DEPARTMENT OF COMMERCE · UNITED STATES OF AMERICA

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2910

Calcium Hydroxyapatite

This Standard Reference Material (SRM) is intended primarily for use in evaluating the physical and chemical properties of calcium apatites of biological, geological, and synthetic origins. The composition of SRM 2910 slightly deviates from the theoretical compositional formula for calcium hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$. The compositional formula of SRM 2910 obtained from chemical analyses and charge balance, normalized to 6 phosphate groups (PO_4 + HPO_4) per formula unit is given in Comment 2, page 3. SRM 2910 is a high-purity powder form of calcium hydroxyapatite with crystal sizes of about 0.1 μm to 0.5 μm. One unit of SRM 2910 consists of 2 g of material.

Certified Values: This SRM is certified for calcium and phosphorus contents, Ca/P molar ratio, specific surface area, and solubility product. Calcium and phosphorus were determined by atomic absorption spectrometry and colorimetry, respectively and are given on page 2. The specific surface area was determined by the BET Method. The solubiity product was calculated with a computer program from measured equilibrium calcium and phosphate concentrations and pH values. The set of thermodynamic equilibrium constants used in the computation is given in Table 1. Details of the methods used for assignment of the certified values are cited in references given under the section on *Certified Values* on page 2.

Material heterogeneity was assessed for each of the certified analytes or physical parameters by means of analysis of variance (ANOVA) [1]. Uncertainties were assessed by use of the CIPM approach [2]. The certified and reference values are expressed as mean value ± expanded uncertainty (*U*) [3].

Reference Values: Additional measurements and data were obtained to further characterize the material and are provided as reference values, which are listed on page 3. Hydrogenphosphate content, water content, and carbonate content were determined using methods and procedures described on page 3. Also, line-profile breadths, relative intensities, lattice parameters, and contents of crystallographically disordered material were determined from X-ray powder diffraction data. Infrared and Raman spectra were also recorded.

Note: The certified and reference values reported as mass fractions are expressed in percent [4].

Expiration of Certification: The certification of SRM 2910 is valid, within the measurement uncertainties specified, until **30 March 2013**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see *Instructions for Use*). This certification is nullified if the SRM is contaminated or modified.

Maintenance of SRM Certification: NIST will monitor representative samples from this SRM lot over the period of its certification. If substantive changes occur that affect the certification before the expiration of certification, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Statistical analysis was provided by E.S. Lagergren of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.C. Colbert and B.S. MacDonald of the NIST Measurement Services Division.

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See Certificate Revision History on Last Page

The SRM was prepared and analyzed by M. Markovic and M.S. Tung of the Paffenbarger Research Center, American Dental Association Health Foundation, and B.O. Fowler of the National Institute of Dental Research, National Institutes of Health, all assigned to the NIST Polymers Division.

The Rietveld analyses and the associated X-ray diffraction measurements, were performed by J.P. Cline of the NIST Ceramics Division.

NOTICE AND WARNING TO USERS

Source of Material: The calcium hydroxyapatite was synthesized at NIST by solution reaction of calcium oxide and phosphoric acid in accordance with the procedure of McDowell et al. [5]. The calcium hydroxyapatite precipitate, approximately 900 g, was dried at 105 °C in air for 1 day. The resulting calcium hydroxyapatite [6,7] is composed of about 75 % (by mass) of the hexagonal form (space groups $P6_3/m$ or specifically $P6_3$ [8]) and of about 25 % (by mass) of the monoclinic form (space group $P2_1/b$ [9]).

Handling and Storage: This SRM should be stored in its original container with the cap tightly closed under normal laboratory conditions of temperature and humidity.

INSTRUCTIONS FOR USE

SRM 2910 can be used without any pretreatment when stored according to the above *Handling and Storage* procedures.

CERTIFIED VALUES

Calcium Content: Calcium was determined by atomic absorption spectroscopy [10]. The certified mass fraction based on twenty replicate measurements is

$$39.15\ \% \pm 0.10\ \%$$

Phosphorus Content: Phosphorus was determined colorimetrically as the phosphovanadomolybdate complex [10,11]. The certified mass fraction based on twenty replicate measurements is

$$18.18\ \% \pm 0.04\ \%$$

Ca/P Molar Ratio: The certified calcium to phosphorus molar ratio based on the determinations of calcium and phosphorus is

$$1.664 \pm 0.005$$

Specific Surface Area: The specific surface area was determined by the triple-point method of Brunauer, Emmett, and Teller (BET Method) [12] with nitrogen as the adsorbate gas and helium as the inert nonadsorbable carrier [6]. The certified specific surface area (area divided by mass) based on twelve replicate measurements is

$$18.3\ \mathrm{m^2/g} \pm 0.3\ \mathrm{m^2/g}$$

Solubility Product: Twelve suspensions containing about 400 mg of the calcium hydroxyapatite in 20 $\mathrm{cm^3}$ of 5.026 $\mathrm{mmol/dm^3}$ phosphoric acid were equilibrated at 37.0 °C ± 0.1 °C for 60 days in a continuously shaken water bath. The mean values of pH and total soluble calcium and phosphate concentrations determined in the supernatants were: pH = 4.685 ± 0.004; c(Ca) = 3.562 $\mathrm{mmol/dm^3}$ ± 0.038; $\mathrm{mmol/dm^3}$; and c(PO_4) = 7.160 $\mathrm{mmol/dm^3}$ ± 0.106 $\mathrm{mmol/dm^3}$ [3]. The thermodynamic solubility product, K_{sp}, of HA is defined as

$$K_{sp}(\mathrm{HA}) = a^5(\mathrm{Ca}^{2+})\, a^3(\mathrm{PO_4}^{3-})\, a(\mathrm{OH}^-)$$

where a is the activity of the ion. The K_{sp} was calculated with a computer program [13] from measured equilibrium calcium and phosphate concentrations and pH values in each sample as input data. The set of thermodynamic equilibrium constants (at ionic strength (I) = 0, 37 °C) used in the computation is given in Table 1. The certified value for the thermodynamic solubility product based on twelve replicate measurements at 37 °C is

$$K_{sp}(\mathrm{HA}) = (2.03 \pm 0.71) \times 10^{-59}$$

REFERENCE VALUES

Hydrogenphosphate Content: The Gee and Deitz method [14] with some modifications [6] was used for determination of the hydrogenphosphate (HPO_4^{2-}) content. The mass fraction of HPO_4^{2-} based on four replicate measurements is

$$0.592\ \% \pm 0.030\ \%$$

Carbonate Content: Carbonate (CO_3^{2-}) content was determined by heating approximately 5 g of the calcium hydroxyapatite sample at 1200 °C to liberate CO_2, which was collected in an absorption cell containing a lithium hydroxide solution. Carbonates in the absorption cell were determined by automatic coulometric titration. Analyses were done at Galbraith Laboratories, Knoxville, TN. The mass fraction of CO_3^{2-} based on twelve replicate measurements is

$$0.032\ \% \pm 0.002\ \%$$

Water Content: The water content was determined from the total mass loss by three different thermal procedures [3,6,7] designated below as (a), (b), and (c). The water content in calcium hydroxyapatite is the difference between the total mass loss and mass loss due to two thermal reactions: (1) hydrogenphosphate pyrolysis into pyrophosphate and (2) thermal reaction of calcium pyrophosphate with calcium hydroxyapatite to form β-tricalcium phosphate. The calculated mass loss from these two thermal reactions is 0.111 % ± 0.006 %; all values for water contents listed below are reduced by 0.111 %. The mass loss (less than 0.02 %) due to thermal decomposition of CO_3^{2-} to CO_2 was neglected.

(a) Thermogravimetry in Nitrogen: Five samples were heated continuously from 30 °C to 850 °C at a rate of 10 °C/min in a nitrogen atmosphere. The mass fraction of water based on five measurements is

$$1.59\ \% \pm 0.05\ \%$$

(b) **Thermogravimetry in Air:** Six samples were heated in air at 850 °C for 16 h to 19 h, then cooled in a desiccator and weighed in the laboratory atmosphere (about 50 % relative humidity) at ambient temperature. The mass fraction of water based on six measurements is

$$1.43\ \% \pm 0.03\ \%$$

(c) Thermogravimetry in Steam: Three samples pressed into pellets were heated in a steam atmosphere (water vapor pressure of 100 kPa) at 1000 °C for 10 h and then cooled and weighed as in procedure (b) above. The mass fraction of water based on three measurements is

$$1.56\ \% \pm 0.03\ \%$$

Comment 1: The water contents in samples weighed at laboratory conditions, procedures (b) and (c), were lower than in the samples heated and weighed in the nitrogen atmosphere, procedure (a), because of fast readsorption of surface water during cooling and weighing in the laboratory atmosphere at ambient temperature; therefore, the water content of 1.59 % determined by procedure (a) is expected to be the most reliable.

Comment 2: The compositional formula of SRM 2910 [6] obtained from chemical analyses and charge balance, normalized to 6 phosphate groups (PO_4+HPO_4) per formula unit is

$$Ca_{9.985}(HPO_4)_{0.063}(PO_4)_{5.937}(OH)_{2.026}(CO_3)_{0.005}(SiO_3)_{0.005} + 0.902\ H_2O$$

Contents of SiO_3^{2-} and trace elements are given in reference 6 along with details about the water component that is primarily adsorbed.

Comment 3: In order to obtain an anhydrous calcium hydroxyapatite from SRM 2910, this SRM should be heated, procedure (b), at 850 °C ± 10 °C in air at normal relative humidity (about 50 %) for 12 h or longer, then cooled in air and stored in a desiccator. This anhydrous calcium hydroxyapatite will have a monoclinic structure and will contain about 0.3 % (by mass) of β-tricalcium phosphate, $\beta\text{-Ca}_3(\text{PO}_4)_2$ [3,6,7]. If procedure (c) is used, the anhydrous monoclinic hydroxyapatite will contain 1 % (by mass) of β-tricalcium phosphate [3,6,7].

X-ray Powder Diffraction: The X-ray powder diffraction (XRD) patterns for the determination of the profile breadth and relative intensity data were collected from six samples on a manual diffractometer using $\text{CuK}\alpha$ radiation. The goniometer had a radius of 185 mm and was equipped with a graphite post monochromator and scintillation detector. The divergence slit was 1° and the receiving slit was 0.3 mm. The scan range for relative intensities was from 3° to 60° 2θ; scan rate was 0.5° 2θ/min. The scan range for profile breadth of reflections (310) and (002) was from 38.5° to 40.5° 2θ and 25. 0° to 26.5° 2θ, respectively; scan rate was 0.03125° 2θ/min.

A X-ray diffraction pattern of the powder is shown in Figure 1. The intensities, reported as relative intensity (I_{rel}) in Table 2, were determined as the background subtracted diffraction line heights measured manually from plotted data relative to the most intense line as 100.

For determination of sample induced profile broadening, reported as Full Width Half Maximum (FWHM), the angular width at half the background subtracted height of at least four scans of the (310) and (002) reflections for each of six samples were obtained by XRD. The scan range for profile breadth of reflections (310) and (002) was from 38.5° to 40.5° 2θ and 25.0° to 26.5° 2θ, respectively; scan rate was 0.031250 2θ/min. The angular width, B (° 2θ), was corrected for the instrument profile function, b (° 2θ), with use of Warren's method [15]; the corrected value of the angular width, β, was calculated from the equation $\beta = (B^2 - b^2)^{1/2}$. A stoichiometric, highly crystalline hydroxyapatite prepared by solid-state thermal reaction [16] was used as a reference substance in the determination of the value of b. The 1/β value for the (310) line reflects the size/strain in the plane perpendicular to the c-axis and 1/β for the (002) line denotes the size/strain along the c-axis. The values for 1/β are

$$1/\beta(310) = 5.5\ (°\ 2\theta)^{-1} \pm 0.2\ (°\ 2\theta)^{-1}$$
$$1/\beta(002) = 8.4\ (°\ 2\theta)^{-1} \pm 0.2\ (°\ 2\theta)^{-1}$$

Rietveld analyses of additional X-ray diffraction data were used for determination of lattice parameters, the Reference Intensity Ratio or I/I_c [17,18], and to determine the quantity of the crystallographically disordered material. This material results from relaxation and dissatisfied bonds at grain boundaries and the particles' surface. It will not diffract in a manner analogous to the bulk and can be considered as an amorphous phase or as a disordered fraction. Disordered fraction is used here rather than amorphous phase to avoid implication of the presence of the well known completely amorphous form of calcium phosphate [19], and because amorphous calcium phosphate should not be present in this SRM 2910 calcium hydroxyapatite since the preparative conditions used preclude its formation [20]. These data were collected on two samples removed from each of four randomly selected bottles. SRM 676, an alumina powder SRM certified for quantitative analysis [21], served as the internal standard. Specimens were prepared at the mass ratio of 65 % hydroxyapatite and 35 % SRM 676 alumina. These XRD data were collected on a diffractometer equipped with a focusing germanium incident beam monochromator, sample spinner and a scanning position sensitive detector. Copper $\text{K}\alpha_1$ radiation, $\lambda = 0.15405945$ nm [22], was used. The scan range was from 17.5° to 152° 2θ; scan times were approximately 6.5 h per sample. General Structure Analysis System (GSAS) [23] was used for a Rietveld refinement of the two phases. Refined parameters included the following: scale factors, crystallite size and strain induced broadening terms of the pseudo-Voigt profile function [24], lattice parameters, sample shift and transparency terms, atomic thermal and position parameters for the silicon, aluminum and oxygen atoms, Lorentz polarization factor and six terms of a background function describing the effects of thermal diffuse scattering and air scattering (incoherent) of the incident beam. Gaussian U, V, and W terms [25] were presumed to be instrumental in origin and fixed at those obtained from a refinement of SRM 660, the lanthanum hexaboride line profile standard [26]. The values for the a and c lattice parameters are

$$a = 0.942253 \text{ nm} \pm 0.000013 \text{ nm}$$
$$c = 0.688501 \text{ nm} \pm 0.000009 \text{ nm}$$

Peak positions are reported in Table 3. The I/I$_c$ was computed from the observed structure factors of the Rietveld analyses. The reflections used were the (211) from the calcium hydroxyapatite of SRM 2910 and the (113) from the alumina of SRM 676. The data were corrected for the amorphous content of the SRM 676 [27]. The value of the I/I$_c$ is

$$1.258 \pm 0.078$$

Crystallographically disordered material fraction was determined from the discrepancy between the ratio of hydroxyapatite to alumina determined from the Rietveld refinements, which is based only on the crystalline material, as opposed to the ratio at which they were prepared, which included the disordered fraction. The crystallographically disordered material fraction of 9.1 % ± 4.1 %, corrected for the water content of 1.59 % ± 0.05 %, is

$$7.5\ \% \pm 4.1\ \%$$

Infrared Spectra: Infrared spectra of the powder suspended in potassium bromide pellets were recorded from 4000 cm^{-1} to 300 cm^{-1} with a spectrophotometer purged with dry air free of carbon dioxide. The instrument spectral slit width was ≈ 6 cm^{-1} above 2000 cm^{-1} and (3 to 5) cm^{-1} below 2000 cm^{-1}. Infrared spectra are shown in Figure 2.

Raman Spectra: Raman spectra of the powder pressed into a pellet were recorded from 4000 cm^{-1} to 50 cm^{-1} with a spectrometer using 488.0 nm wavelength excitation from an argon ion laser and an instrument spectral slit width of 3.5 cm^{-1}. The exciting radiation at a power of 320 mW was focused on the sample pellet and scattered radiation was detected by a photomultiplier cooled to - 25 °C. Raman spectra are shown in Figure 3.

The vibrational origins of all infrared and Raman spectral bands shown in Figures 2 and 3 are given in reference 6. Bands of impurity ions present along with the calcium hydroxyapatite bands in both spectra are identified as follows: In Figure 2, the broad infrared band from about 3700 cm^{-1} to 2500 cm^{-1} and the band at 1625 cm^{-1} arise from H_2O, the 1459 cm^{-1} and 1418 cm^{-1} bands arise from CO_3^{2-}, and the 870 cm^{-1} band arises from HPO_4^{2-}; in Figure 3, the Raman bands at 1005 cm^{-1} and 880 cm^{-1} arise from HPO_4^{2-}.

Table 1. Thermodynamic Equilibrium Constants Used in Computation

No.	Equilibrium	K (I = 0, 37 °C)
1	$H_3PO_4 \leftrightarrows H^+ + H_2PO_4^-$	6.22×10^{-3}
2	$H_2PO_4^- \leftrightarrows H^+ + HPO_4^{2-}$	6.58×10^{-8}
3	$HPO_4^{2-} \leftrightarrows H^+ + PO_4^{3-}$	6.84×10^{-13}
4	$H_2O \leftrightarrows H^+ + OH^-$	2.40×10^{-14}
5	$Ca^{2+} + H_2PO_4^- \leftrightarrows CaH_2PO_4^+$	6.99
6	$Ca^{2+} + HPO_4^{2-} \leftrightarrows CaHPO_4^0$	355
7	$Ca^{2+} + PO_4^{3-} \leftrightarrows CaPO_4^-$	2.90×10^6
8	$Ca^{2+} + OH^- \leftrightarrows CaOH^+$	20

Table 2. 2θ-Values and d-Values Calculated from Unit Cell Parameters Determined by Rietveld Method and Relative Intensities (I_{rel}) Observed from the XRD Pattern of SRM 2910

hkl	° 2θ	d (nm)	I_{rel}
100	10.83	0.816	8
101	16.83	0.526	4
110	18.82	0.471	2
200	21.76	0.408	6
111	22.85	0.389	6
201	25.35	0.351	2
002	25.86	0.344	35
102	28.11	0.317	9
210	28.93	0.308	16
211	31.77	0.281	100
112	32.18	0.278	53
300	32.90	0.272	63
202	34.05	0.263	24
301	35.46	0.253	5
212	39.18	0.2297	6
310	39.80	0.2263	23
221	40.44	0.2229	2
311	41.99	0.2150	6
302	42.31	0.2134	1
113	43.84	0.2063	5
400	44.37	0.2040	1
203	45.30	0.2000	5
222	46.69	0.1944	28
312	48.07	0.1891	13
320	48.59	0.1872	4
213	49.46	0.1841	32
321	50.48	0.1806	16
410	51.26	0.1781	11
402	52.07	0.1755	12
004	53.17	0.1721	14
104	54.43	0.1684	1
322	55.86	0.1645	5
313	57.11	0.1611	4
501	58.03	0.1588	2
412	58.29	0.1582	2
330	58.75	0.1570	1
420	59.93	0.1542	5

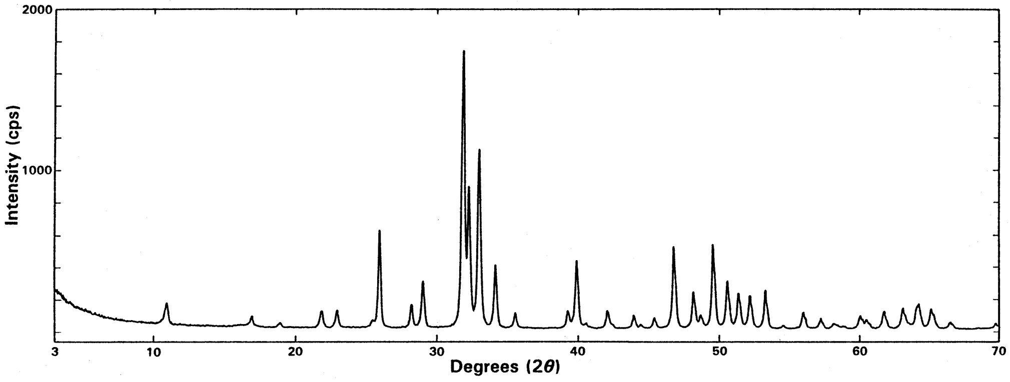

Figure 1. X-ray diffraction pattern of calcium hydroxyapatite SRM 2910 in the 2θ range from 3° to 70°.

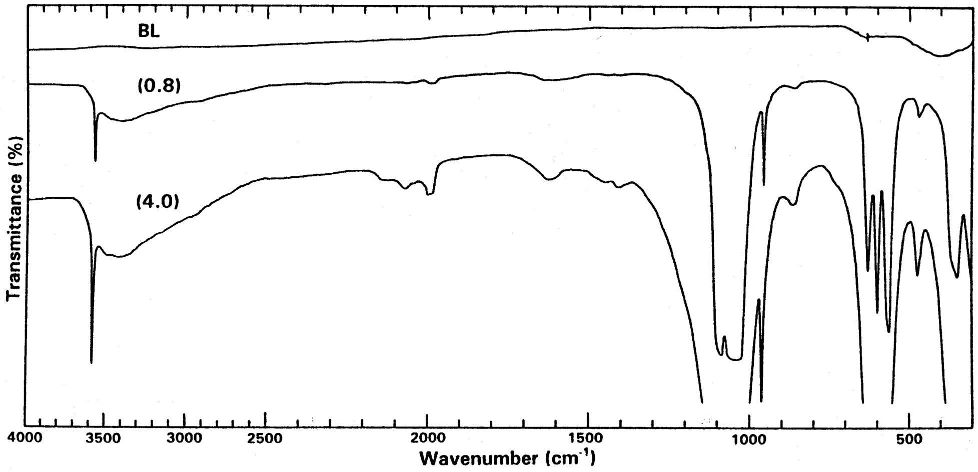

Figure 2. Infrared transmittance spectra of calcium hydroxyapatite SRM 2910 samples in KBr pellets containing 0.8 mg and 4.0 mg of sample *versus* blank KBr pellet. BL is the KBr baseline.

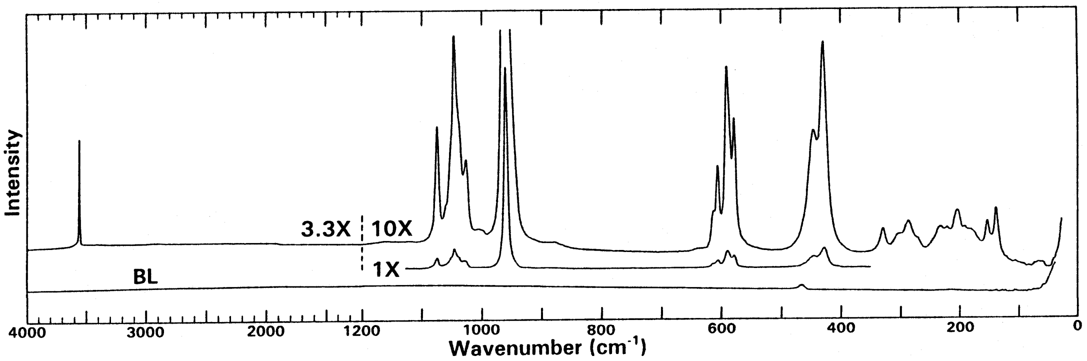

Figure 3. Raman spectra of calcium hydroxyapatite SRM 2910 sample recorded at relative intensities of 1 and 10 in the range below 1200 cm^{-1} and at relative intensity of 3.3 above 1200 cm^{-1}. BL is the instrument baseline from laser reflection off a platinum surface.

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Certificate Revision History: 01 July 2003 (This revision reflects a change in unit size and an extension in the expiration date); 03 November 1997 (Original certificate 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) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.