



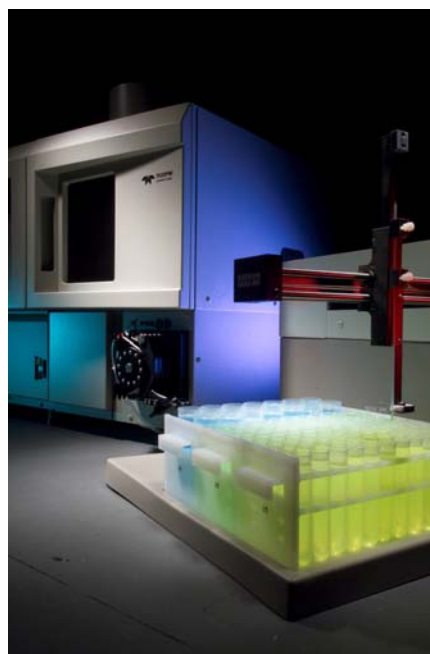
# Determination of the Halogen Elements in the Deep UV Region of the Spectrum by ICP - OES

## Introduction

There are many applications where the ability to measure the halogen elements, at the same time as the metallic elements, is desirable. A few of these include the measurements of Cl and Br in refinery process fluids, the measurement of Cl and Br in waste oils and the determination of Cl<sup>-</sup>, Br<sup>-</sup> and I<sup>-</sup> in food supplements and drinking water.

Historically, the wavelength range of commercially available ICPs has been limited to 165 to 800 nm. This has precluded the determination of the halogens because the primary emission lines for these elements reside between 134 and 154 nm. As a result of this historic limitation, other techniques such as ion chromatography, ion selective electrodes and combustion analysis ( e.g. organic elemental analysis or CHNOX) have been required for the determination of chlorine and bromine in most matrices.

This application note describes a new feature of the Prodigy ICP which permits ppb level determinations of chlorine, bromine and iodine in the deep ultraviolet region of the ICP spectrum. The **Prodigy-H** will be shown to provide the accuracy, precision and detection limits required to measure halogen elements.



*Shown: The Prodigy ICP*

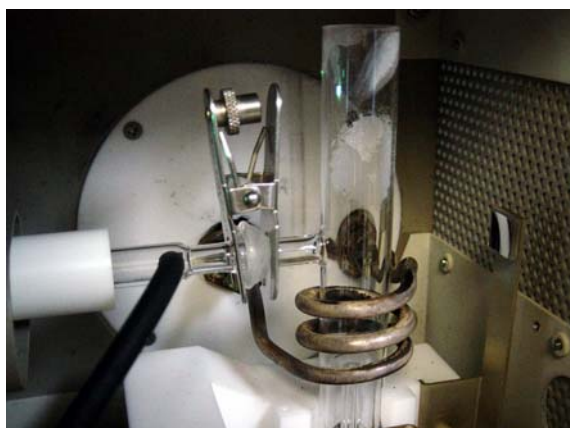
## Experimental

### Instrumental

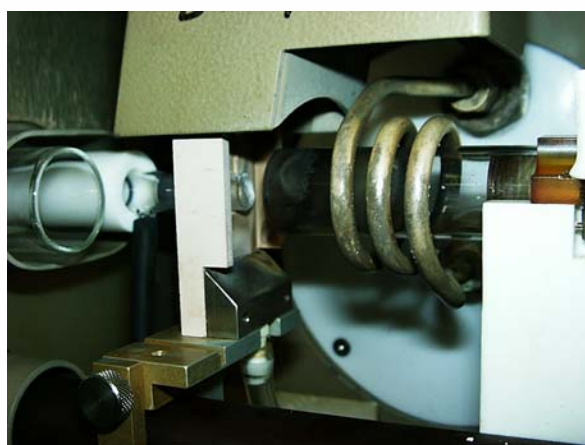
A Teledyne Leeman Labs **Prodigy High Dispersion ICP** equipped with the Halogen Option was used to collect the data for this technical note.

Prodigy-H incorporates several design elements which are critical to effective halogen measurements in the deep UV region of the ICP spectrum. These include:

- **An Ultra-Clean Optical Spectrometer** free from components that can outgas and absorb deep UV photons. Most optical systems contain printed circuit boards and other electronic components which seriously compromise deep UV light transmission.
- **Deep UV Sensitive Detection System.** Prodigy's detection system provides a stable, linear response down to 134 nm. Most solid-state detectors are not sensitive below 160 nm.
- **High Efficiency Purge Path.** With deep UV measurements, it is critical that the light path from the ICP torch to the entrance slit of the spectrometer be fully purged. Even a few millimeters of air will absorb nearly all of the deep UV light. The Radial and Axial Purge Paths used on Prodigy-H are shown in figures 1 and 2 respectively.



*Figure 1. Radial Purge Path*

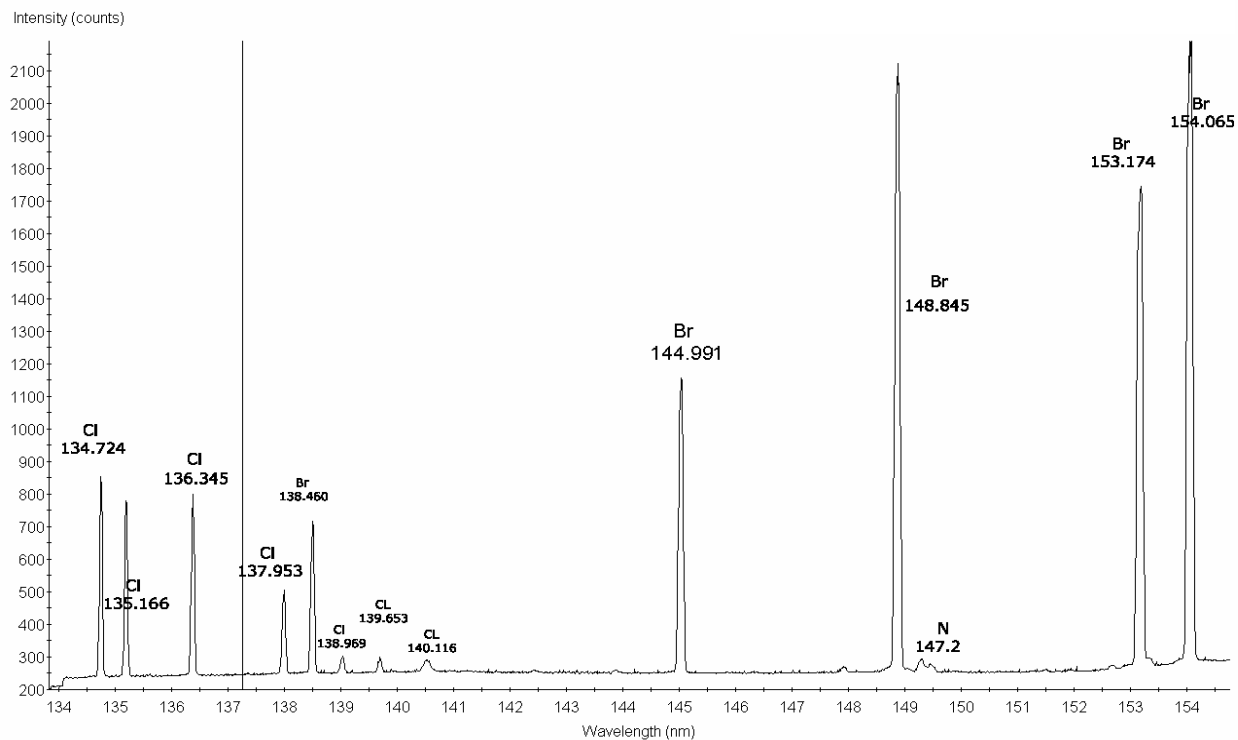


*Figure 2. Axial Purge Path*

Prodigy-H incorporates several other design features that are not only important for the determination of halogens, but are critical to the overall performance of the ICP for traditional metal measurements. A detailed review of these features can be found in the Prodigy brochure as well as at [www.LeemanLabs.com](http://www.LeemanLabs.com).

## Discussion

The spectral region containing the preferred emission wavelengths for Cl, Br are shown in *Figure 3* along with a listing of the preferred wavelengths for Cl, Br and I in *Table 1*.



*Figure 3. Spectrum of Cl and Br Emissions.*

Element	Wavelength, nm
Cl	134.724
Br	154.065
I	183.038

Table 1. Preferred Wavelength for Cl, Br and Iodine

*Figure 4* shows a typical calibration curve for Chlorine in radial viewing mode. The calibration is linear from sub ppm levels up through the top end of this curve at 500 ppm. Higher concentrations were not explored in this work. The calibration data also shows that the deviations between the standard concentrations and the calculated values are small and the %RSDs of the replicate measurements are low.

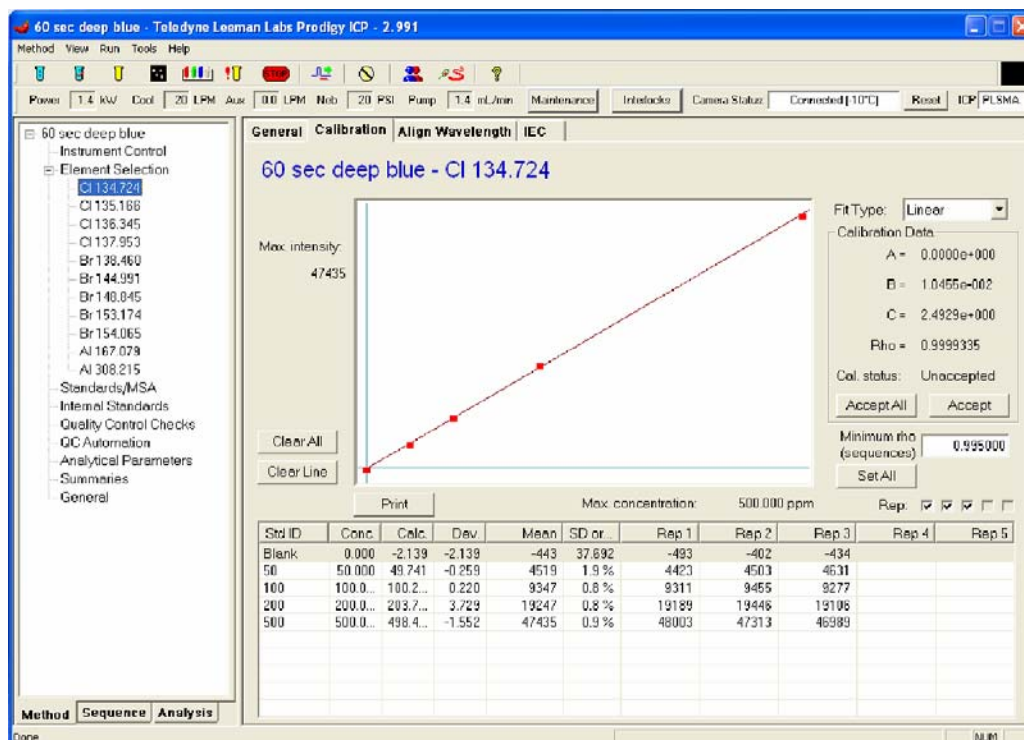


Figure 4. Chlorine Calibration Data at 134.724 nm

In an effort to explore the effectiveness of this new capability two NIST SRMs were analyzed for chlorine. These data are shown in *Table 2*, below.

### Chlorine

	<b>Bovine NIST 1577 A</b>		<b>Oyster Tissue NIST 1566 A</b>
	<b>19.3 ppm</b>		<b>53.7 ppm</b>
	<b>18.4</b>		<b>54.8</b>
	<b>18.5</b>		<b>54.7</b>
	<b>18.4</b>		<b>54.4</b>
	<b>19.1</b>		<b>54.0</b>
	<b>20.0</b>		<b>55.8</b>
<b>Average</b>	<u><b>19.0</b></u>	<b>Average</b>	<u><b>54.6</b></u>
<b>factor</b>	0.75 gms/100 ml 133.33		0.67 gms/100 ml 142.86
<b>Measured Value</b>	<b>2533 ug/gm</b>	<b>Measured Value</b>	<b>7800 ug/gm</b>
<b>Cert Value</b>	<b>2800</b>	<b>Cert Value</b>	<b>8290</b>
<b>% Recovery</b>	<b>90.48</b>	<b>% Recovery</b>	<b>94.09</b>

Table 2.



## Conclusions

Prodigy's Halogen Option provides the analyst access to analytes that have historically required alternative analytical techniques such as ion chromatography or ion selective electrodes. This technical note demonstrates Prodigy-H's fundamental capability to measure the halogen elements in the deep UV region of the spectrum as well as the linearity, accuracy and precision that can be expected from these measurements. The ability to measure Chlorine, Bromine and Iodine at sub ppm levels along with all of the conventional metals will help to improve the efficiency of today's laboratories.