National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 90

Ferrophosphorus
(powder form)

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 Mass Fraction 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.

Table 1. Certified Value for SRM 90

Constituent	Mass Fraction (%)		
Phosphorus	26.17	±	0.04

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 JCGM and NIST Guides [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 [4].

Expiration of Certification: The certification of **SRM 90** is valid indefinitely, within the uncertainty specified, provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Use"). Periodic recertification of this SRM is not required. 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 or register online) will facilitate notification.

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

Review and evaluation of uncertainty was performed by J.R. Sieber of the NIST Chemical Sciences Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 31 May 2019
Certificate Revision History on Page 3

Steven J. Choquette, Director
Office of Reference Materials

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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.

PREPARATION AND ANALYSIS[1]

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.

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

[1] Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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: https://www.nist.gov/pml/pubs/sp811/index.cfm (accessed May 2019).

[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 https://www.nist.gov/srm/upload/SP260-136.PDF (accessed May 2019).

[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 https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2019); 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 https://www.nist.gov/pml/pubs/tn1297/index.cfm (accessed May 2019).

[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: 31 May 2019 (Title update; editorial changes); 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) 948-3730; e-mail srminfo@nist.gov; or via the Internet https://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.