ColdBlock™ Technologies Inc.

APPLICATION NOTE

NIST 694 – Western Phosphate Rock

SUMMARY

The application note summarizes the digestion of NIST 694, a Western Phosphate Rock Certified Reference Material using ColdBlock™ Digestion CB15S Technology.

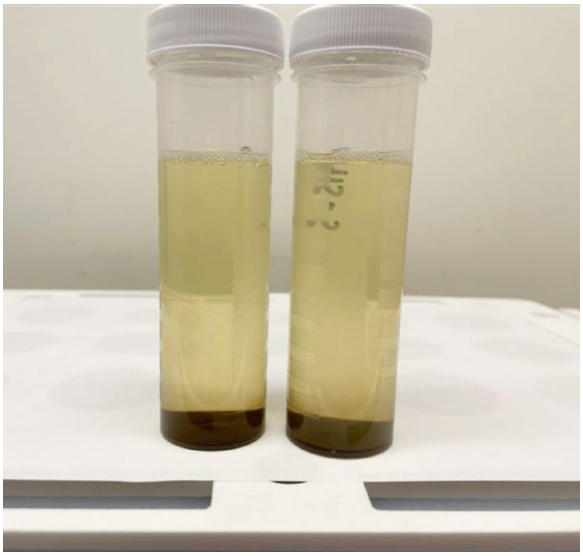
Instrument:	ColdBlock CB15S sample digester technology, chiller, ICP-OES
Published:	September 2022
Digestion Time:	25 Minutes
Acid Used:	Aqua Regia & Tetrafluoroboric Acid
Average ColdBlock Recovery vs. CRM:	■ 107% Calcium ■ 101% Phosphorus ■ 103% Aluminum

METHODOLOGY

1. Chiller temperature was set to 5˚C
2. 0.5g of each sample was weighed and placed into a ColdBlock™ Digestion vessel
3. 20ml Aqua Regia & 3mL Tetrafluoroboric acid (HBF4) was added
4. Sample was digested at 100% power for 5 minutes then power was reduced to 70% and digestion continued for 20 minutes
5. Sample was cooled and bulked to 50mL using 2% HNO3 v/v

DISCUSSION

- After 25 minutes the sample was brown, and a minor amount of solid material remains
- The use of Hydrofluoric acid was investigated and resulted in full recovery of Silicon. Consequently, the formation of insoluble Calcium Fluoride (CaF2) impacted recoveries of Calcium & Aluminum.
- To address the insoluble CaF2, HBF4 was used to fully recover all the Calcium and Aluminum, but negatively impacted Silicon recoveries



NIST 694 is a Western Phosphate Rock -standard reference material by the National Institute of Standards & Technology. It is intended for use in validating analytical methods.

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Results

NIST 694 – Western Phosphate Rock							
Method:	0.5g	20mL Aqua Regia + 3mL HBF4. Digest at 100% for 5 minutes then reduce power to 70% and digest again for 20 minutes.					
Constituent	**Certified Value of Constituents wt.%**	**Element**	**Reference Value (ppm)**	**ColdBlock Average (ppm)**	**Stdev**	**%RSD**	**% Recovery**
Al_2O_3	1.8	Al	9525	**9856**	217.6	2%	**103%**
CaO	43.6	Ca	311609	**332687**	5759.5	2%	**107%**
CdO	0.015	Cd	131	**145**	1.3	1%	**110%**
Cr_2O_3*	0.11	Cr	684	**794**	14.3	2%	**116%**
Fe2O3	0.79	Fe	5525	**5434**	268.6	5%	**98%**
K2O	0.51	K	4234	**4479**	372.6	8%	**106%**
MgO	0.33	Mg	1991	**2339**	127.6	5%	**117%**
MnO	0.0116	Mn	90	**97**	2.5	3%	**108%**
Na_2O	0.86	Na	6380	**6636**	651.6	10%	**104%**
P_2O_5	30.2	P	131799	**133460**	2790.4	2%	**101%**
V_2O_5	0.31	V	1737	**1681**	52.2	3%	**97%**
ZnO*	0.19	Zn	1526	**1705**	95	6%	**112%**
SiO_2	11.2	Si	52360	**13872**	916.4	7%	**26%**

* Indicative Values

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