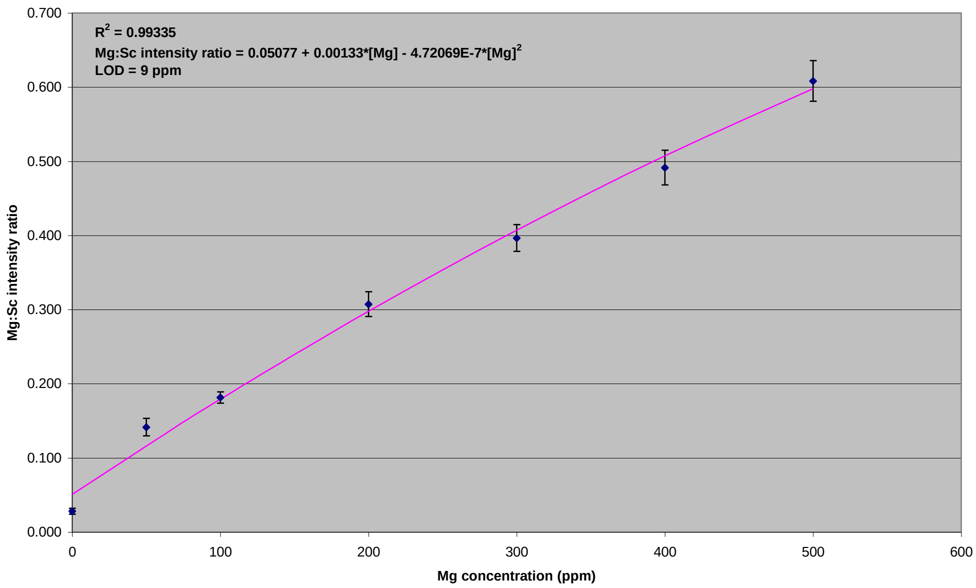
An experimental investigation into the feasibility of using LIBS to determine the Mg concentration of Highly-Active Liquor (HAL)

An experimental investigation was conducted to assess the feasibility of using an ST-LIBS instrument and a FO-LIBS instrument to remotely determine the Mg concentration of Highly-Active Liquor (HAL) over the Mg concentration range of 50 ppm - 500 ppm. Experimental data obtained using a number of simulant (inactive) liquid samples provided by BNFL were used to estimate the measurement precision and limit of detection (LOD) of a LIBS instrument. Coupling of the laser radiation with the liquid samples was found to be a key factor affecting the measurement precision. Better coupling was achieved using 355 nm laser radiation (frequency-tripled Nd:YAG laser) compared to using 1064 nm (fundamental wavelength of a Nd:YAG laser). Using an ST-LIBS instrument fitted with a frequency-tripled Nd: YAG laser (355 nm), an LOD of ~10 ppm and an RSD of ~10% were obtained. It was found that a FO-LIBS instrument utilising 1064 nm laser radiation was not able to produce a sufficiently intense and stable plasma on the surface of the liquid. The available 355 nm laser was not suitable for coupling to an optical fibre and so it was not possible to investigate the feasibility of a FO-LIBS instrument incorporating a 355 nm laser.



Calibration plot for Mg in HAL using Sc as internal standard.
The error bars represent ± 1 standard deviation.

This work demonstrated that LIBS is able to determine the Mg concentration in simulated HAL solutions over the approximate Mg concentration range 50 ppm to 500 ppm. Measurement precision appears to be largely dependent on the efficiency of the coupling of the laser radiation with the liquid sample. The improved coupling of the 355 nm laser light with the liquid samples increased the measurement precision considerably from that obtained using the 1064 nm laser. The better coupling also allowed lower laser pulse energies to be used which helped to reduce disturbance to the liquid (e.g. splashing) which in turn allows higher pulse repetition rates to be utilised. To improve coupling efficiency further, an alternative laser could be utilised which produces an output that matches the absorption band in nitric acid / HAL at ~ 300 nm (e.g. an XeCl laser operating at 308 nm).

From a practical point of view, it would be highly desirable to use an optical fibre to deliver the laser radiation to, and collect the plasma light from, a sample located inside an HA cell. A fibre-optic probe type LIBS instrument, however, has a number of disadvantages. These include i) a limited ability to produce a highly focussed (i.e. small spot size) on the sample, ii) the limited laser pulse energy that may be transmitted, ii) a significantly reduced stand-off distance to the sample (i.e. the fibre-optic probe will need to be close to the sample, possibly less than 100 mm), and the limited transmission of UV light which may

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compromise the detection efficiency of the Mg ionic emission line at ~280 nm). None of these disadvantages are considered to be such that the use of a fibre-optic probe LIBS instrument for this application can be ruled out at this time.

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