

Application Note - The Digestion of Oil Samples

Introduction:

This application describes the digestion of oil samples (used oil, residual oil, etc...) prior to the analysis of metals by ICP-OES. The study entailed the digestion of a Conostan multi-element standard and a **SCP SCIENCE EnviroMAT** Used Oil CRM using a **DigiPREP HT** Graphite Block Digestion System with a Touch Screen Controller.

Sample Type:

- Conostan oil S-21 standard – 100 ppm (Certificate Date: February 2nd, 2010)
- EnviroMAT Used Oil CRM (Certificate Date: May 5th, 2009)
- Sample weight: 0.5g.
- 5 Replicates

Supplies and Reagents:

- 1) **DigiPREP HT** 250 (High temperature graphite digestion block)*
- 2) Touch Screen controller *
- 3) **DigiPREP HT** 250 digestion tubes *
- 4) Spectroflame Modula FMD-07 ICP-OES, Spectro Analytical
- 5) **PlasmaPURE** grade HNO_3 *
- 6) **PlasmaPURE** grade H_2SO_4 *
- 7) Pasteur pipettes
- 8) Rubber bulb
- 9) Analytical balance
- 10) 10 ml graduated cylinder, Corning
- 11) Deionized water
- 12) **DigiTUBE**s 50 ml*
- 13) Cyclonic baffled spray chamber*
- 14) Mini X-Flow nebulizer*
- 15) 1.2 mm Alumina Injector Torch*

Sample Preparation Procedure:

The oil sample is weighed directly into a **DigiPREP HT** 250 digestion tube, followed by the addition of H_2SO_4 (5 ml) and HNO_3 (5 ml). The tubes are placed in the insert rack which is then placed on the **DigiPREP HT** heating block. The heating profile is listed for the touch screen controller. At the second ramp, when the temperature reaches 260°C, 15 ml of HNO_3 is added dropwise. Strong orange fumes will occur. The acid is added until the orange fumes are gone (adding more HNO_3 if necessary). At the final ramp, when the temperature reaches 360°C, 10 ml of HNO_3 is added dropwise. Strong orange fumes will occur. When the solution is evaporated down to approximately 1 ml, another 10 ml of HNO_3 is added dropwise. At this point, the solution should become colorless. Cool the sample and transfer to a 50 ml **DigiTUBE**. Normalize to 50 ml with DI water. The sample is then ready for ICP analysis.

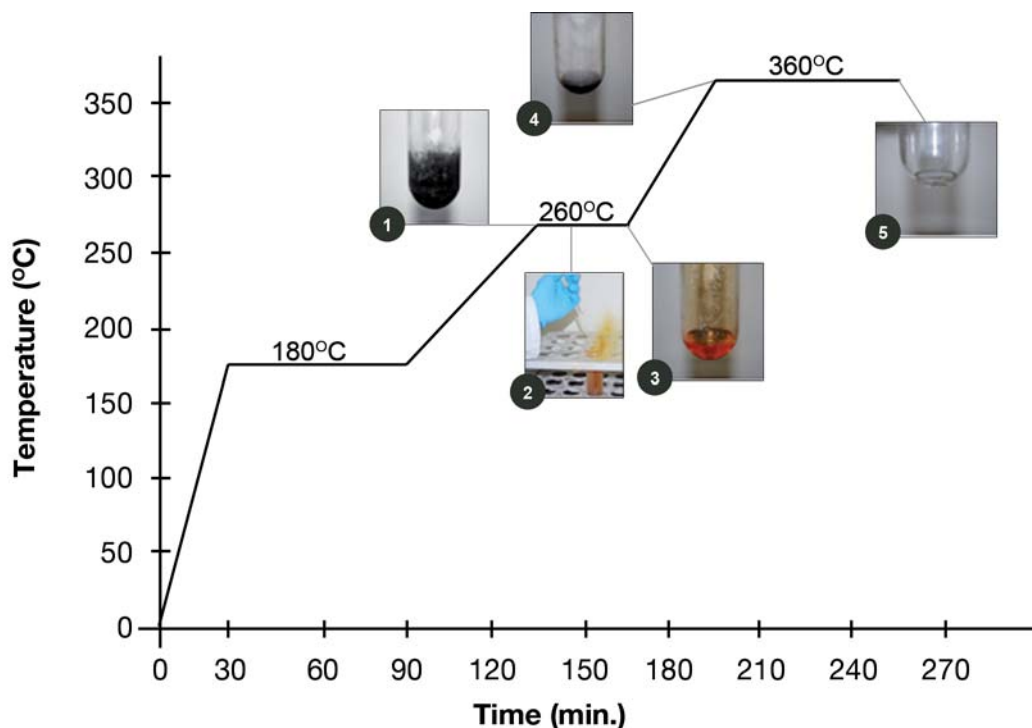
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DigiPREP HT Touch Screen Controlled Heating Program:

STAGE	RAMP TIME (Minutes)	TEMPERATURE (°C)	HOLD TIME (Minutes)
1	30	180	60
2	45	260	30
3	30	360	60

Temperature Profile:



- 1) When the temperature reaches 260°C, the solution should be black.
- 2) The addition of HNO₃ should be done slowly as the reaction is fairly violent as orange fumes will occur.
- 3) At the end of the second hold cycle, the color of the solution should be clear orange.
- 4) Once the temperature reaches 360°C, the volume of the solution should have decreased to approximately 3 ml and should be brown.
- 5) The addition of HNO₃ should be done slowly as the reaction is fairly violent as orange fumes will occur. Once the volume of the solution is down to 1 ml, add an additional 10 ml of HNO₃. At this point, the solution should be straw yellow to colorless. If not, add more HNO₃ until the solution is straw yellow to colorless.

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Recoveries Conostan standard:

Element Symbol	Wavelength (nm)	DigiPREP HT (ppm)	Tolerance (mg / kg)	Certified Value (ppm)	Tolerance (ppm)	Recovery (%)
Ag	328.07	101	3	100	1	101
Al	396.15	100	5	100	2	100
Ba	455.40	101	4	100	1	101
Cd	228.80	95	4	100	2	95
Cr	283.56	99	8	100	2	99
Cu	324.75	100	4	100	3	100
Fe	259.94	100	4	100	2	100
Mg	285.21	101	3	100	3	101
Mn	257.61	94	8	100	2	94
Ni	221.65	103	10	100	2	103
Pb	220.35	98	2	100	5	98
V	309.31	96	5	100	3	96
Zn	213.86	98	10	100	1	98

Recoveries EnviroMAT Used Oil standard:

Element Symbol	Wavelength (nm)	DigiPREP HT (mg / kg)	Tolerance (mg / kg)	Certified Value (ppm)	Tolerance (mg / kg)	Recovery (%)
Ag	328.07	10.3	4.8	13	3	80
Al	396.15	17.9	4.0	14	3	128
Ba	455.40	7.8	2.4	9.0	0.5	86
Ca	396.85	80.3	5.6	72	5	112
Cd	228.80	17.1	3.2	15	1	114
Cr	283.56	16.9	2.4	15	2	112
Cu	324.75	3729	452	3132	226	106
Fe	259.94	68.2	9.0	59	6	116
Mg	285.21	11.8	2.0	11	1	108
Mn	257.61	21.1	1.6	18	1	117
Ni	221.65	48.1	8.4	45	3	107
Ti	334.94	9.8	2.6	9	2	109
V	309.31	9.2	3.2	7	0.5	132
Zn	213.86	19.2	3.0	16	2	120

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Conclusion

As shown in the graph above, the interval of confidence for *Digi*PREP HT values (red square) overlap the interval of confidence for the *Enviro*MAT CRM values (blue diamond). Therefore, the method is working properly.

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