



Application of the MCN-6000 for Determination of Trace Elements in Concentrated Acids

MCN-6000 with ICP-MS

Problem: The trace element content of high purity semiconductor reagents is of critical importance in determining device quality. This analysis can be very extremely difficult being limited often by contamination, available sample volume and also by difficulties in determining traces of certain elements which suffer from problematic spectral interferences.

Solution: The unique desolvating microconcentric nebulizer, the MCN-6000 from CETAC is a highly efficient nebulization system designed specifically for small volume work with ICP-MS. Combining the fundamental microvolume capabilities of the MCN-100 with the ultimate desolvation performance of CETAC's proprietary microporous membrane technology, the MCN-6000 delivers performance of high sensitivity and extremely low levels of interferences. The system is designed for ultratrace work and being fabricated from totally inert materials and additionally incorporates an enclosed micro autosampler.

Analytical advantages:

- w Microvolume sampling at 10-100 uL / min.
- w Sensitivity enhancement of about an order of magnitude.
- w Reduction in polyatomic ion interferences.
- w Direct analysis of aggressive reagents.

Analytical performance: The totally inert build of the MCN-6000 allows for the direct analysis of many aggressive acid matrices. Acids such as HCl, HNO₃, and even HF can be determined at extremely high matrix levels. The system achieves excellent precision on such materials due to the aerosol generation and the buffering effect of the membrane. Table 1 illustrates the precision achieved in three acid matrices with spikes at the 50 ng/L level.

<u>Element</u>	<u>69% HNO3</u>	<u>35% HCL</u>	<u>49% HF</u>
	<u>%RSD</u>	<u>%RSD</u>	<u>% RSD</u>
V51	0.92	0.91	0.97
Cr 52	0.51	1.83	0.46
Fe 56	1.57	1.9	1.81
Ni58	1.57	1.17	0.75
Co59	0.45	1.28	0.59
Cu63	4	3.15	4.5
As75	1.97	-	0.42
Cd111	8	3.94	0.5

Recoveries of added elements in such acids is an excellent measure of analytical accuracy when no reference materials are available. Recoveries at the 500 ng/L level are illustrated for all three acids on Table 2.

Element	69% HNO3	35% HCl	49% HF
V51	561	495	514
Cr52	522	497	528
Ni58	666	472	487
Co59	493	490	497
Cu63	536	536	558
As75	673	-	517
Cd111	502	429	558

Table 2. Recoveries from spikes at 500ng / L level in concentrated acids.

The element suite selected represents some of the most difficult elements to be determined principally because of the high levels of polyatomic interferences, the desolvation system however is so efficient that the interferences are analytically insignificant and good recoveries and linear calibrations can be achieved for all the elements to low ppt levels. Calibrations for a range of elements are illustrated on Figures 1a-c particularly impressive is the curve for V⁵¹ in 35% HCl which exhibits linearity of 0.9995 down to 0.01ug/L level especially so given the high chloride matrix and the resultant removal of the ClO⁺ interference at mass 51

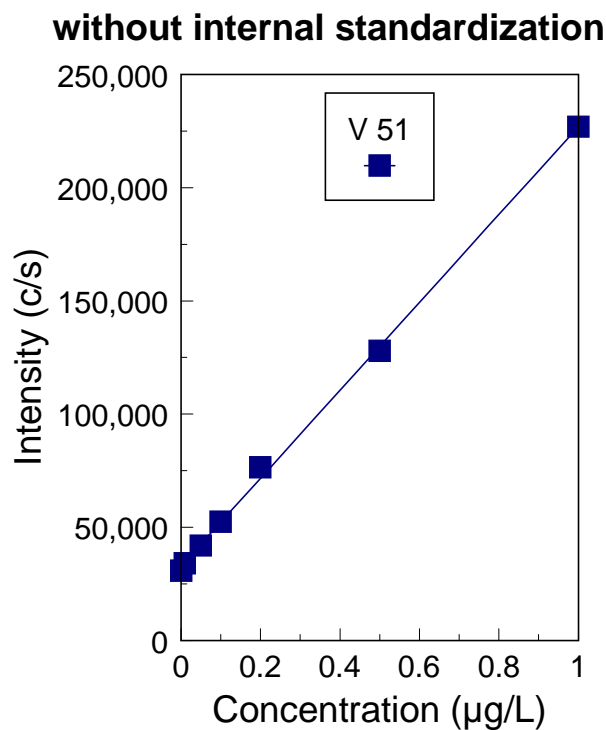


Figure 1a. Calibration for V⁵¹ in 35% HCl without internal standardization.

The calibration for Co in conc HF shown on Figure 1b shows linearity down to base level with no significant contribution from ArF^+ . The sensitivity enhancement of the system is illustrated in Figure 1c. for As in concentrated HNO_3 where the As can be measured to below ng /L levels.

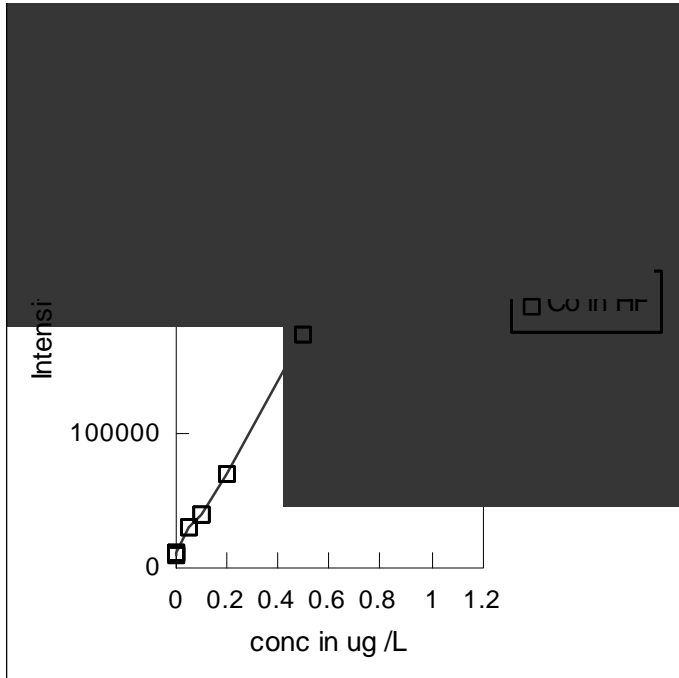


Figure 1b. Calibration for Co in 49% HF.

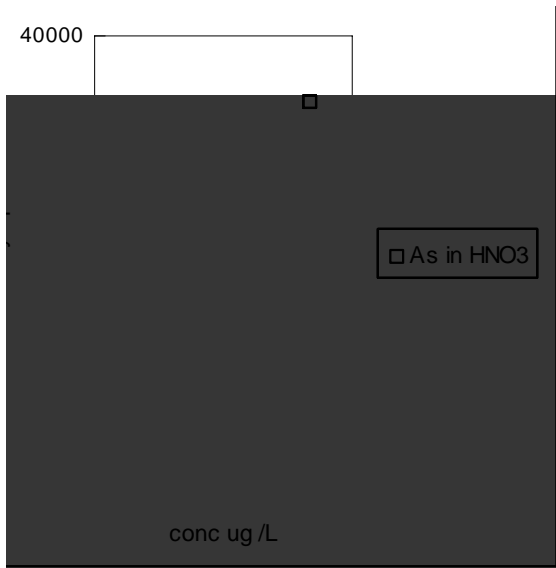


Figure 1c. Calibration for As in 60% HNO_3 .

Summary: The high efficiency of aerosol production of the coupled to the membrane desolvation allows extremely low levels of trace elements to be determined in even the most aggressive reagents using the MCN-6000. Interferences are reduced to nominal levels allowing access to transition elements and sensitivity is at least an order of magnitude better than with conventional systems. Specific details of the analytical performance of the MCN-6000 on Fe, K and Ca can also be found on applications note RAB/MCN6/3/96.

- References:
1. Performance characteristics of a desolvating microconcentric nebulizer, CETAC MCN 6000 with ICP-MS. CETAC Rapid Applications Bulletin RAB/MCN6/1/96
 2. Determination of Fe, K and Ca using the CETAC MCN 6000. CETAC Rapid Applications Bulletin RAB/MCN6/3/96.