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THE CANISTER TEST FOR EVALUATING THE COKING PROPERTIES OF METALLURGICAL COALS

by

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INTRODUCTION

The canister test enables the coking properties of small samples (∿2 lb) of metallurgical coals to be evaluated under the conditions of heat transfer that obtain in the CANMET 12-inch movable-wall coke oven. It should find application, for example, in the evaluation of exploration drill-core samples, in the evaluation of a multiple of blend possibilities prior to 500 lb confirmatory tests, and in research programs such as the addition of antifissuring agents and formed coal to coke oven blends.

The test involves carbonizing the coal samples in cylindrical perforated canisters placed within a coal matrix in the 12-inch oven. After carbonization and pushing, the canisters are retrieved and the resultant coke cylinders (in effect, two half-oven pieces) are shattered. Disintegration indices from the shatter test are correlated with ASTM stability and hardness factors.

The test was conceived with the development of the controlled bulk-density side-charge box for use in the 12-inch oven. Several materials for canister fabrication were tried (for example, asbestos), but canisters made from perforated mild steel sheet with end caps were found to be most successful in spite of the possibility of undesirable heat transfer effects because of conduction. This technique for carbonizing small samples was first used in a program involving coke manufacture from de-watered coal-oil slurries[1], but the dynamic coke reactivity method (reaction with carbon dioxide at 1050°C while mildly tumbling) used for evaluating the resultant cokes could not be correlated to meaningful coke quality factors. This difficulty led to the development of a physical disintegration test for evaluating cokes from the canisters which could be related directly to ASTM stability and hardness factors[2].

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One of the best known early methods for evaluating the physical properties of small coke samples is known as the Blayden Micro-Hardness Test[3]. In this test, coke (2g) is shattered in a tube (1-in dia. x 12-in long, 25 rpm, 800 revolutions) rotating like a propellor and containing 12 steel balls (5/16-in diameter). The test results were expressed as the percentage of the original coke remaining on No. 25 and 72 British Standard sieves. The Blayden Micro-Hardness Test has been modified and extended at Bituminous Coal Research, Inc., Pittsburgh. Small coal samples are carbonized (at a rate of approximately 30 - 40°F through the fluid range) in cylinders surrounded by coke breeze. The micro-hardness test is done on a composite sample of selected size fractions after carbonization and the percent size reduction representing the change in average particle diameter of the tumbled coke is correlated linearly with the ASTM stability factor. This technique has also been adopted successfully by Dominion Foundries and Steel Limited, Hamilton, and a linear correlation between the BCR stability and ASTM stability as determined in the CANMET 12-inch oven is available[5]. Ignasiak and Berkowitz[6] have recently reported a bench-scale method for preparing structurally-homogeneous coke discs under an applied pressure, a coke strength index being obtained from the results of a tumble test using a porcelain jar containing steel balls.

METHOD

(i) Canister Charging

The canisters (3-in dia. x 11.5-in long, approximately 40 percent void area) hold approximately 80-in^3 of coal. Coal is prevented from passing through the perforations in the canisters by a paper lining. The sample (1050g) prepared to a size consist of 80-85 percent minus 1/8-in gives a canister bulk density of 50 lb/ft^3. Canisters are labelled with metal tags for retrieval after carbonization.

(ii) Carbonization

The canisters (up to 22 per carbonization test) are placed in rows and surrounded by the matrix coal in the side-charge box (9.4 ft^3, 1/2-in plywood ends, 1/8-in Masonite sides). Four rows of canisters are separated from one another and from the top and bottom of the box by approximately 3-in of matrix coal.

The side-charge box is inserted into the 500-lb oven and the resultant coke is pushed from the oven and water-quenched approximately 30 minutes after a centre-oven temperature of 1850^{o}F has been reached. The coke is dried overnight.

(iii) Physical Testing of the Canister Coke

After drying, the coke cylinders from the canisters are weighed to give the coke yield. Half of the coke cylinders (half-oven piece), weighing 300 ± 10g are then taken for the physical disintegration test. A preliminary shattering of the sample is done by dropping a weight (15kg) onto the sample down a pipe (5.25-in dia. x 3-ft long). This procedure is usually repeated. The coke is screened through 1-1/2, 1, 3/4, 1/2, 1/4 and 1/6-in sieves, recording the mass retained on each sieve. The material passing the 1/6-in sieve is discarded. The remainder is placed in a shatter box (12-in x 7-in x 9-in high) and shattered again for 20 minutes (720 cycles per minute) using a paint shaker. The appropriate disintegration conditions had been ascertained from plots of shattering and breakage against time, which showed that disintegration was essentially complete after 15-18 minutes. The resultant coke is then screened again, using the same sieves as above, and the amount of minus 1/6-in material is taken as the difference between the mass of material charged to the shatter box and the cumulative amount of material on the sieves. The number of coke pieces retained on each sieve is recorded.

(iv) Calculation of Disintegration Indices

An approximate value for the apparent specific gravity of the shattered coke is found by displacement of water in a graduated cylinder (most cokes tested had an apparent specific gravity within the range 0.8 - 1.2g/cc).

The disintegration index (ψ) corresponding to the ASTM stability factor is given by the ratio (expressed as a percentage) of the mean coke lump diameter (plus 1/6-in material) after shattering and the mean lump diameter before shattering. This ratio is similar to the index adopted by the Bituminous Coal Research, Inc., (mentioned previously), which expresses the change in average particle diameter. The mean lump diameter based on volume of the coke pieces retained on each sieve is calculated as follows:

Mean volume of each coke piece retained on sieve

$$= \frac{\text{mass of coke pieces on sieve}}{\text{number of pieces x A.S.G.}}$$

$$V = \frac{m}{n \times A.S.G.}$$

For a spherical shape factor:

$$V = \frac{4}{3}\pi\left(\frac{d}{2}\right)^3$$

Where d is the mean diameter of coke pieces on the screen

$$d^3 = 1.91\ V$$

$$3 \ln d = \ln 1.91\ V$$

$$d = e^{\left(\frac{\ln 1.91\ V}{3}\right)}$$

If $(x_1, x_2, x_3, \ldots x_n)$ is the number of lumps of coke of diameters $(d_1, d_2, d_3 \ldots d_n)$, then the mean coke lump diameter is given by

$$\bar{d} = \frac{\sum_1^n d_n x_n}{\sum x}$$

and the disintegration index corresponding to the ASTM stability factor is expressed by

$$\psi = \frac{\bar{d} \text{ after shattering}}{\bar{d} \text{ before shattering}}$$

The repeatability of the method for duplicate samples is illustrated in Figure 1.

The disintegration index (θ) corresponding to the ASTM hardness factor is expressed as a percentage of the cumulative mass retained on the sieves after shattering.

(v) Correlation of Canister Disintegration Indices with ASTM Stability and Hardness Factors

Correlation data between canister disintegration indices (ψ and θ) and hardness factors (S and H) were obtained from canister tests using

metallurgical coal blends which had previously been carbonized at the 500-lb scale in the movable-wall oven.

Because the rate of heat transfer during coal carbonization affects the quality of the resultant coke, it is necessary to state the carbonization conditions under which the tests were done. Standard conditions in the CANMET 12-inch oven are coking rate:1.2-in per hour, oven bulk density:51 lb/ft^3, and carbonization until the attainment of a centre temperature of $1850^{o}F$.

Figure 2 shows the correlation obtained between ψ and S, the canister stability index and the ASTM stability factor. Statistical regression analysis of the data yielded the line of best fit as a parabolic relationship:

$$\psi = -140.500 + 7.880S - 0.067S^2$$

Differentiation of this equation yields:

$$\frac{d\psi}{dS} = 7.880 - 0.134S$$

Equating $d\psi/dS$ to zero indicates a maximum at $\psi = 91.3$ when $S = 58.8$ in the parabola drawn through the datum points. Because values of $\psi > 91.3$ were obtained, the line of next best fit (a semi-logarithmic relationship) was ultimately used for the ψ - S regression equation (the correlation coefficient for this equation was > 0.9):

$$\psi = -229.829 + 80.284 \ln S$$

Approximately 86 percent of the datum points in Figure 2 fall within $\pm$ 3 units of S of the semi-logarithmic regression.

Figure 3 shows the correlation obtained between θ and H, the canister hardness index (percent cumulative mass greater than 1/6-in) and the ASTM hardness factor. Statistical regression analysis of the data yielded the line of best fit as a linear relationship:

$$\theta = -43.060 + 1.630 H$$

Although 83 percent of the datum points fall within $\pm$ 3 units of H of the linear regression, the correlation coefficient (0.56) is low because the range of experimental values of θ (56-72) and H (56-71) is narrow.

ACKNOWLEDGEMENTS

The writer acknowledges with gratitude the major contribution made to the development of the canister test method by Mr. D. DiCredico, formerly Technologist in the Canadian Metallurgical Fuel Research Laboratory, ERL,

CANMET, and now with the Iron Ore Company of Canada, Labrador City, Newfoundland. Mr. DiCredico developed the method of expressing the canister disintegration index (ψ) in terms of mean lump coke diameters based on volume and obtained the correlation equations between ψ and S, and θ and H. The writer also wishes to thank Mr. R.S. Gale for assisting Mr. DiCredico. Mr. K.F. Hampel, Technologist, is continuing with this program.

As alluded to on the first page, the basic philosophy behind this test procedure was conceived by J.C. Botham and E.W. Montgomery.

REFERENCES

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2. D. DiCredico, Canadian Metallurgical Fuel Research Laboratory, Energy Research Laboratories, CANMET.
   Development of the physical disintegration test will be given in a later report. The work is being continued by K.F. Hampel.

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4. R. Moses, Bituminous Coal Research, Inc., Pittsburgh, Private Communication.

5. Minutes of the Canadian Carbonization Research Association, Fifty-Seventh Technical Committee Meeting, September 4-5, 1975.

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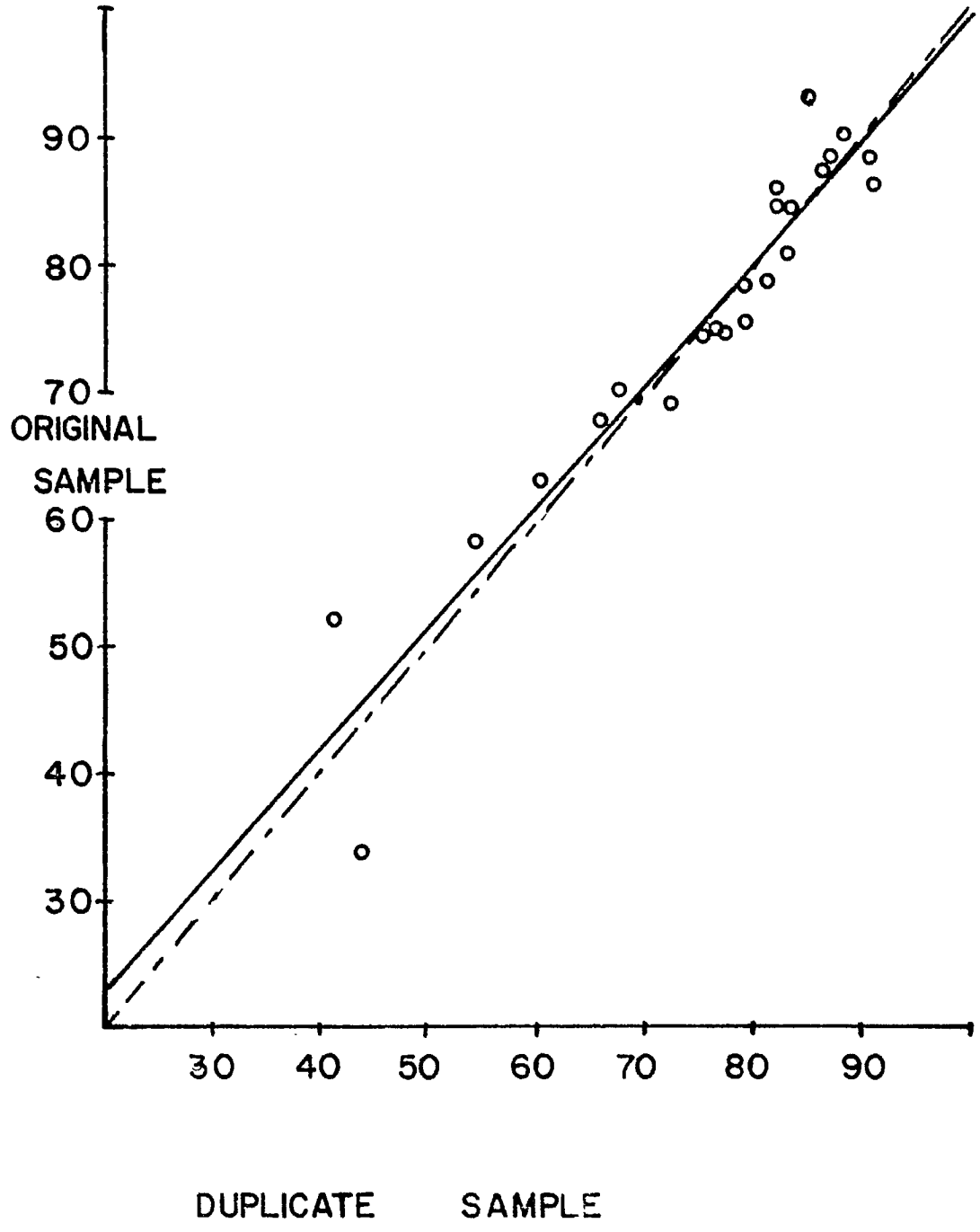

FIGURE 1

REPEATABILITY OF DISINTEGRATION INDICES
FROM DUPLICATE CANISTER TESTS

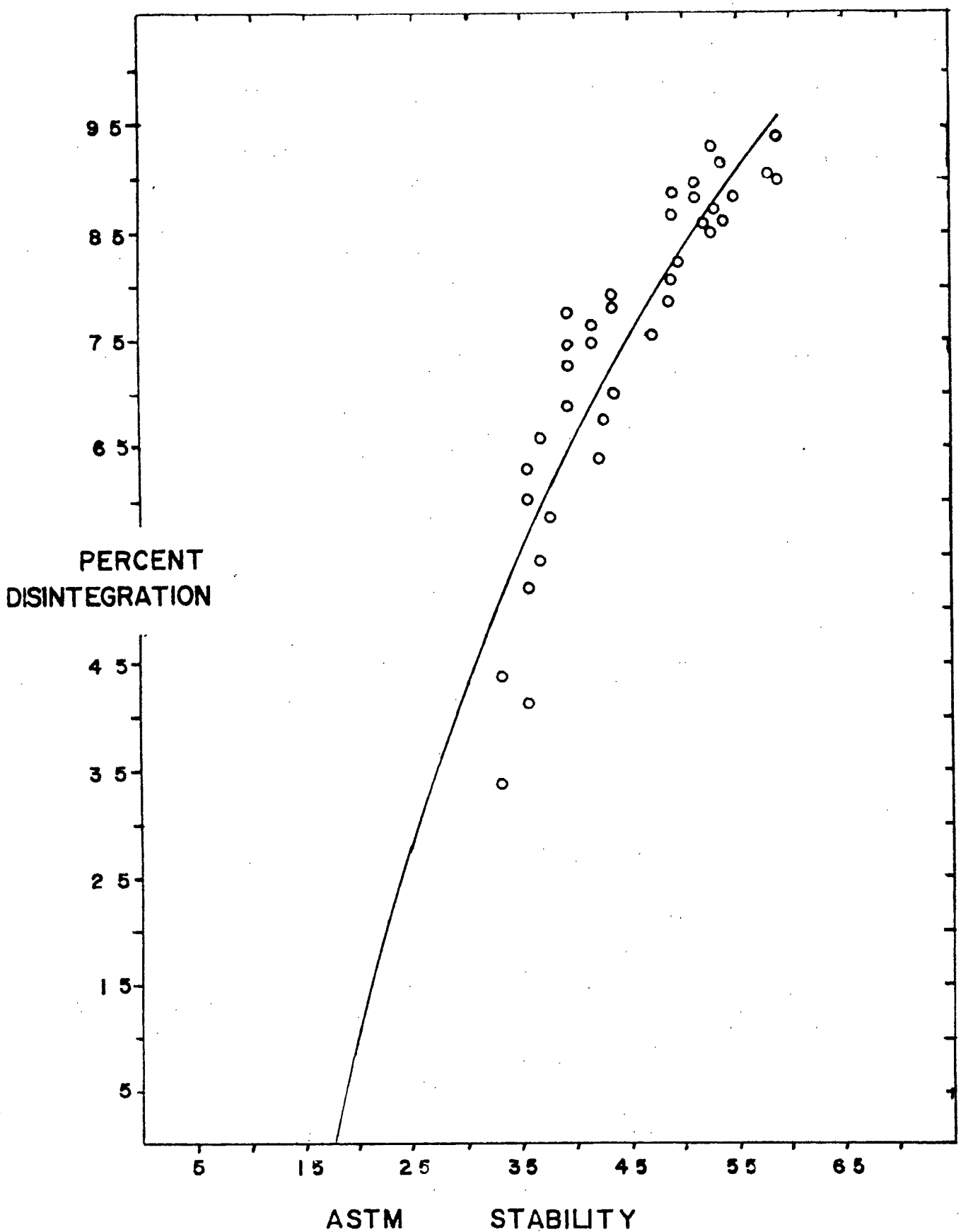

FIGURE 2

CORRELATION BETWEEN CANISTER STABILITY INDEX
AND ASTM STABILITY INDEX

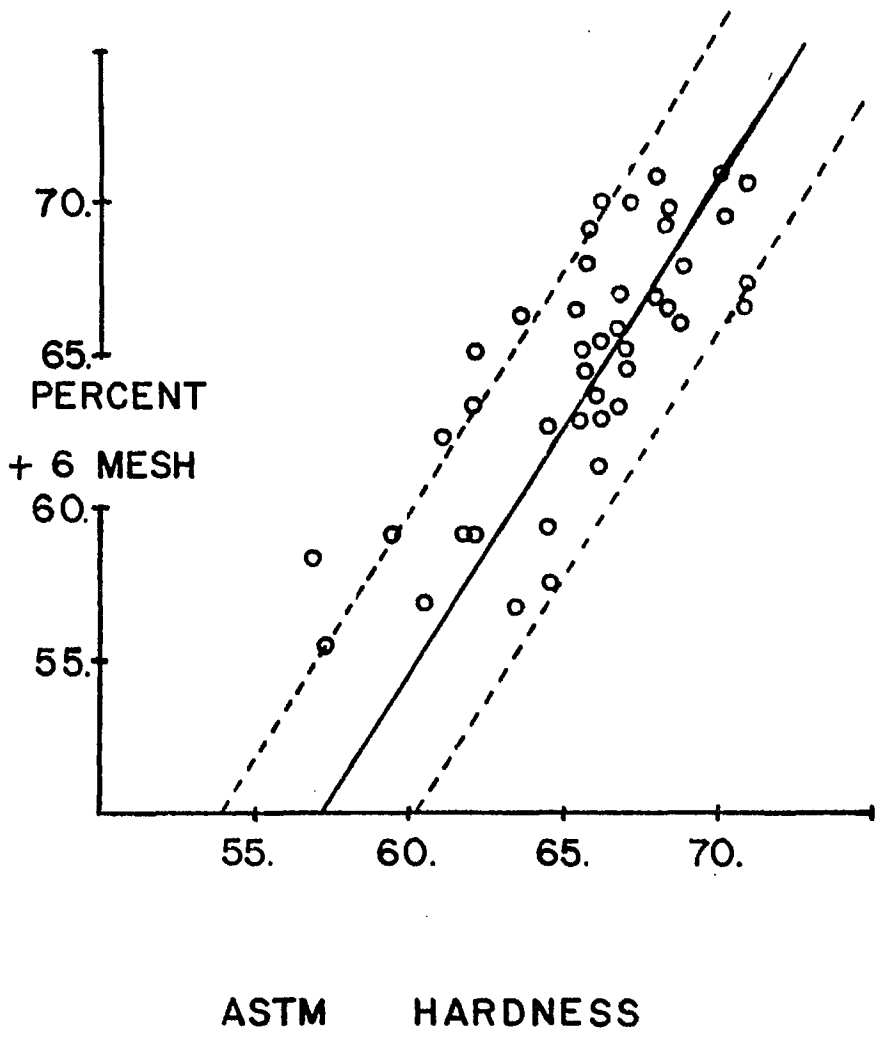

FIGURE 3

CORRELATION BETWEEN CANISTER HARDNESS INDEX
AND ASTM HARDNESS INDEX