EDXRF APPLICATION NOTE

ANALYSIS OF METALS IN AEROSOLS ON AIR FILTERS

#1549

SCOPE

Measurement of metals, especially toxic heavy metals, in aerosols on air filters is demonstrated. Simple linear empirical calibration is discussed, and performance is shown for measurement precision and typical detection limits.

BACKGROUND

Element analysis of aerosols and particulate matter released in smokestacks and other industrial gas discharge is vital to ensure that environmentally acceptable levels of toxic and hazardous elements are released properly into the air. Monitoring smoke or other gaseous waste is important in many areas, such as industrial manufacturing, coal-fired power plants, chemical and plastics production, etc., in order to minimize air pollution and the release of toxic metals in compliance with US EPA and other world and regional or local governing regulations. As a tool to help ensure compliance and proper release, Rigaku offers the NEX DE EDXRF analyzer with 60 kV excitation source and high resolution and throughput Si Drift Detector, giving the analysts and technician alike a fast, simple, yet powerful means for monitoring elemental analysis of air filters.

INSTRUMENTATION

Model:	Rigaku NEX DE
X-ray tube:	12W 60kV Ag-anode
Detector:	High Throughput SDD
Sample Type:	Nuclepore® polycarbonate Aerosol membranes
Film:	Prolene (4um)
Analysis Time:	1200 sec **(**300 sec per measurement condition)
Environment:	Helium (for optimum light element performance)
Autosampler:	10-position 40mm Automatic Sample Changer

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SAMPLES

Air filter samples were prepared by MicroMatter, a global supplier of XRF aerosol air filter standards. The filter material used was Nuclepore polycarbonate aerosol membrane. Teflon membrane material is not recommended, as Teflon film shows a background three times higher than other membrane materials. The filter membrane is supported by a 40mm diameter plastic outer ring. Samples lie flat and smooth in the 10-position autosampler, covering the analysis aperture.

CALIBRATION

While heavier loadings may require a curved fit, analysis of the typical loadings shown here in the µg/cm^2 range show a linear XRF response, and so single-point calibrations are suitable. A single-point linear empirical calibration was made for each element using the Standard Value loading levels shown in the RECOVERY and REPEATABILITY section.

RECOVERY and REPEATABILITY (Precision)

To demonstrate typical measurement recovery and instrument precision, each calibration standard was measured in 10 repeat analyses using a total analysis time of 1200 sec (300 sec per measurement condition). Fewer measurement conditions may be used depending on overall composition of the filter loadings.

Units: µg/cm^2

Measurement Condition	Element	Standard Value	Average Value	Standard Deviation	% RSD
Low-Z	Al	54.3	53.9	0.18	0.3
	Si	31.6	31.9	0.04	0.1
Low-Mid-Z	Cr	46.2	45.9	0.06	0.1
	Ba	41.3	41.6	0.02	<0.1
High-Mid-Z	Ga	23.8	23.4	0.05	0.2
	As	25.5	25.2	0.04	0.2
	Se	19.9	20.0	0.03	0.2
	Hg	32.2	31.9	0.07	0.2
	Pb	60.3	60.7	0.09	0.1
High-Z	Ag	22.6	22.5	0.06	0.3
	Cd	48.1	48.2	0.09	0.2

TYPICAL DETECTION LIMITS

To determine the Lower Limit of Detection (LLD) using the empirical method, ten repeat analyses of a blank filter membrane sample were measured and the standard deviation calculated. The LLD is then defined as three times the standard deviation. This approach ensures that analyses above the determined LLD are measuring signal above background. The following typical LLDs were determined using 300 sec per measurement condition. Actual detection limits may vary based on analysis time used, combinations of elements present and filter membrane material used.

Element	LLD (ng/cm^2)
Al	12.6
Si	5.7
Cr	3.3
Ba	3.6
Ga	3.0
As	1.2

Element	LLD (ng/cm^2)
Se	1.5
Hg	3.6
Pb	2.7
Ag	25
Cd	45

Note: 1ng/cm^2 = 0.001 ug/cm^2
1ug/cm^2 = 1000 ng/cm^2

CONCLUSION

The Rigaku NEX DE combines filtered direct excitation with a high performance SDD detector capable of 500,000+ cps throughput to deliver excellent sensitivity for the measurement of metals in aerosols on air filters. The NEX DE analyzer is capable of elemental analysis from Na – U, making the XRF technique ideal for other elements on air filters, as well. For the measurement of light elements Na-Cl, helium purge is used to remove air from the optical path and optimize analysis sensitivity. Elements from Cr and above can be easily measured in air without the need for helium purge.

The NEX DE analyzer also gives the user a very versatile tool not only for monitory air filters, but also elemental analysis of solids, powders and liquids. This power and simplicity makes the analyzer an ideal tool for screening and monitoring particulate aerosol filers to help ensure industrial processes are environmentally sound and meet regulatory compliance.

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