DIAL
Diagnostic Instrumentation & Analysis Laboratory
Mississippi State, MS 39762-5932

A Fieldable Air-ICP for On-line Monitoring of Toxic Metals

Increasing regulatory demands requiring significant reductions in the emission of hazardous air pollutants have led to the need for techniques capable of providing real-time monitoring of toxic metals in combustion gas streams. These waste streams range from coal-fired boilers, municipal waste combustors to plasma vitrification systems used for the remediation of low level radioactive waste. A lack of a fieldable continuous emission metal monitor (CEM) has been recognized as a significant gap in the available technology. The air-ICP has been designed to fill this gap.

Unlike the standard laboratory ICP, the air-ICP readily tolerates the introduction of molecular gas samples as well as a significantly higher water and particle loading. It has the additional advantage of operating both as argon-air ICP or as an all-air system, that is, operating without the use of argon, a significant reduction in operating costs.

The introduction of air into the plasma results in a totally different spectra from the line spectra seen in an argon ICP. The emission spectra consists molecular bands (e.g. OH, NO, N_2+) in the wavelength regions of interest for metal detection (200 - 350 nm). These increased interferences place a more stringent requirement on the resolution of the

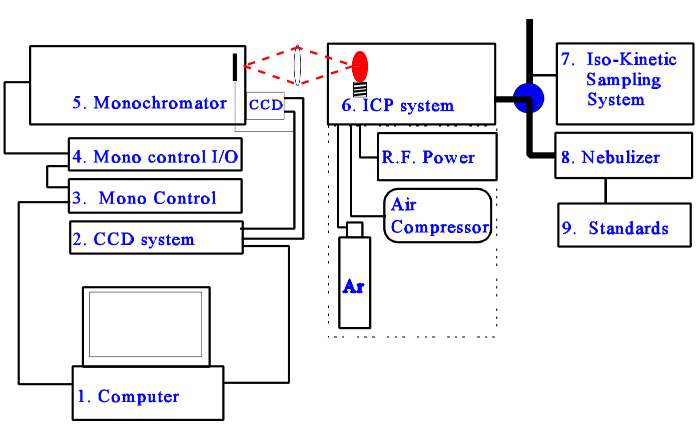

Figure 1. Air-ICP system schematic.

detection system dictating the use of larger instruments. In fact, the presence of numerous possible molecular interferences and the lack of a large database characterizing the ICP emissions expected from molecular offgas systems dictated the use of a sequential detection system rather than, for example, a polychromator. The extractive sampling techniques used by an ICP introduces the sample stream into a controlled environment where matrix effects are minimized and the plasma properties are stabilized. Any matrix effects that remain may be dealt with through the use of internal standards or standard addition methods in the sampling apparatus. Options that are impractical for in-stack methods.

Table 1. Comparison of Air-ICP and Ar/Air-ICP Detection Limits

Element	Air-ICP		Argon/Air-ICP		(Ar/Air)/Air
	ppm	μg/m³	ppm	μg/m³	ratio
Be (313.04)	0.00084	0.2	0.00021	0.049	4
Cd (228.80)	0.03	7.5	0.012	3	2.5
Co (345.32)	0.0072	1.8	0.0033	0.82	2.2
Cr (359.35)	0.0027	0.68	0.0012	0.29	2.3
Hg (253.65)	0.26	66	0.051	13	5.1
Mg (279.55)	0.00056	0.14	0.0003	0.076	1.8
Ni (341.48)	0.0048	1.2	0.0017	0.43	2.8
Pb (405.78)	0.011	2.7	0.011	2.6	1
Sb (206.83)	0.66	170	0.23	59	2.9
Sr (407.77)	0.000035	0.009	0.000023	0.006	1.5

A schematic of the system is shown in Figure 1. One major advantage of our instrument is the substantial reduction achieved in the size of the components. This allows us to interface the instrument directly at the stack, eliminating the difficulties associated with the use of long heated sample lines and the delays inherent in taking the sample back to the lab for analysis.

For argon/air operation, the only modification is an increase in rf power (1.5 - 2 kW). For complete air operation, the ICP is started using argon and progressively switched over to air. A small air compressor is used to supply the gas. The only modifications required to achieve a successful transition are increased gas flow rates (to prevent torch failure) and a small modification to the load coil, the number of turns is increased from 3.2 to 4.7. Calibration is performed using a Cetac (Model U-5000AT) ultrasonic nebulizer.

To further reduce instrument size and overall portability while improving the resolution, we are in the process of upgrading our detection system to the UV-HiRIS, a fiber-optic interferometer, presently under development by Dr. David Baldwin, Ames National Laboratory. This instrument is compact, thermally and vibrationally stable. It uses a combination of an acousto-optic tunable filter and a Fabry-Perot interferometer to provide the resolution of a 1.5 m monochromator in a 10 kg package.

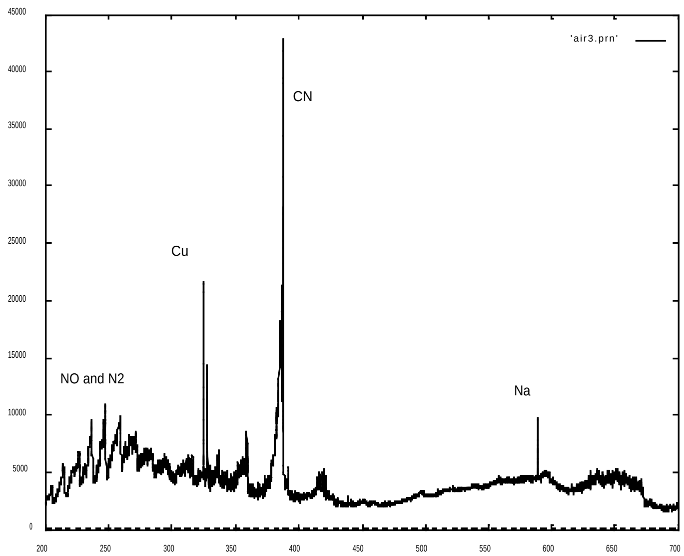

Figure 2. Spectra emissions from off-gas from dc plasma vitrifier.

Figure 2 gives a spectral scan of a gas sample extracted from the offgas stream from the plasma vitrifier at MSU. This sample was taken from the off-gas stream of a plasma vitrifier (transferred dc arc). No material was being vitrified at the time. The scan indicates the presence of significant molecular contribution from NO, N_2, and CN. The sensitivity of the air-ICP is amply demonstrated by the only metal contribution evident, Cu. This is produced by the high current densities present in the arc which constantly erode and vaporize the torch's copper electrode.

In summary, a fieldable air-ICP system has been described. The air-ICP can operate either as an argon/air mixture (sample air-based) or solely on air. The combination of the solid-state air-ICP and the HiRIS spectrometer represent the final phase of size reduction and will reduce the overall size and weight of the complete air-ICP package substantially (from original ~1000 kg, present 250 kg, to ~150 kg) while improving the resolution and therefore detection limits. This instrument has moved the ICP from being an analytical laboratory system to a truly portable instrument capable of providing continuous real-time data for both regulatory compliance and process control. This allows the instrument to sample molecular gas waste streams at the stack on a real-time basis.

Specifications of a Fieldable Air-ICP

- ICP, 27.12 MHz solid-state rf generator and matching unit (SEREN I3500)
    - RF output power 3.5 kW into 50 ohms
    - output impedance 50 ohms
    - connector type HN
    - input power 185-250, 50/60 Hz, single phase, 35 amps
    - Dimensions, full rack, 14" H x 19" W x 21"D
    - Weight approx. 57 kgs
- Ultrasonic nebulizer, CETAC U-5000AT
- Air compressor, small bottle compressed argon.
- 1 m SPEX, Model 1704 monochromator with a Spectrum One CCD detector (to be replaced by HiRIS weight approx. 10 kg).
- Isokinetic Sampler, under development. Weight approx. 20 kg.

Additional information about this system, you may contact Dr. John Plodinec at:

Diagnostic Instrumentation and Analysis Laboratory
Post Office Box MM
Mississippi State, MS 39762-5932

Phone: 662-325-2105
FAX: 662-325-8465
Email: dial@dial.msstate.edu