Low-level Elemental Analysis of Filters

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Objective

XRF analysis of liquids is common but the risk of a sample spill is never totally avoided. Using filters instead has several advantages:

- The samples can be measured in vacuum
  - No He necessary
  - Better detection for light elements
- No sample breakages
- Filters produce less waste
- Less sample is needed

Usually an ED-XRF system is used for filter analysis, because high power WD-XRF instruments might damage the filter.

However, it can be tricky to measure certain elements due to the configuration of most ED-XRF spectrometers. Low levels of heavier elements can be hard to achieve, if the excitation energy is not sufficient or sum peaks and escape peaks interfere with the signal. The characteristic lines of lighter elements of interest (S, P, Cl) often interfere with the L-lines of most X-ray tubes, so measuring lower concentrations is challenging.

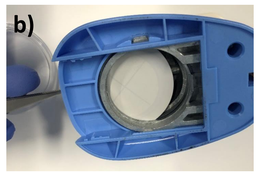
These challenges are overcome when using a low power WD-XRF system. Here we demonstrate the analysis of low levels of Br and Cl using a 400W WD-XRF spectrometer, Bruker's S6 Jaguar.

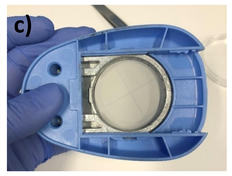
Sample Preparation and Standards

Known amounts of solutions with defined concentrations of Cl and Br were filtered through a to make standards.

For unknown samples, the same amount as for the standards were filtered

The filter was dried and cut to the right size (hole punch, 38 mm)

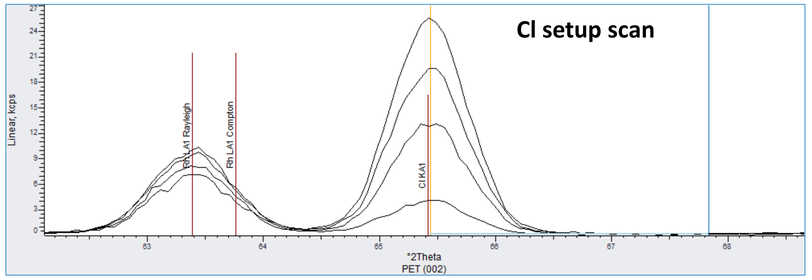


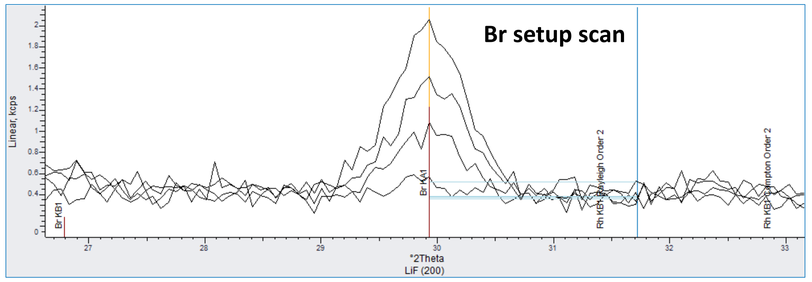


	Std-1	Std-2	Std-3	Std-4	Std-5	Std-6
Cl [PPM]	100	200	300	400	500	700
Br [PPM]	5	10	15	20	25	35

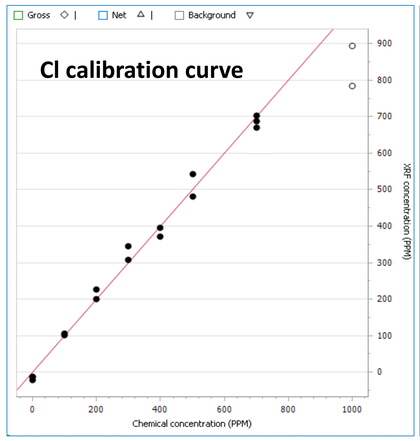
Method

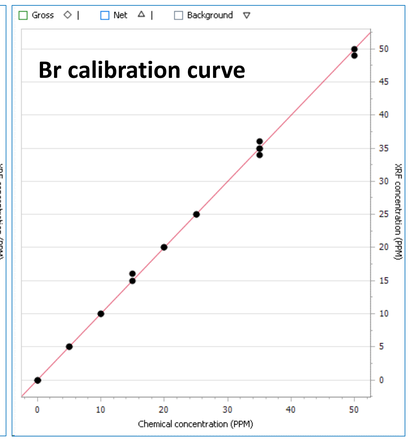
The method was simple using default crystals as commonly used in the industry – PET for Cl, Li200 for Br – and measuring peak / background for 120 s / 30 s for Cl and 180 s / 60 s for Br.





The calibration curves show great correlation and a small standard deviation.





Calibration Coefficients	
Standard deviation	22.6 PPM
Squared correlation coefficient	0.99189

Calibration Coefficients	
Standard deviation	0.3 PPM
Squared correlation coefficient	0.99959

Results

To check the results 2 samples were tested and a repetition test was run. The control samples show great accuracy of the calibration. The repeatability is outstanding and shows that samples can be remeasured without having to worry about radiation damage to the sample.

		Cl (PPM)	Br (PPM)
C1	Given	630	38
	S6	633.5	38.2
C2	Given	400	20
	S6	388.1	20.1

Rep #	Cl (PPM)	Br (PPM)	Cl (kCps)	Br (kCps)
1	631	38	21.107	1.115
2	632	38	21.147	1.118
3	632	38	21.160	1.114
4	633	38	21.195	1.124
5	633	38	21.172	1.122
6	634	38	21.204	1.125
7	635	38	21.257	1.125
8	634	39	21.203	1.127
9	633	39	21.195	1.126
10	635	39	21.251	1.128
11	635	38	21.231	1.123
12	635	39	21.259	1.128
AVG	634	38	21.198	1.123
SD	1.4	0.5	0.0	0.0
RSD	0.22%	1.28%	0.22%	0.42%

Conclusions

- The S6 Jaguar is the perfect instrument when high resolution WD-XRF measurements of radiation-sensitive samples is needed
- This measurements are straightforward and low levels of challenging elements like Cl, P and S or elements heavier than Rh can analyzed with a few clicks.
- The principle of this application can easily be transferred to water treatment processes, measuring biofluids and pharmaceutical solutions and petrochemical products.
- The detection limit and the statistical error are below 1 ppm.

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