Weathering characteristics of coating-grade asphalts

Jones, P. M.; Gibbons, E. V.

Publisher's version / Version de l'éditeur:

Proceedings of the Eighth Annual Conference of the Canadian Technical Asphalt Association: 1963-11, 8, pp. 89-102, 1965-03-01

NATIONAL RESEARCH COUNCIL
CANADA

DIVISION OF BUILDING RESEARCH

WEATHERING CHARACTERISTICS OF COATING-GRADE ASPHALTS

BY

P. M. JONES AND E. V. GIBBONS

REPRINTED FROM
PROCEEDINGS OF THE EIGHTH ANNUAL CONFERENCE OF THE CANADIAN TECHNICAL ASPHALT ASSOCIATION, VOL. VIII, NOVEMBER 1963, P. 89 - 102.

RESEARCH PAPER NO. 241
OF THE
DIVISION OF BUILDING RESEARCH

OTTAWA

PRICE 25 CENTS

MARCH 1965

NRC 8399

This publication is being distributed by the Division of Building Research of the National Research Council. It should not be reproduced in whole or in part, without permission of the original publisher. The Division would be glad to be of assistance in obtaining such permission.

Publications of the Division of Building Research may be obtained by mailing the appropriate remittance, (a Bank, Express, or Post Office Money Order or a cheque made payable at par in Ottawa, to the Receiver General of Canada, credit National Research Council) to the National Research Council, Ottawa. Stamps are not acceptable.

A coupon system has been introduced to make payments for publications relatively simple. Coupons are available in denominations of 5, 25 and 50 cents, and may be obtained by making a remittance as indicated above. These coupons may be used for the purchase of all National Research Council publications including specifications of the Canadian Government Specifications Board.

Weathering Characteristics of Coating-Grade Asphalts

By P. M. Jones and E. V. Gibbons*

Few of the residual asphalts produced from crude oil sources are suitable for roofing purposes because of their wide variations in quality. Those that are selected are further processed and segregated to provide the coating grade asphalts used in the Canadian roofing industry.

Final processing involves air blowing the soft residue. The resultant product usually has a softening point between 210 and 230°F and a penetration at 77°F of between 15 and 25 dmm. To provide satisfactory service the asphalt must possess suitable consistency characteristics for the preparation of shingles and other products. It must also be capable of resisting changes in its properties upon exposure to the weather. The nature of these changes has been studied and discussed by many workers: Abraham[1] in summarizing the work of several workers describes the changes as oxidation, dehydrogenation, carbonization, polymerization and evaporation; Beitchman[2], Kleinschmidt and Snoke[3], Galloway[4], Pfeiffer[5] and others have described some of the changes that occur to asphalts during exposure. In general, a loss in weight was observed and the asphaltene fraction increased at the expense of the maltene fraction. Greenfeld[6] has made a detailed study of the chemical changes two asphalts undergo upon weathering. He concludes that the changes are complex and numerous, that oxidation occurs resulting in the formation of volatile products such as carbon dioxide and water, acidic water soluble products and a product that is insoluble in water an n-pentane. Stewart[7] used infrared analysis to study chemical changes and noted an increase in the carbonyl content as degradation proceeded. Wright and Campbell[8] have used this change in carbonyl content as a means of measuring degradation in studies of the parameters in accelerated weathering. A review of these and many other studies on weathering of asphalts has recently been made by Martin[9].

Although most of these studies have investigated the chemical nature of the changes accompanying weathering, the most important effect from a practical point of view is the increase in hardness of the asphalt. It has been shown by Mertens and Greenfeld[10] that this increase depends upon the nature of the crude from which the asphalt is obtained and upon the method of manufacture. The asphalt hardens progressively until it is unable to withstand strains imposed by thermal and moisture changes or wind vibrations and cracking or fracture results. When this occurs the waterproofing ability of the asphalt coating is lost and deterioration of the underlying fabric may be accelerated. Hardening of the asphalt may also result in the loss of protective mineral granules from the surface so that more asphalt is exposed to radiation, which further accelerates degradation. The nature of the physical and chemical changes that give rise to these effects is known in general, but little information is available of the way in which they are related to the physical properties and chemical composition of the materials.

Mertens[11] has reported a correlation between weatherometer life and the dispersibility of the asphaltenes. This concept has been used by Jones[12]

*P. M. JONES and E. V. GIBBONS, Division of Building Research, National Research Council, Ottawa, Ont.

in studies with coating grade asphalts. A good correlation is found between the dispersibility of the asphaltenes and the durability of coating grade asphalts produced from straight run, crude blend, oil fluxed residues and residue catalyzed with ferric chloride.. No correlation was found for asphalts catalyzed with phosphoric acid and each of the production methods gave different correlation equations.

Heithaus[13] claims that the range of the dispersibility values in Mertens' study is too small to be of major practical importance; that penetration has been correlated with durability and that maltene viscosity is correlated with durability[14]. In an extensive study Hamada[15] has obtained correlations between durability and penetration, ductility, softening point and density and also gives the following relationship: if flash point (°C) — 2 × softening point (°C) $\geqslant$ 120°C, the probability of having a good weather resistant asphalt is high.

At the Division of Building Research of the National Research Council of Canada a study has been made of 15 coating-grade asphalts. This report presents a summary of their properties and the influence of these properties upon their performance as weather resistant coatings.

MATERIALS AND METHODS OF TEST

Fifteen asphalts were obtained in 1959 from widely distributed crudes used in the production of asphalt shingles and other prepared roofing and siding products. Ten were from Canadian crudes, three from Venezuela and two from the Middle East. The properties and sources of the asphalts are listed in Table I; it may be noted that several are from the same source —for example, asphalts No. 3 and 7 are both from Lloydminster, with a three-year time interval, and asphalts No. 8 and 14 are from southern Alberta crudes. The original sample No. 14 (durability 22 cycles) was improved by different blending techniques to produce No. 8, which has a durability of 50 cycles.

CHEMICAL ANALYSIS

A chromatographic procedure (Kleinschmidt[16]) was used to separate the asphalts into components. The asphaltene fraction was separated as that portion insoluble in n-pentane, and the remaining maltene fraction was further subdivided by liquid chromatography. An activated fullers earth column was used, white oils being eluted with n-pentane, dark oils with methylene chloride, and resins with water-saturated methyl ethyl ketone. The final fraction, removed with a mixture of acetone and chloroform, was included with the resin fraction for purposes of reporting. The composition of the asphalts before and after weathering as determined in this manner is shown in Table II. The elemental composition of the asphalts was kindly determined by Mr. W. J. Montgomery of the Fuels and Mining Practice Division of the Department of Mines and Technical Surveys and is summarized in Table III.

PREPARATION AND EXPOSURE OF TEST PANELS

The asphalts were exposed for natural and accelerated studies as films $2\frac{1}{4}$ in. by $5\frac{1}{2}$ in. by 25 mils on aluminum panels prepared by the hydraulic press method[17]; a teflon-coated glass fabric is used instead of dextrin paper to keep the asphalts from adhering to the heated plates. In the accelerated durability test four panels were exposed in a single-arc weatherometer operated in accordance with ASTM D529-59T, Cycle A; the panels are subjected to 51 minutes of carbon-arc radiation, producing a black panel temperature of 140°F, followed by nine minutes of radiation with water at 45 $\pm$ 5°F sprayed onto the panels. The hourly cycle is repeated 22 times to produce a single daily cycle, and daily cycles are repeated five days a week. To overcome variations in the day-to-day performance of the weatherometer the panels were exposed at different times. This method of exposure has been found to be statistically the equivalent of exposing one panel of each asphalt in four different machines at the same time[18].

During exposure the panels were rotated according to the plan described in ASTM D529-59T and were tested for the development of flaws every five cycles with a high voltage probe[19]. A photograph of the flaws was examined with a 60-square grid and the panel was considered to have failed when 50 per cent of the squares showed at least one flaw.

The operation of the weatherometer equipment produced results comparable with those reported by Mertens[11] who reported a correlation of $Y = 223.8X - 86.1$, where Y is the durability and X is the dispersibility of the least soluble asphaltenes. A correlation using the fifteen asphalts described previously and exposed in the equipment at NRC produced a correlation of $Y = 211.9X - 84.9$. This indicates that one asphalt tested in the two different laboratories would differ by about six cycles.

Natural durability studies were made on eleven of the asphalts. Four panels of each asphalt were exposed at an angle of 45 deg. to the vertical, facing south, at three exposure sites operated by the Division of Building Research[20]. The panels were examined at regular intervals in a manner similar to that used for the accelerated durability tests.

DISCUSSION OF RESULTS FROM ACCELERATED DURABILITY STUDIES

Although the crudes were from widely different sources, they were processed to meet specifications so that they present a very narrow range of physical characteristics. Some studies by Wikinson, Striker and Traxler[21] have resulted in suggested criteria for establishing the quality of asphalts with respect to durability. These limits are shown in Table IV, and if applied to the asphalts under study would eliminate all but No. 24, and 10. Greenfeld[22] reports that all but one of the 15 asphalts he studied would be excluded by these limits.

In an effort to obtain the relationship between physical properties and durability, studies have been made on the correlation coefficients. This is a measure of the degree of correlation between the measured values and the equation of a straight line that best fits these values. The correlation coefficient r cannot exceed +1 nor be less than —1 in value. A value of +1 denotes perfect functional relationship between **x** and **y**, an increase in **x** being associated with an increase in **y**. A value of —1 is also a perfect functional relationship, with increase in **x** associated with a decrease in **y**. When **r** is zero there is no relation between **x** and **y** and values of **r** between +0.5 and —0.5 are considered to indicate no significant relationship between **x** and **y**.

This approach was used on the best straight lines that could be plotted between durability and the following: softening point, penetration at 32, 77 and 115°F, ductility, density, weight loss at 10 cycles, the ratio of resin to asphaltenes, resins plus asphaltenes to total oils, carbon to hydrogen, and the sum of carbon plus hydrogen. The values of the various equations and **r** are shown in Table V. The results show that no significant correlation exists between durability and most of the physical and chemical parameters measured. There is some significant correlation with penetration at 77 and 115°F. A plot of these values in Figure 1 indicates a wide scatter so that the correlation may be one of coincidence.

An excellent correlation exists between ductility at 77°F and durability. This is shown in Figure 2 in which records for 13 of the 15 asphalts are plotted. Asphalts No. 1 and 14 were omitted as showing extreme results, so that the value of r was increased from 0.81 to 0.95. The equation for the line $\mathbf{Y} = 23.1\mathbf{X} + 8.4$, where **Y** equals durability in days, and **X** equals ductility in cm. Shown also in Figure 2 are the lines of two times the standard error of estimate, which for durability is 4.9 cycles. With the above equation an estimate of durability can be made within two standard errors at the 99 per cent confidence level.

This correlation may encourage more study of the ductility test, possibly including microductility as a method of producing a rapid evaluation of the durability of asphalts. A high degree of precision is required, however, because the range of ductility is only 3 cm for these coating-grade asphalts.

RESULTS OF NATURAL DURABILITY TESTS

The panels have now been exposed for about 18 months. At two of the sites, Halifax and Ottawa, very few failures have been detected, in contrast with those at Saskatoon where there has been a high degree of failure. Table VI records the degree of failure at the three sites.

From these results it is evident that Halifax does not present a very severe exposure for asphàlts probably the result of the presence at the roof site of a large amount of fly ash, which coats the panels and reduces degradation. It is of interest to note that failures at Ottawa and Halifax are of the same order, as is predicted by accelerated durability results. The asphalts that fail are those with poor accelerated durability performance. The rapid degradation at Saskatoon is particularly surprising. These panels were exposed in August and first examined in November, when very little failure was detected. In February, however, most had failed badly, indicating the severe effect of cold weather upon asphalt. Only those showing good durability in the accelerated test withstood this cold test, with the single exception of the Pembina specimen, which also failed.

The wide variation in performance at the three exposure sites makes it apparent that any attempt to obtain direct correlation between natural and accelerated durability is complex. Certain general statements can, however, be made. There appears to be some relationship between weight losses on natural and accelerated weathering, irrespective of the type of asphalt, and it also appears that failure is not always due to the attainment of a certain weight loss. This was shown in the panels from Halifax where weight losses in excess of those recorded in accelerated tests are found without failure. The rate of weight loss at the Ottawa site appears to be such that 5 cycles in the weatherometer is the same as 3 months' exposure at the test site (after the initial loss and for the particular time of exposure).

A word of caution should be added at this point to prevent any gross extrapolation of results. The accelerated test is a laboratory test that divides asphalts into groups, based on their ability to withstand the test. It does not follow that an asphalt with a rating of 60 cycles is three times better than one having a rating of 20 cycles. Only actual performance of the shingles can indicate this. The natural weathering test is an unrealistic one; it uses asphalt on a metal panel for purposes of testing, and introduces thermal strain into the asphalt that would not normally be present with a prepared roofing product.

CONCLUSION

The complex nature of asphalt and the conditions under which it degrades makes any correlation study very difficult. Performance of the materials on natural and accelerated weathering indicates that a wide variation exists between various asphalts. Most physical and chemical tests show very small differences, but because of the narrow range of properties these are not suitable for durability ratings. The ductility of asphalts, although covering a very small range, has an excellent correlation with durability rating. It is suggested that some consideration should be given to the use of the ductility test or a modified, more sensitive, ductility type test as a measure of the durability of coating grade asphalts.

REFERENCES

1. Abraham, H. Asphalts and allied substances, 6th Ed. V. 5, p. 298-304, New York, Van Nostrand, 1960.
2. Beitchman, B. D. Infra-red spectra of asphalts. J. Res., N.B.S., V. 63A, n. 2, 1959, p. 189-193.
3. Kleinschmidt, L. R. and H. R. Snoke. Effect of light and water on the degradation of asphalt. J. Res., N.B.S., V. 63C, n. 1, 1959, p. 31-36.
4. Galloway, R. M. Chemical composition of asphalt related to road service, A.C.S. Symposium on Chemistry and Composition of Asphalts, V. 3, n. 2, April 1958, p. A53-A59.
5. Pfeiffer, J. P. The properties of asphaltic bitumen, p. 172, New York, Elsevier, 1950.
6. Greenfeld, S. H. Chemical changes occurring during the weathering of two coating-grade asphalts. A.C.S. Symposium on Fundamental Nature of Asphalt, V. 5, n. 4A, Sept. 1960, p. A89-A106.
7. Stewart, J. E. Infra-red spectra of chromatographically fractionated asphalts. J. Res., N.B.S., V. 58, n. 5, 1957, p. 265-269.
8. Wright, J. R. and P. G. Cambell, Determination of oxidation rates of air blown asphalts by infra-red spectroscopy. J. Appl. Chem., V. 12, 1962, p. 256-266.
9. Martin, K. G. A review of the durability of roofing bitumens. J. Inst. Petrol, V. 47, n. 454, 1961, p. 321-328.
10. Mertens, E. W. and S. Greenfeld. Some qualitative effects of composition and processing on weatherability of coating-grade asphalts. A.S.T.M., S.T.P. No. 280, 1959, p. 20-28.
11. Mertens, E. W. Predicting weatherability of coating grade asphalts from asphaltene characteristics. A.S.T.M. Bulletin No. 250, 1960, p. 40-44.
12. Jones, T. K. The prediction of coating grade asphalt weatherability from asphaltene dispersibility data. Paper presented to A.S.T.M. Annual Meeting, Symposium on Recent Research on Bituminous Materials, June, 1963.
13. Heithaus, J. J. Measurement and significance of asphaltene peptization. J. Inst. Petroleum, V. 48, n. 458, 1962, p. 45-53.
14. Heithaus, J. J. Physical factors affecting the weather resistance of asphalt coatings. Industrial and Engineering Chemistry, V. 1, n. 3, Sept. 1962, p. 149-152.
15. Hamada, M. Durability of asphaltic materials. Proceedings, Arch. Inst. of Japan, V. 69(1-5), 1961, p. 53-72.
16. Kleinschmidt, L. R. Chromatographic method for refractionation of asphalt into distinctive groups of components. J. Res., N.B.S., V. 154, n. 3, 1955, p. 163-166.

17. Greenfeld, S. H. A method for preparing uniform films of bituminous materials. A.S.T.M. Bulletin No. 193, 1953, p. 50-53.

18. Greenfeld, S. H., E. W. Mertens and P. W. John. A statistical study of weatherometer data on coating grade asphalts. A.S.T.M., S.T.P. No. 280, 1959, p. 29-37.

19. Jones, P. M. Comparison of two spark-gap testers for asphalt films. Materials Res. and Standards, V. 3, n. 4, 1963, p. 293-295.

20. Gibbons, E. V. Outdoor exposure sites of the national research council. Chemistry in Canada, V. 12, n. 3, 1960, p. 44-48.

21. Wilkinson, C. E., R. N. Traxler and L. Striker. Criterion for the evaluation of roof coating asphalts. A.S.T.M., Bulletin No. 230, 1958, p. 42-44.

22. Greenfeld, S. H. Characteristics of fifteen coating-grade asphalts. J. Res., N.B.S., V. 64C, n. 4, p. 299-305, 1960.

TABLE I

CHARACTERISTICS OF ASPHALTS

Asphalts (a)	Source	S.Pt.°F (b)	Penetration at (c) 32°F dmm	77°F dmm	115°F dmm	S.G. at 77°F (d)	Ductility at 77°F (e)	Flash Point °F	Fire Point °F (f)	Durability (g)
1	Pembina	225.9	11.7	16.2	25.7	0.9993	1.7	535	605	75
2	Tiajuana 2nd sample	220.6	15.7	20.5	32.5	1.0142	2.5	570	630	70
3	Lloydminster 2nd sample	209.1	9.7	13.0	27.0	1.0382	2.4	490	555	69
4	Tiajuana 1st sample	217.6	13.7	18.8	29.7	1.0184	2.8	565	645	68
5	Marlago	228.9	9.3	13.2	21.5	1.0182	2.4	575	645	66
6	Arabian	223.7	11.3	14.5	23.7	1.0250	2.5	605	680	61
7	Lloydminster 1st sample	233.2	9.0	12.0	21.7	1.0342	1.7	460	530	58
8	Selected Southern Alberta Crudes	231.4	10.5	15.3	24.0	1.0259	1.6	520	575	50
9	South Saskatchewan Pipeline 1st sample	230.9	12.0	14.7	26.5	1.0268	1.8	530	595	48
10	West Spur Pipeline	227.3	12.0	18.2	28.5	1.0000	1.7	555	650	43
11	Coleville 2nd sample	224.1	9.5	12.5	22.8	1.0349	1.5	520	565	38
12	South Saskatchewan 2nd sample	231.4	8.7	12.8	23.0	1.0360	1.2	515	585	35
13	Coleville 1st sample	230.7	9.5	11.7	23.0	1.0300	1.1	520	580	29
14	Miscellaneous Southern Alberta Crudes	221.2	12.2	15.3	26.2	1.0180	1.6	540	630	22
15	Kuwait	227.8	7.3	9.8	16.2	1.0520	0.5	570	655	21

(a) Asphalts numbered in order of decreasing durability.
(b) A.S.T.M. D36-26 - Test for softening point of bituminous materials.
(c) A.S.T.M. D5-59T - Penetration of bituminous materials.
(d) A.S.T.M. D71-52 - Test for Specific Gravity of asphalts and tar pitches.
(e) A.S.T.M. D113-44 - Test for ductility of bituminous materials.
(f) A.S.T.M. D92-57 - Test for flash and fire points by Cleveland Open Cup.
(g) 50 per cent Failure with 60 - square grid 51-9 cycle.

TABLE II

CHANGES IN COMPONENTS WHEN EXPOSED TO ACCELERATED

WEATHERING

Asphalt and Durability		Asphaltenes Bef. Exp.	Asphaltenes (a) Failure	Resins Bef. Exp.	Resins (a) Failure	Dark Oils Bef. Exp.	Dark Oils (a) Failure	Water White Oils Bef. Exp.	Water White Oils (a) Failure	Per cent Wt. Loss at Failure
1	75	42.1	51.0	25.6	17.8	16.2	15.8	19.1	15.4	9.4
2	70	37.7	47.6	18.2	12.2	24.0	20.1	19.4	20.0	10.4
3	69	43.4	54.6	18.1	19.8	23.0	13.6	13.8	11.9	5.0
4	68	39.5	48.9	22.3	21.9	20.1	14.9	18.5	14.3	8.0
5	66	40.8	48.2	19.3	16.0	24.7	22.6	16.4	13.2	6.6
6	61	42.5	50.7	23.9	15.8	24.3	23.8	11.7	9.8	7.9
7	58	45.2	55.8	19.9	17.1	17.4	12.5	17.0	14.6	5.4
8	50	42.6	52.9	20.7	19.2	21.2	13.2	17.5	14.7	7.4
9	48	41.8	53.2	23.9	17.9	31.1	16.6	14.2	12.1	7.2
10	43	38.8	47.4	27.7	20.5	18.3	16.6	19.9	15.5	7.4
11	38	44.6	51.4	18.5	9.1	21.9	22.9	15.5	16.6	5.6
12	35	44.1	54.5	16.0	15.7	26.0	16.9	14.2	12.9	6.4
13	29	44.7	53.3	17.5	17.1	24.0	17.7	15.1	12.3	3.2
14	22	38.9	45.3	20.5	19.4	20.7	18.3	21.9	17.0	3.8
15	21	48.6	57.2	23.5	17.3	19.2	16.1	8.5	9.4	1.9

(a) 51-9 cycle 50 per cent failure using 60-square grid.

TABLE III

CHEMICAL ANALYSES OF ASPHALTS BEFORE EXPOSURE

Asphalt	(a) Durability	Carbon per cent	Hydrogen per cent	(b) Oxygen per cent	Sulphur per cent	Nitrogen per cent	(c) C/H	C+H
1	75	86.5	11.1	1.3	0.6	0.4	0.65	97.6
2	70	85.7	10.6	0.9	2.7	0.5	0.67	96.3
3	69	83.9	10.2	0.5	4.9	0.4	0.69	94.1
4	68	85.9	10.5	0.2	2.8	0.5	0.68	96.4
5	66	85.8	10.3	-	3.2	0.5	0.69	96.1
6	61	85.7	10.3	-	3.9	0.3	0.69	96.0
7	58	84.3	10.0	0.7	4.5	0.4	0.70	94.3
8	50	85.2	10.2	0.4	3.3	0.4	0.69	95.4
9	48	84.9	10.3	0.4	3.9	0.5	0.69	95.2
10	43	86.7	10.9	0.9	1.0	0.4	0.66	97.6
11	38	84.6	10.0	1.1	3.7	0.5	0.70	94.6
12	35	84.8	10.0	0.3	4.3	0.5	0.71	94.8
13	29	85.2	10.2	0.4	3.6	0.5	0.70	95.4
14	22	85.3	10.5	0.8	3.0	0.4	0.68	95.8
15	21	85.4	9.6	-	5.1	-	0.74	95.0

(a) 51-9 cycle 50 per cent failure using 60-square grid
(b) Oxygen by difference from average values
(c) Atomic ratio

TABLE IV

CRITERIA OF WILKINSON CONTROL FOR DURABILITY

Property	Limits
Softening point	210 - 230°F
Penetration at 32°F	10 minimum dmm
Penetration at 77°F	18 - 25 dmm
Penetration at 115°F	24 - 40 dmm
Flash Point (Cleveland Open Cup)	500 minimum °F
Specific Gravity (77°F)	0.990 to 1.040

TABLE V

EQUATIONS AND CORRELATIONS OF ASPHALT CHARACTERISTIC AND DURABILITY

Characteristic	Equation	Correlation Coefficient r
Softening Point	Y = 224.3 - 0.8X	-0.25
Penetration at 32°F	Y = 3.9X + 7.6	+0.46
Penetration at 77°F	Y = 3.2X + 3.5	+0.51
Penetration at 115°F	Y = 1.2X + 22.0	+0.71
Ductility at 77°F	Y = 25.1X + 5.7	+0.81
Density	Y = 662.4 - 602.0X	-0.34
Wt. loss at 10 cycles	Y = 66.7 - 14.6X	-0.14
$\frac{\text{Resins}}{\text{Asphaltenes}}$	Y = 119.1X - 9.2	+0.26
$\frac{\text{Resins and Asphaltenes}}{\text{Total oils}}$	Y = 701.5 - 484.5X	-0.15
$\frac{\text{Carbon}}{\text{Hydrogen}}$	Y = 161.7 - 155.0X	-0.6
Carbon + hydrogen	Y = 6.0X - 529.5	+0.23

Note: In all cases Y = durability
X = characteristic

TABLE VI

PERCENTAGE OF FAILURE ON NATURAL DURABILITY

	Halifax					Saskatoon					Ottawa				
Asphalts	3mos	6mos	9mos	12mos	18mos	3mos	6mos	9mos	12mos	18mos	3mos	6mos	9mos	12mos	18mos
1	1.67	3.34	5.01	5.01	5.01	3.34	50+F				3.34	8.3	15.0	16.3	22.1
3	3.34	5.01	5.01	5.01	5.01	6.68	13.4	15.0	115.0	50+F	5.01	8.3	11.7	10.8	36.7
4	5.0	6.7	6.7	6.7	6.7	3.34	6.7	16.1	15.0	50+F	6.7	8.3	8.3	8.3	10.0
5	11.7	11.7	11.7	11.7	11.7	10.0	50+F				15.0	20.0	40.1	35.9	50+F
6	6.7	6.7	6	6.7	6.7	16.7	50+F				15.0	25.1	26.7	24.2	30.9
7	0	0	0	0	21.7	0	50+F				3.3	3.3	20.0	18.0	50+F
9	6.7	8.3	8.3	8.3	25.1	0	50+F				6.7	8.4	16.7	16.7	46.7
10	5.0	3.3	3.3	3.3	13.4	3.3	50+F				10.0	11.7	15.0	15.0	36.7
13	8.4	21.7	26.7	26.7	50+F	3.3	50+F				15.0	23.4	50+F		
14	3.3	11.7	15.0	50+F		0	50+F				3.3	3.3	15.0	50+F	
15	0	11.7	18.4	15.9	50+F	8.4	50+F				1.7	13.4	50+F		

F - denotes failure

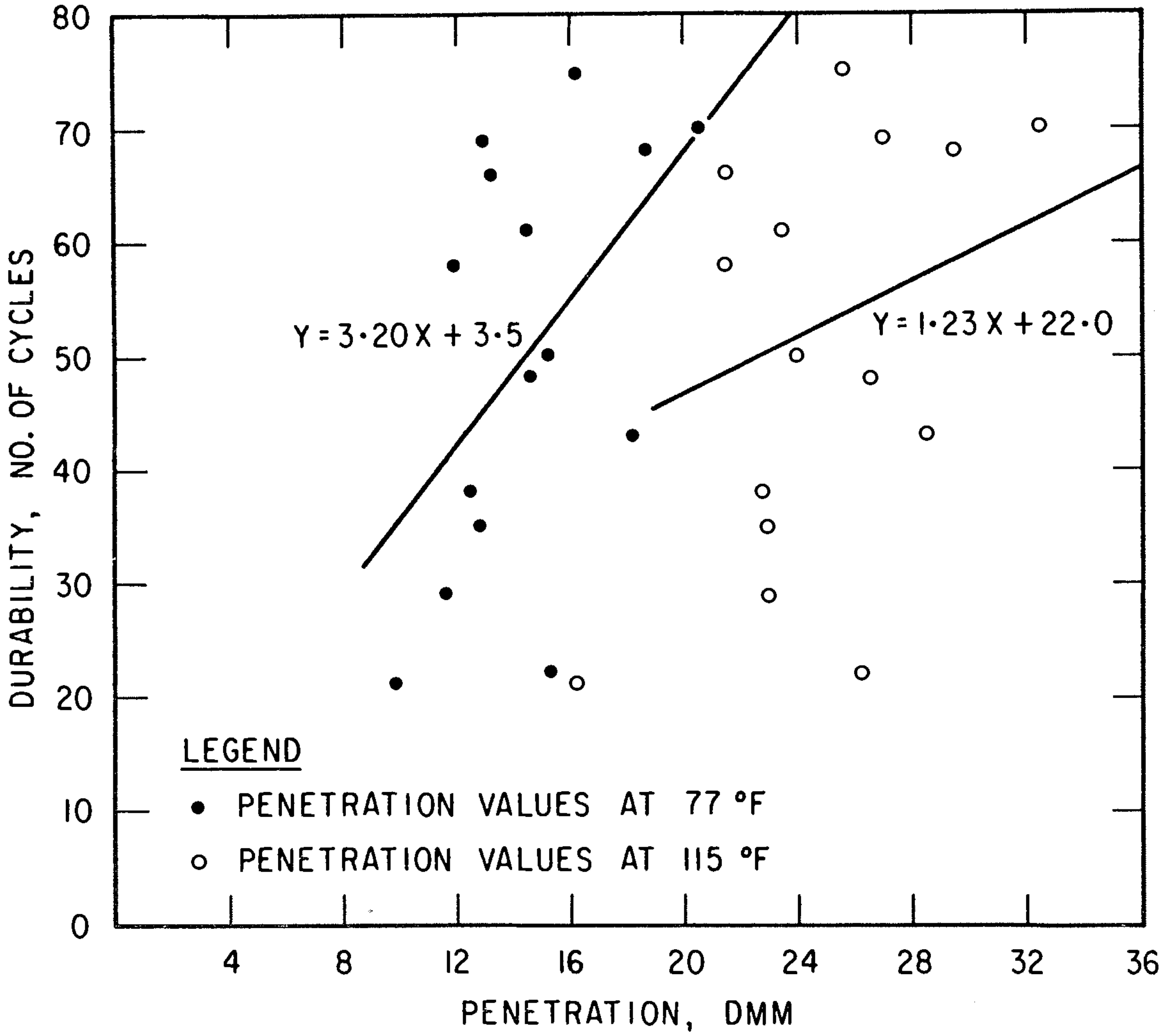

FIGURE 1
CORRELATION OF PENETRATION WITH DURABILITY

BR 3001-1

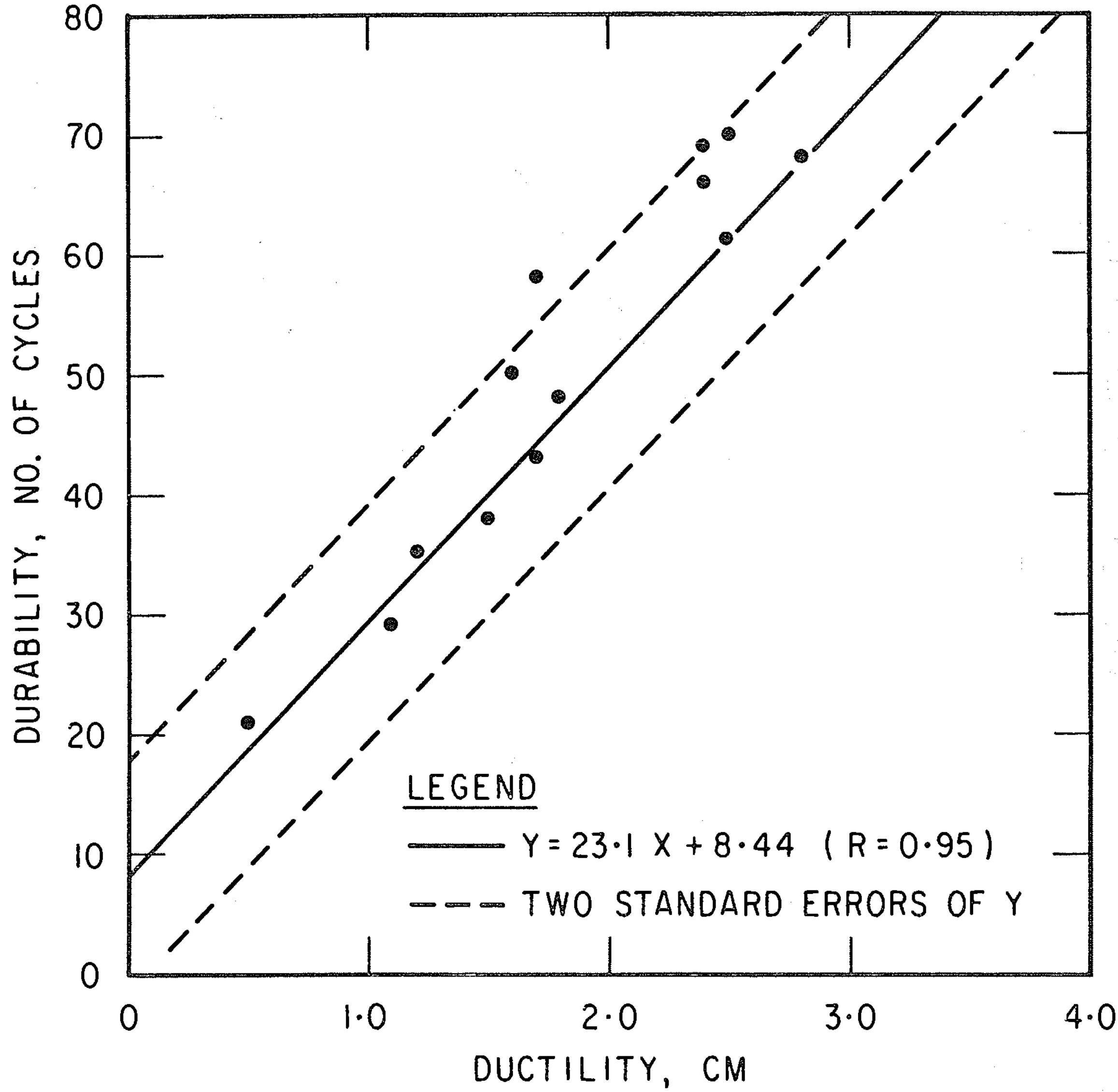

FIGURE 2
CORRELATION OF DUCTILITY WITH DURABILITY

BR 3001-2