Communications to the Editor

Chem. Pharm. Bull.
31(4)1419—1421(1983)

FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY COUPLED WITH SOLVENT EXTRACTION / FLOW INJECTION ANALYSIS

Koreharu Ogata,* Shinzo Tanabe and Toshio Imanari
Faculty of Pharmaceutical Sciences, Chiba University,
1-33, Yayoi-cho, Chiba-shi, Chiba

A system is described which permits the simple and rapid determinations of traces of metals in less than 200 µl of sample solution by flame atomic absorption spectrophotometry coupled with solvent extraction/flow injection analysis (FAAS-Ex/FIA).

In its application to zinc, the sampling rate was 60 samples/h and the detection limit was 10 ppb (2 ng, zinc).

KEYWORDS —— flame atomic absorption spectrophotometry; solvent extraction/flow injection analysis; determination of zinc; phase separator

Atomic absorption spectrophotometry (AAS) is essential for the determination of metals in environmental or biological samples; in particular, flame AAS (FAAS) is widely used on account of its simplicity of operation and sufficient sensitivity.

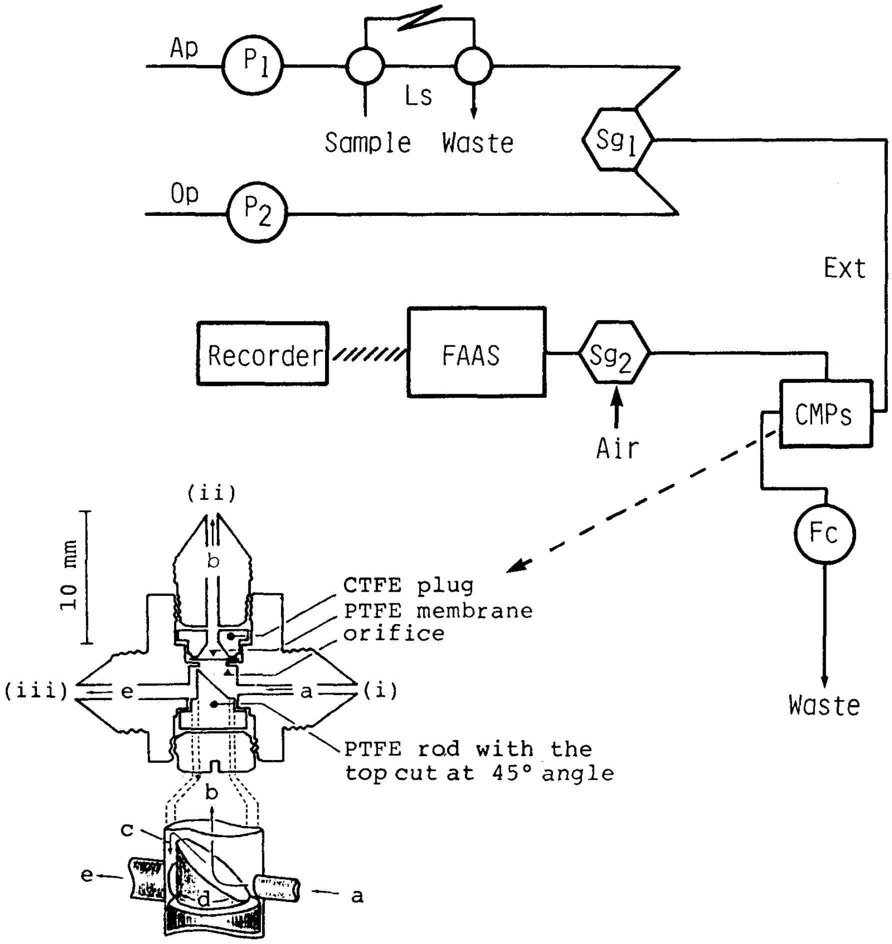
However, it is often necessary to employ a tedious manual extraction, in cases where interfering substances are present in sample solutions or concentration of metals is low. To overcome these difficulties, solvent extraction/FIA (Ex/FIA) may be applicable.

Recently, the study on the Ex/FIA coupled with FAAS using a specially devised manifold was reported,$^{1)}$ but this method was not a completely continuous flow system and operating conditions were rather complicated.

In this experiment, we tried to connect FAAS directly with Ex/FIA as a complete flow system. The flow rate of Ex/FIA during the operation is generally lower than the aspiration rate of the spectrophotometer. To combine both systems directly, it is important to develop a suitable method for delivering the organic stream emerging from the Ex/FIA into the nebulizer of the spectrophotometer. This problem was solved by introducing an "air-compensation method".$^{2)}$ It is also necessary to concentrate analyte solutions by Ex/FIA to lower detection limits of FAAS. For the purpose, the phase volume ratio (Op/Ap: Op; organic phase, Ap; aqueous phase) had to be kept as small as possible in this system and the phase separator had to separate two phases under high flow rate conditions. The chamber membrane type phase separator devised by us (CMPs)$^{3)}$ satisfied the requirements.

Experimental conditions of the system were established by using zinc as a model element, considering the problems mentioned above. The flow diagram of FAAS coupled with Ex/FIA system and the construction of CMPs are illustrated in Fig.1.

NII-Electronic Library Service



Chamber Membrane Type Phase Separator3)

(i)inlet of Op and Ap segments, (ii)outlet of Op, (iii)outlet of Ap and excess Op, (a)flow route of two segments, (b)flow route of Op, (c) and (d)flow routes of Ap and excess Op.

CTFE; poly(monochlorotrifluoroethylene)

PTFE; poly(tetrafluoroethylene)

Fig.1. Flow Diagram of FAAS-Ex/FIA System and Construction of CMPs

A sample solution (200 µl) containing zinc [prepared with 1.3 M ammonium chloride buffer (pH 9.0) containing 0.1 % N,N-diethyl dithiocarbamate (DDTC)] is injected into Ap stream [1.3 M ammonium chloride buffer (pH 9.0), 2.0 ml/min, pump P_1; Seishin PSD-3.2] by loop sampler(Ls). Ap stream meets Op stream [methyl isobutyl ketone (MIBK), 1.1 ml/min, pump P_2; the same as P_1 type] at the segmentor(Sg_1).$^{4)}$ The DDTC-zinc complex is extracted away from Ap segments in the extraction tube (Ext; 0.5 mm i.d. x 100 cm) to Op segments. The flow rate of separated Op is adjusted at 0.9 ml/min by the controller(Fc).$^{5)}$ The gap in flow rate between the Ex/FIA and FAAS is supplemented by spontaneous aspiration of air at the segmentor(Sg_2).

Analytical conditions by FAAS(Shimadzu AA-650) are as follows:acetylene; 2 l/min, air; 10 l/min for the nebulizer, 100 mm lamina flow burner height; position 4.0 mm, sample flow rate; 3.5 ml/min, wavelength for zinc; 213.86 nm, measurement mode; background correction mode by D_2 lamp.

In this method, the concentration factor of about 2 was attained, and the sensitivity was 3 times higher than that of FAAS at the same aspiration rate as the flow rate of separated Op from Ex/FIA(0.9 ml/min). The sampling rate was 60 samples/h and the detection limit was 10 ppb (2 ng, zinc). The calibration curve for zinc was linear in the range of 20 - 800 ppb. The relative standard deviations(RSD) were 2.7 % and 1.5 % for 50 ppb and 100 ppb zinc, respectively.

It was proved that the present method was useful in the determination of zinc in 200 µl of sample solution, and might be used for determining traces of other metals by choosing suitable solvent systems.

REFERENCES

1) L. Nord and B. Karlberg, Anal. Chim. Acta, 125, 199 (1981).
2) N. Yoza, Y. Aoyagi and S. Ohashi, Anal. Chim. Acta, 111, 163 (1979).
3) K. Ogata, K. Taguchi and T. Imanari, Anal. Chem., 54, 2127 (1982).
4) J. Kawase, Anal. Chem., 52, 2128 (1980).
5) K. Ogata, K. Taguchi and T. Imanari, Bunseki Kagaku, 31, 641 (1982).

(Received January 14, 1983)