Technical Note: 40861

High Throughput Soils Analysis with the iCAP 6500 - Elemental Analysis in Less than 20 Seconds

Key Words

- iCAP 6500 Radial
- Environmental Analysis
- Farming
- High Speed
- High Throughput
- Soil Analysis
- Soil Nutrients

Introduction

Commercial and private analytical laboratories are becoming leaner, more cost effective and with a need for faster, more accurate analyses than ever before. The need is seated in the free-market system with competition dictating that we become better, faster and cheaper all at the same time.

The drive to become more cost-conscious combined with faster, accurate analysis in smaller laboratories is also having an effect on the instrument manufacturers who are influenced by the customers to provide these enhancements. The result of the changes in instrument design have produced spectrometers that are smaller in size, accurate, fast and versatile with computer control as standard providing easier and more accessible instrument control.

The most popular ICP of choice for environmental commercial labs was Thermo Scientific's ICAP 61e. These instruments offered rapid analysis times and good sensitivity coupled to a robust design which was sometimes limited by the fixed channel optical designs. The iCAP 6000 Series ICP spectrometers are a natural replacement for the 61e. They offer even faster analysis times and 2-3 times lower detection limits while the CID detector allows the choice of all analytical lines between 167 and 847nm.

Agricultural Sampling and Analysis

Farming is becoming an increasingly high-tech business with economic and environmental pressures requiring accelerated growth and increased yields. To provide these conditions, farmers use specialized targeted fertilization methods to ensure that their fields receive the ideal amount and type of nutrients with the minimum waste.

To achieve this, they sample the fields and tag the samples with Global Positioning System (GPS) coordinates. The samples are analyzed and the data is uploaded to the farmer's GPS system which directs the distribution system to deliver the correct soil nutrient formula to the exact locations to promote ideal growing conditions.

Principles

Soils analysis laboratories are prime examples of cost-effective, rapid analysis. They serve the farming community by analyzing surface soil samples for majors, minors, traces and micro-nutrient elements to determine the correct type and amount of fertilizer needed for that patch of arable land. Due to the high sampling errors involved in surface sampling, highly accurate and low-level analyses are not priorities but speed is. Large numbers of individual samples are analyzed for trends rather than absolute percentages and the analysis time desired is less than 30 seconds per sample.

Soils analysis includes the analysis of major, minor and trace elements in the same sample. To perform effective high speed soils analysis with an ICP there are a number of specific requirements for the instrument:-

1. Simultaneous analysis of all analytical lines.
2. Short exposure times - while retaining sufficient intensity to produce an accurate analysis.
3. The instrument should have the ability to integrate high and low intensity wavelengths in a single run.
4. Simultaneous background analysis - essential for accurate analysis and short integration times.
5. Arranging the programmed analysis to achieve the most efficient use of time.
6. Fast peristaltic pump speed to reduce the dead time between autosampler and plasma.
7. An instrument capable of rapid sample introduction and plasma equilibration with minimized washout time.

The iCAP 6500 Radial ICP Spectrometer is ideal for this type of analysis. It meets all of the above criteria and in addition can be operated with a large capacity autosampler further enhancing the flexibility.

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Instrumentation

The iCAP 6500 Radial

The iCAP 6000 series is the first generation of the Thermo Scientific new breed of ICP emission spectrometer, designed specifically for low cost of ownership through low gas consumption and reduced servicing costs. The instrument has the smallest footprint of any ICP on the market but has a high performance Echelle optical design with a unique charge injection device (CID) detector. Advancements in CID technology allow this detector to feature higher sensitivity and lower noise than any of its predecessors.

The iCAP 6500 Radial View plasma was selected for this application to minimize matrix interference.

Instrument Hardware

Parameter	Setting
Plasma view	Radial
Nebulizer	V-Groove
Torch center tube	2 mm ID
Mixing chamber	Glass Cyclonic
Sample pump winding	Yellow/Blue
Integration time	1 second
Repeats per analysis	1
Sample uptake time	3 seconds*

* The sample introduction tubing is shortened as much as possible to reduce flush time.

Plasma Parameters

Parameter	Setting
R.F. forward power	1150 watts
Coolant flow	12 L/min.
Auxiliary flow	0.5 L/min
Nebulizer flow	0.65 L/min.
Sample uptake rate	80 rpm (11 ml/min.)
Sample volume per analysis	about 2 ml

Elements analyzed

Element	Example Wavelength (nm)
B	249.773
Ca	315.887
Cu	224.700
Fe	259.837
K	769.896
Mg	279.079
Mn	293.930
Na	589.592
P	213.618
S	182.624
Zn	206.200

Method

The sample preparation method is based on an ammonium acetate extraction of the soil sample.

The soil samples were air dried overnight then ground after removing stones and foreign matter. A 5g aliquot was taken from each and placed into a plastic 30ml vial. A 20 ml solution of 1M ammonium acetate solution (1M, neutralized with acetic/ammonia) was added. The sample was shaken up several times and left to react overnight.

The solution was shaken several times more then allowed to settle for half an hour. This resultant solution was filtered, made up to 250 ml with deionized water then analyzed.

Results

The sample analysis was optimized to maximize sample throughput. Minimum length of capillary tubing was used to reduce sample uptake delays and integration times were reduced to a minimum while still maintaining data quality. The results of the speed optimization show that using a single replicate for 10 samples gives a total analysis time of 1 minute and 55 seconds.

This equates to a total time per sample of 11.5 seconds per sample. The following table (Figure 1) shows the single replicates of the 10 sample analysis.

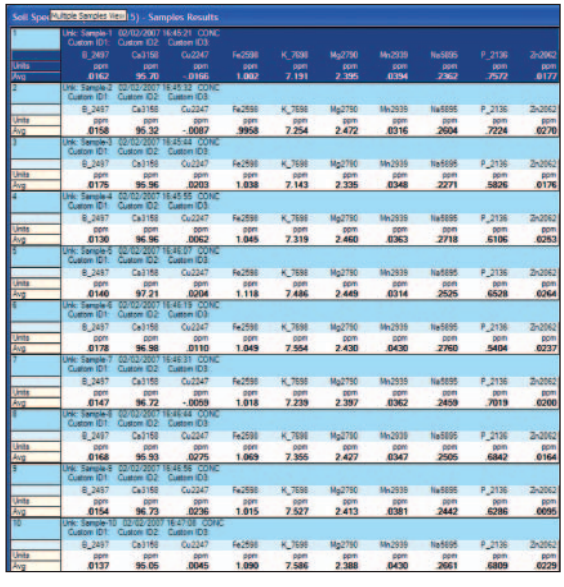

Figure 1: Single Replicates of a 10 Sample Analysis

Results table (mg/L in solution)

		B_2497	Ca3158	Cu2247	Fe2598	K_7698
2 Feb 2007 16:45:21	Sample-1	0.02	95.7	-0.02	1.00	7.2
2 Feb 2007 16:45:32	Sample-2	0.02	95.3	-0.01	1.00	7.3
2 Feb 2007 16:45:44	Sample-3	0.02	96.0	0.02	1.04	7.1
2 Feb 2007 16:45:55	Sample-4	0.01	97.0	0.01	1.05	7.3
2 Feb 2007 16:46:07	Sample-5	0.01	97.2	0.02	1.12	7.5
2 Feb 2007 16:46:19	Sample-6	0.02	97.0	0.01	1.05	7.6
2 Feb 2007 16:46:31	Sample-7	0.01	96.7	-0.01	1.02	7.2
2 Feb 2007 16:46:44	Sample-8	0.02	95.9	0.03	1.07	7.4
2 Feb 2007 16:46:56	Sample-9	0.02	96.7	0.02	1.02	7.5
2 Feb 2007 16:47:08	Sample-10	0.01	95.1	0.00	1.09	7.6

		Mg2790	Mn2939	Na5895	P_2136	Zn2062
2 Feb 2007 16:45:21	Sample-1	2.40	0.04	0.24	0.76	0.02
2 Feb 2007 16:45:32	Sample-2	2.47	0.03	0.26	0.72	0.03
2 Feb 2007 16:45:44	Sample-3	2.34	0.03	0.23	0.58	0.02
2 Feb 2007 16:45:55	Sample-4	2.46	0.04	0.27	0.61	0.03
2 Feb 2007 16:46:07	Sample-5	2.45	0.03	0.25	0.65	0.03
2 Feb 2007 16:46:19	Sample-6	2.43	0.04	0.28	0.54	0.02
2 Feb 2007 16:46:31	Sample-7	2.40	0.04	0.25	0.70	0.02
2 Feb 2007 16:46:44	Sample-8	2.43	0.03	0.25	0.68	0.02
2 Feb 2007 16:46:56	Sample-9	2.41	0.04	0.24	0.63	0.01
2 Feb 2007 16:47:08	Sample-10	2.39	0.04	0.27	0.68	0.02

Similar results are being routinely achieved in a commercial laboratory. A currently installed iCAP 6500 Radial in a soils laboratory situation produces the following sets of times:

A total of 310 unknown samples and quality control standards are typically analyzed in one hour. This averages out to approximately 11 to 12 seconds a sample. If sulfur is required in the method, 310 samples and QCs will be analyzed in 95 minutes. This averages out to just over 18 seconds a sample.

Both of the above real-life examples were well below the expected 30 second mark for the speed requirement and, as the table of results shows, the results are precise and stable especially when the short flush and integration times are taken into account.

Conclusions

The iCAP 6500 proved to be eminently suitable for providing rapid, accurate results.

The high sensitivity of the optical system and CID camera allowed the integration time to be reduced to a minimum while still retaining the capacity to produce credible results. Although the iCAP is well known for its sensitivity and low detection limits, the disparate concentrations of the soil samples, from trace to major, proved to be no obstacle.

In addition, the iCAP provides several benefits for busy commercial soils analysis labs. Analysis times are greatly reduced compared to conventional instruments allowing greater sample throughput, reduced instrumentation requirements and lower overall operating costs.

In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

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