U. S. Department of Commerce
Maurice H. Stans
Secretary

onal Bureau of Standards
M. Branscomb, Director

Certificate of Calibration

Standard Reference Material 725

for

Isomer Shift of Iron Compounds

Sodium Pentacyanonitrosylferrate II[a] (Sodium Nitroprusside)

Use

This standard reference material is issued for the calibration of the isomer shift of iron compounds and alloys and to provide a uniform basis for presentation of Mossbauer isomer shift data. The center of the sodium nitroprusside doublet at 25.0 °C is defined as zero isomer shift, from which all isomer shifts of iron-bearing materials should be measured. This provides a suitable "benchmark" for the isomer shift of iron compounds. Since the peak positions of Standard Reference Material 1541, iron foil, are measured with respect to this standard, the iron foil also can serve as a "benchmark."

Certified Value

The isomer shift of this standard has an average value of 0.0000mm/s at 25 ± 0.2 °C. For discussion of uncertainty see below.

The isomer shift of this standard is compared to that of the National Bureau of Standards Primary Standard which has the value of zero mm/s isomer shift.

Statement of Uncertainties

Combined unmeasured systematic errors for the isomer shift are not expected to exceed +0.0005 or −0.0005. For a description of these errors see reference [1].

The portion of variability due only to heterogeneity of the material for the size of platelet indicated below is less than 0.1 percent which is negligible in comparison to other sources of variability.

The standard deviation of a single measurement was found to be 0.0005 mm/s. This estimate which is based on 24 independent measurements (23 degrees of freedom) is a measure of reproducibility between replicate measurements. The standard deviation of the certified average value, derived from this estimate, is 0.0001 mm/s. The 95 percent confidence

(over)

interval for the mean is approximately ± 0.0002 mm/s. The total uncertainty must be increased to take into account the systematic errors.

Description

This standard reference material was prepared from a single crystal of the compound sodium pentacyanonitrosylferrate (II) dihydrate, $Na_2[Fe(CN)_5NO]\cdot 2H_2O$. The purity of this compound, also known as sodium nitroprusside, meets the specifications of the American Chemical Society for reagent-grade materials as verified by quantitative analysis of the main constituents, but as heavy metals. The standard is in the form of a platelet of dimensions 1 × 1 × 0.0775 ± 0.003 cm that has been cut from a large single crystal. The 1 × 1 cm surface is parallel to the 010 crystal plane (a<b<c) within ± 2 degrees of arc. The opposite 1 × 1 cm surfaces are parallel to within 0.001 cm with a surface finish of 0.5 μm. The natural iron concentration is 25.0 ± 1.0 mg/cm^2. The resonant spectra have considerable line-broadening due to the thickness of the absorber. The experimental line width (full width at half maximum) is approximately 0.289 mm/s for a ^{57}Co-Pd source. The Mossbauer effect, background corrected, is approximately 17 percent for the NBS spectrometer.

Additional Information

The velocity scale should be calibrated using iron foil, Standard Reference Material 1541 or by other means, but for a check of the Mossbauer spectrometer, the separation of the peaks is 1.7015 ± 0.0002 mm/s at 25.0 ± 0.2 °C (24 determinations). Error is the standard deviation of the average. The standard deviation for a single determination is 0.001 mm/s. See note on supersession given below.

The constant acceleration optical-interferometric-feedback spectrometer was used to make these measurements. [1].

These data were derived from a least squares fitting of a Lorentzian function to the separate peaks. No interrelation between the peak positions was assumed. Therefore, this SRM is issued provisionally pending completion of a study of systematic errors due to the possibility of alternate mathematical models to that used in the analysis of the spectrum.

Acknowledgments

The single crystal platelets of sodium nitroprusside were prepared by the Isomet Corporation of Palisades Park, New Jersey. The calibration of the crystals was made at the National Bureau of Standards, Institute for Materials Research, by J. J. Spijkerman, F. C. Ruegg, J. C. Travis, and J. R. DeVoe. An independent method of measurement was used by L. H. Bennett and L. J. Swartzendruber, of the Alloy Physics Section, Metallurgy Division. [2]

Cooperating Laboratories[b]

Prof. R. Fritz
Westfalische Wilhelms Universität
Munster, Germany

Prof. James Mullen
Purdue University
Lafayette, Indiana

Dr. Ronald W. Grant
North American Rockwell
Science Center
Thousand Oaks, California

Prof. Paul Flinn
Carnegie Institute of Technology
Pittsburgh, Pennsylvania

Prof. U. Gonser
Institut für Metallphysik und Metallknude
Universität des Sarlandes
66 Saarbrucken 15 Germany

The overall coordination of the technical work leading to the certification of this SRM was performed by J. R. DeVoe, Chief, Radiochemical Analysis Section.

[a]This nomenclature is based upon the oxidation states of this compound as implied by interpretation of Mossbauer spectral data.

[b]The quadrupole splitting quoted in this certificate was determined by NBS. The values obtained by the cooperating laboratories are not given, but agree with the NBS value within the cooperator's measured uncertainties.

*The details of the instrumentation and of preparation and measurement of all the Mossbauer standards are given in references [1, 2]. For information on work done in other laboratories see the citations in reference 1.

References

[1] DeVoe, J. R., Spijkerman, J. J., Travis, J. C., Standards for Mossbauer Spectroscopy of Iron Compounds, NBS Spec. Publ. 260-20 (A revised publication will appear in June 1971).

[2] Swartzendruber, L. J. and Bennett, L. H., J. Res. NBS, Vol 74A, No. 5, 691-697 (Sept.-Oct. 1970).

Directions for Use

The standard reference material in the form of a single crystal platelet is sandwiched between two pieces of 4 mil polyethylene film. It is recommended that the platelet remain in this moisture-proof container. If this precaution is observed, the life of the standard should be indefinite. A suitable mounting consists of two concentric aluminum rings approximately 1.25 inches in diameter. The crystal, encased

in the plastic envelope, is placed between the rings and the assembly is fastened with small screws, thereby clamping the envelope firmly between the rings. Both rings have an inside diameter of 0.75 inch to expose the crystal and to prevent crushing of its edges. This procedure was followed in obtaining the data at NBS on this standard reference material.

Place the standard reference material as the absorber at 25.0 °C in the Mossbauer spectrometer and take sufficient transmitted counts of the 14.4 keV gamma ray from an iron-57 source to obtain counting statistics to the required degree of uncertainty. Determine the peak positions of the quadrupole doublet. The use of digital computation techniques is recommended. Divide the distance between the peaks by two and assign this as the value for isomer shift indicated above. Replace the standard crystal with the compound whose isomer shift is to be measured as the absorber in the spectrometer. Measure the peak position parameters as a difference between that of the sample under study and the mid-point between the peaks of the standard.

Washington, D. C. 20234 J. Paul Cali, Chief
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(This certificate supersedes certificate of July 28, 1967.)