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Direct Injection Nebulization for Sample Introduction in
Inductively Coupled Plasma-Mass Spectrometry**

by

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A COMPARISON OF CONTINUOUS PNEUMATIC NEBULIZATION AND FLOW INJECTION-DIRECT INJECTION NEBULIZATION FOR SAMPLE INTRODUCTION IN INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY

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ABSTRACT

Samples containing Ni, Cd, Pb, and U were analyzed eighteen times over a two-month period using inductively coupled plasma-mass spectrometry (ICP-MS). Sample introduction was accomplished by either flow injection-direct injection nebulization (FI-DIN) or continuous pneumatic nebulization (CPN). Using comparable instrumental measurement procedures, FI-DIN analyses were 33% faster and generated 52% less waste than CPN analyses. Instrumental limits of detection obtained with FI-DIN and CPN were comparable but not equivalent (except in the case of Pb) because of nebulizer-related differences in sensitivity (i.e., signal per unit analyte concentration) and background. Substantial and statistically significant differences were found between FI-DIN and CPN Ni determinations, and in the case of laboratory waste samples, there were also small but statistically significant differences between Cd determinations. These small (2 to 3%) differences were not related to polyatomic ion interference (e.g., $^{95}\text{Mo}^{16}\text{O}^+$), but in light of the time and waste savings to be realized, they should not preclude the use of FI-DIN in place of CPN for determination of Cd, Pb, U, and similar elements present at trace concentrations.

KEYWORDS

ICP-MS; Sample Introduction; Flow Injection; Analytical Waste Reduction

INTRODUCTION

Since its commercial introduction in 1985, inductively coupled plasma-mass spectrometry (ICP-MS) has matured rapidly; so much so that the technique is now employed routinely in many laboratories for the determination of trace elements in industrial, biomedical, and terrestrial samples.¹ The technique's growth has been largely due to the intrinsic strengths of ICP-MS, e.g., instrument detection limits of $\mu\text{g L}^{-1}$ or better, but even as these strengths have been exploited by many, some prominent weaknesses remain. One such weakness, not solely restricted to ICP-MS, is sample introduction inefficiency. One group of analytical chemists have, in the past, referred to sample introduction as the "Achilles' heel" of atomic spectroscopy,² and for that reason, many investigators have proposed and tested a variety of "ideal" sample introduction devices³⁻⁶ which have achieved varying degrees of success.

Despite this research, continuous pneumatic nebulization (CPN) of dissolved analytes remains very popular in ICP spectrometry because it works well in a broad range of applications.⁷⁻⁸ However, normal pneumatic nebulizers, e.g., glass concentric types, utilize only 1 to 10% of the sample uptake. The remaining portion of the consumed sample (primarily water and mineral acid) goes directly to laboratory waste. This creates a secondary waste stream that would be considered, at minimum, corrosive by regulatory standards, and in the worst case, could also be toxic and/or radioactive. Although the worst case is unlikely to be encountered in most laboratories, it is of special significance and concern to the United States Department of

Energy (DOE) and its contractors, most of whom analyze materials that are hazardous and radioactive, thereby creating analysis waste with the same characteristics.

Given the special circumstances encountered by the DOE, and in light of the many analyses performed per annum in support of DOE operations (over 400,000 for environmental management alone),⁹ elimination or reduction of the analytical waste stream would significantly reduce waste management costs, thereby liberating resources (staff and financial) for more pressing concerns. For this reason, we have investigated methods for eliminating waste from ICP spectrochemical measurements. Until recently, this objective would have had little chance of success, but development of the direct injection nebulizer (DIN)^{10,11} and its combination with flow injection (FI) have provided a new and singular opportunity for waste reduction. We have studied the performance and waste-reducing characteristics of the FI-DIN system by direct comparison against CPN, and we describe the conduct and results of this investigation in the subsequent sections of this report.

EXPERIMENTAL

Apparatus. A PlasmaQuad 2 ICP-mass spectrometer (Fisons Instruments, Winsford, UK) with a high-performance interface and extended dynamic range was used in this work. The quadrupole mass analyzer was operated exclusively in the multiple ion monitoring (i.e., "peak hopping") mode, and ion signals were detected by pulse counting. Other instrument operating conditions are listed in Table 1.

"Normal" sample introduction was studied by continuously delivering sample solutions to a V-groove pneumatic nebulizer (provided by Fisons). Sample delivery was accomplished with

an autosampler and peristaltic pump (Model 222 and Minipuls 3, respectively, Gilson Medical Electronics, Middleton, WI). A jacketed Scott double-pass spray chamber (provided by Fisons), cooled to 278 K with recirculating water, was combined with the V-groove nebulizer to desolvate the aerosol before injection into the plasma. Sample solutions were also introduced to the ICP-mass spectrometer through a direct injection nebulizer system (Microneb 2000, CETAC Technologies, Omaha, NE) driven by a dual-piston HPLC pump (also supplied by CETAC). Each sample was loaded into the flow injection loop using the Gilson autosampler and peristaltic pump, and all replicate integrations were obtained with a single 500 μL aliquot of sample. An 18 MOhm-cm water carrier (Nanopure water polisher, Barnstead/ Thermolyne, Dubuque, IA) was used with the FI system.

Standards and Samples. Nickel, cadmium, lead, and uranium were selected as analytes for this study on the basis of regulatory interest, atomic number, isotopic composition, and anticipated blank limitations. Multielement calibration standards containing these analytes were prepared by mixing and diluting single-element solution standards (High Purity Standards, Charleston, SC) such that the calibration standards contained 0.5 or 10 $\mu\text{g L}^{-1}$ of each analyte, 5 $\mu\text{g L}^{-1}$ each of Rh and Lu, and 2% v/v nitric acid. A diluent blank containing 5 $\mu\text{g L}^{-1}$ each of Rh and Lu and 2% v/v nitric acid was also prepared. All solutions were prepared in acid-cleaned class A volumetric glassware by using a calibrated electronic digital pipette (EDP Plus, Rainin Instruments, Woburn, MA), high-purity nitric acid (Optima grade, Fisher Scientific, Pittsburgh, PA), and 18 MOhm-cm water.

Standard Reference Material (SRM) 1643c ("Trace Elements in Water," National Institute of Standards and Technology, Gaithersburg, MD) and a commercial calibration verification

solution (ICPMS-2A, Spex Industries, Edison, NJ) were used as test samples to compare CPN and FI-DIN determinations against certificate analyte concentrations. Two laboratory waste solutions (obtained from our ICP-emission spectroscopy laboratory and designated as Waste #37 and Waste #40) were used as test samples to compare CPN and FI-DIN analysis of "real samples." All four samples were spiked with Rh and Lu and diluted, per the previously noted specifications, either 10-fold (SRM 1643c), 100-fold (Waste #40), 500-fold (Waste #37), or 1000-fold (ICPMS-2A) such that ion counting rates were below the upper limit of the detection system. Each diluted sample contained $5 \mu\text{g L}^{-1}$ each of Rh and Lu, as well as 2% v/v nitric acid.

Analysis Procedure. The ICP-mass spectrometer was started per the manufacturer's instructions and allowed to warm up for 30 minutes to one hour. After the warmup period, a test solution containing $10 \mu\text{g L}^{-1}$ each of Li, Mg, Co, Y, In, La, Lu, Pb, and U was nebulized to allow optimization of instrument sensitivity and short-term measurement reproducibility. The test solution was also used to calibrate the instrumental mass-to-charge ratio (m/z) scale.

Once sensitivity, reproducibility, and mass scale calibration were satisfactory, the calibration and analysis sequence listed in Table 2 was initiated. Signals for $^{58,60}\text{Ni}$, ^{103}Rh , ^{111}Cd , ^{175}Lu , $^{206,208}\text{Pb}$, and ^{238}U in each solution were integrated five times in succession at 30 s per replicate, where Rh was used as an internal standard for Ni and Cd, and Lu was used as an internal standard for Pb and U. The CPN and FI systems were rinsed thoroughly with 10% v/v high-purity nitric acid between analyses.

Data Analysis. For each sample introduction system, the analysis procedure was repeated six times within a two month period. Eighteen analyses of the post-calibration blanks and test samples were completed. The analysis data were pooled, and a mean and standard deviation

were calculated for each determination. The standard deviation of each pooled blank determination was multiplied by three to obtain instrumental limits of detection. In the case of the blanks and test samples, Student's t^{12} was calculated to facilitate detection of significant differences between FI-DIN determinations, CPN determinations, and certificate analyte concentrations. The 95% confidence interval was used in all statistical comparisons.

RESULTS AND DISCUSSION

The principal objective of this study was to compare important analytical method selection criteria (e.g., precision) for CPN and FI-DIN sample introduction. These criteria, which are discussed below, can be difficult to determine as they are influenced by many experimental factors. However, use of identical calibration and analysis procedures, combined with the consistent use of replicate integrations and integration times, facilitated comparison by eliminating variables that were not directly related to the properties of the sample introduction systems. The effects of other factors, e.g., day-to-day variations in instrument sensitivity, were mitigated by the use of internal standardization.

Waste Volume and Analysis Time. Per sample waste volume was calculated by dividing the volume of waste collected by the number of solutions analyzed in the course of a calibration and analysis sequence. In the case of CPN, the waste (unconsumed sample and rinsate) was collected from the spray chamber drain; in the case of FI-DIN, the waste was collected from overflow of the FI manifold. By this method, we found that 7.1 mL of waste was produced per sample using CPN, while only 3.4 mL of waste was produced per sample using FI-DIN. Much of the FI-DIN waste was due to overflow and the volume of tubing between the sample and the

injection loop; however, ~ 0.5 mL of the waste volume was due to control logic that allowed the peristaltic pump to operate while the FI valve was in the inject state (thereby sending unconsumed sample directly to waste). With different EPROMs (Erasable, Programmable Read-Only Memory) in the DIN control module, this feature can be disabled.

Taking into account the inefficiency of the control logic, we attribute the waste savings resulting from FI-DIN introduction to two factors: (i) the DIN consumed 2.7 mL less solution and generated no waste over the 2.85 min required for signal integration and (ii) use of the flow injection manifold eliminated the need to fill and rinse the volume of tubing between the sample and nebulizer, reducing waste by ~ 1 mL. Further opportunities for waste reduction, e.g., reducing dead volume or using smaller injections, were certainly available but were not studied for this report.

Per sample analysis time was computed by dividing the time required to complete the calibration and analysis sequence by the number of solutions analyzed. By this method, we found that a CPN analysis required 7.5 min per sample, while an FI-DIN analysis required only 5.0 min per sample. The 2.5 min savings in analysis time was due solely to reduced rinseout time, which is less in FI-DIN because only the injection loop requires rinsing (i.e., the nebulizer is rinsed constantly by the carrier solution). Further time savings were possible (e.g., by using a smaller injection loop), but these would be modest relative to the savings realized here since 3.5 min per sample was required for FI-DIN signal stabilization and integration, independent of injection volume.

Limits of Detection and Sensitivity. The instrumental detection limits (IDLs) achieved using FI-DIN and CPN, as listed in Table 3, appear similar despite the fact that the background

equivalent concentrations (BECs) differed significantly at the 95% confidence interval. However, with the exception of Pb, F-tests¹² indicated statistically significant differences in the standard deviations of the FI-DIN and CPN BEC determinations. This finding implies significant differences as well in the IDLs for Ni, Cd, and U. In light of these differences, it was concluded that CPN yielded better detection limits for Cd, which is not generally prone to memory effects (i.e., sample-to-sample carryover), while FI-DIN yielded better detection limits for U (which is refractory and, thus, prone to memory effects). These observations are consistent with other reports, suggesting that DIN is less prone to memory effects than CPN.¹⁰

The systematic differences shown in Table 3 for Ni BEC and IDL were also important, but in this case, the differences were probably not due to memory effects. Increased solvent loading (such as that characteristic of the DIN)¹⁰ can raise plasma potential^{13,14} and increase the exposure of the Ni sampling interface to acid vapors. Both of these factors will accelerate interface erosion, and accelerated erosion would elevate the Ni BEC as shown. This conclusion is confirmed by our observation that samplers used with the DIN tend to have shorter lifetimes than those used with normal pneumatic nebulizers. It is, however, interesting to note that higher erosion rates may yield some analytical advantages, e.g., less refractory element memory, by reducing the rate at which salts condense upon the sampling interface.¹⁵

Two other factors that influence detection limits (and subsequent comparisons) are absolute sensitivity, i.e., the slope of an instrumental calibration curve (signal per unit analyte concentration), and relative sensitivity, i.e., the signal per mole of an analyte isotope, normalized to that of a reference isotope. As absolute sensitivity decreases, BEC and IDL increase so long as the background signal remains constant, and when relative sensitivity varies, the BEC and IDL

for an analyte will change relative to that of another analyte, even if the absolute sensitivity for one remains constant.

Absolute sensitivity data obtained from FI-DIN and CPN determinations of the analytes are given in Table 4. Relative sensitivity data for repeated analysis of the instrument tuning solution are presented in Figure 1. Figure 1 clearly indicates little difference in relative sensitivity between the two nebulizers except, perhaps, at low atomic numbers (e.g., Li and Mg). As such, relative sensitivity changes could not have influenced the differences in BEC and IDL. However, statistically significant differences were observed between FI-DIN and CPN absolute sensitivities for all analytes except Ni. This difference ($\text{CPN/FI-DIN} \approx 1.5$), although significant, was not sufficient to explain the changes in BEC and IDL. As such, we concluded that the observed changes were due to a combination of sensitivity differences and background variations, both related to the choice of nebulizer.

Accuracy and Precision. Sample analyses obtained using CPN and FI-DIN are compared in Tables 5 through 8. The data in Table 5 were collected to establish the accuracy of the instrument calibration procedure. These data indicate statistically significant biases in the determinations of Ni and U, although the bias in the U determination was small ($\sim 2\%$) at the "as-analyzed" U concentration (i.e., $10.07 \mu\text{g L}^{-1}$ after sample dilution). The FI-DIN and CPN determinations of Ni differed significantly, and F-tests indicated significant differences in the precision of the Ni and Pb determinations. There was also a small but statistically significant difference ($\sim 2\%$) between the certificate Cd concentration and the Cd determination by CPN; however, the difference between the FI-DIN and CPN determinations was not significant.

The data in Table 6 were collected to document the accuracy of the instrumental

determinations by using a standard reference material. Significant differences between the FI-DIN and CPN Ni determinations were found, as were significant differences in the precision of the Ni and Pb determinations. Relative to the certificate concentrations, small but significant biases (~ 5%) were found in the determinations of Cd and Pb (U was not certified or detected); however, there were no significant differences between the FI-DIN and CPN determinations of these analytes. In light of the calibration data (Table 5), this may suggest that other factors (e.g., evaporation) contributed to the positive biases. This suggestion is especially reasonable when the age of the standard (over 2 years) is taken into account.

The data in Tables 7 and 8 were collected to document the comparison between FI-DIN and CPN analyses of "real" waste samples. These data indicate statistically significant differences between the FI-DIN and CPN determinations of Ni and Cd, though the difference in the Cd determinations was fairly small (~ 2%). There were also significant differences in the precision of Ni, Pb (Waste #37 only), and U (Waste #40 only) determinations, though the coefficient of variation for the Pb and U determinations was, at worst, only 6%. The discrepancy in the Ni determinations was expected, especially at the "as-analyzed" concentrations of Ni (which were close to the Ni IDL). Examination of the sample spectra ruled out the possibility of spectral interference (e.g., $^{95}\text{Mo}^{16}\text{O}^+$) on ^{111}Cd . Thus, other factors such as matrix effects or systematic bias (see Table 5 and the associated discussion) may have been the source of the small Cd discrepancy.

CONCLUSIONS

The data we have collected indicate that significant reductions in waste volume and

analysis time were realized when FI-DIN was used in place of CPN for ICP-MS sample introduction. The FI-DIN and CPN determinations of Cd, Pb, and U compared reasonably well with available certificate analyte concentrations, but more significantly, differences between FI-DIN and CPN ICP-MS determinations of Cd, Pb and U were small or statistically insignificant. Other figures of merit (detection limit and precision) were also very similar for the two sample introduction techniques, and these data, considered together, indicate that FI-DIN could be used interchangeably with CPN for elemental analysis by ICP-MS.

Further reductions in waste and analysis time could be achieved by means of different flow injection protocols; for instance, it was possible, based on the solution delivery rate and per sample analysis time, to reduce the sample loop size to 200 μ L. The logic of the peristaltic pump control circuit could have also been changed such that the pump was off when the FI manifold was in the "inject" state. Neither of these changes would have affected the figures of merit presented herein, but they would certainly have decreased waste volume and analysis time per sample. Use of multiple small injections per sample (e.g., 50 μ L per integration) could have also reduced waste and analysis time, but given the higher degree of signal transience, this could have compromised figures of merit. We chose not to accept such compromises in this study, but alternative FI strategies are certainly worthy of study, especially by those who characterize acutely hazardous materials.

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Table 1. Typical Operating Conditions

	CPN	FI-DIN
Loop Fill Period (s)		30
Loop Fill Rate (mL min ⁻¹)		3.0
Loop Inject Period (s)		225
Uptake Delay (s)	75	45
Rinse Delay (s)	180	30
Liquid Delivery Rate (mL min ⁻¹)	1.0	0.05
<u>Argon Flow Rates (L min⁻¹):</u>		
Coolant	13.6	13.0
Auxiliary	1.55	0.6
Nebulizer	0.85	0.2
DIN Argon Pressure (kPa)		483
Plasma Forward Power (kW)	1.35 ^a	
<u>Ion Optical Settings (V):</u>		
Extractor	-190	
Collector	10	
L1	-10	
L2	-30	
L3	0	
L4	-40	
Pole Bias	1	
Detector	-2300	

^a This entry and those subsequent were used with both nebulizer types.

Table 2. Instrumental Calibration and Sample Analysis Sequence

Sample ID	Sample Type
2% nitric acid	calibration blank
0.5 $\mu\text{g L}^{-1}$ Ni, Cd, Pb, and U	blank subtracted standard
10 $\mu\text{g L}^{-1}$ Ni, Cd, Pb, and U	blank subtracted standard
2% nitric acid	memory check
2% nitric acid (3X)	post-calibration blank
1:10 SRM 1643c (3X)	blank subtracted sample
1:1000 ICPMS-2A (3X)	blank subtracted sample
2% nitric acid	memory check/blank
1:500 Waste #37 (3X)	blank subtracted sample
1:100 Waste #40 (3X)	blank subtracted sample

Table 3. Comparison of Background Concentrations and Detection Limits

Element	<u>Background Concentration ($\mu\text{g L}^{-1}$)</u>		<u>Instrument Detection Limit ($\mu\text{g L}^{-1}$)</u>	
	FI-DIN	CPN	FI-DIN	CPN
Ni ^a	3.2	0.84	1	0.5
Cd	0.027	0.012	0.04	0.02
Pb ^b	0.073	0.11	0.1	0.2
U	0.003	0.008	0.01	0.02

^a Based on ⁵⁸Ni.^b Based on ²⁰⁸Pb.

Table 4. Comparison of Absolute Instrument Sensitivity

Element	<u>Sensitivity (ions s⁻¹ per µg L⁻¹)</u> ^a	
	FI-DIN	CPN
Ni ^b	6700 ± 4200	10400 ± 2300
Cd	2190 ± 760	3200 ± 600
Pb ^c	8700 ± 1900	11900 ± 1400
U	10800 ± 2200	16200 ± 1300

^a Mean and standard deviation of 6 determinations.^b Based on ⁵⁸Ni.^c Based on ²⁰⁸Pb.

Table 5. ICP-MS Analysis of Calibration Verification Standard

Element	<u>Analyte Concentration (mg L⁻¹)</u>		
	FI-DIN ^a	CPN ^a	Certificate
Ni ^b	11 ± 1	13 ± 2	9.94
Cd	10.0 ± 0.2	10.1 ± 0.2	9.93
Pb ^c	10.0 ± 0.2	11 ± 1	10.08
U	9.9 ± 0.2	9.9 ± 0.2	10.07

^a Mean and standard deviation of 18 analyses.^b Based on ⁵⁸Ni.^c Based on ²⁰⁸Pb.

Table 6. ICP-MS Analysis of NIST SRM 1643C

Element ^a	<u>Analyte Concentration ($\mu\text{g L}^{-1}$)</u>		
	FI-DIN ^b	CPN ^b	Certificate
Ni ^c	57 ± 9	80 ± 20	60.6
Cd	13.0 ± 0.3	12.9 ± 0.4	12.2
Pb ^d	37 ± 1	38 ± 3	35.3

^a Uranium was neither detected or certified in the standard.

^b Mean and standard deviation of 18 analyses.

^c Based on ^{58}Ni .

^d Based on ^{208}Pb .

Table 7. ICP-MS Analysis of Waste #37

Element	<u>Analyte Concentration (mg L^{-1})</u>	
	FI-DIN ^a	CPN ^a
Ni ^b	0.8 ± 0.4	0.4 ± 0.1
Cd	1.31 ± 0.03	1.34 ± 0.03
Pb ^c	1.6 ± 0.1	1.62 ± 0.05
U	3.20 ± 0.09	3.19 ± 0.09

^a Mean and standard deviation of 18 analyses.

^b Based on ^{58}Ni .

^c Based on ^{208}Pb .

Table 8. ICP-MS Analysis of Waste #40

Element	<u>Analyte Concentration (mg L⁻¹)</u>	
	FI-DIN ^a	CPN ^a
Ni ^b	0.44 ± 0.03	0.6 ± 0.2
Cd	0.065 ± 0.002	0.067 ± 0.002
Pb ^c	0.71 ± 0.02	0.71 ± 0.03
U	0.62 ± 0.02	0.60 ± 0.01

^a Mean and standard deviation of 18 analyses.

^b Based on ⁵⁸Ni.

^c Based on ²⁰⁸Pb.

Figure 1. Relative Sensitivity versus m/z for FI-DIN and CPN

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