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SUPERCEDING
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NATIONAL MILITARY ESTABLISHMENT SPECIFICATION

MANGANESE, POWDERED (FOR USE IN AMMUNITION)

This specification was approved by the Departments of the Army, the Navy, and the Air Force for use of procurement services of the respective Departments.

A. APPLICABLE SPECIFICATIONS AND OTHER PUBLICATIONS

A-1. *Specifications.*—The following specifications, of the issue in effect on date of invitation for bids, form a part of this specification:

U. S. ARMY SPECIFICATION

50-0-1—General Specification for Ammunition except Small Arms Ammunition.[1]

100-2—Standard Specification for Marking Shipments by Contractors.[1]

NAVY DEPARTMENT SPECIFICATION

General Specifications for Inspection of Material.[2]

FEDERAL SPECIFICATION

RR-S-366—Sieves; Standard, Testing.

A-2. *Other publications.*—The following publications, of the issue in effect on date of invitation for bids, form a part of this specification:

BUREAU OF SUPPLIES AND ACCOUNTS PUBLICATION

Navy Shipment Marking Handbook.[2]

INTERSTATE COMMERCE COMMISSION REGULATIONS

Regulations for the Transportation of Explosives and Other Dangerous Articles, etc.

B. GRADES

B-1. This specification covers the following grades of manganese powder, as specified in the contract or order (see par. H-2):

Grade I—99.5 percent total manganese.
Grade II—98.0 percent total manganese.
Grade III—95.0 percent total manganese.

C. MATERIAL AND WORKMANSHIP

C-1. *Grade I.*—Grade I manganese shall be ground to the proper granulation in an inert atmosphere.

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[1] Applicable only to Army purchases.
[2] Applicable only to Navy purchases.

D. GENERAL REQUIREMENTS

D-1. See section E.

E. DETAIL REQUIREMENTS

E-1. The material shall comply with the requirements shown in table I.

TABLE I.—*Detail requirements.*

	Grade I Percent (minimum)	Grade II Percent (minimum)	Grade III Percent (minimum)
Total manganese	99.5	98.0	95.0
Free metallic manganese	99.0		
Granulation: [1]			
Through No. 200 sieve		98.0	99.0
Through No. 230 sieve	100		
Through No. 325 sieve	98		
Particles smaller than 10 microns diameter	[2] 50		

[1] See par. H-3.
[2] Maximum.

F. METHODS OF SAMPLING, INSPECTION, AND TESTS

F-1. *Size of lot.*—Maximum, 1,000 pounds. Unless otherwise specified in the contract or order, the lot shall consist of material from not more than one manufacturing batch of manganese powder. (See par. H-6.)

F-2. *Sampling.*—Five containers shall be taken, at random, from the lot. If there are fewer than five containers in the lot, all the containers shall be taken for sampling. A ¼-pound sample shall be taken by means of a sampling thief, from each container so chosen. The samples shall be placed in a clean dry container, stoppered tightly and mixed thoroughly to form a composite sample representative of the lot. The composite sample shall be labeled to identify the sample with the lot represented.

F-3. *Inspection.*—

F-3a. *Army.*—Inspection shall be made in accordance with the requirements of U. S. Army Specification 50-0-1, and shall be made at the point of delivery unless otherwise specified in the contract or order.

F-3b. *Navy.*—Unless otherwise specified in the contract or order, inspection shall be made at the point of delivery.

F-4. *Tests.*—The laboratory tests shall be made in accordance with the following paragraphs. For Navy purchases, the tests shall be made at a Government laboratory unless otherwise specified in the contract or order.

F-4a. *Total manganese.*—Place an accurately weighed portion of approximately 0.25 gm. of the sample in a 750-ml. Erlenmeyer flask and add 50 ml. of nitric acid solution having a specific gravity of 1.135. When the sample has dissolved, add 1 gm. of sodium bismuthate of at least 80-percent purity. Bring the liquid to the boiling point and allow to boil for 3 to 5 minutes. If the precipitated oxides of manganese or the color of permanganic acid does not persist through this boiling, add more sodium bismuthate and boil the mixture again. Add sufficient sulfurous acid to clear the solution. Dilute the solution to a volume of 250 ml. with nitric acid solution having a specific gravity of 1.135, and cool to 10° to 15° C. Add 8 gm. of sodium bismuthate and agitate the mixture briskly for 1 minute. Dilute the mixture with 250 ml. of cold water and filter immediately through a layer of prepared asbestos supported on a perforated porcelain plate resting in a large glass funnel, taking care not to allow the filter to run dry during the filtration. Wash the flask and filter with cold 3-percent nitric acid solution until the washings are entirely colorless, again taking care not to allow the filter to run dry. Treat the combined filtrate and washings immediately as follows: Add to the filtered solution of permanganic acid an accurately weighed portion of approximately 9.1 gm. of solid ferrous ammonium sulfate taken from a bottle of the well-mixed salt; stir the mixture briskly, and as soon as reduction is complete and the salt dissolved, titrate the excess of ferrous salt with approximately $N/10$ potassium permanganate solution. Obtain the permanganate value of the ferrous salt as follows: Place 250 ml. of cold nitric acid solution having a specific gravity of 1.135 in a 750-ml. Erlenmeyer flask and add 2 gm. of sodium bismuthate; agitate the mixture vigorously for 1 minute. Dilute with 250 ml. of cold water and immediately filter through a layer of prepared asbestos as previously described; add to the filtrate, first 50.00 ml. of approximately $N/10$ potassium permanganate solution, and then an accurately weighed portion of approximately 3.0 gm. of the solid ferrous ammonium sulfate; dissolve the salt and titrate immediately with more of the $N/10$ potassium permanganate solution. Make the corrections for the excesses of permanganate used to cause the end point colors in both titrations. Calculate the percent total manganese as follows:

$$\text{Percent total manganese}=\frac{1.099\ N\left\{\frac{BC}{D}-E\right\}}{W}$$

where N=normality of the potassium permanganate solution

B=ml. of potassium permanganate solution required for reaction with D gm. of ferrous salt

C=gm. of ferrous salt added to reduce the permanganate formed from the manganese in the sample

D=gm. of ferrous salt used for determining the permanganate value of the ferrous salt

E=ml. of potassium permanganate solution used in back titration of the excess ferrous salt present after reduction of the permanganate formed from the manganese in the sample.

W=gm. of sample.

F-4b. *Free metallic manganese.*—Determine the free metallic manganese content by the hydrogen evolution method, using a suitable apparatus for dissolving the sample and measuring the gas evolved. The apparatus shown on figure 1 has been found satisfactory, and its use is described below. Assemble the apparatus as shown on figure 1, attaching the various parts to suitable supports. Turn the three-way stopcock to the A position, raise the leveling bulb until the meniscus of the water in the gas measuring buret is at the 0-ml. mark, and turn the stopcock clockwise to the B position. Place 100 ml. of a 5- to 8-percent acetic acid solution, previously saturated with hydrogen, in the reaction flask, and attach the reaction flask to the apparatus by means of the two-holed rubber stopper. Adjust the position of the thermometer in the rubber stopper so that the bulb of the thermometer dips into the acid solution. Weigh accurately a portion of approximately 1.0 gm. of the sample and wrap in cellophane or paper in such a manner as to make the following operations possible. Remove the reaction flask from the apparatus and place the wrapped sample in the neck of the flask without allowing the sample to fall into the acid. Reattach the reaction flask to the apparatus, again taking care not to allow the sample to fall into the acid. Allow the sample to remain in the neck of the flask for at least 10 minutes or until the atmosphere above the acid becomes saturated with water vapor. Read the temperature and turn the three-way stopcock clockwise to the C position. Cause the sample to fall into the acid by hitting the bottom of the reaction flask against the palm of the hand. Shake the flask to cause the wrapping on the sample to open and the manganese to come in contact with the acid. As the evolution of gas proceeds, lower the leveling bulb so that the level of the water in the gas measuring buret remains at approximately the same level as the water in the leveling bulb. When the reaction is complete, cool the acid solution in the reaction flask to its temperature prior to the reaction and keep at this temperature for 10 to 15 minutes. Adjust the level of the water in the leveling bulb to the same height as the meniscus of the water in the gas measuring buret. Read the volume of gas in the buret and correct for any error in the graduation of the buret. Read the gas temperature with a thermometer close to the buret. Read the barometric pressure on a mercurial barometer accurate to 0.1 mm. and corrected for temperature. Calculate the percent free metallic manganese as follows:

$$\text{Percent free metallic manganese} = \frac{273.1 \times 0.2450 \; V \; (P-p)}{760 \; (273.1 + T) W}$$

where V = ml. of hydrogen evolved
P = corrected barometric pressure
p = vapor pressure of water in mm. at temperature T
T = gas temperature in degrees C.
W = gm. sample

F-4c. *Granulation.*

F-4c (1). *Sieve analysis.*—Nest the specified sieve or sieves, conforming to Federal Specification RR-S-366, on a bottom pan. Place a weighed portion of approximately 50 gm. of the sample on the upper sieve, cover this, and shake for 10 minutes by hand or 5 minutes by means of a single-eccentric type mechanical shaker which imparts to the sieves a rotary motion and tapping action of uniform speed of 300 ± 15 gyrations and 150 ± 10 taps of the striker per minute. Weigh the amounts retained or passing through the sieve(s), and calculate to percentages as required.

F-4c (2). *Determination of the sub-sieve particle size distribution.*

F-4c (2) a. *Apparatus.*

F-4c (2) a (1). *Andreasen pipet.* Use the Andreasen pipet and its accessory equipment shown in figures 2 and 3. For each pipet used, accurately determine the volume of the cylinder with the pipet in place, the drop in liquid level in the cylinder due to the withdrawal of 10 ml. aliquots, and the volume of the 10 ml. chamber in the pipet. To facilitate and control the withdrawal of aliquots from the cylinder and to permit the rapid emptying of the pipet in preparation for the next aliquot, a convenient suction-pressure system, illustrated in figures 2 and 3 may be used. This consists of a Y tube, connected by means of rubber tubing to a rubber bulb and to the Andreasen pipet.

F-4c (2) a (2). *Dispersing solution.*—Dissolve four gm. of dioctyl sodium sulfo succinate (Aerosol O. T.) in approximately 4 liters (1 gal.) of distilled water. Add 0.5 gm. of potassium chromate to the resulting solution and mix thoroughly. Determine the density and absolute viscosity of this solution at 25° C.

F-4c (2) a (3). *Wash solution.*—Dilute 10 ml. of the dispersing solution to 500 ml. with distilled water.

F-4c (2) a (4). *Crucibles.*—Prepare Gooch crucibles of satisfactory filtering properties by depositing a thin layer of fine asbestos on a thin layer of coarse asbestos in the crucible. Dry the crucibles at 105° C. to constant weight, cool and weigh. The crucibles can be used for four to six determinations before being discarded. Fine porosity fritted glass crucibles may be used if it is first established that the finest particles in the sample will be retained on the crucible.

F-4c (2) a (5). *Filter flasks.*—Fasten four small filter flasks to a ringstand and attach to the suction by means of a manifold. Use stopcocks on each line of the manifold to permit individual control of the filter flasks.

F-4c (2) a (6). *Dispersing tube.*—Use a tube (see figs. 2 and 3) approximately ⅝ inch inner diameter and 5 inches long, equipped with a ground glass stopcock. This tube may be improvised by removing the capillary tip from a 100-ml. buret and cutting the graduated cylinder at the 88-ml. mark.

F-4c (2) a (7). *Dispersing brush.*—Use a brush with tufted tip. This brush shall fit snugly into the dispersing tube.

F-4c (2) a (8). *Heavy-duty motor.*—Use a heavy-duty motor equipped with a check to grip the wire end of the brush. The motor shall be capable of 900 r. p. m.

F-4c (2) b. *Procedure.*—Before starting the test, calculate the time at which an aliquot is to be taken from the pipet to determine particles smaller than 10 microns diameter (see par. H-4) using the following formula:

$$\text{Time (minutes)} = \frac{306.1\ VH}{7.20 - D}$$

where V = absolute viscosity of suspending medium in poises.

H = distance between surface of liquid and pipet tip in cm. before each sample is drawn.

D = density of suspending medium in gm. per ml.

Add approximately 7 ml. of the dispersing solution and 0.05 gm. of potassium chromate crystals to the dispersing tube. Then add a portion of 10.00 gm. of the sample of manganese powder to the tube. Insert the brush into the dispersing tube, and work up and down so as to disperse the sample throughout the solution. With the tufted tip of the brush in contact with the base of the tube, directly above the closed stopcock, attach the wire handle of the brush to the chuck of the heavy-duty motor. By means of a clamp, fix the dispersing tube to the stand supporting the motor to prevent the tube from rotating with the brush. Immerse the lower half of the tube in a beaker of cold water to prevent overheating as the suspension is stirred by the brush. Start the motor and stir the suspension for 15 minutes at a rate of approximately 900 r.p.m. Turn off the motor and detach the brush and dispersing tube from the motor and its supporting stand.

Insert a large funnel in the mouth of the cylinder of the Andreasen pipet. Hold the dispersing tube and contents (brush and suspension) over the funnel; open the stopcock of the tube and permit the suspension to run into the cylinder. Close the stopcock and add approximately 10 ml. of dispersing solution to the dispersing tube by means of a wash bottle. Move the brush up and down to loosen any manganese particles clinging to it; then open the stopcock. Repeat this procedure until both tube and brush have been thoroughly washed, as indicated by the absence of manganese particles in the dispersing solution when transferred from the dispersing tube to the cylinder. Add dispersing solution to the cylinder of the Andreasen pipet until, with the pipet inserted in place, the suspension is brought to the mark. Replace the pipet by a thermometer. Note the temperature of the suspension and allow hot or cold tap water to flow over the outside walls of the cylinder until the temperature of the suspension is 25.0 ± 0.5° C.; use the thermometer to stir the suspension vigorously during this process. Remove the thermometer and place the pipet with stopcock closed, in the cylinder. After making certain that the vent to the liquid chamber is closed shake the assembly in an inverted position for approximately 2 minutes. Take care to assure a uniform dispersion by sufficient shaking and inverting of the assembly. Upright the cylinder, open the stopcock of the pipet, and open the air vent. Blow out any unmixed suspension which may be in the stem. Place the assembly in a constant temperature bath maintained at 25.0 ± 0.5° C., fasten firmly to a rigstand, and note the exact time. It is important that the minimum possible time elapse between the cessation of shaking and the noting of the time. Let the suspension stand for the calculated time. During this time make the connection between the reservoir and the pipet by means of the length of flexible rubber tubing provided for this purpose. Seven seconds before the calculated time for taking the aliquot, start sucking up the sample as follows: Compress the bulb, close the open end of the Y tube, and control the rise of liquid in the pipet by slowly releasing the bulb. After the liquid has been drawn up to the 10-ml. mark of the pipet, close the stopcock of the pipet and open the end of the Y tube. To expel the liquid from the pipet, reverse the stopcock, close the open end of the Y tube, and compress the bulb gently. Wash the pipet bulb twice after the aliquot has been expelled by opening the pinch clamp (fig. 2) and allowing 2 to 3 ml. of solution to pass from the reservoir to the pipet bulb. Expel the washings in the same manner as the aliquot. Catch the aliquot and washings in a 50 ml. beaker. Transfer the aliquot and washings quantitatively to a tared Gooch crucible or fritted-glass crucible in position on a suction flask, using a rubber tipped stirring rod and a jet of dispersing solution from a wash bottle to effect complete transfer. Wash the contents of the crucible with three 5-ml. portions of wash solution and then three 5-ml. portions of acetone. Air dry the crucible, then dry for 15 minutes at 100° to 105° C., cool in a desiccator and weigh. Calculate the increase in weight as percent suspended particles in the aliquot finer than 10 microns as follows:

$$\text{Percent finer than 10 microns} = \frac{100\,AV}{BW}$$

where A = gm. of suspended particles in aliquot taken at time calculated for 10-micron diameter

B = ml. of aliquot taken (see par. F-4c (2) b)

V = ml. of suspension in Andreasen pipet at start of test (see par. F-4c (2) b)

W = gm. of sample.

F-5. *Rejection and resubmission.*—If the composite sample fails to comply with any of the specified requirements, the entire lot shall be rejected. The contractor shall have the option of having a partial or complete analysis made on each container in the lot at no expense to the Government. The contractor may then remove defective portions of the lot and resubmit the lot for acceptance.

G. PACKAGING, PACKING, AND MARKING FOR SHIPMENT

G-1. *Packing.*—Unless otherwise specified in the contract or order, powdered maganese shall be packed in plain steel drums conforming to the requirements of Specification 37D of Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc. Each drum shall have a removable head and shall be airtight when the cover is secured in place. The gross weight of the container and contents shall not exceed 110 pounds.

G-2. *Marking.*—Shipments shall be marked to insure safe handling as required by Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc, and in addition shipments for the Army shall be marked in accordance with the requirements of U. S. Army Specification 100-2; for the Navy, in accordance with the requirements of the Navy Shipment Marking Handbook. Any special marking shall be specified in the contract or order.

H. NOTES.

H-1. *Use.*—Grade I manganese power covered by this specification is intended for use in the manufacture of delay powder. Grades II and III are intended for use in pyrotechnic mixtures.

H-2. Requests, requisitions, schedules, and contracts or orders should contain the following features:

(a) Title, number, and date of the specification.
(b) Grade required. (See par. B-1.)
(c) Special marking required. (See par. G-2.)

H-3. Manganese powder complying with the granulation requirement specified in paragraph E-1 may not be suitable for use in all types of delay powder. Special requirements for particle-size should be stated in the contract or order.

H-4. If it is desired to determine "percent finer than" values for particle sizes other than 10 microns the following method of calculating the time for taking aliquots may be used:

$$\text{Time} = \frac{KH}{d^2}$$

$$\text{and } K = \frac{3.061 \times 10^4\ V}{(D_1 - D_2)}$$

where d = specified diameter of particles, in microns
V = absolute viscosity of the suspending medium, in poises
H = distance between liquid surface and pipet tip when aliquot is drawn in cm.
D_1 = density of particle in gm. per ml.
D_2 = density of liquid in gm. per ml.
Time = interval starting immediately after placing the assembly in the constant temperature bath at 25° C. and terminating upon the start of removal of the desired aliquot.

The following illustrates the calculations made for the determination of the particle size distribution of manganese (density 7.20) for the diameters selected, using an Andreasen pipet with the following characteristics: volume, 640 ml.; distance from pipet tip to liquid level at start, 18.90 cm.; average decrease in this distance on withdrawal of a 10-ml. aliquot, 0.34 cm. The dispersing solution is the water-aerosol-potassium chromate mixture described above.

Selected diameter d (microns)	$1/d^2$	K	$K1/d^2$	H (cm)	$\frac{KH}{d^2} = T$ (minutes from start of test)
30	0.00111	44.1	0.0490	18.90	0.93
20	.00250	44.1	.110	18.56	2.04
10	.01	44.1	.441	18.22	8.03
5	.04	44.1	1.764	17.88	31.5

Calculate the percentage finer than selected diameter by means of the formula given in paragraph F-4c (2) b by substituting for A the weight of suspended particles in the aliquot taken at the time calculated for the selected diameter.

H-5. Information as to the availability of Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc., may be obtained from the Interstate Commerce Commission, Washington 25, D. C.

H-6. A batch is defined as that quantity of material which has been subjected to some unit chemical or physical mixing process intended to make the final product substantially uniform.

H-7. Copies of National Military Establishment, Joint Army-Navy and Federal specifications (required for Army and Air Force purchases) and U. S. Army specifications may be obtained as indicated in the "Index of United States Army, Joint Army-Navy, and Federal Specifications and Standards." Copies of this index may be obtained from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Services within the Department of the Army and the Department of Air Force will obtain copies of National Military Establishment, Joint Army-Navy, U. S. Army, and Federal specifications through established departmental channels. Both the title and identifying symbol number should be stipulated when requesting copies.

H-8. Copies of National Military Establishment, Joint Army-Navy, and Federal specifications (required for Navy purchases), Navy Department specifications, and the Navy Shipment Marking Handbook may be obtained upon application to the Bureau of Supplies and Accounts, Navy Department, Washington 25, D. C., except that

Naval activities should make application to the Supply Officer in Command, Naval Supply Center, Norfolk 11, Va. Both the title and identifying symbol number should be stipulated when requesting copies of specifications.

H-9. Copies of this National Military Establishment specification required for Army purchases) may be obtained from the Office, Chief of Ordnance, Department of the Army, Washington 25, D. C.

ARMY: OC.
NAVY: OAS.

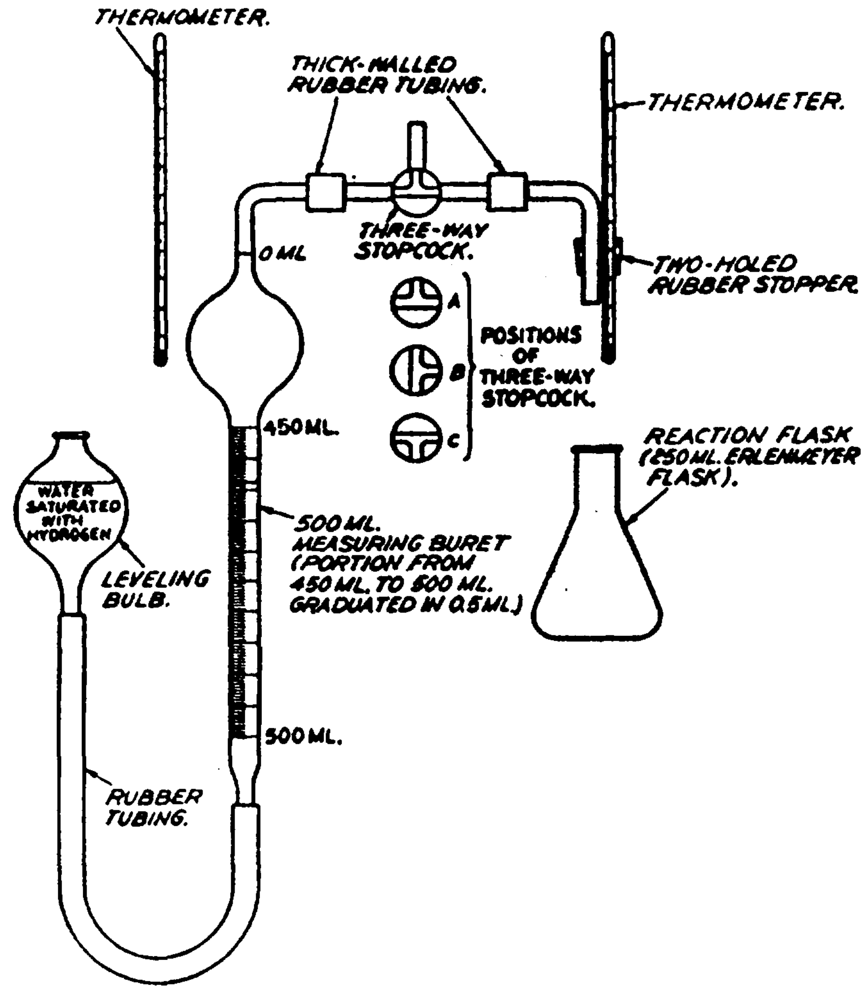

FIGURE 1.—*Assembly of apparatus (for determining free metallic manganese content).*

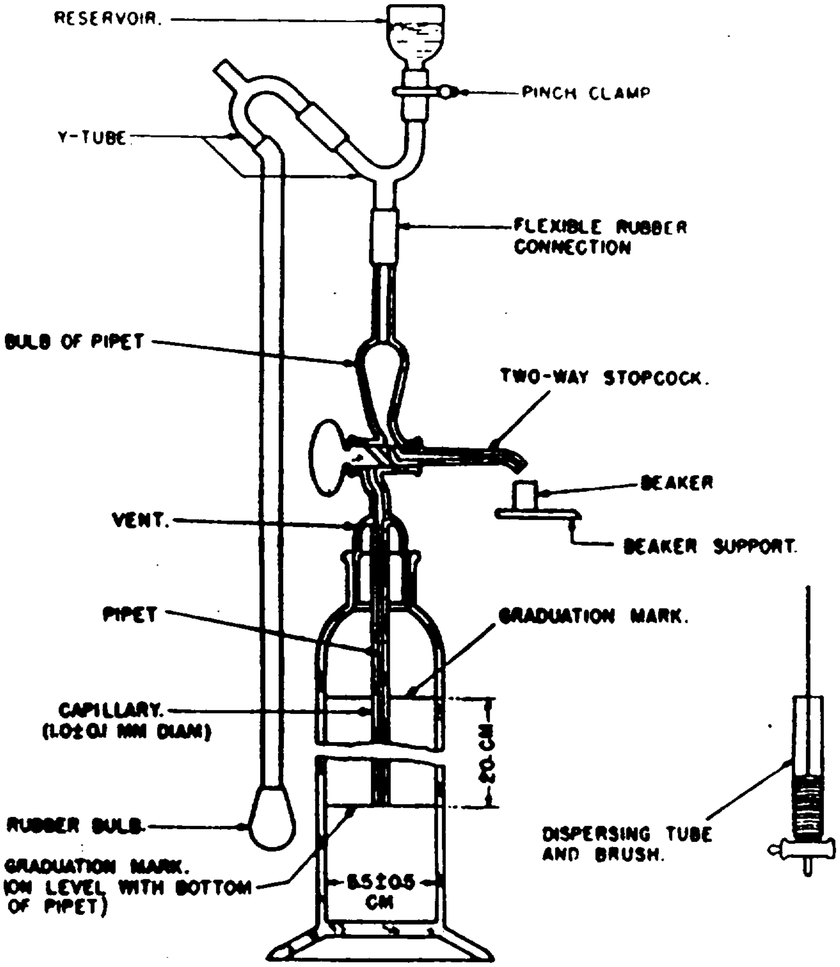

FIGURE 2.—*Andreasen pipet and accessories (sectional view).*

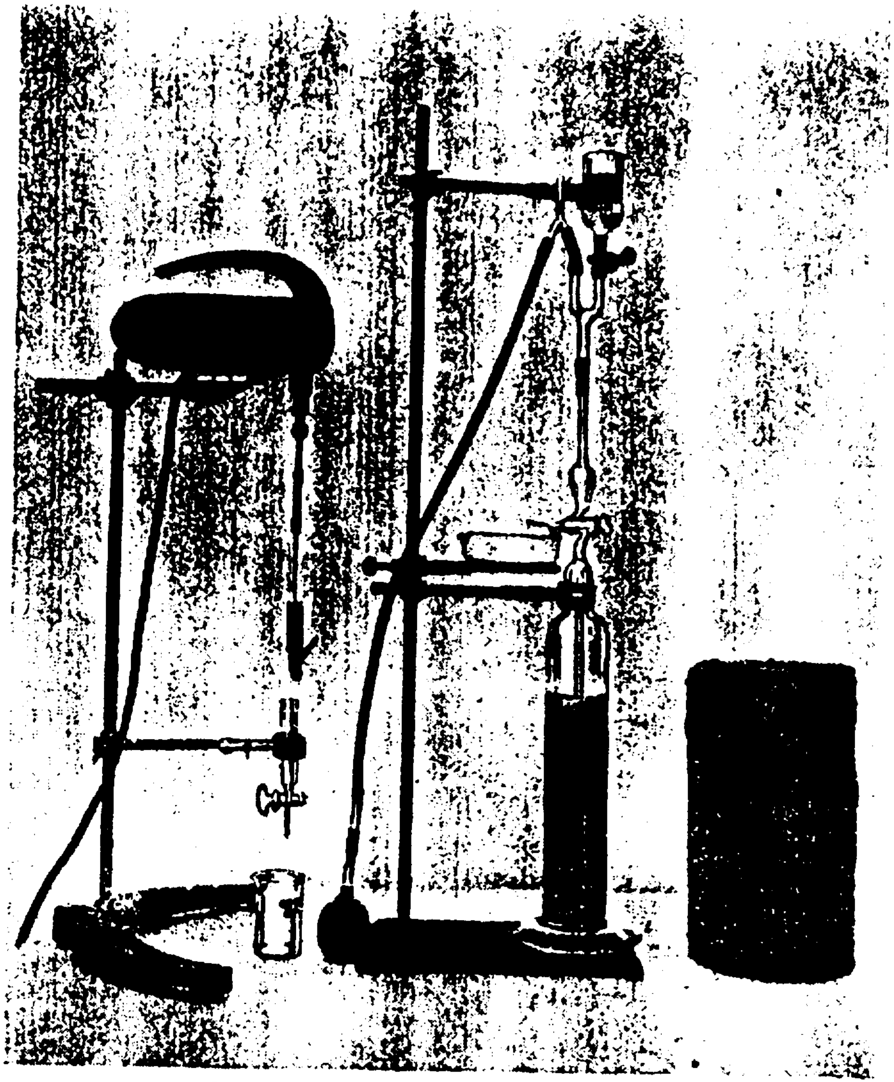

FIGURE 3.--*The Andreasen pipet and accessories.*