

THE USE OF THE BECKMAN FLAME SPECTROPHOMETER IN PLANT AND SOIL ANALYSIS

JOSEPH Q. SNYDER, The Samuel Roberts Noble Foundation, Ardmore

Flame spectra have been used for nearly a hundred years for qualitative analyses, but it was not until the work of Lundegardh (3) in 1929 that they were applied to any extent to quantitative analyses. In 1939, Griggs (2) published the first results of any such application in this country and it has been only in the last few years that the technique of flame photometry has come into general use in the United States.

An excellent discussion of the Beckman Flame Spectrophotometer is presented in a publication by Gilbert, Hawes, and Beckman (1). Several papers (4, 5, 8) have described the application of this instrument to the analysis of various materials. Toth and Prince (6, 7) have described the application of the Perkin-Elmer model 52-A flame photometer to analyses of sodium, potassium, and calcium in soils and plants. The present paper describes the methods in use in this laboratory for the past two years for the analysis of sodium, potassium, calcium, and magnesium in soils and plants using the Beckman instrument.

ANALYTICAL PROCEDURE

The first step in the analysis is the preparation of a solution containing the ions to be analyzed in suitable concentration ranges and as free as possible from interfering substances.

In the preparation of soil extracts, a 6 gm. portion of ground, air-dry soil is placed in a Buchner type funnel with a Corning medium porosity sintered glass plate. This sample is then leached with 50 ml. of a neutral 1 N ammonium acetate solution. The filtrate is suitable for direct flame analysis without further treatment except in the case of very unusual (generally saline) soil.

Plant digests are prepared by a method similar to that of Toth, *et al* (6). Samples are first air dried, ground to 40 mesh, and oven dried to constant weight at 105°C. A sample weighing between 1.8 and 2 gm. is placed in a Pyrex Kjeldahl flask and 15 ml. of concentrated nitric acid are added and allowed to react at room temperature. After the frothing has passed its peak, the flask is heated gently in a hood until the froth subsides. Increased heat is then applied until all the original particles are destroyed and the volume is reduced to about 3 ml. A gelatinous precipitate is present at this point and the solution is an amber to brown color. Five ml. of 70-72% perchloric acid are added and the flask heated until the nitric acid fumes are driven off, the perchloric acid is fuming and the volume is reduced to about 1 ml. Twenty-five ml. of distilled water are added, the solution brought to a good boil and filtered immediately through S & S No. 589 Blue Ribbon filter paper into a 50 ml. volumetric flask, washing residue and filter with boiling water. The solution is cooled to 25°C. and made to volume. If potassium perchlorate precipitates on cooling, the perchlorate may be reduced, the solution may be diluted to about twice the volume required to effect solution, or the sample may be re-ashed using less plant material. The final solution is checked by the flame method and, if necessary, suitable dilutions are prepared for the analyses of the various metallic ions.

The Beckman Flame Spectrophotometer utilizes an oxygen-gas flame into which the sample is sprayed by compressed air. In this laboratory natural gas with a 1050 BTU rating is used at a pressure of 2 cm. of isopropanol. The oxygen pressure which gives the best results is 12 to 14 inches of water and the air pressure used is 19 pounds per square inch. The narrowest slit width is used that will give at high sensitivity a full scale reading with the most

concentrated standard used. The approximate wave lengths (in millimicrons) used for the various determinations are: potassium, 767; calcium, 626; sodium, 589; and magnesium, 370.8. The wave length of maximum emission is checked before each run since changes in the placing of the spectrophotometer and in mirror settings will cause it to vary slightly.

Standard curves are run each time the instrument is operated and points on these curves are checked periodically during the running of samples. Standards containing typical amounts of interfering ions are checked and the amount of interference computed. Concentrations are determined from the standard curves and corrected for interference.

DISCUSSION

The use of this method for soil analysis has been very successful in this laboratory. It has given results comparable to those obtained by other methods and the time required and the costs have been considerably less.

Using flame photometry, The Samuel Roberts Noble Foundation has been able to make a large number of analyses of field-grown crops raised under various conditions and treatments in south-central Oklahoma. In the course of other research, large numbers of plants raised in sand culture have been rapidly and accurately analyzed.

As a check on this over-all method of ashing and analyzing plants, an experiment was run in which the analyses of plant samples fortified with known amounts of various salts were compared with the analyses of duplicate samples receiving no fortification. The results of this experiment are shown in Table I.

TABLE I
Recovery of Metallic Ions from Fortified Plant Samples

Sample	SUDAN GRASS		LOVE GRASS		BERMUDA GRASS		AVERAGE % RECOVERED
	1.6826	1.5384	2.2383	1.5406	1.4215	1.2997	
CALCIUM:							
Fortified	.0097	.0092	.0068	.0064	.0063	.0061	
Unfortified	.0038	.0035	.0010	.0007	.0004	.0004	
Recovered	.0059	.0057	.0058	.0057	.0059	.0057	
Added	.0060	.0060	.0060	.0060	.0060	.0060	
% Recovered	98%	95%	97%	95%	98%	95%	96%
MAGNESIUM:							
Fortified	.0040	.0042	.0042	.0040	.0041	.0040	
Unfortified	.0002	.0002	.0003	.0002	.0002	.0002	
Recovered	.0038	.0040	.0039	.0038	.0039	.0038	
Added	.0040	.0040	.0040	.0040	.0040	.0040	
% Recovered	95%	100%	98%	95%	98%	95%	97%
SODIUM:							
Fortified	.0015	.0014	.0017	.0015	.0024	.0024	
Unfortified	.0005	.0005	.0007	.0005	.0014	.0013	
Recovered	.0010	.0009	.0010	.0010	.0010	.0011	
Added	.0010	.0010	.0010	.0010	.0010	.0010	
% Recovered	100%	90%	100%	100%	100%	110%	100%
POTASSIUM:							
Fortified	.0094	.0092	.0125	.0098	.0104	.0097	
Unfortified	.0058	.0053	.0087	.0060	.0065	.0059	
Recovered	.0036	.0039	.0036	.0038	.0039	.0038	
Added	.0040	.0040	.0040	.0040	.0040	.0040	
% Recovered	90%	98%	95%	95%	98%	95%	95%

(All weights are in grams)

SUMMARY

A method of routine soil and plant analysis involving the use of the Beckman Flame Spectrophotometer is described. The soils are leached with neutral 1 N. ammonium acetate and the leachate analyzed with this instrument. The plant samples are ashed with nitric and perchloric acids and the digest, diluted if necessary, analyzed in the same way. Concentrations are determined from standard curves with corrections applied for interfering ions.

The author gratefully acknowledges the work of the staff of The Samuel Roberts Noble Foundation Laboratory in running by this method the large number of analyses which stimulated the inquiries that prompted the presentation of this paper. The assistance of Mr. Brown W. Scott, Mr. M. K. Patterson, Jr., and Mr. Bob Thompson has been especially helpful.

BIBLIOGRAPHY

1. GILBERT, P. T., JR., HAWES, R. C. and BECKMAN, A. O. 1950. Beckman flame spectrophotometer. *Anal. Chem.* 22: 772-780.
 2. GRIGGS, M. A. 1939. Use of the Lundgardh spectrographic method. *Science* 89: 134.
 3. LUNDGARDH, H. 1929 (Part I) and 1934 (Part II), *Die quantitative Spektralanalyse der Elemente*. Jena: Gustav Fischer.
 4. MOSHER, R. E., et al. 1949. The use of flame photometry for the quantitative determination of Na and K in plasma and urine. *Am. J. Clin. Path.* 19: 461.
 5. ———, BIRD, E. J., and BOYLE, A. J. 1950. Flame photometric determination of calcium in brucite and magnesite. *Anal. Chem.* 22: 715-717.
 6. TOTH, S. J., PRINCE, A. L., WALLACE, A., and MIKKLESON, D. S. 1948. Rapid quantitative determination of eight mineral elements in plant tissue by a systematic procedure involving use of a flame photometer. *Soil Sci.* 66: 459-466.
 - 7: ——— and ———. 1949. Estimation of cation-exchange capacity and exchangeable Ca, K, and Na contents of soils by flame photometer techniques. *Soil Sci.* 67: 439-445.
 8. WEST, P. W., FOLSE, P., and MONTGOMERY, D. 1950. Application of flame spectrophotometry to water analysis, determination of sodium, potassium, and calcium. *Anal. Chem.* 22: 667-670.
-