

SUPPLEMENTAL MW DIGESTION FOR CHALLENGING MATRICES

Solomon Kariuki
UKDRS

Background

Test Name: 11 S MW v. 13

Run: 1

Status: Cancelled

Subcontract:

Lab Name: Select

Guar: 17

GuarM:

Sample Test Result Detail

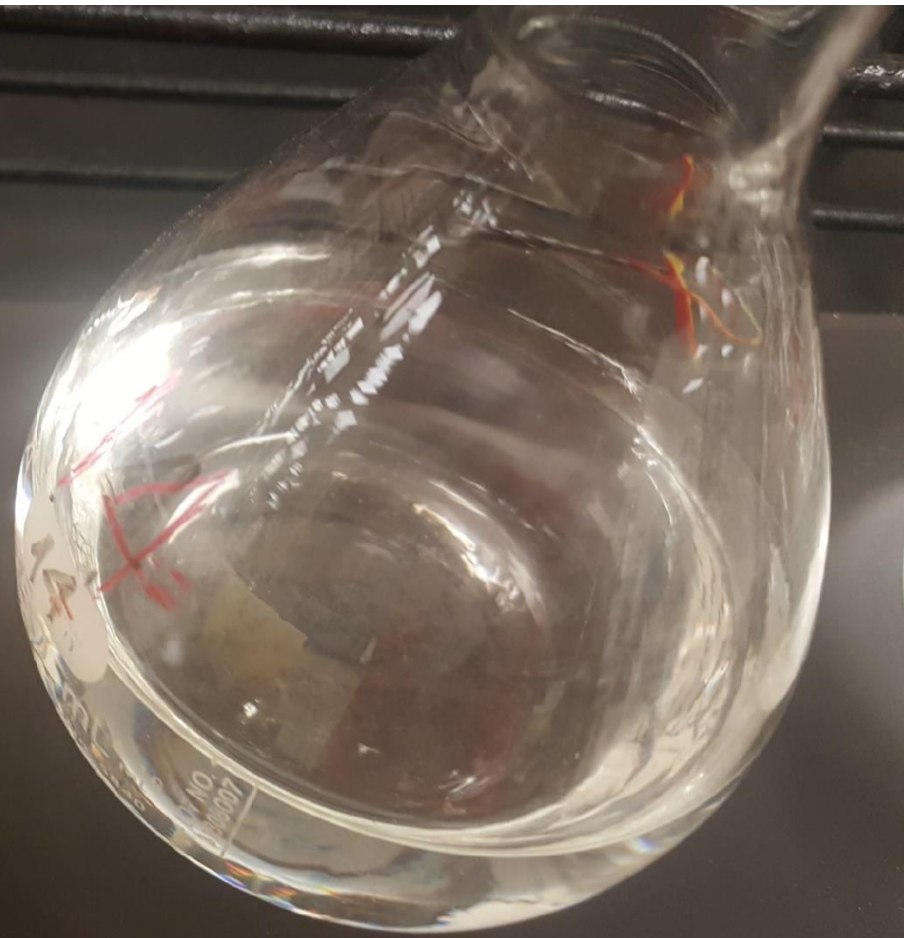
Param Name	Flag	Reported Value	Status	DL	QL	E	R
1_FDOR1_FROR1		x102348.5	Cancelled			<input checked="" type="checkbox"/>	<input type="checkbox"/>
2_FDOR2_FRRE1		153287.5	Cancelled			<input checked="" type="checkbox"/>	<input type="checkbox"/>
3_FDOR3_FROR2		x131312.5	Cancelled			<input checked="" type="checkbox"/>	<input type="checkbox"/>
4_FDOR4_FRRE2		x136817	Cancelled			<input checked="" type="checkbox"/>	<input type="checkbox"/>
5_FDRU1_FRRU1		147949	Cancelled			<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
6_FDRU2_FRRU2		x125014	Cancelled			<input checked="" type="checkbox"/>	<input type="checkbox"/>
Calc High Limit		100	Cancelled			<input checked="" type="checkbox"/>	<input type="checkbox"/>
Calc Low Limit		16	Cancelled			<input checked="" type="checkbox"/>	<input type="checkbox"/>
Determinations		6	Cancelled			<input checked="" type="checkbox"/>	<input type="checkbox"/>
Econvalue		9.5	Cancelled			<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Found		15.1	Cancelled			<input type="checkbox"/>	<input type="checkbox"/>

Background

- Samples consistently deficient on S and Ca with our SOP method
- Sample consistently met guarantee when analyzed by our referee labs
- LabW, block digestion 95°C, 1.5 h, 3mL HNO₃ + 3 mL HCl



Synthetic gypsum



Ammonium sulfate

Manufacturing process...

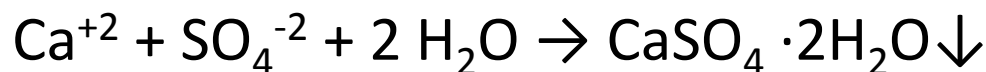
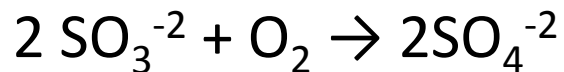
“Sulfur Dioxide from the exhaust is pulled into the reaction volume, where it is reacting with water and the resulting acid dissolves the added limestone”



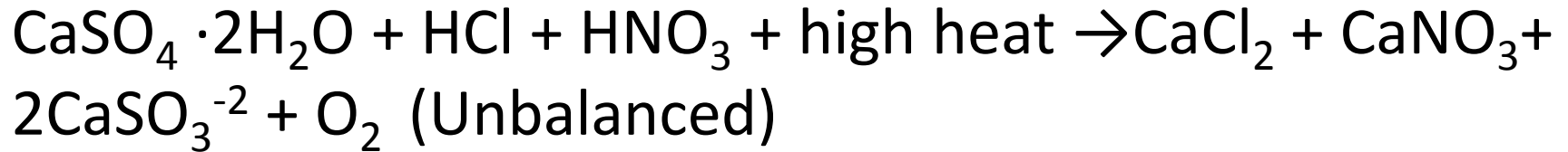
“The initial reaction of the ions generate calcium sulfite”.



“In the presence of higher oxygen, air blown into the reaction tank, the sulfites convert to sulfates”.



Microwave digestion



HIGH-TEMPERATURE VAPORIZATION OF GYPSUM AND ANHYDRITE :
Lunar and planetary institute...NASA Astrophysics Data System

- *Source of SO₂ and SO₃ in the earth atmosphere due to Chicxulub prehistoric impact*
- *Simulation by decomposition of gypsum and anhydrite with Pulse Laser*
- **Possibility of reduction of CaSO₄ to CaSO₃**

MW Digestion Parameters

0.25 g sample + 6 mL HNO₃ + ≥ 2 hours

Pre-digestion + 6 mL HCl

Stage 1:

Power, 1600 W @ 100%

Temperature, 160°C

Ramp, 20 min: Hold, 20 min

Stage 2:

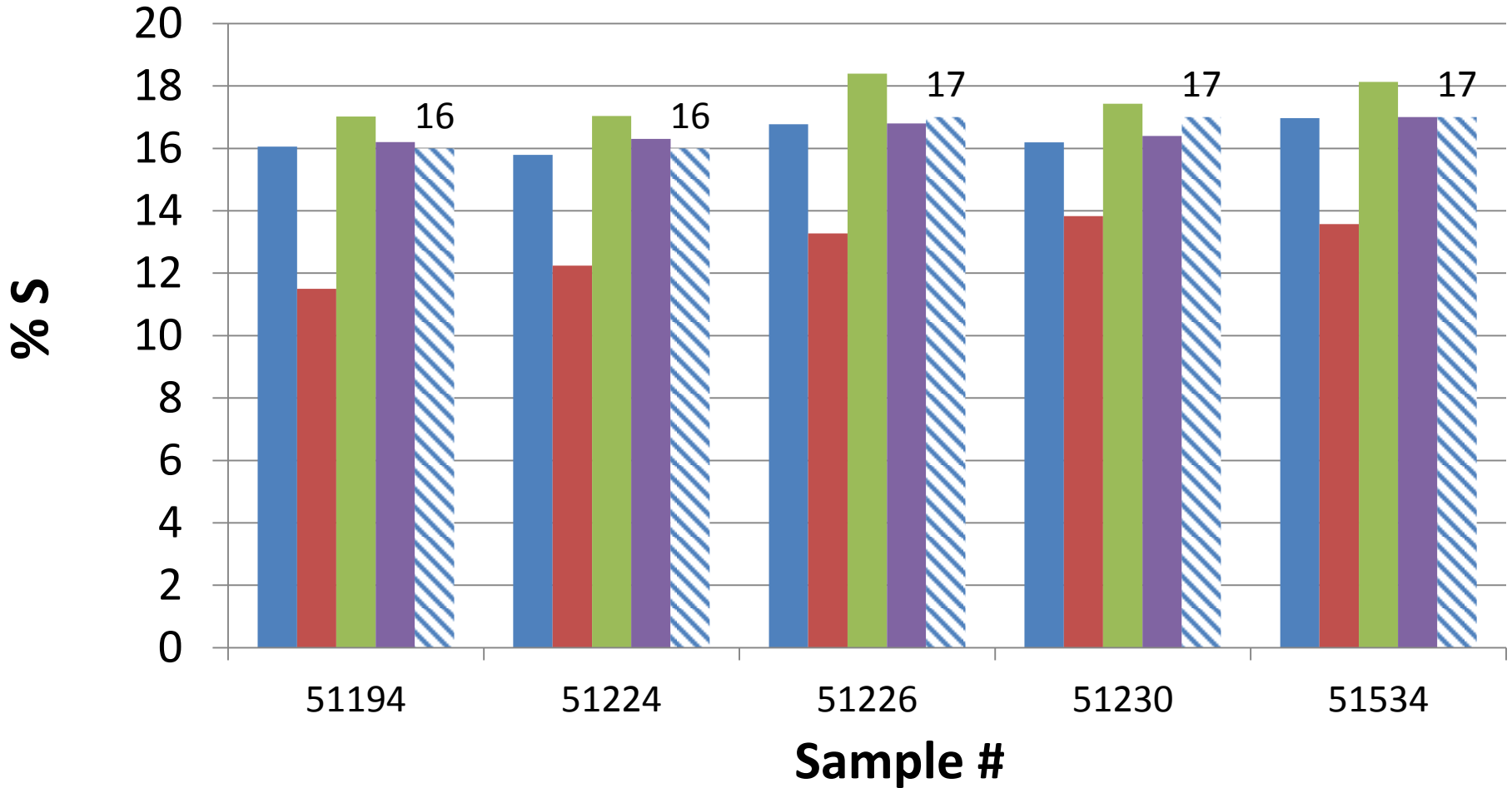
Power, 400 W @ 100%

Temperature, 75°C

Ramp, 5 min: Hold, 10 min

4 Methods Comparison of Total S Analysis

UKExpt UKSOP LabW LabTC Guarantee



4 Methods Comparison of Ca Analysis

UKExpt UKSOP LabW LabTC Guarantee

