NATO STANDARD

AOP-4300

ENERGETIC MATERIALS, TEST PROCEDURES FOR ASSESSING THE QUALITY OF ALUMINIUM POWDER FOR USE IN ENERGETIC MATERIAL FORMULATIONS

EDITION A VERSION 1
MAY 2018

NORTH ATLANTIC TREATY ORGANIZATION

ALLIED ORDNANCE PUBLICATION

Published by the
NATO STANDARDIZATION OFFICE (NSO)

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NORTH ATLANTIC TREATY ORGANIZATION (NATO)

NATO STANDARDIZATION OFFICE (NSO)

NATO LETTER OF PROMULGATION

24 May 2018

1. The enclosed Allied Ordnance Publication AOP-4300, Edition A, Version 1, ENERGETIC MATERIALS, TEST PROCEDURES FOR ASSESSING THE QUALITY OF ALUMINIUM POWDER FOR USE IN ENERGETIC MATERIAL FORMULATIONS, which has been approved by the nations in the CNAD Ammunition Safety Group AC/326, is promulgated herewith. The agreement of nations to use this publication is recorded in STANAG 4300.

2. AOP-4300, Edition A, Version 1, is effective upon receipt.

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Zoltán GULYÁS
Brigadier General, HUNAF
Director, NATO Standardization Office

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RECORD OF SPECIFIC RESERVATIONS

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CHAPTER 1 INTRODUCTION

1.1. AIM

The aim of this agreement is to standardize test methods for assessing the quality of aluminium powder used in the manufacture of energetic material formulations (i.e., explosives, propellants and pyrotechnics). The use of these methods should facilitate cross procurement and provide means by which nations can satisfy themselves that aluminium received from abroad has been tested by acceptable means. These methods are not intended for use as quality control purposes during the manufacture of aluminium powder.

1.2. AGREEMENT

Participating nations agree to use the test procedures described for assessing the quality of aluminium powder when required by the procuring nation.

1.3. WARNING

This agreement calls for the use of substances and test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves the user from the statutory obligations relating to health and safety at any stage during use.

1.4. RELATED DOCUMENTS

1. ASTM B 330, Standard Test Method for Fisher Number of Metal Powders and Related Compounds, 2007.

2. British Standard Institute BS 4359: Part 2, Determination of the specific surface area of powders: Recommended air permeability methods, 1985.

3. ASTM B 214, Standard Test Method for Sieve Analysis of Metal Powders, 2007.

4. ASTM E 11, Standard Specification for Wire Cloth and Sieves for Testing Purposes, 2004.

5. ASTM D 480, Standard Test Methods for Sampling and Testing of Flaked Aluminum Powders and Pastes, 1988 (reapproved in 2003).

6. International Standard ISO 3923/1, Metallic Powders – Determination of Apparent Density – Part 1: Funnel Method, 2008.

7. ASTM B 417, Standard Test Method for Apparent Density of Non-Free-Flowing Metal Powders Using the Carney Funnel, 2000 (reapproved in 2006).

8. International Standard ISO 3923/2, Metallic Powders – Determination of Apparent Density – Part 2: Scott Volumeter Method, 1981.

9. ASTM B 329, Standard Test Method for Apparent Density of Metal Powders and Compounds Using the Scott Volumeter, 2006.

10. International Standard ISO 3953, Metallic Powders – Determination of Tap Density, 2011.

11. AOP-4682, Edition A Version 1, Energetic Materials, Test Methods for Ingredients.

12. ASTM E 101, Test Method for Spectrographic Analysis of Aluminum and Aluminum Alloys by the Point-To-Plane Technique, 1991.

13. ASTM E 34, Standard Test Methods for Chemical Analysis of Aluminum and Aluminum-Base Alloys, 1994 (reapproved in 2002).

ANNEX A PHYSICAL AND CHEMICAL PROPERTIES

A.1. DESCRIPTION OF ALUMINIUM

The aluminium powder used in the manufacture of energetic material formulations (i.e., explosives, propellants and pyrotechnics) for military purposes is prepared by atomization, stamping or grinding of aluminium metal. Atomization produces a spheroidal or elongated particle, stamping yields a particle described as "flake" and grinding yields irregularly shaped, elongated particles.

A.2. TESTS TO BE APPLIED

The tests to be applied will depend upon the type and the specification of the aluminium powder being procured. The end use of the material determines the specification of its physical and chemical properties. Because of the great variety of aluminium powders available, this document does not seek to identify various grades and types. Specifications for properties such as particle shape, size and size distribution, chemical purity, etc. shall be made in the purchaser's order.

A.3. LIST OF PHYSICAL TESTS

1. Visual examination
2. Sieving
3. Average particle size
4. Particle size distribution
5. Apparent density
6. Tap density

A.4. LIST OF CHEMICAL TESTS

1. Volatile matter
2. Matter soluble in diethyl ether
3. pH of aqueous extract
4. Matter insoluble in hydrochloric acid
5. Gritty particles
6. Free metallic aluminium
7. Elemental impurities

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ANNEX B TEST PROCEDURES

B.1. VISUAL EXAMINATION

A sample of the aluminium powder will be examined under a microscope of suitable magnification to permit clear viewing of individual particles. Atomized particles shall have an approximately spherical shape. Flake particles shall consist of irregular, flattened particles with frayed, irregular contours. Ground particles shall consist of irregular, elongated particles.

B.2. SIEVING

The sieve test shall be conducted using a Tyler Ro-Tap testing sieve shaker and US standard sieves (RR-S-366) or equivalent. The stack requires sieves with the coarsest sieve on top and the finest sieve at the bottom. Place a catch pan at the bottom of the stack. Weigh to the nearest 0.02 g a 50-g sample and brush onto the uppermost sieve. Place a sieve cover on top of the stack and lock stack on the sieve shaker. Shake for 15 minutes. Carefully brush material passing the bottom sieve onto a weighing pan and weigh to the nearest 0.01 g. Add material from the next higher sieve and weigh. Continue until all the material has been weighed. Multiply each weight by 2 to obtain weight cumulative percent passing at each sieve level. Repeat analysis if total recovered weight is less than 99 percent. Sieving flake aluminium can sometimes cause problem. In such cases, wet the sample with white spirit (also known as mineral spirits or petroleum spirits) and wash the material through the sieve using a jet of this liquid. Wash the material retained in the sieve with acetone, dry and weigh.

B.3. AVERAGE PARTICLE SIZE

B.3.1. General

The average particle size shall be determined using the Fisher sub-sieve sizer procedure described hereafter. ASTM B 330 (“Standard Test Method for Fisher Number of Metal Powders and Related Compounds”) may be used as an acceptable equivalent procedure. Another acceptable alternate procedure is described in British Standard Institute BS 4359: Part 2 (“Determination of the specific surface area of powders: Recommended air permeability methods”).

B.3.2. Checking of apparatus

Check water level in standpipe of pressure regulator. Adjust water level as required. Check drying agent in rear of cabinet. Remove and dry in oven if indicator is pink. Check sample packing assembly to see that the pointer tip coincides with the baseline on the calculator chart. Check calibration with Fisher sub-sieve sizer calibrator. If calibration is off, adjust flowmeter wires according to calibration instruction.

B.3.3. Procedure

Attach porous plug to plug manipulator. Center a filter paper disc over end of sample tube and push plug into sample tube with perforated surface against filter paper disc. Remove plug manipulator and place sample tube in vertical position on metal post of support stand to force plug to proper height inside sample tube. Weigh to nearest 0.01 g a sample of powder equal in grams to the true density of sample (2.7 g). With a funnel, transfer weighed sample to sample tube. Tap side of tube to settle powder. Center another filter paper disc over the top of the tube and force a second plug (perforated surface against paper disc) into tube. Push plug and disc downward until powder is compacted against lower plug and disc. Place sample tube on post under rack and pinion with lower plug in contact with post. Lower rack until upper plug is in contact with bottom of the rack. Turn pinion knob by applying standard torques until sample is packed to optimum porosity. Shift calculator chart laterally until tip of pointer coincides with the sample height curve. Place sample tube (without disturbing sample) between rubber cushion supports. Clamp upper tube down until airtight seal is obtained at both ends. Turn on switch. When liquid level in manometer has reached maximum height, turn pinion knob until bar on rack is at bottom of meniscus in manometer. Read particle size at tip of pointer. Fine powders may require 5 minutes or more running time before maximum height is reached. If the average particle size is 0.2 to 20 microns, read chart directly. If the average particle size is between 20 and 50 microns, turn range control indicator to left and double chart readings.

B.4. PARTICLE SIZE DISTRIBUTION

B.4.1. General

The size distribution of spheroidal particles shall be determined using a Malvern particle size analyzer as described hereafter. Other brands of particle size analyzers based on the phenomenon of low-angle forward-scattering light from a laser beam, such as Microtrac, are acceptable. Particle size distribution may also be determined by applying ASTM B 214 ("Standard Test Method for Sieve Analysis of Metal Powders"). Specifications for sieves are reported in ASTM E 11 ("Standard Specification for Wire Cloth and Sieves for Testing Purposes"). ASTM D 480 ("Standard Test Methods for Sampling and Testing of Flaked Aluminum Powders and Pastes") describes methods to determine the percent of coarse particles retained on a sieve by wet-screening techniques.

B.4.2. Procedure

The Malvern particle size analyzer shall be mounted with a suitable focal lens and a small volume (15 mL) non-recirculating cell. Install the non-recirculating cell against the collecting lens of the Malvern particle size analyzer and fill it with isopropanol. Prepare a light dispersion of aluminium powder (2-5 g) in mechanically stirred isopropanol (about 50 mL) contained in a 150-mL beaker sitting in an ultrasonic bath. Do a background reading of the cell. Using a dropper, rapidly add a few drops of the dispersion into the cell until the obscuration is between 0.1 and 0.4 and then do the measurement. Selecting the model independent analysis package on the Malvern instrument, determine particle size distribution and average particle size on a volume basis.

B.5. APPARENT DENSITY

B.5.1. Funnel method

The apparent density of powder that flows freely through a 5-mm orifice shall be determined in accordance with ISO 3923/1 ("Metallic Powders – Determination of Apparent Density – Part 1: Funnel Method"). ASTM B 417 ("Standard Test Method for Apparent Density of Non-Free-Flowing Metal Powders Using the Carney Funnel") is an acceptable equivalent procedure.

B.5.2. Scott volumeter method

The apparent density of powder that does not flow freely through a 5-mm orifice shall be determined in accordance with ISO 3923/2 ("Metallic Powders – Determination of Apparent Density – Part 2: Scott Volumeter Method"). ASTM B 329 ("Standard Test

Method for Apparent Density of Metal Powders and Compounds Using the Scott Volumeter") is an acceptable equivalent procedure.

B.6. TAP DENSITY

B.6.1. General

The tap density shall be determined using calibrated rubber-stoppered measuring cylinders either of 25 mL in capacity and approximately 15 cm in length or of 100 mL in capacity and approximately 25 cm in length. The upper surface of a wooden base stand is covered with hard leather. A guide is provided by placing two wooden filter rings, clamped one above the other, on the same support. The lower ring is positioned to limit the travel of the cylinder to 6.5 cm. ISO 3953 ("Metallic Powders – Determination of Tap Density") is an acceptable equivalent technique.

B.6.2. Procedure for blown grades

Transfer 20 ± 0.1 g of the sample to the 25-mL cylinder and close with the rubber stopper. Place the cylinder into the wooden stand and drop it vertically 30 times from a height of 6.5 cm on to the leather pad. Level off the surface of the powder by the minimum amount of side-tapping and read off the volume (V_1) occupied by the powder.

B.6.3. Procedure for flake grades

Transfer 15 ± 0.1 g of the sample to the 100-mL cylinder and add 60 mL of 92% v/v methylated spirits (also known as denatured alcohol). Stopper cylinder and shake for about 5 minutes. Wash down any material adhering to the walls of the cylinder with a jet of methylated spirits and allow cylinder to stand in a vertical position for 1 hour. Read off volume (V_2) occupied by the aluminium powder.

B.6.4. Calculation

$$\text{Apparent density for blown grades (g/mL)} = \frac{20}{V_1}$$

$$\text{Apparent density for flake grades (g/mL)} = \frac{15}{V_2}$$

B.7. VOLATILE MATTER

Heat a weighing bottle in a drying oven at about 105°C for 2 hours. Cool in a desiccator for 30 minutes with cover off. Weigh bottle and cover to the nearest 0.1 mg. Record as W1. Add approximately 5 g of the sample to the weighing bottle and weigh to the nearest 0.1 mg with cover in place. Record as W2. Remove cover and place bottle and cover in drying oven or preferably in dry nitrogen stream at 105°C for 3 hours. Remove bottle and cover from oven and allow to cool in desiccator for 30 minutes. Place cover on bottle and reweigh to the nearest 0.1 mg. Record as W3. Calculate the following quantity:

$$\text{Volatiles (\%)} = \frac{(W2 - W3) \times 100}{W2 - W1}$$

B.8. MATTER SOLUBLE IN DIETHYL ETHER

Dry a clean 400-mL flask in an oven at 90°C and cool in a desiccator to constant weight. Weigh to the nearest 0.1 mg and record as W1. Weigh about 10 g of the sample to the nearest 0.1 mg and extract for 4 hours with 100 mL of diethyl ether using a Soxhlet extraction apparatus with a Whatman single thickness, fat-free extraction thimble or equivalent. After extraction has been completed, evaporate the diethyl ether almost to dryness using a boiling water bath. Dry residue in an oven at 90°C to a constant weight and then cool in a desiccator. Weigh and record this as W2. Run a blank with each group of the tests and record this as W3. Calculate the following quantity:

$$\text{Soluble matter (\%)} = \frac{((W2 - W1) - W3) \times 100}{\text{Weight of sample}}$$

Note: As diethyl ether is flammable, appropriate safety precautions against accidental ignition must be taken. Furthermore, peroxides contained in ether can constitute an explosive hazard.

B.9. PH OF AQUEOUS EXTRACT

The determination of pH shall be done using any standard pH meter accurate to 0.05 pH unit and calibrated using suitable buffer solutions. Transfer 5 ± 0.1 g of the sample to a 250-mL beaker, add 50 mL of water and stir for 2 minutes. Allow powder to settle for about 1 minute and determine the pH of the aqueous extract.

B.10. MATTER INSOLUBLE IN HYDROCHLORIC ACID

Transfer 10 ± 0.1 g of the sample to a 600-mL beaker and add 50 mL of water. Add hydrochloric acid (d = 1.18 g/mL) dropwise, controlling the reaction by the rate of addition of the acid until the sample dissolves. Heat the beaker gently for 10 minutes. Dilute with water, allow any solid matter to settle and decant the clear liquid. Wash the residue with water and repeat the decantation. Transfer the residue to a 400-mL beaker, add 20 mL of 50% sodium hydroxide solution and boil gently for 10 minutes. Dilute the solution immediately with 200 mL of water, allow the residue to settle and carefully decant the supernatant liquid. Repeat the washing twice with water and finally with 2% hydrochloric acid solution. Filter any residue in the beaker through a No. 40 Whatman filter paper or equivalent and wash well with hot water. Dry the filter paper and ignite it in a tared (W1) ignited porcelain crucible at a temperature of 800°C for 30 minutes. Cool in a desiccator for 30 minutes and reweigh (W2). Retain the residue and crucible for the determination of gritty particles as described in Test Procedure B.11. Calculate the following quantity:

Total insoluble matter (%) = (W2 - W1) x 10

B.11. GRITTY PARTICLES

Prepare aqua regia by cautiously mixing one volume of nitric acid (d = 1.42 g/mL) with three volume of hydrochloric acid (d = 1.18 g/mL). Transfer the residue from Test Procedure B.10 to a 100-mL beaker, add 5 mL of aqua regia and boil gently for 5 minutes. Allow to cool. Dilute the contents of the beaker with water and neutralize with sodium hydroxide solution. Pour the liquid through a 63-µm sieve or equivalent, ensuring all the residue is transferred to the sieve. Wash the residue thoroughly with water and dry the sieve in an oven at 103 ± 2°C. Gently brush the material retained on the sieve with a small soft brush. Transfer any residue retained on the sieve to a tared watch glass (W1) and weigh (W2). Transfer the residue to the 250-µm sieve or an equivalent and again gently brush with a soft brush. Transfer any residue retained to a tared watch glass (W3) and weigh (W4). Calculate the following quantities:

Gritty particles retained on the 63-µm sieve (%) = (W2 - W1) x 10

Gritty particles retained on the 250-µm sieve (%) = (W4 - W3) x 10

B.12. FREE METALLIC ALUMINIUM

B.12.1. General

The free metallic aluminium shall be determined by measuring the volume of gas evolved when a weighed quantity of aluminium powder is reacted with a sodium hydroxide solution as per the following equation:

$2\ Al + 2\ NaOH + 2\ H_2O \rightarrow 2\ NaAl_2 + 3\ H_2 \uparrow$

A method equivalent to the procedure described hereafter will be acceptable. This procedure also appears in AOP-4682 (“Energetic Materials, Test Methods for Ingredients”).

B.12.2. Apparatus

The basic apparatus is shown in Figure 1. It is advisable to house the apparatus in a separate room, free from draughts and away from direct sunlight and source of heat.

B.12.3. Reagent

Sodium hydroxide solution, 20% m/v.

B.12.4. Calibration of receiver

Liquids may be introduced into the receiver (R) by attaching a tube to the outlet of tap S2 which dips into a vessel containing the required liquid, turning taps S1 and S2 to the appropriate positions and applying suction to the open arm of tap S1. Thoroughly clean the receiver (R) with chromosulfuric (chromic) acid and rinse well with water. Fill the 1-L bottle (B) about three-quarters full with water. Ensure that the open arm of tap S2 is free from liquid by applying gentle pressure to the open arm of tap S1 to blow out any trapped liquid. Turn tap S2 to connect the receiver (R) with the bottle (B) and apply gentle pressure (using the manometer (M) as guide) via tap S3 to force water into the receiver (R) until it is filled to the mark (A). Check that no air bubbles are trapped on the walls of the receiver (R). Close tap S2 and release the pressure on tap S3. Open tap S2 to the exit arm and run out the volume of water, contained in the receiver (R), into a suitable tared receiver (W1 ± 10 mg) and weigh (W2 ± 10 mg). Note the temperature of the water (T_1°C).

$$\text{Volume of receiver (mL)} = V_1 = \frac{W2 - W1}{d_1}$$

where:

d_1 = Density of water at temperature T_1°C

B.12.5. Procedure

Ensure that the bottle (B) is at least three-quarters full of water. Open tap S1 to the atmosphere and adjust tap S2 to connect the bottle (B) with the receiver (R). Open tap S3 and apply gentle pressure (using the manometer (M) as guide) via the open arm of S3 to force water into the receiver (R) until it is filled to just above the mark (A). Close tap S2 and release the pressure on tap S3. Carefully adjust tap S2 to allow the level of water in the receiver (R) to fall to coincide with the mark (A). Adjust tap S1 so that both the receiver (R) and the 100-mL reaction vessel (X) are connected to the atmosphere. Fill the burette with the sodium hydroxide solution, adjust the level to zero and wash any excess alkali from the tip of the burette to prevent any premature reaction with the sample. Weigh accurately 0.355-0.365 g (W3) of the sample and transfer quantitatively to the reaction vessel (X). Add 20 mL of water to the reaction vessel (X) and immediately connect it firmly to the apparatus. Allow the reaction vessel (X) and its contents to reach equilibrium and, after ensuring that the level of water in the receiver (R) is still at the mark (A), adjust tap S1 so that the receiver (R) is connected to the reaction vessel (X) only. Close tap S3 and adjust tap S2 to connect the receiver (R) with the bottle (B). Record the temperature (T_2°C) shown by the thermometer (C). Check the position of all taps and run the sodium hydroxide dropwise into the reaction vessel (X), controlling the rate of addition of alkali very carefully to prevent gas being evolved too quickly. If necessary, the reaction flask may be cooled by immersing it in a beaker of cold water. Maintain the pressure in the receiver (R), as indicated by the manometer (M), slightly below atmospheric pressure by opening tap S3 from time to time during the evolution of hydrogen. Continue the addition of alkali until a total of 20.0 mL has been added to the reaction vessel (X). Swirl the contents of the reaction vessel (X) to ensure that no unreacted sample remains. Allow the apparatus to stand for approximately 1 hour in order to allow the temperature to reach equilibrium. Note the temperatures (T_3°C) and (T_4°C) indicated by the thermometers (C) and (D), respectively. Adjust the level of the water in the receiver (R) to that of the water in the manometer (M) by releasing or applying pressure to the open end of tap S3. Close tap S2. Owing to the capillary effects, the liquid levels in the receiver (R) and the manometer (M) are in equilibrium when the bottom of the meniscus in the manometer (M) is level with the top of the meniscus in the receiver (R). Turn tap S1 to open the receiver (R) to the atmosphere and run the remaining water, from the receiver (R) through tap S2, into a tared 250-mL conical flask (W4 ± 10 mg). Weigh the flask immediately (W5 ± 10 mg). Record the atmospheric pressure (P).

B.12.6. Calculation

Volume of water remaining in receiver (mL) = $V_3 = \frac{W5 - W4}{d_2}$

where:

d_2 = Density of water at temperature T_4°C

Uncorrected free metallic aluminium (%) = $A = \frac{(P - x - y) \times (V_1 - V_2 - V_3) \times 0.02886}{(273.15 + T_4) \times W3}$

where:

P = Atmospheric pressure (mmHg)
x = Water vapour pressure at temperature T_3 (mmHg)
y = Barometric correction for brass scale (mmHg)
V_1 = Volume of receiver (mL)
V_2 = Volume of sodium hydroxide solution added (mL)
V_3 = Volume of water remaining in the receiver (mL)
T_4 = Ambient temperature of apparatus (°C)
W3 = Weight of sample (g)

Note: $0.02886 = \frac{273.15\ \text{K} \times 26.98\ \text{g/mol} \times 100}{760\ \text{mmHg} \times 22400\ \text{mL} \times 1.5\ \text{mol}}$

When quantities of elemental zinc and silicon are present in the sample, the following correction must be made:

Corrected free metallic aluminium (%) = A - (% zinc x 0.27) - (% silicon x 1.28)

Note: The values 0.27 and 1.28 derive from the ratio of the amounts of hydrogen formed and the ratio of the atomic weights of the elements Al, Zn and Si.

$Zn + 2\ NaOH + 2\ H_2O \rightarrow Na_2Zn(OH)_4 + H_2 \uparrow$

$0.27 = \frac{(1\ \text{mol} / 1.5\ \text{mol})}{(65.38\ \text{g/mol} / 26.98\ \text{g/mol})}$

$Si + 2\ NaOH + H_2O \rightarrow Na_2SiO_3 + 2\ H_2 \uparrow$

$1.28 = \frac{(2\ \text{mol} / 1.5\ \text{mol})}{(28.09\ \text{g/mol} / 26.98\ \text{g/mol})}$

B.12.7. Dead space correction

It is advisable to try to ensure that the final temperature T_3 is within 0.2°C of the initial temperature T_2, otherwise the dead space correction will be significant and must be taken into consideration.

Volume of hydrogen evolved = (V1 - V2 - V3) + C

where C is the volume correction to be applied due to the expansion or contraction of gas in the dead space.

If T_2 differs from T_3, then:

$$C = \frac{(V_4) \times (T_2 - T_3)}{273.15}$$

where:

V_4 = Volume of dead space (≈ 60 mL)

If T_3 is greater than T_2, then C is negative.

Note: The volume of dead space (V_4) corresponds to the volume of air above the test sample in the 100-mL reaction vessel (X) after 20 mL of water and 20 mL of sodium hydroxide have been added.

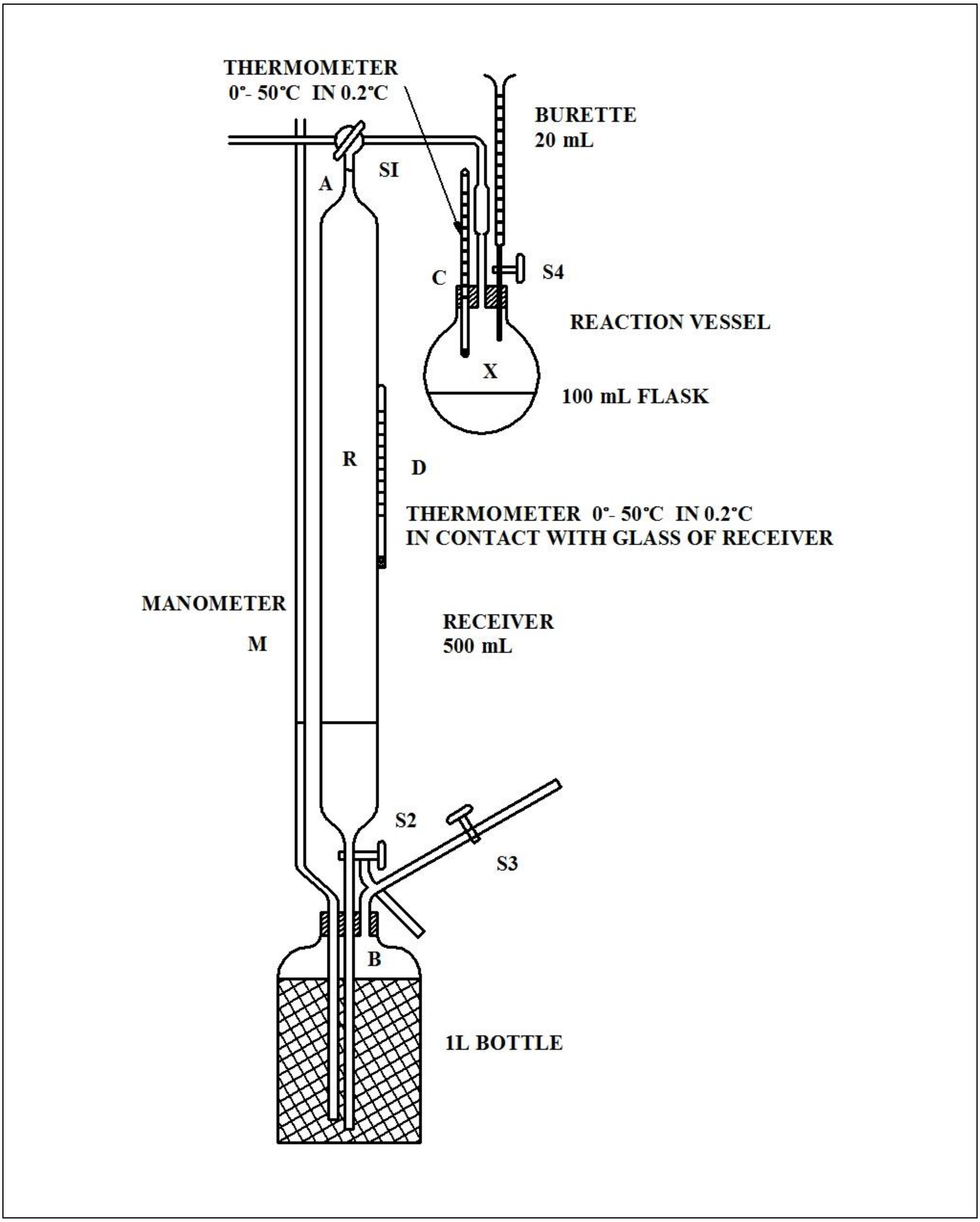

Figure 1: **Hydrogen evolution apparatus for the determination of free metal**

B.13. ELEMENTAL IMPURITIES

The percent of elemental impurities such as silicon, copper, iron, zinc, magnesium, lead, chromium, etc. shall be determined in accordance with ASTM E 101 ("Test Method for Spectrographic Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique") or an equivalent procedure. Alternate techniques to determine the elemental impurities include atomic absorption spectroscopy (AAS) or chemical analysis in accordance with ASTM E 34 ("Standard Test Methods for Chemical Analysis of Aluminum and Aluminum-Base Alloys") or an equivalent procedure.

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