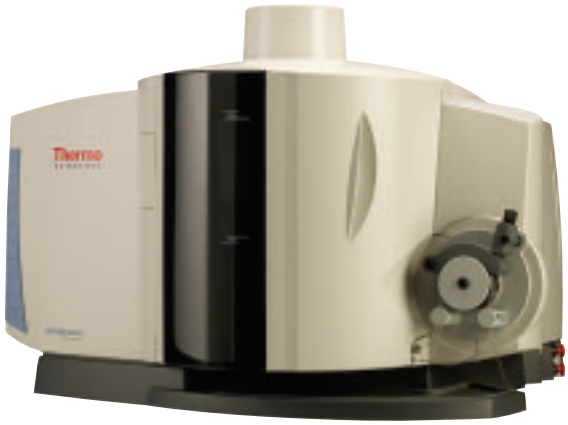
Application Note: 40960

The iCAP 6000 Series ICP with Ultrasonic Nebuliser Accessory

Key Words

- iCAP Duo
- Ultrasonic Nebuliser
- Increased sensitivity



Introduction

The use of an Ultrasonic Nebuliser (USN) in conjunction with an optical ICP system has long been accepted as a simple and cost effective way to increase sensitivity and decrease detection limits. Conventional pneumatic nebulisers are generally only 2 to 3 % efficient under normal operating conditions. The CETAC U5000AT+ Ultrasonic Nebuliser converts more of the liquid sample into a usable aerosol, with an efficiency of 10 to 15 %. This note will determine the effectiveness of the CETAC AT5000+ using a multi-element method by directly comparing the results to the standard sample introduction of the iCAP (concentric nebuliser and cyclonic spraychamber). The method development is minimal to reflect real world use, and to demonstrate if there is a clear advantage to using such an accessory.

Principle of Operation of a USN

The instrument (iCAP) peristaltic pump introduces liquid sample across an oscillating piezoelectric transducer. The oscillations disperse the sample into a fine aerosol, which is swept out of the spray chamber by a flow of argon gas from the ICP (this is provided by the nebuliser gas line). The aerosol then passes through a heated tube and an electrothermally cooled condenser. An integrated drain pump removes the condensed sample solvent and any excess sample liquid from the spray chamber. After the condenser, the dried aerosol particles are swept by the nebuliser gas to the ICP for analysis.[1]

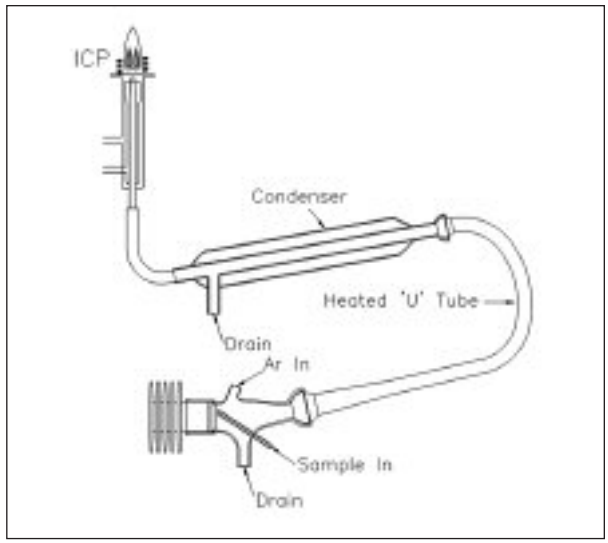


Figure 1: Schematic of CETAC USN

The CETAC USN couples to the iCAP very easily, using the nebuliser push fit connectors for the USN gas flow and coupling the sample flow directly to the torch. This allows for changeover from the standard SIK to the USN, in seconds.

Method Development

The iCAP 6500 Duo was used for this experiment, with the standard aqueous sample introduction kit. A multi-element method was created to cover the entire range of the spectrum. The same parameters were used for both the standard sample introduction kit (SIK) and the USN test, with the exception of the nebuliser gas flow, which was optimised using the Optimise Source tool.

Parameter	Setting
Pump Tubing	Tygon Orange/White sample White/White drain
Pump Rate	45 rpm
Nebuliser	Concentric/USN
Nebuliser Argon Pressure	0.3 L/min for USN, 0.75 L/min for SIK
Spraychamber	Cyclonic
Centre Tube	2 mm
Torch Orientation	Duo
RF Forward Power	1150 W
Coolant Gas Flow	12 L/min
Auxiliary Gas Flow	0.5 L/min
Integration Time	15 seconds

Table 1: Instrument Parameters

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Optimise Source allows for unattended automated method development by optimising the RF power, Nebuliser Flow, Pump Speed, Auxiliary Gas Flow, Coolant Gas Flow (and Radial Viewing Height for Radial instruments) using all or a selection of method elements, with one of three options for Best Signal, Best Signal/Background Ratio or Best Detection Limit (√SBR). Best DL was chosen in this case to optimise the nebuliser flow on the standard SIK and the USN. Figure 2 below shows the subaray plot for Aluminium 167.079 nm from the nebuliser optimisation using the standard SIK method. The Optimise Source samples have full flexibility as normal samples, with adjustable centre integration and background correction points. At the end of the automated routine, the analyst is offered the option to update the method with the optimised values, allowing the new method to be used immediately. Optimising the nebuliser gas flows took just 4 minutes using the automated routine, which enables unattended method development, as it simply aspirates a method solution.

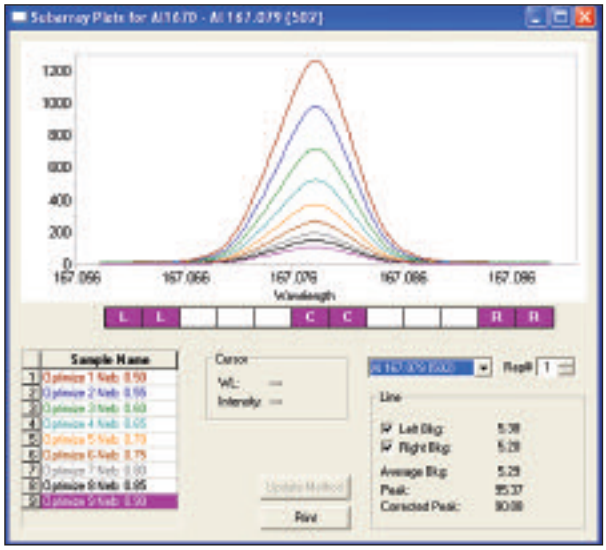

Figure 2: Optimise Source plot for the nebuliser gas flows

Results & Discussion

The Optimise Source routine found that 0.75 L/min was optimum for the standard SIK method and 0.3 L/min for the USN method.

A blank (Deionised Water) and a 0.5 ppm standard were used to calibrate the instrument.

Table 2 below shows the increase in sensitivity from the standard SIK to the USN over the range of elements. The counts for the 0.5 ppm standard have been bank corrected to demonstrate the actual increase in sensitivity. The deionised water sample was not pre-treated in any way before analysis, which explains the poor Boron sensitivity, as most of it is lost down the drain. For Boron analysis to be successful on USN, it is normal practice to treat samples with 0.2 % mannitol and use a 1 % tartaric acid rinse between samples to aid the wash out.

	SIK	USN	
	0.5ppm Standard	0.5ppm Standard	Times Increase in Sensitivity with USN
Element	CTS/S	CTS/S	
Ag3280	5513.351	50706.34	9.2
Al1670	156.4023	3552.7901	22.7
As1937	53.8533	710.3539	13.2
B_2497	1745.1	88.1	0.1
Ba4554	324269.12	1756783.6	5.4
Be3130	115067.8	1937927.88	16.8
Ca3968	466915.8	3172240	6.8
Cd2288	907.2834	13036.488	14.4
Co2286	348.1686	5834.703	16.8
Cr2677	1943.1946	32540.4869	16.7
Cr2835	3143.1	48197.28	15.3
Cu3247	11779.9	57043.97	4.8
Fe2599	2629.3175	34407.43	13.1
Mg2795	94421.13	1578158.4	16.7
Na5895	53601.5	488558	9.1
Ni2216	461.531	8875.072	19.2
Ni2316	198.609	3365.4635	16.9
S_1807	91.318	862.035	9.4
Sb2068	100.2599	1031.3819	10.3
Si2516	901.639	7623.39	8.5
Sn1899	62.7455	1176.765	18.8
Ti3349	21813.657	208483.05	9.6
Tl1908	108.3712	1310.0869	12.1
V_3093	8282.34	92769.43	11.2
Zn2138	1723.26	26656	15.5

Table 2: Sensitivity of 0.5 ppm standard on standard SIK and USN

After calibration, the blank was analysed 10 times in a single sample to ascertain an instrument detection limit. Table 3 below shows the Detection Limits gained from both of the methods. As above, because of the lack of pre-treatment, the Boron detection limit is extremely high at 148 ppb. Contamination of the blank (with Ca, Na and Mg) and the USN (with Cu) and was also noted, which explains the high detection limits for those elements.

	SIK	USN
	Detection Limit	Detection Limit
Ag3280	0.72	0.24
Al1670	1.34	0.14
As1937	8.5	1.04
B_2497	1.68	148.39
Ba4554	0.02	0.02
Be3130	0.03	0.03
Ca3968	1.65	3.28
Cd2288	0.78	0.1
Co2286	1.55	0.26
Cr2677	0.74	0.09
Cr2835	0.78	0.09
Cu3247	0.26	1.31
Fe2599	0.62	0.13
Mg2795	0.29	0.25
Na5895	1.46	0.62
Ni2216	2.17	0.4
Ni2316	2.72	0.38
S_1807	5.37	0.87
Sb2068	5.53	0.61
Si2516	2.13	2.03
Sn1899	5.35	0.39
Ti3349	0.08	0.03
Tl1908	2.45	0.76
V_3093	0.34	0.08
Zn2138	5.2	0.96

Table 3: Detection Limits in ppb

Conclusions

The use of the CETAC AT5000+ USN was a simple and efficient to gain an average 12 fold improvement in sensitivity over a full range of elements. Without doubt, the detection limits produced here can be improved by optimising the method per element, but even as a mutli element method, the results are impressive and offer an easy alternative to improving detection limits without considering alternate techniques such as GFAAS and ICPMS.

Acknowledgements

1 CETAC Technologies, 14306 Industrial Rd., Omaha, Nebraska 68144, USA