NIST NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
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Standard Reference Material® 2910b
Hydroxyapatite
CERTIFICATE OF ANALYSIS

Purpose: The certified values delivered by this Standard Reference Material (SRM) are intended for evaluating the physical and chemical properties of apatites with biological, geological or synthetic origins.

Description: A unit of SRM 2910b consists of one bottle containing approximately 2 g of hydroxyapatite powder. The calcium to phosphorus (Ca/P) molar ratio for SRM 2910b is consistent with the theoretical Ca/P molar ratio of 1.67 for hydroxyapatite and a compositional formula of $Ca_{10}(PO_4)_6(OH)_2$.

Certified Values: Certified values for constituents of SRM 2910b are reported in Table 1 as mass fractions (expressed as a percent) and Table 2 as Ca/P molar ratios on an as-received basis. 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 [1]. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST.

The measurand is the total mass fraction for each constituent listed in Table 1. The certified values are metrologically traceable to the International System of Units (SI) derived unit for mass fraction, expressed as percent. The calcium and phosphorous content were determined by X-ray fluorescence (XRF) and inductively coupled plasma optical emission spectroscopy (ICP-OES). Twenty four measurements were performed by XRF on twelve units of SRM 2910b and twelve measurements were performed by ICP-OES on six units of SRM 2910b. Each certified value is the estimate of the mean of a random effects model fitted to the data from the two methods. The estimates and associated uncertainties are calculated using the Bayesian inference paradigm [2]. The uncertainty listed with each certified value is an interval calculated in a consistent manner with the ISO/JCGM Guides [3,4], and it expresses contributions from all recognized sources of uncertainty, including differences among analytical methods, differences among bottles, dispersion of values resulting from sample preparation and replicated measurement, preparation and measurement of calibrants, analytical calibration functions, assay of primary materials, and balance calibration. The nominal coverage for each interval is 95 %. The measurand is the certified Ca/P molar ratio presented in Table 2 and is derived from the determinations of calcium and phosphorus. The certified values are metrologically traceable to the SI derived unit for mass, expressed as the ratio of the mass fractions.

Additional Information: Values of potential interest are provided in Appendix A. Additional information is provided in Appendix B.

Period of Validity: The certified values delivered by **SRM 2910b** are valid within the measurement uncertainty specified until **28 July 2036**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm). Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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Table 1. Certified Mass Fraction Values

Constituent	Mass Fraction (%)	95 % Coverage Interval
Calcium (Ca)	39.24	38.60 to 39.75
Phosphorus (P)	18.09	17.75 to 18.39

Table 2. Certified Ca/P Molar Ratio

Constituent	Molar Ratio	95 % Coverage Interval
Ca/P	1.676	1.639 to 1.714

Safety: Consult the Safety Data Sheet (SDS) for hazard information.

Storage: The material should be kept in its original bottle, tightly closed, and stored in a desiccator.

Use: A minimum sample mass of 500 mg of material is necessary for values in this certificate to be valid within stated uncertainties. The contents of the bottle should be shaken well before each use, the bottle closed tightly immediately after use and stored as described above.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Oct 2026).

[2] Gelman, A.; Carlin, J.B.; Stern, H.S.; Dunson, D.B.; Vehtari, A.; Rubin, D.B.; *Bayesian Data Analysis*; 3rd ed., CRC Press (2014).

[3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the expression of uncertainty in measurement*; Joint Committee for Guides in Metrology (JCGM) (2008) ; available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Oct 2026); see also 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/special-publication-811 (accessed Oct 2026).

[4] JCGM 101:2008; *Evaluation of Measurement Data — Supplement 1 to the "Guide to the Expression of Uncertainty in Measurement" — Propagation of Distributions using a Monte Carlo Method*; Joint Committee for Guides in Metrology (JCGM) (2008) available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Oct 2026).

[5] McDowell, H.; Gregory, T.M.; Brown, W.E.; *Solubility of the $Ca_5(PO_4)_3OH$ in the System $Ca(OH)_2$•H_3PO_4•H_2O at 5, 15, 25 and 37 °C*; J. Res. Natl. Bur. Stand., Vol. 81A, pp. 273–281 (1977); available at http://nvlpubs.nist.gov/nistpubs/jres/081/2/V81.N02.A05.pdf (accessed Oct 2026).

[6] Dong, C.; *PowderX: Windows-95-based program for powder X-ray diffraction data processing*; J. Appl. Cryst., Vol. 32, p. 838 (1999).

[7] Huffman, Jr., E.W.D.; *Performance of a New Automatic Carbon Dioxide Coulometer*; Microchemical Journal, Vol. 22; pp. 567–573 (1977).

[8] Boniface, H.J.; Jenkins, R.H.; *The Determination of Carbon in Steel by Coulometric Titration in Partially Aqueous Medium*; Analyst, Vol. 96, pp. 37–46 (1971).

Certificate Revision History: 22 October 2026 (Change of period of validity; format update; editorial changes); **16 December 2015** (Corrected methods for antimony and bismuth; editorial changes); **04 September 2015** (Original certificate date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Values of Potential Interest: X-Ray Diffraction (XRD) measurements and carbonate content analysis were carried out to demonstrate that SRM 2910b is consistent with the mineral hydroxyapatite. Values of potential interest for XRD peak positions and lattice parameters are reported in Table A1 and Table A2, respectively, and the carbonate content as measured is below the limit of quantitation (0.40 %). A value of potential interest to users is considered to be a value that may be of interest to the SRM user, but for which insufficient information is available to assess the uncertainty associated with the value [1]. Values of potential interest cannot be used to establish metrological traceability.

Table A1. Peak Positions using CuKα Radiation, λ = 0.154 059 3 nm

hkℓ	2θ	*d* (nm)	I (relative)
100	10.83	0.816 0	10
101	16.83	0.526 4	3
110	18.82	0.471 1	2
200	21.77	0.408 0	6
111	22.85	0.388 9	5
201	25.35	0.351 1	2
002	25.85	0.344 4	34
102	28.10	0.317 3	8
210	28.93	0.308 4	15
211	31.76	0.281 5	100
112	32.17	0.278 0	48
300	32.90	0.272 0	60
202	34.04	0.263 2	21
301	35.45	0.253 0	4
212	39.18	0.229 8	6
310	39.80	0.226 3	22
221	40.44	0.222 9	2
311	41.99	0.215 0	6
302	42.31	0.213 5	1
113	43.83	0.206 4	5
400	44.37	0.204 0	1
203	45.28	0.200 1	4
222	46.68	0.194 4	28
312	48.07	0.189 1	20
320	48.59	0.187 2	4
213	49.45	0.184 2	30
321	50.48	0.180 7	15
410	51.26	0.178 1	11
402	52.06	0.175 5	12
004	53.14	0.172 2	13
104	54.41	0.168 5	1
322	55.85	0.164 5	6
313	57.10	0.161 2	4
501	58.03	0.158 8	2
412	58.28	0.158 2	1
330	58.75	0.157 0	1
420	59.93	0.154 2	4

Table A2. Lattice Parameters

	Lattice Parameter (nm)
ab	0.942 27
c	0.688 86

X-Ray Diffraction: The diffraction Instrumental analysis was performed on a Rigaku SmartLab. The XRD was equipped with a Cu rotating anode system, operated at 45 kV, 200 mA, producing CuKα radiation with diffracted-beam graphite monochromator. The instrument was configured to use a Bragg-Brentano geometry. A scintillation detector was used. The incident slit was set to 1 mm with 1 mm and 0.15 mm receiving slits of RS1 and RS2, respectively. Data were collected with a step width of 0.01° 2θ and a count time of 4 seconds per step. The data analysis was performed using PowderX, a freely available software package [6].

Carbonate Analysis: Carbonate content in candidate SRM 2910b was tested by Galbraith Laboratories (Knoxville, TN) using a Carbonate as Carbon quantitative method [7,8]. The analytical method liberates carbon dioxide from the sample through acidification and heat. Carbon dioxide-free nitrogen sweeps the evolved carbon dioxide through a scrubber and into an absorption cell, which automatically titrates coulometrically. This method determines carbon dioxide, carbonic acid, bicarbonate ion, and carbonate ion in a variety of materials, including aqueous, non-aqueous liquids and solids. The values are provided as mass fractions for carbon and can be converted to mass fractions carbonate by multiplying by ratio of the molecular mass of carbonate to the atomic mass of carbon (4.996).

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APPENDIX B

Preparation of Materials: The hydroxyapatite powder was produced at NIST in collaboration with the ADAF Volpe Research Center. The powder was synthesized by the solution reaction of calcium hydroxide and phosphoric acid [5]. In brief, approximately 2.5 L of distilled water was boiled for 60 minutes in a 5 L three-neck round bottom flask equipped with an overhead stirrer, reflux condenser coupled to a CO_2-absorbing NaOH trap, and ports for introducing titrant and nitrogen gas (purified by passing through 0.01 M KOH solution). Approximately 120 g of calcium oxide (prepared from calcium carbonate by heating at 1100 °C for 24 h) was added to the water and mixed vigorously. Phosphoric acid (0.5 $mol \cdot L^{-1}$) was then added to the calcium oxide/calcium hydroxide slurry at a rate of 0.6 mL/min and to a final calculated Ca/P molar ratio of 1.67 (approximately 2.5 L of phosphoric acid). The mixture was refluxed for 2 days. After refluxing, the solution was cooled and the precipitated solid phase settled, the supernatant decanted, and an equal volume of boiled distilled water was added and further refluxed for another 2 days. This washing and boiling procedure was repeated four times until the pH of the supernatant was >6 which was the condition where residue traces of dicalcium phosphate were converted into hydroxyapatite. Six individual batches were synthesized, totaling approximately 1 kg of hydroxyapatite. The batches were combined in 3 L of deionized carbonate free water under boiling conditions and nitrogen atmosphere and mixed with an overhead stirrer for 5 days. The final precipitate was dried at 105 °C under vacuum for 5 days.

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