

Arsenic Speciation in FGD Wastewater Using LC-HG-AFS

Application Note APP164

The determination of Arsenic in wastewater from industrial sources is a challenging application. In recent years wastewater treatment plants have been installing technologies to capture arsenic and other heavy metals. The efficiency of these treatment processes is dependent on the form of arsenic present and as such there is a need to speciate arsenic upstream and downstream of pollution abatement technology so the process can be optimized.

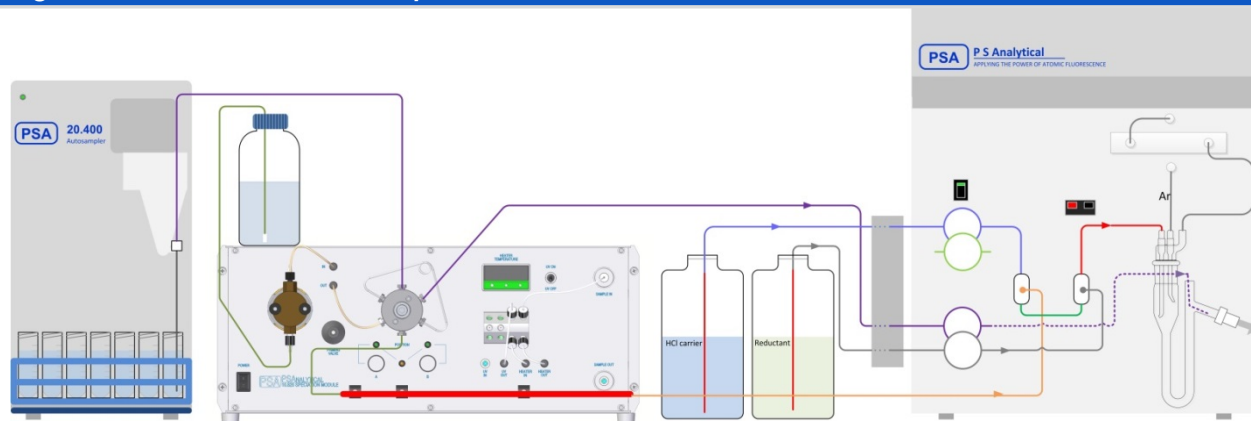
Instrumentation

The determination of arsenic species in FGD wastewater was achieved using a strong anion exchange (SAX) column (PSA C1) with sodium phosphate buffer at a pH of 6.2 (see table 1). Figure 1 shows the instrumental set-up. A 50 μ L injection loop should be used at the 6-port valve to minimize the amount of sample loaded onto the column. Samples should be filtered using a 0.2 micron prior to injection.

Table 1 Chemicals

Solution	Preparation	Pump tubing, pump speed
HCl carrier	250 mL concentrated hydrochloric acid topped up to 1 L with deionised water.	red-red, 50 %
Reductant	Dissolve 4 g NaOH in 900 mL water and add 7 g sodium borohydride. Top up to 1 L.	grey-grey, 100 %
Mobile phase/ sample carrier	20 mM sodium phosphate, pH 6.2	1 mL/min

Figure 1 Instrumental set up



www.psanalytical.com

PSA

P S Analytical

UK: Tel. +44 (0) 1689 891 211

USA: Tel: +1 954 429 1577

SEA: Tel: +61 (0) 428 226 674

E-mail: psa@psanalytical.com

E-mail: usa@psanalytical.com

E-mail: sea@psanalytical.com

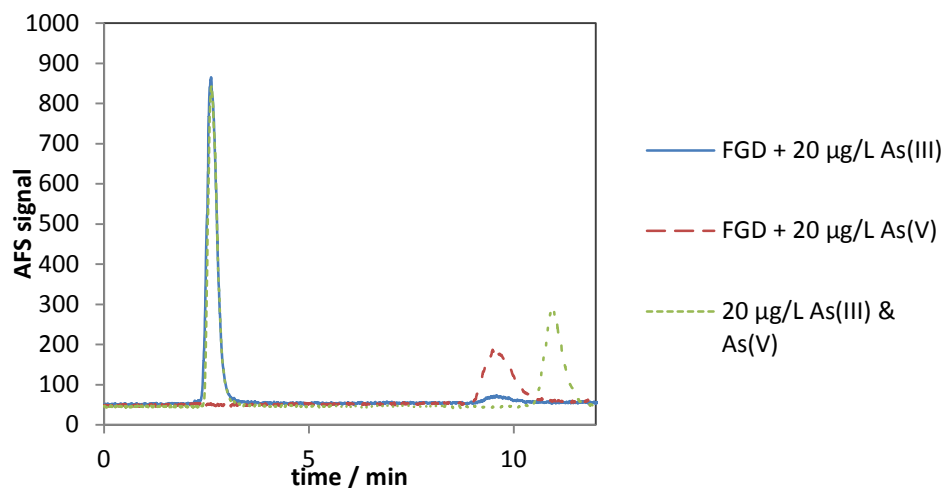
Results

A synthetic FGD wastewater matrix was prepared as outlined in Table 2. Because of the complicated sample matrix the retention time for As(V) was found to shift to a retention time of 9.43 min in the FGD waste water compared to a retention time of 10.87 min in the standard. In principle this can be overcome by diluting the sample with water prior to injection and by using a gradient pump so that the phosphate concentration is increased to 100 mmol l⁻¹. In this case the As(V) will elute earlier and the retention time shift will be minimized

Table 2 Synthetic Wastewater Sample

Component	Target Concentration mg/L	Reagent Used
Ca	2000	CaCl ₂
Mg	1000	MgCl ₂
Na	500	Na ₂ SO ₄
K	100	KBr
Cl	1000	CaCl ₂ /MgCl ₂
SO ₄	1200	Na ₂ SO ₄
Br	2000	KBr

Figure 2 chromatogram of synthetic FGD waste water spiked with As(III) and As(V) compared to a 20 µg/L As(III) and As(V) standard



	Spike recovery (AsIII) / %	Spike recovery (AsV) / %
Synthetic FGD waste water	101.3 ± 5.9 %	100.96 ± 5.6 %

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UK: Tel: +44 (0) 1689 891 211

USA: Tel: +1 954 429 1577

SEA: Tel: +61 (0) 428 226 674

E-mail: psa@psanalytical.com

E-mail: usa@psanalytical.com

E-mail: sea@psanalytical.com

Conclusions

In this application we have demonstrated that accurate determinations of arsenite and arsenate can be achieved in a FGD wastewater matrix. A synthetic wastewater was spiked with a known mass of both species and good recoveries were obtained indicating no matrix interference. A retention time shift was observed for arsenate in the sample matrix due to the high levels of dissolved salt in the sample. This however did not affect the accuracy of the result. A detection limit of 0.3ng/ml and 1.0ng/ml was obtained for arsenite and arsenate respectively



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USA: Tel: +1 954 429 1577

SEA: Tel: +61 (0) 428 226 674

www.psanalytical.com

E-mail: psa@psanalytical.com

E-mail: usa@psanalytical.com

E-mail: sea@psanalytical.com