U.S. Department of Commerce
C. William Verity
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 120c

Florida Phosphate Rock

This Standard Reference Material (SRM) is intended for use in validating analytical methods and in calibrating instruments used in the analysis of this type of phosphate rock. SRM 120c consists of ground phosphate rock; approximately 70% passes through a 75 μm (200 mesh) screen.

Table 1 lists the certified total concentrations of constituents.

Table 1. Certified Total Concentrations[1]

Constituent[2]	Concentration, percent by weight[3]	Constituent[2]	Concentration, percent by weight[3]
Al_2O_3	1.30 ± 0.04	Na_2O	0.52 ± 0.02
Fe_2O_3	1.08 ± 0.03	TiO_2	0.103 ± 0.006
K_2O	0.147 ± 0.004	U_3O_8	0.0135 ± 0.0002
MnO	0.027 ± 0.002	V_2O_3	0.016 ± 0.002

1 Based on a minimum sample size of 0.5 gram dried at 105 °C for 2 hours.
2 Methods used are listed under "Analytical Methods for Constituents in Table 1."
3 Except for U_3O_8, the certified total concentration of each constituent is based on the agreement of results by at least two independent analytical methods. At least one method known to yield a total concentration was performed at NBS. U_3O_8 was determined at NBS by isotope dilution mass spectrometry, a method of known accuracy. The uncertainties for all constituents, except K_2O and U_3O_8, are two times the standard deviation (S.D.) of the average. The uncertainties for K_2O and U_3O_8 have been expanded to four times the S.D. of the average concentration. For K_2O this allows for possible systematic errors, for U_3O_8 it allows for possible sample heterogeneity.

Table 2 lists the AFPC method-dependent concentrations of constituents.

Table 2. AFPC Method-Dependent Concentrations[1]

Constituent[2]	Concentration, percent by weight[3]	Constituent[2]	Concentration, percent by weight[3]
Al_2O_3 (soluble)	1.18 ± 0.04	Fe_2O_3 (soluble)	1.02 ± 0.02
CaO	48.02 ± 0.17	K_2O (soluble)	0.110 ± 0.005
CdO	0.0010 ± 0.0002	MgO	0.32 ± 0.01
CO_2	3.27 ± 0.12	P_2O_5	33.34 ± 0.06
F	3.82 ± 0.02	SiO_2	5.5 ± 0.2

1 Based on a minimum sample size of 0.5 gram dried at 105 °C for 2 hours.
2 Analytical methods used are listed under "Analytical Methods for Constituents in Table 2."
3 The method dependent concentrations are the average concentrations calculated from the analyses by six to ten laboratories of the Association of Florida Phosphate Chemists (AFPC) using AFPC methods. A copy of the current AFPC methods manual may be purchased for approximately $10.00 (in 1988) from: Association of Florida Phosphate Chemists, P.O. Box 1645, Bartow, Florida 33830.

Except for CO_2, the uncertainties represent two standard deviations of the averages. The uncertainty for CO_2 has been expanded to four times the S.D. of the average concentration to allow for possible sample heterogeneity

Gaithersburg, MD 20899
February 29, 1988

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Statistical evaluation of the data was performed by R.C. Paule of the National Measurement Laboratory.

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

Analytical Methods for Constituents in Table 1.

Al_2O_3	1. Flame Emission Spectrometry --total dissolution method using HNO_3, HF, $HClO_4$ 2. Instrumental Neutron Activation
Fe_2O_3	1. X-ray Fluorescence Spectrometry 2. Atomic Absorption Spectrometry --heat sample in aqua regia and continue as in AFPC 7B
K_2O	1. Flame Emission Spectrometry --total dissolution method using HNO_3, HF, and $HClO_4$ 2. Instrumental Neutron Activation 3. X-ray Fluorescence Spectrometry
MnO	1. Atomic Absorption Spectrometry --heat sample in 5 parts HCl to 12.5 parts HNO_3 (AFPC 12A) 2. Instrumental Neutron Activation
Na_2O	1. Atomic Absorption Spectrometry --heat sample in 1 + 1 HCl (AFPC 10A) or heat in aqua regia 2. Instrumental Neutron Activation
TiO_2	1. Instrumental Neutron Activation 2. X-ray Fluorescence Spectrometry
U_3O_8	Isotope Dilution Thermal Ionization Mass Spectrometry
V_2O_3	1. DC Plasma Spectrometry -- heat sample in aqua regia 2. Instrumental Neutron Activation

Analytical Methods for Constituents in Table 2.

Al_2O_3 (soluble)	1. Atomic Absorption Spectrometry --heat sample in 1 + 1 HCl (AFPC 8B)
CaO	1. EDTA Volumetric --heat sample in HNO_3 (1 + 1) + HCl (AFPC 16C) 2. Gravimetric Sulfate -- heat sample in HCl (1 + 1), filter, add H_2SO_4 (AFPC 16A)
CdO	1. Atomic Absorption Spectrometry --heat sample in HCl, HNO_3 (AFPC 12A)

CO_2	1. Gasometric -- (AFPC 17B) 2. Gravimetric -- (AFPC 17A)
F	1. Specific Ion Electrode -- (AFPC 18B) 2. Volumetric--(AFPC 18A)
Fe_2O_3 (soluble)	1. Atomic Absorption Spectrometry --heat sample in 1 + 1 HCl (AFPC 7B)
K_2O (soluble)	1. Atomic Absorption Spectrometry --heat sample in 1 + 1 HCl (AFPC 11A)
MgO	1. Atomic Absorption Spectrometry --heat sample in 1 + 1 HCl (AFPC 9A)
P_2O_5	1. Molybdovanadophosphate Spectrophotometry --heat sample in HNO_3 (1 + 1) + HCl (AFPC 3C) 2. Molybdovanadophosphate Spectrophotometry --heat sample in aqua regia (AOAC 2.2021)
SiO_2	1. Atomic Absorption Spectrometry -- HF digestion and H_3BO_3; or 1 HNO_3 + 1 HCl + $HClO_4$ + H_3BO_3 2. Gravimetry -- $HClO_4$ digestion with H_3BO_3 and add HF to SiO_2

ADDITIONAL INFORMATION ON THE COMPOSITION

The constituents listed in Table 3 are not certified, but are included for information only.

Table 3. Non-certified Concentrations of Constituents

Constituent	Concentration, percent by weight	Constituent	Concentration, percent by weight
As_2O_3	(0.0009)	PbO	(0.003)
CoO	(0.001)	S (total)	(0.37)
Cr_2O_3	(0.01)	SO_4 (total)	(1.07)
CuO	(0.0016)	SrO	(0.1)
Eu_2O_3	(0.0005)	ZnO	(0.009)
MoO_3	(0.002)		
NiO	(0.004)		

PREPARATION, TESTING, AND ANALYSIS: The material for SRM 120c was prepared by the American Cyanamid Company. The material was ground and blended. Approximately 70% passed through a 75 μm (200 mesh) screen. Homogeneity testing was performed by P.A. Pella and A. Marlow using x-ray fluorescence spectrometry. In these tests, duplicate 500-mg samples were taken from randomly selected bottles and fused with $Li_2B_4O_7$. Comparison of within-sample variability to between-sample variability indicated no significant differences in the Al, Ca, K, P, Si, and Ti concentrations.

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

- Agrico Chemical Co., South Pierce Chemical Works, Bartow, FL, E.D. Germain.

- Agrico Chemical Co., Agrico Mining Co., Mulberry, FL, D.G. Sebastian.

- American Cyanamid Co., Lonesome Plant, Bradley, FL, C. Ware, J.M. Stergar.

- CF Industries Inc., Central Phosphates, Inc., Plant City, FL, T.A. Edwards.

- Estech Inc., Bartow, FL, W.L. Hall.

- Gardinier Inc., Tampa, FL, M.I. Hoke, A. Egitto, L. LeFever.

- International Minerals and Chemical Corp., IMC Fertilizer Group, Bartow, FL, J.E. Gibson, M.K. Dooley, C.S. MacConnell, L.K. Oliver, F.E. Patrick, W.T. Sherrill, M.C. Talbot, H. Williams.

- International Minerals and Chemicals Corp., New Wales Operations, Mulberry, FL, C.W. Wilks.

- Occidental Chemical Co., Florida Operations, White Springs, FL, J.A. Neil.

- Thornton Laboratories, Inc., Tampa, FL, L.H. Isham.

- National Bureau of Standards, Center for Analytical Chemistry, Gaithersburg, MD, T.A. Butler, J.W. Gramlich, R.M Lindstrom, L.A. Machlan, A. Marlow, P.A. Pella, T.C. Rains, and T.A. Rush.