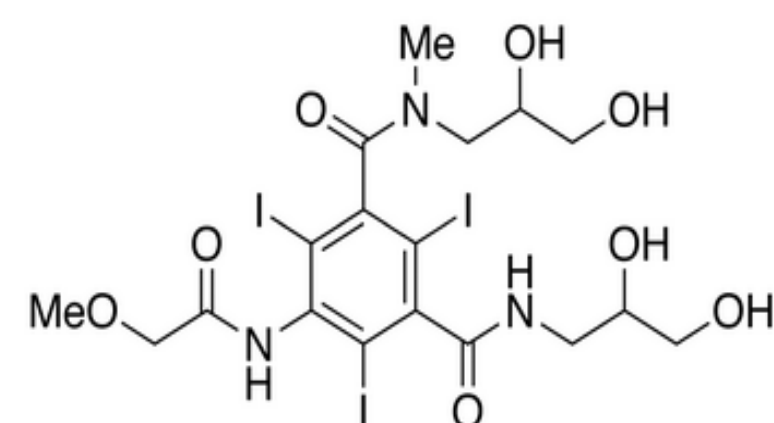


## Introduction

Iopromide is an iodinated contrast medium (ICM) which is used to image internal body organs and blood vessels by either X-ray or computerized tomography (CT) scans. Iopromide is generally given to patients in g/L concentrations and is excreted within 24 hours in the patient's urine[1]. The structure of iopromide is shown below:



The presence of iopromide in surface waters and wastewaters has been widely reported as ranging from several ng/L to as much as 10 µg/L in sewage treatment plant effluents [2-4]. Furthermore, ICMs are known to be resistant to sewage treatment and studies have shown they are relatively poorly removed by conventional treatment processes [2, 5-7]. Due to its presence and environmental persistence, it has also been suggested that iopromide be used as a potential indicator of wastewater contamination [8].

Most analytical methods developed for iopromide and other ICMs involve the use of LC coupled to a mass spectrometer, generally a triple quadrupole mass spectrometer [9-13]. This poster describes the optimized conditions for sensitive and reproducible analysis of sub-ppb levels of iopromide in water extracts, using an Agilent 1260 Bio-inert LC coupled to an Agilent 7700x ICP-MS. With the use of a 500 µL injection volume we have established a lower method reporting limit of 0.1 ppb for iopromide in our diluted methanol extracts in our assay; this corresponds to a lower MRL of 2 ppt in our environmental water samples.

## Instrumentation

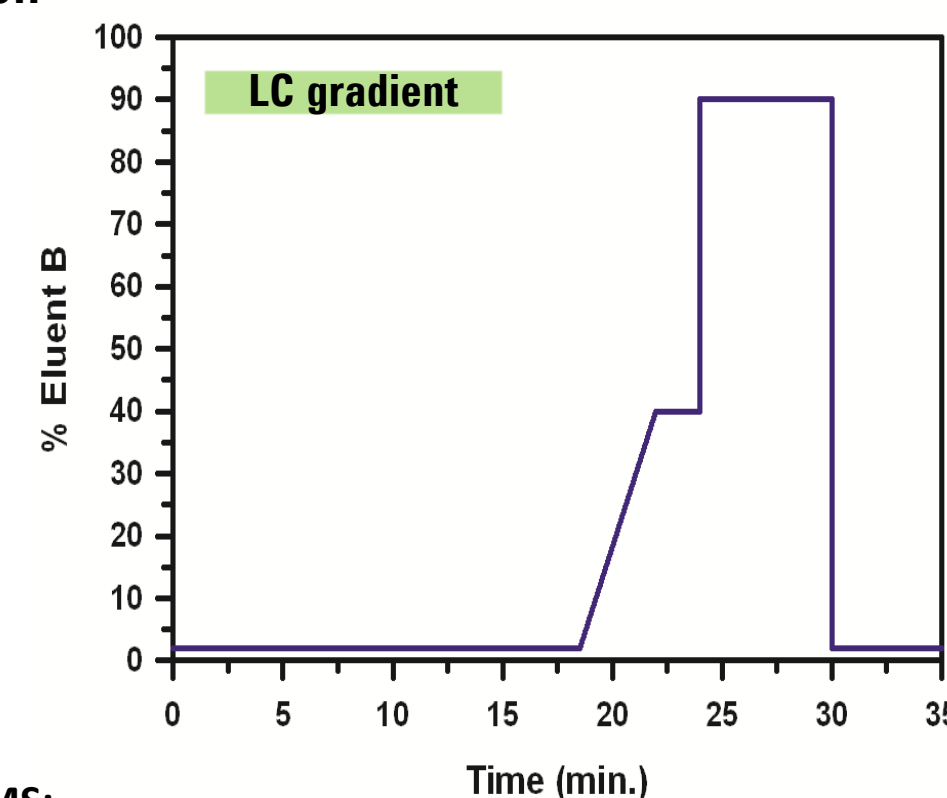
### Sample preparation

Water samples were filtered through 0.7 µm filters and then extracted using an automated SPE system. 200 mg hydrophilic-lipophilic balance (HLB) cartridges were first preconditioned with 5 mL of MTBE, followed by 5 mL of methanol and 5 mL of HPLC grade water. 1.0 L of each sample was then loaded onto a cartridge at a flow rate of 15 mL/min, after which the cartridges were rinsed with HPLC grade water followed by drying with nitrogen gas for 30 minutes. All adsorbed analytes were then eluted into 15 mL graduated conical tubes with 5 mL of methanol followed by 5 mL of 10/90 (v/v) methanol/MTBE solution. The eluent was then evaporated to a total volume less than 100 µL under flowing nitrogen followed by reconstitution to 1.0 mL total volume using methanol. 50 µL of this extract was then diluted with 950 µL of HPLC grade water to give the final extract used for IC-ICP-MS analysis.

### Instrument Configuration and Conditions

#### Liquid Chromatograph:

- Agilent 1260 Bio-inert quaternary pump
- Agilent 1260 Bio-inert HiP autosampler equipped with 2.0 mL loop, 30 second H<sub>2</sub>O needle wash, 500 µL sample injection volume
- Dionex AG-16 4x50 mm guard column & Dionex AS-16 4x250 mm analytical column
- 1.0 mL/min flow rate; Eluent A = MQ H<sub>2</sub>O, Eluent B = 100 mM NaOH



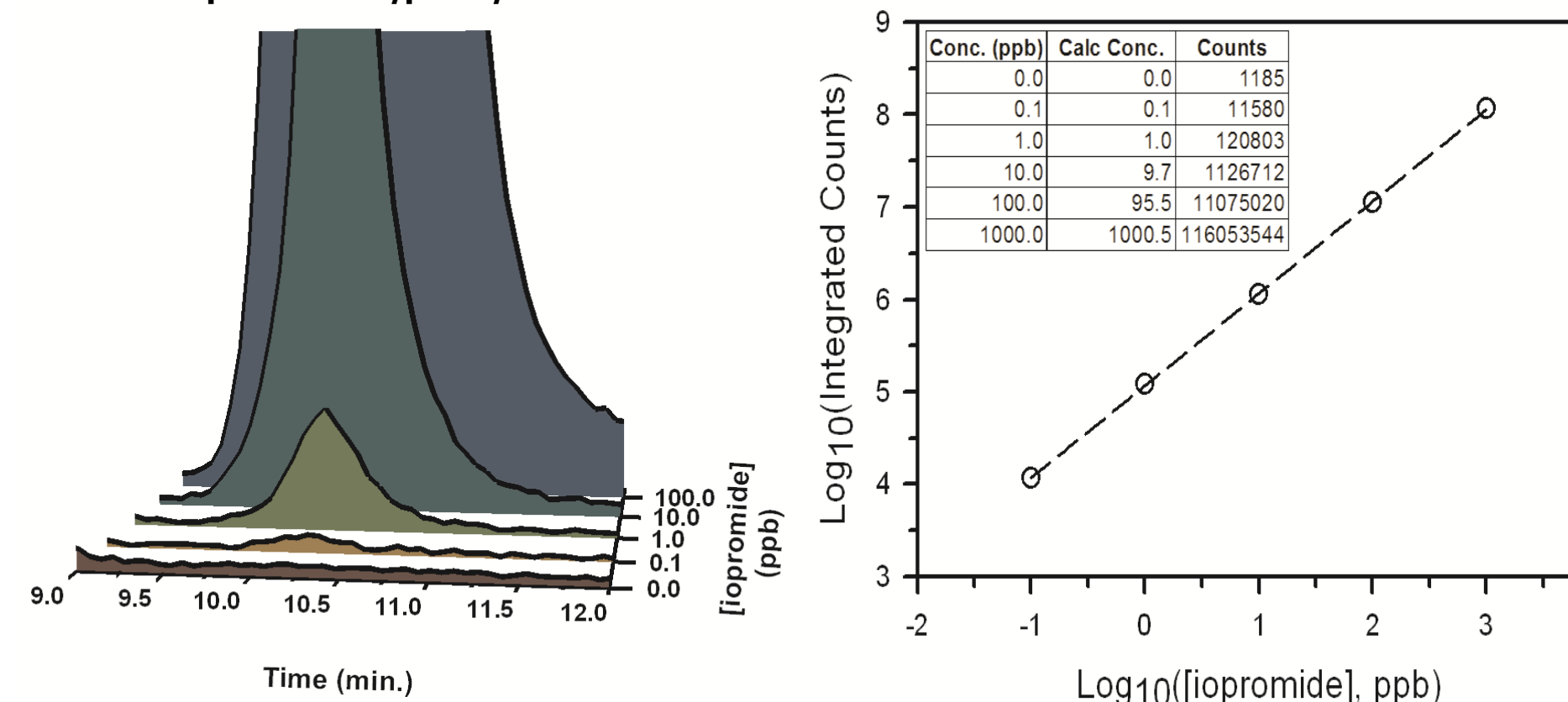
#### ICP-MS:

- Agilent 7700x ICP-MS
- HMI Sample Introduction; 0.6 L/min dilution gas, 0.5 L/min carrier gas, 9.0 mm sample depth
- Helium collision gas mode; 3.5 mL/min He flow
- 2 second integration time for <sup>127</sup>I in TRA mode
- Calibration standards prepared in MQ H<sub>2</sub>O with [iopromide] of 0.0, 0.1, 1.0, 10.0, 100.0, and 1000.0 ppb

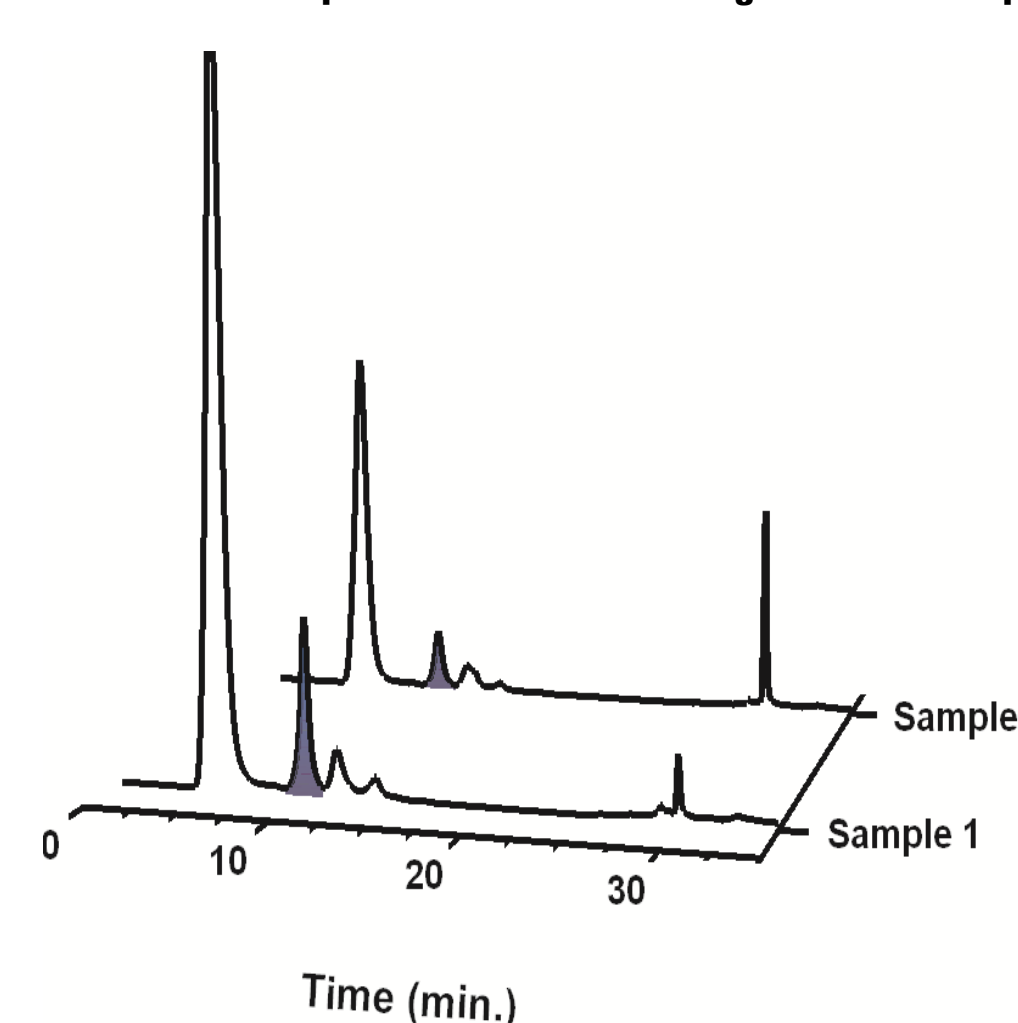
## Results and Discussion

### Analysis of Calibration Standards & Samples

Using the LC gradient described here, iopromide elutes from the column at 10.1 minutes, and we have successfully quantitated iopromide in all non-zero standards we have prepared for this study. Our calibration curve is linear from 0.1 ppb to 1000.0 ppb - this range covers the range in which iopromide is typically encountered in environmental waters.



In addition to iopromide, several other iodine containing compounds were detected in our chromatograms, as shown below. These unknown species can be quantified based on their iodine content, using compound-independent calibrations (CIC), where the iodine content of an unknown compound is calibrated using the iodine response for a known compound, in this case iopromide. ICP-MS is ideally suited for analysis using CIC, as the high temperature ICP ion source ensures that the elemental response of the target element (iodine in this case) is essentially independent of the compound in which the target element is present.



### Summary of Results

The analysis of our sample extracts indicated concentrations of iopromide in our extracts ranging from sub-ppb up to approximately 250 ppb; these values correspond to concentrations of iopromide in our environmental waters ranging from 4 ppt to 5 ppb. The wide dynamic range of our ICP-MS assay allowed for all samples to be analyzed with one single calibration curve.

Although this work focused on iopromide, we were also able to detect the presence of other iodinated organics present in our sample extracts, as evidenced by the peaks in the EIC for m/z 127 (I<sup>+</sup>). The iodine content of these peaks can be quantitated using compound-independent calibrations (CICs), since the instrumental response to I essentially does not depend on the organic nature of the iodinated species entering the plasma.

Our future work on this topic will include expanding our method to quantitate more ICMs in environmental waters and to examine the fate of these species during oxidative water treatment processes.

## Conclusions

We have successfully quantified iopromide in a series of environmental water extracts using an Agilent 1260 LC coupled to an Agilent 7700x ICP-MS. This experimental arrangement allowed for us to establish an analytical method with a lower method reporting limit (MRL) of 0.1 ppb iopromide in extracts prepared via automated SPE. The use of the HMI interface allowed for an extended (>24 hr.) analysis to be completed (using non-volatile eluents) with minimal matrix deposition on the interface cones, and the use of He collision gas mode provides effective removal of polyatomic interferences. Not only does our work confirm and quantitate the presence of iopromide in these environmental samples, it indicates the presence of other iodinated organic compounds in these samples that are likely from anthropogenic sources and may prove to be biologically active.

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## For More Information

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