Application Note - Digestion of Nickel Alloy

Introduction:

This study was performed to investigate trace elements in nickel alloy. The effectiveness of the *NOVA WAVE* microwave digestion was evaluated through comparison with known values.

Sample Type:

Nickel alloy (Ni 200) – 0.1 g 2 replicates

Supplies and Reagents:

NOVA WAVE Model SA Microwave Digestion System *
 Quartz Vessels, Calibrated at 50 ml *
 Teflon[®] Caps and Safety Pressure Release Caps (30 bar - 435 psi) *
 PlasmaPURE HNO₃ (70%)
 PlasmaPURE HCI
 Analytical Balance 4 Decimal Places, Mettler-Toledo
 ICP-AES Spectrometer, Spectro Analytical Inc., Ciros Model
 Nebulizer, Mini X-Flow *
 Torch, with Alumina Injector, 1.2mm diameter *
 Baffled Cyclonic Spray Chamber*
 Graduated Cylinder, Corning, 10 ml
 Volumetric flask, Corning, 50 ml
 * Manufactured by SCP SCIENCE

Sample Preparation Procedure:

A sample of 0.1 g was added to a 9:3 HNO₃: HCl mixture. The sample was allowed to stand at room temperature for 20 minutes. The vessel was then placed in the microwave digestion system and digested following the temperature profile below. At the end of the digestion, the vessel was vented and transferred to a 50 ml Teflon volumetric flask. Although no silicates are noticeable, two drops of HF were added to ensure total digestion and the solution was stirred manually for about 1 minute. The solution was normalized to 50 ml with DI water and analyze on the ICP-OES. The results are given as an average of 2 replicates.

NOVA WAVE Heating Program:

Stage	Ramp Time	Temperature	Hold Time
	(minutes)	(°C)	(minutes)
1	10	220	20

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Results for Ni alloy

Element	λ	NOVA WAVE (%)	Limiting Chemical Composition (%)
S	178.0	0.0055	0.01
Si	251.6	0.0673	0.35
Mn	257.8	0.3511	0.35
Fe	259.5	0.0659	0.40
Cu	324.8	0.2611	0.25
Ni+Co	N/A	99.2491	99.0 (min. value)

Discussion/Conclusion

Reverse aqua regia is an excellent acid combination for the digestion of nickel alloy. The solutions are green-clear after the digestion and the concentrations of trace elements are equal or below the maximum values. The Ni+Co value was obtained by subtraction from the other impurities.

References

EPA 3051 Microwave digestion method Certificate of Analysis for Ni 200 (Huntington Alloys)



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