U. S. Department of Commerce
John T. Connor, Secretary
National Bureau of Standards
A. V. Astin, Director

# Certificate of Calibration

## Standard Reference Material 725
## for
## Mossbauer Differential Chemical Shift for Iron-57
## Disodium Pentacyanonitrosoferrate Dihydrate
## (Sodium Nitroprusside)

This Standard Reference Material was prepared from a single crystal of the compound disodium pentacyanonitrosoferrate dihydrate ($Na_2Fe(CN)_5NO.2H_2O$). 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 should not be considered as entirely free from impurities such as heavy metals. It is in the form of a platelet of dimensions 1x1x0.0775 $\pm$0.003 cm that has been cut from a large single crystal. The 1x1 cm surface is parallel to the 100 crystal plane within $\pm$2 degrees of arc. The opposite 1x1 cm surfaces are parallel to within 0.001 cm with a surface finish of 20 microns. The natural iron concentration is 25.0 $\pm$4 percent mg/cm$^2$.

The chemical shift of this Standard is compared to that of the National Bureau of Standards Primary Standard, consisting of the average chemical shift of ten platelets which is given the value of zero chemical shift. The National Bureau of Standards dual-spectra Mössbauer spectrometer [1, 2] was used. This Standard has an average value for the chemical shift of 0.0000 $\pm$0.0002 cm/s at 25.0°C. This uncertainty is expressed as the standard deviation of a single determination derived from single measurements on 50 platelets. The resonant spectra have considerable line-broadening due to the thickness of the absorber. The experimental line width (full width at half maximum) is 0.0305 $\pm$0.0004 cm/s. The line width corrected for thickness broadening is 0.0202 cm/s. The Mössbauer effect is 17 percent.

To allow the user to calibrate the velocity scale of a Mössbauer spectrometer, the electric quadrupole splitting of the Standard Reference Material was measured. The velocity scale of the dual-spectra comparison spectrometer was calibrated by measurement of the electric quadrupole splitting of the Primary Standards using an optical interferometric technique. The average value of the electric quadrupole splitting for this Standard is 0.1726 $\pm$0.0002 cm/s at 25.0°C where the uncertainty is expressed as the standard deviation of a single determination derived from single measurements on 10 platelets.

The details of the instrumentation and of preparation and measurement of the sodium nitroprusside crystals are given in NBS Miscellaneous Publication 260-13 entitled "Mössbauer Spectrometry Standard for Chemical Shift of Iron Compounds."

The single crystal platelets of sodium nitroprusside were prepared by the Isomet Corporation of Palisades Park, New Jersey. The calibrations of the crystals were made within the National Bureau of Standards Institute for Materials Research, by J. J. Spijkerman, F. C. Ruegg, D. K. Snediker, and W. L. O'Neal of the Radiochemical Analysis Section, James R. DeVoe, Chief.

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REFERENCES:

[1] Radiochemical Analysis: Activation Analysis, Instrumentation Radiation Techniques, and Radioisotope Techniques, July 1963 to June 1964, James R. DeVoe, Editor, NBS Tech. Note 248, pp 25-37 (1964). Available from Superintendent of Documents, Government Printing Office, Washington, D. C. 20402, 50 cents a copy.

[2] Radiochemical Analysis: Activation Analysis, Instrumentation, Radiation Techniques, and Radioisotopes Techniques, July 1964 to June 1965., James R. DeVoe, Editor, NBS Tech. Note 276, pp 74-110 (1966). Available from Superintendent of Documents, Government Printing Office, Washington, D. C. 20402, $1.00 a copy.

WASHINGTON, D. C.
April 6, 1966

W. Wayne Meinke, Chief
Office of Standard Reference Materials

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## Directions for Use

The Standard Reference Material in the form of a single crystal platelet is sandwiched between two pieces of a 4 mil polyethylene film. Due to the advisability of keeping it free from moisture it is recommended that the platelet not be removed from this container. 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 together 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 was the procedure used in taking all of the data at NBS on this Standard Reference Material.

Place the Standard Reference Material as the absorber at 25.0° C in the Mössbauer 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 position of the electric quadrupole splitting. The use of digital computation techniques are recommended. Divide the distance between the peaks by two and assign this as the value for chemical shift indicated above. Replace the Standard crystal with the compound whose chemical shift is to be measured as the absorber in the spectrometer. Measure the peak position parameters as a difference between that of the unknown compound and the mid-point between the peaks of the Standard.