Vapor-phase Sample Supply of Metals as β-Diketonates in Atomic Absorption Spectroscopy[1]

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In atomic absorption and other flame spectroscopy, the nebulization technique has been widely employed to supply the analytical flame with liquid samples. Instead of the conventional nebulization, though, methods have recently been reported in which metal fumes or vapors are introduced to flames or absorption cells.[2] In the present work a volatilizer-premix burner system has been developed and a vapor-phase sample supply has been extended by the use of some β-diketonates of metals.[3]

The volatilizer-premix burner was constructed with a volatilizer unit and a pre-heated mixing chamber-type premix burner unit, which were connected to each other in a gas-tight manner. The volatilizer unit consisted of a volatilization chamber (a Pyrex tube, 1 cm I.D. × 13 cm L.) and an electric heater. This chamber was used with or without a small column packed in its rear end which worked as a "spoiler." The temperature inside the volatilization chamber was adjusted to any level between 100 and 250°C. The premix burner unit consisted of a metal mixing chamber with an electric heater and a 5 cm-slot burner head. The mixing chamber was heated to any temperature in the 150—250°C range. Dried nitrogen (up to 170 m*l*/min) was supplied to the volatilization chamber. Air (4.8 *l*/min) and acetylene (1.2 *l*/min) were supplied to the mixing chamber. A Perkin-Elmer Model 303 atomic absorption spectrophotometer was used to record the absorptions at the most sensitive line of each metal. Samples were prepared by dissolving the laboratory-prepared trifluoroacetylacetonates of copper, chromium and iron[4] in chloroform and commercially obtained acetylacetonates of copper and chromium in a chloroform-methanol mixture. Samples of 0.2—10 μ*l* (containing about 0.1—5 μg metal) were injected by a microsyringe into the volatilization chamber through a silicone rubber septum.

Higher temperatures of the volatilization and mixing chamber, and larger nitrogen flow rates, generally gave higher peak absorptions. The analytical curves obtained with a 1 cm glass-microbead column spoiler are shown in Fig. 1. The absolute sensitivity was found to be considerably higher than that estimated with the conventional nebulization (about eight-fold in the case of chromium). With a 4 cm gas-liquid partition column spoiler (30% SE-30 on Shimalite W), the metal β-diketonates were completely separated from the solvent and from each other. Background absorption caused by chloroform was eliminated. These results indicate that the vaporphase sample supply in atomic absorption spectroscopy makes possible a rapid, highly sensitive, and interference-free determination of metals as volatile β-diketonates and possibly also as organometallic compounds. We have also noted the possibility of identifying any particular ligand of metal chelates.

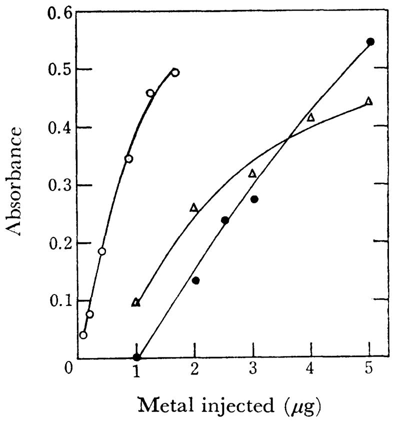

Fig. 1. Analytical curves for chromium acetylacetonate (○), copper trifluoroacetylacetonate (●), and iron trifluoroacetylacetonate (△).

	○	●	△
Volatilization chamber temp. (°C)	202	180	220
Mixing chamber temp. (°C)	200	238	199
Nitrogen flow rate (m*l*/min)	168	164	164

Precision in C.V.(%): 3.4(○), 13(●), 4.4(△)

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