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2. ORIGINATOR'S REPORT TITLE Development of Porosity Test, Benite Strands				5. TEST DAY MO. YR. COMPL. 10 62	
				6. PROGRAM OR WEP. SYST.	

24. OUTLINE, TABLE OF CONTENTS, SUMMARY, OR EQUIVALENT DESCRIPTION:

The present method for evaluating the functioning quality of benite strands is the closed bomb test for quickness. This report describes a non-destructive, faster and simpler porosity test which correlates very closely with the quickness test. The results obtained by each test from twenty lots of benite (representing satisfactory quality) were compared. A highly significant correlation coefficient of 0.92 was found. The regression equation with associated confidence limits was derived. This equation, which relates quickness and porosity results, can be used to predict one value from the other. Arrangements for using the porosity test in process control and for further comparisons with the closed bomb quickness test have been made.

CONCLUSIONS

1. A simple technique for porosity testing was developed.
2. The porosity test correlated closely with the quickness test when applied to twenty lots of benite strands.
3. The porosity test, fast in terms of elapsed and working times, is simple in terms of equipment, standardization and calculations required for its installation, maintenance and operation. This technique is applicable for the non-destructive testing of a wide variety of Ordnance materials and the apparatus can be modified to accommodate essentially any size or shape of sample.

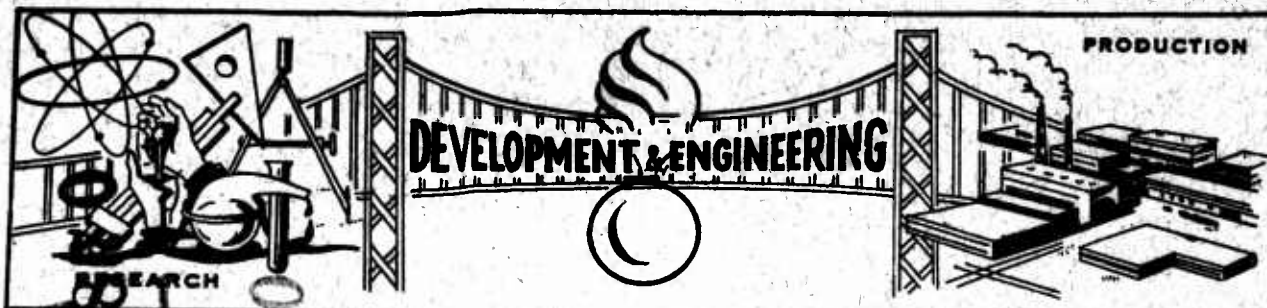
General Technical Data,
Reliability Evaluation
1. GENERAL CLASS NAME COMPLETE

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22. SIGNED / S/ Arthur Koop	23. IDEP CONTRACTOR	SUBCONTRACTOR
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CARD 1
OF 1
2. REPT. NO. 347
CLASS. 25.00.00-N5-01
SUB-CLASSES ORIG. SEQ.

36-1-5



TECHNICAL REPORT 3022

DEVELOPMENT
OF
POROSITY TEST

I. APPLICATION TO BENITE STRANDS

BY
MILTON ROTH



OCTOBER 1962

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TECHNICAL REPORT 3022
AMMUNITION GROUP

DEVELOPMENT
OF
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I. APPLICATION TO BENITE STRANDS

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The author wishes to thank the following personnel in the Explosives and Propellants Laboratory, Feltman Research Laboratories:

Mr. John Dipman -- for conducting porosity tests and making calculations.

Mr. Lester Shulman -- for providing results on the closed bomb quickness tests.

SECTION I

INTRODUCTION

Benite, an igniter material containing black power ingredients in a matrix of nitrocellulose, is used as a replacement for black powder in some cannon primers. This replacement material is usually in strands, 0.08-inches in diameter and up to 18 inches long (Reference 10).

Chemical analyses and dimensional measurements proved inadequate for predicting the performance of benite, therefore, it was necessary to supplement these tests with the closed bomb quickness test. Although the closed bomb quickness test is very reliable and its adoption gave satisfactory quality control, the method is slow, complicated and tedious.

Visual and microscopic examination of the strands revealed qualitative differences in surface texture, and it appeared reasonable to postulate that these differences affected burning rate. Porosity testing seemed logical for quantitatively evaluating such surfaces, and a literature search was made for information on this technique. Methods were found for ceramics and similar vitrified materials, but there has been little work on Ordnance materials. This report describes the adaptation of a porosimeter used for ceramics to determine the porosity of benite and includes a comparison with the quickness values obtained in the closed bomb.

SECTION II

SUMMARY

The present method for evaluating the functioning quality of benite strands is the closed bomb test for quickness. This report describes a non-destructive, faster and simpler porosity test which correlates very closely with the quickness test. The results obtained by each test from twenty lots of benite (representing satisfactory and unsatisfactory quality) were compared. A highly significant correlation coefficient of 0.92 was found. The regression equation with associated confidence limits was derived. This equation, which relates quickness and porosity results, can be used to predict one value from the other. Arrangements for using the porosity test in process control and for further comparisons with the closed bomb quickness test have been made.

SECTION III

CONCLUSIONS

1. A simple technique for porosity testing was developed.
2. The porosity test correlated closely with the quickness test when applied to twenty lots of benite strands.
3. The porosity test, fast in terms of elapsed and working times, is simple in terms of equipment, standardization and calculations required for its installation, maintenance and operation. This technique is applicable for the non-destructive testing of a wide variety of Ordnance materials and the apparatus can be modified to accommodate essentially any size or shape of sample.

~~SECTION IV~~
RECOMMENDATIONS

Actions Taken

1. A program was initiated to obtain additional data on production runs of benite prior to incorporating the test into the benite specification MIL-B-45451B (Ord). This program will include samples which are first tested for porosity and then fired in the closed bomb.
2. A survey was made to determine the applicability of porosity testing to other materials of Ordnance interest. Combustible cartridge case materials will be studied to evaluate their suitability as predicted from this test.

SECTION V

STUDY

Background

1. The burning of propellants and igniters is a function of chemical composition and physical structure. By fixing the size and shape of a given composition burning can be controlled. When structural defects are produced in the manufacturing process, the burning becomes erratic and a suitable test is needed for process control.

2. Difficulty in control of burning was frequently encountered in the production of benite, and chemical analyses were valueless for predicting such defects. As a result, the closed bomb quickness test was incorporated in the specification to control functioning quality. Although this quickness test is reliable, it is expensive in terms of equipment costs and man-hours (Reference 4 & 5). In addition, the sample is consumed in the test, and this may represent a significant cost or present a problem in retesting.

3. Macroscopic and microscopic examination of benite samples revealed that considerable variation existed in the porosity. Since porosity is an important factor among burning properties, it was assumed that a suitable measure of porosity should correlate with burning tests. Based on this assumption, a study of porosity tests seemed desirable.

2. Quadruplicate replications are obtained in less than one hour per sample. Prior to testing, the only conditioning necessary for the sample is equilibrium with room temperature.

3. The test is non-destructive, and only restriction on its use is that the pore volume of the sample must not exceed the capacity of the capillary. However, the capillary and the sample tube may be constructed to accommodate materials of almost any size, shape or pore volume (Reference 11).

Other methods of measuring porosity (such as the air pycnometer), which are dependent upon finding differences in real and apparent density or volume, introduce errors which reduce the accuracy and precision by a factor of 10 or more. Methods based on mercury intrusion (measurement of the amount of mercury forced into pores of a material at specified pressure) require elaborate, expensive apparatus and also depend upon measurements of real and apparent density. Mercury intrusion may be worthy of investigation, however, since it offers the possibility of studying the effect of changes in pressure on the structure of a material.

Discussion of Results

The data obtained from the quickness and porosity tests (Table I) was statistically analyzed by correlation and regression techniques. The correlation coefficient (r) is a measure of the extent of relationship between two variables (quickness and porosity). The regression (least squares line) is the straight line representing the best fit for related data.

The confidence limits for the regression line differ from the confidence limits on the slope. The regression line limits are of interest when the least squares line is to be used for predicting porosity from quickness, while the slope limits are applicable when substituting in the equation for this line which is shown below.

$$\text{Where: } P = 0.017Q - 2.71$$

Q = Quickness value, psi/millisec.

P = Porosity, %

Regardless of the technique used for predicting, the correlation is such that only a small difference between the measured and predicted values is expected.

Table II is designed to give an idea of the savings that can be realized by substituting the porosity test for the closed bomb test. Although the closed bomb will give information unobtainable by the porosimeter, in situations where it is simply desired to evaluate surface quality, or where both tests give similar information, the porosity test is preferable. Such a substitution will save time and money and will free the closed bomb for use in research studies.

Table III presents data showing the relationship between quickness and specific gravity. This study was included since porosity is expected to be inversely related to specific gravity. The correlation coefficient is -0.23 which, although not significant, does indicate the expected inverse relationship. Since specific gravity is a relatively insensitive test, it was not surprising that the correlation was too low to be meaningful.

SECTION V

STUDY

Background

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3. Macroscopic and microscopic examination of benite samples revealed that considerable variation existed in the porosity. Since porosity is an important factor among burning properties, it was assumed that a suitable measure of porosity should correlate with burning tests. Based on this assumption, a study of porosity tests seemed desirable.

4. A literature search revealed a lack of information on determining the porosity of Ordnance materials. However, a method described by the American Society for Testing Materials (ASTM) for vitrified materials (using a McLeod gage type porosimeter) appeared promising (Reference 6). This ASTM method is intended for use with relatively dense materials and is based on the laws of gas expansion. For this work, a readily available, inexpensive core porosimeter (Reference 7) was substituted after introducing a few modifications. A diagram of the apparatus is shown in Figure 2 and its manipulation is described under Procedure in this study.

The sample is inserted in the lower portion of the porosimeter and after replacing the top, the mercury level is raised above the level of the open stopcock. The stopcock is closed, the mercury level is lowered and the air from the pores of the sample is drawn into this Torricellian vacuum. This volume of air is readily measured in the calibrated capillary tube. The pore volume can be checked rapidly by allowing air to be reabsorbed and repeating the cycle of operations.

The advantages in using this method are:

1. Only a single reading is required to determine the pore volume of the sample. The accuracy is about 0.01 ml. (Reference 11) and is independent of the size and porosity of the sample.

2. Quadruplicate replications are obtained in less than one hour per sample. Prior to testing, the only conditioning necessary for the sample is equilibrium with room temperature.

3. The test is non-destructive, and only restriction on its use is that the pore volume of the sample must not exceed the capacity of the capillary. However, the capillary and the sample tube may be constructed to accommodate materials of almost any size, shape or pore volume (Reference 11).

Other methods of measuring porosity (such as the air pycnometer), which are dependent upon finding differences in real and apparent density or volume, introduce errors which reduce the accuracy and precision by a factor of 10 or more. Methods based on mercury intrusion (measurement of the amount of mercury forced into pores of a material at specified pressure) require elaborate, expensive apparatus and also depend upon measurements of real and apparent density. Mercury intrusion may be worthy of investigation, however, since it offers the possibility of studying the effect of changes in pressure on the structure of a material.

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The data obtained from the quickness and porosity tests (Table I) was statistically analyzed by correlation and regression techniques. The correlation coefficient (r) is a measure of the extent of relationship between two variables (quickness and porosity). The regression (least squares line) is the straight line representing the best fit for related data.

In calculating the correlation coefficient and the regression equation, the quickness value was considered as the independent variable or predictor, and the porosity value as the dependent variable. The calculated value for the correlation coefficient was 0.92; and a test of significance (Reference 8) indicated that a real relationship existed between the two variables.

A regression equation with the associated confidence limits was calculated by the method of least squares and fitted to the data in Figure 1. This equation can be used not only for estimating the porosity value when the quickness is known but also for calculating specification limits for porosity which will be equivalent to those used for quickness.

The regression line brings out several interesting points. First, the fact that the line does not pass through the origin is expected since the benite obviously would burn even if nonporous. Second, the slope of the line indicates the effect of porosity on quickness. Thus, for consistent performance it is necessary to control porosity.

Confidence limits for the regression line are shown by the dotted lines in Figure 1. These limits encompass a range within which any single additional data point would fall 95% of the time. These limits are different for each value of the independent variable and are at a minimum for the average of the independent variable. Figure 1 shows that the confidence interval encloses all points and the interval is sufficiently narrow so predictions can be made within small limits.

The confidence limits for the regression line differ from the confidence limits on the slope. The regression line limits are of interest when the least squares line is to be used for predicting porosity from quickness, while the slope limits are applicable when substituting in the equation for this line which is shown below.

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Table III presents data showing the relationship between quickness and specific gravity. This study was included since porosity is expected to be inversely related to specific gravity. The correlation coefficient is -0.23 which, although not significant, does indicate the expected inverse relationship. Since specific gravity is a relatively insensitive test, it was not surprising that the correlation was too low to be meaningful.

Figure 2 shows the apparatus developed for this study. Basically, it is very similar to the commercially available core porosimeter (Reference 7). However, it has been modified to include a sample barrier which prevents the specimen from entering the capillary tube when the mercury level is raised and to provide a straight glass tubing at the top. The graduated portion of the capillary tube also differed from that on the commercial model in that the total capacity was 1.5 mls. rather than 3.5 mls. The barrier was made by pushing three or four points into the wall of the tube near the shoulder, and inserting a flat piece of polyethylene (cut from a bottle and punched with small holes) above these points. This apparatus is suitable for relatively small specimens, but design modifications to accommodate specimens of almost any size or shape can be readily made. With larger apparatus, the vacuum is obtained by a suitable pump rather than by manipulating the mercury reservoir.

Photomicrographs of benite (Figure 3) show cracks, pores and other voids which are measured as porosity if they open on the surface. Since the diameter of benite strands is quite small, it is likely that a great majority of the pores do open on the surface. In any event, surface porosity is an estimate of the overall structure and the functioning quality. The results indicate that the porosity test will distinguish between satisfactory and unsatisfactory material. For example, all samples exceeding the specification limit for quickness were above 13% porosity, while all samples meeting the quickness requirement were below 13% in porosity (Table II).

EXPERIMENTAL PROCEDURE

A. Apparatus

Modified McLeod gage type porosimeter, (Figure 2).

B. Materials

Mercury metal - American Chemical Society Grade

C. Procedure:

Break the benite into pieces about two inches long, and weight out approximately 5 grams. Place the sample in the lower portion of the porosimeter. Any moisture which may be entrapped on the inner surfaces of the porosimeter or on the sample shall be removed before determinations are made. To remove this moisture, raise the leveling bulb until the mercury in the capillary is above the stopcock. Close the stopcock and lower the leveling bulb so that the sample is under vacuum for at least one minute. Any moisture inside the porosimeter vaporizes and by raising the leveling bulb again the gas is collected in the capillary and finally expelled by opening the stopcock. Connect the top of the capillary to a drying column so that only dry air is drawn into the apparatus.

Immediately after removing the moisture, lower the leveling bulb exposing the sample to the dry air for at least one minute. Raise the leveling bulb until the mercury in the capillary is above the stopcock and then close the stopcock. Lower the leveling bulb to expose the entire sample to the vacuum for at least one minute. Raise the bulb again to collect the air in the capillary tube and read the volume when the mercury surface in the

leveling bulb is on a level with the meniscus in the capillary. This is the pore volume.

Make five determinations of the pore volume as quickly as possible using the same sample and consider the average as the pore volume (v). The range of values obtained for pore volume should not exceed 10% of the average. Lack of agreement among these values may be traced to leaks or moisture in the apparatus.*

The total volume (V) of the sample should be measured by a volumeter, or calculated from the specific gravity. The percentage of pore volume shall be calculated as the volume of gas contained in the total volume of the sample:

$$\text{Percentage of pore volume} = 100 \frac{v}{V}$$

where: v = Pore volume of the sample, ml

V = Total volume of the sample, ml

*NOTE: To test for leaks in the apparatus proceed as follows: After taking a reading, expel all gas from the apparatus. Close the stopcock, then raise and lower the leveling bulb several times. The level of the mercury in the capillary should rise to the stopcock when the leveling bulb is returned to its original position.

CALCULATIONS

Calculation of the statistical parameters was based on the following equations (Reference 9):

Correlation Coefficient,

$$r = \frac{\overline{XY} - (\overline{X})(\overline{Y})}{\sqrt{[\overline{X^2} - (\overline{X})^2][\overline{Y^2} - (\overline{Y})^2]}}$$

Least squares line,

$$Y = mX + b$$

Slope,

$$m = \frac{\overline{XY} - (\overline{X})(\overline{Y})}{\overline{X^2} - (\overline{X})^2}$$

Y-intercept,

$$b = \overline{Y} - m\overline{X}$$

Confidence limits on slope,

$$S_m = \frac{m \sqrt{\frac{1}{r^2} - 1}}{N - 2}$$

Confidence limits on line,

$$S_{Yi} = S_Y \sqrt{1 + \frac{1}{N} + \frac{(\overline{X} - X_i)^2}{N [\overline{X^2} - (\overline{X})^2]}}$$

NOMENCLATURE

b = Y-intercept

m = slope

N = Number of data points

r = Correlation Coefficient

S_m = Confidence limits on slope

S_y = Standard error of estimate for dependent variable

S_{yi} = Standard error of estimate for a single value
of the dependent variable

X_i = Particular value of the independent variable

Y_i = Particular value of the dependent variable

$\overline{X}, \overline{Y}$ = Averages of the independent and dependent
variable

$\overline{X^2}, \overline{Y^2}$ = Averages of the squared values of the variables

$\overline{XY}$ = Average of the products of all data points

All calculations apply only to the regression of Y on X
since it was assumed that X (Quickness) is an accepted stand-
ard which Y (Porosity) must predict.

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APPENDICES

APPENDIX A

TABLES

TABLE I

COMPARISON OF QUICKNESS WITH POROSITY OF BENITE

<u>Lot No., (PA)</u>	<u>Mix No.</u>	<u>Quickness (psi/millisec)</u>	<u>Pore Volume (ml/5g)</u>	<u>Porosity (Percent)</u>
PA-6-14	3	1640	0.80	27.21
PA-6-13	3	1320	0.61	20.82
PA-6-14 Sple#	81	1225	0.43	14.63
PA-6-13	2	1205	0.61	20.75
PA-6-15 Sple#	93	1160	0.40	13.61
PA-6-13	4	1145	0.47	15.99
PA-6-19	4	830	0.25	8.47
PA-6-17	2	795	0.36	12.20
PA-6-18	4	785	0.31	10.54
PA-6-10	-	770	0.28	9.52
PA-6-18	3	760	0.27	9.18
PA-6-19	5	750	0.28	9.49
PA-6-19	3	730	0.21	7.12
PA-6-18	1	720	0.35	11.90
PA-6-17	1	715	0.30	10.17
PA-6-16	122	705	0.38	12.93
PA-6-19 Sple#	6	690	0.32	10.85
PA-6-19	1	690	0.26	8.78
PA-6-18	2	680	0.25	8.50
PA-6-19	2	660	0.26	8.81

Correlation Coefficient $r = 0.92$

Regression equation $P = 0.0170 Q - 2.71$

95% Confidence limits
on slope $m = 0.0170 \pm 0.0151$

TABLE II

COMPARISON OF ECONOMIC FACTORS INVOLVED IN THE
CLOSED BOMB AND POROSITY TEST

<u>FACTOR</u>	<u>CLOSED BOMB TEST</u>	<u>POROSITY TEST</u>
Conditioning Time, hrs.	4	None
Total Test Time, hrs.	4	1 (2)
Working Time, hrs.	4	1
Standardization	Required (PATR 2367)	None
Tolerances in Sample Size and Shape	Limited	Flexible
Calculations of Results	Complex	Simple
Instrumentation	Complex (1)	None
Cost of Equipment	Over \$15,000	Under \$100

(1) Closed bomb, piezo-electric gauge, cathode-ray oscillograph and associated electrical apparatus, and a recording camera for making a permanent firing record.

(2) Time for five replications.

TABLE III

COMPARISON OF QUICKNESS AND SPECIFIC GRAVITY

<u>Lot No., (RAD)</u>	<u>Quickness (psi/millsec)</u>	<u>Specific Gravity</u>
2-3 (61)	946	1.577
1-10 (62)	879	1.625
1-9 (62)	850	1.619
1-2 (62)	848	1.537
1-11 (62)	848	1.611
1-1 (62)	837	1.570
1-13 (62)	836	1.592
2-6 (62)	806	1.538
2-1 (61)	801	1.607
2-2 (61)	798	1.628
1-19 (62)	791	1.581
1-3 (62)	773	1.564
2-7 (62)	717	1.589
1-16 (62)	713	1.550
1-7 (61)	710	1.611
2-11 (62)	698	1.577
2-1 (62)	691	1.578
3-1 (62)	678	1.580
1-18 (62)	656	1.620
1-17 (62)	633	1.615
2-10 (62)	576	1.640

Correlation coefficient = -0.23

APPENDIX B

FIGURES

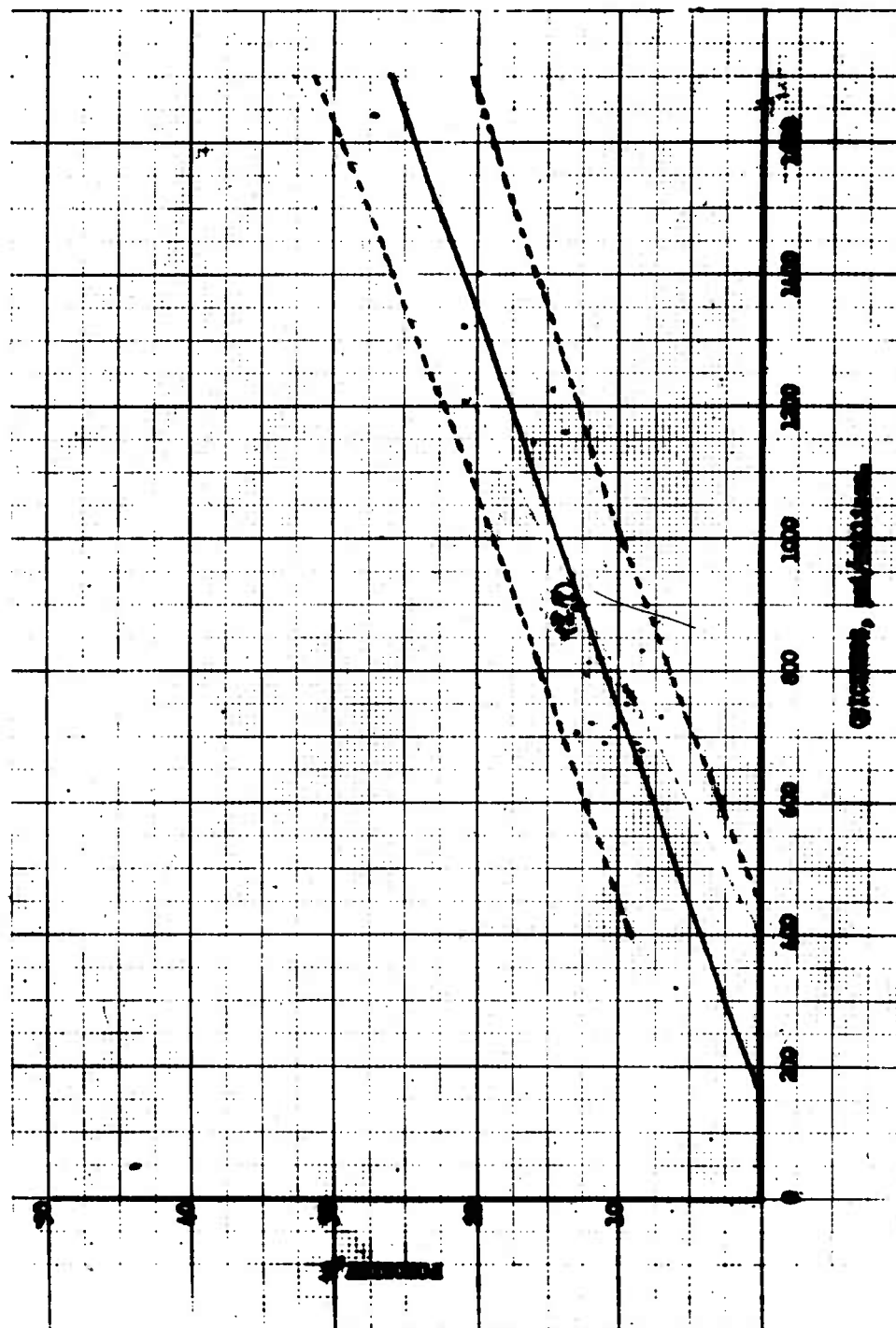


Figure 1 - Scatter Diagram and Regression Line of Porosity on Quickness

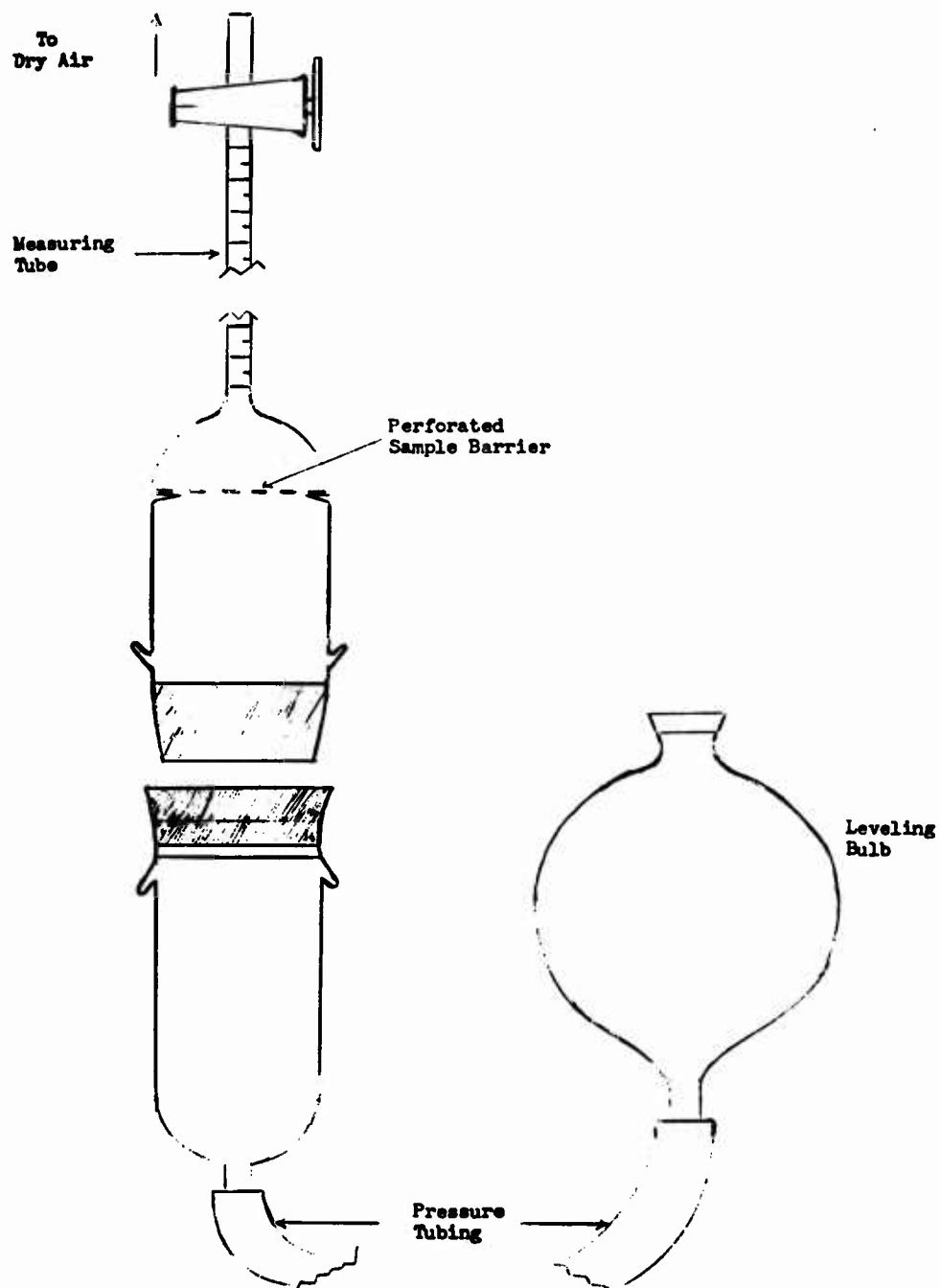


Figure 2. Porosimeter

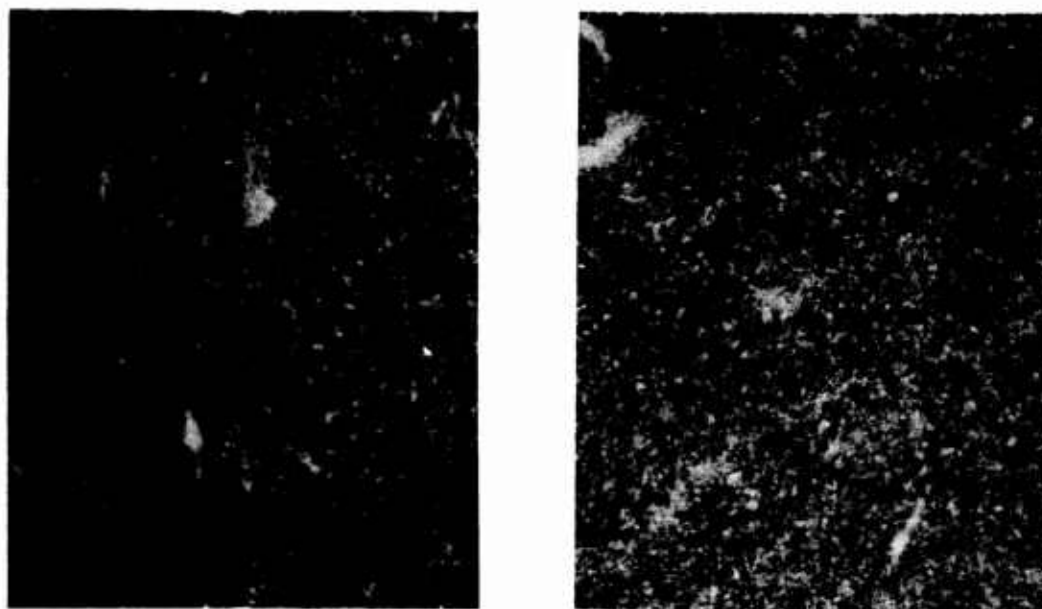


Figure 3. Benite Sections Showing Porous Structure

ABSTRACT DATA

ABSTRACT

Accession No. _____ AD _____

Picatinny Arsenal Dover, New Jersey

DEVELOPMENT OF POROSITY TEST: APPLICATION TO BENITE STRANDS

Milton Roth

Technical report 3022, October 1962,
16 pp, figures, tables. Unclassified
report from the Process Engineering
Laboratory of the Ammunition Group.

The present method for evaluating the
functioning quality of benite strands is
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1. Porous materials--Test
methods.
2. Benite--Porosity.
- I. Roth, Milton.

UNITERMS

Porosity
Benite
Quickness
Testing
Closed bomb test
Roth, M.

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(over)

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1. Porous materials—
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I. Roth, Milton

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