

DEVELOPMENT AND TESTING IN THE LABORATORY AND GREENHOUSE OF A
METHOD USING TETRAPHENYLBORON FOR THE DETERMINATION
OF EXTRACTABLE SOIL POTASSIUM

By

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TABLE OF CONTENTS

Chapter	Page
I. INTRODUCTION	1
II. REVIEW OF LITERATURE	3
Potassium Fixation	3
Extraction of Soil Potassium	3
Measurement of Extracted Potassium	4
Flame photometry	5
Cobaltinitrite methods	6
Tetraphenylboron methods	7
III. MATERIALS AND METHODS	10
Soils	10
Analytical Methods	11
Greenhouse Study	12
Methods of Statistical Analysis	12
IV. RESULTS AND DISCUSSION	16
Development and Testing of the Tetraphenyl-	16
boron Method	
Method of addition of tetraphenylboron	16
Time-temperature relationships	17
Reaction of medium	17
Interferences	20
Extract: precipitating agent ratio	22
Proposed procedure	25
Preparation of solutions	25
Comparison of tetraphenylboron and flame	
photometer methods	26
Greenhouse Experiment	33
V. SUMMARY AND CONCLUSIONS	44
VI. LITERATURE CITED	46
APPENDIX	49

LIST OF TABLES

Table	Page
I. Soil Characteristics as Determined by Laboratory Analyses	13
II. A Comparison of Soil Potassium Values Obtained by the Tetraphenylboron and Beckman Flame Photometer Methods for Six Extracting Agents (5:1 Solution-Soil Ratio)	27
III. A Comparison of Soil Potassium Values Obtained by the Tetraphenylboron and Beckman Flame Photometer Methods for Six Extracting Agents (7:1 Solution-Soil Ratio)	28
IV. A Comparison of Soil Potassium Values Obtained by the Tetraphenylboron and Beckman Flame Photometer Methods for Six Extracting Agents (10:1 Solution-Soil Ratio).	29
V. Relationship of Kinds and Concentrations of Extracting Agents to Divergence Between Tetraphenylboron and Beckman Flame Photometer Analyses . .	31
VI. Recovery of Potassium Added to Extracts by Tetraphenylboron and Flame Photometer Analyses	32
VII. Dry Matter Yields of Sugar Drip Forage Sorghum in a Greenhouse Experiment	34
VIII. Analysis of Variance of Forage Sorghum Yields	36
IX. Duncan's New Multiple Range Test of Forage Sorghum Yields	37
X. Milligrams of Potassium Removed by Sugar Drip Forage Sorghum in a Greenhouse Experiment	39
XI. Analysis of Variance of Milligrams of Potassium Removed by Forage Sorghum Plants	41
XII. Duncan's New Multiple Range Test of the Milligrams of Potassium Removed by Forage Sorghum Plants	42
XIII. Profile Descriptions of Soils	50

LIST OF FIGURES

Figure	Page
1. Influence of Method of NaTPB Addition on Potassium Determination with Tetraphenylboron	18
2. Time-Temperature Relationships in the Potassium Determination with Tetraphenylboron	19
3. Effect of Ammonium Ions and Ammonium Ions plus Formaldehyde on Potassium Determination with Tetraphenylboron	21
4. Calcium and Magnesium Interference in Potassium Determination with Tetraphenylboron	23
5. Standard Curve for Potassium Determination with Tetraphenylboron	24

I. INTRODUCTION

The soil is the basic medium for agriculture production. It is the task of the soil scientist to elucidate facts about the soil which will enable him to suggest courses of action to improve soil fertility and productivity. Soil productivity, as a basis of overall agriculture productivity, encompasses many fields of soil study. Not only is the soil scientist concerned with the nutrients present with the soil, but also soil structure, soil morphology, and with various other dynamic systems which influence soil water and aeration relationships. All these factors influence the productivity of the soil. However, most of them can be little more than characterized and the limitations enumerated. Soil fertility is often defined as the ability of the soil to supply mineral nutrients to plants. It is one soil characteristic which may be altered with little difficulty by the addition of essential nutrients to deficient soils.

The soil chemist is concerned with methods of measuring the amounts of essential soil nutrients which may be absorbed by plants. To the layman this would appear to present a rather simple task. To one familiar with soils, the process of quantitatively determining the usable forms of an element essential for plant growth, presents a formidable undertaking. Soil reactions of elements, which are altered by complex forces and phenomena, differ markedly in certain aspects from simple laboratory reactions.

Potassium is classified as one of the macro essential nutrients. It is placed in this major category because of the quantity utilized by plants and because it is deficient in many soils, particularly the coarse

textured ones and those found in humid regions. The determination of available potassium is complicated not only by factors inherent within the soil system, but also by its extremely soluble nature in numerous compounds. This investigation is concerned with the removal of extractable potassium from the soil and the quantitative evaluation of the element in the soil extract. The overall objective is the development of a simple, quick analytical procedure for the determination of potassium in soil extracts.

II. REVIEW OF LITERATURE

Potassium Fixation

The determination of potassium in a soil system is complicated by several factors. One of these is the dynamic state of this element when associated with clay mineral colloids. In a laboratory study, Volk (30)¹ found that as much as 87% of the soluble potassium added to a silty clay soil was converted to a non-extractable form when the soil was alternately wetted and dried. DeTurk et al. (7) stated that potassium fixation was gradual even under moist conditions and that an equilibrium was attained within six months. This equilibrium was altered by moisture content of the soil, soil temperature, and various other factors which could not be controlled. The effect of these soil conditions is very difficult to estimate. Therefore, the estimation of extractable potassium in the soil system can only be partially controlled.

Extraction of Soil Potassium

Another factor of extractable potassium estimation is the extraction process. The extraction is the most important controllable aspect of potassium determination. The amount of potassium which may be extracted varies with the extracting reagent and its concentration. In some instances, the amount also varies with soil-solution ratio and the time of extraction.

Mitchell (15) considered the various extracting reagents commonly used and theorized that normal ammonium acetate is the optimum reagent.

¹Numbers in parenthesis refer to Literature Cited.

His choice was based upon the fact that the ammonium ion is quite similar to the potassium ion in both size and activity. Also, the acetate anion apparently gives little interference with cation determination. The most important criterion in the choice of extracting reagent is determined by plant response. The extracting reagent utilized should remove an amount of potassium from the soil which can be correlated with plant growth. The amount will not be the same for all extracting reagents. However, for a specific reagent one should be able to correlate plant growth and extractable potassium. If this is accomplished, the researcher is justified in using adjective ratings (high, low, and medium etc.,) for soil potassium values.

Voluminous research has been reported concerning plant growth responses in relation to soil tests and fertilizer additions. The most widely known research is concerned with various modifications of the Mitscherlich theory. Mitscherlich's (14) original work was an attempt to relate plant growth to soil nutrient content. The theory was expressed as a mathematical equation. This equation was derived to fit collected data. Bray (5) found a modification of the original Mitscherlich equation more tenable in the case of potassium. On both fertilized and unfertilized soils he reported that a curvilinear relationship exists between replacable potassium and increases in corn yields with potassium fertilization. Magistad et al. (13) have reported data concerning fertilizer applications and the law of diminishing returns.

Measurement of Extracted Potassium

The third factor in the estimation of extractable potassium is the quantitative determination of the element in the soil extract. The fore-

most difficulty in measurement is implied by Jackson (12) when he explained that even though potassium is fixed very readily in soils, most of the compounds of this element are quite soluble in aqueous solution. Thus, the researcher is limited to a small number of insoluble potassium salts, if he uses a gravimetric or turbidimetric procedure.

Flame Photometry

Apparently, the most expedient and precise method for the determination of potassium in solution is emission spectrophotometry. According to Jackson (12), most manufacturers of emission apparatus claim one to three per cent accuracy for the determination of potassium. Jackson stated that this accuracy is dependent upon four main factors:

- (1) Instrumental precision
- (2) Uniformity between standard and test sample
- (3) Amount of the element available for analysis
- (4) The element to be determined.

Of these factors, the second one is the most difficult to control when soil extracts are analyzed.

The factors which limit the wide-spread use of emission spectrophotometry in the determination of soil cations are the cost of the apparatus plus the skill required to maintain and operate the instrument. In smaller laboratories, such as the county soil testing laboratories in Oklahoma, a less expensive method is required. Several gravimetric and turbidimetric methods have been proposed.

Cobaltinitrite Methods

The most popular methods of gravimetric and turbidimetric potassium analysis involve various modifications of the cobaltinitrite precipitation of potassium as an insoluble potassium cobaltinitrite (4, 16, 17). The cobaltinitrite methods are widely accepted and used.

Sodium cobaltinitrite is commonly used as the reagent for the potassium determination. It is a tri-sodium salt of the trivalent cobaltinitrite complex with the chemical formula $\text{Na}_3\text{Co}(\text{NO}_2)_6$ and is soluble in water. The corresponding potassium salt, $\text{K}_3\text{Co}(\text{NO}_2)_6$, is slightly soluble in water and insoluble in alcohol. In the precipitation of potassium with cobaltinitrite, potassium substitutes for sodium in the molecule yielding an insoluble salt with the empirical formula $\text{K}_n\text{Na}_{3-n}\text{Co}(\text{NO}_2)_6$. The fact that the ratio of K to Na may vary, as indicated by the empirical formula, is an objection to the use of cobaltinitrite. Jackson (12) stated that the upper limit of sodium content in these insoluble salts appears to be approximately a 1:1 ratio of K to Na. As this ratio varied from any assumed value for the standard solutions, the determination became less accurate. The substitution of potassium for sodium varied with temperature and became quite erratic at temperatures above 30°C. Temperature control was mandatory for accurate results. Published data (4, 16, 17) indicated that the various cobaltinitrite methods gave tenable results when the temperature was controlled carefully and skilled technicians made the determination. In the county laboratories in Oklahoma, temperature control is difficult and the technicians are not skilled.

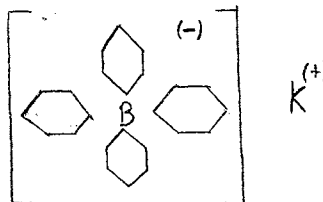
Since the cobaltinitrite procedure has these disadvantages, the development of an inexpensive method of potassium determination is necessary. The method should be insensitive to temperature and adaptable to

use by unskilled technicians.

The Tetraphenylboron Methods

In 1949, Wittig et al. (31), developed the tetraphenylboron complex. Lithium tetraphenylboron ($\text{Li}(\text{C}_6\text{H}_5)_4\text{B}$) was found to precipitate potassium quantitatively. Thus, these workers opened the way for development of several new procedures for potassium determination.

Sunderman and Sunderman (28) gave the following structural formula for potassium tetraphenylboron:



Ievins et al. (11) stated that the crystal configuration for potassium tetraphenylboron was a tetragonally bipyramidal form. The tetragonal lattice had two molecules per cell.

During the past thirteen years, several methods of potassium determination using the tetraphenylboron complex have been proposed (2, 6, 8, 19, 20, 21, 22, 24, 28, 29). These methods usually involve the sodium salt of the complex. Many of the characteristics and interferences of the precipitate formation have also been enumerated.

The suitability of the tetraphenylboron complex for potassium determination may be attributed to the striking dissimilarity of the solubilities of the sodium and potassium salts. The sodium salt of the complex (NaTPB) is quite soluble in aqueous solution, whereas the potassium salt (KTPB) is quite insoluble. Geilmann and Gebauhr (8) calculated the solubility product of KTPB as 2.25×10^{-8} moles/liter at 20°C whereas Rudorff and Zannier (23) gave a value of 3.3×10^{-8} at 25°C .

Gloss (9) listed the following characteristics of the potassium tetraphenylboron precipitate.

- (1) The salts of the tetraphenylboron complex are well defined. The potassium salt corresponds exactly to the formula $K(C_6H_5)_4B$.
- (2) The KTPB salt is much less soluble than other salts used in potassium analyses.
- (3) The KTPB salt is stable to heat.
- (4) The high equivalent weight yields an optimum gravimetric conversions factor.

These characteristics make tetraphenylboron extremely suitable for potassium determinations.

There are several other elements and ions which form insoluble tetraphenylboron salts. Phlaum and Howick (19) stated that ammonium, cesium, mercury, rubidium, silver and thallium form insoluble tetraphenylboron salts. The only one of major consequence in soils is the ammonium ion. It has been suggested by Berkhout and Jongen (2) that formaldehyde will prevent the formation of the insoluble ammonium tetraphenylboron. Amin (1) found that additions of formaldehyde to remove ammonium interference were not completely satisfactory. Certain organic amines and amides may also form insoluble tetraphenylboron salts (19). Some interference in KTPB formation is inherent in solutions containing large amounts of calcium and magnesium. Berkhout and Jongen (2) stated that use of ethylenediaminetetracetic acid (EDTA) would alleviate these interferences. Amin (1) indicated that EDTA completely masked the interference due to chromium, manganese, ferric iron, cobalt, nickel, copper, zinc, cadmium, silver, lead, aluminum, and bismuth.

The several methods presented for potassium determination using tetraphenylboron include gravimetric (8), volumetric, (2, 22, 24), conductometric (21) and turbidimetric (6, 20, 28, 29). The most desirable type of analysis for soil extracts is the turbidimetric, since most laboratories are equipped with a colorimeter which may be adapted to this

type of determination.

De La Rubia and Blasco (6) have presented a turbidimetric method adapted to soils. Their method utilizes the ammonium acetate extract. Power and Ryan (20) reported that they were unable to reproduce the results obtained by De La Rubia and Blasco. Teeri and Segin (29) devised a turbidimetric procedure for potassium estimation in blood serum using a three per cent NaTPB solution in a buffered system. Power and Ryan (20) presented a rather simple turbidimetric procedure. Sunderman and Sunderman (28) gave a detailed investigation of various aspects of turbidimetric potassium determination using NaTPB. Their work involved the use of a five per cent solution of alkaline Na TPB, EDTA, and formaldehyde and is the most comprehensive article published to date. The results obtained by these workers have been readily reproducible with a few modifications.

III. MATERIALS AND METHODS

Soils

Seven soils were selected for this investigation. These soils are representative of series which cover rather extensive areas of Oklahoma. Care was taken to choose soils which should be low in extractable potassium. The texture of the soils varies from sand to clay loam.

Six soils were selected from the central and southeastern part of Oklahoma where rainfall is relatively high. It was assumed that the potassium content of these soils would be low as compared to the soils of more arid areas in western Oklahoma. One extremely coarse textured soil, Brownfield (Miles), was taken in southwestern Oklahoma where rainfall is generally low.

The soils collected were: Brownfield (Miles) sand, Greer county; Bowie sandy loam, Atoka county; Dennis silt loam, McIntosh county; Durant clay loam; Bryan county; Linker loam, Pittsburg county; Parsons clay loam, Muskogee county; Stephenville loamy sand, Lincoln county. The profile descriptions and site locations of the soils used are listed in the appendix.

Approximately 250 pounds of topsoil from each site were brought to Stillwater. These samples were air-dried, processed, and stored in plastic bags until used in the greenhouse and laboratory studies.

Analytical Methods

The soils were characterized by the following chemical and physical measurements: The pH was determined with a Beckman glass electrode pH meter on a 1:1 soil-water paste. The organic matter percentage was obtained by the potassium dichromate wet oxidation method of Schollenberger (25). Total nitrogen content was determined by the Kjeldahl method according to Harper (10). Cation exchange capacity was measured by the ammonium saturation and distillation method of Peech et al. (18). The exchangeable cations (Ca, Mg, K, Na) were determined in the ammonium acetate leachate by use of the Beckman DU Flame Spectrophotometer with photomultiplier. Total phosphorus determinations were accomplished by perchloric acid digestion and the development of the molybdate color complex according to the procedure of Shelton and Harper (26). For total potassium, calcium, magnesium and sodium determination, the soils were digested with perchloric acid and the elements were measured with the Beckman DU Flame Spectrophotometer. Available phosphorus was determined by extraction with 0.1 N acetic acid and the development of the molybdate color complex (10). Mechanical analysis of the soils was accomplished by the Bouyoucos method (3). The results of these analyses are given in Table I.

Extractable potassium was measured by modifications of the procedure reported by Sunderman and Sunderman (28) and the Beckman DU Flame Spectrophotometer. Six extracting reagents (0.1, 0.5, and 3.0 N sodium acetate plus 0.1, 0.5 and 3.0 N sodium nitrate) and three soil-solution ratios (1:5, 1:7, and 1:10) were used.

Greenhouse Study

An experiment was designed to ascertain the affect of four potassium levels on the seven soils collected. The potassium treatments were comprised of 0, 5%, 10%, and 15% of the cation exchange capacity of each soil. The experiment was arranged in a completely randomized block design with three replications of each treatment. The pots were then fertilized with the equivalent of 160 pounds of nitrogen and P_2O_5 per acre and lime was added to 75% of the cation exchange capacity. After fertilization, the soils were allowed to equilibrate moist for six weeks.

Sugar Drip forage sorghum was planted in these pots on October 20, 1961. The sorghum was thinned to four plants per pot when the stand was established. Four harvests were taken from these pots on January 2, February 27, April 18, and May 31. To improve the growth and vigor of the plants, the equivalent of 50 pounds per acre of P_2O_5 was added to all pots after the second harvest.

The plants were harvested by cutting the stalks approximately three inches above the ground. The samples were brought to the laboratory, oven-dried at $90^{\circ}C$, and weighed. The replications were composited, ground, and subsequently digested in nitric-perchloric acid. The cations were determined in the plant ash by means of the Beckman DU Flame Spectrophotometer and photomultiplier.

Methods of Statistical Analysis

Statistical analyses were made on the plant dry weights and the milligrams of potassium removed from the soil by four harvests. The procedure listed by Steele and Torrie (27) for analysis of variance of completely randomized block experiments was followed. There were 28

TABLE I
SOIL CHARACTERISTICS AS DETERMINED BY LABORATORY ANALYSES

Soil	pH	% O. M.			Avail P (Lbs./A)	Cation Exchange Capacity	Exchangeable		
		Total N	Total P				Ca	Mg	Na
Bowie	5.5	1.48	0.06	0.024	7.9	2.5*	1.3	0.9	0.11
Brownfield (Miles)	6.6	0.27	0.02	0.020	38.9	1.4	1.3	0.9	0.04
Dennis	5.8	2.25	0.11	0.048	3.3	8.1	4.3	3.8	0.09
Durant	5.5	2.36	0.11	0.060	25.7	15.1	8.3	4.6	0.35
Linker	5.7	1.40	0.08	0.036	3.7	4.8	2.8	1.4	0.13
Parsons	5.4	2.80	0.14	0.072	5.0	12.4	5.0	5.2	0.26
Stephenville	6.1	1.31	0.06	0.028	2.5	3.9	2.6	1.4	0.09

*Cation exchange capacity and exchangeable Ca, Mg, and Na values are expressed as me. per 100 grams.

TABLE I (CONTINUED)
SOIL CHARACTERISTICS AS DETERMINED BY LABORATORY ANALYSES

Soil	%				Sand	%		Soil Class
	Total Ca	Total Mg	Total Na	Total K		Silt	Clay	
Bowie	0.08	0.29	0.07	0.12	71	22	7	sandy loam
Brownfield (Miles)	0.12	0.33	0.06	0.22	93	4	3	sand
Dennis	0.15	0.86	0.08	0.44	37	54	17	silt loam
Durant	0.26	0.78	0.09	0.48	25	48	27	clay loam
Linker	0.08	0.50	0.07	0.34	47	38	15	loam
Parsons	0.18	1.00	0.08	0.41	21	50	29	clay loam
Stephenville	0.12	0.38	0.08	0.24	81	10	9	loamy sand

different soil-potassium combinations for each of these analyses. This data was also examined for significant differences using Duncan's New Multiple Range test (27).

IV. RESULTS AND DISCUSSION

The current investigation has included both laboratory and greenhouse studies. The primary objective of the laboratory investigation was the development and testing of a new quick-test method for determination of extractable potassium using sodium tetraphenylboron (NaTPB) as a precipitating reagent. The greenhouse study was intended to ascertain plant response to potassium additions.

Development and Testing of the Tetraphenylboron Method

Many factors must be considered in the development of new analytical procedures. The influence of these factors must be determined and adverse affects and interferences must be enumerated along with techniques for their control. The following is a discussion of several factors which were found pertinent in achieving accurate, reproducible results in potassium analysis when sodium tetraphenylboron is used as the precipitating reagent.

Method of Addition of Tetraphenylboron

Sunderman and Sunderman (28) have suggested the use of a five percent solution of alkaline sodium tetraphenylboron (NaTPB) as a precipitating agent. For best results, the writer has found that the sodium tetraphenylboron must be forcefully injected into the precipitation medium. Sunderman and Sunderman have also noted this phenomenon and they used a Folin blow-out pipette to add NaTPB. A five ml. hypodermic syringe is less awkward than a pipette and facilitates precipitation

more rapidly. A comparison of three methods of NaTPB addition is shown in Figure 1.

Time-Temperature Relationships

Time of precipitation was reported as a variable in the use of sodium tetraphenylboron for potassium analysis (28). This has been verified, to some extent, by the present work. The important aspect appears to be a time-temperature interaction. Precipitation reached a maximum within five minutes after injection of the sodium tetraphenylboron if the medium was near 30°C. However, when the standard solutions or media were kept at approximately 25°C, maximum precipitation requires 15 minutes. Since winter to summer room temperatures vary from approximately 25°C to 35°C, it is suggested that 15 minutes be allowed as a precaution to insure maximum turbidity formation. The time-temperature interactions for potassium standards in 0.1 N sodium acetate are graphically represented in Figure 2.

Reaction of Medium

In an acid medium, Sunderman and Sunderman (28) found that the turbidity diminished after approximately 30 minutes. The present work in an alkaline medium showed no noticeable change in turbidity within two hours if the precipitation medium was diluted to ten ml. within 15 minutes after turbidity formation. Acid soil extracts are somewhat difficult to analyze using sodium tetraphenylboron. In an acid medium, potassium tetraphenylboron floccules were observed soon after turbidity formation. These floccules had a tendency to settle out of solution. This necessitated frequent mixing of the turbid solution to estimate the turbidity. If the floccules were kept in solution, further difficulty was encounter-

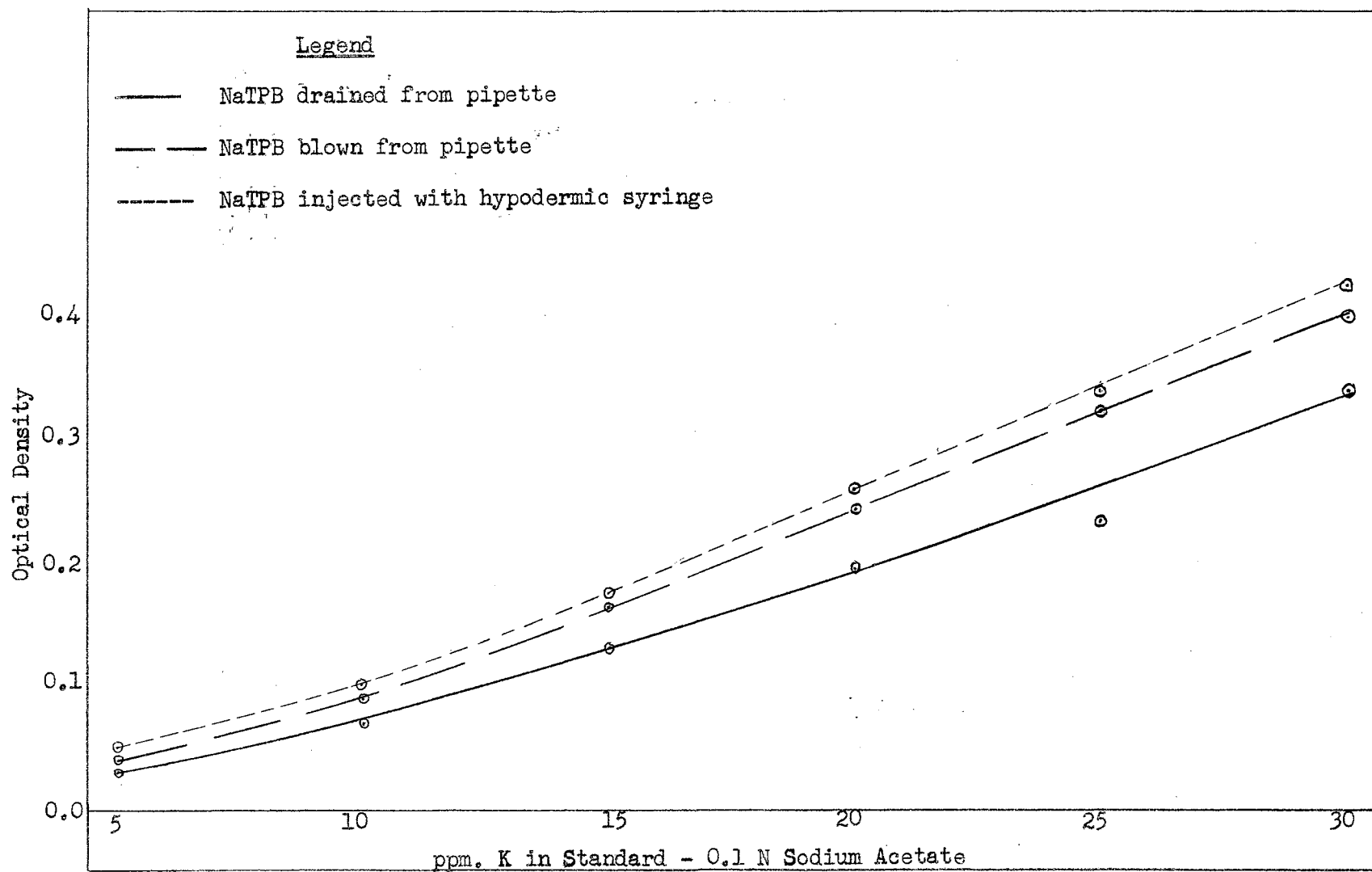


Figure 1. Influence of Method of NaTPB Addition on Potassium Determination with Tetraphenylboron

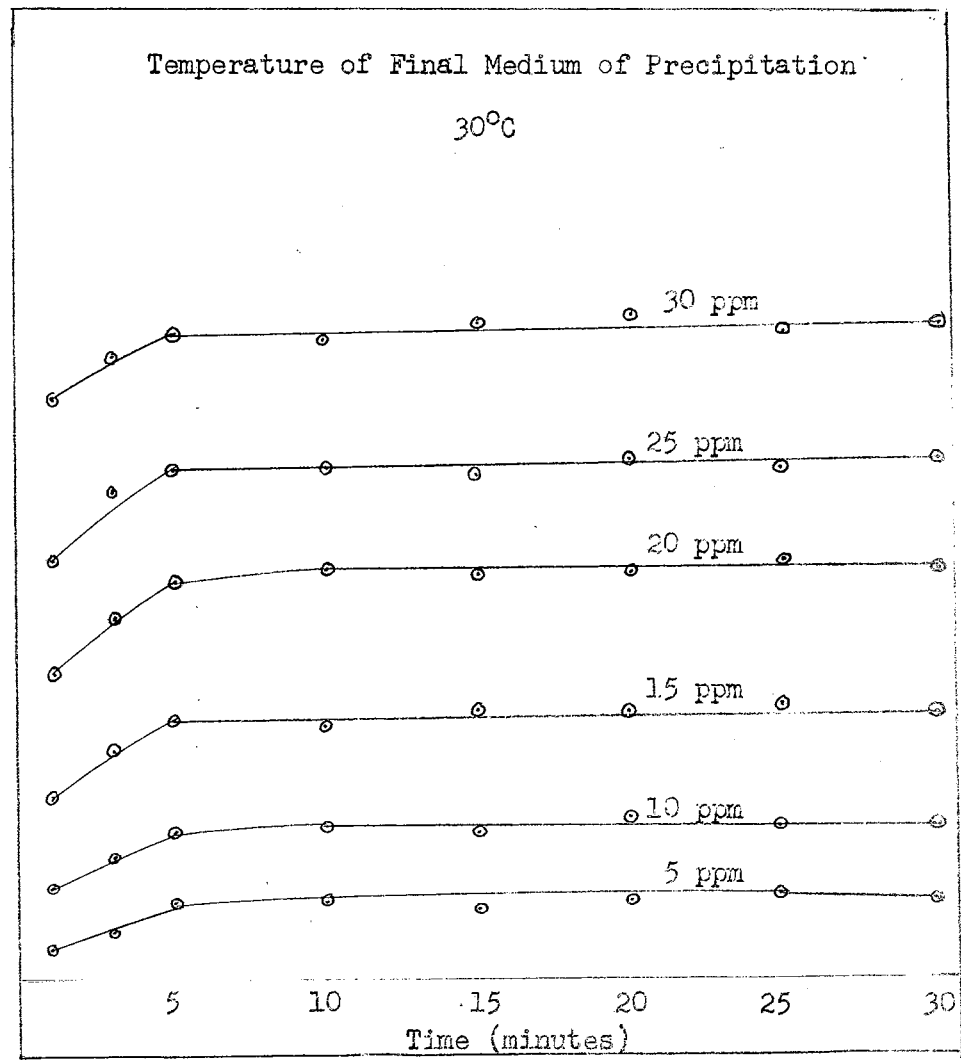
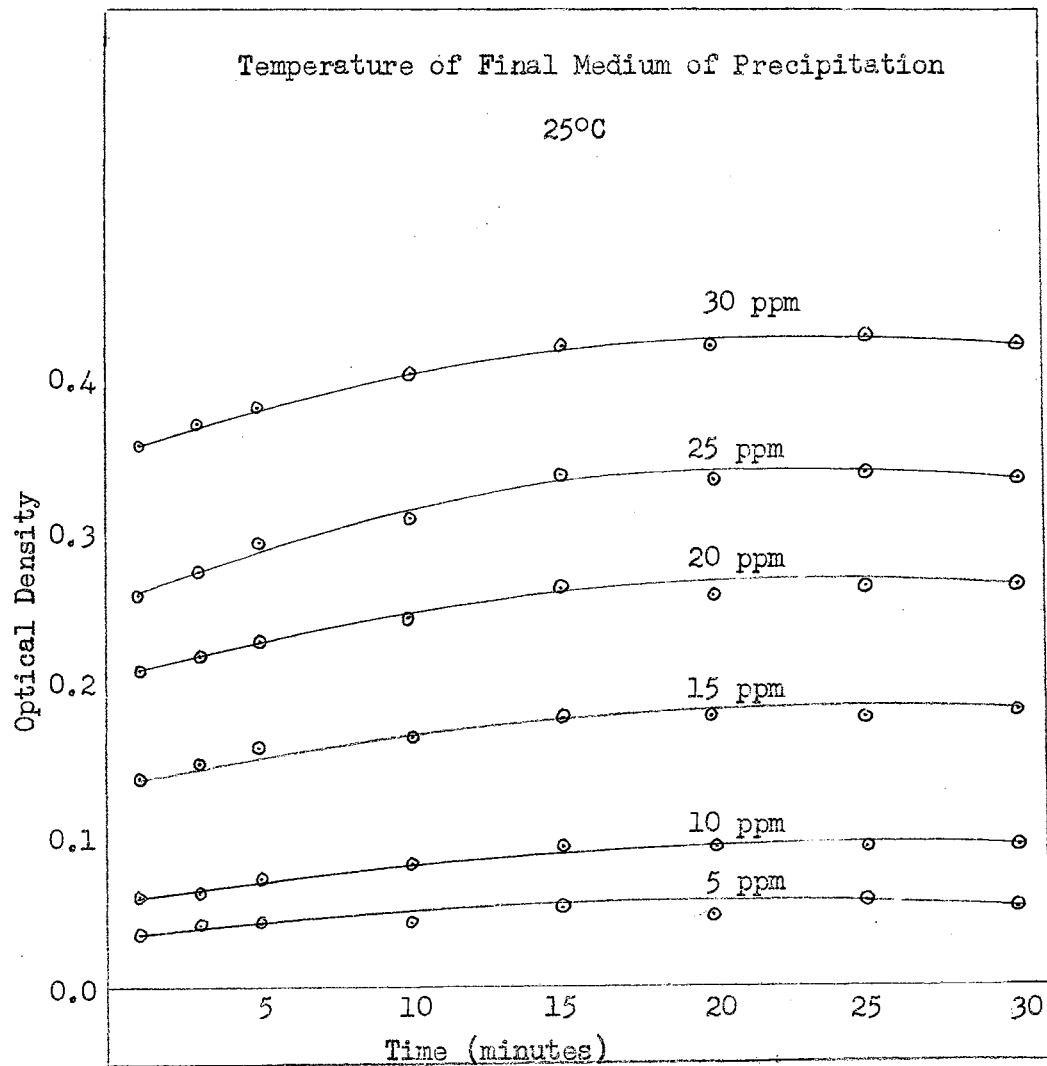


Figure 2. Time-Temperature Relationships in the Potassium Determination with Tetraphenylboron

ed in estimating the turbidity because the large flake-like floccules caused erratic galvanometer readings. As the floccules rotated, they presented varying areas to the light source and changed the optical density readings. Standard curves of potassium in acid media were very difficult to reproduce, presumably because of floccule formation.

Interferences

Certain interferences are encountered in the determination of potassium with sodium tetraphenylboron. Ammonium ions in the medium are the most difficult interference to control. The ammonium ions react with sodium tetraphenylboron to form an insoluble ammonium tetraphenylboron which causes high readings. Formaldehyde was used to complex the ammonium ions as suggested by Berkhout and Jongen (2). However, ammonium concentrations greater than five ppm. could not be effectively masked by the use of formaldehyde. The ammonium interference and the influence of formaldehyde are presented in Figure 3. It is noted that the curve for standard solutions plus three ppm. of ammonium plus $\frac{1}{2}$ ml. of formaldehyde is very similar to the standard curve.

An interference is inherent in soil extracts due to the presence of calcium and magnesium. A concentration of less than 50 ppm. of these elements caused little or no error. When the concentration of calcium and magnesium reached 200 ppm., an interference occurred. This interference was difficult to detect in any one group of standards. However, if several replications were run on standards with and without calcium and magnesium, the interference was evident. Optical density values were increased in the solutions containing calcium and magnesium. This interference was alleviated by the addition of ethylenediaminetetracetate

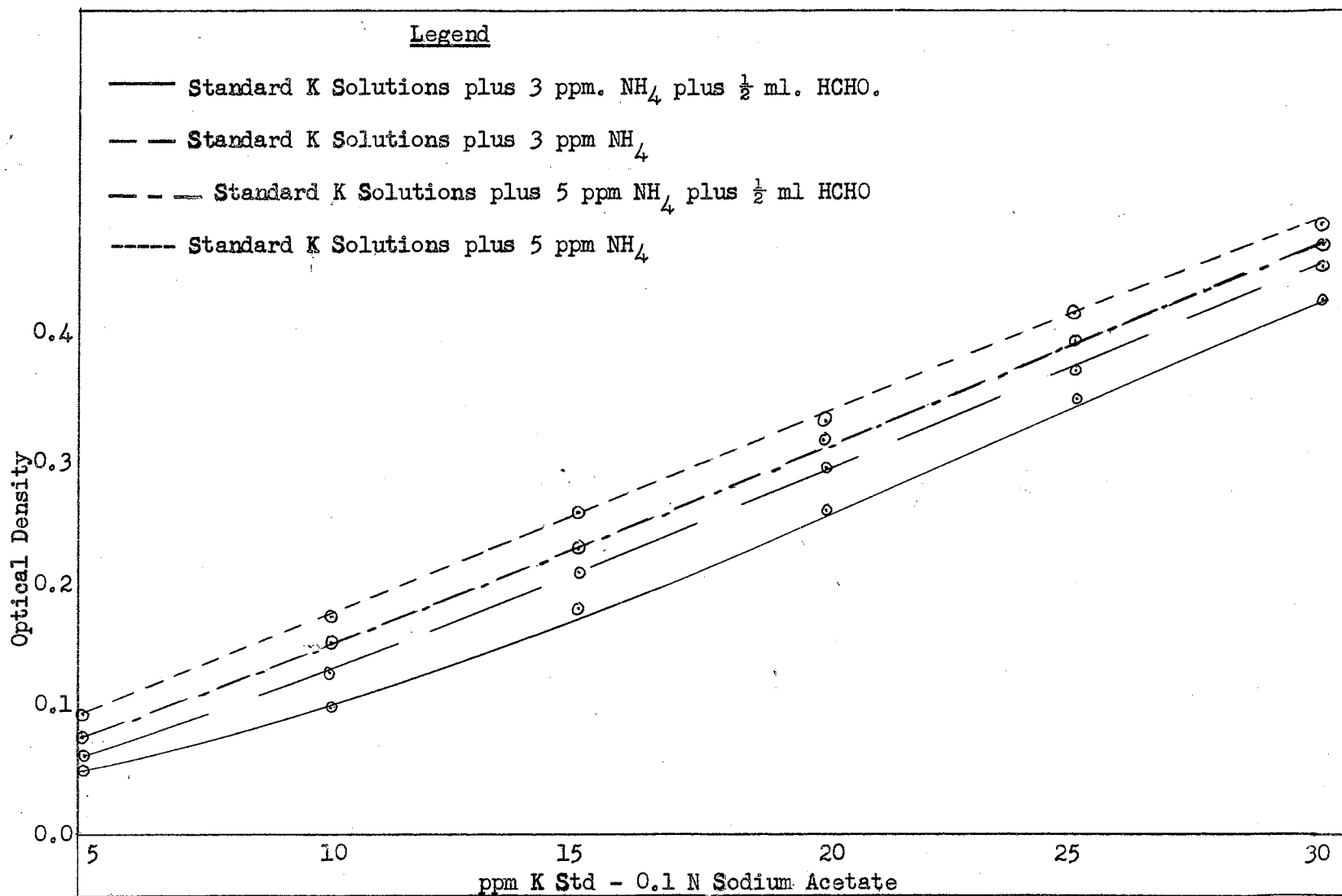


Figure 3. Effect of Ammonium Ions and Ammonium Ions plus Formaldehyde on Potassium Determination with Tetraphenylboron

(EDTA) to the precipitation medium (1, 2). Graphic representation of this interference and its control are found in Figure 4. It is doubted that the interference of calcium and magnesium in any one soil extract would alter the readings enough to change the interpretation of the values. Since EDTA complexes several other elements, which may or may not be present in soil extracts, it is suggested that it be added for overall precision in this method.

Extract:Precipitating Agent Ratio

The ratio of extract to precipitating reagent was very important. Attempts to develop turbidity in five and ten ml. aliquots of extract failed. It was determined that a one ml. aliquot of extract, which contained from five to thirty ppm. of potassium, was suitable. The one ml. sample was diluted to three ml. upon the addition of EDTA and formaldehyde. Thus, the final precipitation medium had a three ml. volume. After the injection of one ml. of NaTPB, the sample was diluted to ten ml. for turbidity estimation. This was the optimum dilution for estimating the turbidity with the Bausch-Lomb Spectronic 20 colorimeter using 12 mm. diameter tubes. With this dilution, the optical density values ranged from approximately 0.05 for the five ppm. standard to approximately 0.45 for the 30 ppm standard.

The concentration of potassium in the soil extract was restricted to values between five and 30 ppm. If extracts or standards containing less than five ppm were analyzed, the results were very erratic and in some instances deviated markedly from potassium values determined by flame photometry. Potassium values greater than 30 ppm., readings which were also somewhat erratic and the curve tended to flatten. The most precise values were obtained when the amount of potassium in the soil

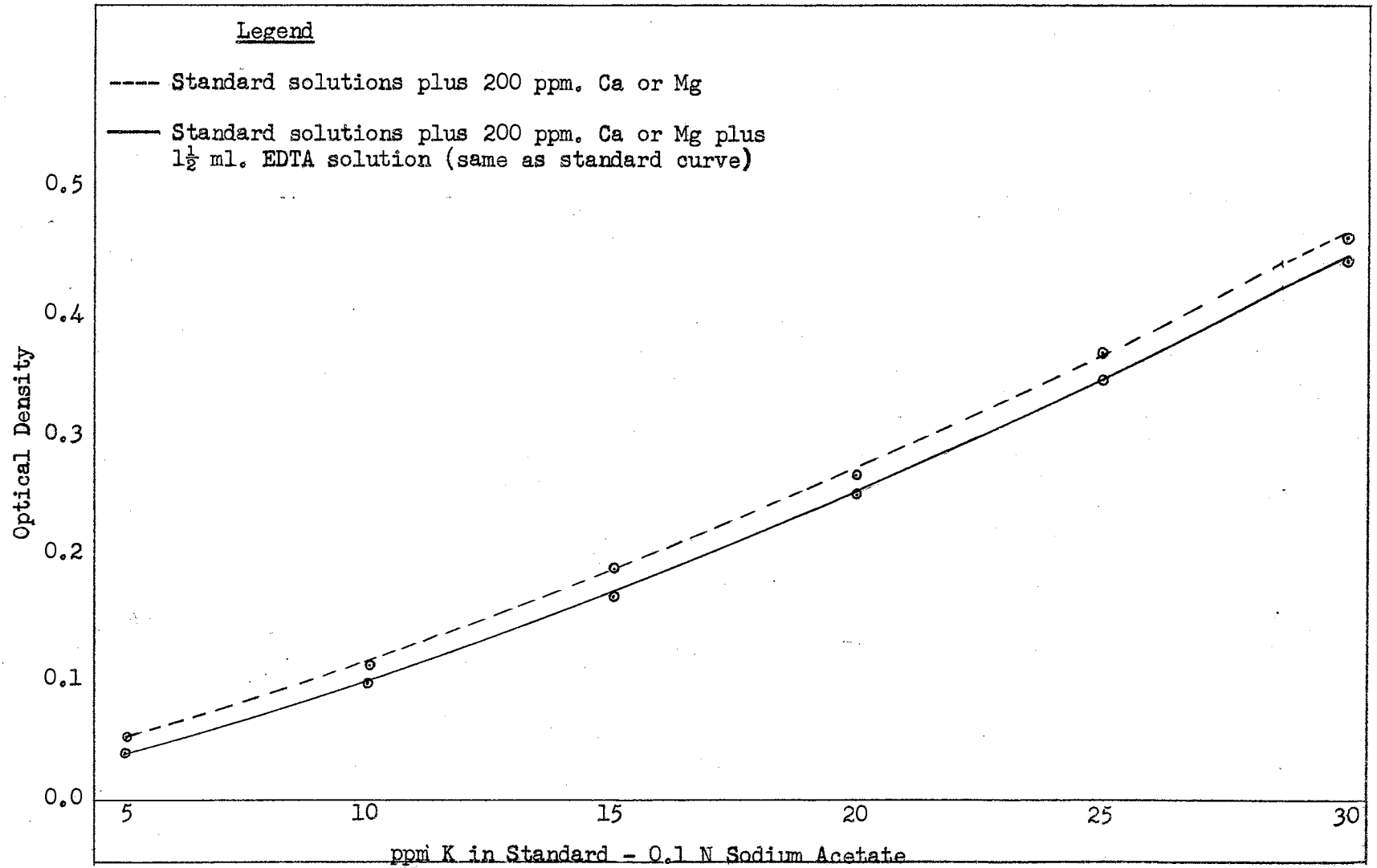


Figure 4. Calcium and Magnesium Interference in Potassium Determination with Tetraphenylboron

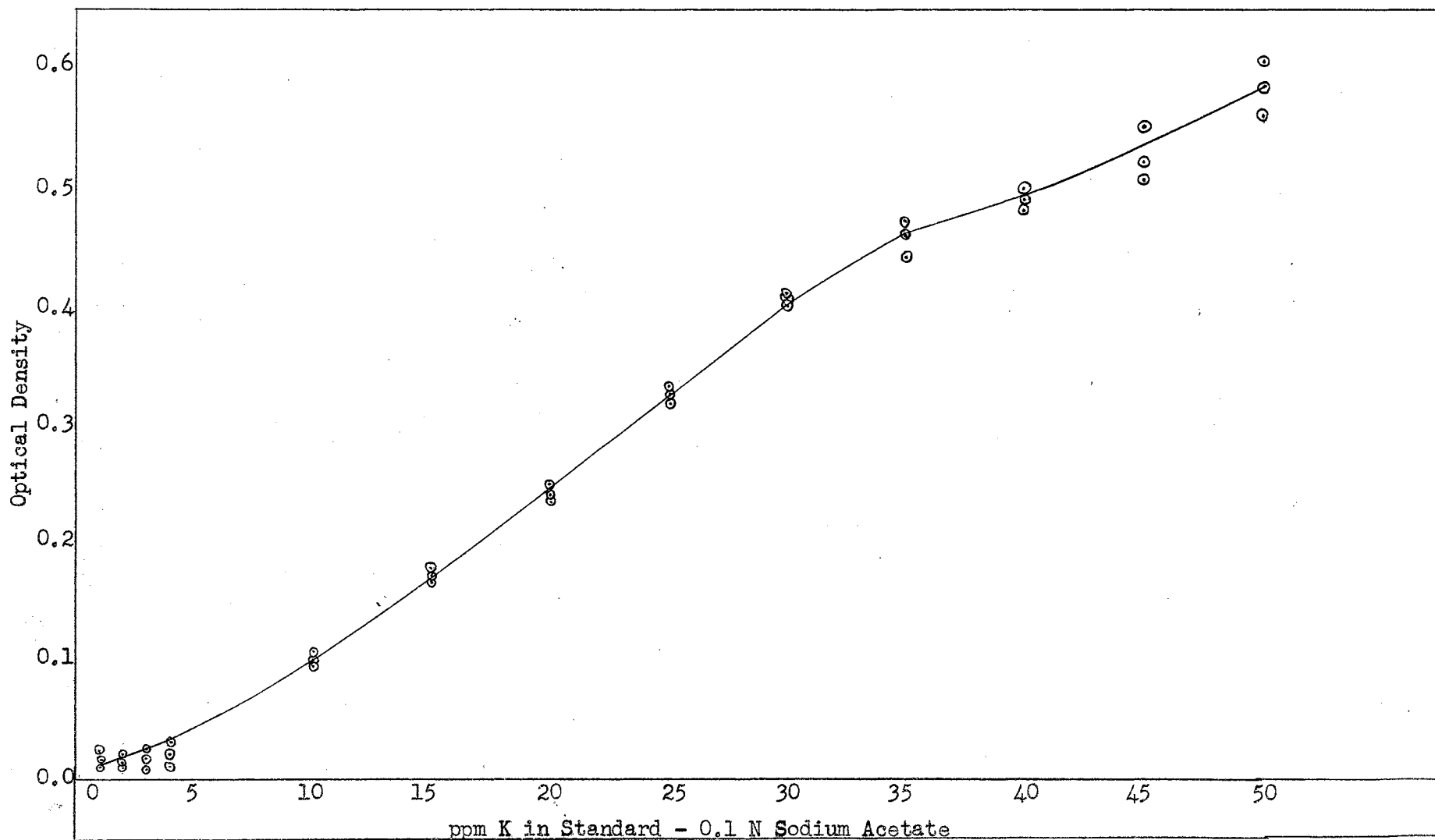


Figure 5. Standard Curve for Potassium Determination with Tetrphenylboron

extract was between five and thirty ppm. A standard potassium curve (potassium standards in 0.1 N sodium acetate) is presented in Figure 5.

From these experiments plus the suggestions of Sunderman and Sunderman (28), a procedure has been devised for analysis of potassium in alkaline media with sodium tetraphenylboron as the precipitating reagent. The method apparently was insensitive to temperature if reaction time is adequate.

Proposed Procedure

- (1) The soil is extracted by shaking five grams of soil in a extracting solution of 0.1 N sodium acetate for five minutes. The amount of extracting solution may vary from 25 to 50 ml. depending upon the soil-solution ratio desired.
- (2) The extract is filtered into a suitable container.
- (3) One ml. of the extract is transferred to a ten ml. volumetric flask.
- (4) Two ml. of a solution composed of two parts of two percent alkaline EDTA and one part of 40 per cent formaldehyde are added to the extract.
- (5) One ml. of alkaline five percent sodium tetraphenylboron is forcibly injected into the medium using a hypodermic syringe; the mixture is allowed to stand for 15 minutes.
- (6) The turbid solution is diluted to volume (ten ml.) with distilled water and the turbidity is estimated with a suitable colorimeter at 525 millimicrons wave length using 12 mm. diameter colorimeter tubes.
- (7) The ppm. of extracted potassium are found by reference to a standard curve made by developing the turbidity in standard solutions of 5, 10, 15, 20, 25, and 30 ppm. of potassium in 0.1 N sodium acetate.

Preparation of Solutions

- (1) The five percent solution of alkaline sodium tetraphenylboron is prepared by adding five grams of reagent grade sodium tetraphenylboron to 50 ml. of distilled water, then adding four ml. of 0.25 N sodium hydroxide, and diluting to 100 ml. with distilled water.

- (2) The EDTA-formaldehyde solution is made as follows: Two parts of alkaline two percent EDTA, prepared by dissolving two grams of the disodium salt in 50 ml. of distilled water containing two ml. of 0.25 N sodium hydroxide and bringing to 100 ml. volume, is added to one part of 40 percent formaldehyde (reagent grade formalin). The two reagents must be mixed each day. Do not use a mixture which has been prepared for longer than six hours.

The sodium tetraphenylboron solution and the EDTA were stable for a period of approximately two weeks if they were refrigerated. The reagents should not be taken from the refrigerator, allowed to warm, and then returned to the refrigerator. The mixture of EDTA and formaldehyde should not be allowed to stand mixed. According to Schall (24), formaldehyde reacts with sodium hydroxide present in the EDTA solution and will no longer complex ammonium ions.

Comparison of Tetraphenylboron and Flame Photometer Methods

Six soils in three replications were extracted with 0.1, 0.5, and 3.0 N sodium acetate and sodium nitrate using 5:1, 7:1, and 10:1 solution-soil ratios. The various extracts were then analyzed by the tetraphenylboron procedure and with the Beckman DU Flame Spectrophotometer with photomultiplier. The Beckman apparatus was adjusted to give a full scale deflection with solutions of 0 to 30 ppm of potassium. Results given in Tables II, III, and IV are reported as ppm. of potassium in each soil as determined by the specific extractants and ratios.

The tetraphenylboron procedure gave results most nearly conforming to those derived through flame photometry when the soil was extracted with 0.1 N sodium acetate. If 0.1 N sodium nitrate was used, results were similar, but showed greater deviation from flame analysis. As the concentration of the extracting reagent increased, the divergence between

TABLE II

A COMPARISON OF SOIL POTASSIUM VALUES OBTAINED BY THE TETRA-PHENYLBORON AND BECKMAN FLAME PHOTOMETER METHODS FOR SIX EXTRACTING AGENTS. (5:1 SOLUTION-SOIL RATIO).

Soils	<u>Extracting Agents</u>											
	0.1N NaAc		0.5N NaAc		3.0N NaAc		0.1N NaNO ₃		0.5N NaNO ₃		3.0N NaNO ₃	
	TPB*	Flame**	TPB	Flame	TPB	Flame	TPB	Flame	TPB	Flame	TPB	Flame
Bowie	45	50	25	45	55	40	30	35	70	45	60	65
Brownfield (Miles)	75	75	100	95	125	90	75	70	120	90	115	135
Dennis	40	35	45	60	110	75	45	45	70	65	125	120
Durant	40	45	70	65	130	95	40	45	90	80	140	140
Parsons	55	45	80	70	95	75	45	45	115	75	120	130
Stephenville	55	50	75	60	100	65	45	50	65	65	100	95

* TPB-Sodium tetraphenylboron.

** Beckman DU Flame Spectrophotometer with photomultiplier.

TABLE III

A COMPARISON OF SOIL POTASSIUM VALUES OBTAINED BY THE TETRA-PHENYLBORON AND BECKMAN FLAME PHOTOMETER METHODS FOR SIX EXTRACTING AGENTS. (7:1 SOLUTION-SOIL RATIO).

Soils	Extracting Agents											
	0.1N NaAc		0.5N NaAc		3.0N NaAc		0.1N NaNO ₃		0.5N NaNO ₃		3.0N NaNO ₃	
	TPB*	Flame**	TPB	Flame	TPB	Flame	TPB	Flame	TPB	Flame	TPB	Flame
Bowie	42	35	35	49	63	49	28	35	49	42	42	56
Brownfield (Miles)	77	84	105	98	140	81	77	77	84	91	133	147
Dennis	42	42	49	70	119	84	49	49	84	70	105	126
Durant	49	49	77	77	133	98	42	49	98	91	140	140
Parsons	63	49	77	77	119	84	56	56	115	75	91	105
Stephenville	49	56	42	70	119	77	42	64	63	70	91	84

* TPB-Sodium tetraphenylboron

** Beckman DU Flame Spectrophotometer with photomultiplier

TABLE IV

A COMPARISON OF SOIL POTASSIUM VALUES OBTAINED BY THE TETRA-PHENYLBORON AND BECKMAN FLAME PHOTOMETER METHODS FOR SIX EXTRACTING AGENTS. (10:1 SOLUTION-SOIL RATIO).

Soils	<u>Extracting Agents</u>											
	0.1N NaAc		0.5N NaAc		3.0N NaAc		0.1N NaNO ₃		0.5N Na NO ₃		3.0N NaNO ₃	
	TPB*	Flame**	TPB	Flame	TPB	Flame	TPB	Flame	TPB	Flame	TPB	Flame
Bowie	50	40	20	50	50	50	40	40	50	50	50	70
Brownfield (Miles)	80	90	110	110	130	100	60	80	100	110	120	150
Dennis	60	60	40	70	130	90	50	50	70	70	100	140
Durant	60	60	50	90	150	110	40	50	90	100	140	160
Parsons	70	50	70	100	130	90	60	60	100	90	100	120
Stephenville	50	60	50	70	110	80	40	50	70	70	100	110

* TPB-Sodium tetraphenylboron

** Beckman DU Flame Spectrophotometer with photomultiplier

the two methods usually tended to increase. The overall summation of the divergence was positive because the tetraphenylboron method often gave higher results, especially when 3.0N sodium acetate was the extractant. (Table V). It is suspected that certain organic substances, such as amides and amines, which are likely to be removed from soils by higher concentrations of alkaline agents, were responsible for these positive deviations. The deviations were most notable in the extracts of 3.0 N sodium acetate which was the most alkaline agent used. These extracts showed some discoloration which was presumably due to an organic component. These deviations could be inherent in either the tetraphenylboron procedure or in the Beckman flame photometer analysis. An insoluble organic-tetraphenylboron complex, formed during the precipitation could account for the higher values in the tetraphenylboron analysis. High sodium levels could cause depressed sensitivity of the flame photometer. The flame apparatus was sluggish and less sensitive to the controls when high sodium content extracts were analyzed.

A procedure was devised to ascertain the recovery of standard amounts of potassium added to randomly selected extracts. The potassium fortified extracts were analyzed by the tetraphenylboron procedure and by flame analysis. The assumed final concentration of potassium in the extract was computed by adding the Beckman values for potassium in the original extract to the ten ppm. of added potassium. The Beckman values were used for the original concentration of potassium since the tetraphenylboron method gave erratic results at concentrations of less than five ppm. The recovery average of 99.7% for the tetraphenylboron procedure indicated that little interference was encountered. The flame apparatus averaged 100.1% recovery (Table VI).

TABLE V

RELATIONSHIP OF KINDS AND CONCENTRATIONS OF EXTRACTING AGENTS
TO DIVERGENCE BETWEEN TETRAPHENYLBORON AND
BECKMAN FLAME PHOTOMETER ANALYSES

Solution Soil Ratio	<u>Extracting Agents</u>					
	0.1N NaAc	0.5N NaAc	3.0N NaAc	0.1N NaNO ₃	0.5N NaNO ₃	3.0N NaNO ₃
5:1	+1.7*	0.0	+21.2	-1.6	+18.3	-4.1
7:1	+1.2	-9.3	+36.7	-6.0	+8.0	-9.4
10:1	+1.7	-25.0	+30.0	-6.7	-1.7	-20.3
Mean	+1.53	-11.43	+29.63	-4.77	+8.2	-11.27

* Plus (+) values indicate higher potassium values for the tetraphenylboron method and negative values (-) indicate lower values for tetraphenylboron. All values are expressed in ppm.

TABLE VI

RECOVERY OF POTASSIUM ADDED TO EXTRACTS BY TETRAPHENYLBORON
AND FLAME PHOTOMETER ANALYSES

Extract No.	ppm. K in Extract		ppm. K Added	ppm. K Recovered		Percent Recovery	
	TPB*	Flame**		TPB	Flame	TPB	Flame
1.	3	1	10	11	11	100.00	100.00
2.	12	11	10	21	22	100.00	104.80
3.	6	6	10	15	16	93.75	100.00
4.	16	14	10	24	23	100.00	95.83
5.	4	1	10	12	12	109.10	109.10
6.	8	8	10	17	17	94.44	94.44
7.	12	13	10	24	23	104.34	100.00
8.	17	17	10	26	26	96.29	96.29

* TPB-Sodium tetraphenylboron

** Beckman DU Flame Spectrophotometer with photomultiplier

Greenhouse Experiment

Forage sorghum dry matter yields are listed by replications which were summed over the four harvests (Table VII). The analysis of variance for these data is shown in Table VIII. The statistical analysis of the plant yields indicated significant differences among treatments at the one percent probability level. The 28 treatments were the various soil-potassium combinations. The treatments are compared by Duncan's New Multiple Range Test in Table IX.

Total milligrams of potassium removed by the forage sorghum plants is given in Table X. These figures were obtained by summing replications over the four harvests. The analysis of variance of these data indicated significant differences among treatments at the one percent probability level (Table XI). Duncan's New Multiple Range Test for these data is shown in Table XII.

Yield data gave little correlation between plant growth and the potassium treatments for any of the soils. Various explanations for this fact might be advanced. It is possible that the upper limit of potassium fertilization was reached with the five percent level of exchangeable potassium addition (K_1). The K_1 level, when summed over all soils, gave the highest yields. The possibility of other limiting factors of plant growth, including soil volume and certain light conditions present in the greenhouse, cannot be disregarded.

The amount of potassium removed by the plants varied directly with the amount of potassium added to the soils. There was evidently luxury consumption of potassium since yields did not follow this same trend.

TABLE VII

DRY MATTER YIELDS OF SUGAR DRIP FORAGE SORGHUM IN A GREENHOUSE
EXPERIMENT (EXPRESSED IN GRAMS PER POT)

<u>Treatment</u>	<u>Rep I</u>	<u>Rep II</u>	<u>Rep III</u>	<u>Mean Yield</u>
Stephenville K ₃	46.7	34.1	42.5	41.2
Durant K ₁	35.8	35.8	41.4	37.7
Parsons K ₁	34.1	43.2	34.6	37.3
Stephenville K ₁	33.3	33.2	41.1	35.9
Parsons K ₂	37.6	36.6	31.0	35.1
Bowie K ₁	37.9	29.8	35.6	34.4
Stephenville K ₀	30.0	42.4	30.6	34.3
Stephenville K ₂	29.9	33.7	39.4	34.3
Parsons K ₀	36.9	30.8	35.2	34.3
Durant K ₀	30.6	26.8	42.0	33.1
Bowie K ₃	32.3	29.2	37.3	32.9
Bowie K ₂	30.4	28.7	39.4	32.8
Durant K ₂	38.8	24.1	34.6	32.5
Dennis K ₂	31.8	26.7	37.6	32.0
Dennis K ₃	32.7	28.4	33.8	31.6
Bowie K ₀	24.7	37.5	32.5	31.6
Dennis K ₁	31.0	26.7	35.2	30.9
Linker K ₁	20.4	34.1	37.9	30.8
Durant K ₃	17.9	29.0	44.2	30.4
Dennis K ₀	32.0	24.6	32.8	29.8
Linker K ₃	23.8	29.7	33.7	29.1
Linker K ₀	26.0	18.0	36.6	26.9
Parsons K ₃	29.7	27.1	22.2	26.3

TABLE VII (CONTINUED)

<u>Treatment</u>	<u>Rep I</u>	<u>Rep II</u>	<u>Rep III</u>	<u>Mean Yield</u>
Linker K ₂	18.5	18.4	31.1	22.7
Brownfield K ₁	13.8	18.6	24.4	18.9
Brownfield K ₂	13.6	17.9	23.5	18.3
Brownfield K ₀	16.8	15.3	17.8	16.6
Brownfield K ₃	10.1	13.4	14.0	12.5

TABLE VIII
ANALYSIS OF VARIANCE OF FORAGE SORGHUM YIELDS

Source	df	SS	MS	F
Total	83	6167.19	-----	-----
Replications	2	511.55	255.78	7.11**
Treatment	27	3712.70	137.50	3.82**
Error	54	1942.94	35.98	-----

** Denotes significance at the 1 percent probability level.

TABLE IX

DUNCAN'S NEW MULTIPLE RANGE TEST OF FORAGE SORGHUM YIELDS

<u>Treatment</u>	<u>Mean</u>
Brownfield K ₃	12.5
Brownfield K ₀	16.6
Brownfield K ₂	18.3
Brownfield K ₁	18.9
Linker K ₂	22.7
Parsons K ₃	26.3
Linker K ₀	26.9
Linker K ₃	29.1
Dennis K ₀	29.8
Durant K ₃	30.4
Linker K ₁	30.8
Dennis K ₁	30.9
Bowie K ₀	31.6
Dennis K ₃	31.6
Dennis K ₂	32.0
Durant K ₂	32.5
Bowie K ₂	32.8
Bowie K ₃	32.9
Durant K ₀	33.1
Parsons K ₀	34.3
Stephenville K ₂	34.3
Stephenville K ₀	34.3
Bowie K ₁	34.4
Parsons K ₂	35.1

TABLE IX (CONTINUED)

<u>Treatment</u>	<u>Mean</u>
Stephenville K ₁	35.9
Parsons K ₁	37.3
Durant K ₁	37.7
Stephenville K ₃	41.2

Note: Any two means not paralleled by the same line are significantly different at the 5% probability level.

TABLE X

MILLIGRAMS OF POTASSIUM REMOVED BY SUGAR DRIP FORAGE
SORGHUM IN A GREENHOUSE EXPERIMENT

<u>Treatment</u>	<u>Rep I</u>	<u>Rep II</u>	<u>Rep III</u>	<u>Mean Yield</u>
Stephenville K ₃	1263	1015	1271	1183
Parsons K ₁	868	1140	941	983
Durant K ₁	897	953	1083	978
Stephenville K ₂	845	916	955	905
Linker K ₃	683	935	1076	898
Dennis K ₃	889	880	868	879
Parsons K ₃	956	893	743	864
Stephenville K ₁	809	792	986	862
Durant K ₂	988	646	940	858
Durant K ₃	603	751	1219	858
Parsons K ₂	892	927	711	843
Dennis K ₂	818	630	951	800
Durant K ₃	685	673	946	768
Dennis K ₁	787	670	828	762
Stephenville K ₀	674	904	645	741
Linker K ₂	693	519	1000	738
Durant K ₀	606	655	912	724
Bowie K ₂	611	654	874	713
Linker K ₁	462	754	845	687
Parsons K ₀	751	545	755	684
Bowie K ₁	645	571	675	630
Dennis K ₀	608	577	621	602
Linker K ₀	557	414	784	585

TABLE X (CONTINUED)

<u>Treatment</u>	<u>Rep I</u>	<u>Rep II</u>	<u>Rep III</u>	<u>Mean</u>
Brownfield K ₂	415	554	690	553
Bowie K ₀	432	580	551	521
Brownfield K ₁	325	528	678	510
Brownfield K ₀	430	439	454	441
Brownfield K ₃	340	442	494	425

TABLE XI
ANALYSIS OF VARIANCE OF MILLIGRAMS OF POTASSIUM
REMOVED BY FORAGE SORGHUM PLANTS

Source	df	SS	MS	F
Total	83	3,732,537	-----	-----
Replications	2	338,315	169,157.5	10.47**
Treatment	27	2,521,376	93,384.3	5.78**
Error	54	872,846	16,163.8	-----

** Denotes significance at the 1 percent probability level.

TABLE XII

DUNCAN'S NEW MULTIPLE RANGE TEST OF THE MILLIGRAMS OF
 POTASSIUM REMOVED BY FORAGE SORGHUM PLANTS

<u>Treatment</u>	<u>Mean</u>
Brownfield K ₃	425
Brownfield K ₀	441
Brownfield K ₁	510
Bowie K ₁	521
Brownfield K ₂	553
Linker K ₀	585
Dennis K ₀	602
Bowie K ₁	630
Parsons K ₀	684
Linker K ₁	687
Bowie K ₂	713
Durant K ₀	724
Linker K ₂	738
Stephenville K ₀	741
Dennis K ₁	762
Bowie K ₃	768
Dennis K ₂	800
Parsons K ₂	843
Durant K ₃	858
Durant K ₂	858
Stephenville K ₁	862
Parsons K ₃	864
Dennis K ₃	879

TABLE XII (CONTINUED)

<u>Treatment</u>	<u>Mean</u>
Linker K ₃	898
Stephenville K ₂	905
Durant K ₁	978
Parsons K ₁	983
Stephenville K ₃	1183

Note: Any two means not paralleled by the same line are significantly different at the 5% probability level.

V. SUMMARY AND CONCLUSIONS

A turbidimetric quick-test procedure for the estimation of potassium in soil extracts was developed using sodium tetraphenylboron as a precipitating agent. Six soils were collected and used to compare this method with a standard procedure using the Beckman DU Flame Spectrophotometer with photomultiplier. The soils were extracted with 0.1, 0.5, and 3.0 N sodium acetate and sodium nitrate using three solution-soil ratios (5:1, 7:1, and 10:1). The potassium content of these extracts was then estimated using the turbidimetric sodium tetraphenylboron procedure and the Beckman DU Spectrophotometer.

A greenhouse experiment was designed to study soil test-plant growth correlations. The seven soils were fertilized with potassium by additions of 0, five, ten, and 15 percent of the cation exchange capacity. Sugar Drip forage sorghum was used as the indicator crop. Four harvests were taken and dry-matter yields plus the potassium content of the plants were determined.

From the results of this investigation, the following conclusions seem justifiable:

- (1) A method, which involved the use of sodium tetraphenylboron, compared favorably with the Beckman DU Flame Spectrophotometer with photomultiplier in the quantitative estimation of potassium in soil extracts.
- (2) Ammonium interference to potassium determination with sodium tetraphenylboron could not be effectively masked

by additions of formaldehyde, if ammonium ion concentration was greater than five ppm.

- (3) Formation of the maximum turbidity using sodium tetraphenylboron was affected by both time and temperature.
- (4) Little yield response was shown to potassium fertilization of Sugar Drip forage sorghum in the greenhouse.
- (5) Sugar Drip forage sorghum exhibited "luxury consumption" of potassium in the greenhouse.

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A P P E N D I X

TABLE XIII
PROFILE DESCRIPTIONS OF SOILS

Brownfield Sand¹

Collected by: Dr. Robert M. Reed and A. C. Bennett

Location: The description was made near the location where soil was taken from the greenhouse and laboratory experiments. Collected at the Agronomy Research Station at Mangum, Oklahoma in an area adjacent to the cotton fertility study southeast of the station headquarters.

Site: Cultivated area; no previous fertilization. The land is nearly level, having a surface gradient of about $\frac{1}{2}$ percent.

Profile:

A _{1p}	0-6"	Brown (7.5YR 5/4) sand, brown (7.5YR4/3) when moist; structureless; slightly firm, hard when dry; permeable; pH 6.5; gradual boundary.
A ₁	6-18"	Light brown (7.5YR 6/3) loamy fine sand, light brown (7.5YR 5/3) when moist; structureless; very friable; freely permeable; pH 6.5; grades to layer below.
B ₂	18-38"	Reddish brown (6YR 5/3) sandy clay loam, reddish brown (6YR 4/3) when moist; weak medium subangular blocky;

¹Profile description by H. M. Galloway, formerly soil scientist with Oklahoma State University and Soil Conservation Service.

firm; porous and permeable; pH 6.5; contains a few fine, reddish yellow specks around the fine pores; grades to layer below.

- B₃C 38-46" Light brown (7.5YR 6/4) light sandy clay loam, light brown (7/5YR 5/4) when moist; weak medium subangular blocky; firm; porous and permeable; pH 6.5; grades to layer below.
- C₁ 46-66" Brown (7.5YR 5/3) light sandy clay loam, brown (7.5YR 4/3) when moist with numerous medium to coarse, faint light-gray mottles and strong brown specks; weak medium subangular blocky; firm to friable; pH 7.5; occasional soft fine black pellets and ferruginous films; grades to layer below.
- C_{ca} 66-86" Light brown (7.5YR 6/3) loamy sand, light brown (7.5YR 5/3) when moist with considerable coarse quartz sand and seams of brown sandy clay loam; occasional concretions of CaCO₃; pH 8.0; calcareous in seams; occasional soft black concretions and ferruginous films; mass of material averages fine sandy loam when crushed; grades to layer below.

Bowie Fine Sandy Loam¹

Collected by: Roy M. Smith and A. C. Bennett

Location: West of Bentley, Oklahoma on county road. Sec. 13, T4S, R12E.

Site: Old field covered with annual weeds. Two percent slope.

Profile:

- | | | |
|-----------------|--------|---|
| A ₁ | 0-7" | Light brownish gray (10YR 6/2) light fine sandy loam, dark grayish brown (10YR 4/2) moist; structureless fine to medium granular; soft dry, very friable moist; pH 5.5; clear boundary. |
| A ₂ | 7-16" | Very pale brown (10YR 7/3) light fine sandy loam, pale brown (10YR 6/3) moist; fine to medium granular to structureless; soft dry, very friable moist; pH 4.8; clear boundary. |
| B ₂₁ | 16-28" | Brownish yellow (10YR 6/8) light sandy clay loam, yellowish brown (10YR 5/8) moist; few fine faint pale brown mottles; weak medium subangular blocky structure; hard dry, friable moist; pH 4.3; gradual boundary. |
| B ₂₂ | 28-40" | Brownish yellow (10YR 6/8) sandy clay loam, yellowish brown (10YR 5/8) moist; many fine and medium red, yellowish red, gray and pale brown mottles; porous massive to weak medium subangular blocky structure; hard dry, friable moist; pH 4.3; gradual boundary. |

¹Profile description by Roy M. Smith, soil scientist with Oklahoma State University.

- B₃ 40-50" Brownish yellow (10YR 6/8) light sandy clay loam, yellowish brown (10YR 5/8) moist; many coarse and medium distinct mottles of yellowish brown, red and gray; porous massive structure; slightly hard dry, friable moist; pH 4.1; clear boundary.
- C_u 50" Coarsely mottled with pale gray, red, yellowish red and strong brown coarse clay loam; massive structure; pH 4.2.

Dennis Silt Loam¹

Collected by: Ruel Bain, Dr. Robert M. Reed, and A. C. Bennett.

Location: Two and four tenths miles S of Muskogee-McIntosh Co. line at S edge of Council Hill, Oklahoma, 30 feet E of highway 72. NW $\frac{1}{4}$, Sec. 17, T12N, R16E.

Site: Field has been incultivation in the past; two percent slope.

Profile:

- A_{1p} 0-7" Very dark grayish brown (10YR 3/2) moist, silt loam; moderate coarse and medium granular; friable moist; pH 5.4; abrupt boundary.
- A₁ 7-11" Very dark brown (10YR 2/2) moist, heavy silt loam or light silty clay loam; moderate coarse, medium, and fine granular; pH 5.3; gradual boundary.
- A₃ or B₁ 11-15" Very dark brown (10YR 2/2) moist, light silty clay loam; common fine mottles of yellowish brown (10YR 5/4) moist; moderate to strong coarse, medium, and fine granular; many worm casts; some reddish and black

¹Profile description by Ruel Bain, formerly soil scientist with Oklahoma State University.

- concretions to 1/8" in diameter; pH 5.3; clear boundary.
- B₂ 15-35" Strongly mottled with browns, yellowish browns, strong browns, pale browns largely on 10YR hue, silty clay loam or silty clay; many black concretions, pH 5.3.
- B₃ 35-51" Same as above horizon except slightly finer texture and appearance or light olive gray (5Y 6/2) moist, and light brownish gray (2.5Y 6/2) moist, making up approximately 20% of surface color (or by volume); black concretions larger; pH 5.3.

Durant Clay Loam¹

Collected by: Roy M. Smith and A. C. Bennett.

Location: Center, SW $\frac{1}{4}$, SE $\frac{1}{4}$, SE $\frac{1}{4}$, Sec. 30, T5S, R10E, Bryan Co., Oklahoma.

Site: Range site

Profile:

- A_{1p} and 0-10"
A₁ Gray (10YR 5/1) clay loam, very dark gray (10YR 3/1) moist; weak medium granular structure; dry hard, friable moist; pH 5.5; clear boundary.
- B₁ 10-18" Brown (10YR 5/3) silty clay loam, dark brown (10YR 4/3) moist; common to many reddish brown to strong brown mottles; moderate fine and medium subangular blocky structure; very hard dry, friable moist; pH 4.6; gradual boundary.
- B₂₁ 18-30" Light olive brown (2.5YR 5/4) clay, olive brown (2.5YR 4/4) moist; many prominent strong brown, yellowish red and gray mottles; few gravel; moderate medium

¹Profile description by Roy M. Smith, soil scientist with Oklahoma State University.

- blocky structure; very hard dry, very firm moist;
pH 5.3; gradual boundary.
- B₂₂ 30-36" Same as B₂₁ but has a few black Fe and Mn concretions;
separated for purpose of analysis; mottles similar to
above horizon but larger in size; pH 6.2.
- B₃ 36-46" Olive (5Y 5/3) clay, olive (5Y 4/4) moist; few
yellowish brown mottles; moderate medium and coarse
blockly structure; very hard dry; pH 6.5; gradual
boundary.
- C 46-54" Olive (5Y 5/4) light silty clay, olive (5Y 5/3) moist;
massive structure; extremely hard dry; few weak CaCO₃
concretions; pH 7.0.

Linker Loam

Collected by: Ruel Bain, Dr. Robert M. Reed, and A. C. Bennett.

Location: Six to seven miles NW of McAlester, Oklahoma in an area
mapped Hanceville fsl in Pittsburg Co. survey. Collected 30 feet N.,
30 feet E. of SW corner of section 18, T6N, R14E.

Site: Old field land covered by Broomsedge. Shallow on sandstone, three
percent slope.

Profile:

- A₁ 0-6" Dark grayish brown (10YR 4/2) loam, very dark grayish
brown (10YR 3/2) moist; weak medium and fine granular
structure; a few sandstone fragments up to $\frac{1}{4}$ " in
diameter; pH 5.6; clear boundary.

¹Profile description by Ruel Bain, formerly soil scientist
with Oklahoma State University.

- A₂ 6-12" Brown (10YR 4/3) sandy loam, dark brown (10YR 3/3) moist; weak medium granular to structureless; pH 4.3; clear boundary.
- B₁ 12-21" Yellowish brown (10YR 5/4) very light sandy clay loam, yellowish brown (10YR 4/4) moist; few fine mottles of brownish yellow (10YR 6/6), yellowish brown (10YR 5/6) moist; weak medium and fine granular structure; pH 4.3.
- B₂₁ 21-28" Brown (10YR 5/3) light sandy clay loam with about 40% of surface pale brown (10YR 6/3), dark brown (10YR 4/3) moist with the 40% brown (10YR 5/3) moist; pH 4.4.
- D_r 28"-- Strongly mottled with yellowish brown (10YR 5/8), brownish yellow (10YR 6/8), light yellowish brown (10YR 6/4), and yellowish red (5YR 4/6) sandstone fragments up to 3" in diameter. Some have black interior; pH 4.4.

Parsons Clay Loam¹

Collected by: Ruel Bain, Dr. Robert M. Reed and A. C. Bennett.

Location: Two miles north of Muskogee-McIntosh Co. line at S edge of Council Hill, Oklahoma, W on gravel road 350 yards, 30 feet N of bar ditch in SE₄, SE₄, Sec. 14, T13N, R15E.

Site: Nearly level to slightly depressional area; very wet at time of sampling. Old field area with annual vegetation and water grass.

Profile:

- A_{1p} 0-6" Grayish brown (2.5Y 5/2) clay loam, very dark grayish brown (10YR 3/2) moist; a few distinct fine mottles of

¹Profile description by Ruel Bain, formerly soil scientist with Oklahoma State University.

yellowish brown (10YR 5/6); weak coarse, medium and fine granular-irregular clods when dry; very hard dry; pH 5.4.

- A₂ 6-9" Light brownish gray (10YR 6/2) silt loam, very dark grayish brown (10YR 3/2) moist; a few fine distinct brownish yellow (10YR 6/8); weak coarse medium and fine granular-irregular clods when dry; very hard dry; pH 5.0; abrupt boundary.
- B₂₁ 9-18" Brown or dark brown (10YR 4/3) moist clay; strongly mottled with grayish brown (10YR 5/2), light gray (10YR 7/1), yellowish brown (10YR 5/6) brownish yellow (10YR 6/8), dark brown (7.5YR 4/4); blocky to massive structure; extremely hard dry; many Fe and Mn concretions; a few fine roots; pH 5.2; gradual boundary.
- B₂₂ 28-41" Strongly mottled brown (10YR 4/3), light gray, yellowish brown, brownish yellow, dark brown clay (as above horizon); blocky to massive structure; extremely hard dry; many black specks and concretions; pH 5.4; gradual boundary.
- B_{3C} or C 50-54" Brown (10YR 5/3) clay; mottled as above; blocky to massive structure; extremely hard dry; many black specks and concretions; pH 5.3.

Stephenville Loamy Sand¹

Collected by: Roy M. Smith and A. C. Bennett.

Location: Ten miles south, 6 miles W of Chandler in Sec. 32, T12N, R3E.

¹Profile description by Roy M. Smith, soil scientist with Oklahoma State University.

Site: Virgin timber; blackjack and post-oak.

Profile:

- A₁ 0-6" Brown (10YR 5/3) light loamy fine sand, dark brown (10YR 3/3) moist; weak fine to medium granular structure; slightly hard dry, very friable moist; pH 6.2; clear boundary.
- A₂ 6-15" Light brown (7.5YR 6/4) light loamy fine sand, dark brown (7.5YR 4/4) moist; structureless to fine granular; slightly hard dry, very friable moist; pH 6.2; clear boundary.
- B₂ 15-26" Red (2.5YR 4/6) sandy clay loam, dark red (2.5YR 3/8) moist with few small black Fe and Mn concretions and few pale yellow splotches; weak medium subangular blocky; hard dry, friable moist; pH 5.3; gradual boundary.
- BC 15-26" Red (2.5YR 4/6) light sandy clay loam, dark red (2.5YR 3/8) moist with many medium black Fe and Mn concretions and chips of weathered sandstone, few yellowish splotches; porous massive structure; hard dry, friable moist; pH 5.5; clear boundar.
- C 30-34" Reddish brown (5YR 4/4) and yellowish red (5YR 4/8) dry; fine sandy loam and weathered sandstone chips; hard dry; contains 50% dime to dollar size Fe and Mn concretions; pH 6.0.

VITA

Allison Carr Bennett

Candidate for the Degree

of

Master of Science

Thesis: DEVELOPMENT AND TESTING IN THE LABORATORY AND GREENHOUSE
OF A METHOD USING TETRAPHENYLBORON FOR THE DETERMINATION
OF EXTRACTABLE SOIL POTASSIUM

Major: Agronomy (Soils)

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