



**रक्षा मंत्रालय**  
**MINISTRY OF DEFENCE**

**संयुक्त सेवा स्पेसिफिकेशन**  
**JOINT SERVICES SPECIFICATION**

**ON**

**ALUMINIUM POWDER, HEAVY GRADE 1**

**मानकीकरण निदेशालय**  
**रक्षा उत्पादन विभाग**  
**रक्षा मंत्रालय**  
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**नई दिल्ली – 110 011**

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**DEPARTMENT OF DEFENCE PRODUCTION**  
**MINISTRY OF DEFENCE**  
**'H' BLOCK, NIRMAN BHAWAN PO**  
**NEW DELHI - 110 011**

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**RECORD OF AMENDMENTS**

Amendment		Amendment pertains to : Sl. No. / Para No. / Column No.	Authority	Amended by	Signature & Date
No.	Date			Name & Appointment (IN BLOCK LETTERS)	

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**0. FOREWORD**

0.1 This Joint Services Specification has been prepared by Armament Standardisation Sub Committee on the authority of the Standardisation Committee, Ministry of Defence.

0.2 This specification has been approved by the Ministry of Defence and is mandatory for use by the Defence Services.

0.3 (a) First revision was done in the year 1994.

(b) This Specification is a revision of JSS 1370 - 03 : 2007 (Revision No. 2) and supersedes the same.

0.4 This specification would be used for Manufacture, Supply and Quality Assurance of Aluminium Powder, Heavy Grade 1.

0.5 Quality Assurance Authority for the item covered by this specification is the Controller, Controllerate of Quality Assurance (Military Explosives), Aundh Road, Pune - 411 020. Enquiries regarding technical parameters shall be addressed to the Quality Assurance Authority, while other enquiries shall be referred to :-

The Director,  
Directorate of Standardisation,  
Ministry of Defence,  
'H' Block, Nirman Bhawan PO,  
New Delhi - 110 011.

0.6 Non registered users can obtain the following on payment :-

(a) **Copies of IS from :-**

Bureau of Indian standards,  
Manak Bhawan,  
9, Bahadur Shah Zafar Marg,  
New Delhi - 110 002.

or

Their regional / Branch offices.

(b) **Copies of JSS / JSG from :-**

The Director,  
Directorate of Standardisation  
Standardisation Documents Centre,  
Ministry of Defence  
Room No. 05, 'J' Block,  
Nirman Bhawan PO,  
New Delhi - 110 011.

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0.7 Indian Standard (IS) specifications are available free of cost for registered users on :-

Directorate of Standardisation Website

**[www.ddpdos.gov.in](http://www.ddpdos.gov.in)**

For registration visit our website.

0.8 This specification holds good only for the supply order for which it is issued.

0.9 **Directorate of Standardisation Website.** All the approved JSS / JSGs are available on the Directorate of Standardisation Website **[www.ddpdos.gov.in](http://www.ddpdos.gov.in)**. Defence Organisations desirous of accessing a copy of this document are requested to approach the Directorate of Standardisation for obtaining user id / password to access the website.

1. **SCOPE.** This specification is meant to govern manufacture, supply and quality assurance of Aluminium Powder, Heavy, Grade1 suitable for use in the manufacture of incendiary compositions, Aluminised explosives and pyrotechnic compositions.

2. **RELATED SPECIFICATIONS / DOCUMENTS**

2.1 Reference is made in this specification to :-

<b>Sl. No.</b>	<b>Specification No. &amp; Year</b>	<b>Nomenclature</b>
(a)	IS 138 : 1992 (Third Revision) Reaffirmed 2009, AMD 1	Ready Mixed Paint, Marking, for Packages and Petrol Containers - Specification.
(b)	IS 438 : 2006 (Second Revision) Reaffirmed 2011	Aluminium Powder for Explosives and Pyrotechnic Compositions - Specification.
(c)	IS 460 (Part 1) : 1985 (Third Revision) Reaffirmed 2008 AMD 1	Specification for Test Sieves : Part I Wire Cloth Test Sieves.
(d)	IS 2339 : 2013 (First Revision)	Specification for Aluminium Paint for General Purposes, in Dual Container.
(e)	JSG 0112 : 1997 (Revision No. 1)	General Methods of Test and Assessment of Impurities in Chemicals / Materials used in the Manufacture of Explosives and Ammunition.

3. **MATERIAL**

3.1 Aluminium Powder, Heavy, Grade 1 shall consist of powdered Aluminium or Aluminium alloy of the type known as 'blown'. It shall be in the form of compact, irregular and elongated particles and shall not be in the form of flakes. It shall be free from foreign matter and visible impurities.

3.2 The material shall conform to the appropriate sieving requirements as per clause 7.4 for a particular size.

4. **MANUFACTURE.** Aluminium Powder, Heavy, Grade 1 shall be manufactured by a process which has received authoritative approval. The Quality Assurance Officer / Quality Assurance Authority shall be informed regarding the process used and shall be given prior notification of any proposed deviations therefrom. All deviations from approved process, however slight, shall be recorded immediately and all material affected shall be set aside, pending decision of the Quality Assurance Authority.

5. **TENDER SAMPLE.** The contractor / supplier shall submit a tender sample of 250 g free of all charges and conforming to this specification to the Quality Assurance Authority / Quality Assurance Officer as stated in the contract.

6. **PRE - INSPECTION OF STORES / CONSIGNMENT**

6.1 Manufacturers / Contractors must satisfy themselves that the stores are in accordance with the terms of the contract and fully conform to the required specification, by carrying out a thorough pre-inspection of each lot before actually tendering the same for inspection to the Quality Assurance Officer nominated under the terms of the contract. A declaration by the contractor that a necessary pre - inspection has been carried out on the stores tendered will be submitted alongwith the challan. The declaration will also indicate the method followed in carrying out pre - inspection showing the features checked / tested and will have the test certificate attached to the challan / declaration.

6.2 If the Quality Assurance Officer finds that the pre - inspection of the consignment as required above has not been carried out, the consignment is liable for rejection.

7. **QUALITY ASSURANCE**

7.1 **Inspection.**

7.1.1 Aluminium Powder, Heavy, Grade 1 and the packages in which it is packed shall be subject to inspection by and to the approval of the Quality Assurance Officer / Quality Assurance Authority.

7.1.2 Samples of the material and of the packages may be taken from any portion of the batch / lot / consignment.

7.2 **Sampling.** Normally two representative samples each of 250 g shall be drawn from each batch / lot / consignment of supply / manufacture. However, the number of samples to be drawn shall be at the discretion of the Quality Assurance Officer / Quality Assurance Authority.

7.3 **Criteria for Conformity.** If on examination, any sample is found not to conform to this specification, the whole batch / lot / consignment may be rejected.



7.4 **Test Requirements.** Samples taken from any portion of the batch / lot / consignment of material shall conform to clause 3 and 4 and in addition, shall conform to the following test requirements :-

**Table : Test Requirements of Aluminium Powder, Heavy, Grade 1**

<b>Sl. No.</b>	<b>Characteristic</b>	<b>Passing Standard</b>	<b>Test Method</b>
(a)	Matter soluble in Ether, % by mass	Max. 0.25	Appendix 'A'
(b)	Volatile matter at 105 °C ± 2 °C, % by mass	Max. 0.05	1(a) of JSG 0112
(c)	Gritty matter, % by mass	Max. 0.05	6 of JSG 0112
(d)	Sieving requirement of gritty particles.		
	(i) Retained on 425 micrometre IS Sieve	Nil	18 of JSG 0112
	(ii) Number of particles retained on 250 micrometre IS Sieve, per 10 g sample	Max. 5	18 of JSG 0112
(e)	pH of water extract	Min. 5.5 Max. 7.5	5 (b) of JSG 0112
(f)	Silicon and compounds of Silicon (other than grit) calculated as Silicon, % by mass	Max. 0.5	Appendix 'B'
(g)	Copper and compounds of copper, calculated as metallic copper, % by mass	Max. 0.25	Appendix 'C'
(h)	Iron and compounds of Iron calculated as metallic Iron, % by mass	Max. 1.0	Appendix 'D'
(j)	Zinc and compounds of Zinc calculated as metallic zinc, % by mass	Max. 0.25	Appendix 'E'
(k)	Magnesium and compounds of Magnesium calculated as metallic Magnesium, % by mass	Max 0.2	Appendix 'F'

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<b>Sl. No.</b>	<b>Characteristic</b>	<b>Passing Standard</b>	<b>Test Method</b>
(l)	Metallic impurities other than Aluminium and metals referred above (Sl. Nos. f to k), % by mass	Max. 0.5	Appendix 'G'
(m)	Free metallic Aluminium, % by mass	Min. 98.0	Appendix 'H'
(n)	Apparent density		
	(i) Size 2 mm/425 micrometre	Min. 0.9 g/cm <sup>3</sup>	Appendix 'J'
	(ii) All other sizes	Min. 1.0 g/cm <sup>3</sup>	
(p)	Sieving requirements :-		
	1. Size 2 mm / 425 micrometre		
	(i) Retained on 2 mm IS Sieve	Nil	18 of JSG 0112
	(ii) Retained on 425 micrometre IS Sieve, % by mass	Min. 80	-do-
	2. Size 600/150 micrometre		
	(i) Retained on 600 micrometre IS Sieve	Nil	18 of JSG 0112
	(ii) Retained on 150 micrometre IS Sieve, % by mass	Min. 90	-do-
	3. Size 425/125 micrometre		
	(i) Retained on 600 micrometre IS Sieve	Nil	-do-
	(ii) Retained on 425 micrometre IS Sieve % by mass	Max. 10	-do-
	(iii) Retained on 125 micrometre IS Sieve % by mass	Min. 90	-do-

<b>Sl. No.</b>	<b>Characteristic</b>	<b>Passing Standard</b>	<b>Test Method</b>
	4. Size 425 micrometre		
	(i) Retained on 425 micrometre IS Sieve	Nil	-do-
	(ii) Retained on 125 micrometre IS Sieve, % by mass	Max. 60	-do-
	5. Size 250 micrometre		
	Retained on 250 micrometre IS Sieve	Nil	-do-
	6. Size 125 micrometre		
	Retained on 125 micrometre IS Sieve	Nil	-do-
	7. Size 125/63 micrometre		
	(i) Retained on 125 micrometre IS Sieve	Nil	18 of JSG 0112
	(ii) Retained on 63 micrometre IS Sieve % by mass	Min. 30 Max. 60	-do-
	8. Size 63 micrometre		
	Retained on 63 micrometre IS Sieve	Nil	-do-

**Note.** Retention of Powder up to one per cent may be permitted on the designated Sieve, wherever given as 'Nil' provided the retained material pass the next coarser Sieve specified in IS Sieves.

**8. WARRANTY.** The stores supplied against the contract shall be deemed to have been warranted against defective material and performance by the contractor / manufacturer for a period of 12 months from the date of receipt of the stores at the consignee's end and if during this period any of the stores supplied is found defective the same shall be replaced by the contractor / manufacturer free of charges at the consignee's premises.

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**9. PACKAGING**

9.1 The material shall be packed in sound, clean, dry, air - tight mild steel drums painted inside and outside with Aluminium paint as per IS 2339. The quantity to be packed will be as agreed to between the purchaser and the contractor.

9.2 Any other form of packages shall have the prior approval of the Quality Assurance Officer / Quality Assurance Authority.

9.3 The inclusion of any foreign matter or impurities in any of the packages shall render the whole batch / lot / consignment liable to rejection.

**10. MARKING**

10.1 All packages containing the material shall be indelibly and legibly marked with the following details :-

- (i) Nomenclature and Specification No. of the Material.
- (ii) Name and Address of the Consignee.
- (iii) A.T. or S.O. No. and Date.
- (iv) Consignment No.
- (v) Lot / Batch No. and Date of Manufacture.
- (vi) Gross and Net Mass.
- (vii) Consecutive No. of Package and Total Number of Packages in the Consignment.
- (viii) Date of Supply.
- (ix) Manufacturer's Name and Initial or his Recognized Trade Mark.

10.2 In addition to the above, the Quality Assurance Officer may suggest some more markings / identifications suitable at the time of inspection.

10.3 The paint used for marking should conform to IS 138 and to the satisfaction of the Quality Assurance Officer / Quality Assurance Authority.

**11. SAFETY OF OPERATIONS.** Nothing in this specification shall relieve the manufacturer / supplier / contractor / user of his responsibility for the safety of operations in manufacture, storage, transit or use of this store.

**12. DEFENCE STORES CATALOGUE NUMBER**

12.1 Defence Stores Catalogue Number allotted to this store is :-

<b>Sl. No.</b>	<b>Item / Store</b>	<b>DS Cat. No.</b>
(a)	Aluminium Powder, Heavy Grade 1 (Size 2mm)	1370 - 000 093
(b)	Aluminium Powder, Heavy Grade 1 Size 600 / 150 Micrometer	1370 - 000 271
(c)	Aluminium Powder, Heavy Grade 1 Size 425 / 125 Micrometer	1370 - 000 272
(d)	Aluminium Powder, Heavy Grade 1 Size 425 Micrometer	1370 - 000 273
(e)	Aluminium Powder, Heavy Grade 1 Size 250 Micrometer	1370 - 000 274
(f)	Aluminium Powder, Heavy Grade 1 Size 125 Micrometer	1370 - 000 275
(g)	Aluminium Powder, Heavy Grade 1 Size 125 / 63 Micrometer	1370 - 000 276
(h)	Aluminium Powder, Heavy Grade 1 Size 63 Micrometer	1370 - 000 277

**13. SUGGESTIONS FOR IMPROVEMENT**

13.1 Any suggestion for improvement in this document may be forwarded to :-

The Director,  
Directorate of Standardisation,  
Ministry of Defence,  
'H' Block, Nirman Bhawan PO,  
New Delhi - 110 011.

**A. DETERMINATION OF MATTER SOLUBLE IN ETHER**

**A.1 Procedure.**

A.1.1 Place 10 g of sample in a 250 ml conical flask (previously rinsed with Ether) with 100 ml of Ether and shake occasionally over a period of 1 hour. Filter by decantation into a tared flask, wash three times with successive portions of 20 ml hot Ether, combine the filtrates, distil off the Ether on a hot water bath, dry for 1 hour in the boiling water oven and weigh.

A.1.2 Carry out a blank determination on the Ether and correct the mass of dried residue accordingly.

A.1.3 Express the corrected mass of dried residue as a percentage on the mass of the sample taken.

**APPENDIX 'B'**

**B. DETERMINATION OF SILICON CONTENT**

**B.1 Procedure.**

B.1.1 Place about 2g of the sample accurately weighed in a 600 ml beaker and add 50 ml of distilled water. Then add 5 ml of concentrated Hydrochloric acid and warm the liquid until the reaction commences. When the first violent evolution of gas has subsided, add further portions of Hydrochloric acid up to a total of 25 ml. When all solid has dissolved, except for a little waxy matter, add Sulphuric acid (12 ml of concentrated Sulphuric acid diluted to 30 ml with distilled water) and evaporate the liquid on sand tray or hot plate until copious fumes are evolved. Cool the residue, dilute with 200 ml of distilled water and warm with occasional stirring until the white solid has dissolved. Allow the contents of the beaker to stand overnight. Filter off the insoluble residue next morning on a No. 42 Whatman filter paper. Wash with 2 percent Sulphuric acid solution several times (Retain the filtrate for the determination of Copper, Iron and Zinc contents). Extract the residue insoluble in Sulphuric acid in a 100 ml beaker with 20 ml of hot 20 per cent Ammonium acetate solution. After 30 minutes extraction, filter the liquid through a No. 41 Whatman filter paper, wash the residue once with 20 per cent Ammonium acetate solution, dry, ignite in a porcelain crucible and weigh (M1). Warm the ash with 10 ml of Aqua - regia for 5 minutes, dilute the solution and filter it through a 9 cm, No. 40 Whatman filter paper. Wash the residue with distilled water, dry and ignite gently. Transfer the ash into a 150 ml Monax beaker, add 20 ml of 50 per cent Sodium hydroxide solution and boil gently for exactly 10 minutes. Wash out the solution and the residue into a tall 400 ml beaker containing about 200 ml of distilled water. Allow the solid matter to settle for 15 minutes, syphon or decant off the clear liquid and add a further 200 ml of distilled water. Repeat the washing by decantation once more and then filter off the residue on a 9 cm, No. 40 Whatman filter paper. Wash the residue with hot water and finally with a 2 % solution of Hydrochloric acid. Dry the residue, ignite in a porcelain crucible and weigh the ash (M2). The loss in mass (M1- M2) is due to silica derived from elementary Silicon originally present as such. Calculate the Silicon present from the mass of silica and express it as a percentage on the mass of the sample taken.

**B.1.2 Calculation.**

$$\begin{array}{l} \text{Silicon Content,} \\ \text{\% by Mass} \end{array} = \frac{(M1 - M2) \times 0.467 \times 100}{\text{Mass of the sample taken}}$$

**C. DETERMINATION OF COPPER CONTENT**

**C.1 Procedure.**

C.1.1 Saturate the filtrate reserved for Copper, Iron and Zinc determination (See Appendix 'B' for Silicon content) with Hydrogen sulphide for 20 minutes. Filter off any precipitate on a 9 cm, No. 40 Whatman filter paper, and wash with Hydrogen sulphide water (Retain the filtrate for further determination of Iron and Zinc contents). Dry the residue and ignite in a tared crucible. Cool in a desiccator and weigh the residue of cupric oxide. Calculate the mass of metallic Copper contained in the cupric oxide so obtained and express it as a percentage on the mass of the original sample taken.

**C.1.2 Calculation.**

$$\text{Copper Content, \% by Mass} = \frac{\text{Mass of CuO} \times 0.80 \times 100}{\text{Mass of the sample taken}}$$

C.2 Confirm the presence of Copper by dissolving the residue in Nitric acid and making alkaline with Ammonia, when the characteristic blue colouration should be observed.



**APPENDIX 'D'**

**D. DETERMINATION OF IRON CONTENT**

**D.1 Reagents.**

- (a) Stannous chloride solution - 6 g stannous chloride and 6 ml of concentrated Hydrochloric acid diluted to 1 litre.
- (b) Mercuric chloride solution - Saturated.
- (c) Indicator - Diphenylamine dissolved in concentrated Sulphuric acid to give a 1 % solution.

**D.2 Procedure.**

D.2.1 Boil off the Hydrogen sulphide from the filtrate (in a 600 ml beaker) obtained after precipitating Copper sulphide (See Appendix 'C' for copper content). Then add 3 ml of concentrated Nitric acid and boil for about 2 minutes. Allow the liquid to cool a little and then dilute the solution to about 300 ml. While the solution is still hot, cautiously add 30 per cent caustic soda solution with vigorous stirring until all the Aluminium hydroxide first precipitated is just re - dissolved (about 60 ml is required). Keep on the water bath until the brown precipitate of Ferric hydroxide has settled and filter off on a 9 cm, No. 54 Whatman filter paper (Reserve the filtrate for determination of Zinc content). Wash the precipitate once with hot distilled water and dissolve it in about 30 ml of warm Hydrochloric acid (1:1). Heat the solution to the boiling point and add stannous chloride solution drop by drop until the yellow colour due to Ferric ions just disappears. After cooling to room temperature, add 5 ml of Mercuric chloride solution in order to oxidise the slight excess of stannous chloride, allow the liquid to stand a few minutes, add 2 or 3 drops of Diphenyl - amine indicator and titrate with N/10 Potassium dichromate solution. The end point is indicated by the appearance of a deep blue colour. Carry out a blank titration using similar quantities of all reagents used and apply the necessary correction to the original titration. Calculate the Iron equivalent from the volume of Potassium dichromate used and express it as a percentage on the mass of the original sample taken.

**D.2.2 Calculation.**

If the corrected titre value is V ml and the factor of the N/10 Potassium dichromate solution is f, then

$$\text{Iron Content, \% by Mass} = \frac{V \times f \times 0.0056 \times 100}{\text{Mass of the sample taken}}$$

**E. DETERMINATION OF ZINC CONTENT**

**E.1 Procedure.**

E.1.1 Pass Hydrogen sulphide slowly for 15 minutes into the filtrate from the Ferric hydroxide precipitate (See Appendix 'D' for Iron content). Set the liquid aside for two hours in order to allow any precipitate to settle. Filter off any precipitate on a 9 cm, No. 54 Whatman filter paper and wash once with Hydrogen sulphide water. Dissolve the precipitate in 20 ml of warm Hydrochloric acid (1:1) and wash with distilled water into a 150 ml beaker. Boil the solution gently and reprecipitate by addition of Ammonium chloride and dilute Ammonia to the solution until it is just alkaline to litmus paper, boil for a minute or two and filter off the precipitate of Aluminium hydroxide on a small, No. 41 Whatman filter paper. After washing twice with hot distilled water discard the residue and make the filtrate just acidic with Acetic acid. Saturate the solution with Hydrogen sulphide, allow the precipitate to settle and filter it off on a 9 cm, No. 40 Whatman Filter paper. Wash the precipitate once with Hydrogen sulphide water, moisten the paper with a few drops of saturated Ammonium nitrate solution, dry, ignite, cool in a desiccator and weigh. The colour of the ash - yellow while hot, confirms the presence of Zinc. Calculate the zinc equivalent from the mass of the ignited precipitate and express it as a percentage on the mass of the original sample taken.

**E.1.2 Calculation.**

$$\begin{array}{l} \text{Zinc Content,} \\ \text{\% by Mass} \end{array} = \frac{\text{Mass of ZnO} \times 0.80 \times 100}{\text{Mass of the sample taken}}$$

**APPENDIX 'F'**

**F. DETERMINATION OF MAGNESIUM CONTENT**

**F.1 Reagents.**

(a)	Bromate - bromide solution	0.458 g Potassium bromate, 4 g Potassium bromide and 0.1 g Potassium Hydroxide per litre (1 ml = 0.05 g Magnesium)
(b)	Sodium thiosulphate	4.10 g Sodium thiosulphate per litre
(c)	Sodium Tartrate	20 % solution
(d)	Potassium cyanide	30 % solution
(e)	8 - Hydroxy quinoline (oxine)	2 % solution in alcohol
(f)	Potassium iodide	10 % m/v solution
(g)	Indigo Carmine	1 % solution

**F.2 Procedure.**

F.2.1 Treat about 2g of the sample accurately weighed with 40 ml of 20 per cent Sodium hydroxide solution, dilute to 150 ml and boil. Filter off the residue on a prepared Gooch crucible and wash with hot water containing 0.2 % of Sodium hydroxide. Extract the residue with 15 ml hot dilute (1:1) aqua regia and wash with hot water. Boil the extract for about 1 minute, cool and make up to 100 ml in a standard flask. Pipette 10 ml into a 150 ml beaker, add 5 ml of water, 5 ml of 20 per cent Sodium tartrate solution and 1 drop of Phenolphthalein (1 %). Neutralise with 2N Sodium hydroxide and add 2 ml in excess. Add 2 ml of 30 per cent Potassium cyanide solution and 2 ml of 2 per cent alcoholic oxine solution. Stir to induce precipitation. Allow to stand for 10 minutes and again stir for 1 minute. Heat to 60 °C and cool. Filter off the precipitate with the aid of suction on a G - 3 sintered glass crucible with an asbestos pad and wash with 120 ml of a solution composed of 500 ml of alcohol, 430 ml of distilled water, 50 ml of 20 % Sodium tartrate solution and 20 ml of 2N Sodium hydroxide, in 10 portions. Extract the precipitate with 20 ml of hot 1:1 Hydrochloric acid and 30 ml of hot distilled water, receiving the extract in a conical flask. Cool and add 3 to 4 drops of indigo - carmine solution. Titrate slowly with Bromate - bromide solution until the liquid is golden yellow, free from any green tinge, adding a further 2 drops of indigo - carmine solution just before the endpoint if necessary, and then add a further 2 ml of Bromate - bromide solution in order to provide excess. Note the total quantity added (V1 ml). Without delay, add 10 ml of 10 % Potassium iodide solution and a few drops of freshly prepared starch solution and titrate the liberated Iodine with N/10 Sodium thiosulphate solution (factor f) until the blue colour is just discharged. Note the quantity of N/10 Sodium thiosulphate used (V2 ml).

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F.2.2 **Calculation.**

$$\begin{array}{l} \text{Magnesium Content,} \\ \text{\% by Mass} \end{array} = \frac{(V_1 - V_2) \times f \times 0.00005 \times 1000}{\text{Mass of the sample taken}}$$

F.3 Carry out a control determination on another 10 ml portion of solution to which has been added 2 ml of a solution containing 2.05 g of Magnesium sulphate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) per litre. This will be equivalent to the addition of 0.2 % Magnesium to the sample.

F.4 The difference between the two percentages of Magnesium thus obtained should be exactly equal to the added percentage of Magnesium.

F.5 **Alternate Method - I.**

**Sub : Determination of Magnesium as an impurity present in Aluminium powder by using Atomic Absorption Spectrophotometer.**

Instrumental Conditions :

Photocathodo lamps	-	Mg
Wavelength	-	285.2 nm
Flame	-	Acetylene - Air Flame
Working range	-	0.1 to 0.5 ppm

**Preparation of Sample Solution.**

Weigh 500 mg of sample in a glass beaker and add 100 ml of HCl solution (1 : 1). Boil it for 15 - 20 min. Confirm that the sample has been completely dissolved in HCl solution. Cool it and dilute to 250 ml in volumetric flask.

**Preparation of Standard Solution.**

Prepare 0.1 to 0.5 ppm solution of Magnesium from 1000 ppm standard solution (MERCK)

**Analytical Procedure.**

First introduce the standard solutions to the air acetylene flame one by one and measure the absorbance. Then introduce the sample solution to the flame and measure the absorbance.

Plot the graph of absorbance versus concentration. Calculate the concentration of Magnesium present in the sample from the graph.

**APPENDIX 'G'**

**G. DETERMINATION OF OTHER METALLIC IMPURITIES**

**G.1 Procedure.**

G.1.1 Place about 2g of the sample accurately weighed in a 600 ml beaker, cover with 50 ml of warm distilled water and add Hydrochloric acid gradually until the metal has dissolved (10 ml of acid is usually sufficient). Add 2 ml of Nitric acid to oxidise iron, evaporate the solution to dryness and remove Nitric acid by a further evaporation after addition of 5 ml of Hydrochloric acid. Heat the solid residue at 120 °C for an hour, cool and dissolve in 10 ml of Hydrochloric acid and dilute with 300 ml of water. Add 5g of Ammonium chloride, boil the solution and add dilute Ammonia in slight excess. Boil for a further 5 minutes after which filter off the precipitated Aluminium and Iron hydroxide on a 12.5 cm, No. 41 Whatman filter paper. Wash the precipitate with distilled hot water containing 2 percent of Ammonium chloride. Evaporate the filtrate and washings to about 50 ml in a beaker, make just acidic with Hydrochloric acid, boil for a few minutes, and then make just alkaline with Ammonia in order to precipitate any Aluminium remaining. Filter through a 9 cm, No. 41 Whatman filter paper, evaporate the filtrate to dryness in a beaker, transfer the solid mechanically to a tared Platinum crucible using moist filter paper to transfer the last traces of solid. Burn the paper and volatilise the solid Ammonium Chloride by gentle heating. Sulphate the residue with two drops of Sulphuric acid, ignite at red heat, cool and weigh. Correct this mass for the amount of residue found in the blank determination and for the amounts of Zinc and Copper originally found in the sample (Assure these are present as Copper oxide and Zinc Oxide). Discard if remainder after corrections for Zinc, Copper and blank is not greater than 10 milligrams. Express the residue of Sodium sulphate as percentage on the mass of the sample taken.

**G.1.2 Calculation.**

$$\text{Other Metallic Impurities as Sodium, \% by Mass} = \frac{\text{Mass of the Residue} \times 0.3234 \times 100}{\text{Mass of the sample taken}}$$

**H. DETERMINATION OF METALLIC ALUMINIUM CONTENT**

H.1 Weigh 0.1 g of Aluminium powder into a small glass tube of convenient length closed at one end. Introduce the tube containing the powder into the decomposition vessel which may be small 257 ml (2 ounce) bottle. Add 20 ml of 10 per cent Sodium hydroxide solution into the vessel and connect it to the Nitrometer with due regard for air - tightness. Make sure that the air in decomposition vessel is initially at atmospheric pressure. Expel all air completely from Nitrometer by bringing the acidulated water level coloured with a drop of Methyl red. Surround the decomposition vessel with water. Note the temperature of the water jacket around the Nitrometer and the waterbath around the decomposition vessel. It should almost be the same. Tilt the decomposition vessel in such a way that the Sodium hydroxide finds its way into the tube containing the Aluminium powder, at the same time opening the passage to the Nitrometer. When the reaction ceases, adjust the pressure very nearly to the atmospheric pressure and allow it to stand for one hour. Add a drop or two of Methyl red in the cup of the Nitrometer to serve as an index for the pressure of the gas in the Nitrometer. Find out the position by trial at which the liquid in the Nitrometer cup does not move up or down the bore of the stopper when the Nitrometer is opened to the atmosphere. This operation is to be repeated after putting the gas in the Nitrometer in communication with that in the decomposition vessel to ensure that the air shut off in the latter is at atmospheric pressure. Read the volume of the gas. Read the temperature of the water at jacket and the water bath of the decomposition vessel. Note the barometric height and the temperature.

**Note.**

- (i) Correct the volume of Hydrogen for the volume of gas produced by metallic Zinc only. (This correction may be ignored if metallic Zinc present in the sample is less than 0.1 per cent.
- (ii) 100 g of pure metallic Aluminium will produce 124580 ml of Hydrogen measured at N.T.P.

Calculate the percentage metallic Aluminium content, on the grease - free material, as follows :-

Let  $M$  = the mass of the sample used.

$v$  = the volume in ml of the Hydrogen produced corrected as regards calibration of the Nitrometer.

$t_0$  = the temperature in degree Celsius at the commencement of the evolution.

$t$  = the temperature in degree Celsius at the instant of measurement of the volume of Hydrogen produced.

$p$  = the vapor pressure in mm of 10 % NaOH solution at  $t$  °C.

$P$  = the corrected atmospheric pressure in mm at the instant of measurement of the volume of Hydrogen produced.

$G$  = the percentage of grease.

(i) Correct the volume of Hydrogen,  $v$  for change of temperature ( $t_0 - t$ ). Let  $V$  be the corrected volume, then assuming the volume of air originally present in the apparatus is approximately 50 ml.

$$V = v + (t_0 - t) \times 0.2 \text{ ml}$$

(ii) Calculate the volume  $V_{100}$  of Hydrogen at N.T.P. liberated by 100 g of the sample.

$$V_{100} = \frac{V(P - p) \times 273 \times 100 \text{ ml}}{(273 + t) \times 760 \times M}$$

(iii) Correct this volume for the volume of Hydrogen produced by the Zinc present in 100 g of the sample.

(iv) The volume of Hydrogen produced by the metallic Aluminium in the sample is then  $V_2$ .

(v) Calculate the percentage metallic Aluminium present in the sample using the theoretical volume of hydrogen produced by 100 g of pure Aluminium :-

$$Al = \frac{V_2 \times 100}{124580}$$

(vi) Express this as a percentage of grease free material.

$$\text{Percentage of Al} = \frac{[v + 0.2(t_0 - t)](P - p) \times 273 \times 100 \times 100 \times 100}{(273 + t) \times 760 \times M \times 124580 \times (100 - G)}$$

Where  $G$  is the percentage of grease present.

**J. DETERMINATION OF APPARENT DENSITY**

J.1 20 g of the material are introduced into a stoppered glass cylinder, graduated in half milliliter, the cylinder being approximately 15 cm (6 inches) high and having an internal diameter of 2 cm (0.8 inches). The cylinder is dropped vertically 30 times from a height of 6.5 cm (2.5 inches) on to a hard leather pad. The surface of the column of Aluminium powder is leveled by the minimum amount of side tapping, and the total volume is read.

J.2 The above procedure is conveniently carried out by sliding through two wooden filter stand rings clamped one above the other on the same support, the lower being so placed as to limit the travel of the cylinder to 6.5 cm.

$$\text{Apparent Density} = \frac{\text{Mass g/cm}^3}{\text{Volume}}$$