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Improving Precision of Rubber Test Methods: Part 1-Hardness

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ABSTRACT

Many interlaboratory test programmes (ITPs) on rubber test methods have been carried out during the last 10 years. ITPs have been organized within ISO TC 45 and also in several countries such as USA, UK and Sweden.

Most of these ITPs have shown that the repeatability and the reproduceability are poor for many rubber test methods.

In an attempt to do more than just determine the poor precision, the authors chose four methods and decided to study them in order to identify the factors giving poor precision, thereby improving the methods.

This first part contains the work done on hardness tests, both IRHD and Shore. The results show that many factors are contributing to the variation in test results, but the main factor is the influence of the operator. If we eliminate the influence of the operator and perform the test under correct temperature, load and time, we can achieve a much improved repeatability and reproduceability. This indicates that there is a need to develop more automatic, computerized hardness testers which can be sold for reasonable prices.

1 BACKGROUND

At the beginning of the 1980s it was decided to include within IS0 TC45: Rubber and Rubber Products, a precision clause in all testing method standards. The precision clauses were established by carrying out interlaboratory test programme (ITPs) to establish the ***repeatability*** (within laboratory) and the ***reproduceability*** (between laboratories) for the test methods.

In 1981 IS0 published a standard for determination of the precision of test methods, IS0 5725-86.' In 1984 TC 45 published a technical report, IS0 TR 9272,[2] for guidance on how to establish precision data for rubber test methods. IS0 TC 45 has since then carried out about 25 interlaboratory test programmes.

This work inspired researchers in Sweden to start an interlaboratory test programme organized by the Swedish National Testing Institute. During the years 1982-1988, 14 interlaboratory tests were carried out. For two of the methods a retest was done. Up to 25 laboratories participated in these interlaboratory tests.

All these interlaboratory tests within ISO and in Sweden have shown that the spread in the test results is worse than anyone could have expected.

At the same time, the requirements for the products have increased, which means that we need to be able to test the properties of rubber materials with a higher accuracy than before. It must not be the case that what we measure mainly reflects the spread in the testing and does not show the variations in the material tested.

2 THE PURPOSE OF THE PROJECT

The purpose of the project was to achieve a lower spread in test results, within and between laboratories, for the test methods under study. The results from this project will be presented to the Swedish Standards Institution and to IS0 as a basis for improving the test method standards.

This project was started in 1989.

3 PARTICIPATING COMPANIES

The following companies have participated and financed this project:

—Alfa-Laval Materials AB
-Forsheda AB
—Horda Compound AB
-Skega AB
—Statens Provningsanstalt

—Sunnex AB
-Trelleborg Tndustri AB
-Viskafors AB
-Volvo Flygmotor AB
-Volvo PV AB, materiallab
—Värnamo Gummifabrik AB
-Saab-Scania AB, Scaniadivisionen

4 THE ORGANIZATION OF THE PROJECT

4.1 General organization

The following methods have been used during the project:

-hardness, normal and micro IRHD, according to IS0 48” and Shore according to IS0 7619[4]
-tensile test, according to IS0 37’
-heat ageing, according to IS0 188”
-temperature retraction test, TR, according to IS0 2921[7]

Good background material was obtainable for all of these methods as all of the tests have been studied one or more times by interlaboratory trials.

The test methods have been studied by investigating the influence of different factors on the spread in test results.

At the beginning of the project a visit was paid to all participating companies to make up an inventory of the type of test instruments that are being used. Some preliminary interlaboratory tests and other measurements were also made.

4.2 Organization of the hardness part

For hardness, the following influencing factors were studied:

-equipment
-operator
-temperature
-thickness
-applied load (Shore)

Equipment and operator influence were studied by one operator visiting all participating companies and using a normal IRHD tester and a Shore tester.

The influence of temperature and applied load were studied in one laboratory, and the influence of thickness only by looking at literature references.

5 HARDNESS RESULTS

How the reproduceability was calculated is shown in Appendix 1. The hardness reproduceability determined in earlier ITPs[8,9] is shown in Table 1 for comparison.

TABLE 1
Hardness Reproduceability

	Mean	s^a	R^b	$(R)^c$
1983 ITP				
IRHD-normal	62.2		4.9	
IRHD-micro	63.8		5.5	
Shore	62.5		7·2	
1987 *ITP*				
IRHD-normal	60.5	1·4	4·1	6.8
IRHD-micro	60.8	1.9	5.5	9·0
Shore	60.9	2·6	7.3	12·0

[a] s = Standard deviation.
[b] R = Reproduceability in actual units of measurement.
[c] (R) = Reproduceability as a percentage of the measured value.

Note: The 1983 ITP had 25 participants; the 1987 ITP had 20 participants, and the participants were divided in two groups with each group making the measurements on the same test pieces.

The hardness long- and short-term repeatability determined in an IS0 ITP from 1991 are shown in Table 2 and Appendix 2.

5.1 Equipment used

The equipment used for the precision tests is shown in Table 3.

5.2 Test conditions

The test conditions in the different laboratories were as shown in Table 4.

TABLE 2
Hardness Repeatability

	Mean	s^a	r^b	$(r)^c$
1991 ITP, long-term (months)				
IRHD-Normal	60·0	0.72	2·0	3·4
Shore	60.9	0·76	2.1	3.5
1991 ITP, short-term (days)				
IRHD-Normal	59.9	0·43	1.2	2·0
Shore	60.3	0·58	1.6	2.7

[a] *s* = Standard deviation.
[b] *r* = Repeatability in actual units of measurement.
[c] (r) = Repeatability as a percentage of the measured value.

Note: The results are calculated from three Swedish participants in an IS0 ITP. Eleven rubber samples were measured with 1 or 2 days interval for short-term repeatability and after 1, 3 and 6 months for long-term repeatability. The results shown are the mean values for three companies.

Most of the laboratories did not use talc for lubrication of the test pieces as required by the standard. The operator who visited all participating laboratories did use talc during his measurements, but the author has not seen any evidence that using talc should improve the reproduceability.

5.3 Test results normal IRHD

A full summary of all measurements is presented in Appendix 3.

5.3.1 *Equipment and operator influence*

Table 5 shows the mean values, standard deviation and reproduceability results from normal IRHD measurements, when investigating equipment and operator influence. Figures 1-4 show the variation between laboratories graphically.

The results show more variation when the same operator is using the same hardness tester compared to the results obtained when the operator is using different hardness testers. This is probably because the hardness tester transported round to the participating laboratories was brand new and had a problem with friction inside the dial gauge.

The results indicate that the equipment has less influence than the operator on the test results.

TABLE 3
Equipment Used

Company	*N-IRHD*	*Cal*[a]	*M-IRHD*	*Cal*[a]	*Shore*	*Cal*[a]	*Stand*
1	Wallace	*Y*	Wallace	*Y*	Zwick	*Y*	*N*
2	Wallace	*Y*	Wallace	*Y*	Zwick	*N*	*Y*
3	Wallace	*Y*	Wallace	*Y*	Shore	*Y*	*N*
4	Wallace	*Y*	Wallace	*Y*	Zwick	*N*	*N*
5	Wallace	*Y*	Wallace	*Y*	Zwick	*N*	*N*
6	Wallace	*Y*	Wallace	*Y*	Zwick	*Y*	*N*
7	Wallace	*Y*	Wallace	*Y*	Zwick	*Y*	*Y*
8	Wallace	*Y*	Wallace	*Y*	Zwick	*N*	*N*
9	Wallace	*Y*	Wallace	*Y*	Zwick	*Y*	*Y*
10	Wallace	*Y*	Wallace	*Y*	Shore	*N*	*N*
11	Wallace	*Y*	Wallace	*Y*	?	*N*	*N*
12	Wallace	*N*	Wallace	*N*	Zwick	*N*	*N*
13	Wallace	*Y*	Wallace	*Y*	?	*N*	*N*
14	Wallace	*Y*	—		Shore	*N*	*N*

[a] Cal = Calibrated between 6 and 12 months before the test.

5.3.2 Influence of temperature

The influence of temperature was examined in two ways. When visiting the different participating laboratories, the temperature was recorded when doing the hardness tests. These temperatures were plotted in a diagram against the mean hardness (see Fig. 5). Later, one test was

TABLE 4
Test Conditions

Company	*Date*	*Temperature (°C)*	*Talc*
1	16 Mar. 1990	22.0	*N*
2	27 Apr. 1990	24.5	*N*
3	27 June 1990	23·5	*N*
4	20 Mar. 1990	22·5	*N*
5	29 Aug. 1990	24·5	*N*
6	15 Mar. 1990	23-O	*Y*
7	15 July 1990	24.5	*N*
8	27 Aug. 1990	22·5	*N*
9	4 Apr. 1990	22·5	*Y*
10	20 Apr. 1990	24-O	*N*
11	30 Aug. 1990	22.5	*N*
12	29 Aug. 1990	23-O	*N*
13	4 Sep. 1990	21.5	*N*
14	2 Apr. 1991	21.0	*N*

TABLE 5
Equipment and Operator Influence: Normal IRHD

	Mean	s^a	R^b	$(R)^c$
Different operators and different hardness testers	59·8	1.07	3·0	5·1
Different operators and the same hardness tester	59.2	0·90	2.6	4·3
One operator and different hardness testers	59.4	0·51	1.4	2-4
One operator and the same hardness tester	59.1	O-6.5	1·8	3·1

[a] s = Standard deviation.
[b] ***R*** = Reproduceability in actual units of measurement.
[c] ***(R)*** = Reproduceability as a percentage of the measured value.

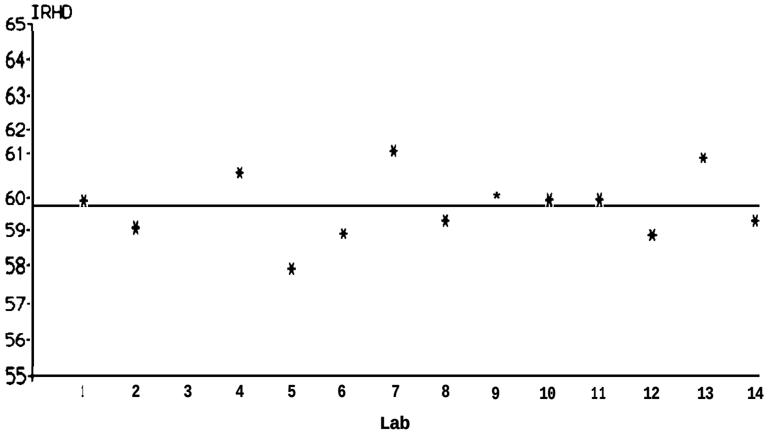

Fig. **1.** Normal IRHD, different operators, different hardness testers (mean values for five materials).

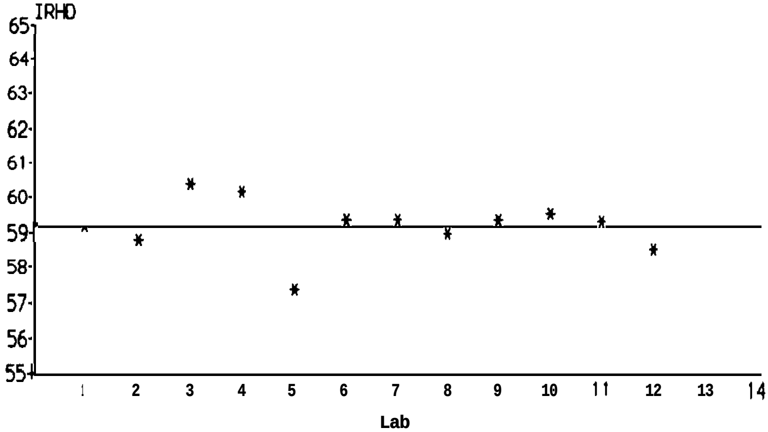

Fig. 2. Normal IRHD, different operators, same hardness tester (mean values for five materials).

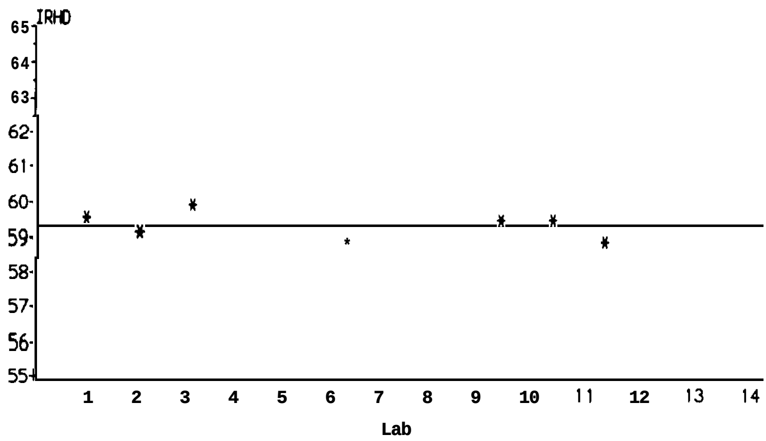

Fig. 3. Normal IRHD, one operator, different hardness testers (mean values for five materials).

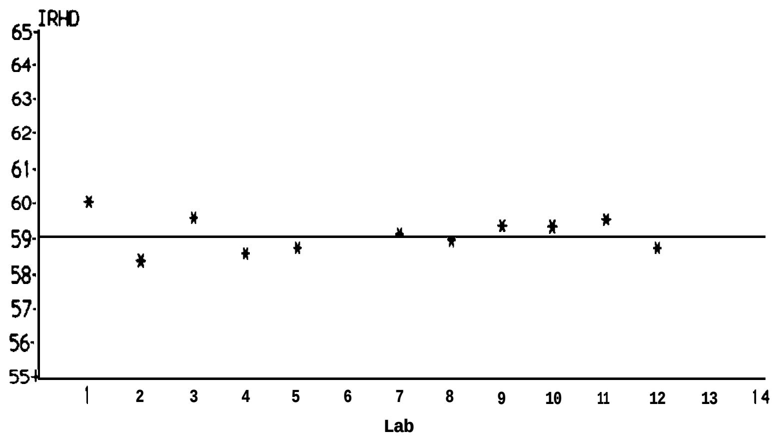

Fig. 4. Normal IRHD, one operator, same hardness testers (mean values for five materials).

performed in one laboratory, in a temperature chamber, at temperatures of 15, 20, 25 and 30 "C; the results are shown in Table 6 and Fig. 6. The regression line shows the hardness to vary by 0·13 IRHD/°C.

5.3.3 Influence of thickness

No experimental tests were performed to investigate the influence of thickness on hardness as references to earlier work were available. Brown reports a diagram with 'effect of test piece thickness on hardness reading', in the physical testing of rubber.'" Tangorra in Italy has carried out extensive theoretical and experimental research," leading to

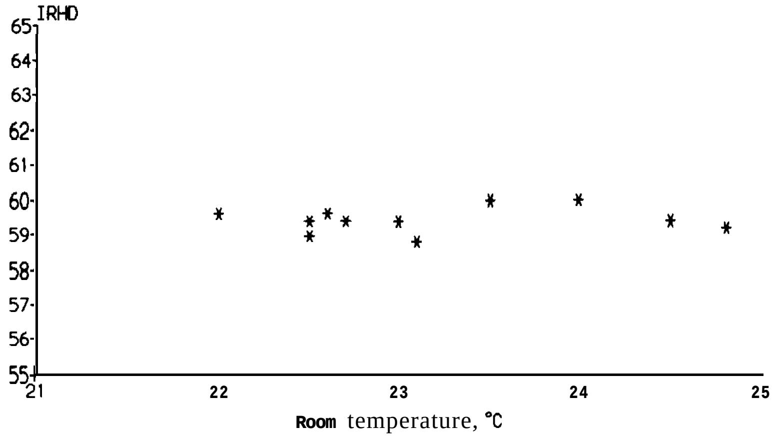

Fig. 5. Normal IRHD, one operator, different hardness testers: temperature influence.

TABLE 6
Hardness as a Function of Temperature

Temperature (°C)	*Sample 1 IRHD*	*Sample 2 IRHD*	*Sample 3 IRHD*	*Sample 4 IRHD*	*Sample 5 IRHD*	*Mean IRHD*
15	*58*	*50*	*68*	79	44	59.8
20	56	49	66	78	43	58.4
25	56	49	66	78	43	58.4
30	56	48	65	76	43	57.6

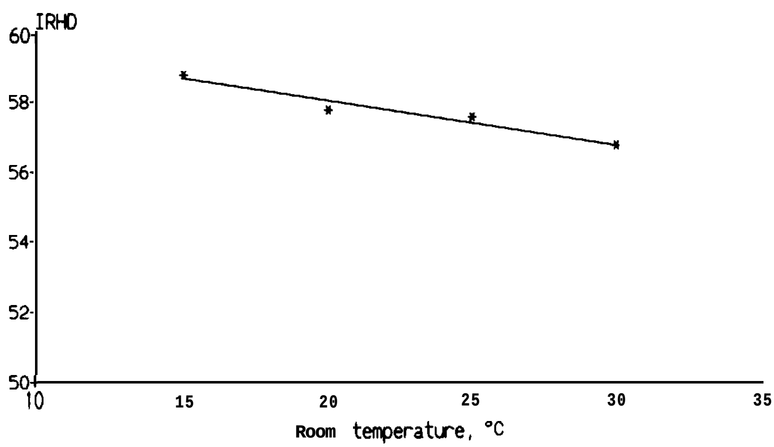

Fig. 6. Normal IRHD, different room temperature (mean values for five materials).

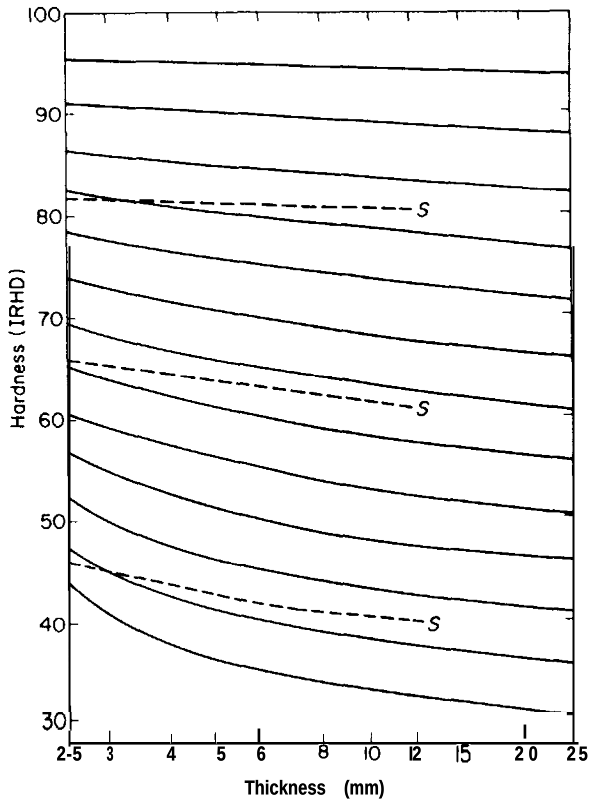

Fig. 7. Effect of test piece thickness on hardness readings from the physical testing of rubber.

the inclusion in the Italian standards[12] of diagrams suitable for the correction of IRH because of non-standard thickness.

5.3.4 Time influence

Figure 8 was prepared to see if the time lapse from the first test to the last test had any influence or the hardness results: no clear trend can be seen.

5.4 Micro IRHD test results

A full summary of all measurements is presented in Appendix 4.

5.4.1 *Equipment and operator influence*

Table 7 shows the mean values, standard deviation and reproduceability results from micro-IRHD measurements, when investigating equipment and operator influence. Figures 9 and 10 show the variation between

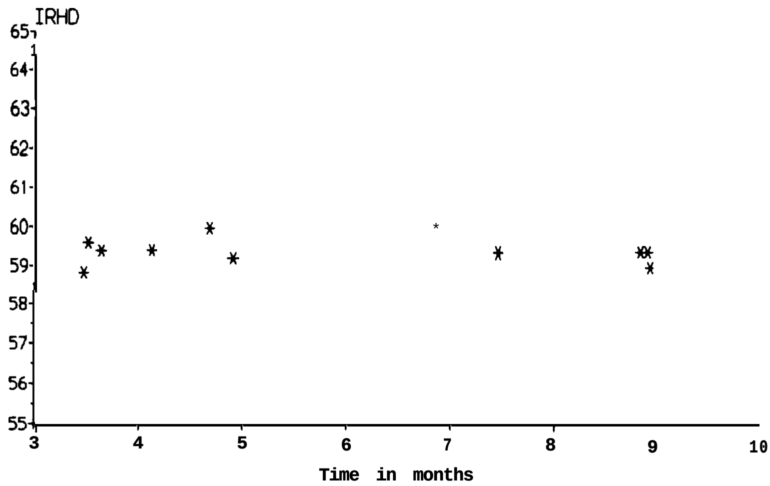

Fig. 8. Normal IRHD, one operator, different hardness testers: time influence.

TABLE 7

Equipment and Operator Influence: Micro IRHD

	Mean	s^a	R^b	(R)
Different operators and different hardness testers	62.2	1.14	3-2	5.2
One operator and different hardness testers	62·0	0·99	2·8	4.5

[a] s = Standard deviation.
[b] **R** = Reproduceability in actual units of measurement.
[c] ***(R)*** = Reproduceability as a percentage of the measured value.

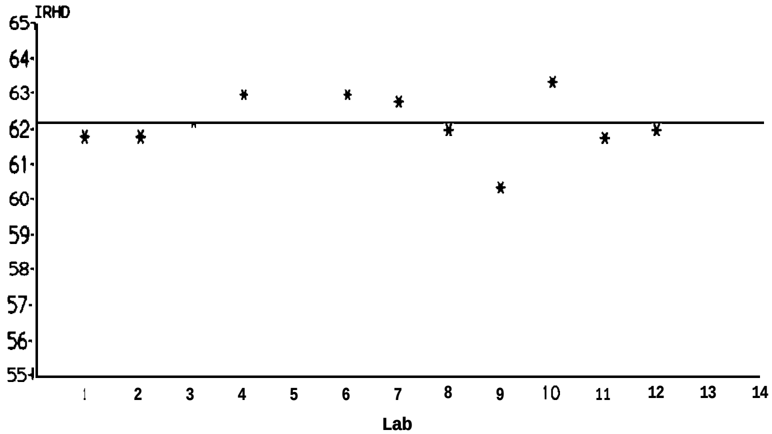

Fig. 9. Micro IRHD, different operators, different hardness testers (mean values for five materials).

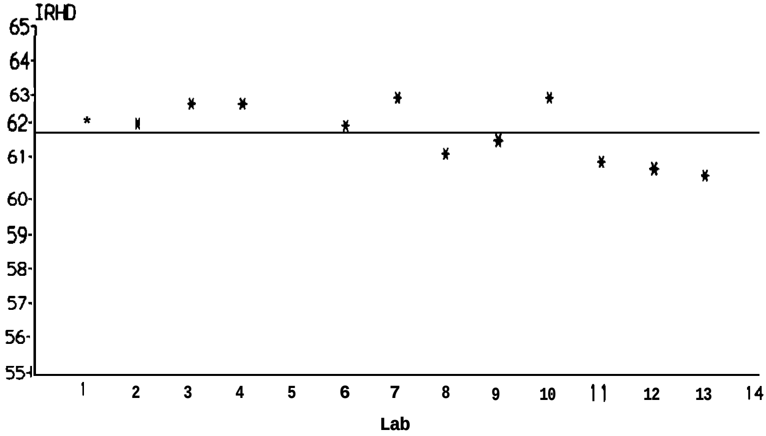

Fig. 10. Micro IRHD, one operator, different hardness testers (mean values for five materials).

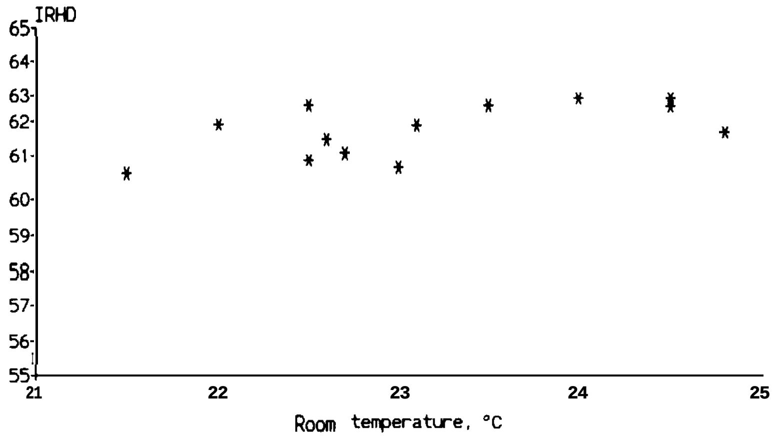

Fig. 11. Micro IRHD, one operator, different hardness testers: temperature influence.

TABLE 8
Equipment and Operator Influence: Shore A

	Mean	S^a	R^b	(R)
Different operators and different hardness testers	61·0	1.61	4.6	7·5
Different operators and the same hardness tester	59·7	1.12	3.2	5·3
One operator and different hardness testers	61·2	1·10	3·1	5·1
One operator and the same hardness tester	61.3	0·65	1·8	3·0

[a] s = Standard deviation.
[b] R = Reproduceability in actual units of measurement.
[c] ***(R)*** = Reproduceability as a percentage of the measured value.

laboratories graphically. The results indicate that the operator has less influence than the equipment on the test results.

5.4.2 Influence of temperature
The temperature influence was examined in one way. When visiting the different participating laboratories, the temperature was recorded when doing the hardness tests. These temperatures were plotted against the mean hardness (see Fig. 11).

5.5 Test results: Shore

A full summary of all measurements is presented in Appendix 5.

5.5.1 Equipment and operator influence
Table 8 shows the mean values, standard deviation and reproduceability results from Shore A measurements, when investigating equipment and operator influence. Figures 12, 13 and 14 show the variation between laboratories graphically. It can be seen that the operators and the instruments seem to contribute about equally to the variation in results.

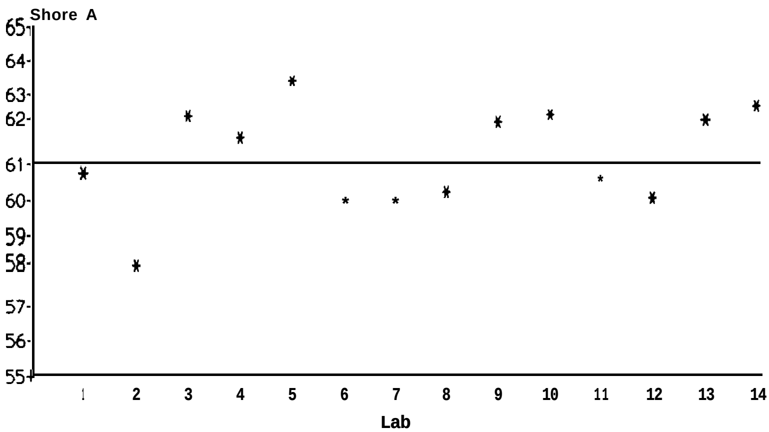

Fig. **12.** Shore A, different operators, different hardness testers (mean values for five materials).

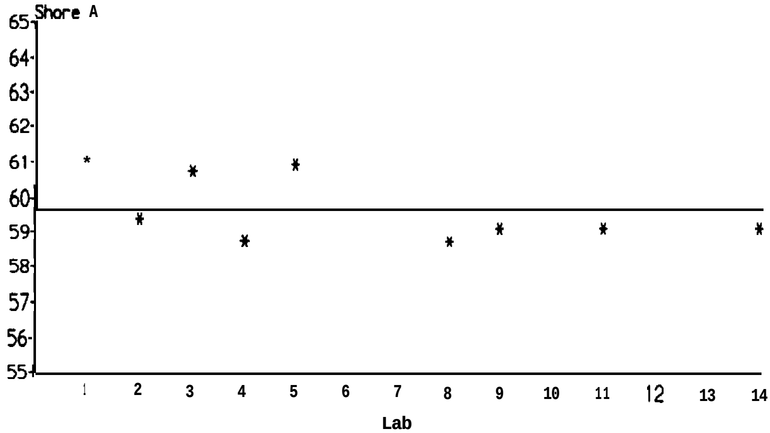

Fig. 13. Shore A, different operators same hardness tester (mean values for five materials).

5.5.2 *Influence of thickness*

No experimental tests were carried out to investigate the influence of thickness on hardness, as references to earlier work was found. Bassi *et al.* presented a paper Shore A Hardness and Thickness.‘”

5.5.3 *Influence of applied load*

It was first established what load different operators applied on the hardness tester when measuring Shore A by hand. Ten operators made

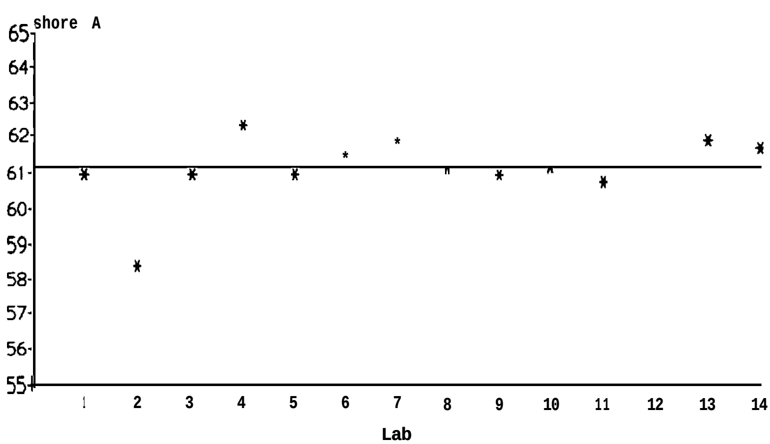

Fig. 14. Shore A, one operator, different hardness testers (mean values for five materials).

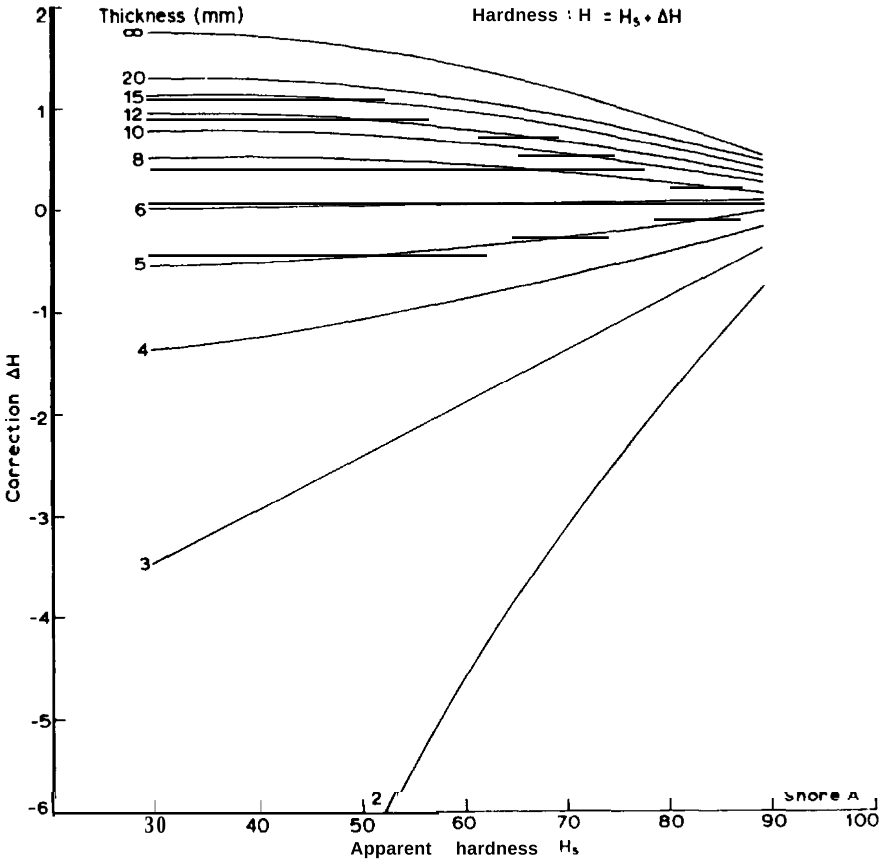

Fig. 15. Influence of thickness: Shore A.

measurements on a balance. The operators used loads from 1·2 kg to 5·6 kg (see Appendix 5). The specified load is 1·0 kg.

To establish how much this influenced the hardness results, two series of measurements were taken with an automatic Shore A Tester, Durotronic 1000 with automatic stand 902, with 1 and 5 kg loads.

TABLE 9
Influence of Applied Load: Shore A

	Applied load	*Mean*	*Increase*
First measurement series (11 samples)	1 kg	60·6	
	5 kg	63-1	2.5
Second measurement series (7 samples)	1 kg	60.6	
	5 kg	62·8	2·2

The first measurement series was made on 11 rubber samples earlier used in an IS0 ITP. The second series was made on a set of 7 Shore hardness reference blocks of silicone rubber.

The results, shown in Table 9 and in Appendix 5, show the hardness to increase from 0·9 to 3·7° Shore A when using a 5 kg load. There was no correlation between the hardness value and the increase in hardness with the 5 kg load.

5.6 Summary of results

The reproduceability results calculated from the ITP made in this project show improved precision values compared to the ITPs from 1983 and 1987. The author has no explanation for this other than that it might be the influence of the operator visiting the different laboratories; so the other operators performed the tests with more care. The results are shown in Table 10 for comparison.

The author has, by analysing all the tests performed in this project, tried to estimate the contribution of different factors to the reproduceability for the three hardness methods. The estimation is shown in Table 11.

It can be seen that the operator is the main factor, except for micro IRHD where not enough tests were performed to really differentiate between equipment and operator, although the tests that were performed point to the equipment.

Another way of looking at this is that if the equipment allows the operators to do the test in different ways, is the equipment to be blamed?

To investigate the smallest possible repeatability, an automatic Shore stand 902 with a digital gauge and measured 11 rubber blocks was used on two different days, giving a constant load (1 kg), a constant loading speed and a constant measurement time (1 s). What is left is mainly the variations in the rubber blocks. The result is compared to three

TABLE 10
Hardness Reproduceability

	N-IRHD	*M-IRHD*	*Shore A*
1983 ITP, *R*[a]	4.9	5·5	7.2
1987 ITP, *R*	4.1	5·5	7·3
Project ITP, *R*	3·0	3.2	4.6

[a] ***R*** = Reproduceability in actual units of measurement.

TABLE 11
Influence of Different Factors

Method	*R, IRHD*	*Remarks*
Normal IRHD		
Equipment	0·4	
Operator	1.6	
Temperature	0·13	per °C
Thickness	2	from 4 to 10 mm at 60-70 IRHD
Micro-IRHD		
Equipment	2.8	
Operator	0·3	
Shore A		
Equipment	1.4	
Operator	1·5	
Load	2.4	from 1 to 5 kg
Thickness	1·5	from 4 to 10 mm at 60-70 Shore A

different laboratories making the same short time repeatability test on the same rubber blocks. The results are shown in Table 12 and Section A2.4.

6 RECOMMENDED ACTIONS

Apart from eliminating the operator influence, which is of importance for all testing, the author suggests the following recommendations for improving the three methods.

TABLE 12
Short Time Repeatability: Shore A

Laboratory	*r*
1 manual	1.48
2	2.18
3	1·05
1 automatic	0·60

Normal-IRHD

-Eliminating the friction inside the dial gauge is an important factor when setting a correct 'zero' value.

-Automatic timing of the zero setting time, 5 s and measuring time, 30 s.

-Automatic lowering of the zero load and measurement load, with an even speed.

Micro-ZRHD

The same items as for normal—IRHD are valid. In addition to this the micro hardness testers used are too complex and difficult to use.

Shore A

-Using a constant load greatly improves the Shore test. This can be done by using a stand, but then the idea of a portable hardness tester disappears. Maybe it is possible to build a small load indicator in a pocket meter.

-Increasing the measurement time, as in DIN, to 3 or 5 s.

-Regular calibration and adjustment, as the load of the spring changes with time.

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APPENDIX 1

HOW TO CALCULATE THE REPEATABILITY AND REPRODUCEABILITY

n = Number of measured values
x_i = Measurement $1, 2, 3, \ldots, n$

$\bar{x}$ = Mean value $\qquad \bar{x} = \frac{\sum x_i}{n}$

$\bar{s}$ = Mean value (pol) $\qquad \bar{s} = \sqrt{\frac{\sum (s_i)^2}{n}}$

The pol mean value is used when calculating mean values of standard deviation and coefficients of variation.

s = Standard deviation $\qquad s = \sqrt{\frac{\sum (x_i) - \bar{x})^2}{n - 1}}$

S_L = Standard deviation between laboratories
s_r = Standard deviation within laboratories

v = Coefficient of variation $\qquad v = \frac{s}{\bar{x}} 100$

v_L = Coefficient of variation between laboratories
r = Repeatability $\qquad r = 2{\cdot}83 s_r$

R = Reproduceability $\qquad R = 2{\cdot}83\sqrt{s_L^2 + s_r^2}$
If the repeatability is not calculated $sr = 0$

Definition: An established value, below which the absolute difference between two 'between-laboratory' test results may be expected to lie, with a specified probability. The probability is normally 95% if nothing else is specified.

(R) = Reproduceability expressed as a percentage of the mean value of the measured values.

Extreme values have been checked with Dixon's Outlier Test.

APPENDIX 2

A2.1 Normal IRHD, long-term repeatability

Time (months)	*Material*											
	1	2	3	4	5	6	7	8	Y	10	11	
Laboratory no. I												
0	55	*44*	53	67	77	55	60	44	66	59	80	60.0
1	55	44	52	67	76	55	60	44	66	57	80	59·6
3	55	43	52	67	76	54	61	43	66	57	80	59.5
6	56	44	54	67	77	55	61	43	66	58	81	60.2
Mean	55.3	43.8	52.8	67·0	76.5	54.8	60.5	43.5	66.0	57·8	80.3	59.8
s	0.50	0.50	0·96	0·00	0.58	0.50	0.58	0·58	0·00	0·96	0.50	0.33
s(pool)	0·59	***R*** total	1.67	***(R)*** total	2·79							
Laboratory no. 2												
0	55	44	53	66	76	55	60	44	65	58	78	59.5
1	55	42	52	66	75	54	60	43	65	57	78	58.8
3	56	45	54	67	75	55	61	45	66	58	78	60.0
6	56	44	53	67	76	55	61	44	66	57	79	59.8
Mean	55.5	43.8	53·0	66.5	75.5	54.8	60.5	44·0	65.5	57.5	78.3	59.5
s	0·58	1.26	0.82	0.58	O-58	O-50	0-58	0.82	0.58	0.58	0.50	0.52
s(POOL)	0·70	***R*** total	1·99	***(R)*** total	3.34							
Laboratory no. 3												
0	56	44	54	67	77	56	61	44	66	56	83	60.4
1	57	44	54	67	76	56	61	44	66	58	82	60·5
3	58	45	54	67	76	56	62	45	66	58	79	60.5
6	58	44	54	68	78	56	62	44	67	59	82	61.1
Mean	57.3	44.3	54.0	67.3	76.8	56.0	61.5	44.3	66·3	57·8	81.5	60·6
s	0.96	0·50	0·00	0·50	0·96	0·00	0·58	0·50	0·50	1·26	1.73	0·33
s(pool)	0·84	***R*** total	2.38	***(R)*** total	3.92							

A2.2 Shore A, long-term repeatability

Time (months)	*Material*										
	1	2	3	4	5	6	7	8	9	10	11	
0	56	46	56	69	80	56	64	4.5	66	58	75	61·0
1	56	46	58	68	79	55	64	46	68	58	76	61·3
3	57	46	57	69	79	56	64	46	68	58	77	61·5
6	57	46	58	69	79	55	64	47	68	58	77	61.5
Mean	56.5	45.8	57.3	68.8	79.3	55.5	64·0	46·0	67.5	58.0	76.3	61.3
s	0·58	0.50	0·96	0·50	0.50	0.58	0·00	0.82	1·00	0·00	0.96	0·26
s(pool)	0·67	***R*** total	1·89	***(R)*** total	3.08							
Laboratory no. 2												
0	54	43	53	66	77	54	60	43	66	55	73	58.5
1	55	44	54	66	75	53	60	43	65	53	73	58.3
3	55	44	55	66	76	54	61	43	66	54	73	58·8
6	56	44	54	66	77	54	61	44	66	53	74	59.0
Mean	55·0	43.8	54.0	66.0	76.3	53.8	60·5	43.3	65.8	53.8	73.3	58.7
s	0.82	0.50	0·82	0·00	0.96	0.50	0.58	0·50	0·50	0·96	0.50	0.32
s(pool)	0·66	***R*** total	1.86	***(R)*** total	3.17							
Laboratory no. 3												
0	58	47	57	69	80	57	63	47	67	58	76	61.7
1	58	48	59	70	81	57	66	48	69	59	79	63.1
3	60	48	60	70	81	58	66	48	69	60	78	63.5
6	59	47	59	70	80	57	65	48	69	59	78	62.8
Mean	58.8	47.5	58.8	69.8	80.5	57.3	65·0	47.8	68.5	59.0	77.8	62.8
s	0·96	0.58	1.26	0·50	0.58	0.50	1.41	0.50	1·00	0.82	1.26	0.74
s(pool)	0·91	***R*** total	2.58	***(R)*** total	4.12							

A2.3 Normal IRHD, short-term repeatability

Time (days)	*Material*										
	1	2	3	4	5	6	7	8	9	10	11	
Laboratory no. 1												
0	55	44	53	67	77	55	60	44	66	59	80	60.0
1	55	44	53	67	77	55	61	44	66	58	79	59.9
Mean	55·0	44·0	53.0	67.0	77.0	55·0	60.5	44.0	66.0	58.5	79.5	60.0
s	0·00	0.00	0·00	0·00	0·00	0·00	0.71	0·00	0·00	0·71	0.71	0.06
s(pool)	0·37	***R*** total	1·05	***(R)*** total	1.74							

A2.3—(*Contd.*)

Time (days)	*Material*											
	1	2	3	4	5	6	7	8	9	10	11	
Laboratory no. 2												
0	55	42	52	66	75	55	60	44	65	58	78	59.1
2	55	43	53	66	76	55	60	44	65	58	78	59.4
Mean	55·0	42.5	52.5	66·0	75.5	55.0	60.0	44·0	65.0	58-O	78·0	59.2
s	0·00	0·71	0·71	0.00	0·71	0·00	0.00	0·00	0·00	0·00	0·00	0.19
s(pool)	0·37	***R*** total	1·05	***(R)*** total	1.76							
Laboratory no. 3												
0	56	44	54	67	77	56	61	44	66	56	83	60·4
2	57	45	54	68	77	56	62	45	67	56	83	60·9
Mean	56·5	44.5	54·0	67.5	77.0	56.0	61.5	44·5	66.5	56·0	83·0	60·6
s	0.71	0·71	0·00	0.71	0.00	0·00	0·71	0.71	0·71	0·00	0.00	0.39
s(pool)	0·52	***R*** total	1.48	***(R)*** total	2.44							

A2.4 Shore A, short-term repeatability

Time (days)	*Material*											
	1	2	3	4	5	6	7	8	9	10	11	
0	56	46	56	69	80	56	64	45	66	58	75	61·0
1	56	45	57	68	80	56	63	45	66	57	76	60.8
Mean	56·0	45·5	56.5	68.5	80·0	56·0	63.5	45·0	66.0	57·5	75.5	60·9
s	040	o-71	0·71	o-71	0·00	0·00	0·71	0·00	0.00	0·71	0·71	0·13
s(pool)	0·52	***R*** total	1.48	***(R)*** total	2.43							
Laboratory no. 2												
0	54	43	53	66	77	54	60	43	66	55	73	58·5
2	54	43	53	65	75	53	59	43	65	53	72	57.7
Mean	54·0	43.0	53.0	65.5	76·0	53.5	59.5	43.0	65.5	54·0	72.5	58.1
s	0·00	0·00	0·00	o-71	1.41	0.71	0.71	0·00	0.71	1·41	0.71	0.58
s(pool)	0·77	***R total***	2.18	(R) total	3.74							
Laboratory no. 3												
0	58	47	57	69	80	57	63	47	67	58	76	61.7
2	58	48	57	69	79	57	63	47	67	58	77	61.8
Mean	58·0	47.5	57·0	69·0	79.5	57.0	63·0	47·0	67.0	58.0	76.5	61·8
s	0·00	0.71	0·00	0·00	0·71	0·00	0·00	0·00	0·00	0·00	0.71	0·06
s(pool)	0·37	***R*** total	1·05	***(R)*** total	1.69							

A2.4—(*Contd.*)

Time (days)	*Material*											
	1	2	3	4	5	6	7	8	9	10	11	
Laboratory no. 1 Shore automatic												
0	58·0	45.3	55.6	67.6	78.4	55.9	624	45·7	66·2	56·7	75·1	60.6
4	58·0	45.4	55.7	67.5	78.1	55.7	62.0	45·2	66.6	56.2	75.2	60.5
Mean	58·0	45.3	55.7	67.6	78.3	55.8	62.2	45·5	66·4	56·5	75·2	60.6
s	0·00	0.07	0·07	0·07	0.21	0.14	0.28	0·35	0·28	0·35	0.07	0·08
s(pool)	0·21	***R*** total	0·60	***(R)*** total	0.99							

APPENDIX 3

A3.1 Normal IRHD, different operators, different hardness testers

Laboratory	*Material*					
	1	2	3	4	5	*Mean*
1	58	50	67	80	45	60·0
2	58	49	66	79	44	59·2
3	58	50	68	80	43	59.8
4	59	51	68	81	45	60.8
5	56	49	65	78	42	58·0
6	57	49	67	79	43	59·0
7	59	52	69	81	46	61.4
8	57	50	67	79	44	59.4
9	58	50	68	80	44	60.0
10	57	51	68	79	45	60.0
11	58	49	68	80	45	60.0
12	56	0	67	79	43	59.0
13	59	50	70	81	46	61.2
14	56		68	79	44	59·4
Mean	57.6	50.0	67·6	79.6	44.2	59.8
s	1·09	0·88	1.22	0.93	1.19	
s(pool)	1·07	***R*** total	3.03	***(R)*** total	5.06	

A3.2 Normal IRHD, different operators, same hardness tester

Laboratory	*Material*					
	1	2	3	4	5	*Mean*
1	57	49	68	79	43	59.2
2	57	49	66	79	43	58.8
3	58	51	68	80	45	60·4
4	58	51	68	80	44	60·2
5	54	48	65	78	42	57.4
6	57	49	68	80	43	59.4
7	57	50	67	79	44	59.4
8	57	49	67	79	43	59·0
9	57	50	67	79	44	59.4
10	57	50	68	80	43	59.6
11	58	49	67	79	44	59.4
12	57	49	66	78	43	59.6
13						
Mean	57.0	49·5	67.1	79.2	43.4	59.2
s	1·04	0·90	1·00	0.72	0.79	
s(pool)	0·90	***R*** total	2·55	***(R)*** total	4·30	

A3.3 Normal IRHD, one operator, different hardness testers

Laboratory	*Material*					
	1	2	3	4	5	*Mean*
1	57	50	67	80	44	59.6
2	58	49	66	79	44	59.2
3	58	51	67	80	44	60·0
4	57	50	67	80	43	59.4
5	57	50	67	79	44	59.4
6	56	49	67	79	43	58.8
7	57	50	67	79	44	59-4
8	57	50	67	79	44	59.4
9	57	50	67	80	44	59·6
10	57	50	67	79	45	59.6
11	56	49	67	79	44	59·0
12	57	50	67	79	44	59.4
13						
Mean	57-o	49.8	66.9	79.3	43.9	59.4
s	0.60	0·58	0·29	o-49	0·51	
s(pool)	0·51	***R*** total	144	***(R)*** total	2.42	

A3.4 Normal IRHD, one operator, same hardness tester

Laboratory	*Material*					
	1	2	3	4	5	*Mean*
1	57	49	68	80	43	59.4
2	56	48	66	79	43	58.4
3	57	49	68	80	44	59·6
4	57	48	66	80	42	58.6
5	57	49	66	79	43	58.8
6	57	49	68	79	43	59·2
7	57	50	66	79	44	59·2
8	57	49	66	80	43	59-o
9	57	50	67	80	43	59·4
10	57	49	68	80	43	59·4
11	57	50	67	80	44	59·6
12	56	50	66	79	43	58·8
13						
Mean	56.8	49.2	66.8	79.6	43.2	59.1
s	0.39	0.72	0·94	0·51	0·58	
s(pool)	0.65	***R*** total	1·85	***(R)*** total	3.13	

APPENDIX 4

A4.1 Micro IRHD, different operators, different hardness testers

Laboratory	*Material*					
	1	2	3	4	5	*Mean*
1	60	52	69	81	47	61.8
2	60	54	69	79	47	61.8
3	60	52	69	83	47	62.2
4	63	53	71	81	47	63.0
5						
6	61	52	72	83	47	63.0
7	61	52	71	83	47	62·8
8	60	52	70	81	47	62.0
9	60	51	68	78	45	60·4
10	62	53	72	83	47	63.4
11	60	51	71	81	46	61.8
12	60	52	70	81	47	62.0
13	59	52	71	82	47	62.2
Mean	60.5	52-2	70.3	81.3	46.8	62.2
s	1.09	0-83	1.29	1·61	0·62	
s(pool)	1·14	***R*** total	3.23	***(R)*** total	5·20	

A4.2 Micro IRHD, one operator, different hardness testers

Laboratory	Material					
	1	2	3	4	5	Mean
1	62	52	70	81	46	62.2
2	61	52	70	80	47	62.0
3	61	53	70	83	47	62.8
4	62	53	71	81	47	62.8
5						
6	60	52	71	81	47	62.2
7	61	53	72	82	47	63.0
8	60	51	69	80	47	61.4
9	61	52	69	80	47	61.8
10	61	52	72	83	47	63.0
11	60	51	70	80	45	61.2
12	59	51	69	80	46	61.0
13	59	51	69	80	45	60·8
Mean	60.6	51·9	70.2	80·9	46.5	62.0
s	1·00	o-79	1.11	1.16	0·80	
s(pool)	0.99	***R*** total	2.79	***(R)*** total	4·50	

APPENDIX 5

A5.1 Shore A, different operators, different hardness testers

Laboratory	Material					
	1	2	3	4	5	Mean
1	59	52	67	80	46	60.8
2	56	49	65	78	43	58.2
3	61	53	69	81	48	62.4
4	60	53	69	80	47	61.8
5	61	55	70	81	50	63.4
6	58	50	67	80	44	59·8
7	59	51	67	77	45	59.8
8	59	51	67	78	46	60.2
9	61	53	69	81	47	62.2
10	62	54	69	79	48	62-4
11	60	51	67	79	45	60·4
12	59	51	66	79	45	60·0
13	60	53	69	81	48	62-2
14	61	53	70	81	48	62-6
Mean	59.6	52.0	67-8	79.5	46.3	61-0
s	1.56	1.68	l-48	1.33	1.93	
s(pool)	1.61	***R*** total	4.55	***(R)*** total	7.46	

A5.2 Shore A, different operators, the same hardness tester

Laboratory	*Material*						*Load on tester N*
	1	2	3	4	5	*Mean value*	
1	60	52	68	80	46	61.2	14
2	59	49	66	78	45	59.4	13
3	60	52	68	79	45	60·8	56
4	58	49	65	78	44	58.8	12
5	60	52	68	79	46	61·0	19
6							
7							
8	58	49	65	78	44	58·8	12
9	56	50	67	78	45	59·2	28
10							
11	59	49	66	78	44	59.2	16
12	57	51	67	78	45	59.6	29
13							
14	58	49	67	78	44	59.2	12
Mean	58.5	50·2	66.7	78.4	44-8	59·7	21.1
s	1.35	1.40	1·16	0.70	o-79		
s (pool)	1.12	*R* total	3.16	(R) tot.	5.30		

A5.3 Shore A, one operator, different hardness testers

Laboratory	*Material*					
	1	2	3	4	5	*Mean*
1	60	52	68	80	45	61.0
2	57	49	65	78	43	58.4
3	59	51	68	80	47	61·0
4	61	53	70	81	47	62.4
5	60	51	67	79	48	61·0
6	59	53	69	80	46	61.4
7	60	53	69	80	47	61.8
8	60	52	68	79	47	61.2
9	60	52	68	80	45	61.0
10	61	52	69	79	45	61.2
11	60	51	68	80	45	60.8
12	60	52	68	80	46	61.2
13	60	53	69	81	47	62.0
14	60	53	69	80	47	61·8
Mean	59.8	51·9	68.2	79.8	46.1	61.2
s	0·97	1.14	1.19	0.80	1.33	
s(pool)	1·10	*R* total	3.12	(R) total	5.10	

A5.4 Shore A, one operator, the same hardness tester

Days	Material											
	1	2	3	4	5	6	7	8	9	10	11	
1	56	46	56	69	80	56	64	45	66	58	75	61·0
2	56	45	57	68	80	56	63	45	66	57	76	60·8
3	56	46	58	68	79	55	64	46	68	58	76	61.3
4	57	46	57	69	79	56	64	46	68	59	76	61.5
5	57	46	57	69	19	56	64	46	68	58	77	61.5
6	57	46	57	68	80	55	63	46	68	58	77	61·4
7	57	45	58	69	79	55	64	47	68	58	77	61·5
8	57	46	58	68	79	56	64	45	68	57	78	61·5
Mean	56·6	45·8	57·3	68·5	79.4	55·6	63·8	45·8	67·5	57·9	76.5	61·3
s	0·52	0·46	0·71	0·53	0·52	0·52	0·46	0·71	0·93	0·64	0.93	0·27
s(pool)	0·65	*r* total	1.84	(r) total	3·00							

A5.5 Shore A: influence of different load

The same operator and the same hardness tester
Instrument used: Shore Durotronic 1000 digital Shore A with automatic stand 902

Load (kg)	*Material*											
	1	2	3	4	5	6	7	8	9	10	11	*mv*
Material set 1: *ISO ITP set*												
1	58.0	45·3	55·6	67·6	78.3	55.9	62.4	45·7	65.6	56·7	75·1	60.6
5	60·7	48.9	59.3	69.4	79·2	58.5	64·0	49.0	69.0	58.8	77·3	63.1
Difference	2.7	3.6	3·7	1.8	0·9	2.6	1·6	3·3	3.4	2.1	2.2	2·5
Material set 2: Shore reference blocks												
1	34.6	38.1	53.8	59.1	70·6	79.7	87·9					60·5
5	36.2	41·2	55.1	60.8	73·7	82.3	90·0					62·8
Difference	1.6	3.1	1·3	1·7	3.1	2.6	2.1					2·2