

99-1 (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 in workplace atmospheres: OSHA ID-108, OSHA Sampling and Analytical Methods, Salt Lake City (1990).
-----------------------------	-----------------------------------------------------------------------------------------------------------------------------------------

Summary: Air is drawn through a midjet fritted glass bubbler containing 3mM NaHCO₃ / 2,4 mM Na₂CO₃ solution to collect bromine. The sample solution is analysed for bromide and bromate by IC. Bromine in air concentrations calculated from the bromide results are reported. Bromine in air concentrations calculated from the bromate results are used as a cross-check to test for possible interference by bromide.

SAMPLING

Sampler type	Midget fritted glass bubbler
Sampling substrate	3mM NaHCO ₃ / 2,4 mM Na ₂ CO ₃ solution (10 – 15 ml)
Recommended flow rate	0,5 l/min
Recommended sampling time	15 min – 4 h
Recommended volume	-

TRANSPORT AND STORAGE

Description/conditions of transport and storage incl. specific issues	The collection solution is transferred into a 20 ml glass scintillation vial for transport to the laboratory, rinsing the bubbler into the sample vial with unused collection solution (2-3 ml), closing tightly with a Teflon-lined cap and sealing around the caps with vinyl or waterproof tape to prevent leakage. Samples may be stored under normal laboratory conditions for a period of at least 30 days. (Sample stability tests yielded recoveries of 101,9 %, 104,2 %, 100,7 % and 88,9 % after 1, 5, 15 and 30 days respectively for samples stored at normal laboratory temperatures.)
------------------------------------------------------------------------------	-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------

ANALYSIS

Sample preparation	There is no sample preparation, but the total volume of each sample solution measured with a graduated cylinder before analysis, since it varies according to the amount of evaporation of the collection solution that occurs during sampling and the precise volume of unused collection solution used to rinse out the bubbler after sampling. If there is suspended particulate, an aliquot of the sample solution is filtered using a syringe filter for analysis.
Analytical technique	Analysis is for bromide by chemically-suppressed IC and conductimetric detection. Stoichiometric factors of 1,2 and 3,75 are applied to bromide and bromate results, respectively, to take account of the disproportionation that occurs ($3 \text{ Br}_2 + 6 \text{ OH}^- \rightarrow 3 \text{ H}_2\text{O} + 5 \text{ Br}^- + \text{BrO}_3^-$).

METHOD EVALUATION DATA	
Range studied	0,052 – 0,205 ppm (0,34 – 1,37 mg/m ³)
Sampling bias	-
Analytical bias	-
Method bias	- 5,6 %
Sampling precision	-
Analytical precision	-
Method precision	6,7 %
Limit of quantification	0,9 µg per sample
Overall uncertainty (EN 482)	19 %
Expanded uncertainty (prEN 482)	16 – 22 %
INFORMATION IN RELATION TO THE VALIDATION	
Is the sample dissolution procedure described widely applicable?	not applicable
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?	Breakthrough tests were performed. No breakthrough was found after 120 min for a concentration of 1,25 mg/m ³ . Breakthrough was determined to be 2,4 % after 240 min.
Was temperature and RH considered, where appropriate?	not applicable
EVALUATION	
Rating category	A 2
Rationale for rating	<p>Up to date methodology, detailed method description, overall uncertainty and expanded uncertainty requirements met, range studies does not cover 0,1×LLV.</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>If present in the air, Br⁻ (e.g. from HBr) gives a positive interference. However, results determined from Br⁻ are confirmed by determining BrO₃⁻, the other product of disproportionation of bromine.</p> <p>Use of liquid in glass bubblers for sample collection is a disadvantage, as spillage or breakage can occur if precautions are not taken.</p>
Similar methods	None.