

78-2 (2004)	Antimony and compounds (except stibine) (as Sb)
CAS N°: 7440-36-0	EINECS N°: 231-146-5
EC-LV (8 h): - Lowest European LV (8h): 0,5 mg/m³ Highest European LV (8h): 0,5 mg/m³	EC-STLV: - Lowest European STLV: 1 mg/m³ Highest European STLV: 2 mg/m³

SUMMARY OF THE METHOD

Language: English	Reference: Metals and metalloids in workplace air by X-ray fluorescence spectrometry , MDHS 91, Methods for the Determination of Hazardous Substances, HSL (1998).
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Summary: Air is drawn through an MCE or other suitable filter mounted in an inhalable sampler. Dust and fume collected on the filter is analysed by XRFS using either the K α or L β line. The XRF spectrometer is calibrated with filters prepared by sampling dust of known composition from an atmosphere generated in a dust chamber. A 'ratio monitor standard' is used to minimise the effects of instrument drift.

SAMPLING

Sampler type	Multi-orifice sampler, IOM sampler or CIS, as described in MDHS 14/2 (now MDHS 14/3)
Sampling substrate	MCE filter or other suitable membrane filter
Recommended flow rate	2 l/min (multi-orifice sampler and IOM sampler) 3,5 l/min (CIS)
Recommended sampling time	K α line: 2 – 8 h, L β line: 30 min – 8 h
Recommended volume	-

TRANSPORT AND STORAGE

Description/conditions of transport and storage incl. specific issues	The sample filter is transferred to a filter transport cassette for transport to the laboratory, or it is transported in the sampler used for sampling or its internal filter cassette, after sealing with suitable plastic caps. There is a risk of sample loss from the filter if it is not handled carefully during transportation, especially for higher sample loadings, but once in the laboratory samples are stable indefinitely.
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ANALYSIS

Sample preparation	not applicable
Analytical technique	Analysis by XRFS.

METHOD EVALUATION DATA

Range studied	Not applicable – see limit of quantification.
Sampling bias	Overall uncertainty calculation: < 5 % (according to EN 13890) Expanded uncertainty calculation: included in sampling precision
Analytical bias	< 10 % (estimated)
Method bias	-
Sampling precision	Overall uncertainty calculation: < 5,3 % (according to EN 13890) Expanded uncertainty calculation: 9,0 % (incorporates bias uncertainty)

METHOD EVALUATION DATA (continued)				
Analytical precision	K α line: 4,2 – 8,2 %, L β line: 2,8 – 6,1 %			
Method precision	-			
Limit of quantification	K α line: 5 μ g per filter, L β line: 1 μ g per filter			
Overall uncertainty (EN 482)	K α line:			
	0,1 \times LV 34 %	0,5 \times LV 28 %	2 \times LV 28 %	UK LV (0,5 mg/m ³)
	L β line:			
	0,1 \times LV 31 %	0,5 \times LV 27 %	2 \times LV 26 %	UK LV (0,5 mg/m ³)
Expanded uncertainty (prEN 482)	K α line:			
	0,1 \times LV 25 %	0,5 \times LV 24 %	2 \times LV 24 %	LLV and HLV
	L β line:			
	0,1 \times LV 24 %	0,5 \times LV 23 %	2 \times LV 23 %	LLV and HLV
INFORMATION IN RELATION TO THE VALIDATION				
Is the sample dissolution procedure described widely applicable?	not applicable			
Does the analysis include wall deposits, where applicable?	no			
Was a test gas atmosphere used, where applicable?	not applicable			
How was the recovery determined?	The effect of particle size and depth effects was assessed and provided the physical size of collected particles is < 2,5 μ m and < 500 μ g of sample is collected, the analytical bias is expected to be < 10 %.			
Was the sampler capacity or breakthrough volume determined?	A maximum sample loading is specified of 0,5 mg for a 25 mm filter or 1 mg for a 37 mm filter.			
Was temperature and RH considered, where appropriate?	not applicable			

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EVALUATION	
Rating category	A 1
Rationale for rating	<p>Up to date methodology, detailed method description, overall uncertainty and expanded uncertainty requirements met</p> <p>The overall uncertainty data above have been determined from the analytical bias and precision data given in HSL back-up data report IS/97/07 using the calculation method and sampling bias and precision estimates given in EN 13890. The expanded uncertainty data have been calculated using the method described in the EU mandated report <i>Analytical methods for chemical agents</i>. The data are for samples with the minimum recommended air volumes of 240 l for the Kα line and 60 l for the Lβ line.</p>
Observations	If dust deposited on the internal surfaces of a sampler forms part of the sample, XRFS filter analysis results can only be regarded as suitable for screening purposes. The method only gives quantitative results for certain sampler types.
Similar methods	None.