POTASSIUM ESTIMATION

FIXATION AND RELEASE IN LOWER

FRASER VALLEY SOILS

by

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ABSTRACT

A study of the potassium status of some Lower Fraser Valley soils was undertaken in order to supply data which would aid in the interpretation of fertility experiments and chemical tests for available soil potassium. Since the rapid chemical tests for available potassium now used in the Lower Fraser Valley leave much to be desired, several promising methods were studied and compared to a standard ammonium acetate extraction gravimetric analysis method for determining exchangeable potassium.

The Morgan and Spurway rapid methods for available potassium estimation were not found precise or accurate enough for the purposes of the study. A technique described by Peech and English, which involves extraction of available potassium with normal sodium acetate of $pH4.8$ and subsequent precipitation of potassium as the di-potassium sodium cobaltinitrite in formaldehyde-isopropyl alcohol solution was tested. A modification of this method was adapted to estimation by the photometer and results for exchangeable potassium, although lower, correlated well with those of the standard determination. A technique which is more rapid and better adapted to studies in which potassium is the only cation to be tested is that described by Bray, who recommends extraction with sodium nitrate and precipitation of potassium with sodium cobaltinitrite in ethyl alcohol solution and subsequent photometric

determination. This method extracted slightly more pot-
assium than that of Peech but also showed a high corre-
lation with the standard method used. Both and Bray and
Peech procedures were found adaptable to line chart
estimation for routine soil testing work.

The Bray method was compared to the standard ammonium
acetate-gravimetric procedure in potassium fixation and
release studies in limed and unlimed soils. Two upland
soils and three recent alluvial soils, on which fertility
experiments with oats had been conducted, were studied. A
greenhouse experiment using lettuce was designed to indicate
any existing relation between chemical studies and plant
response. In the laboratory fixation was induced by
alternately wetting and drying the soils at 110° C. All
soils were found to have high fixing potentials when
measured by both extraction methods, although the fixation
was higher when determined by the standard method. The
effect of lime on fixation in both the laboratory and green-
house soils was determined. The soils generally showed an
apparent release of potassium when measured by the ammon-
ium acetate-gravimetric method and a fixation by the sodium
nitrate technique. These differences were considered to be
due to the relative replacing powers of Na^+ and NH^+ when
Ca^{++} or H^+ dominate the exchange complex. Thus the extraction
of potassium by Na^+ may be inhibited when calcium is the
complementary ion on the colloid. The potassium status of

the upland soils was less affected by lime than the lowland soils when fixation or release due to lime was measured by either method.

The soils studied failed to release any potassium from the fixed form when leached of their exchangeable potassium and stored for three months in a moist condition, or when wetted and dried at 110 C.

No relation was found to exist between exchangeable potassium or the relative potassium fixing and supplying powers of the soils, and oat response to potassium in the field or lettuce growth in the greenhouse.

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INTRODUCTION

Potassium is an essential element for plant growth
and the quantity available in the soil is therefore of
great importance. In the Lower Fraser Valley, chemical
tests designed to estimate the amount of available
potassium in the soil are often employed in making
potassium fertilizer recommendations. The adequacy of
these tests depends on their accuracy and upon how they
are interpreted. It is evident that the tests employed
leave much to be desired and lack of correlation with
plant growth is frequently noted.

It appears that potassium fixation and release may
be important in interpreting the results of chemical
tests and plant response to potassium fertilizer appli-
cation. There is a possibility that a high potassium
fixation by some soils may mask response to potassium.
Other soils, which give a low test for available pot-
assium, may have high supplying potentials and show no
response to potassium. Some investigators have produced
convincing evidence that lime will induce potassium
fixation. Little is known about the effect of lime on
the potassium status of the Lower Fraser Valley soils.

Recommendations for potassium fertilization in the
Fraser Valley have stressed the need of heavy applications
on the light upland soils and light applications on the

on the heavier alluvial soils. Some fertility experiments indicate that the greatest response to potassium application occurs on the lowland soils. Further, when little or no response to potassium is obtained its application is often discontinued. Whether this policy is wise is a matter of contention.

These considerations prompted the investigations included in this thesis. In undertaking the work it was hoped to establish a reliable and accurate method for estimating available potassium in Lower Fraser Valley soils and to provide some information which will assist in the interpretation of fertilizer experiments.

REVIEW OF LITERATURE

Potassium Fixation and Release

It is generally agreed that there are three forms
of potassium important for soil productivity; these are
the water soluble, exchangeable and fixed forms. The
main plant sources of potassium are believed to be the
water soluble and exchangeable forms. When the normal
equilibrium of these three forms in the soil is disturbed,
there is a conversion from one form to another. The
conversion of a portion of the soluble potassium, added
to the soil as fertilizer, to a non-exchangeable form
was noted by Dyer (14) in 1894 and Freer and Erb (15)
in 1918. This conversion of potassium has been termed
"fixation" and is defined by Joffe and Levine (25) as
"The conversion of soluble and replaceable potassium
into a form which is not released by the cations generally
employed in exchange reaction." Upon the removal of
exchangeable potassium from the soil by plants or by
leaching, fixed potassium is converted to the exchange-
able form. This process is called potassium "release."

Biological

Jenny (22) investigated the effects of organisms
on leaching of potassium from artificial and natural
soils. He found that systems inoculated with micro-
organisms lost less potassium by leaching than did

sterile systems. The addition of lime to inoculated systems further reduced the amount of potassium leached, while lime added to sterile systems increased the quantity leached. Jenny concluded that the lime caused a release of fixed and exchangeable potassium which was utilized by the organisms and fixed. The smaller loss of potassium from the limed inoculated series he attributed to the greater biological activity afforded by the lime.

Blume and Purvis (4) found potassium fixation highest in soils high in organic matter. They suggested that this may be due to the effect of micro-organisms. They also found an inverse relationship between water soluble and fixed potassium and since micro-organisms utilized the water soluble potassium in the soil they suggested that this may be indirect evidence of biological fixation.

Organic matter is incapable of fixing potassium and depresses its fixation in mineral soils according to Gourley and Wander (19) and Joffe and Levine (26). The latter workers state that the high exchange capacity of organic matter causes competition between the organic and mineral complexes for added potassium. Less potassium enters the mineral complex and consequently less is fixed.

Wood et al. (59) discounts the conception that biological fixation is of any consequence in the reversion of potassium to an unavailable form. Fixation of potassium was found to occur after boiling the soil samples in normal nitric acid. Furthermore, samples treated with 30% hydrogen peroxide fixed as much potassium as did untreated samples.

Phosphate Complexes

Joffe and Kolodny (24) report that iron, aluminum, calcium and magnesium phosphates are capable of fixing considerable amounts of potassium. A sample of 1.39 gms. of aluminum phosphate to which 20.28 mgms. potassium chloride was added fixed 4.20 mgms. of potassium when alternately wetted and dried at room temperature, and 11.77 mgms. when alternately wetted and dried at 70°C.

In field studies these workers found that fixation was higher in soils which received applications of acid phosphate, potassium chloride and lime than in soils which received acid phosphate and potassium chloride alone. They believed that the increase in pH afforded by the lime increased the phosphate mobility and hence the amount of potassium fixed. Although no explanation is advanced to clarify the mode of fixation from the results of the experiment, the following facts are emphasized:

1) It appears that any condition that facilitates the release of phosphate and its recombination with various cations favors the fixation of potassium.

2) Soils subjected to meteorological conditions of wetting and drying will fix potassium.

3) The quantity of potassium fixed, in part at least, will depend on the phosphate in circulation.

Other work showing the effect of the phosphate ion on potassium fixation is that of De Turk et al. (12) who found that more potassium is fixed when added as mono-potassium acid phosphate than as potassium chloride. When added as the acid phosphate a large proportion of the potassium became exchangeable; but when added as the chloride, the greatest proportion of potassium remained water soluble. The resulting high exchangeable potassium in the samples with added phosphate was considered the reason for higher fixation in the acid phosphate treated samples. These workers explained that the phosphate probably replaced hydroxide groups on the clay colloid, resulting in an extra valence which could accomodate the potassium ions. The field results of Joffe and Kolodny could readily be explained on this basis.

Micaceous Minerals.

It has been established, (26), (53), (41), that potassium fixation can be induced artifically by alternately wetting and drying soils or purified clay colloids. Volk

(53) found that little or no fixation occured under moist conditions, but when the soil was dried considerable fixation occured. Alternately wetting and drying at high temperatures was found to be the most rapid method of inducing fixation. Volk attributed fixation to the drying process. By X-ray examination of the .002 mm. fraction he found that some added potassium had reacted with colloidal silicates to form muscovite.

Page and Baver (36) studied ionic size in relation to fixation of cations by colloidal clay. They suggested that there is an entrapment of the potassium ions caused by the contraction of the expansible portions of the lattice of montmorillonite minerals on drying.

Considerable fixation was found to occur under moist conditions by Wood and De Turk (50 and Fine (15), when soil was stored a sufficient time to allow the apparently slow fixation process to occur.

Seatz et al. (45) found from X-ray studies that fixation tended to be higher in soils containing the highest proportion of mica and montmorillonite types of clay minerals.

Joffe and Levine (26) discovered that all the potassium released from seracite by electrodialysis is re-fixed on the addition of KCl solution upon alternately wetting and drying. They concluded that the potassium came from the mineral as part of its structure and suggested, in this instance, that potassium is fixed as part of the mineral structure.

The same investigators (31) found that pyrophyllite (the hydrous mica mineral from which montmorillonite and bentonite are built) did not fix potassium unless ground in a ball mill. The grinding increased both the exchange capacity and the amount of potassium which could be fixed. This was presumed to be due to the increase in surface area and number of interstices exposed by the grinding process.

They produced further indirect evidence that potassium is formed as part of the mineral structure by showing that ammonium fixation follows the same general pattern as potassium fixation. They quoted the work of Gruner (20) who treated vermiculite with ammonium hydroxide and converted it to ammonium mica. The ammonium became part of the mineral structure.

Joffe and Kolodny (25) studied the effect of heat on the potassium fixing powers of soils and montmorillonite. It has been shown by X-ray studies that the structure of montmorillonite is destroyed at 550°C. and the fixing powers of both soils and montmorillonite were destroyed at this temperature. This indicated that the crystalline clay colloids which have a definite lattice structure are capable of fixing potassium. Their work further showed that heating up to 200°C. increased the fixing powers of both soils and montmorillonite. They concluded that - "The rapid aging due to heating of certain active spots such as cavities,

lattice distortions, irregularities of the colloidal clay surface, holding exchangeable potassium ions, may also result in potassium fixation."

Bray (5), in a study of colloids of some Illinois soils, postulated that fixation of potassium is an adsorption process. More mature soils (more highly weathered) were found to fix greater amounts of potassium than more recent (relatively unweathered) soils. Furthermore, upper horizons showed in general more fixing power than lower, less weathered horizons. As the result of X-ray and petrographic studies Bray suggests that mica-like minerals, through direct alteration, form a beidellite-type mineral, probably a mica-beidellite colloidal partical. During this alteration process the mica loses its potash and takes on water and in this form has exchange properties. Prior to physical weathering the beidellite is still attached to the mica; after weathering much of the mica is broken off, and forms a part of the superfine fraction. These resulting beidellite-like particals, since part of their formation is due to a loss of potassium, would be expected to furnish conditions in which potassium could be fixed with varying degrees of tenacity:- the reforming of an old mineral, not the building of a new one as Volk (52) suggested.

Commenting on Volk's work, Bray claims that if potassium

were converted back to muscovite, it would be too insoluble
to take part in an equilibrium reaction between the non-
replaceable and replaceable forms.   Bray found the conver-
sion of potassium from the fixed to the replaceable form
slow compared to the usual base exchange reactions of
zeolite or montmorillonite type of materials.   This would
conform to the secondary type of mineral mentioned above.   /
"The partially weathered surface of practically any primary/
silicate mineral may offer an excellent opportunity for
movement of potassium both in and out of the lattice layers
made less compact by hydration and oxidation, but still
compact enough to prevent the usual speed of replenishment
shown by montmorillonite systems."

Joffe and Levine, in a series of articles (26), (27),
(28), (30), (31), proved almost conclusively that potassium
must be in the exchangeable form before fixation can occur.
They offered the following evidence in support of this theory:

1) A simple linear relationship is found to exist
    between the amount of potassium entering the
    exchange complex and the amount fixed. As the
    amount of potassium entering the complex increases,
    the amount fixed increases.

2) The rate of change of increase of potassium
    fixation with increased application reaches a
    maximum when the quantity of potassium is equal

to the exchange capacity of the colloid. When
a small quantity of potassium, in relation to the
exchange capacity, is added, the exchange capacity
evidently is not the limiting factor and a large
proportion is fixed. When a large amount of potass-
ium in relation to the exchange capacity is added,
only a small proportion of potassium is fixed,
although the absolute amount fixed is greater.

3) As long as the complex is saturated with potassium
the magnitude of fixation is unaffected no matter
how much potassium is added.

4) When potassium is added to samples of Manalto
colloid and hydrogen bentonite of different·
weights but with equal exchange capacity, fixation
is found to be equal.

5) The fact that organic matter reduces potassium
fixation by competing with the mineral complex
for potassium emphasizes the role of the mineral
exchange complex in the fixation process.

From the discussion above it is clear that the pheno-
menon of potassium fixation is not fully understood. However,
it seems probable that potassium is fixed mainly in the
interstices of weathered primary silicate minerals. If
such is the case there is no definite boundary between the

exchangeable and moderately available forms of potassium,
but merely a gradual increase in the tenacity with which
potassium is held. The contention that potassium is fixed
biologically or as a phosphate complex has been discredited
by the majority of investigators and is likely that they
play only a minor part in potassium fixation.

It is also apparent that potassium release occurs when
the quilibrium of the soil potassium is disturbed by removal
of the exchangeable form and the amount of potassium released
is then, directly related to the amount in the exchange
complex. Whether or not release will be rapid will depend
on the tenacity with which the fixed potassium is held. This
will be governed to a large degree by the amount of potassium
in the fixed form.

Practical significance

From a practical standpoint potassium fixation might be
regarded as the process which causes added potassium to be
rendered unavailable to the plant. Potassium release, on
the other hand, is the process which provides potassium to
the plant from the fixed or unavailable state. It is clear
then, that high potassium fixation in some cases, would be a
handicap. On the other hand, if potassium fixating powers
of the soil are low, there will be leaching losses of added
potassium and luxury consumption by the plant will be
encouraged.

Hoagland and Martin (21) conducted experiments in which soils were depleted of exchangeable potassium by continued cropping. They concluded that some soils would not maintain a "Physiologically effective" concentration of potassium in the soil solution and thus fixed potassium was of little practical value. According to these workers, when the exchangeable potassium is utilized by plants the solubility of the non-replaceable form of potassium will determine the potassium supplying power of the soil. They found that fixed potassium was unavailable to plants. Kolodny (29) corroborates this finding.

Under field conditions, where only one crop per year was grown, Wood et al. (60) demonstrated that the replaceable potassium content of a soil is higher in the spring than in the fall, but varies little from spring to spring. Thus, a constant reversion of potassium from the fixed to exchangeable form is indicated. This reversion keeps pace, more or less, with cropping and leaching, but lags during the growing season with more rapid potassium removal.

The cumulative potassium release is much higher from a potassium fertilized soil than an unfertilized soil, according to Wood and De Turk (59). The potassium added and not used by the crop is prevented from leaching by fixation. Later, it is able to provide the plant with potassium much more rapidly than in the case of the soil to which no potassium

has been added. In a laboratory experiment potassium was added to a soil as potassium chloride at the equivalent of one ton per acre. From one half to three quarters of the portion fixed was recovered in less than a year, when all replaceable potassium was removed at each of five successive leachings. In the soils studied it was estimated that under field conditions a crop would remove only one fifth of the exchangeable potassium per year; hence reversion would be expected proceed at a fairly steady rate and keep the exchange complex constantly supplied with potassium.

Seatz et al. (45) found that, at higher rates of potassium application, a lower percentage of potassium is recovered than at low rates of application and concluded that the amount of potassium fixed is related to the amount applied. Peech (40) supports Seatz and further points out that a heavy application of potassium fertilizer may, on some soils, cause a fixation process tending, in effect, to lower the available potassium.

Ayres (3) and others (33), (21), (59), have shown that, with an increased concentration of potassium fertilizer, there is increased sorption of potassium on the exchangeable colloid. According to Levine and Joffe (30) increased sorption will cause increased fixation.

Reitemeier (42), Attoe and Truog (3), and Rouse (44) have found positive correlation between crop extracted potassium and moderately available or acid soluble potassium.

From the foregoing, it is evident that soils vary considerably in their potassium supplying powers. In some cases a positive correlation has been found between crop growth and exchangeable potassium, but more frequently no positive relationship has been noted. It appears therefore, that exchangeable potassium is not always a reliable criterion. of a soil's potassium status with regard to crop production.

Effect of Lime

There has been a great deal of controversy regarding the effects of lime on potassium fixation and on the potassium of the exchange complex. One school of thought follows Leibig's contention that addition of lime liberates potassium to the soil solution. This view is held by many modern investigators and is strongly supported by Jenny (22).

Experiments carried out by Jenny with purified soil colloids, permutites, natural and artificial soils, revealed in all cases that lime liberated adsorbed potassium in large quantities. The more acid the soil, the more potassium was liberated by liming. He found that calcium chloride, calcium carbonate, and calcium hydroxide all liberated potassium from the complex in the presence of all important anions usually found in the soil solution.

Worsham and Sturgis (61), working with Missippi Delta soils found that calcium salts liberated potassium from a 100% base saturated soil. Calcium carbonate and tri-calcic

phosphate liberated only small amounts of potassium.

Many papers dealing with the effect of lime on potassium fixation conclude that lime induces fixation. MacIntire et al. (32) found, in lysimeter studies with Tennessee soils, that every application of lime effected a repression on the outgo of potassium, the effect becoming greater with increased rates of liming. The variously limed soils in this experiment were given 200 pound applications of potassium chloride yearly for six years. Periodically the amount of potassium in the leachate was determined.

Allaway and Pierre (1) found that non-calcareous soils fix little potassium, and that an excessive application of lime contributes to potassium fixation with a resulting low potassium fertility.

Other workers (48), (4), (15), (45), (17), have obtained similar results in both field and laboratory studies. The general viewpoint held by these workers is that lime is capable of inducing potassium fixation and that overliming of some soils is likely to result in potassium deficiencies.

Several investigators have offered explanations of the effect of lime on potassium fixation. Wicklander (56) found that cations with properties and ionic size similar to potassium decrease fixation. Others increase fixation, the extent being determined by the relative ease with which they are displaced by potassium. Ammonium, potassium and hydrogen

systems result in low potassium fixation while magnesium, barium, calcium, and sodium systems result in high fixation. Wicklander contends that cations adsorbed or added, decrease the fixation according to their replacing power or affinity for the colloid. If the competing ions are capable of filling the vacant places on the mineral lattice resulting from the release of fixed potassium, they will exhibit greater influence on subsequent additions of potassium. This will result in a low fixation.

Peech and Bradfield (39) studied the effect of lime and neutral calcium salts upon the solubility of soil potassium in some Florida soils. They demonstrated that the amount of potassium adsorbed by a colloid clay complex is greatly increased by increasing the degree of calcium saturation. Peech (37), in a later paper, found that the exchangeable potassium content generally increases with increased pH or degree of base saturation, even where similar amounts of potassium have previously been applied. He indicates that potassium replaces calcium more readily than it does hydrogen due to the relative position of potassium, hydrogen, and calcium in the lyotropic series. Since there are greater amounts of calcium present at higher pH values the efficiency of the complex for absorbing potassium will be greatly enhanced.

Levine and Joffe (31) contend, however, that it is not

the increase in calcium on the complex that allows more potassium to be adsorbed, but the resulting increase in pH. They found that when the calcium ion is increased at any one pH, the fixation of added potassium is reduced. This, they claim, is due to the calcium being more successful in competing for the exchange position. With an increase in pH there is an increase in the amount of potassium which will enter the complex, and hence an increase in the amount of potassium which will become fixed.

Ayres, (3), in a study of sorption of potassium in Hawaiian soils, produced evidence indicating that the concentration of potassium added is of equal if not greater importance than the degree of calcium saturation in increasing the potassium on the complex.

The above review illustrates the divergence of opinion regarding the effect of lime on soil potassium. It would seem safe to conclude that this effect will vary from one soil to another.

Methods of potassium extraction and analysis.

Standard Methods

Extraction

The most common extracting solutions used for the displacement of exchangeable cations include neutral normal ammonium acetate, neutral 0.5 N ammonium chloride, normal barium acetate, barium chloride, sodium acetate, and sodium nitrate. Some workers have used acid extractants, the most common being 0.5 N acetic acid, 0.1 N and 0.05 N hydrochloric acid.

Schollenberger and Simon (47) point out that exact equivalents cannot be expected when comparing the amounts of bases extracted by different solutions. Golden et al. (18) verified this using soils varying in texture, pH range, organic matter and carbonate content and leaching them with ammonium acetate, ammonium chloride, barium chloride, potassium acetate, barium acetate, ammonium formate, acetic acid and 0.1 N hydrochloric acid. The potassium acetate proved to give the highest results for total exchangeable bases while ammonium acetate, barium acetate, and ammonium chloride gave variable potassium results when compared to the other extractants. In general the acetate ion, regardless of the cation attached to it, produced the highest and most consistant results for both exchangeable bases and total exchange capacity. The acid extractants tested gave extremely low results.

Results.

From the results obtained by Golden et al. (18) and other investigators (26), (53), (48), (60) the neutral normal ammonium acetate extracting solution seems the most favorable for potassium studies. Advantages of this extractant are outlined in detail by Schollenberger and Dreibelbis (46).

The extraction of exchangeable bases with ammonium acetate is usually accomplished by a leaching procedure. Various leaching methods are described in the literature. Peech (38) leached 25 gms. soil in a Buchner funnel with small portions of ammonium acetate until a volume of 225 ml. was obtained. Joffe and Levine (26) used a similar method for extracting exchangeable potassium. Volk (54) extracted potassium by agitating 30 gms. of soil with 450 cc. ammonium acetate for 15 minutes and leaching ten times with 25 cc. portions of the solution.

Schollenberger and Dreibelbis (46) recommends leaching the soil in a closed system to prevent loss of ammonium and to prevent carbon dioxide from entering the system and thus affecting the bases dissolved. He reports higher results by this method than by open system techniques. A soil to ammonium acetate ratio of 1:7.5 or 1:10 was found to extract all exchangeable bases when leaching was continued for a minimum of four hours. Leaching for over 24

hours increased the amount of cations extracted.

The leaching procedure described by Scholl-
enberger and Dreibelbis is less troublesome than and does
not require the continuous attention of the other methods
described.

Analysis

Many methods of analysis have been adapted to
soil potassium determinations. The classical gravimetric
method using platinic chloride as the precipitant is not
used extensively due to the high cost of the reagent. The
most commonly used techniques involve the precipitation of
potassium as the dipotassium sodium cobaltinitrite with
subsequent gravimetric or volumetric determinations.
Wander (55) describes a colorimetric method using potassium
dichromate to oxidize the cobaltinitrite precipitate.
Titration of the precipitate with ceric sulphate is des-
cribed by Brown (10). The gravimetric method and volu-
metric method using potassium permanganate as the oxidant
described by Wilcox (57) are probably the most widely used.

Wilcox reports an absolute accuracy of ±0.05
mgms potassium by both the gravimetric and volumetric tec-
hniques. Temperature of precipitation is important and
variations from 20°C. will produce erroneous results. The
precipitation is very sensitive to the presence of ammonia.

Calcium and magnesium to 0.2N, barium to 0.1N, and sodium to 1.0N are reported to have little effect. The presence of acetate causes high results and sulfate and chlorine in high concentrations cause low results.

Rapid Methods

Extraction

Rapid methods of extracting and determining available plant nutrients often sacrifice accuracy for rapidity. During recent years, however, methods have been developed which satisfy both of these requisites. Bray (6) points out that a successful rapid test must conform to certain requirements. Firstly, the extracting solution and procedure used should extract all or a proportionate part of the available forms of a nutrient from the soil. Secondly, the amount of the nutrient in the extract should be measured with reasonable accuracy and speed. Finally, the amounts extracted should be correlated with the growth and response of individual crops to that nutrient under various conditions.

Bray (7) found a 20% sodium nitrate solution satisfactory for potassium extraction. He refers to the potassium extracted by this solution as exchangeable potassium and has used it widely in correlation studies with plant response. The extraction procedure entails shaking 5 gms of soil with 10 mls of the extracting sol-

ution for one minute in a small vial. The system is then
filtered and the filtrate taken for analysis.

Morgan (34) recommends extraction of available
nutrients with normal sodium acetate buffered at pH4.8
using a 1:2 soil to extractant ratio. Detailed studies of
the sodium acetate method include that of Tinsley and Pizer
(42) who found that Morgan's solution extracted an average
of 46% of the exchangeable potassium determined by extr-
action with 0.5N acetic acid. On sandy soils the exchange-
able potassium extracted was as high as 55%. The same
investigators studied the effect of time of shaking the
soil with the sodium acetate solution on the amount of
nutrients extracted. The percentage increase of potassium
extracted due to increasing time from one minute to 15 min-
utes averaged 2.9 for the 17 soils tested. They found that
a 1:10 soil to extractant ratio removed an average of 7.3%
more potassium than a 1:2 ratio. The Morgan method was
compared to the acetic acid method on a series of soil
samples. As the percent potassium extracted by the acetic
acid method increased from 8 to 50% the potassium extracted
by the sodium acetate method increased from 3 to 32%.

Peech suggests using a 1:5 soil to extractant
ratio and shaking 10 grams of soil with 50 mls of Morgan's
sodium acetate extractant for 30 minutes on an automatic
shaking machine. The system is then filtered and tests for
the exchangeable bases are conducted, using the filtrate.

Analysis

Almost without exception some form of the cobalt-initrite precipitation is used for the rapid estimation of soil potassium. The main differences in the various procedures are in the ratio of sodium nitrite to cobaltic nitrite used in the preparation of the precipitating reagent and the type of alcohol used to decrease the solubility of the dipotassium cobaltinitrite precipitate. Some workers use formaldehyde to minimize the interference of ammonia and gum arabic to keep the precipitate in suspension for photmetric determination.

Methods of estimating the concentration of the turbid dipotassium cobaltinitrite solutions vary. The majority of workers use colour or line charts, or compare the turbidity to standard solutions. Recently methods of photometric determination, with a resulting increase in accuracy, have been developed.

Peech and English (40) claim that not enough emphasis has been laid on attempts to minimize the errors in the analytical procedure. They point out that the main difference between the rapid and more tedious techniques is that the rapid tests are used in aliquots of a soil extract without subsequent treatment. Thus, erroneous results may readily be caused by interfering ions and they attempted to minimize these effects by introducing slight changes in classical techniques. Thus, they found that the

effect of organic matter was reduced by addition of organic matter was reduced by addition of activated charcoal to the extracting system and that the use of iso-propyl alcohol instead of ethyl alcohol increased the temperature range over which consistent results could be obtained. However, iso-propyl alcohol decreased the sensitivity of the determination. This was improved by increasing the ratio of sodium nitrite to cobaltic nitrite and their concentrations. Ammonia interference was eliminated by the use of formaldehyde.

The Peech and English method of potassium precipitation is an adaption of the two layer technique. Two milliters of soil extract and 6 drops of formaldehyde are added to a large vial and allowed to stand for 5 minutes. One milliliter of the sodium cobaltinitrite solution and 2 mls of iso-propyl alcohol are added. The latter is added slowly so as to form a layer. The two layers are mixed rapidly and uniformly by swirling for 30 seconds. In 25 minutes the turbidities are compared with standards over light using lines of 1, 2 and 3 mms in thickness drawn on cellulose acetate paper.

This method has been adapted to photometric estimation by several workers, Eldon (43), Wolf & Ichisaka (58), and is found to be quite satisfactory. Wolf and Ichisaka (58) used gum arabic as a protective colloid to aid in the formation of uniform small sized particles of

precipitate that remain in suspension for a long time.

Bray (7) describes a more rapid method of potassium estimation. The soil extract is forced into the ethyl alcohol-cobaltinitrite solution by means of a hypodermic syringe, insuring rapid and complete mixing of the two solutions. The turbid solution is then read on the photometer and the concentration is found by comparing the photometer reading to a standard chart. Bray states that the standard chart should be checked each time a new reagent is used, and prior to each set of determinations and that the temperature should not vary more than a few degrees.

Other methods include those of Spurway, (49), Morgan (34), Truog (52) and Thorton (51), most of which are adaptable to use in the field. All of these techniques use the same principles for potassium precipitation as described above. Available information indicates that they are somewhat less accurate than the Peech and Bray methods and their various modificiations.

EXPERIMENTAL

Study of Methods of Potassium Analysis

The volumetric and gravimetric procedures of potassium analysis described by Wilcox (57) and the rapid methods of Spurway (49), Morgan (34), Peech and English (40) and Bray (7) were studied using standard potassium solutions.

Volumetric Method

Table 1 shows the recovery of standard potassium samples by the volumetric method described by Wilcox (57). The percent potassium recovered at the 1.5 mgm level is low. Although this method is accurate within the desired ±2% range at levels of 4.5 mgms to 9 mgms of potassium it proved longer and not as well adapted to the laboratory conditions as the Wilcox gravimetric technique (57).

TABLE 1

Recovery of Potassium Standards: Volumetric

Method.

Potassium added as KCl mgms	Potassium Recovered mgms		Means	Percent Recovery
	1	2		
1.5	1.196	1.201	1.198	79.8
4.5	4.585	4.583	4.584	101.8
7.5	7.477	7.480	7.478	99.6
9.0	9.080	9.112	9.096	101.0

Gravimetric Method

Wilcox reports a recovery of ±2% by the gravimetric procedure between 2 and 15 mgms of potassium present. It is evident in Table 11 that the desired accuracy was not obtained with potassium standards containing less than 4.5 and above 15 mgms. Between 4.5 and 15 mgms the reproducibility and absolute accuracy was 0.08 mgms or 2.6×10^{-3} me of potassium.

TABLE 11
Recovery of Potassium Standards: Gravimetric Method.

Potassium added as KCl mgms	Potassium Recovered mgms				Means	Percent Recovery
	1	2	3	4		
1	.757	.634	.932	.777	.775	77.5
3	2.581	2.792	2.751	2.944	2.767	92.4
4.5	4.463	4.482	4.459	4.468	4.468	99.3
6	5.783	5.921	5.940	5.896	5.885	98.1
9	9.211	9.106	8.860	8.904	9.020	100.2
12	12.120	12.189	12.309	12.001	12.155	101.2
15	15.228	15.263	15.360	15.413	15.331	102.1
18	17.664	17.318	17.412	17.841	17.559	97.5

Effect of Common Soil Cations.

The effect of cations commonly found in soil extracts on the accuracy of the gravimetric procedure is given in Table 11. One-tenth normal magnesium and calcium and 0.75 N sodium had no effect on the accuracy of the determination

while higher concentrations interfered. Since these cations are seldom found in such high concentrations in soil extracts their removal will not generally be required.

Removal of ammonium is imperative, since its prsence, even in small amounts will cause erroneous results. It is desirable to test the solution for ammonia with Nessler's reagent prior to the addition of the rtrisodium cobalt-initrite solution. This is particularly important in laboratories where ammonia fumes are likely to be present.

TABLE 111

Effect of Common Soil Cations on the Recovery of Potassium: Gravimetric Method.

Potassium in Standards = 9 mgms.

Cation added	Concentration	Potassium Recovered mgms		Means	Percent Recovery
		1	2		
Ca	0.1N	9.090	9.202	9.146	101.7
	0.3N	10.106	9.412	9.759	108.5
Mg	0.1N	9.262	9.034	9.148	101.6
	0.3N	9.820	9.614	9.717	108.0
Na	0.75N	8.920	9.111	9.015	100.1
	1.00N	9.203	9.312	9.257	103.0

Turbidimetric Methods

Spurway's Method

The rapid method of potassium estimation outlined by Spurway (49) was tested on standard solutions and acetic

acid soil extracts. A great deal of variability in the
turbidity of replicate samples was noted. The standards,
when compared to the Spurway chart and prepared line charts,
showed that only a narrow range of concentrations could be
estimated. Further study of the method was considered
unwarranted as it did not show the degree of accuracy and
precision required.

Morgan's Method

The Morgan (34) method of potassium estimation
was found to be less variable than that of Spurway. Stand-
ard solutions of 20, 40, 60 and 80 ppm potassium averaged
readings of 1, 2, 6, and 10 units respectively on the
Morgan chart. These readings were found to vary, however,
if the cobaltinitrite-sodium nitrite ratio varied or if old
sodium cobaltinitrite solution was used.

The procedure was modified for estimation on the
photometer. One milliter of soil extract or standard
solution was transferred to a photometer micro tube. Two
tenths of a milliter of Morgan's reagent "A" (sodium cobalt-
initrite solution) and 2 mls of reagent "B" (iso-propyl
alcohol and formaldehyde) were added. The solution was
allowed to stand one minute and was then mixed thoroughly
by shaking for 30 seconds with a rotary movement.

When the solution was read on the photometer
between five and fifteen minutes after shaking less vari-

ation occured than when left for longer intervals. This
is illustrated in Table lV.

TABLE lV

Effect of Time of Standing on Photomoter* Readings:
Modified Morgan Method.

Standard Concentration ppm	Photometer Readings (Log Scale) Time Interval in Minutes		
	5	10	15
20	15.9	15.6	15.0
20	11.4	11.0	10.0
20	11.7	11.6	10.2
50	52.2	51.8	51.0
50	54.0	53.6	51.8
50	51.5	51.2	50.1

The variation in photometer readings on replicates of
each standard is shown in Table V. The variation is often
as large as four units on the photometer - logarithmic
scale and occasionally varies by seven. Figure 1 shows
the average of the four readings for each standard plotted
against the standard concentration. The points do not fall
close enough to a straight line to give accurate results.
For higher accuracy larger number of replicates would be
required.

*Fisher A C Model Electrophotometer

FIG. I   Morgan Turbidometric Method

Standard curve showing potassium concentrations

plotted against photometer readings

TABLE V

Replicate and Average Photometer Readings
for Standard Potassium Solutions: Mod-
ified Morgan's Method.

Standard concen- trations ppm	Replicate Photometer Readings (Log Scale)				Means
	1	2	3	4	
20	16.9	12.4	12.9	14.2	14.1
40	39.3	32.0	36.5	35.3	35.9
50	52.2	54.0	51.5	50.3	52.0
60	57.2	65.0	61.0	64.8	62.0

Peech's Method

Peech's and English's (40) turbidimetric method
for potassium estimation was tested on standard potassium
solutions using the photometer. The variations between
photometer readings for each standard were fairly large
and averages of photometer readings plotted against con-
centrations of the standards did not fall on a straight
line. These errors were apparently due to uneven mixing
of the alcohol with the formaldehyde-cobaltinitrite solution.
Peech points out that experience in mixing the solutions is
required in order to obtain duplicate results.

It was found that this difficulty can be overcome
by a slight modification of the Peech procedure. If the soil

extract formaldehyde solution is foreed into the alcohol-cobaltinitrite solution by means of a pipette, a rapid and complete mixing is ensured. With this modification the variations between replicate readings between any one standard are not more than 3.5 divisions and usually less than 3 divisions on the logarithmic scale as shown in Table Vl. As evident in Fig. 11 the standard graph is a straight line, and hence follows Beer's Law, between 20 and 60 ppm. The solution should stand 25 minutes between mixing and reading on the photometer for most accurate results. This is illustrated in Table Vll.

TABLE Vl

Replicate and Average Photometer Readings for Standard Potassium Solutions: Modified Peech Method.

Standard concentrations ppm	Replicate Photometer Readings (Log Scale)				Means
	1	2	3	4	
10	4.1	4.0	3.5	4.4	4.0
20	27.8	27.0	26.5	26.7	27.0
30	38.0	33.5	36.0	35.7	35.8
40	47.5	51.0	48.0	48.7	48.8
50	56.6	58.0	57.5	55.9	57.0
60	67.0	64.5	65.3	64.0	65.2
70	70.0	70.6	72.8	70.6	71.0

FIG. II Modified Peech Turbidometric Method

Standard curve showing potassium concentration

plotted against photometer readings

TABLE V11

Effect of time of Standing on Photometer Readings:
Modified Peech Method.

Photometer Readings (Log Scale)

Standard Concentration ppm	Time Intervals in Minutes		
	15	25	35
30	36.8	36.0	35.9
30	34.0	33.5	33.5
30	38.6	38.0	37.8
50	57.1	56.6	56.4
50	58.8	58.0	57.5
50	57.8	57.5	57.5

Peech reports that ammonia does not influence the determination when present in quantities lower than 1000 pounds per acre. This was confirmed for quantities up to 750 lbs per acre which were found to have no effect in this trial. However, at 1000 lbs. per acre, a small effect was noted.

The Peech method is readily adapted to the estimation of potassium by use of line charts. Although the method outlined by Peech was tried, more consistant estimations over a wider range of potassium concentrations were possible when the line chart technique described by Bray was used. The accuracy compares with that reported in the following section.

Bray's Method

Of these tested the turbidimetric method of potassium analysis described by Bray (7) proved to be the most precise. The variations seldom exceeded two units on the logarithmic scale as shown in Table Vlll. Figure lll illustrates that Beer's Law is satisfied between 20 and 60 ppm. when average photometer readings are plotted against standard concentrations. Table lX indicates that the most precise results are obtained when the solution is read on the photometer between 5 and 15 minutes after precipitation.

The effect of ammonia was tested and it was found that the equivalent concentration of 500 lbs. per acre and above resulted in high readings on the photometer, Ammonium was added to the greenhouse soils at the rate of 300 pounds per acre nitrogen and had no effect on the Bray determination eight weeks later. It is probable that the ammonium present in field soils would not affect this determination. However, further tests should be made at lower concentrations to definitely establish the level at which interference can be expected.

The use of the line chart technique for estimating the potassium described by Bray proved to give reasonably reliable and reproducible results. The accuracy of potassium estimation by this means would undoubtedly
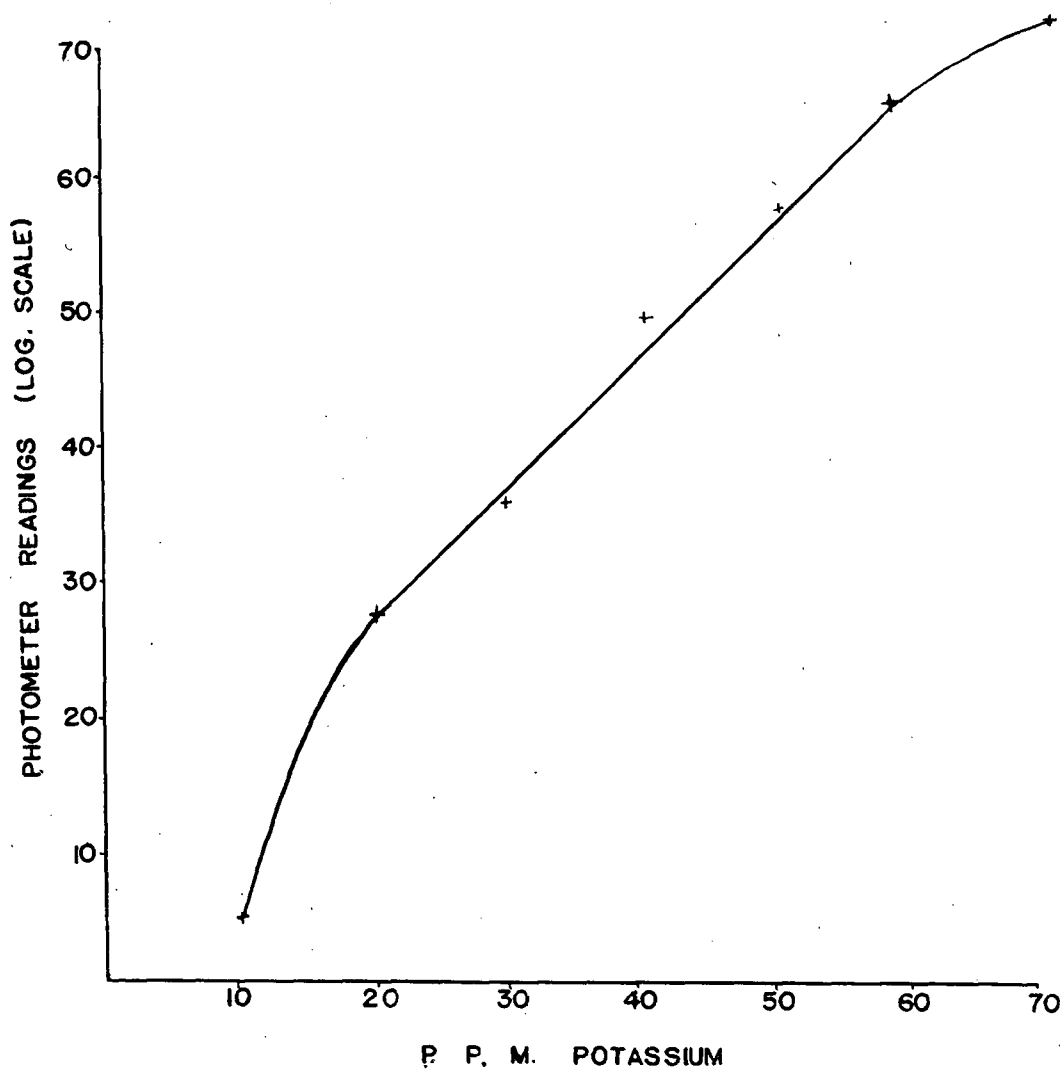
FIG. III   Bray Turbidometric Method

Standard curve showing potassium concentration

plotted against photometer readings

increase with the experience of the worker. The variation
between operators is minimized, since each individual must
prepare his own standard chart. Standard solutions of 10,
20, 30, 40 and 50 ppm of potassium are prepared in tri-
plicate and precipitated by Bray's technique. The average
of the number of milliliters of each concentration required
to obscure a standard line is plotted against the concen-
tration of the solution. The average of duplicate deter-
minations from each soil extract treated in the same way
is then read directly in parts per million from the graph.
In Figure 1V it will be noted that the most accurate
estimations are possible between 10 and 50 ppm. A satis-
factory estimate of exchangeable potassium for routine
soil testing work is possible by this method.

TABLE V11l

Replicate and Average Photometer Readings for
Standard Potassium Solutions: Bray Method.

Standard Concentrations ppm	Photometer Readings (Log scale)				Means
	1	2	3	4	
10	6.0	6.8	6.5	6.6	6.5
20	15.0	15.0	16.2	14.0	15.0
30	21.5	22.0	23.0	23.5	22.5
40	30.4	31.0	30.0	31.1	30.6
50	38.5	37.5	38.5	36.5	37.7
60	45.2	45.0	44.8	44.6	44.9
70	46.0	47.5	48.6	46.1	47.1

FIG. IV Bray Line Chart Method

Standard curve showing standard potassium concentrations

plotted against the amount of turbid $K_2NaCo(NO_2)_6$

solution required to obscure a standard line

TABLE 1X

Effect of Time of Standing on Photometer Readings:
Bray Method.

Standard Concentration ppm	Photometer Readings (Log Scale) Time Interval in Minutes		
	5	10	15
20	15.0	14.8	13.8
20	15.0	14.8	13.6
20	16.2	15.9	15.0
50	38.5	38.2	37.6
50	37.5	37.4	36.8
50	38.5	38.3	37.7

The Bray determination is extremely sensitive to temperature variation. Accurate replicate results could be obtained only when the temperature of the reagents used was between 18° and 22° C. The reagents are readily kept at this temperature by storing them in a water bath.

Since the Spurway and Morgan potassium tests were found to give less precise results than the other rapid methods tested, further studies are confined to the Bray and modified Peech techniques. The gravimetric procedure is used for estimating the ammonium acetate extracted potassium.

Study of Methods of Potassium Extraction.

Ammonium Acetate Extraction

Preparation of the Ammonium Acetate Filtrate for Potassium Analysis

The ammonium acetate filtrate was prepared for analysis by a variation of Schollenberger and Dreibelbis (47) method. The solution from the ammonium acetate leaching was transferred to a 600 ml. beaker and evaporated to dryness on a steam bath. The sides were washed down with hot water from a wash bottle and the system evaporated again. This procedure was repeated until a foamy residue resulted, thereby hastening the subsequent removal of organic matter. Organic matter was then destroyed by evaporating 10 c.c. portions of 6% hydrogen peroxide and 6 drops of nitric acid until a white residue remained.

The residue was taken up with 15 c.c. of 6 N nitric acid and 50 ml. water. This solution was heated to 60° - 70° C. and filtered to remove the silica, after which the filter paper was washed with hot water from a wash bottle. The resulting filtrate was tested for ammonia with Nessler's reagent and evaporated to dryness on a steam bath. If a positive Nessler's test resulted the ammonia was driven off by flaming the residue with a bunsen burner.

Completeness of Potassium Removal from Soils

The completeness of removal of exchangeable potassium from the soil by ammonium acetate was studied. In the determination of exchange capacity the time of leaching was found to have no effect between 4 and 10 hours. This would indicate that all exchangeable potassium is removed by one leaching. To ensure complete potassium removal, four soils of varying textures were leaching twice with 500 mls of ammonium acetate. No potassium was detected in the second leachates.

Table X indicates the completeness of removal of added potassium by ammonium acetate. It will be noted that errors due to both extraction and analysis do not exceed ± 2%.

TABLE X

Recovery of Potassium Added to the Soil:
Ammonium Acetate Extraction.

Soil Texture	Potassium Added mgms/100gms	Original Exchangeable Potassium mgms/100 gms	Potassium Recovered mgms/100gms	Percent Recovery
Sandy Loam	6	11.090	17.201	99.3
			16.892	98.8
Clay Loam	6	19.781	25.390	98.3
			25.382	98.4
Loam	4	9.480	13.232	98.2
			13.290	98.6

Rapid Extracting Methods

From the results of Tinsley and Pizer (50), ex-
traction of potassium by Morgan's with sodium acetate
for longer periods than 15 minutes failed to give sig-
nificant increases in the amounts of potassium dissolved.
The 30 minute shaking recommended by Peech was thus
considered adequate for the soils studied.

The effect of time of shaking of the potassium
extracted by sodium nitrate was tested. A clay and
sandy loam soil were each shaken in the sodium nitrate
solution for periods of 1, $1\frac{1}{2}$, 2, $2\frac{1}{2}$ and 3 minutes. No
significant differences in photometer readings were
noted when the leachates were determined for potassium
by the Bray technique.

Comparison of The Modified Peech, Bray and Standard
Methods.

In Table Xl, the amounts of exchangeable potassium
extracted and determined by the modified Peech and Bray
procedures are compared to that extracted and determined
by the standard ammonium acetate-gravimetric method.
At the lower levels of potassium the variation of both
rapid methods from thestandard determination is small.
However, at higher levels only slightly more than half
of the potassium extracted by ammonium acetate was

removed by the rapid methods. The sodium acetate re-commended by Peech and English, extracts slightly less potassium than the sodium nitrate used by Bray. Both methods show a high significant correlation to the standard technique as shown in Figures V and V1.

It is evident that both rapid methods give fairly accurate and reproducible estimations of exchangeable potassium. The Peech method, while longer than that of Bray, has an advantage in that all the exchangeable cations may be determined on the leachate. For studies of potassium only, the Bray technique would be the most satisfactory.

Bray's rapid method for the estimation of exchange-able potassium is further compared to the standard ammonium acetate-gravimetric method in potassium fixation and release studies.

TABLE X1

Table Comparing the Potassium Extracted and
Determined by the Bray, Modified
Peech and Standard Methods.

Exchangeable Potassium   (me/100gms)

Soil	Standard Method	Bray Method	Peech Method
3	.902	.501	.346
4	.361	.262	.208
14	.365	.292	.228
12	.174	.176	.095
16	.796	.476	.364
11	.085	.112	.077
19	.818	.547	.453
15	.504	.305	.318
20	.961	.621	.595
21	.385	.296	.265

FIG. V Graph showing relation between standard ammonium acetate

extraction with Wilcox Gravimetric Analysis and

sodium nitrate extraction with Bray Turbidometric Analysis

FIG. VI Graph showing relation between standard ammonium acetate
extraction with Wilcox Gravimetric Analysis and sodium
nitrate extraction with Modified Peech Turbidometric Analysis

Potassium Fixation and Release Studies of Some Lower Fraser Valley Soils

Soils sampled and sampling procedure

The sampling areas were chosen in order to give a range of both soil type and crop response to potassium. All sites chosen were on or adjacent to areas where rod-row experiments with oats had been conducted by the Dominion Experimental Farm during the past four years. A Munroe silty clay loam, a Munroe silt loam, and a Ladner clay were sampled to represent the lowland soils. A Whatcom loam and a Lynden silt soam were sampled to represent the upland soils. Five samples of the plowed layer and five of the subsoil to nine inches below the plowed layer, each were composited and mixed well on a tarp in the field. A sample of about 50 pounds was taken to the laboratory, quickly air dried and stored in sacks. Samples for analysis were screened through a 2 mm seive and stored in cardboard cartons.

A description of the soils and notes on oat response to potassium fertilizer is given in the Appendix.

Exchange Capacity and Base Saturation

The Schollenberger and Dreibelbis (47) method for estimating total exchange capacity and base unsaturation was tested. After leaching the soil with ammonium acetate

The excess ammonium was removed by leaching with neutral 80% ethyl alcohol. The ammonium remaining on the soil complex was determined by the Kjehldal method. The milli-equivalents of ammonium per 100 gms of soil was taken as the total exchange capacity.

Exchangeable hydrogen was determined on the ammonium acetate filtrate by titrating to pH 7 with ammonium hydroxide using the glass electrode. The milli-equivalents of ammonium required was taken as the milli-equivalents of exchangeable hydrogen.

This method was not found to be precise and in order to obtain a better estimation of base unsaturation the method described by Brown (9) was used. In this method exchangeable hydrogen is determined by shaking 2.5 gms of soil in 25 mls of neutral normal ammonium acetate intermittently for one hour. The pH of this system is then determined with the glass electrode and the milli-equivalents of exchangeable hydrogen read directly from a titration curve. The curve is prepared by titrating 1.0N acetic acid with dilute ammonium hydroxide using the glass electrode and plotting pH against milli-equivalents of base required.

Total exchange capacity, base saturation, pH and exchangeable potassium determined by the Bray and standard methods are given in Table Xll. The exchange-

able potassium generally increases with the degree of base saturation in the surface soils. In the sub-soils, however, even though the base saturation is higher than in the surface soils the potassium is lower in every case.

The response of oats (see Appendix) to potassium application bears no apparent relationship to the exchangeable potassium. This is not surprising, since the exchangeable potassium in the soils is fairly high. Thus, it was thought desireable to study the relative potassium supplying powers of these soils.

Potassium Supplying Powers

A laboratory study of the relative potassium supplying powers of the soils was made by a modification of the method of Bray and De Turk (8). These workers found that if soils were leached of all their replaceable potassium and stored under moist conditions for long periods of time potassium would be released from the fixed form. The release of potassium in soils from which the exchangeable potassium had not been leached supplied insignificant amounts of potassium during moist storage.

Thirty 35 gm samples of both surface and sub-soil of each soil type were leached with 500 mls ammonium acetate. An additional 100 cc ammonium acetate was then leached through the soils and analysed for potassium to ensure complete removal. The excess ammonium was removed with

neutral 80% ethyl alcohol and the soils transferred to petri dishes. Two samples each of the surface and sub-soils were stored for one month, two month, and three month periods. Moisture was maintained at 30% by weight with distilled water. After storage the soil was leached with ammonium acetate and the potassium determined by the gravimetric procedure.

In no cases did the soils studied supply detectable amounts of potassium when stored under moist conditions for periods of one, two, and three months. To determine whether a more drastic treatment would cause potassium release, some of the soils were leached with ammonium acetate, washed clear of excess ammonium with 80% ethyl alcohol and alternately wetted and dried 10 times at 100° C. The soils were then releached with ammonium acetate and the filtrate prepared for potassium analysis. Again, no potassium was detected.

The negative results of this experiment does not necessarily mean that under field conditions of alternate wetting and drying, freezing and thawing over long periods these soils will not supply potassium from the fixed form. From the results reported by Clarke (11) and from the results of fertility trials the opposite is probably true.

Due to the negative results obtained by use of the standard method study of potassium release by the Bray technique was not attempted.

TABLE XII

Table Showing Chemical Properties of the Soils Studied

Soil	Depth	pH	Total Exchange Capacity me/100 gms	Exchange-able Bases me/100 gms	Exchange-able Hydrogen me/100 gms	Percent Base Satur-ation	Exchangeable Potassium me/100 gms	
							Standard Method	Bray Method
Munroe SiCL	0-9"	5.20	29.69	16.99	12.70	57.1	.902	.501
	9-15"	5.30	28.51	18.51	10.00	65.0	.361	.292
Ladner C	θ-9"	5.30	23.72	11.02	12.70	46.6	.365	.262
	9-15"	4.98	23.01	14.51	8.50	63.0	.174	.176
Munroe SiL	0-9"	5.60	34.14	22.44	11.70	65.8	.796	.476
	9-15"	5.65	21.23	14.93	6.30	70.4	.085	.112
Whatcom L	0-9"	5.72	21.97	13.87	8.10	63.2	.818	.547
	9-15"	5.90	20.28	12.28	8.00	60.5	.504	.305
Lynden SL	0-9"	5.90	19.72	13.42	6.30	68.0	.961	.621
	9-15"	5.82	14.22	7.97	6.25	56.0	.385	.296

Potassium fixation

Studies were carried out to determinee the magnitude
of potassium-fixation on a limed and unlimed series of
soils. The limed series was brought to pH 8.2 - 8.4
with calcium hydroxide. The amount of calcium hydroxide
required was determined by use of buffer curves as outlined
by Dunn (13). These were prepared by shaking 10 gms. of
each soil with varying increments of 0.05 N calcium
hydroxide in 50 ml. or more of solution for half an hour.
The pH of the resulting systems was plotted against the
gms. of calcium hydroxide used. Fixation was induced
artificially by a modification of the method outlined by
Joffe and Levine (1).

Thirty-five milliliters of a potassium chloride
solution was added to 35 gms of soil in a petri dish.
The concentration of the potassium chloride solution was
prepared so that 1000 parts of potassium per million
parts of soil was added. The system is then alternately
wetted and dried give times in an oven at 110° C. Thirty-
five milliliters of distilled water was used for each
wetting.

Following the final drying 20 gms of the oven dry
soil was leached with 200 mls of ammonium acetate and the
filtrate prepared for gravimetric analysis. Two five
gram samples were extracted and analyzed by the Bray tech-
nique. The sum of the potassium added and the initial

exchangeable potassium minus the potassium extracted was taken as the amount of potassium fixed. From Table Xll it is evident that a considerable proportion of the added potassium was fixed when determined by both the ammonium acetate and sodium nitrate methods. However, the magnitude of the fixation was greater when determined by the former technique. Furthermore, those soils which showed highest fixation by the ammonium acetate method gave the lowest fixation with sodium nitrate extraction. The opposite condition was also apparent.

The effect of lime on potassium fixation is summarized in Table Xlll. Again, the effects observed were different for the two extractants. No final explanation can be given for the apparent release of potassium due to lime as indicated by the ammonium acetate extraction and the increase in fixation due to lime when the sodium nitrate was used. However, it appearsthat the explanation lies in the relative replacingpowers of NH_4^+ and Na^+ when Ca^{++} or H^+ ions dominate the exchange complex. Apparently, the replacing power of sodium is higher when H^+ is present in high concentrations. Although no other evidence of this was found in the literature review, it is known that the apparent replacing power of ions is affected by the nature of the ions present on the complex. This suggests that in studies of potassium fixation and release special attention should be given, not only to the extractant,

TABLE X111

Potassium Fixed by Alternately Wetting and Drying
the soils at 110 C. after the Addition of
.256 me/100 gms Potassium.

Soil	Potassium Fixed, me/100 gms	
	Standard Method	Bray Method
Unlimed series		
Munroe SiCL	2.015	0.860
Ladner C	1.469	1.177
Munroe SiL	1.954	0.680
Whatcom L	1.737	0.946
Lynden SL	1.819	0.773
Limed Series		
Munroe SiCL	1.984	1.212
Ladner C	1.648	1.229
Munroe SiL	1.951	1.181
Whatcom L	1.580	1.211
Lynden SL	1.441	1.079

TABLE X111a

Increase (+) or decrease (-) in potassium fixed
Caused by Lime Application

Soil	Standard Method	Bray Method
MunroeSiCL	-.031	+.352
Ladner C	+.179	+.452
MunroeSiL	-.003	+.501
Whatcom L	-.157	+.265
Lynden SL	-.378	+.306

but also to the nature of the ions on the complex.

No definite conclusions are possible regarding the effect of lime on the potassium status of the soils studied. However, it is noted that the apparent potassium release due to lime, as indicated by the ammonium acetate extraction, is greatest in the two Upland soils and the potassium fixed due to lime using the sodium nitrate extractant is least in these soils. It appears that lime had less effect on the potassium status of the Upland soils than on the Lowland soils studied.

Greenhouse Experiment

A greenhouse experiment was designed to study the effect of lime on potassium fixation and to relate this effect, if any, to chemical tests. It was also hoped to establish the level of exchangeable potassium at which luxury consumption by plants occurs and to determine the validity of laboratory studies on fixation and release of potassium by correlation with plant growth.

The surface horizons of the five soils under study were used. A limed series was brought to a pH or 8.2 - 8.4 with calcium hydroxide as described above. Since time was limited only one crop could be grown. Thus, in order to conform to the objects of this experiment it was necessary to use a small volume of soil. Grand Rapids leaf

lettuce was the crop chosen for the experiment because it grows rapidly and the visual symptoms of a potassium deficiency are readily apparent.

Waxed cylindrical cardboard cartons 3" high and 4" across were used. In order to increase the volume for root development the bottoms were covered with 200 gms of acid leached quartz sand. Two hundred gms of $\frac{1}{4}$" mesh oven dry equivalent soil was placed over the sand. Three replicates of a limed and unlimed series of each soil were prepared in this manner.

Nutrients were added in solution in amounts suggested by Jenny (23). Potassium was not added to any of the soils. The nutrient solutions were made up of the following salts:

Major elements	gms/liter
NH_4NO_3	80.05
$NH_4H_2PO_4$	115.04
H_2SO_4 (45%)	1.66 ml
Minor elements	gms
H_3BO_3	2.83
$MnCl_2.4H_2O$	1.81
$Zn(C_2H_3O_2)'.3H_2O$	0.172
$CuSO_4.5H_2O$	0.08
$H_2MoO_4.H_2O$	0.09
Iron tartrate	5.00

Nitrogen and phosphoric acid were applied at a rate equivalent to 300 lbs per acre corresponding to Jenny's $N_3 P_3 K_0$ application. Jenny found that an $N_3 P_3 K_2$ application produced optimum lettuce yield upon a wide variety of soils. Minor elements were applied at the rate of $\frac{1}{2}$ mls solution per pot. In the unlimed series 0.04 gms calcium sulfate was applied in solution to each container. In all cases nutrient solution was applied before planting.

Prior to transplanting from flats, three random samples of 5 week oldlettuce consisting of 12 plants each were analyzed for potassium. Four lettuce plants from the flats were then transplanted into each carton. The plants were watered frequently with small amounts of water to prevent leaching of the nutrients. Petri dishes were placed below the cartons so that any leachate could berreturned to the soil.

In order to determine the level of potassium at which nutrient deficiency symptoms occur a series of acid leached quartz sand cultures were prepared. Four hundred grams of sand was added to each of thirty of the cardboard containers. Nutrient solution and calcium sulfate was added as above. Ten levels of potassium were prepresented with three replicates of each level. The amounts added as potassium chloride in solution were equivalent

to 0, .125, .250, .375, .500, .625, .750, .875, 1.00 and
1.50 milli-equivalents of potassium per 100 gms of soil.
These values were used in order to include the range of
exchangeable potassium of the soils studied. The lettuce
failed to grow in the sand, presumably due to the lack of
aeration because of its fine texture. This portion of
the experiment was therefore discontinued.

The plants were harvested on March 6 after 8 weeks
growth. At this time some deficiency symptoms, presumed
to be due to low potassium, were showing in some of the
containers. The green matter was weighed, dried for 48
hours at 70°C and weighed again for oven dry weight. The
dry matter was then moistened with concentrated sulfuric
acid and digested by the dry combustion method at 500°C
in a muffle furnace. The ash residue was taken up with
concentrated nitric acid and water and filtered. The
filtrate was made up to a volume of 250 mls. Aliquots
were taken, evaporated to dryness in a steam bath, and the
residue analyzed for potassium by the Wilcox gravimetric
procedure.

The soils were analyzed for potassium before and
after the experiment by both the standard ammonium acetate-
gravimetric and Bray methods. The amount of potassium
fixed or released during the experiment was calculated with
respect to both techniques.

Greenhouse Experiment

Pertinent information obtained from the greenhouse experiment is given in Table XlV.

When the dry weight yields of lettuce are compared with the percent potassium in the plants, it is evident that enough potassium was available in all soils so that it was not a limiting factor. For example, in the plants giving the lowest yield, the percent potassium present in the plant was higher than in plants showing higher yields. This confirmed the results of chemical tests and field fertility trials which indicated that these soils were high in available potassium. The decrease in yield due to liming was significant at $p = .05$ for the Munroe soils and the Lynden soils. The reason for this decrease was not evident, although it may have been that the lime caused a fixation of some of the minor elements, even though they were added to all soils. Some of the minor elements, when present in threshold quantities are apt to result in arrested growth. Lime caused a decrease in the percent potassium absorbed in all cases except on the Munroe silty clay loam.

TABLE XIV

Table Showing Oven Dry Yield of Lettuce and Potassium Absorbed

Sample	Average oven dry weights gms.		Average potassium absorbed by plants m.e.		Percent potassium in plants	
	Limed	Unlimed	Limed	Unlimed	Limed	Unlimed
Munroe SiCL	.1757	.4294	.134	.304	2.87	2.76
Ladner C	.3653	.3990	.183	.217	2.02	2.13
Munroe SiL	.2836	.4959	.158	.413	2.18	3.26
Whatcom L	.2761	.3122	.272	.352	3.86	4.40
Lynden SL	.3882	.5141	.358	.525	3.60	3.95

Potassium Fixed or Released by the Greenhouse Soils.

Studies were made on the fixation and release of potassium and the effect of lime on the potassium status of the greenhouse soils, to which no potassium had been added. The amount of original exchangeable potassium plus the amount absorbed by the plants minus the exchangeable potassium in the soil after growth was taken as the amount of potassium fixed. Negative values represent potassium supplied. The potassium fixed or released in the unlimed and limed series as determined by the Bray and standard methods is reported in Table XV.

TABLE XV

Potassium Fixed (+) or Released (-) in the Limed and Unlimed series as determined by the Standard and Bray Methods : Greenhouse Soils.

| | Standard Method | | Bray Method | |
Soil	Unlimed me/200gms	Limed me/200gms	Unlimed me/200gms	Limed me/200gms
Munroe SiCL	-.011	-.026	+.094	+.336
Ladner C	-.062	-.038	-.154	+.046
Munroe SL	-.018	-.026	-.178	+.326
Whatcom L	-.036	-.065	-.138	+.054
Lynden SL	-.022	-.080	-.194	-.104

It is noted that the ammonium acetate extractant indicated a release of potassium in all soils while the

sodium nitrate showed a release in four of the five

unlimed soils and a fixation in four of the five limed

soils. These facts support the explanation advanced to

explain the laboratory studies, that the replacing power

of Na^+ for potassium is lower when Ca^{++} dominates the

exchange complex.

The potassium released due to lime is highest in

the two Upland soils when the ammonium acetate extract-

ant is used, while the apparent fixation due to lime

was least in these soils when the sodium nitrate extract-

ant was employed. This corroborates the previous evidence

that lime has less effect on the potassium status of the

upland soils than on the recent alluvial soils.

SUMMARY AND CONCLUSIONS

1. Rapid methods for potassium estimation were tested for accuracy and precision using standard solutions. The Spurway (49) and Morgan (34) rapid tests were not found precise enough for this investigation. The Bray (7) method and a modification of the Peech and English (40) technique proved satisfactory and were further tested by comparison to the standard ammonium acetate-gravimetric method.

2. The exchangeable potassium of ten soil samples was extracted and determined by both the Bray and Peech procedures and showed a high significant correlation to the potassium extracted and determined by the ammonium acetate-gravimetric method. It was found that the Peech method was more suitable when cations in addition to potassium were to be studied. However, the Bray technique was more adaptable to special potassium studies.

3. Both rapid methods are adaptable to line chart estimation. Of the line chart methods tested a technique described by Bray (7) was considered the most suitable for routine soil testing work.

4. The soils studied, when leached free of exchangeable potassium, did not release detectable amounts of

fixed potassium when stored in a moist condition for
three months, or when alternately wetted and dried ten
times at 110 $^{\circ}$C.

5. A greenhouse experiment was conducted to study
the effect of lime on potassium fixation and to show if
any relationship existed between chemical studies and
plant growth. The potassium in all the soils was ample
for optimum lettuce growth, hence, the experiment
confirmed the results of field and chemical tests in
illustrating the high available potassium content of these
soils. Decreases in yield due to lime application in the
greenhouse were attributed to lime induced fixation of
some of the minor elements.

6. The magnitude of fixation of added potassium in
a limed and unlimed series of soils was studied in the
laboratory and the potassium fixed and released by the
limed and unlimed greenhouse soils was calculated. In the
laboratory considerable fixation of added potassium was
noted when determined by both the ammonium acetate and
sodium nitrate extractions. The fixation was greatest
when measured by the ammonium acetate extraction and those
soils in which fixation was highest by this method tended
to be lowest by the sodium nitrate technique. Both in the

laboratory and greenhouse soils the lime caused an apparent release of potassium when the ammonium acetate extractant was used, and a fixation of potassium by the sodium nitrate technique. The reason for this difference was thought to lie in the relative replacing powers of NH_4^+ and Na^+ when Ca^{++} or H^+ dominate the exchange complex.

7. Lime application did not influence the potassium status of the upland soils to such an extent as the lowland soils.

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(i)

APPENDIX

Soil and Area Descriptions
and Notes on Fertilizer Response.

1. Location - Farm of G. & H. Thomson, Chilliwack.

General description - This farm is allotted almost

entirely to dairying. Hay, pasture, corn and oats

are the main crops in rotation. The sampled area

is semi-depressional and imperfectly drained.

Relatively little fertilizer and no lime has been

added, whereas manure has been applied fairly

regularly. A rod row fertility test with oats was

conducted on the sample site in 1949 and indicated

that the soil was of high fertility for oats.

Nitrogen and phosphorous applications resulted

in a good increase in yield over the check plots

while potassium application was responsible for

only a small increase.

Soil description* - Munroe silty clay loam.

    0 - 9" - cultivated depth; moist; friable silt loam,
            fine nuciform to coarse nuciform structure;
            color dry - light grey-brown (6/2 10 yr)

*Profile descriptions based on N.S.S.C. recommendations

(35);colors based on Maunsell Color Chart.

    9 - 13" -massive, mottled silty clay loam, pale
            brown, color dry (6/3, 10 yr)

    13"   - massive, highly mottled light grey silty
            clay loam.

(ii)

2. Location - Farm of H. Lancaster, Matsqui.

General description - Dairying is the main enterprise
on this farm. Flax, turnips, potatoes, and canning
corn are cash crops grown in rotation with oats,
hay and pasture. The tested site is a lower portion
of a gently undulating area and is imperfectly
drained. Manure is added regularly to this soil.
It has received two commercial fertilizer appli-
cations in the past five years, a 4:10:10 and
calcium cyanamide. The rod row oat fertility
trial conducted in 1949 at this site indicated only
a moderate fertility for oats. Marked response to
nitrogen at all rates of application and response
to potassium at lower application rates only, were
noted. Phosphate fertilizer resulted in only small
increases in yield over the check plots.

Soil description - Ladner clay.

0 - 8" - cultivated depth; moist; firm clay, coarse
nuciform to medium nuciform aggregates;
color dry - light grey (7/2, 10 yr),
aggregates hard when dry.

8 - 15" - massive mottled clay; color dry - very
pale brown (7/3, 10 yr)

15" - - massive, highly mottled, saturated clay

(iii)

3. Location - Farm of C.E. Greer, Huntington.

   General description - As above, dairying is the main

   enterprise on this farm. Hay, pasture, clover,

   oats and corn are grown in the rotation. The

   sampled area is flat and moderately well drained.

   Apart from manurial applications no fertilizer or

   lime has been added to this soil. The oat fertil-

   ity trial in 1950 on this site indicated only

   moderate fertility with a marked response to

   nitrogen and phosphorous application. Potash

   response was evident at the lower application

   rates.

   Soil description - Munroe silt loam

   0 - 8" - cultivated depth: moist; friable silt

           loam, medium granular to small blocky

           aggregates, high organic matter; dry color -

           grey brown (5/2, 10 yr)

   8 - 15"- fairly massive friable silt clay loam;

           dry color - light grey (7/2 10 yr)

   15"   - massive silty clay loam, slightly mottled

4. Location - Farm of H.F. Smith, Abbotsford.

   General description - The land at this site has been

   cropped since 1945. Strawberries and daffodils

   are grown as cash sidelines; while oats, clover,

   hay, and pasture are the main crops grown to

(iv)

support a small dairy herd. The tested site is on a shallow knoll in a well drained position. Small amounts of manure have been added to the sampled site but no lime. or commercial fertilizer has been added. The oat fertility trial indicated a definite response to nitrogen and phosphorous application and little or no response to potassium.

Soil description - Whatcom silt loam

0 - 9" - cultivated depth; moist; very friable loam, granular to fine nuciform structure, light brown concretions common; dry color- yellowish brown (5/4, 10 yr)

9 - 20"- friable loam, medium granular structure, concretions; dry color - light yellowish brown (6/4, 10 yr)

20-30" - massive, fairly compact, very fine sandy loam, slightly mottled, yellowish brown (5/4, 10 yr)

30"   - massive compact sandy clay loam, light grey brown (6/2, 10 yr)

5. Location - Farm of F. Gaspar, Abbotsford.

General description - The land, which at this site is flat and well drained, was cleared in 1945 and has since been cropped to strawberries, broccoli,

(v)

oats and clover. The land was heavily fertil-
ized in the years when strawberries and broccoli
were grown. No lime has been added. The oat
fertility trials in 1949 indicate no residual
effect from past fertilization. A strong nit-
rogen and phosphoric acid response was evident.
Response to potassium application was high at
low nitrogen levels and at lower application
rates.

Soil description - Lynden sandy loam.

0 - 9" - cultivated depth; moist; very friable
silt loam with fine nuciform structure;
some pebbles; color dry - yellowish
brown (5/4, 10 yr)

9 - 18"- friable silt loam, fine to medium
nuciform structure, some pebbles;
color dry - yellowish brown (5/4, 10yr)

18" - stratified silt loam, loamy sand, and
fine gravels.