U. S. Department of Commerce
Peter G. Peterson
Secretary

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 120b

Phosphate Rock

(Florida)

This standard is a finely powdered material intended for use in checking chemical methods of analysis and in calibration with optical emission and x-ray spectrometric methods of analysis.

See ADDENDUM* (Over) for Uranium (Radium and Thorium)
(All results are based on samples dried for 1 hour at 105 °C.)

Percent by Weight

ANALYST*	P_2O_5	CaO	SiO_2	F	Soluble Fe_2O_3	Soluble Al_2O_3	MgO	Na_2O	MnO	K_2O		TiO_2	CO_2	CdO
1	34.51[a]	49.42[b]	4.70[c]	3.82[d]	1.10[e]	1.09[f,g]	0.29[h]	0.33[f]	0.032[i]	0.12[f,j]	--	0.15[k]	--	0.002[l]
2	34.51[m]	49.35[m]	4.73[n]	3.79[m]	1.10[h]	1.07[h]	.28[h]	.36[h]	.031[h]	.12[j]	0.09[o]	--	2.76[p]	.002[h]
3	34.66[n]	49.38[m]	4.67[q]	3.83	1.09[h]	1.07[h]	.30	.36[h]	.032[h]	.12[j]	.098[o]	.15	2.79	.002[h]
4	34.67[r]	49.47[m]	4.69[q]	3.81[s]	1.13[h]	1.04[h]	.28[h]	.35[h]	.032[h]	--	.087[o]	.15[k]	2.78[p]	.003[h]
5	34.57	49.32[m]	4.63[q]	3.86	1.06[h]	1.05[h]	.25[h]	.34[h]	--	--	.085[o]	--	2.83	--
6	34.48[m]	49.45[m]	--	3.92[s]	1.14[m]	1.07[t]	--	--	--	--	--	--	--	--
Average	34.57	49.40	4.68	3.84	1.10	1.06	0.28	0.35	0.032	0.12	0.090	0.15	2.79	0.002

[a] Phosphorus precipitated with magnesia mixture, ignited and weighed as $Mg_2P_2O_7$.

[b] Calcium precipitated as oxalate, ignited and weighed as CaO.

[c] Sample fused with Na_2CO_3, silica precipitated with ZnO and dehydrated with HCl. Traces of SiO_2 recovered by H_2SO_4 dehydration.

[d] Fluorine distilled into NaOH solution and precipitated as lead chlorofluoride. Chloride is precipitated with excess $AgNO_3$ and excess $AgNO_3$ is titrated with standard KCNS solution.

[e] $SnCl_2$ reduction – $K_2Cr_2O_7$ titration.

[f] Flame emission spectrometry with repetitive optical scanning.

[g] A value of 1.13 percent was obtained for total Al_2O_3 by gravimetry.

[h] Atomic absorption spectrometry.

[i] KIO_4 spectrophotometric method.

[j] Sample digested with mixed acids for 1 hour. Determination completed by atomic absorption spectrometry.

[k] H_2O_2 spectrophotometric method.

[l] Polarographic method.

[m] Volumetric method.

[n] Gravimetric method.

[o] Sample digested with dilute HCl or aqua regia for 15 minutes. Determination completed by atomic absorption spectrometry.

[p] CO_2 absorbed and weighed.

[q] Dehydration with $HClO_4$ in presence of boric acid.

[r] Molybdovanadophosphate spectrophotometric method.

[s] Distillation – titration with standard thorium nitrate solution.

[t] Aluminum precipitated with 8 hydroxyquinoline and weighed.

Washington, D.C. 20234
July 31, 1972
ADDENDUM* (Over)
July 31, 1979

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of O. Menis and J. I. Shultz.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. E. Michaelis and C. L. Stanley.

PREPARATION, TESTING, AND ANALYSIS: The material for this standard was prepared by the American Cyanamid Company. Eighty five percent of the lot was made to pass 200 mesh sieve and some blending was done at the plant. Final sieving and blending operations were accomplished at NBS.

Homogeneity testing was performed by S. D. Rasberry, C. E. Fiori, and J. McKay with x-ray fluorescence analysis. Calcium and phosphorus determinations were made on 14 samples representative of the top and the bottom of seven containers. The size of the samples taken for analysis was approximately 35 mg. The maximum variations in concentration among samples were within 0.09 percent for CaO and 0.12 percent for P_2O_5.

The laboratories and analysts cooperating in the analytical program for certification were:

1. R. K. Bell, E. R. Deardorff, E. J. Maienthal, T. C. Rains, T. A. Rush, and S. A. Wicks, Analytical Chemistry Division, Institute for Materials Research, National Bureau of Standards.
2. J. Padar, Agrico Chemical Co., Division of Continental Oil Company, Pierce, Florida.
3. D. B. Underhill, Borden Chemical Co., Plant City, Florida.
4. C. C. Thornton, Thornton Laboratories, Inc., Tampa, Florida.
5. W. W. Harwood, R. M. Lynch and H. N. Gomez, International Minerals and Chemical Corp., Bartow, Florida.
6. J. A. Sielski, American Cyanamid Co., Brewster Plant, Bradley, Florida.

*ADDENDUM

Uranium has been determined at NBS by thermal ionization mass spectrometry, E. L. Garner and L. A. Machlan, and the following certification is made:

	Value, $\mu g/g$	Estimated Uncertainty[a]
Uranium	128.4	$\pm$ 0.5

[a]The estimated uncertainty is based on judgment and represents an evaluation of method imprecision and material variability.

(NOTE: On similar phosphate rock materials, a value of 127 $\mu g/g$ for uranium was reported in Ref. 1; additionally, values of 17 $\mu g/g$ for thorium and 43 pCi $^{225}Ra/g$ for radium also were reported.)

Ref. 1 Agr. Food Chem., 16, No. 2, 1968 (p232)