

FLOW INJECTION ANALYSIS

A New Approach to Soil and Plant Analysis

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Introduction

Agronomists and environmental scientists often need larger numbers of chemical analyses of water, soil, soil extracts or plant digests in the course of their research. Some of this analytical work can be sent to central laboratories, which have automated analytical equipment such as a soil testing laboratory, but the majority of the work is generally carried out in the home laboratory with as much use of labour saving devices as the method of analysis permits. There is constant demand for methods which increase the speed of analysis without being prohibitively expensive. The purpose of this paper is to examine one recent technique--flow injection analysis, which was first proposed in 1975 by Ruzicka and Hansen.

This very simple method utilizes the rapid injection of a small volume of aqueous sample into a continuous moving, turbulently flowing, carrier stream of a reagent. If the speed of flow of the moving carrier stream is fast enough for turbulent rather than laminar flow, the injected carrier stream forms a zone which is then transported towards a detector, which continuously records the absorbance, or changes in electrode potential, etc. As the sampling period is very short, usually less than two seconds, a very high sampling rate can be achieved. This in turn allows the use of a quickly moving carrier which in contrast to the Autoanalyzer concept does not need to be segmented by air.

The beneficial effect of air segmentation is so obvious that the necessity of introducing air bubble was never really doubted although the drawbacks of its presence in the flowing stream are well known: (a) due to compressibility of air, the stream tends to pulsate rather than flow regularly, (b) streams have to be debubbled before entering the flow cell or before repumping, (c) the size of the air bubbles has to be controlled for faster sampling rates, (d) the pressure drop - and flow velocities - vary in the presence of air for various tubing materials (Chaney, 1967). Although it might appear that the role of the bubble is to divide the stream into a number of slugs which then do not mix, the main function of the air segments is in fact to cause wall friction with

resulting turbulent, rather than laminar flow even at low pumping velocities (Gerke and Ferrari, 1967). Yet it is known that at higher pumping rates, and with decreased diameter of tubing a sufficient turbulence will be produced to avoid the laminar flow which is responsible for carry-over. At conventional sampling rates, however, the consumption of the sample may exceed its availability and the consumption of the reagents would become uneconomical. The instant discrete sampling, on the other hand, with five times higher rate of analysis would still give the same reagent consumption even at five times faster reagent flow.

Ruzicka and Hansen (1975) showed that the Flow Injection Method had advantages over other analytical systems in both sampling rate and in the low cost and simplicity of the apparatus required. Later work (Ruzicka and Stewart, 1975, 1976) showed that the method could be applied to spectrophotometric determination of phosphorus, nitrogen, chlorine and to simultaneous nitrogen and phosphorus in plant digests and water samples at sampling rates ranging from 180 to 400 samples per hour.

The apparatus needed for the Flow Injection Method is very simple and consists of:

- 1) a peristaltic pump capable of pumping up to 10 mls/minute and with the possibility of pumping up to 5 tubes at once,
- 2) a flow through cuvette (similar to Helma type 178-QS which has a light path of 10 mm, volume 0.08 ml),
- 3) a spectrophotometer connected to a
- 4) recorder with the capability of a full scale deflection in less than 0.5 seconds,
- 5) a manifold made entirely of polyethylene and tygon tubing of the non-collapsible wall type of 0.95 to 1.20 mm i.d.

An example of the various manifolds used in P and N analyses are shown in Figure 1 (a) and (b). Similar simple manifolds are described for N and Cl in the references. Most colorimetric methods of analysis used in soil and plant analyses could be adapted to Flow Injection Analysis thereby increasing the analytical capability of many small laboratories. The cost of such adaptation would be small as most laboratories already have most of the equipment required. In addition to the published work ongoing research has demonstrated that the method also can be used in continuous dialysis and in conjunction with ion selective electrodes including the air gap electrode used in NH_3 analysis.

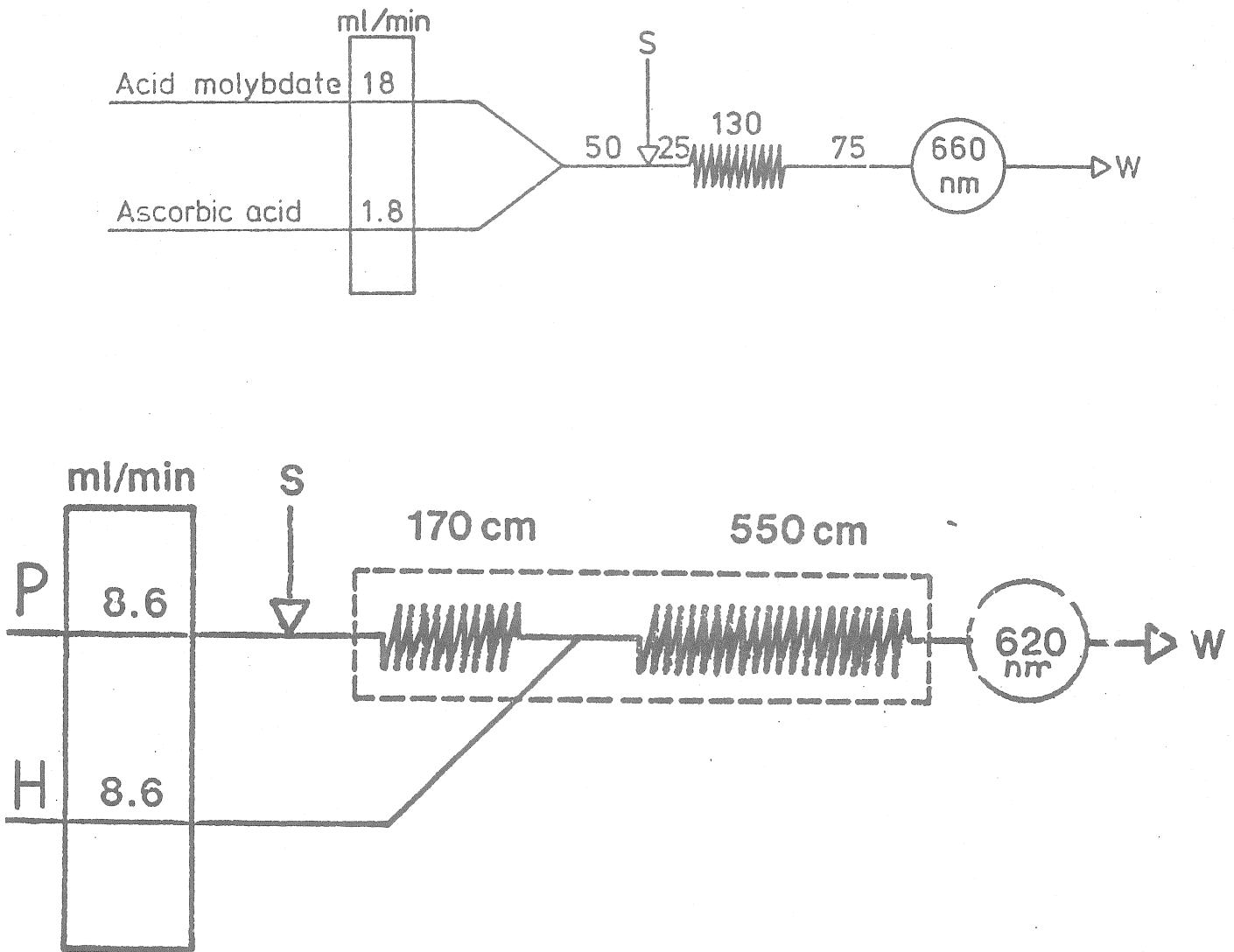


Figure 1. Flow Diagrams: (a) Phosphorus Manifold - Molybdate and ascorbic acid are pumped and jointly form a carrier stream into which 0.50 ml of sample (S) is injected. After passage through a mixing coil the absorbance of molybdenum blue is measured in a flow curvette at 660 nm and the stream continues to waste (W). The total length of the analytical line is 230 cm of which the coil constitutes 130 cm. The tubing had an inner diameter of ~ 1.00 mm. (b) Nitrogen Manifold - Alk. Phenol (P) and Hypochlorite (H) solutions are pumped at equal rates. S is the point of sample introduction (0.3 or 0.5 ml). The dotted line encloses the part of the manifold immersed in the water bath. Tubing as before.

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