Direct Reading
Echelle ICP

High Throughput Sample Analysis with the Leeman Labs Direct Reading Echelle ICP

Application Note 1019

LEEMAN LABS, INC

Value Through Innovation

The Direct Reading Echelle ICP

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Introduction

Many high throughput labs are required to analyze hundreds of samples a day. Included in this category are oil, geological, agricultural, food and environmental labs. If the element suite is large enough, then the only ICP capable of handling the sample load is a fixed-channel simultaneous. A Simultaneous Leeman Labs Direct Reading Echelle (DRE) ICP combines the high resolution and dispersion of echelle optics with high gain detectors to provide the fastest sample throughput possible. ICPs using array type detectors simply cannot match the speed of the PMT based DRE, particularly when the samples are at low concentrations in a complicated matrix. Low UV sensitivity combined with the lack of signal multiplication works against the array-based systems in these high throughput applications.

The sample analysis time of a simultaneous (multi-channel) DRE is approximately one minute, including uptake and rinse. The Direct Reading Echelle (DRE) High-Throughput (HT) ICP with a new version of software, some changes to the basic analysis sequence and modifications to sample introduction hardware, has reduced analysis time by almost 50%. This results in a dramatic improvement in productivity for the high volume lab — running nearly twice as many samples as they currently do in a given day.

Instrumentation

In order to determine the increase in sample throughput, the modifications were made to a multi-channel DRE operating in a high volume oil lab. Performing wear metals in oil analysis, the lab analyzes about 300 samples per day. Each sample is analyzed for 11 elements, which are listed in Table 1 along with wavelengths and background correction points. The integration time used was 3 seconds with a 1 second background correction. Without modifications, the analysis time for a single sample was 72 seconds.

The high throughput modifications were tested with real samples submitted to the lab for analysis. The test was conducted by first running the day's

Element	Wavelength, nm	Background Correction Wavelength, nm
Cu	324.754	324.717
Fe	259.940	259.910
Cr	267.716	267.685
Al	308.215	308.250
Si	288.158	288.125
P	220.353	220.378
Mo	277.540	277.508
Na	589.592	589.660
Sn	189.926	189.904
Ni	231.604	231.577
Zn	206.200	206.224

Table 1 Elements, Wavelengths and Background Correction Points

Table 2 Analysis Conditions	
RF Power	1.1 kW
Coolant Flow	18 LPM
Auxiliary Flow	0.5 LPM
Nebulizer Pressure	45 PSI
Pump Rate	1.1 ml/min

sample load using the modified sample introduction system but none of the software changes. The sample load consisted of 300 samples plus periodic QC standards, which made the total number of analyses to be 320. The analytical conditions used are listed in Table 2. The entire sample load was run without a single QC failure, indicating the modified sample introduction system and the V-groove nebulizer had the necessary stability.

Once the lab's work was complete, the software modifications were made. These consisted of routines designed to accelerate the uptake and rinse cycle of each sample. By moving the sample tip back to the rinse before analysis is complete and increasing the speed of the peristaltic pump, 30 seconds could be removed from the analysis time. With all the modifications, the analysis time was reduced to 42 seconds per sample.

Results

The first batch of samples analyzed by the lab was re-analyzed with the high throughput modifications. Comparisons of some of the results obtained are shown in Table 3. These data are corrected for the 1:4 dilution performed on the original oil samples. In addition, these data are rounded to the nearest integer, being the standard practice for this laboratory.

The data indicates there is no difference in accuracy between the standard and high throughput set-ups. The data for all elements correlated extremely well between the two analysis runs. Figures 1 and 2 demonstrate the excellent correlation between the standard and high throughput mode for Fe ($r = 0.9997$) and Pb ($r = 0.9986$), respectively. All the elements demonstrated similar degrees of correlation over the entire concentration range found in the samples. The accelerated uptake and rinse times are sufficient to prevent any carryover between samples. This is extremely important since real samples such as these may differ significantly in concentration. During the second set of analyses, the same QC standard frequency was used and no failures occurred. This indicates the modified set-up is just as stable as the standard one. And best of all, the analysis time was reduced to 42 seconds a sample. At this analysis rate, a total of approximately 550 analyses could be completed in the time taken to run the 320 under the original set-up.

Standard and High Throughput Mode Results Comparison

Sample	1002		1004		1006		1008	
Element	STD	HT	STD	HT	STD	HT	STD	HT
Cu1	15	13	16	16	3	3	2	2
Fe2	4	4	33	33	10	9	17	17
Cr4	0	0	2	2	0	0	0	0
Al3	2	1	14	12	3	3	0	0
Si2	6	5	26	25	7	7	3	3
Pb1	1	1	1	1	2	2	1	1
Mo3	1	2	2	2	3	3	4	4
Na2	4	2	1	0	3	2	2	2
Sn1	1	1	1	1	1	1	1	1
Ni3	0	0	0	0	0	0	0	0
Zn3	1108	1084	915	896	1046	1048	1295	1295

Sample	1027		1029		1031		1033	
Element	STD	HT	STD	HT	STD	HT	STD	HT
Cu1	4	4	3	3	0	0	7	7
Fe2	67	66	67	65	3	3	15	16
Cr4	1	1	1	1	0	0	1	1
Al3	2	3	2	2	1	1	7	8
Si2	16	18	16	17	11	12	18	16
Pb1	1	1	1	1	0	0	1	1
Mo3	1	2	1	2	1	1	98	97
Na2	0	0	0	0	0	0	2	2
Sn1	0	1	1	1	0	1	1	1

Table 3 Standard and High Throughput Data Comparison

STD = Standard Analysis Speed
HT = High Throughput Mode

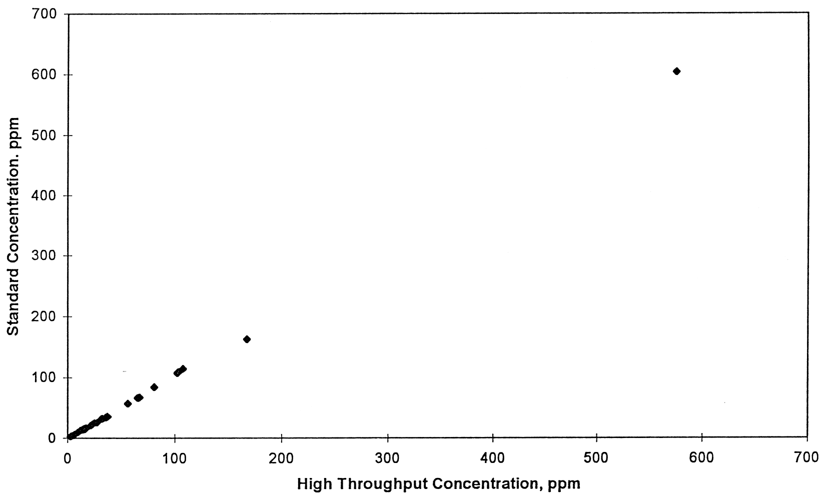

Figure 1 Fe Correlation Data

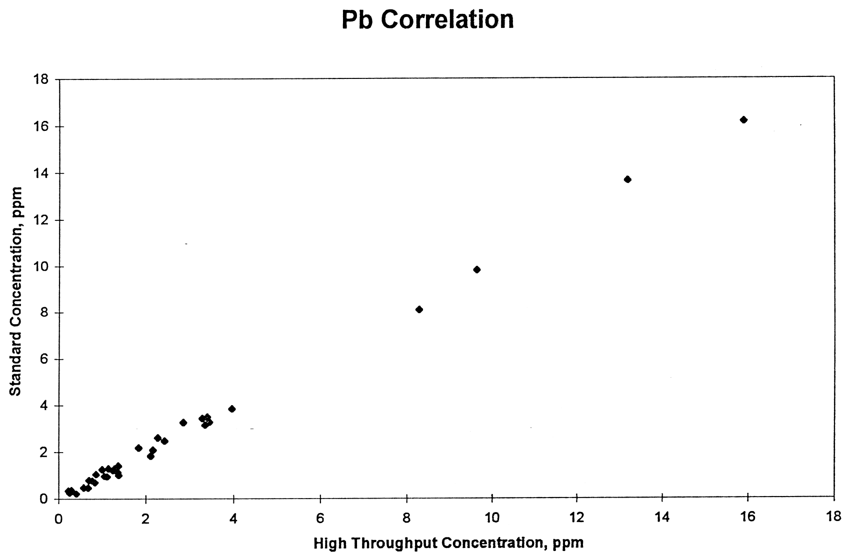

Figure 2 Pb Correlation Data

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