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NEW METHODS OF ELEMENTAL ANALYSIS FOR SOILS AND PLANT TISSUES AT THE COLLEGE OF TROPICAL AGRICULTURE

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Among the several criteria for fertilizer recommendations elemental analysis is one of the most important. In the past elemental analysis was performed by wet chemical methods. Usually only one or at least a few elements were determined for each sample. Also, data for only the elements specifically determined was obtained. This meant that the fertilizer recommendation which was based on the elemental analysis was only as complete as the analysis itself allowed. The obvious answer, then, to the shortcomings of fertilizer recommendations based on elemental analyses was to determine all of the elements present in a sample. However, determining all of the elements in a sample by wet chemical methods meant first a complete qualitative analysis to see which elements were present and then a quantitative analysis for the elements which were found to be present. If this could be accomplished then the fertilizer recommendation could be based on the interaction of the elements present rather than the absolute concentration of a few elements.

Until recently, there were two main stumblingly blocks to the study of elemental interactions in soils and plant tissues. The first was the enormous amount of time and expense involved in making a complete analysis. Secondly, because of the time and expense involved in the accumulation of data, there has not been enough good data collected to statistically determine the implications of elemental interactions. Many researchers are now taking advantage of the newly established emission spectrographic services on the mainland. Emission spectroscopy offers a fast and inexpensive way to accumulate vast quantities of elemental data which can then be treated statistically. As data accumulates elemental interactions according to soils, crops, varieties, climate, etc. will become better understood.

Emission spectroscopy also has its drawbacks as well as its good points. For one thing the samples have to be passed into solution. Then because of strict upper limits of concentrations, many dilutions must be made before the actual analysis can be carried out. Because of this, sample preparation can be tedious, time consuming, and prone to errors. Also, like wet chemical analysis, emission spectroscopy is a destructive method of analysis, that is, the sample is consumed.

Another method of elemental analysis which has been in use for about twenty years now is x-ray fluorescence. As the sample is bombarded by x-radiation the elements making up the sample are induced to radiate x-rays whose energies and wavelengths are characteristic of the elements present; hence, the sample is caused to fluoresce x-rays. Because, for all practical purposes, the sample remaining the same after analysis as before, x-ray fluorescence is a non-destructive method of analysis. The sample can be analyzed by x-ray fluorescence over and over again and can then be either stored or used for some other purpose.

Until about two years ago, x-ray fluorescence instruments were capable of qualitatively scanning the elements, usually from aluminum through all of the elements on the periodic table beyond uranium. Particular elements were analyzed quantitatively one at a time. Although x-ray fluorescence was faster than wet chemical methods it was a slow procedure because each element had to be compared against chemically analyzed standards. Since standardization was a problem most x-ray fluorescent users stuck to the analysis of only one or two elements at a time.

This meant that elemental interactions were generally not studied by x-ray fluorescence.

Within the past two years a x-ray fluorescence instrument has been developed which will simultaneously analyze up to twenty-three elements. This completely automated instrument is called an x-ray Quantometer by the company who manufactures it. Although, the instrument costs about $100,000.00 its speed is such that on a per sample bases the cost is very low. For example, the cost of operation plus the amortization of the initial cost over five years comes to about $240.00 per working day. During a normal working day 240 samples can be analyzed at one dollar per sample. Assuming that fifteen elements for each sample were analyzed, there would be 1,500 elemental analyses made per day at a cost of six and two-thirds cents per element.

The Department of Agronomy and Soil Science recently purchased an x-ray Quantometer which is capable of simultaneously analyzing Na, Mg, Al, Si, P, S, K, Ca, Ti, Mn, Fe, Cu, Zn and Mo plus any other element above Mg on the periodic table. The instrument is also capable of qualitatively scanning for all elements on the periodic table above Mg. Sample preparation for the x-ray Quantometer is relatively simple. The main requirements for samples are: they must be solid, their size can vary from a minimum of 1 1/4" to a maximum of 2 3/8" in diameter and can be any thickness up to 1 3/8", the samples' surface must be flat, smooth and representative of the elemental composition of the entire sample, and finally, the sample must be stable in a vacuum because the analyses are carried out under a vacuum.

For most routine plant tissue analyses the sample is dried at a standard temperature, finely ground, pelletized and analyzed. Most soil analyses will be handled in a similar manner. Once the sample is automatically taken into the Quantometer the actual analysis takes about thirty seconds. For improved statistics resulting in greater accuracy the sample may be analyzed as many times as the operator wishes. At the conclusion of each analysis a number representing the concentration of each element is printed out by a teletype while at the same time the data is punched into a paper tape.

The operator can inspect the data on the teletype at anytime, however, since the teletype data only represents the elemental concentrations and are not the actual concentration, it is used to insure flawless operation of the instrument and for future reference. The punched paper tape, on the other hand, is used to automatically punch computer cards. Because of a phenomenon called matrix effects each element in the sample effects the analysis of every other element. Therefore, the elemental concentrations are not proportional to the size of the numbers representing each element. At the end of each day as many as 1,500 elemental analyses may be completed, most of which have been completed three or more times to get good statistics. The staggering quantity of data each day coupled with the fact that each element for each analysis must be mathematically treated with respect to every other element present, makes the use of a computer imperative.

For the first time, the College of Tropical Agriculture will not be burdened with collection elemental data but will be confronted with the analysis of the data. We visualize setting up a data bank with a computerized retrieval system. Then present and past data will be statistically treated so that elemental interactions will be more thoroughly understood. In the future we hope to set up computer programs which will use the data from a farmer's crop and/or soil to directly print out a fertilizer recommendation. With the speed of the system

the recommendation should be in the hands of the farmer only a few days after he has submitted the samples for analysis.

Unfortunately, the Quantometer is not the complete answer to the problems of elemental analysis. For example it is incapable of analyzing elements below sodium on the periodic table. This means that nitrogen, carbon and boron must still be analyzed by other means. The Quantometer cannot determine the valence states of the elements or how they are atomically bound. Only after an exhaustive study of a large accumulation of data will the actual availability of essential plant nutrients be inferred.

The Quantometer is a fast and accurate elemental analyzer of macro (large or bulk) samples. There is vast knowledge to be learned from the elemental analysis of micro (very small) samples. As a few examples of micro analysis: The root soil interface, root hair analysis, the effectiveness of foliar sprays, effects of insect damage, etc. To do this type of analysis the University of Hawaii is expecting in the near future to install a scanning electron microscope with an energy dispersive x-ray analyzer. In essence the energy dispersive x-ray analyzer will have capabilities similar to the Quantometer only on a micro basis. The analyzer will quantitatively detect all elements from sodium to uranium on the periodic table. All elements within that range will be simultaneously "seen". Since a whole soil aggrate, a whole leaf, insect, etc. can be inserted into the instrument, the elemental study is actually done on an <u>in siter</u> (in place) basis.

With a scanning electron microscope the actual positions or distributions of the elements on a sample's surface can be visualized. By visualized we mean that if desired a micrograph of a given sample area can be taken and then each element of interest can be visualized one at a time over the same sample area. Just as an example, it has been generally believed that sugar cane leaf speckle was caused by silicon deficiency resulting in manganese toxicity. However, the scanning electron microscope with an energy dispersive x-ray analyzer has shown that the speckle was very high in silicon concentration and the manganese concentration did not seem to vary on or off the speckle. We have no ready explanation for this phenomenon at this time but it is sure that sugar cane leaf speckle will be investigated in great detail as soon as the scanning electron microscope becomes operational.