

Application Note - Digestion of CONOSTAN® oil S-21

Introduction:

- 1) This study evaluated the effectiveness of the **NOVAWAVE** microwave digestion system. The study entailed the digestion of CONOSTAN oil S-21 standard.

Sample Type:

CONOSTAN oil S-21 standard – 500 ppm (Certificate Date: March 1, 2010)

- Sample weight: 0.1g
- 12 Replicates

Supplies and Reagent:

- 2) **NOVAWAVE SA** Model*
- 3) Quartz Vessels, 75ml*
- 4) Teflon® Caps and Safety Pressure Release Caps (pre-set release pressure at 435 psi)*
- 5) **PlasmaPURE** HNO₃ (70%), 10ml*
- 6) Analytical Balance 4 Decimal Places, Mettler-Toledo
- 7) Spectroflame Modula FMD-07 ICP-OES, Spectro Analytical
- 8) Crossflow Nebulizer, Mini X-Flow*
- 9) Alumina Injector Torch, 1.2 mm*
- 10) Cyclonic Spray Chamber, Baffled*
- 11) Graduated Cylinder, 10 ml
- 12) Volumetric Flask, 100 ml

* Manufactured by **SCP SCIENCE**

Sample Preparation Procedure:

The oil standard sample was weighted on a 4 place analytical balance directly into the 75ml quartz vessels. The 75ml vessel was selected for the extra head space. After adding 10ml of the HNO₃, the samples were allowed to sit at room temperature for 10 to 15 minutes. They were digested in the **NOVAWAVE** following the instrument's heating profile noted below. After cooling, samples were then normalized to 100 ml with DI H₂O in a volumetric flask.

Discussion:

At the end of the digestion and after cooling to room temperature, the vessels were safely vented in a fume hood. For an optimal recovery of boron, it is recommended that the vessels are placed in a cold (4C°) water bath for 10 minutes prior to normalization to 100 ml with DI H₂O. Recoveries are reported on 12 replicates.



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A new category of Automated Microwave Digestion

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NOVAWAVE Heating Program:

STAGE	RAMP TIME (Minutes)	PRESSURE (PSI – LIMIT)	TEMPERATURE °C	HOLD TIME (Minutes)
1	20	435	200	15

Recoveries:

EI.	AMU	NOVAWAVE (PPM)	STD DEV. (PPM)	CERT. VALUE (PPM)	TOLERANCE PPM	RECOVERY (%)
Ag	328.07	504	35	500	1	101%
Al	396.15	486	19	500	1	97%
B	249.68	513	25	500	1	103%
Ba	455.40	470	24	500	1	94%
Ca	396.85	517	27	500	1	103%
Cd	228.80	487	28	500	1	97%
Cr	283.56	503	20	500	1	101%
Cu	324.75	494	16	500	1	99%
Fe	259.94	487	22	500	1	97%
Mg	285.21	483	15	500	1	97%
Mn	257.61	509	18	500	1	102%
Mo	281.61	503	28	500	1	101%
Na	589.59	493	25	500	0	99%
Ni	221.65	474	26	500	1	95%
P	213.62	492	21	500	1	99%
Pb	220.35	529	42	500	2	106%
Sn	283.99	504	24	500	2	101%
Ti	334.94	521	16	500	0	104%
V	309.31	495	18	500	1	99%
Zn	213.86	486	19	500	1	97%

References:

Certificate of Analysis Conostan Standard S-21:500 ppm lot number 21393500

USEPA 3051 MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS.

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