

Optimized power-supply – miniaturized electrothermal vaporizer – microplasma tandem through precise temperature control for the introduction of liquid microsamples into the μ CCP

(Activity 1.4, CO-UBB)

In this activity, the temporal evaporation of several elements from the Rh microfilament was investigated using the programmable Tenma 72-13360 power supply (TENMA Inc., China), controlled through a program written in LabVIEW. The 10 μ L liquid microsample was introduced *via* the miniaturized SSETV device (Babeş-Bolyai University, Cluj-Napoca) and electrothermally evaporated from a 250 μ m diameter, 99.9% purity Rh microfilament (Goodfellow, Cambridge, UK) by heating for 10 s at 1500 $^{\circ}$ C (2.3 V and 4.56 A). Prior to evaporation, the microsample was dried at 80 $^{\circ}$ C for 180 s by applying a potential of 0.25 V and a current of 1.93 A. Three-dimensional episodic emission spectra (signal – wavelength – time) were recorded simultaneously using the Maya2000 Pro microspectrometer (Ocean Optics, Dunedin, USA), with an integration time of 100 ms per spectrum. Spectroscopic observation of the microplasma was performed at a height of 0.8 mm above the Mo tip microelectrode on which the microplasma is formed.

Thermal calibration of the Rh microfilament was performed over two temperature ranges, 50–600 $^{\circ}$ C and 800–1600 $^{\circ}$ C, by measuring the filament temperature using IR sensors 3ML-CF1 and 1MH1-CF3 (Optris GmbH & Co. KG, Berlin, Germany). The TENMA 72-13360 power supply allowed temperature control of the Rh microfilament with a precision better than 15 $^{\circ}$ C and an accuracy of at least $\pm$ 20 $^{\circ}$ C at a target temperature of 1500 $^{\circ}$ C, maintained for 7 seconds after approximately 3 seconds of heating time required for the filament to reach the set temperature. A bidirectional SMCEVT307–5 DZ–01 F–Q valve powered by the HY3003 Mastech power supply (Premier Farnell, Leeds, UK) was integrated into the argon flow line to control the direction of the gas during the drying and vaporization steps.

Figure 1 illustrates the episodic emission spectra recorded following the evaporation of a 10 μ L sample containing 0.1 mg L⁻¹ Cd, Zn, and Hg, 1 mg L⁻¹ Cu and Pb, and 2 mg L⁻¹ Se. Figure 2 presents the transient emission signals of the elements recorded at their most sensitive spectral lines. The emission spectrum of arsenic was recorded separately using a 2 mg L⁻¹ single-element solution due to the spectral interference between the As line at 228.812 nm and the Cd line at 228.802 nm.

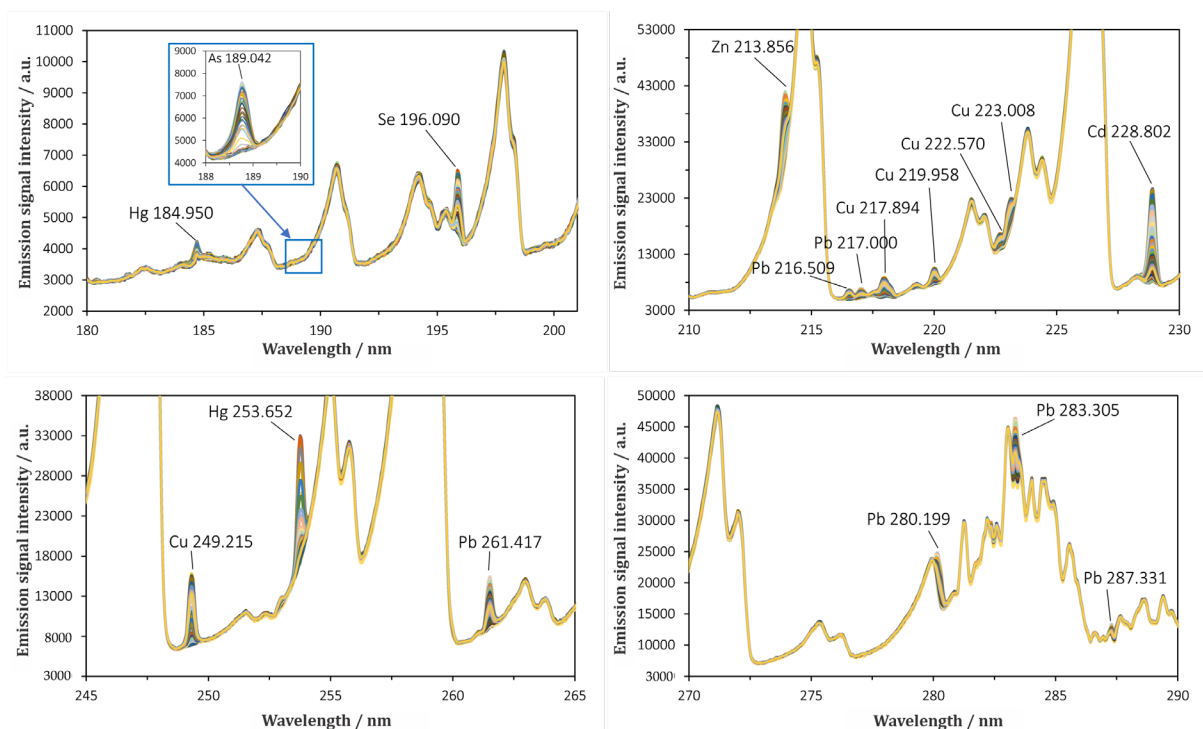


Figure 1. Episodic emission spectra of a solution containing 0.1 mg L^{-1} Cd, Zn, and Hg; 1 mg L^{-1} Cu and Pb; and 2 mg L^{-1} Se, recorded using the SSETV- μ CCP-OES method. Arsenic was measured using a single element solution with a concentration of 2 mg L^{-1} . The operating conditions were: 15 W plasma power, 150 mL min^{-1} Ar 5.0 flow rate, 0.8 mm observation height above the Mo tip microelectrode, 100 episodic spectra recorded with 100 ms integration time, and filament heating at $1500 \pm 20 \text{ }^{\circ}\text{C}$ for 10 s. (AC. Mot, A.-I. Dudu, T. Frentiu, D. Petreus, E.-A. Levei, Z. Stupar, M. Frentiu, E. Covaci. *J. Anal. At. Spectrom.*, 2025, Advance article, DOI:10.1039/D5JA00297D)

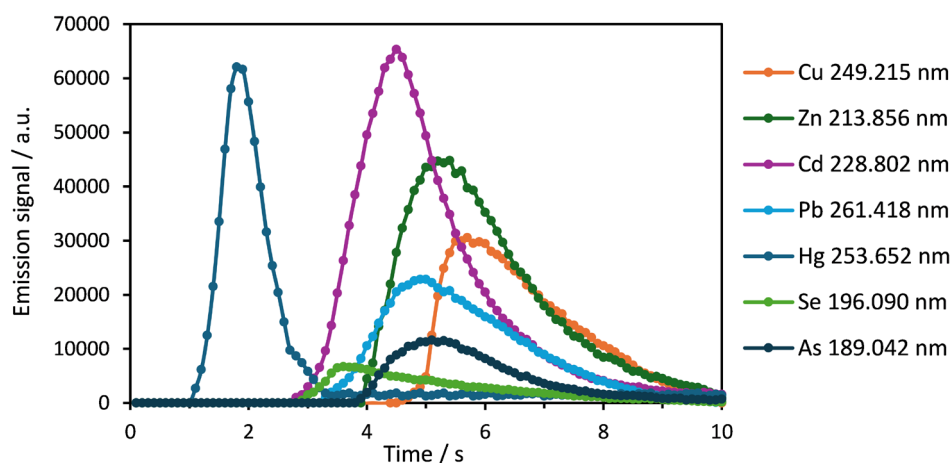


Figure 2. Transient signals of the elements recorded at their most sensitive spectral lines for a $10 \text{ } \mu\text{L}$ sample containing 0.1 mg L^{-1} Cd, Zn, and Hg; 1 mg L^{-1} Cu and Pb; and 2 mg L^{-1} Se. Arsenic was measured using a 2 mg L^{-1} single element solution. Measurement conditions were: sample drying at $80 \text{ }^{\circ}\text{C}$ for 180 s, sample vaporization at $1500 \pm 20 \text{ }^{\circ}\text{C}$ for 10 s, plasma power of 15 W, argon flow rate of 150 mL min^{-1} , observation height of 0.8 mm above the molybdenum microelectrode tip, and acquisition of 100 episodic spectra with an integration time of 100 ms per episodic spectrum. (AC. Mot, A.-I. Dudu, T. Frentiu, D. Petreus, E.-A. Levei, Z. Stupar, M. Frentiu, E. Covaci. *J. Anal. At. Spectrom.*, 2025, Advance article, DOI:10.1039/D5JA00297D)

Based on the episodic emission spectra, it was concluded that the microplasma provides sufficient excitation for emission lines of elements with excitation energies below 7.5 eV. Spectroscopic observation at 0.8 mm above the microplasma allowed the simultaneous recording of the emission spectra of all elements. Although different transient evaporation behaviors were observed, especially for Hg, the experimental data indicated that heating the filament to 1500 ± 20 °C for 10 seconds enables the recording of transient emission signals for elements with excitation energies up to 7.5 eV. This represents a major practical advantage, as it requires only simple instrumentation. Consequently, simultaneous determination of at least seven elements of interest for environmental monitoring and food control can be achieved, which will be further investigated in the following activities and stages.

Results: Power supply - optimized miniaturized electrothermal vaporizer tandem operation for the simultaneous determination of Cd, Cu, Zn, Hg, Pb, Se, and As by SSETV- μ CCP-OES for environmental and food control