National Institute of Standards & Technology

Certificate

Standard Reference Material® 2818

Rockwell Hardness 15N Scale - High Range (Nominal 91 HR15N)

Serial Number: SAMPLE

This Standard Reference Material (SRM) is a transfer standard intended primarily for use in the calibration and verification of the performance of Rockwell hardness equipment using the Rockwell Hardness 15N scale (HR15N). A unit of SRM 2818 is a steel test block, nominally 64 mm in diameter and 15 mm thick, having a polished test surface described by a micro-engraved circle 52 mm in diameter as shown in Figure 1. Each SRM unit bears a unique serial number on the edge of the block.

SRM 2818 is individually certified by performing seven hardness measurements at specific locations on the test surface as indicated in Figure 1. Recommended test conditions, procedures, and precautions for the proper use of this SRM are provided in this certificate. In order to perform calibration or verification measurements over the full usable range of the HR15N scale, this SRM may be used in conjunction with SRM 2816 Rockwell Hardness 15N Scale - Low Range (Nominal 72 HR15N) and SRM 2817 Rockwell Hardness 15N Scale - Mid Range (Nominal 83 HR15N).

Expiration of Certification: The certification of **SRM 2818** is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). Accordingly, periodic recalibration or recertification of this SRM is not required. However, the certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this material over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Hardness measurements leading to the certification of this SRM were performed by R.D. Jiggetts and S.R. Low of the NIST Metallurgy Division.

Statistical analysis was performed by W.S. Liggett of the NIST Statistical Engineering Division.

Geometric verification of the NIST Rockwell diamond indenter used in the certification of this SRM was conducted by J.F. Song and T.V. Vorburger of the NIST Precision Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Frank W. Gayle, Chief
Metallurgy Division

Gaithersburg, MD 20899
Certification Issue Date: 08 December 2011

Robert L. Watters, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

This SRM is made of steel, and thus is susceptible to scratches, dents, and other mechanical damage, particularly on the top and bottom surfaces, as well as being susceptible to corrosion from moisture and reaction to skin oils. The SRM is wrapped in corrosion inhibiting paper and packaged in a protective wooden box.

Storage: When not in use, the SRM should be rewrapped and stored in the box or in a manner having equivalent or better mechanical and corrosion protection.

Handling: The user should wear protective gloves, such as lint-free cotton or powder-free latex gloves, while handling the SRM. If gloves are not worn, the SRM should be cleaned with ethyl alcohol or other suitable cleaning agent and dried thoroughly before storing. (See "Cleaning the SRM," below.) The SRM should be held only by the round edge to avoid excessive contact with the top and bottom surfaces.

Use: The SRM is intended for use with all equipment capable of conducting an HR15N indentation hardness test. The HR15N hardness test is defined as requiring the use of a Rockwell spheroconical diamond indenter and the following test forces: a preliminary force of 29.4 N (3 kgf) and an additional test force of 117.7 N (12 kgf) that, when combined with the preliminary force, generates the total test force of 147.1 N (15 kgf). The Rockwell hardness tester should be set up and operated in accordance with the recommendations of the manufacturer and as required by applicable test method standards. All hardness measurements must be made within the circle engraved on the test surface of the SRM.

Test Environment: It is recommended that the hardness tester to be calibrated or verified be kept in a temperature- and humidity-controlled environment maintained at 23 °C ± 2 °C (73 °F ± 5 °F) and a relative humidity of 50 % or less. The tester must also be in a location that is free from shock or vibration that could affect the hardness measurements.

Block Support Anvil: Use of a flat anvil is recommended (i.e., an anvil that can support the SRM for indentation anywhere on its test surface). However, when calibrating or verifying a hardness tester, the same anvil must be used with the SRM that will be used on the tester after the calibration or verification.

Cleaning the Tester Anvil and Indenter: The anvil and indenter diamond tip should be thoroughly cleaned per recommendations from the manufacturer. In the absence of cleaning instructions from the manufacturer, it is recommended that the anvil and indenter be cleaned with ethyl alcohol and dried using a lint-free paper towel or cloth. Then the surfaces should be blown clean of dust using filtered air, such as from a commercial compressed air can or bottle. **DO NOT** blow clean by mouth.

Cleaning the SRM: Prior to use, it is recommended that the SRM be cleaned. A recommended method for cleaning the SRM is to gently wipe the top and bottom surfaces with clean cotton or a non-abrasive cloth thoroughly wetted with ethyl alcohol. The metal surfaces should immediately be dried using a soft lint-free paper towel or cloth before the alcohol evaporates. This cleaning must be performed in a manner that prevents a residue from remaining on the top and bottom surfaces. The alcohol cleaning should be followed by blowing the surfaces clean of dust with filtered air. The top and bottom surfaces should **NOT** be touched after cleaning.

Seating the Anvil and Indenter: Prior to making hardness indentations on the SRM, the tester anvil and indenter must be adequately seated. This can be accomplished by performing Rockwell hardness tests on a material using the highest test force the tester is capable of applying. The seating tests should be repeated until successive measurement values show no trend of increasing or decreasing hardness.

Placement of the SRM on the Anvil: Immediately before placing the SRM on the anvil, the top surface of the anvil and the bottom surface of the SRM should be cleaned as described above. The SRM should be placed at once on the anvil before dust can return. The top test surface of the SRM should be blown free of dust prior to testing and occasionally during the period of use. The SRM should be slid several times back and forth over the surface of the anvil to help seat the block on the anvil. Any time the SRM is lifted from and replaced on the anvil, the procedure described above in this paragraph should be repeated.

Preliminary Indentation: When a flat anvil is used, at least one preliminary HR15N test should be performed at any location on the test surface of the SRM. The preliminary test will help seat the SRM on the anvil. The measured hardness value of the preliminary test should be ignored. A preliminary indentation is not necessary when using a spot anvil.

Indentation Location: The user is cautioned not to make any indentation such that it contacts a previous indentation, the engraved circle, or the NIST logo; doing so may damage the indenter. No indentation should be made outside the engraved circle. Under no circumstance shall an indentation be made on the bottom surface of the block.

Test Cycle: This SRM unit has been certified by performing Rockwell 15N scale hardness tests using a specific test cycle. The HR15N test cycle may be characterized by specifying four test cycle parameters that have been determined to have a significant influence on the measurement results. The four parameters are: (1) preliminary test force dwell time; (2) total force dwell time; (3) recovery dwell time; and (4) indenter velocity during application of the additional force. These parameters are defined as follows:

> ***Preliminary Test Force Dwell Time*** – the time period beginning when the preliminary force is fully applied and ending when the first baseline measurement of indenter depth is taken.
>
> ***Total Force Dwell Time*** – the time period during which the total force is fully applied.
>
> ***Recovery Dwell Time*** – the time period beginning when the additional test force is fully removed returning the applied force to the preliminary force level and ending when the final measurement of indenter depth is taken.
>
> ***Additional Force Indenter Velocity*** – the indenter velocity during the application of the additional force. For Rockwell 15N scale tests, it is the final 25 % of the total force application that has the most significant effect on the measurement result.

The test cycle parameters used for the certification of this SRM are given in Table 1. To minimize the uncertainty in the hardness measurement, a test cycle should be used that replicates, as closely as possible, the SRM certification test cycle parameters. Deviations from the SRM test cycle in dwell times or force application rate will result in measured hardness values that deviate from measurements made using the SRM certification test cycle.

Table 1. Specifications for the SRM Test Cycle Parameters

Parameter	Value	NIST Tolerance
Preliminary Test Force Dwell Time	3 s	± 0.1 s
Total Force Dwell Time	5 s	± 0.1 s
Recovery Dwell Time	4 s	± 0.1 s
Average Indenter Velocity [during the final 25 % of total force application]	13 µm/s to 24 µm/s	

Indentation Spacing: Rockwell hardness measurements can be influenced by nearby indentations that were previously made. The amount that a measurement can be affected is dependent on the distance between indentations and the number of surrounding indentations. The measured hardness increases as the distance between indentations becomes smaller and the number of surrounding indentations is increased.

For this SRM, NIST studies have shown that when a test is made too close to previously made indentations, the resulting hardness value may be higher than the true hardness. The user should consider this possible elevation of hardness when making measurements on this SRM. Table 2 lists the effect of indentation-to-indentation spacing for this SRM. These stated elevations in hardness are given to illustrate the possible effects of making tests near previously made indentations, and should not be used as correction values for measurements made too close to other indentations.

Table 2. Effect of Indent Spacing

Spacing distance in an indentation-filled test area (center to center)	Increase in measured hardness
2.0 mm	+0.03 HR15N
1.5 mm	+0.05 HR15N

Certified Average Hardness Value: The certified average hardness value is an estimate of the average hardness across the test surface of the block. Hardness varies slightly across the surface due to material non-uniformity and can be modeled by a mathematical function. The average hardness value is defined as that hardness function integrated over the test block surface divided by the surface area. The certified average hardness value is based on the seven NIST hardness measurements and on the material uniformity as discussed below. This average value is defined as the hardness function integrated over the test block surface divided by the surface area, and as such, is the average of the predicted hardness values for *all* test surface locations, and *not* the arithmetical average of the seven NIST measurements. However, since the locations chosen for the seven NIST measurements provide a good representation of the surface, the certified average hardness is close in value to the arithmetical average. The NIST certified average hardness value is only somewhat analogous to the certified value assigned to a commercially calibrated hardness test block that is typically calculated as the arithmetical average of several calibration measurements.

Certified Average Hardness Value: SAMPLE ± 0.21 HR15N

Uncertainty of the Certified Average Hardness Value: The expanded uncertainty, U, in the certified average hardness was calculated using $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO Guide [1], and $k = 2$ is the coverage factor. The value of u_c represents the combined effect of the individual sources of uncertainty listed in Table 3. These sources include uncertainties due to deviations of the NIST standardizing tester and indenter from the defined requirements for the Rockwell hardness test (forces, depth measurement, test cycle, and indenter shape) [2]. The value of u_c is calculated as follows:

$$u_c = \sqrt{\sigma_1^2 + \sigma_2^2 + \sigma_3^2 + \sigma_4^2} \ .$$

Table 3. Sources of Uncertainty for the Certified Average Hardness Value

Type		Uncertainty Source	Standard Uncertainty
σ_1	A	Material Uniformity and Measurement Repeatability	± 0.008 HR15N
σ_2	A	Day-to-Day Reproducibility	± 0.027 HR15N
σ_3	B	NIST Standardizing Hardness Tester	± 0.019 HR15N
σ_4	B	NIST Standardizing Indenter	± 0.100 HR15N
u_c		Combined Standard Uncertainty	± 0.106 HR15N
U		Expanded Uncertainty	± 0.21 HR15N

The expanded uncertainty, U, provides an indication of the accuracy of NIST's estimate of the true value of the average hardness of this SRM. The uncertainty should not be interpreted as the range in expected hardness values that would be measured across the test surface, nor is it a limit of acceptable hardness values for verifying Rockwell hardness equipment. Similarly, σ_1, σ_2, σ_3, and σ_4 are uncertainty components that contribute to the overall uncertainty in the certified average hardness value and are not ranges of expected hardness results due to each source of uncertainty.

The certified average hardness value provides the user with a comparison value for performing indirect verifications [2] of Rockwell hardness machines for the HR15N scale. In doing the comparison, the user cannot measure the surface-wide average hardness, only the hardness values at a collection of properly-spaced points. This distinction leads to two modes of certification, which in the case of this SRM differ only in the uncertainty due to material uniformity and measurement repeatability. The first mode is certification of the average discussed above. The second mode is prediction of what hardness readings NIST would obtain with its machine and indenter were it to measure the points chosen by the user. Let the number of points chosen by the user be denoted by n. For the second mode of certification, the certified value is the same as in the first mode, but the uncertainty due to material uniformity and measurement repeatability is 0.022*sqrt (1/7 + 1/n).

Certification Procedure: The NIST standardizing hardness tester is a specially designed machine using directly loaded dead-weights for applying the required forces, and a laser interferometry displacement sensor for measuring the depth of indentation. The geometry of the NIST standardizing diamond indenter was verified using a stylus instrument [3]. The indenter measurements and expanded uncertainties are 197.09 µm ± 0.64 µm for the tip radius and 119.96° ± 0.02° for the cone angle.

Several test blocks were randomly selected from each production batch, and hardness measurements were made over the test surface of each SRM using the NIST standardizing hardness tester and diamond indenter. Hardness was measured by indentation tests in accordance with the Rockwell Hardness 15N scale principle [2] and following the test cycle given in this certificate. By analyzing the hardness profile of this set of test blocks, information on material uniformity was developed. NIST determined that the hardness varies across the surface of each block and varies differently from block to block. This hardness variation is due primarily to the nature of steel and the manufacturing process.

Rockwell Hardness 15N scale measurements were then made at eight locations on the test surface of each SRM as illustrated in Figure 1. The first indentation, labeled "Seat," was made to ensure that the new test block was adequately seated on the anvil. "Seat indentation" data are potentially erroneous; thus, the resulting hardness value from this first indentation was discarded. The seven hardness tests that followed, labeled 1 through 7, were used in the determination of the SRM certified average hardness values for that SRM. The seven NIST measurement values and locations are given in Table 4. The x, y coordinate system is such that location $x = 0$, $y = 0$ is at the block center (NIST indentation 4) and oriented with the NIST logo at the bottom of the block as illustrated in Figure 1.

Table 4. NIST Hardness Measurements for Individual Test Block Locations

Location	*x*	*y*	Hardness Value (HR15N)
1	– 10	17	SAMPLE
2	20	0	SAMPLE
3	– 10	– 17	SAMPLE
4	0	0	SAMPLE
5	10	17	SAMPLE
6	10	– 17	SAMPLE
7	– 20	0	SAMPLE

Traceability: This SRM can be used to demonstrate that a user's measurements are traceable to the HR15N scale as determined by NIST; however, the procedures required to demonstrate traceability are varied depending on the user's needs. In general, acceptable transfer of the NIST hardness values to the user can be accomplished in three ways: (1) *verification within tolerances* – verifying that the user's measurements are in agreement with NIST certified values within acceptable tolerance limits; (2) *verification within uncertainties* – verifying that the user's measurements are in agreement with NIST certified values within the uncertainty ranges of the user's and NIST's measurements; and (3) *calibration* – calibration of the performance of the user's hardness machine to the NIST certified values by making appropriate mathematical corrections, and then verifying that the user's corrected values are in agreement with NIST certified values within the uncertainties of the user's and NIST's measurements. Each of the three methods requires differing levels of effort by the user to determine the accuracy in their measurements.

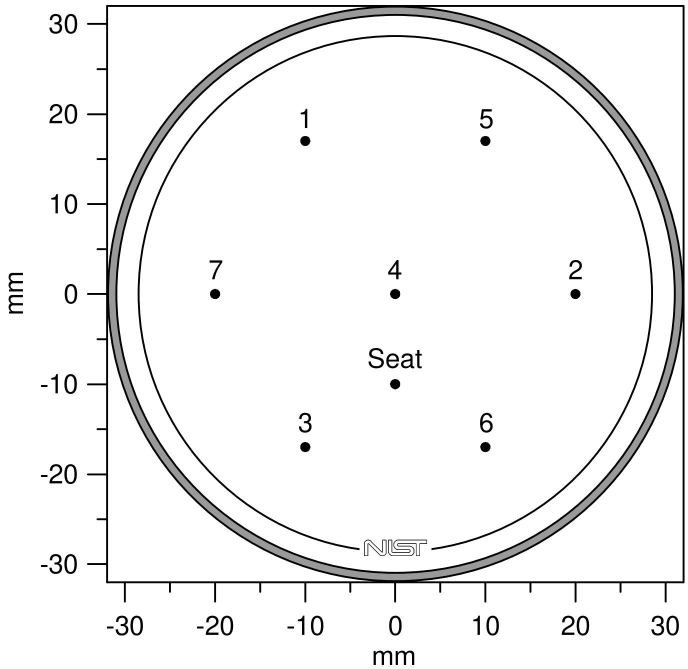

Figure 1. Test surface of SRM indicating the location and sequence of certification indentations (shown as black dots), and illustrating the appearance of the engraved circle and NIST logo. The first indentation, labeled "Seat", was made to ensure that the new test block was adequately seated on the anvil. The indentations labeled 1 through 7 indicate the indentation sequence used in certification.

REFERENCES

[1] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Dec 2011); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed Dec 2011).

[2] ASTM E18; *Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials*; Annu. Book ASTM Stand.

[3] Song, J.F.; Rudder, F.F. Jr.; Vorburger, T.V.; Smith, J.H.; *Microform Calibration Uncertainties of Rockwell Diamond Indenters*; J. Res. Natl. Inst. Stand. Technol., Vol. 100 (1995).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.