

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 Concentrations¹

<u>Constituent</u> ²	<u>Concentration,</u> <u>percent by weight</u> ³	<u>Constituent</u> ²	<u>Concentration,</u> <u>percent by weight</u> ³
Al ₂ O ₃	1.30 \pm 0.04	Na ₂ O	0.52 \pm 0.02
Fe ₂ O ₃	1.08 \pm 0.03	TiO ₂	0.103 \pm 0.006
K ₂ O	0.147 \pm 0.004	U ₃ O ₈	0.0135 \pm 0.0002
MnO	0.027 \pm 0.002	V ₂ O ₃	0.016 \pm 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 U₃O₈, 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. U₃O₈ was determined at NBS by isotope dilution mass spectrometry, a method of known accuracy. The uncertainties for all constituents, except K₂O and U₃O₈, are two times the standard deviation (S.D.) of the average. The uncertainties for K₂O and U₃O₈ have been expanded to four times the S.D. of the average concentration. For K₂O this allows for possible systematic errors, for U₃O₈ it allows for possible sample heterogeneity.

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

Table 2. AFPC Method-Dependent Concentrations¹

<u>Constituent</u> ²	<u>Concentration,</u> <u>percent by weight</u> ³	<u>Constituent</u> ²	<u>Concentration,</u> <u>percent by weight</u> ³
Al ₂ O ₃ (soluble)	1.18 \pm 0.04	Fe ₂ O ₃ (soluble)	1.02 \pm 0.02
CaO	48.02 \pm 0.17	K ₂ O (soluble)	0.110 \pm 0.005
CdO	0.0010 \pm 0.0002	MgO	0.32 \pm 0.01
CO ₂	3.27 \pm 0.12	P ₂ O ₅	33.34 \pm 0.06
F	3.82 \pm 0.02	SiO ₂	5.5 \pm 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 CO₂, the uncertainties represent two standard deviations of the averages. The uncertainty for CO₂ has been expanded to four times the S.D. of the average concentration to allow for possible sample heterogeneity.

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 ₂ O ₃	1. Flame Emission Spectrometry --total dissolution method using HNO ₃ , HF, HClO ₄ 2. Instrumental Neutron Activation
Fe ₂ O ₃	1. X-ray Fluorescence Spectrometry 2. Atomic Absorption Spectrometry --heat sample in aqua regia and continue as in AFPC 7B
K ₂ O	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 ₂ O	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 ₃ O ₈	Isotope Dilution Thermal Ionization Mass Spectrometry
V ₂ O ₃	1. DC Plasma Spectrometry -- heat sample in aqua regia 2. Instrumental Neutron Activation

Analytical Methods for Constituents in Table 2.

Al ₂ O 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 ₂ SO 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 ₂ O ₃ (soluble)	1. Atomic Absorption Spectrometry --heat sample in 1 + 1 HCl (AFPC 7B)
K ₂ O (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 ₂ O 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 ₃ BO ₃ ; or 1 HNO ₃ + 1 HCl + HClO ₄ + H ₃ BO ₃ 2. Gravimetry -- HClO ₄ digestion with H ₃ BO 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

<u>Constituent</u>	<u>Concentration, percent by weight</u>	<u>Constituent</u>	<u>Concentration, percent by weight</u>
As ₂ O ₃	(0.0009)	PbO	(0.003)
CoO	(0.001)	S (total)	(0.37)
Cr ₂ O ₃	(0.01)	SO ₄ (total)	(1.07)
CuO	(0.0016)	SrO	(0.1)
Eu ₂ O 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 μ m (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 $\text{Li}_2\text{B}_4\text{O}_7$. 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.