MIL-P-670A <u>10 February 1966</u> SUPERSEDING JAN-P-670 27 August 1948

MILITARY SPECIFICATION

PHOSPHORUS, RED, STABILIZED

1. SCOPE

1.1 Scope. This specification covers one grade of aluminated red phosphorus furnished in powdered form, as hereinafter specified.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein:

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieves, Standard for Testing Purposes

S STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes MIL-STD-125 - Marking for Shipment and Storage

FBC 6810

(Copies of specifications, standards, drawings and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publication. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated the issue in effect on date of invitation for bids or request for proposal shall apply.

INTERSTATE COMMERCE COMMISSION

49-CFR-71-90 - Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles

(The Interstate Commerce Commision regulations are now a part of the Code of Federal Regulations (1949 Edition-Revised 1950) available from the Superintendent of Documents, Government Printing Office, Washington 25, D.C. Orders for the above publication should cite "49 CFR 71-90").

AMERICAN SOCIETY FOR TESTING AND MATERIALS

ASTM Standard D942-50

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa. 19103).

3. REQUIREMENTS

3.1 Granulation.

3.1.1 <u>Before alumination</u>. Based on the selective sedimentation test described in paragraph 4.5.1(a), the phosphorus shall contain a maximum of 3 percent of particles of less than 10 microns diameter.

3.1.2 <u>After alumination</u>. When tested in accordance with 4.5.1(b), the powder shall pass through the following sieves:

Thru U. S. Standard Sieve No.	Percent
100	98.0 min.
140	85.0 min.
325	50 ± 20

Sieves shall conform to RR-S-366.

3.2 Oxidation. Based on the Norma-Hoffman oxidation bomb test described in 4.5.2, the phosphorus shall show the following oxidation characteristics.

3.2.1 <u>Induction Period.</u> The minimum induction period shall be 20 hours.
3.2.2 <u>Oxidation rate.</u> The maximum decrease in pressure per hour for a
5 gram sample shall be 1.4 psi for the automatic recording method and 1.6
psi for visual method.

3.3 Copper and iron. When tested in accordance with 4.5.3, the maximum content of either copper or iron shall be 50 parts per million. The product of the copper content and iron content. each expressed in parts per million, shall not exceed 125.

3.4 Total phosphorus. When tested in accordance with 4.5.6, the total phosphorus content shall be a minimum of 93 percent.

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3.4.1 <u>White phosphorus</u>. When tested in accordance with 4.5.7. the content of white phosphorus shall be a maximum of 0.01 percent.

3.5 Alumina. When tested in accordance with 4.5.5, the alumina content shall be 2,5 \pm 0.5 percent calculated as $A1(OH)_3$.

3.6 The phosphorus, red, stabilized powder shall be free flowing and free from lumps.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection. requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4..2 Lot. Unless otherwise specified in the contract or order, a lot shall consist of 1,000 pounds or fraction thereof of the material submitted for inspection at one time.

4.3 Sampling. Samples taken by the inspector for the purpose of tests prescribed in this specification shall be selected in a manner as to correctly represent the material furnished and avoid needless destruction of finished material when samples representative of the material are available from other sources.

4.3.1 <u>Before alumination</u>. Remove approximately 100 grams, dry weight, of sample from the process filter cake just prior to acumination and place in a glass-stoppered bottle. Seal the bottle with wax and label to show the material, manufacturer, plant, number of pounds in the lot, lot number, and the contract or order number.

4.3.2 <u>After alumination</u>. Select a minimum of 10 percent of the containers in the lot or one container if there are less than 10 containers in the lot. Take from each selected container a sample of approximately 100 grams and place in a glass-stoppered bottle, Seal the bottle with wax, and label to show the material, manufacturer, plant, number of pounds in the lot, lot number, and contract or order number, All applicable acceptance tests shall be made on every primary sample. Laboratory examination of the samples of the finished product shall be made within 5 hours after removal from the sealed bottles.

4.4 Examination. Examination shall be made in accordance with the requirements of MIL-STD-105.

4.5 Test procedures.

4.5.1 <u>Granulation</u>.

(a) <u>Before alumination</u>.

(1) Preparation of sample. Dry the sample, taken in accordance with 4.3.1, for 4 hours in a vacuum oven at 60° \pm 2°C.

(2) Selective sedimentation test for particles of les. than 10 microns diameter.

(2.a) <u>Apparatus</u>. Andreasen Sedimentation Pipet (Figure 1). This apparatus is available commercially (see 6.3).

(2.b) <u>Dispersing solution.</u> Dissolve 2 grams of Calgon in 2 liters of water,

(2.c) <u>Principle.</u> The method is based upon Stokes' equation relating to the rate of sedimentation of solid particles of various sizes falling freely in a liquid medium (see 6.4).

(2.d) Procedure. Mix the red phosphorus thoroughly and weigh a 5 gram sample into a 250 ml. beaker. Add approximately 200 ml. of dispersing solution and mix with a stirring rod for 2 minutes. Transfer to the cylinder of the Andreasen pipet apparatus and wash all the material from the beaker into the cylinder with dispersing solution contained in a wash bottle. Add sufficient dispersing solution to the cylinder so that the level is at the 20 cm. mark when the pipet is in place. Close the vent hole of the apparatus and shake thoroughly for 5 minutes. Place in a constant temperature bath at 25°C and record the zero time. At the end of 46 minutes, open the vent hole, and draw up 10 ml, of liquid into the pipet by applying gentle suction. Close the stopcock when the 10 ml. mark is reached. Turn the 3-way stopcock and drain off the solution from the pipet into a tared 15 ml. sintered glass crucible of fine porosity. Rinse the pipet with dispersing solution contained in a wash bottle and collect the rinsings in the crucible. Place the crucible in a suction apparatus, turn on the suction, allow to drain, and wash several times with water. Dry in a vacuum oven at 60° C for 3 hours, cool, and weigh. Calculate the percent of particles of less than 10 microns as follows:

$$P = \frac{10 \text{ RV}}{W}$$

where

P = percent of particles of less than 10 microns
R = grams of residue in crucible
V = volume of cylinder (to 20 cm. mark) in ml.
W = grams of red phosphorus in cylinder

(b) <u>After alumination.</u> Nest the specified sieves in the proper order above a receiving pan and place a weighed portion of approximately 100 grams of the dried sample on the upper sieve. Cover and shake for 30 minutes using a mechanical shaker (Rotape or equal Calculate the weight of the material passing through each sieve as a percentage of the total weight of sample received.

4.5.2 Oxidation

(a) <u>Apparatus</u>

Norma-Hoffman oxygen bomb (as described in ASTM Method 942-50 (see 6.5)

Recording pressure gauge (see 6.6).

Copper or bronze tubing for connecting the bomb to the automatic recorder (this tubing is not needed if the readings are made visually). The tubing is 3/16 inch in internal diameter and 9 feet long (total length), It contains a helix (5 turns, 2-1/2 inches in diameter) to provide flexibility. It has couplings at its ends and a valve a few inches from the recorder to bleed off the oxygen.

Special 50 ml. beaker. Eliminate the lip and protruding rim of a 50 ml. beaker by shaping in a flame. This treatment is necessary to permit the beaker to fit into the bomb.

(b) <u>Procedure.</u> Place a flat wad of pyrex wool (0.1 to 0.2 grams) into the bottom of the bomb and add 0.5 ml. of water measured with a pipet. Weigh a 5 gram sample into the special 50 ml. bealer and cover with a watch glass. Place the beaver on top of the glass wool. Bolt the bowp securely and flush with oxygen, bringing the bomb pressure up to 50 psi and releasing three times. Allow 45 to 50 psi pressure to remain and place the bomb in a bath maintained at 110° plus or minus 2°C. Release the pressure intermittently to exactly 50 psi until there is no further increase. Starting at this time, record the pressure using the automatic recorder. Al alternate (but less desirable) procedure is to make the readings visually at 1/2 hour intervals for 1-1/2 hours and at 2 hour intervals thereafter. Plot cumulative pressure drop against time (see figure 2) considering the zero time to be 3/4 hour after the bomb is placed in the bath. The curve is characterized by a portion of moderate slope containing a linear portion, followed by a portion of a greater slope which also contains a linear portion and which tapers off. The induction period is defined as the abcissa (AE') of the intersection of the two linear portions extended (BB' and MM'). The oxidation rate is defined as the slope of the second linear portion (MM') (see 6.7).

4.5.3 <u>Copper.</u>

(a) <u>Reagents.</u>

Sodium diethyldithiocarbamate solution (0.1 percent), Dissolve 0.5 gram of sodium diethyldithocarbamate in water and dilute to 500 ml. Store in an amber bottle.

Ammonium citrate solution (40 percent). Dissolve 200 grams of ammonium citrate $((NH_4)_2HC_6H_5O_7)$ in about 400 ml. of water, make just ammoniacal to litmus paper with ammonium. hydroxide (1 to 1), wash into a 500 ml. separator funnel, add 5 ml. oc diethyldithiocarbamate solution, and shake with 25 ml. of chloroform for 1 minute, Drain off and discard the chloroform layer, and dilute to 500 ml. with water.

EDTA (disodium dihydrogen ethylenedinitrolotetraacetate) (5 percent). Dissolve 10 grams of EDTA in water and dilute to 200 ml.

Standard copper solution (1 ml. = 0.01 mg. of (Cu). Dissolve 0.1964 gram of copper sulfate $(CuSO_4.5H_2C)$ in water, add 5 ml. Of hydrochloric acid, and dilute to 1 liter in a volumetric flask. Pipet 50 ml. into a 250 ml. volumetric flask and dilute to the mark.

(b) <u>Preparation of calibration curve.</u> Pipet 1.0, 2.0, 3.0, 4.0, and 5.3 ml. of standard copper solution into 400 ml. beakers, and add 25 ml. of water and 2 ml. of perchloric acid. Carry along a reagent blank. Add 5 ml. of ammonium citrate solution (40 percent), 1 ml. of EDTA solution (5 percent) and 2 drops of phenol red indicator. Add ammonium hydroxide until the solutions just change to red , cool , and wash into 125 ml. separatory funnels whose sterns have been dried with wads of filter paper. Add 5 ml. of sodium diethyldithiocarbamate solution (0.1 percent), and 10 ml. of chloroform and shake for 1 minute. Drain the chloroform layer into dry 25 ml. volumetric flasks, extract twice more with 5 ml. portions of chloroform, and dilute the combined extracts to 25 ml. with chloroform. Measure the transmittance at 435 millimicorns with a spectrophotometer that has been set to 100 percent transmittance with the reagent blank. Plot mg. of copper against percent transmittance on semi-log paper.

(c) <u>Procedure.</u> Weigh a 1 gram sample into a 400 ml. beaker, and add 10 ml. of water and 35 ml. of nitric acid. Carry along a reagent blank. Boil down to a volume of about 20 ml. and add 2 ml. of perchloric acid. Evaporate to fumes of perchloric acid, and fume for 1 to 2 minutes. cool to room temperature. Add 25 ml. of water, 5 ml. of ammonium citrate solution (40 percent) and 1 ml, of EDTA solution (5 percent), and proceed as described in paragraph 4.5.3 (b). Convert the reading to mg. of copper by consulting the calibration curve, Calculate as follows:

$$PPM of Cu = \frac{mc. of Cu \times 1000}{grams of sample}$$

4.5.4 <u>Iron.</u>

(a) <u>Reagents.</u>

Hydroxylamine hydrochloride solution (5 percent) . Dissolve 25 grams of hydroxylamine hydrochloride in water and dilute to 500 ml.

Ortho-phenanthroline solution (0.2 percent). Dissolve 1 gram of ortho-phenanthroline in water and dilute to 500 ml . Store in a dark bottle.

Sodium citrate solution (20 percent). Dissolve 100 grams of sodium citrate ($Na_3C_6H_5O_7\cdot 2_{L_1}$ in water and dilute to 500 ml.

Standard iron solution (1 ml. = 0.1 mg. of Fe). Dissolve 0.1000 gram of pure iron (National Bureau of Standards Sample 55c) in 50 ml. of hydrochloric acid by heating on the hot plate in an Erlenmeyer flask. Add 3 ml. of hydrogen peroxide (30 percent), boil 10 minutes, cool, and dilute to 1 liter in a volumetric flask,

(b) Preparation of calibration curve. Transfer 0.5, 1.0, 1.5, 2.1, 2.5, and 3.0 ml, of standard iron solution to 100 ml. volumetric flasks and add 35 ml. of water. Carry along a reagent blank. Add 10 ml. of hydroxylamine hydrochloride solution (5 percent) and allow to stand for 15 minutes. Add 10 ml. of ortho-phenanthroline solution (0.2 percent) and 15 ml. of sodium citrate solution (20 percent) and dilute to the mark. Allow to stand for 1 hour and measure the transmittance at 510 millimicrons with a spectrophotometer that has been set to 100 percent transmittance with the reagent blank. Plot mg. of iron against percent transmittance on semi-log paper.

(c) <u>Procedure.</u> Clean 400 ml, beakers (preferably unscratched) and pyrex watch glasses with hydrochloric acid. Weigh a 0.5 gram sample, and add 10 ml. of water and 35 ml. of nitric acid. Boil down on the hot plate at fairly high heat with the watch glass in place to the sudden appearance of red nitrogen dioxide fumes at a volume of about 1 ml. and immediately remove the beaker from the hot plate. Just prior to the appearance of the nitrogen dioxide, the solution will start to foam. The beaker should be removed from the hot plate before the foaming has ceased. If the solution is heated too long (to the point at which white fumes appear) the determination should be discarded. Carry along a reagent blank prepared by adding 35 ml. of nitric acid to 10 ml, of water and evaporating just to dryness. After removing the beakers from the hot plate, allow to cool, add 75 ml. of water, and boil down to about 35 ml. with the watch glass in place. Cool to room temperature (but not lower), add 10 ml. of hydroxylamine hydrochloride solution (5 percent) and allow tc stand for 15 minutes. Add 10 ml. of orthophenanthroline solution (0.2 percent) and 15 ml. of sodium citrate solution (20 percent). dilute to 100 ml. in a volumetric flask, and allow to stand for 1 hour. Measure the transmittance at 510 millimicorns with a spectorphotometer that has been set at 100 percent transmittance with the reagent blank. Convert the reading to mg. of iron by consulting the calibration curve. Calculate as follows:

PPM of Fe =
$$\frac{m_{1.} \text{ of Fe x 1000}}{\text{srams of sample}}$$

4.5.5 <u>Alumina.</u>

(a) <u>Reagents</u>

5-Hydroxy quinolilne solution (2.5 percent). Dissolve 12.5 grams of 3-hydroxyquinoline in 25 ml. of glacial acetic acid by warming gently on the hot plate. Dilute to 500 ml. with water. Filter just before using if not clear.

Wash solution. Add 12 ml. of 3-hydroxyquinoline solution to about 500 ml. of water, add 30 ml. of ammonium hydroxide (1 to 1), and dilute to 500 ml. Prepare just before using.

(b) <u>Procedure.</u> Weigh a 1 gram sample into a covered 400 ml. beaker and add 10 ml. of water and 35 ml. of nitric acid. Boil down to a volume of about 20 ml. and acid 5 ml. of perchloric acid. Evaporate to strong fumes of perchloric acid and fume for 1 minute. Add about 50 ml. of water and if the solution is not clear, filter, and wash with water. Dilute to about 200 ml. with water and add 10 ml, of 8-hydroxyquinoline solution (2.5 percent). Make just alkaline to litmus paper with ammonium bydroxide (1 to 1 and add 20 ml. of ammonium hydroxide (1 to 1) in excess, Wash down and remove the litmus paper. Heat to 60° to 70°C (use 2 thermometer) and digest at that temperature for 30 minutes. Cool to about 15°C, in a cold water bath and filter through a tared sintered glass crucible of medium porosity. Transfer the precipitate to the crucible and wash four times with the wash solution. Finally wash twice with cold water Dry at 135°C for 90 minutes, cool, and weigh Calculate as follows:

Percent A1(OE)₃ = $\frac{16.93 \times f.rams \text{ of precipitate}}{3 \text{ rams of sample}}$

(c) <u>Microscopic examination of red phosphorus</u>. Examine the red phosphorus under a microscope to make sure that the aluminum is not present as metallic aluminum powder.

4.5.6 Total phosphorus.

(a) <u>Reagents.</u>

Magnesia mixture. Dissolve 50 grams of magnesium chloride (NgCl₂.6H₂O) and 100 grams of ammonium chloride in about 500 ml. of water, add ammonium hydroxide in slight excess, allow to stand overnight, and filter if a precipitate appears. Make just acid with hydrochloric acid and cilute to 1 liter.

Ammonium citrate solution (20 percent). Dissolve 200 grams of ammonium citrate $(NH_{\ell})_2HC_5H_5O_7$) in vater and dilute to 1 liter.

(b) <u>Procedure.</u> Transfer a 1 gram sample to a 400 ml. beaker and add 10 ml. of water and 35 ml. of nitric acid. Cover with a watch glass, warm on the lot plate until the vigorous reaction ceases, and then boil down to a volume of approximately 20 ml. Add 10 ml. of perchloric acid and boil down to a volume of 3 or 4 ml. cool, add water, and dilute to 500 ml. in a volumetric flask. Pipet a 25 ml. aliquot (equivalent to $^{0.05}$ gram of sample) into the original beaker , and add 50 ml. of water, 15 ml. of ammonium citrate solution (20 percent) , and 150 ml. of ma gnesium mixture. Add ammonium hydroxide (1 to 1 with stirring until the precipitate starts to appear, stop the addition of the ammonium hydroxide, and stir for about 2 minutes. Add 25 ml. of ammonium hydroxide (1 to 1) in excess and allow to stand overnight. Filter through a Whatman No. 40 filter paper, wash the precipitate into the filter paper with ammonium hydroxide (1 to 50), and then wash three t imes with ammonium hydroxide (1 to 50). Dissolve the precipitate off the filter paper into the orignal 400 ml. beaker with two 12 ml. portions of hydrochloric acid (1 to 1) and then wash several times with hot hydrochloric acid (1 to 50). Add 1 ml. of ammmonium citrate solution and 4 ml. of magnesia mixture and again precipitate by adding ammonium hydroxide as previously described. Add 10 ml. excess ammonium hydroxide (1 to 1) and allow to stand overnight. Filter through a Whatman No. 40 filter paper, transfer the precipitate to the filter paper with ammonium hydroxide (1 to 50), and wash five times with ammonium hydroxide (1 to 50). Transfer the filter paper and precipitate

to a tared porcelain crucible, and char and burn off the filter paper at low heat. Ignite over a Meker burner until the precipitate is white, then ignite over a blast burner for 15 minutes. Cool and weigh as $Mg_2P_2O_7$. Calculate as follows:

> Percent total phosphorus = <u>27.83 x grams of precipitate</u> grams of sample in aliquot

4.5.7 White phosphorus.

(c) <u>Preparation of test paper</u>. Dissolve 10 grams of copper sulfate ($CuSO_4 \cdot 5H_2O$) in 100 ml. of water. Dip pieces of filter paper into this solution and allow to dry in air. Gut the dried paper into strips about 0.6 cm. vide and 10 cm. long.

(b) <u>Preparation of standards</u>. Prepare a solution containing 1 gram of white phosphorus per 200 ml. of carbon disulfide. By diluting this solution, prepare a series of four permanent standards ranging in concentration from 0.0005 to 0.0025 gram of white phosphorus per 100 ml. (other standards can be prepared as desired). Store these solutions in the dar .

(c) <u>Procedure</u>. Weigh 20 grams of the aluminated sample into a flash having a ground-glass stopper and add approximately 50 ml. of carbon disulfide. Snake and mix thoroughly and allow to stand overnight in the dart. Fulter through a large sintered glass crucible of medium porosity, wash thoroughly with carbon disulfide, and dilute the filtrate to 100 ml. in a glass-stoppered cylinder with carbon disulfide. Determine the white phosphorus concentration by immersing strips of test paper in the unknown and standard solutions for one minute and visually comparing the brown stains produced. Calculate as follows:

5. PREPARATION FOR DELIVERY

5.1 Packing

5.1.1 Lovel C. Unless otherwise specified, the red phosphorus shall be packed in hermetically seeled tinned containers of approximately 10 pounds capacity so constructed as to insure acceptance by common or other carriers to insure safe delivery at destination at lowest transportation costs without assessment of penalty charges for improper packing. Containers shall comply with Interstate Commerce Commission Regualtions (see Code Fed . Reg. CFR 71-90) and regulations of carriers as applicable to the mode of transportation.

5.2 Marking .

5.2.1 <u>Mulitary</u>. In addition to any special marking required by the contract or order, shipping containers shall be marked in accordance with MIL-STD-129.

5. NOTES

6.1 Intended use. The stabilized red phosphorus covered by this specification is intended for use in pyrotechnics.

6.2 Ordering data. Purchasers should exercise any desired options cellered herein and procurement documents should specify the following:

(a) Title , number, and date of this specification.

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..3 Fisher Scientific Co. 14-232, Catalogue 03, and Scientific Oldos A custus Co. JP-3250, Crtalogue 63.

6.4 Studes' equation is as follows:

$$D = \sqrt{\frac{13nh \times 10^6}{(c_s - d_1)}} t$$

mere D = diameter of particles in microns

n = viscosity of liquid in centipoises

h = height of fall in cm.

ds = density of solid particles in grams per co

d1 = density of liquid in grams per ce

0 = acceleration due to gravity in cm. per second per second

v = time of fall in seconds

In order to determine the time for the settling of particles of 10 microns, the above equation was solved for t after making the following substitutions: D = 10, n = 0.094, h = 20, $c_s = 2.20$, $d_1 = 0.997$, g = 940.

6.5 The Norma-Hoffman oxygen bomb may be obtained from the Precision Scientific Co., 73414, Catalogue No. 40, and Fisher Scientific Co., 13-555-200, Catalogue 63.

6.6 The recording pressure gauge may be obtained from the Fisher Scientific Co., 13-555-130, or 13-555-135, or 13-555-140, Catalogue 63.

6.7 The linear portions of the graph described in 4.5.2 follow the Ceneral formula y = sx + b and y = mx + n, for the first and second portion, respectively. At the point of intersection the abcissa x would equal $\frac{n-5}{s-m}$. By definition, the constant m is the slope of the second linear portion. For each equation the constants s and b and m and n can be determined by substitution of a series of pairs of x and y values obtained as the experimental data. By the method of least squares, the "best values" for the constants can be obtained. Consult any standard textbook on statistical methods for decailed explanation of this method. Preparing activity: Custodian: Army - MU Army - MU Navy - WP Project No. 6810-0235 Review activities: Army - MU Navy - WP User activities: Army - MI

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Figure 1 - Andreasen Sedimentation Pipet

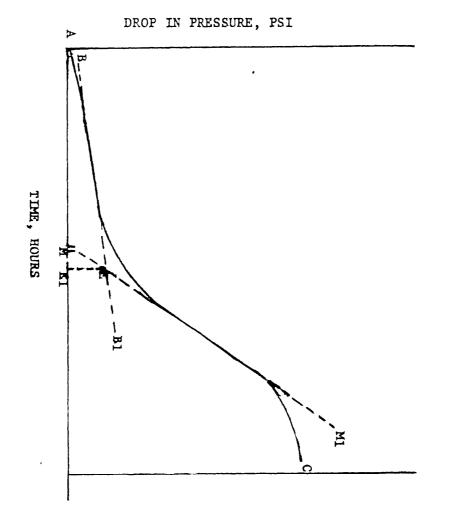


Figure 2 - Typical bomb oxidation curve

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SPECIFICATION ANAL	YSIS SHEET	Form Approved Budget Bureau No. 119-R004
	INSTRUCTIONS	
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