National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 90

Ferrophosphorus

This Standard Reference Material (SRM) is ferrophosphorus, an alloy used in manufacturing steel. SRM 90 is intended for use in calibration and the evaluation of chemical and instrumental methods of analysis. A unit of SRM 90 consists of a bottle containing approximately 75 g of powder.

Certified Values: The certified value for phosphorus in SRM 90 is reported in Table 1. The value is reported as a mass fraction [1]. Value assignment categories are based on the definition of terms and modes used at NIST for chemical reference materials [2]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or taken into account. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories using instrumental and classical test methods.

Expiration of Certification: The certification of **SRM 90** is valid indefinitely within the measurement uncertainties specified provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Use"). 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.

Measurements for value assignment of SRM 90 in 1928 were performed by H.A. Bright of the National Bureau of Standards (NBS, now NIST). Analytical determinations for certification of SRM 90 were performed by the following laboratories: Booth, Garrett, and Blair, Philadelphia, PA; Ledoux and Company, New York., NY; Southern Manganese Corporation, Anniston, AL; Carnegie Steel Company, Duquesne, PA; Carnegie Steel Company, Braddock, PA; Electro Metallurgical Company, New York, NY; Electro Metallurgical Company, Niagara Falls, NY. Test methods used by NBS and collaborating laboratories for value assignment are provided in Appendix A.

Statistical consultation for the value assignment of SRM 90 was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Review and evaluation of uncertainty was performed by J.R. Sieber of the NIST Analytical Chemistry Division.

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

Stephen A. Wise, Chief
Analytical Chemistry Division

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See Certificate Revision History on Page 2

Robert L. Watters, Jr., Chief
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INSTRUCTIONS FOR USE

The material for SRM 90 was tested using gravimetric methods and a specimen quantity of 1.5 g. Therefore, a mass of 1.5 g is known to provide results that can be related to the assigned value and its uncertainty. When not in use, the material should be stored in its original container in a cool, dry location.

Table 1. Certified Value for SRM 90

Constituent	Mass Fraction[(a)] (%)
Phosphorus	26.17 ± 0.04

(a) The certified value is a weighted average of the results of analyses performed according to the methods listed in Appendix A. The uncertainty of the certified value is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO Guide [3]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is calculated, at the level of one standard deviation, by combining a between-method variance and a pooled, within-method variance. The coverage factor, $k = 2$, was chosen to approximate a 95 % level of confidence level [4].

REFERENCES

[1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at: http://ts.nist.gov/WeightsAndMeasures/Metric/mpo_pubs.cfm (accessed Mar 2010).

[2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136, U.S. Government Printing Office: Washington, DC (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/upload/SP260-136.PDF (accessed Mar 2010).

[3] JCGM 100:2008; *Evaluation of Measurement Data – Guide to the Expression of in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Mar 2010); 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://physics.nist.gov/Pubs/ (accessed Mar 2010).

[4] Rukhin, A.L.; Vangel, M.G.; *Estimation of a Common Mean and Weighted Mean Statistics*; J. Am. Stat Assoc., Vol. 93, No. 441, pp. 303−308 (1998).

[5] Lundell, G.E.F., Hoffman, J.I.; *The Analysis of Phosphate Rock*; J. Assoc. Off. Agric. Chem., Vol. 8, No. 2, pp. 184−206 (1924).

Certificate Revision History: 30 March 2010 (This revision updates the certificate to current NIST standards including assessment of uncertainty based on re-evaluation of the original results); 01 October 1928 (Original certification date)

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

APPENDIX A

Test Methods Employed at NBS and the Collaborating Laboratories

1. Fusion with Na_2O_2 followed by precipitation with molybdate, then double precipitation with $MgCl_2$ and weighing as $Mg_2P_2O_7$ [5].

2. Dissolution in H_2SO_4 (specific gravity = 1.84), followed by method 1.

3. Fusion with Na_2O_2 followed by precipitation and weighing as $(NH_4)_3PMo_{12}O_{40}$.

4. Dissolution in H_2SO_4 (specific gravity = 1.84), followed by double precipitation with $MgCl_2$ in the presence of citric acid and weighing as $Mg_2P_2O_7$.

5. Dissolution in $HClO_4$, followed by method 1.

6. Dissolution in HNO_3: HF, followed by method 1.