

99-2 (2004)	Bromine
CAS N°: 7726-95-6	EINECS N°: 231-778-1
EC-LV (8 h): 0,7 mg/m ³ Lowest European LV (8h): 0,66 mg/m ³ Highest European LV (8h): 0,7 mg/m ³	EC-STLV: - Lowest European STLV: 0,66 mg/m ³ Highest European STLV: 2 mg/m ³

SUMMARY OF THE METHOD

Language: English	Reference: Bromine and Chlorine: NIOSH 6011, NIOSH Manual of Analytical Methods, Cincinnati (1994).
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Summary: Air is drawn through a silver membrane filter to collect bromine, using a PTFE prefilter to trap particulate bromide. Both filters are mounted in an opaque, three-piece, 25 mm carbon-filled polypropylene sampling cassette with a 50 mm extension, using porous plastic support pads to support both filters. After sampling, the silver membrane filter is extracted with 6 mM Na₂S₂O₃ and the sample is analysed for bromide by IC.

SAMPLING

Sampler type	Three-piece, 25 mm carbon-filled polypropylene sampling cassette with a 50 mm extension
Sampling substrate	Silver membrane filter
Recommended flow rate	0,3 – 1 l/min
Recommended sampling time	< 8 h
Recommended volume	-

TRANSPORT AND STORAGE

Description/conditions of transport and storage incl. specific issues	Sampling cassettes are sealed with plastic plugs for transport to the laboratory. Samples have been found to be stable for up to 60 days at 25°C (99,2 ± 10,1% recovery).
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ANALYSIS

Sample preparation	The sampling cassette is opened under very dim or red light and the silver filter is transferred to an amber bottle. 6 mM Na ₂ S ₂ O ₃ (3 ml) is added and the collected bromine is extracted by standing for at least 10 min with occasional swirling. The sample solution is then made to 10 ml by addition of H ₂ O (7 ml). The PTFE prefilter may be prepared and analysed for particulate bromides or it may be discarded.
Analytical technique	Analysis by chemically-suppressed IC and conductimetric detection. A pre-column is used before the separator column to remove Ag ⁺ and Ag(S ₂ O ₃) ₂ ³⁻ , which can cause deterioration in separator column performance.

METHOD EVALUATION DATA

Range studied	0,07 – 1,42 mg/m ³
Sampling bias	-
Analytical bias	-
Method bias	- 1,2 %

METHOD EVALUATION DATA (continued)	
Sampling precision	-
Analytical precision	-
Method precision	6,8 %
Limit of quantification	5,3 µg per sample
Overall uncertainty (EN 482)	15 %
Expanded uncertainty (prEN 482)	17 %
INFORMATION IN RELATION TO THE VALIDATION	
Is the sample dissolution procedure described widely applicable?	yes
Does the analysis include wall deposits, where applicable?	not applicable
Was a test gas atmosphere used, where applicable?	yes
How was the recovery determined?	From the results of test gas atmosphere experiments.
Was the sampler capacity or breakthrough volume determined?	not determined
Was temperature and RH considered, where appropriate?	The method was evaluated by sampling generated atmospheres of Br ₂ at both high (80%) and low (20%) relative humidities. The effect of temperature was not studied.
EVALUATION	
Rating category	A 1
Rationale for rating	<p>Up to date methodology, detailed method description, overall and expanded uncertainty requirements met.</p> <p>The overall uncertainty data above have been calculated from NIOSH overall recovery and precision data using the formula in EN 482. The expanded uncertainty data have been calculated using the method described in the EU mandated report <i>Analytical methods for chemical agents</i>.</p>
Observations	<p>NIOSH report an LOD of 1,6 µg per sample. The LOQ above has been calculated from this by multiplying by 10/3.</p> <p>There is only one supplier of silver membrane filters.</p> <p>H₂S gives a negative interference. HBr gives a positive interference.</p> <p>The method is also applicable to the determination of Cl₂.</p>
Similar methods	None.