XVI IMEKO World Congress
Measurement - Supports Science - Improves Technology - Protects Environment ... and Provides Employment - Now and in the Future
Vienna, AUSTRIA, 2000, September 25-28

EFFECTS OF SOME INFLUENCE PARAMETERS ON THE CALIBRATION METHOD FOR IRHD ELASTOMER HARDNESS TESTERS

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Abstract: The method for hardness measurement expressed in International Rubber Hardness Degree - IRHD - of rubber and plastics is described in the standard ISO 48 [1]. Rising requirements to the accuracy of hardness measurements have led to the result that verification/calibration methods for the testers acknowledged by a standard are needed. Results of these investigations will be used as a basis for the establishment of the relevant international standard which is in preparation. Some experiments for IRHD measurement by "Low-hardness test" - method L and "Microtest" - method M are presented in this paper. Practical measurements with different indenters and variation of other measurement conditions were carried out. For the obtained results regression evaluations were applied to determine the sensitivity coefficients for the influence quantities. Finally, the effects of all influence quantities on the uncertainty of hardness testers and on the calibration uncertainty were calculated and summarized.
Keywords: Hardness, rubber, IRHD, uncertainty

1 INTRODUCTION

Measurement methods for the hardness of vulcanized or thermoplastic rubber expressed in International Rubber Hardness Degree - IRHD are defined in ISO 48 [1]. Due to rising requirements to the accuracy of rubber hardness measurements the manufacturer and users of rubber hardness testers recently took an increasing interest in generally accepted calibration methods for the corresponding testers.

Therefore the DIN NMP 434 "Testing of the physical properties of rubber and elastomers" in Germany elaborated a calibration instruction for rubber hardness testers which is now dealt with in the ISO/TC 45 "Rubber and rubber products", subcommittee SC 2 "Physical testing and analyses". The planned adoption of this calibration regulation will contribute to guarantee stipulated accuracy levels of the measuring devices and thus to raise the confidence in hardness measurements of rubber and plastics.

In this paper results of the investigation of calibration methods for IRHD testers according to the methods L and M are presented.

2 METROLOGICAL REQUIREMENTS

Four different measurement methods are defined in the standard ISO 48 [1]. These methods are used for the determination of the hardness of vulcanized or thermoplastic rubbers on flat surfaces. The methods differ primarily in the diameter of the indenting ball and in the magnitude of the indenting force. The hardness values are expressed in International Rubber Hardness Degree - IRHD. In this paper between these scales special attention is devoted to the measuring methods "Low-hardness test" - method L - and "Microtest" - method M. The application ranges of these methods on the IRHD scale are as follows:

method L 10 IRHD to 35 IRHD
method M 35 IRHD to 85 IRHD

A short overview on calibration methods for IRHD hardness testers is given in [3].

The following requirements shown in Table 1 for hardness testers which perform the methods L and M according to ISO 48 should be fulfilled.

Table 1. Metrological requirements to IRHD testers according to the methods L and M

Measurand		method L	method M
ball diameter of indenter	d, mm	5,00 ± 0,01	0,395 ± 0,005
outer diameter of pressure foot	d_o, mm	20 ± 1	3,35 ± 0,15
hole diameter of pressure foot	d_i, mm	6 ± 1	1,00 ± 0,15
force on pressure foot	F_p, N	8,3 ± 1,5	0,235 ± 0,030
contact force	F_c, N	0,30 ± 0,02	0,0083 ± 0,0005
total force	F_t, N	5,70 ± 0,03	0,1533 ± 0,0010
duration of application of F_c	t_c, s	5 + 1	5 + 1
duration of application of F_t	t_t, s	30 + 1	30 + 1
indentation depth of indenter	h, mm	Δh = ± 0,01	Δh = ± 0,01

3 ESTIMATION OF THE UNCERTAINTY OF ISO IRHD TESTERS

Relying on the tolerances for IRHD testers according to ISO 48 the uncertainty of these testers can be estimated on the basis of the ISO guide [2]. The essence of this guide is to sum up the components (input quantities) of the uncertainty of measurement in form of variances $u^2(x_i)$. Because in our case it is justified to assume that the input quantities have a rectangular distribution (pessimistic assumption) we get:

$$u^2(x_i) = a_i^2 / 3 \qquad (1)$$

a_i - halfwidth of the distribution interval of the i-th component
Then the standard uncertainty u(H) is obtained from the standard uncertainties $u(x_i)$ of the input quantities with the help of the sensitivity coefficients $\partial H/\partial x_i$:

$$u^2(H) = \Sigma(\partial H / \partial x_i)^2 u^2(x_i) \qquad (2)$$

Under consideration of the expansion factor k = 2 the expanded uncertainty U is obtained:

$$U = k\ u(H) \qquad (3)$$

3.1 Determination of sensitivity coefficients

The application of the ISO guide [2] requires an as realistic as possible determination of the sensitivity coefficients $\partial H/\partial x_i$ between the output quantity hardness H and the input quantity x_i.
The sensitivity coefficients for the influence quantities indentation depth h and contact force F_c were calculated from an approximation of the measurement equation IRHD = f(h). For the quantities total force F_t, ball diameter d and application time t_t of the total force the sensitivity coefficients were determined under variation of the influence quantities.

The definition equation of IRHD

$$IRHD = \frac{100}{\sqrt{2p}} \int_{-\infty}^{x} e^{-x^2/2} dx \qquad (4)$$

for the range of IRHD(L) was approximated as

IRHD(L) = 76,135 – 52,1051 h + 14,9245 h^2 – 1,59929 h^3

From this approximated relationship the sensitivity coefficients $\partial H/\partial h_i$ -5,70, -12,71 and -17,22 IRHD(L)/mm for 10, 20 and 25 IRHD(L) were obtained.

In order to investigate the relationship IRHD = f(d) indenting balls with diameters 4 mm, 4,5 mm, 5 mm, 5,5 mm and 6 mm were used. For this aim rubber samples with the hardness values 10,5 IRHD(L), 14,2 IRHD(L), 22,2 IRHD(L) and 25,6 IRHD(L) were measured each with the above mentioned indenting balls. Fig. 1 shows the thus determined dependence of the hardness IRHD(L) on the ball diameter d.

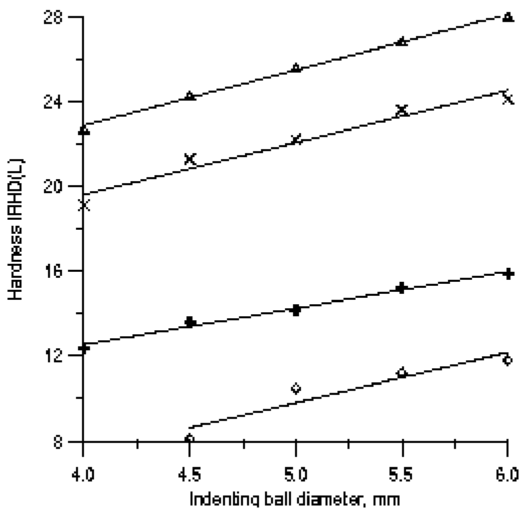

Figure 1. Dependence of the hardness IRHD(L) on the ball diameter d

From the functions depicted in Fig. 1 the sensitivity coefficient $\partial H/\partial d$ = f(IRHD) was calculated as a regression function $\partial H/\partial d = 4{,}64 - 0{,}3353\ H + 0{,}0102\ H^2$ (cf. Fig. 2).

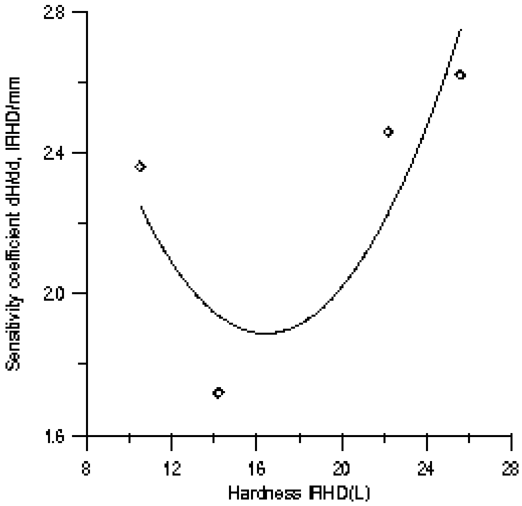

Figure 2. Relationship of the sensitivity coefficient $\partial H/\partial d$ = f(IRHD)

In Fig. 3 the dependence of the hardness IRHD(L) on the application time of the total force in a range t_t = 0 ... 35 s is presented. This relationship was determined on a sample with 14,2 IRHD(L).

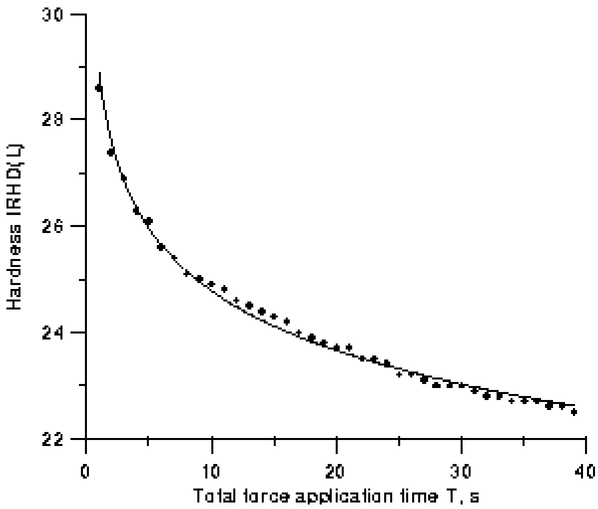

Figure 3. Dependence of the hardness IRHD(L) on the application time t_t of total force
The sensitivity coefficient $\partial H/\partial t_t$ is obtained from the derivative of the curve in Fig. 3 at t_t = 30 s.

By carrying out the same experiment with samples of different hardness we get the relationship $\partial H/\partial t_t = f(IRHD(L))$ (Cf. Fig. 4)

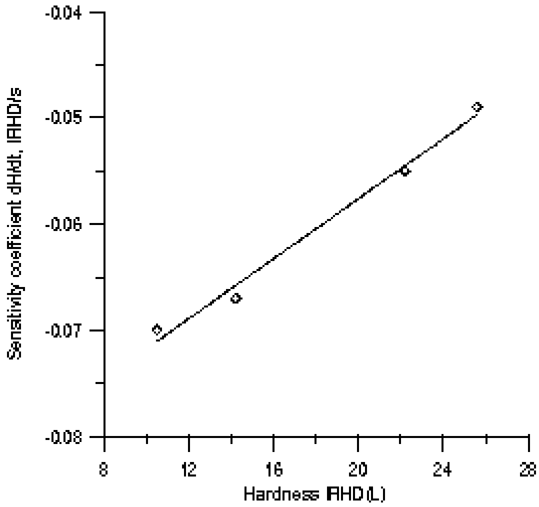

Figure 4. Relationship of the sensitivity coefficient $\partial H/\partial t_t = f(IRHD(L)$

From Fig. 4 we clearly see that the sensitivity coefficient $\partial H/\partial t_t$ slightly increases with increasing hardness IRHD(L).

In order to determine the sensitivity coefficient $\partial H/\partial F_t$ the total Force F_t was varied by 10 % of the nominal value. The result of this experiment is given in Table 2.

3.2 Estimation of the uncertainty

In Table 2 the uncertainty of IRHD(L) testers according to ISO 48 is estimated on the basis of equ. (2). The indicated limit values a_i fulfil the requirements of ISO 48 and the sensitivity coefficients $\partial H/\partial x_i$ are determined as described in 3.1.

Table 2. Estimation of the uncertainty of IRHD(L) rubber hardness testers

Influence quantity x_i	Limit value a_i	$u^2(x_i)= a_i^2/3$	$\partial H/\partial x_i$ at different hardness levels IRHD(L)			Contributions to variance $u^2(H)$ hardness levels IRHD(L)		
			10	20	25	10	20	25
F_t, N	0,03	$3{,}0.10^{-4}$	-4,37	-4,52	-3,16	0,00573	0,00613	0,00299
F_c, N	0,02	$1{,}3.10^{-4}$	10,25	9,94	5,90	0,01366	0,01284	0,00453
h, mm	0,01	$3{,}3.10^{-5}$	-5,70	-12,71	-17,22	0,00107	0,00533	0,00979
d, mm	0,01	$3{,}3.10^{-5}$	2,31	2,02	2,64	0,00018	0,00013	0,00023
t_t, s	0,5	$8{,}3.10^{-2}$	-0,07	-0,06	-0,05	0,00041	0,00030	0,00021
Uncertainty u						0,15	0,16	0,13
Expanded uncertainty U (k = 2)						0,3	0,3	0,3

From Table 2 we understand that the influence of the contact force F_c and of the indentation depth h of the indenter on the uncertainty are dominating. As expected, the influences from the ball diameter d and from the application time of the total force Tt are mastered well. If the requirements of ISO 48 on IRHD(L) rubber hardness testers are fulfilled the uncertainty of the tester can be estimated approximately as U = 0,3 IRHD(L). This value does not contain influences from the test specimen and the environmental conditions.

According to the same method the influences quantities on the uncertainty of IRHD(M) rubber hardness testers were analyzed.The results of this investigation are summarized in Table 3.

We clearly see that the uncertainty of the IRHD(M) rubber hardness testers is dominated by the influence from the depth measuring system. This influence could be reduced if for the limit value of the indentation depth h a smaller value than $a_i = 0{,}01$ mm is realized. At this point one must still investigate whether a higher accuracy of the depth measuring system under consideration of the properties of the to be tested samples can be utilized. Compared with IRHD(N) and IRHD(L) the influence of the ball diameter at IRHD(M) (d = 0,4 mm) is much larger. Therefore, in contrast with ISO 48 the tolerance of the ball diameter was halved to ± 2,5 µm because a tolerance of ± 5 µm led to deviations of 0,5

IRHD(M). This requirement to the ball manufacture can be fulfilled rather easily and does not cause a serious raise of costs.

Table 3. Estimation of the uncertainty of IRHD(M) rubber hardness testers

Influence quantity x_i	Limit value a_i	$u^2(x_i)= a_i^2/3$	$\partial H/\partial x_i$ at different hardness levels IRHD(M)			Contributions to variance $u^2(H)$ hardness levels IRHD(M)		
			40	60	80	40	60	80
F_t, mN	1,0	$3,3.10^{-7}$	-181,93	-181,93	-181,93	0,01102	0,01102	0,01102
F_c, mN	0,5	$8,3.10^{-8}$	378,31	378,31	378,31	0,01192	0,01192	0,01192
h, mm	0,01	$3,3.10^{-5}$	-152,41	-276,13	-365,64	0,76655	2,51618	4,41186
d, µm	2,5	2,08	-0,07	-0,07	-0,06	0,01019	0,01019	0,00755
t_t, s	0,5	$8,3.10^{-2}$	-0,08	-0,07	-0,06	0,00053	0,00041	0,00030
Uncertainty u						0,89	1,60	2,11
Expanded uncertainty U (k = 2)						1,8	3,2	4,2

4 CALIBRATION METHOD OF THE RUBBER HARDNESS TESTERS

The analysis of the error model presented in 3.3 shows that the calibration of the indentation depth measuring system and the test forces is most important. The calibration instruments must have a sufficient small uncertainty. An uncertainty amounting from 1/4 until 1/10 of the tolerances specified in Table 1 is considered as satisfying. For the different measurands the following calibration instruments as listed in Table 4 may be used.

Table 4. Calibration instruments used for the different measurands

Measurand	Calibration instrument
Indentation depth h	Special calibration device with length measuring system
Contact and total force F_c, F_t	Weighing instrument
Ball diameter d	Indicating micrometer
Outer and inner diameter of pressure foot	Vernier caliper
Application time of total force t_t	Stop watch

Table 5 contains an estimation of the uncertainty of measurement for the case that as limit values a_i the uncertainties are used which at present are kept at calibrations of IRHD hardness testers. Therefore these values can be interpreted as calibration uncertainties.

Table 5. Calibration uncertainties of IRHD hardness testers - methods L and M

Measuring method	At direct calibration realized uncertainty of measurement					Calibration uncertainty (k=2), IRHD
	U_{Ft}, mN	U_{Fc}, mN	U_h, mm	U_d, mm	U_t, s	
IRHD(L)	3,2	2,1	0,0027	0,002	0,1	0,1
IRHD(M)	0,2	0,2	0,0027	0,002	0,1	0,5 ... 1,2

On the calibration device for the indentation depth the measuring axes of the reference length measuring system and of the to be calibrated hardness tester must be aligned. As reference system digital length measuring screws with an uncertainty U = 1 µm to 2 µm are suited.

The calibration device for the test force contains a force measuring device and a feeding device. As Force measuring device a force transducer or also a balance can be used. If a balance is used the measured mass m under application of the value of local gravitational acceleration g_{loc} (uncertainty < 10^{-3}) must be converted into the force. The measuring axes of the force measuring device and of the hardness tester must be aligned vertically.

5 CONCLUSION

The analysis of the influencing quantities on the uncertainty of IRHD rubber hardness testers - methods L and M - shows that the direct calibration of the indentation depth and the test forces is especially important. For the fulfilment of the tolerances of corresponding hardness testers according to ISO 48 the reachable uncertainty is given. Appropriate calibration methods with a reasonable ratio between calibration uncertainty and tester tolerance of 1/4 to 1/10 can be realized.

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