acid number, Max.	64	33	Appendix A
3.	Matter insoluble in carbon tetrachloride, percent, Max.	2	3	Appendix B
4.	Water content percent, Max.	2	5	Appendix D
5.	Flame test	Brick red		Appendix E
6.	Granulation			
	(a) Passing through 250 micron I.S. Sieve percent.	none (lump)	75 min	
7.	Softening point	For information		Appendix F

Matter insoluble in cc14/chloroform.

9. PACKING AND MARKING

9.1 Packing

9.1.1 The calcium resinate Type I and Type II shall be supplied in sound, clean, dry, air-tight packages which may consist of a suitable heat sealed polythene bag of film thickness 0.13 mm, placed inside an air-tight container containing an approved quantity.

9.1.2 Supplies offered in any other packages shall have the prior approval of the Inspecting Officer.

9.2 Marking

9.2.1 The packages constituting a consignment shall each be legibly and durably marked with the following details :

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(a) Nomenclature and specification No. of the store

(b) Name and address of the consignee

(c) A/T / S.O. No. and date

(d) Consignment No.

(e) Distinctive lot No. and date of manufacture

(f) Consecutive No. of packages and total No. of packages

(g) Gross and net mass

(h) Date of supply

(j) Contractor's initial or recognised trade mark.

9.2.2 Pre-cautionary marking : Each container shall be marked "Flammable" and "Store in a cool place away from fire and open flame".

9.2.3 In addition to above, the Inspecting Officer may suggest some more marking/identifications at the time of inspection.

9.2.4 The inclusion of any foreign matter or impurities in any of the packages shall render the whole consignment liable to rejection.

Surjit Singh

(Dr. SURJIT SINGH)
DIRECTOR
CONTROLLER OF INSPECTION (MILITARY EXPLOSIVES)

10. APPENDICES

APPENDIX 'A'

DETERMINATION OF ACID NUMBER

Transfer approx. 2 g of the sample, accurately weighed to 250 ml Erlemeyer flask. Add 25 ml of carbon tetrachloride and allow to stand for 1 hour with occasional swiriling. Add 25 ml of 95 % ethyl alcohol and 2 ml of phenolphthalein. Titrate with standard 0.1 N NaOH to a definite pink end point. Deduct a blank obtained by titrating a solution containing 25 ml carbon tetrachloride, 25 ml of alcohol and 2 ml of pehnolphthalein . Calculate as follows :

$$\text{Acid No.} = \frac{\text{ml of NaOH} \times \text{Normality of NaOH} \times 56.1}{\text{mass of sample.}}$$

APPENDIX 'B'

DETERMINATION OF CARBONTETRACHLORIDE INSOLUBLE MATTER

CORBON TETRACHLORIDE / CHLOFORM.

Weigh accurately 2.0 + 0.1 g of sample and transfer it to 250 ml beaker. Add 50 ml of carbon tetrachloride and allow to stand for one hour while swirlling occasiorally. Filter through a Gooch crucible (see note for preparation) transfer and wash the precipitate with carbon tetrachloride. Preserve the filtrate for calcium determination. Dry the crucible at 105°C for one hour, cool and weigh. Calculate as follows :

$$\text{\% carbon tetrachloride insoluble matter} = \frac{\text{mass of residue}}{\text{mass of sample}} \times 100$$

.....9.

NOTE : Preparation of Gooch crucible

Digest the asbestos (which should be of the amphibile variety) with HCl (1 : 3) for 2-3 days. Wash free from acid and digest for a similar period with 10% NaOH solution. Then treat for a few hours with hot alkaline tartarate solution of the strength employed in the sugar determinations** (Old alkaline tartarate solution that have stood for sometime may be used . Then wash free from alkali, digest with dil, HNO_3 (1 :3) for several hours and wash it free from acid, shake with water into a fine pulp. Use this pulp for preparing gooch crucible.

**Alkaline Tartarate solution

Take 17½ g Rochelle salt (sodium potassium tartarate) and 50 g NaOH in water, dilute to 500 ml for two days, filter keep it, and then use.

APPENDIX 'C'

CALCIUM RESINATE CONTENT

Reagents

(a) Buffer solution

Dissolve 67 g of NH_4Cl in 400 ml water. Add 570 ml of NH_4OH and dilute to 1 litre with water.

(b) Erichrome Black T Indicator

Dissolve 0.4 g of the indicator in 100 ml of methyl alcohol.

(c) Standard calcium solution

Dissolve 2.2 g of reagent grade $CaCO_3$ in a mixture of 20 ml of water and 5 ml of HCl by boiling gently on the hot plate. Cool and dilute to 2 litres. Standardise as follows :

Pipette 100 ml aliquot in 400 ml beaker. Add 100 ml water and 5 ml HCl. Heat to 95°C. Add 50 ml oxalic acid solution (2 %) that has also been heated to 95°C. Add NH_4OH dropwise with stirring until the solution smells faintly of ammonia.

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Place on the bench and allow to stand for 2 hours. Filter through a sintered glass crucible (G3) of medium porosity. Transfer the precipitate to the crucible and wash with 0.1 % oxalic acid. Finally wash ppt. with small quantity of water. Place the crucible into 600 ml beaker containing 300 ml of water and 12 ml of H_2SO_4. Stir, heat to about 60°C to dissolve and titrate with 0.1 N $KMnO_4$. Calculate as follows :-

$$\text{g of } CaCO_3 \text{ per ml of solution} = \frac{\text{ml } KMnO_4 \times \text{Normality of } KMnO_4 \times 0.050045}{\text{ml of } CaCO_3 \text{ solution taken for titration.}}$$

(d) EDTA Solution (Versenate solution)

Dissolve 8 g of EDTA (disodium dihydrogen-ethylene diamine tetra acetate) and 0.2 g of $MgCl_2.6\ H_2O$ in water and dilute to 2 litres. Standardise as follows. Pipette 20 ml aliquot of the standardised $CaCO_3$ solution into 250 ml Erlenmeyer flask and add 30 ml water; 10 ml of buffer solution and 5 drops of Eriochrome Black T indicator. Titrate with versenate to a bright blue colour that does not change on the further addition of versenate. Calculate as follows :-

$$\text{Factor (g of calcium resinate per ml of EDTA solution)} = \frac{\text{ml } CaCO_3 \text{ solution x g } CaCO_3 \text{ per ml x } 6.4237}{\text{ml of EDTA solution taken for titration.}}$$

PROCEDURE

Transfer the filtrate from the chloroform insoluble matter to 400 ml beaker and evaporate to dryness by heating on the steam bath. Add 35 ml HNO_3 and cover with watch glass. Boil to dryness by heating on hot plate until the residue is charred. This charring is important. Allow to cool, add 35 ml HNO_3 more and again evaporate till charring. Allow to cool, add 35 ml HNO_3 and 8 ml of

perchloric acid. Cover with watch glass and evaporate to fumes of perchloric acid. Continue fuming until volume is about 1 ml. Open the cover lid and evaporate to dryness. Allow to cool and dissolve the salt in water and dilute to 250 ml in volumetric flask.

Pipette 50 ml aliquot (equivalent to 0.4 g of sample) into 250 ml Erlenmeyer flask and add 10 ml of buffer solution and 5 drops of Eriochrome Black T indicator. Titrate with the EDTA solution to a bright blue colour that does not change on further addition of EDTA solution.

$$\% \text{ Calcium resinate} = \frac{\text{ml of EDTA solution x Factor x 100}}{\text{mass of sample in aliquot.}}$$

ALTERNATE METHOD

Ignite a weighed quantity of sample and extract with HCl. Precipitate calcium by addition of ammonium oxalate. Wash precipitate with hot water till free from oxalate. Dissolve the precipitate in dilute sulphuric acid and titrate with 0.1 N $KMnO_4$.

$$\text{Calcium resinate} = \frac{3.212\ TF}{W}$$

Where T = titre of $KMnO_4$ solution

F = factor of N/10 $KMnO_4$ solution

W = mass of the sample taken

APPENDIX 'D'

DETERMINATION OF WATER CONTENT BY DEAN AND STARK METHOD

Transfer 100 g to 500 ml balloon flask and add 200 ml of reagent grade toluene. Attach to the Dean and stark apparatus and heat the flask on an electric hot plate for 3 hours in such way that the distillate falls from the end of the condenser with the graduated part of the moisture tube at the rate of 2 to 3 drops per second. Midway in the 3 hour period, wash down the condenser with

5 ml chloroform. Do not wash again since the working operation will drive over some water from moisture tube into the flask. Shut off the heat and read the point of contact of the tube meniscus with the wall of the tube and the highest part of the lower meniscus. The difference is water. Calculate as follows :

$$\% \text{ water} = \frac{\text{ml of water} \times 100}{\text{mass of sample}}$$

ALTERNATE METHOD

HOT OVEN METHOD

Heat a flat bottomed clean glass or aluminium dish in the oven adjusted at temperature 100°C ± 5 deg C. Cool it in desiccator and weigh the mass (M1). Place 5 g of sample in the dish; replace the cover and weigh accurately the mass (M2). Uncover the dish and heat the sample in the oven till constant mass is obtained. Cover the dish with the lid and cool in desiccator at room temperature and weigh the mass (M3).

$$\% \text{ water content} = \frac{100 \times (M2 - M3)}{(M2 - M1)}$$

APPENDIX 'E'

FLAME TEST

Ignite 1 or 2 g portion in porcelein crucible until the ash is white or nearly colourless. Cool and moisten the residue with dil. HCl. Evaporate to dryness. Dip the loop of clean platinum wire into residue and hold the loop on blue flame of a bunsen burner. Note if the flame test is characteristic of a calcium as indicated by brick red colour with no perceptible green colouration indicating the presence of barium compound.

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APPENDIX 'F'

DETERMINATION OF THE SOFTENING POINT
(MODIFIED RING AND BALL METHOD)

1. APPARATUS.

(a) Vessle - A glass vessel capable of being heated not less than 8.5 cm in diameter and 10.5 cm in depth from the bottom of the flare (A 600 ml beaker, low form meets this requirement).

(b) Steel Ball
Mass of ball - 345 to 355 g
Diameter of ball - 9.5 mm

(c) Ring

Depth	6.35 ± 0.10	mm
Inside diameter at	bottom ...15.88 ± 0.10	mm
" " "	top .....17.46 ± 0.10	mm
Outside " "	bottom ...19.0 ± 0.10	mm
" " "	top .....20.6 ± 0.10	mm

The bottom of the ring shall be 2.5 ± 0.1 cm above the horizontal plate, which shall be approximately 2.5 cm above the bottom of the container.

A guide for centering the ball shall be used.

2. Preparation of Test Pieces : Heat the material slowly to the minimum temperature at which it can be readily poured; heat the rings to approximately the same temperature, place them on an amalgamated brass plate. Fill the rings carefully with the molten resin avoiding the inclusion of air bubbles, allow to cool to room temperature, cut off the excess material with a warm knife and then stand for a further period of 30 minutes.

3. Method: Fill the glass vessel, to a depth of not less than 9 cm with glycerine and heat to 32° C with constant agitation. Place the filled rings in the recesses of the upper brass plate and centre the balls in the rings. Suspend the assembly in the bath so that the tops of the rings are 5-6 cm below the surface of the glycerine. Hang the thermometer in the middle of the container so that the bulb is on a level with the rings but not touching them. Raise the temperature of the bath at the rate of 5°C per minute (the rate of temperature rise shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for each minute period, after the first three, shall not exceed ± .5°C otherwise tests shall be rejected). The temperature recorded at the instant the test piece touches the lower horizontal brass plate, shall be reported as the softening point.

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Calcium remnate content is approximately 16.2 oz per 1000 cc of solution.

Solution consist of	Batch size	% By weight
Calcium Pentate Precipitate	11 lbs 0 oz	33.00
CTC	17 8 lbs 0 oz	54.35
IPA	3 3 lbs 8 oz	12.65

I-136 co MC 505

	Amount in Batch size	% By weight
Calcium Pentate CTC + IPA solution	1480 cc	10
Strontium Peroxide	13 lb 8 oz	90

16 oz = 1 lbs

CaP	23 oz =	1.4375 lbs
SrO_2	13 lbs 0 oz	13.5 lbs
		14.9375