BERGHOF PRODUCTS + INSTRUMENTS GMBH

Application Note XT8
Microwave Digestion of Low Alloy Steel

Summary

A sample preparation method of low alloy steel for elemental analysis is introduced below. The three alloy samples containing various concentrations of tin are digested using speedwave XPERT in high pressure DAK-100 vessels. During the digestion, the reaction temperature is controlled via contactless in-situ temperature sensor (DIRC) to ensure efficient digestion.

Instrumentation

Microwave Digestion	Rotor and Vessel Type	Liner Type		
	☐ DAP-40X			☐ MiniVessels
	☐ DAP-60X	☐ DAQ-20H		☐ MiniVessels
	☐ DAP-100X	☐ DAQ-22H	☐ DAC-17	☐ MiniVessels
	☑ DAK-100X		☐ MultiTube	☐ MiniVessels

Procedure

Sample Amount	500 mg
Sample Preparation	n/a
Reagent/s [2]	5 ml HNO_3 (65%) and 5 ml HCl (37%)
Experiment	Weigh sample into the vessel. Add reagent/s.
	Shake the mixture carefully or stir with a clean PTFE or glass bar.
	Keep the vessel in the fume hood at least 30 min for pre-reaction.
	Seal and close the vessels as described in the operation manual.
	Start the digestion according to the following program.
	Allow the vessels to cool down to room temperature and open them carefully as described in the operation manual. [1]
	Transfer the sample into centrifugal tubes and dilute them to 25 ml before the analysis.

Temperature Program [2]	Step	T [°C]	p [bar] [3]	Ramp [min]	Hold [min]	Power [%] [4]
	1	150	80	2	5	60
	2	200	80	2	10	60
	3	230	80	2	30	60
	4	50	40	1	10	10

Results	Clear and yellow solutions.

Discussion	In this application, three samples are digested in DAK-100 vessels in speedwave XPERT. These samples are low alloy steel that are iron (Fe) alloyed with carbon (1.13 to 1.25 %) and containing alloying elements, such as tin (Sn), manganese (Mn), nickel (Ni), phosphor (P), chromium (Cr) and silicon (Si). These elements tune the property of the low steel alloys in terms of their corrosion resistance and hardness. It is crucial to determine these elements quantitatively to find out the optimum composition of the sample for the best quality. At this point, microwave digestion provides a fast sample preparation technique for determination of major (sub-percent) to trace (ppm) elements in low alloy steel samples. The HNO_3:HCl (1:1 % v/v) is used to digest 500 mg samples. If necessary, the sample amount can be increased up to 1 g for low alloy steel samples in DAK-100 vessels. HCl will dissolve the iron and the elements given above. Addition of HNO_3 should prevent evaporation of elements as hydride form, such as SnH_4, PH_3 and SiH_4 by oxidizing these elements. These oxides will then be soluble in HCl, except Si. If the element of Si is the purpose of your analysis, HF (1-2 ml) should be also added as a reagent to dissolve the SiO_2. The toxic gases formed during the acid digestion will be collected and removed safely by the fume collection and the ventilation system of the instrument. To conclude, speedwave XPERT provides fast, efficient and safe sample preparation of low alloy steels for elemental analysis.
Notes	[1] To avoid foaming and splashing wait until the vessels have cooled to room temperature (about 20 min). Carefully open the digestion vessel in a fume hood wearing hand, eye and body protection since a large amount of fumes will be produced during the digestion process. [2] This application serves only as a guideline and may need to be optimized for your sample. [3] Pressure is the maximum value given to the program that is limited by the vessel and / or rupture disc specifications. [4] This application is outlined for 4 samples. Increase or decrease the power by 10% per sample, when using more or less sample. Minimum is 40% independent of the sample number.