

## **ESTIMATION OF UNCERTAINTY IN HARDNESS MEASUREMENT OF RUBBER AND OTHER ELASTOPLASTIC MATERIALS**

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**Abstract:** In this paper the approach and results for the estimation of uncertainty in measurements of rubber and plastics are presented. The developed uncertainty guidelines are based on "Guide to the Expression of Uncertainty in Measurement" (ISO GUM). As the most important and widely used hardness measuring methods for rubber and plastics the methods according to Durometer (A and D) and to the International Rubber Hardness Degree (IRHD), scales N, M and L are dealt with. Additionally a new hardness measuring method for soft silicones, called Hardness Very Low (HVL), which is still in the process of standardization, is

considered. According to the calibration methods direct calibration of test force, indentation depth and geometrical deviations of indenters must be carried out. Correspondingly the uncertainty of these direct calibrations have to be determined. In order to indicate the uncertainty based on these direct calibration expressed in hardness units, it is necessary to summarize the uncertainties of the direct calibration with the aid of sensitivity coefficients. Further, indirect calibrations with reference test blocks also require to indicate the uncertainty in hardness units.

**Keywords:** Uncertainty of Measurement, hardness of rubber and plastics

### **1. INTRODUCTION**

In the last years, the efforts of industry to enhance the quality of products have been increasingly supported on the one hand by the development of high-precision measuring methods and measuring devices, and on the other hand, by the international standardization of the basis for quality assurance. During the standardization of the basis for quality assurance, apart from ISO 9001, which describes a model for the quality assurance in the manufacturing, several basic standards have developed and sustainably influenced industrial measurements. Among these are in particular the ISO "Guide to the Expression of Uncertainty in Measurement" [1], which is abbreviated to "GUM", and the standard ISO 17025 [2] on general requirements for the competence of testing and calibration laboratories. On the basis of the GUM in the framework a guideline on the estimation of uncertainty in hardness measurements was elaborated.[3]

### **2. PURPOSE**

While consumers are generally only interested in the properties of the finished product, suppliers are also concerned about the properties of raw and compounded polymers. They wish to control the quality of production processes such as compounding, moulding, extrusion etc., in which by means of different reinforcing fillers and softeners different properties including hardness can be achieved.

The use of well-defined measurement methods makes it possible to compare measurement results, which is one of the basic conditions for international trade. The main measurement methods have been standardized on an international level by the ISO. They mainly include the methods Durometer (Shore) A and D, and the International Rubber Hardness Degree (IRHD), scales N, M, L.

### **3. METHODS**

The GUM was the first document to lay down a general, uniform guideline on how the uncertainty of measurement is to be determined. Basically, the GUM prescribes the following steps:

- Quantify the essential influence quantities of the measurement
- Determine the measurement deviations and the uncertainties of each influence quantity
- Correct for the measurement deviations
- Determine the sensitivity coefficients in order to transform the input quantities into the target quantity
- Summarize the uncertainties of the influence quantities into the combined uncertainty via the sum of their variances
- Calculate an expanded uncertainty for a given confidence level

The following error model is taken as basis. With the aid of the analyzed error influences, the uncertainty of the hardness measuring methods for rubber and plastics can be estimated. The basis of the method is the relationship between the hardness  $H$  and the input quantities  $x_i$ :

$$H = f(x_i), i = 1, \dots, n \quad (1)$$

The components of the uncertainty of measurement are added in the form of variances  $u^2(x_i)$ . As a rectangular distribution of the input quantities was assumed in the present case, the following results

$$u^2(x_i) = \frac{a_i^2}{3} \quad (2)$$

(a - half width of the distribution interval)

The standard uncertainty  $u(y)$  then results from the standard uncertainties of the input quantities:

$$u^2(y) = \sum_i \left( \frac{\partial H}{\partial x_i} \right)^2 u^2(x_i) \quad (3)$$

Taking the expansion factor  $k$  into account, which was set equal to 2, the expanded uncertainty  $U$  is obtained:

$$U = ku \quad (4)$$

#### 4. RESULTS

##### 4.1 Estimation of uncertainty for the hardness measurement methods Durometer A and D

The starting point for the estimation of the uncertainty are the metrological requirements of the measurement methods. For the durometer methods Shore A and D these requirements are laid down in the standard ISO 7619-1 [4]. In Table 1 these metrological requirements are summarized.

**Table 1. Metrological requirements to Shore Durometers types A, D, AO and AM**

Measurement quantity	Unit	Metrological requirement to the method			
		A	D	AO	AM
Shaft diameter of indenter $b$	mm	1.25±0.15	1.25±0.15		0.79±0.025
Indenter radius $r$	mm	-	0.1±0.01	2.5±0.02	0.1±0.01
Cone frustum diameter $d$	mm	0.79±0.01	-	-	-
Cone angle of indenter $\alpha$	°	35±0.25	30±0.25	-	30±0.25
Diameter of pressure foot $f$	mm	18±0.5	18±0.5	-	9±0.3
Area of pressure foot	mm <sup>2</sup>			≥500	
Hole diameter of pressure foot $a$	mm	3±0.1	3±0.1	5.4±0.2	1.19±0.03
Concentricity of pressure foot		Central	Central	Central	Central
Force on pressure foot	kgf	1 <sup>-0/+0.5</sup>	5 <sup>-0/+0.5</sup>	1 <sup>-0/+0.1</sup>	0.25 <sup>-0/+0.05</sup>
Indentation depth $t$	mm	0 to 2.50 $\Delta t = \pm 0.02$	0 to 2.50 $\Delta t = \pm 0.02$	0 to 2.50 $\Delta t = \pm 0.02$	0 to 1.25 $\Delta t = \pm 0.01$
Spring force on indenter $F$	mN	F=550+75H <sub>A</sub> $\Delta F = \pm 37.5$	F=445H <sub>D</sub> $\Delta F = \pm 222.5$	F=550+75H <sub>A</sub> $\Delta F = \pm 37.5$	F=324+4.4H <sub>AM</sub> $\Delta F = \pm 8.8$
Duration time of test force $T$	s	3 or 15	3 or 15	3 or 15	3 or 15

In ISO 868 [5] are given larger tolerances to the durometers as compared with the requirements laid down in ISO 7619-1. So the cone frustum diameter is  $d = 0.79 \text{ mm} \pm 0.03 \text{ mm}$ , the cone angle of the Shore D indenter is  $\alpha = 30^\circ \pm 1^\circ$  and the indentation depth for Shore A and D is  $t = 2,5 \text{ mm} \pm 0,04 \text{ mm}$ . For the measurement method Shore A the sensitivity coefficients  $\partial H / \partial x_i$  for the influence quantities  $F$  and  $t$  were derived from the measurement equations

$$F = 0.55 + 0.075 H_A \quad (5)$$

$$t = 2.50 - 0.025 H_A \quad (6)$$

(H<sub>A</sub> = hardness Shore A). The sensitivity coefficients for the influence quantities  $\alpha$  and  $d$  were determined with indenters which geometry parameters cone angle  $\alpha$  and cone frustum diameter  $d$  were intentionally varied.

For the determination of the sensitivity coefficients  $\partial H / \partial d$  5 different indenters were investigated, which cone frustum diameter were varied in the range from 0.73 mm and 0.65 mm. Basis of the following regression analysis are the measurement results  $\Delta H = f(d)$ . By regression analysis of the suchwise received slopes  $\partial H / \partial d$  the following function was obtained:

$$\frac{\partial H}{\partial d} = -0.0126181 H_A^2 + 1.64264 H_A - 30.647 \quad (7)$$

The relationship between the cone angle  $\alpha$  of the indenter and the hardness value were also received by measurements. 5 different indenters were used, which cone angles  $\alpha$  were varied between  $31^\circ$  and  $39^\circ$ . The following linear function was determined by regression analysis from the slopes  $\partial H / \partial \alpha$ :

$$\frac{\partial H}{\partial \alpha} = -0.0025392 H_A + 0.199376 \quad (8)$$

The influence of the duration time of the test force  $T$  was determined by measurement of the hardness change at a continuous duration time of the test force from 0 s to 30 s on various rubber hardness test blocks. For  $T = 3$  s the following dependence of the sensitivity coefficient  $\partial H / \partial T$  on the hardness  $H$  was received:

$$\frac{\partial H}{\partial T} = 0.0012 H_A + 0.242 \quad (9)$$

In Table 1 for the method Shore A the measurement uncertainty is determined for the case that the metrological requirements to a durometer Shore A according to ISO 7619-1 are fulfilled.

The expanded uncertainties U given in Table 1 can be interpreted as the uncertainties with which the method Shore A is defined in ISO 7619-1.

The analysis of the variances in Table 1 clarifies that the essential influences on the measurement uncertainty result from the measurement deviations of the indentation depth  $\Delta t$ , of the spring force  $\Delta F$  and of the cone frustum diameter  $\Delta d$ . The measurement uncertainty of the method Shore A can be reduced if above all these three influence factors are reduced. Compared with them the influences of the cone angle and of the duration time of the test force are small.

For the measurement method Shore D the sensitivity coefficients  $\partial H/\partial x_i$  for the influence quantities F and t were derived from the measurement equations

$$F = 0.445 H_D \quad (10)$$

$$t = 2.50 - 0.025 H_D \quad (11)$$

( $H_D$  = hardness Shore D). The sensitivity coefficients for the influence quantities  $\alpha$  and r were determined experimentally with indenters which geometry was varied intentionally.

By an investigation of the relationship between the tip radius of a Shore D indenter, which was varied in the range from 0.04 mm to 0.16 mm the hardness  $H_D$  was determined by the linear function

$$\frac{\partial H}{\partial r} = 0.1646 H_D + 70.7016 \quad (12)$$

based on a regression analysis of the slopes  $\partial H/\partial r$  of the curves  $H_D = f(r)$ .

Analogously, the relationship  $\partial H/\partial \alpha = f(H_D)$  was determined by the regression function:

$$\frac{\partial H}{\partial \alpha} = -0.00052 H_D^2 + 0.0615 H_D - 1.0663 \quad (13)$$

For the experimental investigations the cone angle of the indenter was varied in the range from  $26^\circ$  to  $34^\circ$ .

The influence which the duration time of the test force T exerts on the hardness was determined by measuring the hardness change on various rubber hardness test blocks for a continuous duration time of the test force from 0 s to 30 s.

The determination of the measurement uncertainty for the method Shore D is summarized in Table 2 for the case that the metrological requirements according to ISO 7619-1 are fulfilled.

**Table 2. Estimation of the uncertainty of Shore A rubber hardness measuring method**

Influence quantity $x_i$	Limit value $a_i$	$u^2(x_i) = a_i^2/3$	$\partial H/\partial x_i$ at various Shore A hardness levels			Contributions to variance $u^2(H)$ Shore A hardness levels		
			40	60	90	40	60	90
F, N	0.0375 N	0.000469	12.422	12.422	12.422	0.0724	0.0724	0.0724
t, mm	0.02 mm	0.00013	-40	-40	-40	0.2128	0.2128	0.2128
$\alpha$ , °	0.25°	0.020833	0.0978	0.0470	-0.029	0.0002	0.0000	0.0000
d, mm	0.01 mm	0.000033	14.870	22.487	14.985	0.0073	0.0167	0.0074
T, s	0.5 s	0.083	0.279	0.316	0.360	0.0065	0.0083	0.0108
Combined uncertainty u						0.55	0.56	0.55
Expanded uncertainty U (k=2)						1.09	1.11	1.10

**Table 3. Estimation of the uncertainty of Shore D rubber hardness measuring method**

Influence quantity $x_i$	Limit value $a_i$	$u^2(x_i) = a_i^2/3$	Sensitivity coefficients $\partial H/\partial x_i$ at various Shore D hardness levels			Contributions to variance $u^2(H)$ Shore D hardness levels		
			40	60	90	40	60	90
F, N	0.2 N	0.0133	2.247	2.247	2.247	0.0672	0.0672	0.0672
t, mm	0.02 mm	0.00013	-40	-40	-40	0.2128	0.2128	0.2128
$\alpha$ , °	1°	0.33	0.5612	0.7510	0.2554	0.1039	0.1861	0.0216
r, mm	0.01 mm	0.00003	77.282858	80.5779	85.5161	0.1989	0.2162	0.2435
T, s	0.5 s	0.0833	0.8	0.7	0.35	0.0533	0.0408	0.0102
Combined uncertainty u						0.76	0.83	0.74
Expanded uncertainty U (k = 2)						1.53	1.65	1.48

#### 4.2 Estimation of uncertainty for the hardness measuring methods IRHD, scales N, M, and L

For the hardness measuring method IRHD N for the influence quantities total force  $F_T$ , contact force  $F_C$ , indentation depth t, indenter ball diameter d and

duration time of test force  $T$  the uncertainty is determined for the case that the metrological requirements as laid down in ISO 48.[6]

The sensitivity coefficients  $\partial H/\partial x_i$  for the influence quantities indentation depth  $t$  and contact force  $F_C$  were obtained by an approximation of the measurement equation  $IRHD = f(t)$  and for the

quantities total force  $F_T$ , indenter ball diameter  $d$  and duration time of total force  $T$  by experiments under variation of the influence quantities. For the investigation of the relationship  $IRHD = f(d)$  indenter balls with the diameters 1.50 mm, 2.00 mm, 2.50 mm, 3.00 mm and 3.50 mm were used.

**Table 4. Metrological requirements to IRHD hardness testers according to ISO 48**

Measurement quantity	Unit	Metrological requirement to the method			
		N	H	L	M
Ball diameter of indenter $d$	mm	2.5±0.01	1.00±0.01	5.00±0.005	0.395±0.005
Concentricity of pressure foot		Central	Central	Central	Central
Diameter of pressure foot $f$	mm	20±1	20±1	22±1	3.35±0.15
Hole diameter of pressure foot $h$	mm	6±1	6±1	10±1	1.00±0.15
Force on pressure foot $F_f$	N	8.3±1.5	8.3±1.5	8.3±1.5	0.235±0.030
Incremental indentation depth $t$	mm	$t=f(IRHD)$ see ISO* Table 14 $\Delta t=\pm 0.01$	$t=f(IRHD)$ see ISO* Table 15 $\Delta t=\pm 0.01$	$t=f(IRHD)$ see ISO* Table 16 $\Delta t=\pm 0.01$	$t=f(IRHD)$ see ISO* Table 17 $\Delta t=\pm 0.01$
Contact force on indenter $F_C$	N	0.30±0.02	0.30±0.02	0.30±0.02	0.0083±0.0005
Total force on indenter $F_T$	N	5.70±0.03	5.70±0.03	5.70±0.03	0.1533±0.0010
Duration time of total force $F_T$ resp. contact force $F_C$	s	$T_T = 30$ $T_C = 5$			

see ISO\* means draft standard ISO/CD 18898 [7]

**Table 5. Estimation of the uncertainty of IRHD N rubber hardness measuring method**

Influence quantity $x_i$	Limit value $a_i$	$u^2(x_i) = a_i^2/3$	Sensitivity coefficients $\partial H/\partial x_i$ at various IRHD N hardness levels			Contributions to variance $u^2(H)$ IRHD N hardness levels		
			40	60	80	40	60	80
$F_T, N$	0.03 N	0.0003	-4.37	-4.52	-3.16	0.00573	0.00613	0.00299
$F_C, N$	0.02 N	0.00013	10.25	9.94	5.90	0.01366	0.01284	0.00452
$t, mm$	0.01 mm	0.000033	-25.32	-45.86	-65.61	0.02116	0.06940	0.14205
$d, mm$	0.01 mm	0.000033	9.54	7.01	4.48	0.00300	0.00162	0.00066
$T, s$	0.5 s	0.083	-0.027	-0.023	-0.021	0.00006	0.00004	0.00004
Combined uncertainty $u$						0.21	0.30	0.39
Expanded uncertainty $U$ ( $k=2$ )						0.42	0.60	0.78

In order to determine the measurement uncertainty of the scales IRHD M and L analogous investigations as for the uncertainty of IRHD N were carried out. The corresponding results are summarized in the Tables 4 and 5.

**Table 6. Estimation of the uncertainty of IRHD L rubber hardness measuring method**

Influence quantity $x_i$	Limit value $a_i$	$u^2(x_i) = a_i^2/3$	Sensitivity coefficients $\partial H/\partial x_i$ at various IRHD L hardness levels			Contributions to variance $u^2(H)$ IRHD L hardness levels		
			40	60	80	40	60	80
$F_T$ , N	0.03 N	0.0003	-4.37	-4.52	-3.16	0.00573	0.00613	0.00299
$F_C$ , N	0.02 N	0.00013	10.25	9.94	5.90	0.01366	0.01284	0.00452
$t$ , mm	0.01 mm	0.000033	-5.70	-12.71	-17.22	0.00107	0.00533	0.00979
$d$ , mm	0.01 mm	0.000033	2.63	2.49	2.42	0.00023	0.00021	0.00019
$T$ , s	0.5 s	0.083	-0.07	-0.06	-0.05	0.00041	0.00030	0.00021
Combined uncertainty $u$						0.15	0.16	0.13
Expanded uncertainty $U$ ( $k=2$ )						0.29	0.32	0.27

**Table 7. Estimation of the uncertainty of IRHD M rubber hardness measuring method**

Influence quantity $x_i$	Limit value $a_i$	$u^2(x_i) = a_i^2/3$	Sensitivity coefficients $\partial H/\partial x_i$ at various IRHD M hardness levels			Contributions to variance $u^2(H)$ IRHD M hardness levels		
			40	60	80	40	60	80
$F_T$ , N	1.0 mN	3.3 E-7	-181.9	-181.9	-181.9	0.01102	0.01102	0.01102
$F_C$ , N	0.5 mN	8.3 E-7	378.3	378.3	378.3	0.01192	0.01192	0.01192
$t$ , mm	0.01 mm	0.000033	-152.4	-276.1	-365.6	0.76655	2.51618	4.41186
$d$ , mm	2.5 $\mu$ m	2.08	-0.07	-0.07	-0.06	0.01019	0.01019	0.00755
$T$ , s	0.5 s	0.083	-0.08	-0.07	-0.06	0.00053	0.00041	0.00030
Combined uncertainty $u$						0.89	1.60	2.11
Expanded uncertainty $U$ ( $k=2$ )						1.79	3.19	4.23

## 5. DISCUSSION

The analysis of the quantities influencing the uncertainty of the Durometer and IRHD measurement methods shows that a calibration of the indentation depth, of the indenter geometry and of the test force is of particular importance.



**Fig. 1. Calibration of the force measurement system of an IRHD M tester with an electronic balance. The force is applied from the tester to the balance by a pendulum support.**

The fulfilment or reduction of a given limit of measurement uncertainty is proven by calibration. For rubber hardness testers the main calibration

items are the indenter geometry, depth measurement system and the force measurement system. Generally used calibration devices for the indenter geometry are measurement microscopes, for the depth measurement system - a length standard, like an incremental line scale system, and for the force measurement system - a force transducer. In Fig. 1 the force calibration of an IRHD M tester with an electronic balance is depicted. Fig. 2 shows the calibration of the depth measurement system with an incremental line scale system.

## 6. CONCLUSIONS

The presented guidelines show that the calculation of the measurement uncertainty can be carried out without difficulties. The introduction of the guidelines for the determination of the measurement uncertainty creates the preconditions for a further improvement of quality assurance in connection with rubber and plastics products on which the hardness has to be measured. In particular, one requirement given in the basic standards of quality assurance can be fulfilled now: that in practical hardness measurement it must be possible to indicate for each hardness measuring value its uncertainty. If it is necessary to frequently carry out uncertainty calculations it is recommended to convert the guidelines into a software program.

As result of the above mentioned investigations the following uncertainties can be achieved if the metrological requirements of corresponding ISO standards are fulfilled.



**Fig. 2. Calibration of the depth measurement system of a Shore A durometer on the basis of a length standard with a high resolution incremental line scale system**

**Table 8. Measurement uncertainties ( $k = 2$ ) for the Durometer methods Shore A and D achievable on the basis of ISO 7619-1 and ISO 868**

Standard	Measurement method	
	Shore A	Shore D
ISO 7619-1	1.1	1.7
ISO 868	4.2	2.4

**Table 9. Measurement uncertainties ( $k = 2$ ) for the methods IRHD N, L M achievable on the basis of ISO 48**

IRHD scales		
N	L	M
0.8	0.3	4.2

#### REFERENCES

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