Comparison of vessel materials for precise determination of mercury in coal

Introduction

Mercury is a powerful neurotoxic element that is of concern in all its forms. Releases of mercury into the environment are a major concern because of their potential impact, particularly on human health.

Coal contains a variety of toxic elements and mercury is ubiquitous in coal. Since many large power stations burn in excess of a million tons of coal per year their output was identified as the major source of mercury released to the environment.

Coal-rich countries, looking to secure their energy supplies by using domestic coal, will find it increasingly important to control emissions of mercury. In order to effectively determine the mass balance of mercury in power plants, inputs of mercury as well as outputs to the environment must be measured.

Instrumentation

The sample digestions were carried out with the Multiwave 3000 high performance 8-position rotors with simultaneous active pressure control in all vessels using two different vessel types, made by **Anton Paar** GmbH, Graz:

1. High performance Rotor 8XF100
   100 mL PTFE-TFM vessels, 260°C, 60 bar

2. High performance Rotor 8XQ80
   80 mL quartz vessels, 300°C, 80 bar

Determinations were performed on a Perkin Elmer Flow Injection Mercury System (FIMS 400) using the manifold system for vapour generation.

Samples

150-160 mg of two different NIST Standard Reference Materials:

1. Coal 2683b
2. Coal 2692b

Reagents

Mercury standard (1000 μg/mL), PE-Xpress

For XF100:

6 mL HNO_3 (suprapur, 70% (m/v), Seastar)
2 mL HCl (suprapur, 35% (m/v), Seastar)
1 mL HF (suprapur, 64% (m/v), Seastar)
3 mL H_2O (deionized)

For complexation:

6 mL H_3BO_3 (cold saturated)

For XQ80:

6 mL HNO_3 (suprapur, 70% (m/v), Seastar)
4 mL H_2O_2 (suprapur, 30% (m/v), Caledon Labs)

Analytical procedure

1. Weigh in aliquots (150-160 mg) from an air-dried (100°C) sample into Teflon digestion vessels or quartz digestion vessels, respectively.
2. Add the reagents.
3. Digest the samples and the blank.

Digestion Program for XF100 and XQ 80 vessels:

	Power [W]	Ramp [min]	Hold [min]	Fan
1	600	20	10	1
2	800	---	20	1
3	1000	---	20	1
4	0	---	15	3

Due to the use of HF in the XF 100 vessels the digestion program with XF100 vessels is followed by a second complexing step with 6 mL boric acid additionally added to the sample solutions to minimize attack of HF on the volumetric glassware.

Complexation:

	Power [W]	Ramp [min]	Hold [min]	Fan
1	1000	5	15	1
2	0	---	15	3

4. Transfer the solutions quantitatively to 100 mL volumetric flasks and make up to the mark with deionized water.
5. Prepare standard solutions containing 0.1, 0.2 and 0.5 ng / mL by serial dilution of the mercury standard with 5 % (v/v) nitric acid.
6. Determine the trace element content by flow-injection mercury analysis system.

Results

Mercury in coal – result with XF100 vessels

Batch	Hg concentration [μg/kg]	Hg recovery [%]	Certified value [μg/kg]
1-2683	156 ± 4	**173**	90.0
2-2683	116 ± 2	**129**	90.0
1-2692	136 ± 4	**102**	133.3
2-2692	151 ± 4	**113**	133.3

n=3

Mercury in coal – result with XQ80 vessels

Batch	Hg concentration [μg/kg]	Hg recovery [%]	Certified value [μg/kg]
1-2683	86 ± 4	**96**	90.0
2-2683	97 ± 3	**108**	90.0
1-2692	138 ± 3	**104**	133.3
2-2692	131 ± 5	**99**	133.3

n=3

Conclusion

The digestions with XF100 vessels showed poor reproducibility and tended to deliver high values. The vessels gave high and variable blanks even after blank digestion.

This applies only for the determination of mercury whereas other elements have been found with high accuracy in this and other tests.

Digestions with XQ80 vessels delivered superior results for mercury, compared to the XF100 type.

The XQ80 vessels showed consistently near-zero blanks and a greater accuracy for mercury determination.

References

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