

## Application News

Spectroscopy - ICP

No. SCA-115-021

# Determination of Trace Elements in Toluene using ICPE-9000 and Oxygen Kit

### ■ Introduction

To evaluate the elemental pollution in petrochemical field of application, there is the need to measure organic samples in a direct way, as concentrations might be very low. To be able to perform fast multi element analysis Shimadzu's ICPE-9000 is most suitable.

To obtain highest sensitivity measuring organic samples, the Ar/O<sub>2</sub> Mixed Gas Supply Kit is recommended.

With this Kit a special 4-way torch is used, the Quadruple torch (figure 1). An additional gas (Ar + O<sub>2</sub>) is mixed with the carrier gas flow resulting in a high oxidizing power. This allows measuring trace levels of elements by reducing the background noise from the organic solvent, which now is separated as CO<sub>2(g)</sub>.

### ■ System Parameters

To increase sensitivity, different parameters were optimized to obtain lowest limit of detection, in this application focussed on tin (Sn) in toluene matrix.

ICPE-9000 Parameters*	
RF generator power	1.6 kW
Plasma torch	Quadruple torch
Plasma observation	Radial View
Plasma gas (Ar)	20.0 L/min
Auxiliary gas (Ar)	1.40 L/min
Carrier gas (Ar)	0.55 L/min
Additive gas (Ar + O <sub>2</sub> )	0,21 L/min
Sensitivity	High
Exposure time	30 seconds
Repetition	threefold
*using Ar/O <sub>2</sub> Mixed Gas Supply Kit	

Table 1: System configuration & parameters

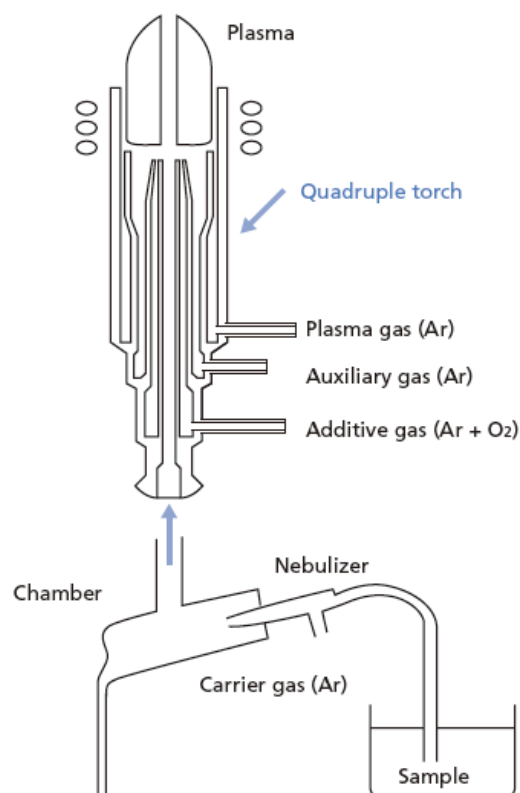


Figure 1: Sample introduction and torch

Depending on several parameters like sample introduction tube length, sample matrix and final method parameters, the rinse times can be optimized with the software integrated time-base-measurement function as described in Application News No. SCA-115-020 "Connection of HVG-1 to ICPE-9000 (Application: Water Analysis)".

## ■ Results

Using the optimized parameters it was possible to measure a calibration containing 5,0 ppb tin as lowest point (figure 2). The LOD estimated by software (3s- criterion) is 2.0 ppb. Another way to calculate LOD is to evaluate the calibration linearity. That is why the LOD is determined according to DIN 32645, additionally. Table 2 shows comparable results of both calculation models.

	LOD	LOQ
ICPEsolution	2,0 ppb (3s)	6,6 ppb (10s)
DIN 32645*	1,6 ppb	6,2 ppb
*result uncertainty 33,33%; $\alpha = 3,00\%$		

Table 2: Statistical data

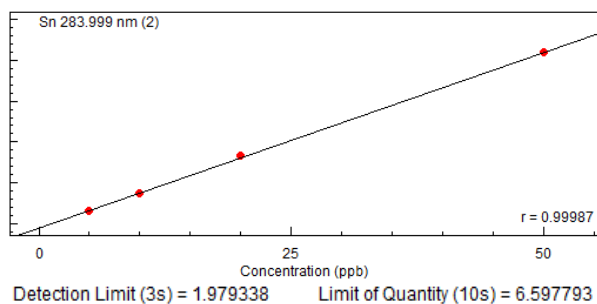


Figure 2: Sn calibration

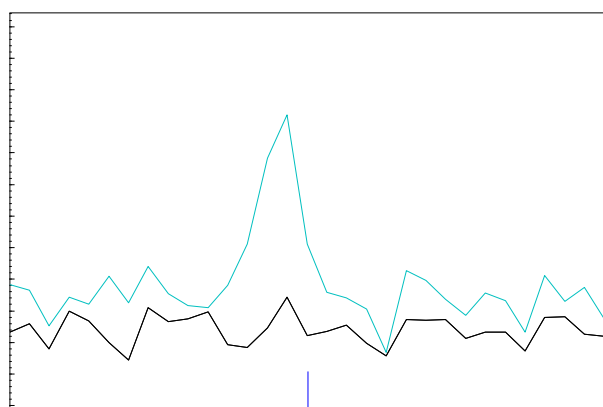


Figure 3: Sn peak profile blank and 50 ppb

## ■ Discussion

As plasma is stable using the solvent toluene and the optimized parameters (table 1), the samples don't have to be diluted in other solvents. With this direct measurement the LOQ is below 7 ppb. A quality control (QC) sample was in the specifications as there always is an uncertainty for QC concentration (10±1 ppb) and the measurement result (13±2 ppb).

Measuring organics the Ar/O<sub>2</sub> Mixed Gas Supply Kit is recommended and ensures a long life time of ICPE-9000 consumables like the Quadruple torch. No carbon deposition can be observed and background noise is decreased a lot, allowing highest sensitivity.

Expanding the measurement range to axial view, an improvement of statistical data is possible. Nevertheless even in radial view Shimadzu's ICPE-9000 offers fast simultaneous measurements in low ppb range for many other elements (figure 4).

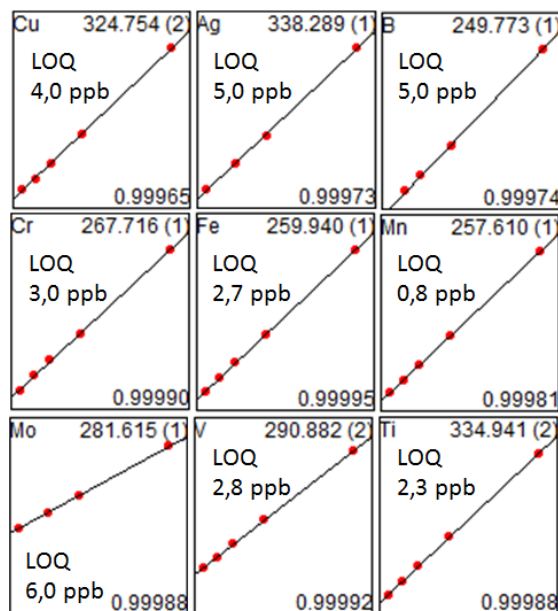


Figure 4: Calibration & LOQ of other elements

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