DESIGN OF A POLYVALENT FLOW INJECTION SYSTEM FOR SPECTROPHOTOMETRIC ANALYSIS OF PLANT LEAVES

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A polyvalent flow injection system was proposed for spectrophotometric determination of iron, copper, manganese and zinc (micronutrients), as well as calcium, magnesium and phosphorous (macronutrients) in plant digests. The aim of this work was to simplify the use of flow injection analysis in laboratories usually processing large lots of samples. It consists of a single system, where it is possible to make different determinations without modifications in the manifold. The same manifold configuration can accommodate a variety of chemistries, without the need for physical reconfigurations.

Determination of total iron was based on reaction between $Fe(II)$ and 1,10 o-phenanthroline, with prior reduction of $Fe(III)$ present in the digest by ascorbic acid. Copper was determined by exploiting the catalytic effect of $Cu(II)$ on the oxidation of ferric ions by thiosulphate; and manganese, by formation of a red complex in alkaline medium between $Mn(II)$ and formaldoxime. Zinc determination involved reaction with potassium cyanate, further destroying of formed Zn-cyanocomplexe by formaldehyde (pH 9.0), and reaction of the freed metal with Zincon. O-cresolphtalein complexone (CPC) was the chromogenic reagent for calcium and magnesium determination, addition of either EGTA or oxine and proper pH adjustment being required for selectivity improvement. The ammonium molybdate/vanadate method was applied to phosphate determination.

Plant materials underwent a dry digestion procedure followed by recovery in 1.0 M HCl and 5-fold dilution with water. Carrier stream (C - 4.0 mL min^{-1}) was 0.2 M HCl. R_1, R_2 and R_3 reagents, 1.0 mL min^{-1} (Fig. 1) were different, depending on the analyte (Tab. 1). The reaction coils, B_1, B_2 and B_3 were 30, 50 and 200 cm long. The wavelength depended on the element to be determined, and the sampling loop L was either 50 cm (Fe, Cu, Mn, Zn and P) or 10 cm (Ca and Mg). Fig. 1 represents the

proposed system, and Tab. 1 shows the reagent concentrations and the linearity of the calibration equations.
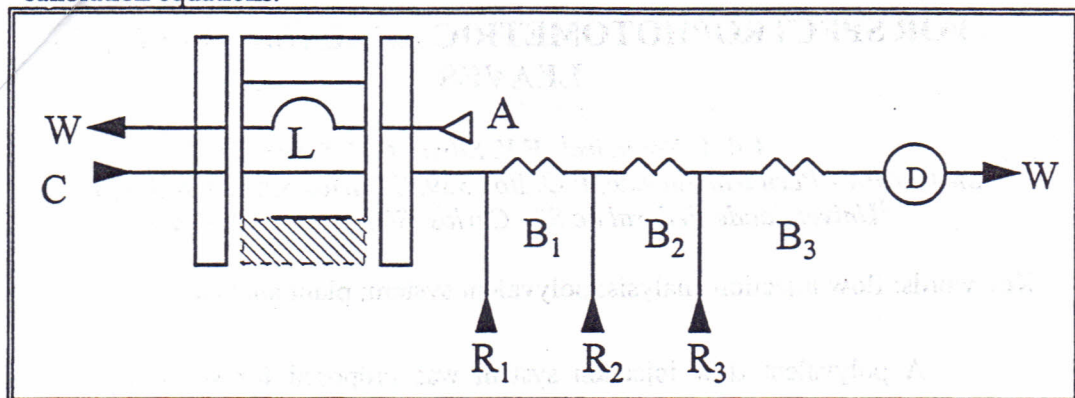
Figure 1. The polyvalent flow injection system. A, sample; L, sampling loop; C, carrier stream (4.0 mL min⁻¹), R_1, R_2, R_3, reagents (1.0 mL min⁻¹); B_1, B_2, B_3 coiled reactors (30, 50 and 200 cm); D, detector; W, waste.

Analyte	δ nm	linearity ($\mu g\ mL^{-1}$)	R_1 (1.0 mL min⁻¹)	R_2 (1.0 mL min⁻¹)	
Iron	512	up to 10.0	1 % (w/v) ascorbic acid	0.25 % (w/v) 1,10 o-phnantroline + 2.0 M NH₄OAc	whater
Zinc	630	2.0	2 % (w/v) ascorbic acid	0.5 % (w/v) KCN + 0.7 M NaOH + 0.5 M Na₂B₄O₇	0.04 % (w/v) Zincon + 1.0 % (w/v) HCHO + 0.02 M NaOH
Manganese	455	2.0	3 % (w/v) ascorbic acid	3 % (w/v) KCN + 2.0 M NaOH	0.5 M formaldoxime
Copper	550	0.6	0.20 M Na₂S₂O₃	whater	0.025 M FeCl₃
Calcium	575	30.0	0.5 M NaOH	0.5 M NH₄/NH₄-pH 10.5.	0.01 % (w/v)(CPC)+ 0.2 % (w/v) oxine
Magnesium	570	10.0	0.5 M NaOH	4.5 mM EGTA + 4.9 mM Ba (II) in 0.5 M NH₄/NH₄-pH 10.5.	0.06 % (w/v) CPC
P - PO₄	420	30.0	whater	1.6 % (w/v) (NH₄)₂MoO₄	0.08 % (w/v) NH₄VO₃

Table 1. Reagent concentrations, wavelengths, and linearity of the calibration equations.

Slight variations in the coefficients of the calibration equations (usually < 3 %) were found after 4-hour working periods. Results were precise (r.s.d. < 0.02), and in agreement with certified values of standard reference materials. When the system was applied to large scale analysis, no major problem did occur, emphasizing the stability, simplicity, robustness and versatility inherent to flow injection analysis. (FAPESP, EMBRAPA).