TB 1049 (1951) USDA TECHNICAL BULLETINS UPDATA
RELEASE OF NATIVE AND FIXED NONEXCHANGEABLE POTASSIUM OF SOILS
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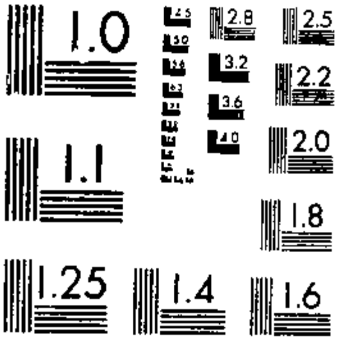

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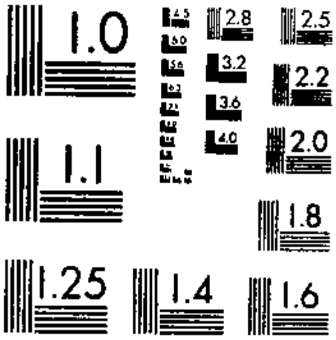

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NATIONAL BUREAU OF STANDARDS-1963-A

Technical Bulletin No. 1049 • October 1951

UNITED STATES
DEPARTMENT OF AGRICULTURE
WASHINGTON, D. C.

Release of Native and Fixed Nonexchangeable Potassium of Soils Containing Hydrous Mica[1]

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CONTENTS

Potassium is required in relatively large amounts by many cultivated plants. In the customary production of crops, potassium is supplied to the plants by inorganic fertilizer, organic materials, and the soil itself. Many soils contain extremely high quantities of potassium, of which only a minute fraction is generally available to plants at a given time. Soils extending over large areas have potassium contents of 1 to 2 percent, which is equivalent to 20,000 to 40,000 pounds of potassium per acre of surface soil, yet usually no more than several hundred pounds of this is currently available to plants. Less weathered soils of this same range of potassium content may contain several times as much available potassium.

Most of the soluble and exchangeable potassium occurring at any instant can be considered as available, but the degree and rate of availability of the nonexchangeable fraction vary enormously among soils. After prolonged periods of cropping without the replenishment of potash from without the soil, some soils will continue to deliver sufficient potassium to plants, while others will become incapable of this. It has

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[1] Submitted for publication May 18, 1951.

[2] Loraine W. Klipp and R. Q. Parks, of this Division, participated in various phases of the work. Soil samples were supplied by members of the agronomy departments of the State agricultural experiment stations of Alabama, Illinois, Maine, Ohio, and Pennsylvania.

been known that the quantities of exchangeable and soluble potassium are inadequate to supply a long succession of crops and that the available quantity must be replenished by release from the nonexchangeable part. Throughout this bulletin, the term "nonexchangeable potassium" refers to potassium that is not comparatively rapidly extracted by neutral normal ammonium acetate solution.

Knowledge of the potassium-supplying capacity of a soil, therefore, is important to the selection of cropping systems, fertilizer usage, and other management practices. It has been generally known that soil associations and regions differ markedly with regard to rate of potassium liberation. More precise understanding of the relative availability of potassium of different soils, especially under actual management practices, and of the source and mechanism of release within the soil is needed.

The organic matter of soils appears not to hold potassium in nonexchangeable forms, and the bulk of the potassium resides in primary and clay minerals. Potassium-bearing feldspars—such as orthoclase, microcline, and some plagioclases—and micas—such as muscovite and biotite—are generally abundant in mineral soils. In the more weathered soils, these minerals occur principally in the silt and finer sand fractions and usually constitute the major part of the total potassium of the soil. Of the three common groups of clay minerals—kaolinite, montmorillonite, and hydrous mica—the last-named is the important carrier of potassium. The possibility of appreciable release of potassium from the clay mineral fraction would therefore appear limited to soils of significant content of hydrous micas.

Surface soils of six experimental fields in five States in the eastern half of the United States, known to contain hydrous mica and having a history of low-potash applications similar to common regional practices, were available in 1944 for greenhouse and laboratory studies.

At one of these locations, Aroostook Farm, Presque Isle, Maine, soil samples were selected from nine plots that had received a wide range of potash additions in the form of fertilizer and organic materials. Many soils fix added potassium in a nonexchangeable condition, especially at higher levels of soluble and exchangeable potassium. The availability of fixed potassium relative to that of the native mineral content is important, both as to the understanding of the mechanism of release and fixation and as to the subsequent production of crops on soils exhibiting such fixation.

The 14 soil samples of this group have been intercompared with respect to their potassium-supplying capacities by a variety of plant, chemical, and mineralogical investigations. The purposes of these experiments included: (1) Comparison of soils, of different regions and origins, having substantial potash contents; (2) relation of mineralogical composition to potassium release; and (3) comparative availabilities of native and fixed potassium. The results are reported in detail in this bulletin.

REVIEW OF LITERATURE

A detailed review of the literature on soil potassium, including that on the release and fixation of nonexchangeable potassium, was prepared recently (*70*).[3] The present review is limited mainly to some work having an immediate bearing on the particular aspects of the experiments reported here.

Hopkins and Aumer (*46*) extracted an Illinois soil with boiling 23-percent HCl. Although their first attempts to start plants on the extracted soil were unsuccessful, alsike and red clover later grew well over a 5-year period. For the first 2 years the tops, and for all years the roots were returned to the soil. The average uptake of potassium during the last 3 years was 50 pounds per acre per year. The conclusion that the liberation of mineral potassium was caused by the action of organic matter decay was not supported by control treatments. Their generalization that all "normal" soils, containing 35,000 pounds or more of potassium per acre, can supply sufficient potassium for crops without the use of potash fertilizer has not been confirmed by subsequent experience.

Fraps (*32*) and Page and Williams (*61*) observed that plants could absorb substantially more potassium from soils than could be accounted for by the reduction in exchangeable potassium content. After Gedroiz (*34*) had replaced the exchangeable cations of a Chernozem by calcium alone, oats and other plants grew as well without added potassium as with it. His assumption, based on this result, that in general the nonexchangeable potassium of soils is adequate for plant growth, appears unjustified.

Martin (*57*) and Hoagland and Martin (*43*) found that during continuous cropping to barley and tomatoes over prolonged periods of time some soils release appreciable quantities of nonexchangeable potassium. Since then the prolonged-cropping technique has become a commonly used procedure for the comparative evaluation of the potassium-supplying-capacity of a soil. For example, alfalfa has been employed in this fashion by Bear, Prince, and Malcolm (*12*), Ladino clover by Chandler, Peech, and Chang (*22*) and by Evans and Attoe (*27*), German millet by Gholston and Hoover (*35*), and Sudan grass and panicum grass by Ayres (*7*). Studies such as these have established the existence of wide differences in the long-time potassium liberation from soils within the same region. Comparison of the results of various workers indicates broad regional soil differences with respect to this property. The possible occurrence of fixed potassium in soils of inadequately characterized history may have modified the liberation rate of the native sources. Seldom has any attempt been made to correlate the supply rate with mineralogical and chemical composition or genesis.

Drake and Scarseth (*26*) found that the response of 13 crop plants to potash applications on Crosby silt loam varied oppositely as their abilities to absorb native potassium. Timothy utilized some potassium unavailable to most of the crops, and carrots, buckwheat, Sudan grass, and wheat also absorbed potassium in excess of the exchange value. Evans and Attoe (*27*) found that Ladino clover extracts more nonexchangeable potassium from soils of high exchange level than oats but less from soils of low exchange level, because of the ability of oats to grow well at low levels of available potassium. Of a number of plant species grown with powdered microcline as the source of potassium, the clovers were the

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[3] Italic numbers in parentheses refer to Literature Cited, p. 42.

more efficient feeders, and it was concluded that, being slower growing plants, they can absorb potassium from a more dilute substrate (*85*). Lewis and Eisenmenger (*54*) observed that of 22 seed plants of differing degree of development grown on Merrimac sandy loam containing ground orthoclase, those of the earlier stages of development were the more efficient in the utilization of potassium from the feldspar.

The observed close agreement between exchangeable and Neubauer potassium values of some soils has led to the assumption that release of nonexchangeable potassium does not occur in the Neubauer procedure (*18*). However, Neubauer values substantially higher than the corresponding exchange values have been obtained for a number of soils (*49, 71, 73, 83*). By three to five successive Neubauer croppings of the same soil sample, Schachtschabel (*76*) and Wiessmann and Lehmann (*90*) observed that some soils release extremely large quantities of potassium. In no case was the exchangeable fraction eliminated by the cropping.

Bray and DeTurk (*19*) concluded that soils could release potassium to the exchangeable form comparatively rapidly when its magnitude was below the equilibrium level, even in the absence of plants. Thus, the moist storage of field samples at equilibrium resulted in no change, while that of samples leached with HCl effected a return to the initial equilibrium level. Ayres (*7*) found that partial removal of exchangeable cations was followed by the release of small amounts of potassium during moist storage and that complete removal resulted in a greater release. Calcium-saturated soil liberated about 2.5 times as much as the hydrogen-saturated soil. The average release during cropping to grass, however, exceeded that during moist storage of hydrogen-saturated soil by about sevenfold.

Peech and Merwin[4] by a particular sequence of leachings with acetates of ammonium and other cations, without any standing between treatments, obtained substantial release of potassium from Honeoye and Dunkirk soils; calcium, magnesium, and sodium acetates, especially, were effective when used between two ammonium acetate leachings. During repeated leachings of four soils with single acetate solutions, sodium released the most potassium; ammonium tended to block release, and thus afforded a sharp distinction between exchangeable and nonexchangeable fractions. This behavior is consistent with the tendency of ammonium to be fixed similarly to potassium (*15, 80*). It was reported by Fine,[5] however, that during moist storage ammonium-saturated soils released about five times as much potassium as hydrogen-saturated soils.

DeTurk, Wood, and Bray (*25*) reported that the exchangeable potassium level of soil of 2 experimental fields was low in October after harvest, but was restored to its equilibrium value by the following May. In a similar study involving 11 field sites, Rouse and Bertramson (*75*) found that at 3 of the locations the exchangeable potassium value was appreciably higher in April than in the preceding September. It has not been established if at least a part of this effect is caused by such

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[4] MERWIN, H. D. THE RELEASE OF NON-EXCHANGEABLE POTASSIUM INTO EXCHANGEABLE FORM IN FOUR NEW YORK SOILS. 1950. [Ph.D. Thesis, Cornell Univ., Ithaca, N. Y.]

[5] FINE, L. O. POTASSIUM FIXATION AND AVAILABILITY. I. INFLUENCE OF FREEZING AND THAWING. II. AVAILABILITY OF FIXED POTASSIUM TO PLANTS. 48 pp., illus., 1941. [Ph.D. Thesis, Wis. Univ., Madison.]

processes as freezing and the leaching of potassium from crop residues. Fine, Bailey, and Truog (*29*) observed that freezing and thawing cycles tended to increase exchangeable potassium in soils of low to moderate fertility and to decrease it in soils of high potassium fertility.

Release of potassium from soils of relatively low exchange level by drying at above-ordinary temperatures has been reported by Bray and DeTurk (*19*), Campanile (*20*), Rouse and Bertramson (*75*), and Walsh and Cullinan (*88*). Possible evidence of a similar release upon air-drying at room temperatures is implicit in some results of Abel and Magistad (*2*), Ayres (*6*), and Seatz and Winters (*77*). Attoe (*3, 4*) definitely established that air-drying of moist, cropped soils can increase the exchangeable potassium content and that the exchange value varies inversely with the relative humidity of the air in equilibrium with the dried soil. The potassium thus released was found to be available to oats; this agrees with the finding of Walsh and Cullinan (*88*) that potassium liberated by drying at 45° C. was available to mustard plants.

Attoe (*4*) suggested a procedure involving alternate drying and wetting and extraction with a salt solution as a possible method for the determination of the potassium-supplying-power of a soil. Subsequently, Evans and Simon (*28*) proposed the use of alternate extractions with 0.5 N HCl and dryings at 80° C. Lee [6] found that soil samples from Illinois experiment fields contained about 200 pounds of exchangeable potassium per acre when they had been thoroughly air-dried in the usual manner, but indicated less than 100, and in two cases less than 10, when the sample was extracted at its field moisture content of 10 to 20 percent. The exchange level did not increase until the moisture was reduced to several percent above that of the air-dry state. Subsequent heating at 105° C. had no further effect on the exchangeable potassium. Release by air-drying has been obtained also by Ayres (*7*) and Larson and Allaway (*52*).

The extraction of potassium not replaceable by the usual salts of cation exchange methods has been effected by strong mineral acids under varying conditions of concentration, temperature, and time. Fraps (*31*), Ayres (*7*), and Attoe and Truog (*5*) used HCl; with Fraps employing concentrated acid at 100° C., Ayres 1 N acid at 95° C., and Attoe and Truog 0.5 N acid with 2 hours of shaking. Boiling 1 N HNO_3 for 10 minutes has been used by Wood and DeTurk (*93*) and Rouse and Bertramson (*75*). Moderate degrees of correlation have been obtained between the values of release by some of these procedures and values by other methods, such as cropping and moist storage.

The earlier studies of electrodialysis of soils indicated a general agreement between exchangeable and dialyzable potassium, but the dialysis periods were comparatively short. Some results such as those of Gilligan (*37*) suggested a higher extraction by dialysis for a 1-day period than by ammonium acetate. Ayres (*7*) and Ayres, Takahashi, and Kanehiro (*8*) increased the duration of electrodialysis considerably—up to 30 days. A quantity of potassium equivalent to the exchangeable fraction was usually liberated the first day, and all soils then yielded nonexchangeable potassium at varying rates that often became constant for a given soil. Ayres found the total 30-day release is re-

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[6] LEE, C. K. A STUDY OF EXTRACTION AND DETERMINATION OF THE AVAILABLE NUTRIENTS IN SOILS. 72 pp., illus., 1948. [Ph.D. Thesis, Ill. Univ., Urbana.]

lated to release by hydrogen-saturated soils during moist storage. Both prolonged electrodialysis and vigorous acid extraction effect extensive soil decomposition.

The solubilities and availabilities of soil-forming potassium-bearing primary minerals have been extensively studied, especially those of feldspars and micas. The potash of all such minerals is available to some extent, but the degree of availability depends on the type of mineral, particle size, and stage of weathering. Most comparisons between potassium feldspars and potassium micas on the basis of equal weight or equal potassium content have indicated a greater effectiveness for the micas (*69*). Blanck (*13, 14*), Plummer (*68*), and Fraps (*30*) found the following order of increasing availability to plants: Microcline, orthoclase, muscovite, biotite. However, the potassium feldspars generally are much more abundant in existing surface soils than the micas.

According to McCaughey and Fry (*55*), microcline in soils shows no chemical alteration, while orthoclase may be of either fresh or extremely altered appearance in young soils. The rate of weathering of plagioclase feldspars, which usually contain small amounts of potassium, varies according to the calcium and sodium content; the high sodium plagioclases, such as albite and oligoclase, occur generally as fresh grains, but the high-calcium species, such as anorthite, may be intensely weathered and difficult to identify. Denison, Fry, and Gile (*24*) established that micas of the Piedmont province soils had undergone severe weathering. Weathered muscovite had increased in water content to about the same extent as it had lost K_2O. Biotite tended to be altered in composition more than did muscovite and to be weathered to a K_2O content of about 4 percent. Barshad (*10*) and Walker (*87*) regard the hydration of micas during weathering as principally a result of replacement of potassium by calcium, magnesium, and hydrogen ions.

Graham (*38*) measured the release of potassium from silt-size fractions of seven soils to soybeans during contact with hydrogen-clay. Only one silt released potassium, two did not, and in the remaining four potassium moved instead from the seeds or plants to the surface of the clay. Olsen and Shaw (*60*) determined the Neubauer values of the 2–10-, 10–20-, and 20–50-micron silt fractions of three soils that had been freed of exchangeable potassium. The seedlings absorbed appreciable amounts of potassium from all fractions, but the extent of release increased with decreasing particle size. Merwin [7] found that by storage while calcium- or hydrogen-saturated, the sand, silt, and clay fractions of four soils contributed 0 to 18, 15 to 51, and 40 to 83 percent, respectively, of the total potassium released. He observed no relationship between the amount of release by sand and silt fractions and their contents of potassium-rich minerals.

Although most workers have studied the contribution of silt and sand minerals to the delivery of potassium, a few have stressed the importance of the clay minerals in the finer fractions (*17, 93*). It was observed by Bray (*17*) that with increasing age of Illinois soils the potassium content of the clay fractions tended to become lower. He postulated a weathering mechanism of 0.1 to 1 micron mica particles, whereby the ends lose potassium, hydrate, and split off to form superfine sheets of

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[7] See footnote 4.

beidellite. Wood and DeTurk (*94*) proposed that in these older soils the potassium of illite particles had been replaced to a greater distance within the particle. The existence of a continuous series of hydrous mica intermediates between the illite and montmorillonite weathering stages has been suggested (*48*). Rouse and Bertramson(*75*) measured the potassium-supplying power of a number of soils by HNO_3 digestion and made X-ray diffraction spectrograms of various clay and silt-size fractions; for the two finest fractions, < 0.2 and 0.2 to 1.0 micron, significant correlations existed between the area under the illite peak of the spectrogram and the HNO_3 release value. Peech and Bradfield (*65*) concluded that the nonexchangeable potassium of severely electrodialyzed Miami B-horizon clay containing 2.36 percent of potassium was less soluble than that of biotite or orthoclase. Fine[8] found the potassium of the < 0.2-micron fraction of Miami A-horizon clay to be unavailable to corn and that of illite of particle size 0.2 to 2.0 microns only slightly available.

The identification and estimation of hydrous micas in soils and their behavior and importance in the potassium economy remain unsatisfactory. Among the reasons for this situation are the following: Diffuse X-ray diffraction spectra; differences in basal spacing (*47*); variations in potassium and water contents (*39, 40*); dissimilarity of hydrous micas of soils and geological deposits; nonuniform distribution within the clay-size fraction (*96*); and possible general occurrence in particles of mixed-layer clay minerals (*42*).

It has been definitely established that potassium is fixed in difficult-to-exchange forms by montmorillonite (*53, 62, 79, 84, 89*), hydrous micas (*79, 89, 91*), and vermiculite (*9*), but not by kaolinite (*44, 84, 86*). Drying appears necessary for the fixation to occur in montmorillonite (*79, 89*) but not in hydrous mica (*79, 91*). As members of these two groups of clay minerals often occur together in soils and as the moisture conditions of soils in the field are extremely variable, it is difficult to predict the behavior of field soil with regard to the fixation of added potash. A necessary condition for fixation is the coexistence of a moderate or high level of readily exchangeable potassium. The various forms of soil potassium comprise an equilibrium system, so that potassium tends to be fixed when the exchange level is above the equilibrium value and to be released when it is below this value (*25*).

Fixation in the expanding 2:1 lattice type of clay minerals is currently regarded as the entrapment of potassium ions between some sheets of particles that do not readily reexpand, because of the presence of critical-size potassium ions and a lack of hydration (*89*). In the nonexpanding or slightly expanding hydrous mica lattices, fixation is probably a restoration of interlayer potassium that had been previously removed by weathering processes or similar leaching stress (*25*).

Chaminade (*21*) considered fixed potassium as virtually unavailable to plants. On the basis of growth of tomato plants with artificially fixed potassium of bentonite as the only source of potassium, Kolodny and Robbins (*51*) concluded that fixed potassium was only very slightly available. However, Fine[8] critized this conclusion because the test plant was a poor feeder, magnesium was not supplied, and the availa-

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[8] See footnote 5.

bility was compared to that of exchangeable potassium supplied in eight times the quantity of fixed potassium. Fine grew tobacco, corn, and wheat with fixed potassium of bentonite as the sole source of potassium; during periods of growth of 7 to 10 weeks, about 25 percent of the potassium became available to the tobacco, and virtually all of it to corn and wheat, which had more thoroughly developed root systems.

DeTurk, Wood, and Bray (*25*) considered that fixation of potassium is not wholly wasteful, as it retards loss by leaching, and some of the fixed potassium is available when the exchange level has become reduced again. According to Walsh and Cullinan (*88*), fixation in soils by wetting and drying cycles is not permanent, as evidenced by the subsequent liberation of fixed potassium after the first mustard crop had been severely deficient. Evans and Attoe (*27*) found that oat plants absorbed appreciable fractions of potassium fixed in soils. York[9] observed the liming of Mardin silt loam to fix a part of potash additions, but the fixed potassium was available during a 6-month period. Attoe (*3*) distinguished between two types of fixation: (a) That in moist soil enhanced by liming and the fixed potassium fairly soluble in 0.5 N HCl; and (b) that by drying independent of pH and the fixed potassium resistant to 0.5 N HCl extraction.

Values for the release of nonexchangeable potassium, from the 14 soil samples under consideration here, by various methods—such as Ladino clover cropping, moist storage, freezing and thawing, electrodialysis, digestion with boiling 1 N HNO_3, and a Neubauer procedure—have already been presented (*72*). Significant bivariate correlations of release values of moderate precision were found among the clover cropping, dialysis, acid, and Neubauer procedures, the most satisfactory agreement being that between the clover and dialysis values.

DESCRIPTION OF SOILS

Wooster silt loam, accession No. E1649, was from plot 14 of experiment 21 on the Frye Farm, Wooster, Ohio. Since 1945 it had received a $N-P_2O_5-K_2O$ application of 20–120–20 pounds per acre in each corn-oats-wheat-clover rotation (*1*). Comparisons of 40–80–40 and 40–80–80 treatments for the 1915–28 period and of 40–120–40 and 40–120–80 treatments for 1929–37 indicate slightly higher yields for the higher potash application, but the differences can be considered significant only for corn. This soil series is reported to be in the medium class of Ohio soils with respect to response to potassium and potash-supplying power (*60*). It was derived from glacial till, and is classified within the Gray-Brown Podzolic great soil group (*78*).

Hagerstown silt loam, E1670, was from plot 23 of the supplementary fertility series at State College, Pa. Since 1922 it had received 6 tons of manure and 24 pounds of P_2O_5 per acre applied to corn and wheat in a corn-oats-wheat-mixed hay rotation (*59*). In the supplementary series, comparisons of 20–72–25, 20–72–50, and 20–72–75 treatments for the period 1922–41 indicate moderate increases in yield for successive

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[9] YORK, E. T. CALCIUM-POTASSIUM INTERRELATIONS IN SOILS AND THEIR INFLUENCE UPON THE YIELD AND CONTENT OF CERTAIN CROPS. 1949. [Ph.D. Thesis, Cornell Univ., Ithaca, N. Y.]

increases in potash additions for all of the rotation crops (*59*). For the nearby Jordan plots on the same soil type, four possible comparisons of treatments, namely 0-0-0 and 0-0-100, 24-0-0 and 24-0-50, 0-48-0 and 0-48-100, 24-48-0 and 24-48-100, for the two periods 1882-1921 and 1922-30 demonstrate appreciable response to potash in every case (*58*). The soil was developed from limestone and is classified as Gray-Brown Podzolic (*78*).

Decatur clay loam, E1563, was taken from section 34 of the Tennessee Valley Substation, Belle Mina, Ala. This section was not in plots but had been cultivated in general farming for 15 years with only one potash application. Experiments on this same soil at the substation indicate that it responds to potash.[10] From 1933 to 1937, of four comparisons of 0 and 400 pounds of KCl, alfalfa showed an appreciable response in three cases. In a cotton-legume-corn 2-year rotation, 1930-38, slight responses to 75 pounds of KCl were indicated generally. With continuous cotton, one series comparing 0, 12, 24, 48, and 96 pounds of K_2O indicated no consistent trend, while another series of 6, 12, 24, and 48 pounds produced a possibly significant slight response. In the growing of cotton only small amounts of potassium are removed from the area. The alfalfa experiments do indicate an appreciable response of this crop to potash, presumably because of the greater removal of potassium. This is a Red Podzolic soil derived from limestone (*78*).

Sable silty clay loam, E1648, from a plot of treatment 7 of the Aledo, Ill., experiment field had received crop residues since 1910 in a rotation involving corn, oats, wheat, and sweetclover (*11*). Comparison of treatment 8, residues-limestone-rock phosphate, with treatment 9, which included these plus 50 pounds K_2O per year, indicated the following response to potash: 1910-42, corn, oats, and wheat showed slight increases; 1939-42, only corn and wheat exhibited slight response. This is a Humic Gley soil that represents stage 1 in the development series of five stages for Illinois soils formed from Peorian loess of varying thickness on Illinoian till, and is reported to be not deficient in available potash (*25*, *93*).

Herrick silt loam, E1646, from a plot of treatment 4 of the Carlinville, Ill., experiment field received rock phosphate from 1910 to 1934 and manure since 1910 in rotations involving corn, oats, wheat, and legume hay (*11*). Comparison of treatments 8 and 9, which were the same as for the Sable soil, indicated response to potash as follows: 1910-42, moderate response for corn and smaller increases for oats, wheat, and hay; 1939-42, this response continued except for oats. This Planosol represents stage 3 of the Peorian loess development series, and it is said to be deficient in potash for the production of high yields (*25*, *93*).

Caribou loam from soil-fertility plots on Aroostook Farm, Presque Isle, Maine, was represented by samples from nine plots receiving differential potash and organic matter applications since 1927 (*23*). The treatments are indicated in table 1.

The 3-year rotation consisted of potatoes, oats, and red clover; the 2-year rotation of potatoes and crimson clover; and the 1-year culture was continuous potatoes. All fertilizer was applied to potatoes. The

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[10] ANONYMOUS. RESULTS TO DATE OF EXPERIMENTS ON SUBSTATIONS AND FIELDS. Ala. Agr. Expt. Sta. Rpt. on Outlying Field Stations, 31 pp., illus. 1938. [Processed.]

TABLE 1.—*Field treatments of Caribou loam samples*

Accession No.	Plot No.	Rotation period	Fertilizer analysis[1]	Other treatment[2]
		Years		
E1548	282	3	4-8-0	Cover crop plowed under.
E1550	281	3	4-8-8	Do.
E1552	274	3	4-8-12	Do.
E1663	363	2	4-8-8	Cover crop removed.
E1664	364	2	4-8-8	Cover crop plowed under.
E1665	365	2	4-8-8	Two cover crops plowed under.
E1666	366	2	4-8-8	Cover crop plus 6 tons straw plowed under.
E1667	367	2	4-8-8	Cover crop plus 20 tons manure plowed under.
E1558	390	1	4-8-8	

[1] One ton per acre in each rotation.
[2] Once in each rotation.

fertilizer rate and analyses listed are those begun in 1939; somewhat different applications were used previously. Before 1935 the green manure crop of the 2-year rotations was a mixture of oats, peas, and vetch. The green manure crop of plot 363 was transferred to plot 365 where it was plowed under with the crop grown there. During the 1927–29 period, 60, 105, and 150 pounds of K_2O provided large successive increases in yield of potatoes but not of oats. From 1930 to 1941, 80, 140, 200, and 280 pounds effected successive increases in potato yields, especially the 80-pound rate, which produced a considerable increase in clover also. The native potash of this soil cannot be depended on for the major portion of the high potash requirements for a large crop of potatoes. Caribou soils are classed as Podzols and were formed from glacial till derived from calcareous shale (*78*).

COMPOSITION OF SOILS

Physical

In table 2 are listed the mechanical analyses for the 14 soil samples,

TABLE 2.—*Particle-size distribution, nitrogen and organic matter contents, and pH values of soils*

Soil series	Accession No.	Mechanical analysis: Sand (0.05–2 mm.)	Mechanical analysis: Silt (2μ–50μ)	Mechanical analysis: Clay (< 2μ)	N	Organic matter	pH value[1]
		Percent	*Percent*	*Percent*	*Percent*	*Percent*	
Wooster	E1649	10.5	74.9	14.6	0.10	1.6	6.4
Hagerstown	E1670	15.0	62.1	22.9	.15	2.9	5.9
Decatur	E1503	22.9	36.2	40.9	.11	[2] 2.0	7.1
Sable	E1648	3.3	70.8	25.9	.21	4.5	5.8
Herrick	E1646	5.2	76.9	17.9	.18	3.1	6.4
Caribou	E1548	30.6	51.2	18.2	.22	4.7	5.3
Do	E1550	32.4	50.0	17.6	.20	4.3	5.2
Do	E1552	30.8	51.2	18.0	.22	4.5	5.2
Do	E1663	33.8	49.1	17.1	.20	4.4	5.2
Do	E1664	35.2	48.6	16.2	.21	4.3	5.1
Do	E1665	32.6	49.4	17.7	.22	5.0	5.1
Do	E1666	28.9	52.7	18.4	.25	6.0	5.1
Do	E1667	30.5	52.1	17.4	.31	6.3	5.0
Do	E1558	33.6	48.7	17.7	.19	3.7	5.0

[1] At a soil-water ratio of 1:2.
[2] Determined by dry-combustion method.

as determined by the pipette method (*50*), organic matter contents as estimated from the loss of weight due to H_2O_2 treatment in the mechanical-analysis procedure, nitrogen contents by a Kjeldahl procedure, and pH values.

The nine Caribou loam samples are uniform in particle-size distribution. The Wooster, Hagerstown, Sable, and Herrick samples have comparatively high silt contents. The highest clay content by far is that of Decatur clay loam. Both the nitrogen and the organic matter contents of the two Caribou samples receiving the highest organic matter additions in the field, E1666 and E1667, are higher than those of the remaining Caribou samples and also of the other soils.

Chemical

The total chemical composition of oven-dry (< 2 mm.) samples of the six soil series, as determined according to Robinson (*74*), is shown in table 3.

Also included is the composition of the silt (2 to 50 microns) and the clay (< 2 microns) fractions of the same soil samples. All of the soils except Decatur contain substantial quantities of potassium, and this is also true of the silt and clay fractions. The potassium contents of the other Caribou samples, not shown, were slightly higher than for the E1548 sample, probably because of exchangeable and fixed residues of potash applications. The occurrence of kaolinite in the clay fraction of the Decatur soil is evidenced by the low silica and high alumina contents of that fraction.

The silt fractions of the same soil samples were subdivided into three size-fractions, 2–10, 10–20, and 20–50 microns. The potash contents of these subfractions, together with those of the silt and clay fractions, are listed in table 4.

The general tendency is for the potash content to increase with decreasing particle size. This trend is relatively gradual except for the Caribou, in which the clay is markedly higher than any of the silt subfractions. Only in the Hagerstown is the clay lower than the silt, which is a reversal of the trend within the silt fraction. The agreement between determined and calculated silt values is satisfactory.

The silt fraction can be subdivided not only by the particle size, but by differences in specific gravity between the various minerals. Silt fractions of samples of Wooster, Hagerstown, Decatur, Sable, and Caribou soils were divided by use of liquids of differing specific gravity into three specific-gravity groups, > 2.70, 2.60–2.70, and < 2.60. These were analyzed for potassium content, and the results are presented in table 5.

The middle, or quartz, fraction has the lowest potash content. Except for the Caribou sample, the light fraction has a higher potash content than the heavy fraction. In the unweathered state, the potash micas have specific gravities > 2.70, but weathering tends to reduce the gravity, so that both the middle and light fractions may contain micas. Also, because of their platy shape, micas tend to settle in liquids as would material with lower actual specific gravity. With due consideration for these effects, it would appear that the feldspar minerals of the light fraction have the highest potash content of the silt minerals.

TABLE 3.—*Chemical composition of soils and of their silt and clay fractions*

WHOLE SOIL

Soil series	Accession No.	K_2O	CaO	MgO	Na_2O	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	MnO	P_2O_5	Ignition loss	Total
		Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
Wooster	E1649	1.87	0.47	0.16	0.91	78.90	3.30	8.63	0.87	0.10	0.15	3.93	[1] 99.48
Hagerstown	E1670	2.27	.16	.11	.17	75.38	3.52	11.13	1.02	.29	.10	5.56	[1] 99.79
Decatur	E1563	.51	.44	.03	.09	69.40	5.28	15.75	1.51	.43	.17	6.86	[1] 100.51
Sable	E1648	1.94	.98	.17	1.14	75.04	3.12	10.11	.60	.06	.15	6.91	[1] 100.39
Herrick	E1646	1.81	1.79	.16	1.19	77.80	2.23	8.99	.89	.04	.19	5.01	[1] 100.16
Caribou	E1548	1.38	.40	.39	1.71	69.40	4.67	12.57	1.05	.07	.25	7.86	[1] 99.88
SILT													
Wooster	E1649	1.82	0.33	0.32	1.04	85.19	1.87	7.72	0.21	0.04	0.06	1.10	99.70
Hagerstown	E1670	2.77	.12	.25	.41	84.20	2.30	7.27	.51	.04	.06	1.56	99.49
Decatur	E1563	.55	.31	.31	.23	75.22	4.24	12.42	.70	.27	.12	5.39	99.76
Sable	E1648	1.97	.65	.32	1.44	84.95	1.08	7.89	.37	.02	.04	.91	99.64
Herrick	E1646	1.89	.83	.28	1.48	85.41	.96	7.45	.42	.01	.10	.74	[1] 99.59
Caribou	E1548	1.28	.27	1.02	2.00	77.70	3.44	11.01	.53	.03	.08	2.25	99.61
CLAY													
Wooster	E1649	2.43	0.31	0.92	0.40	51.69	9.78	23.84	0.75	0.40	0.49	8.79	[1] 99.82
Hagerstown	E1670	1.56	.50	1.56	.57	50.48	8.79	25.52	1.05	.32	.49	10.10	100.94
Decatur	E1563	.76	.46	.67	.12	39.89	10.38	32.54	1.25	.47	.31	13.40	[1] 100.27
Sable	E1648	2.31	1.10	1.58	.32	56.87	8.12	20.04	.79	.12	.45	8.04	[1] 99.76
Herrick	E1646	2.11	1.19	1.76	.37	55.05	9.00	20.84	1.04	.11	.37	8.36	100.20
Caribou	E1548	2.78	.44	2.47	.57	45.22	12.99	23.56	1.27	.11	1.30	9.51	100.22

[1] Includes small content of SO_3.

TABLE 4.—*Distribution of potash in silt and clay fractions*

Separate	Fraction size	K_2O in size-fraction of—					
		Wooster (E1649)	Hagerstown (E1670)	Decatur (E1503)	Sable (E1648)	Herrick (E1646)	Caribou (E1548)
	Microns	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
Clay	2	2.43	1.56	0.76	2.31	2.11	2.78
Silt	2–10	1.98	3.08	.69	2.28	2.17	1.17
	10–20	1.76	2.84	.54	1.99	1.90	1.01
	20–50	1.68	2.54	.38	1.81	1.78	.95
Silt	2–50	1.82	2.77	.55	1.07	1.80	1.28
	[1] 2–50	1.76	2.78	.48	1.04	1.80	1.03

[1] Calculated from the values for the 3 silt fractions.

Cation-exchange capacities and exchangeable-cation contents of the 14 soil samples are listed in table 6.

Exchangeable cations were replaced by leaching 20 gm. of soil in a special glass leaching tube with two 100-ml. portions of neutral N ammonium acetate, the soil remaining ammonium-saturated overnight after percolation of the first portion. Calcium, magnesium, potassium, manganese, and cation-exchange capacity were determined by methods outlined by Peech (*63*). Sodium was determined gravimetrically as

TABLE 5.—*Potash contents of specific-gravity fractions of silts*

Soil series	Accession No.	K_2O content of material of specific gravity				
		Fractions of silt			Whole silt	
		>2.70	2.60–2.70	<2.60	Determined	Calculated [1]
		Percent	*Percent*	*Percent*	*Percent*	*Percent*
Wooster	E1649	1.55	1.14	4.24	1.82	1.09
Hagerstown	E1670	2.64	2.37	4.35	2.77	2.80
Decatur	E1503	.05	.05	.78	.55	.52
Sable	E1648	1.17	.98	2.38	1.07	2.02
Caribou	E1548	1.67	.94	1.28	1.28	1.25

[1] Calculated from the values for the 3 specific-gravity fractions.

TABLE 6.—*Cation-exchange capacity and exchangeable-cation composition of soil samples, in milliequivalents (m.e.) per 100 grams*

Soil series	Accession No.	Cation-exchange capacity	Ca	Mg	K	Na	Mn	H
Wooster	E1649	9.6	6.7	1.25	0.21	0.15	0.026	1.5
Hagerstown	E1670	11.0	6.0	.01	.32	.02	.048	3.9
Decatur	E1503	11.7	11.1	1.04	.28	.10	.000	.0
Sable	E1648	24.7	15.9	5.48	.53	.02	.020	4.0
Herrick	E1646	17.2	13.7	2.50	.24	.10	.039	1.0
Caribou	E1550	14.2	4.4	.60	.18	.08	.029	8.4
Do.	E1548	14.3	5.2	.80	.09	.10	.036	8.6
Do.	E1552	14.5	4.9	.60	.23	.08	.043	8.1
Do.	E1663	13.0	5.2	.04	.22	.31	.041	7.3
Do.	E1664	15.2	5.4	.67	.26	.34	.050	8.4
Do.	E1665	14.5	5.1	.02	.33	.37	.062	8.1
Do.	E1666	10.2	5.9	.68	.03	.30	.003	8.8
Do.	E1667	17.7	7.3	.04	1.17	.40	.071	9.2
Do.	E1558	14.0	4.6	.79	.60	.14	.061	8.8

sodium uranyl magnesium acetate (*67*), and hydrogen by titration of the ammonium acetate extract to pH 7.

The Sable soil has the highest cation-exchange capacity; within the Caribou group the capacity tended to increase with the field application of organic matter. The exchangeable potassium of the Caribou samples exhibited a wide range, 0.09 to 1.17 m.e., as a result of differential field treatments. The straw and manure-plot samples, E1666 and E1667, contained more exchangeable potassium than the continuous-potatoes sample, E1558. For the soils from the other States the range of values was very narrow except for the Sable soil. The trend of accumulation of potassium within the Caribou group was accompanied by a similar trend in exchangeable sodium.

Mineral

The primary mineral composition of the silt fractions of samples of the six soil types was determined by microscopic estimation of the abundance and size of particles of the various minerals (*33, 55*). The percentage estimates of quartz, potash micas, potash feldspars, and plagioclase feldspars are presented in table 7.

TABLE 7.—*Partial mineral composition of silt fractions (2 to 50 microns)*

Soil series	Accession No.	Micas		Potash feldspars		Plagioclase feldspars	Quartz
		Biotite	Muscovite	Microcline	Orthoclase		
		Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
Wooster	E1649	0	1	1	9	10	52
Hagerstown	E1670	0	5	5	20	10	48
Decatur	E1563[1]	1	5	0	0	14	27
Sable	E1648	0	1	5	16	11	55
Herrick	E1646	0	1	3	7	17	45
Caribou	E1548	5	20	1	5	10	20

[1] Appreciable content of siderite and iron oxides.

The abundance of minerals, other than quartz, containing little or no potassium is not indicated. As the plagioclases usually have slight or negligible potassium contents, the appreciable amounts of these minerals shown for all the soils do not necessarily mean a substantial supply of potassium from this source. All soils except the Decatur contain potash feldspars, in a range from low to moderate, the values for both the Hagerstown and Sable being more than 20 percent. Orthoclase exceeds microcline in abundance. Caribou is the only soil possessing a high content of mica, predominantly muscovite. The Wooster, Sable, and Herrick soils are virtually free of micas, while the Hagerstown and Decatur have small contents of muscovite. It is difficult to estimate the abundance of minerals in such mixtures. In addition, mineral particles occur in various stages of weathering, which involve losses of potassium without readily discernible concomitant alterations in crystal properties used for identification.

The clay mineral composition of the < 2-micron clay fractions of the same six soil samples was established by a combination of methods. Kaolinite was determined by a differential thermal analysis procedure (*41*). Montmorillonite was estimated from X-ray diffraction spectrograms of clay that had been solvated with ethylene glycol (*16, 56*) and from the cation-exchange capacity of the clay. The presence of hydrous mica was indicated by the X-ray spectrogram and the content estimated from the potassium content and the exchange capacity. By disregarding the quartz contents the clay mineral portion of the clay fraction was divided into its component minerals (table 8).

The contents of free iron oxides assigned to the Decatur and Hagerstown clays were previously determined in other samples of these soil series. The estimates for hydrous mica, except for the Decatur soil, range from 60 to 90 percent. One of the bases for selection of these six soils for the present investigation was the probable occurrence of substantial amounts of hydrous mica. Only Hagerstown and Decatur clays have appreciable contents of kaolinite, and the high value for Decatur agrees with its low silica to alumina ratio. The two Illinois soils, Herrick and Sable, have the highest montmorillonite contents, and in these soils the beidellite variety probably predominates.

TABLE 8.—*Clay mineral composition of clay fractions* (< *2 microns*) [1, 2]

Soil series	Accession No.	Kaolinite	Hydrous mica	Montmorillonite	Free iron oxides
		Percent	*Percent*	*Percent*	*Percent*
Wooster	E1049	< 10	90	10	0
Hagerstown	E1670	25	70	0	5
Decatur	E1503	50	[3] 40	0	10
Sable	E1648	< 10	60	30	0
Herrick	E1840	< 10	60	30	0
Caribou	E1548	0	80	20	0

[1] Exclusive of quartz content.
[2] Determined by S. B. Hendricks and R. A. Nelson.
[3] A subsequent X-ray spectrogram indicates this value to be too high.

There is a general correlation between the hydrous mica and total potassium contents of the clays. However, the minerals of this group evidently can possess structures and potassium contents lying between those of illite and montmorillonite (*48*). It is to be expected that a reduction in potassium content within this hydrous mica-intermediate weathering sequence would be accompanied by a lesser potassium availability. The indicated clay mineral pattern for the Sable and Herrick soils is similar, but the Sable soil has a slightly higher potassium content and represents a younger stage of development.

RELEASE METHODS AND RESULTS

Greenhouse

Ladino clover was grown on 12 of the soil samples in small ceramic pots with inside dimensions of 5 inches in diameter and 3.5 inches in depth, in two concurrent experiments, Nos. 2 and 3.

In experiment 2, 10 clover plants were grown on 800 gm. (air-dry basis) of Wooster, Hagerstown, Decatur, Sable, Herrick, and Caribou E1550 soils. In experiment 3, 10 clover plants were grown on 700 gm. of Caribou samples E1548, E1552, E1664, E1666, E1667, and E1558. The seeds were inoculated with the proper rhizobia for nitrogen fixation. The initial design of both experiments consisted of two 6 by 6 Latin squares. From the second square of each experiment a row of pots was removed at each even-numbered harvest and the exchangeable potassium and the weight and potash content of the roots determined. Exchangeable potassium was determined, as indicated previously, and the potassium content of tops and roots by a gravimetric cobaltinitrite method (*92*). Additions of Ca-Mg lime, phosphorus, and micronutrient elements were made at the beginning of the experiments where necessary and during the experiments as requirel. The desired pH level was 6 to 6.5. Available phosphorus was maintained at or above the minimum level of 300 pounds of P_2O_5 per acre by a modified Truog method (*64*). Distilled or demineralized water was added to the surface of the soil when required, usually daily. Fifteen harvests were made before discontinuance because of extremely slow growth. Seeding and harvest dates of these two experiments, as well as experiments 5 and 7, are included in table 9.

TABLE 9.—*Harvest dates of greenhouse experiments 2, 3, 5,*[1] *and 7*[2]

Harvest No.	Experiment 2		Experiment 3		Experiment 5		Experiment 7	
	Date	Duration	Date	Duration	Date	Duration	Date	Duration
		Days		*Days*		*Days*		*Days*
Seeding	12-18-44	0	1-26-45	0	4-26-47	0	11-13-47	0
1	3- 3-45	75	4-14-45	78	6-27-47	62	12-24-47	41
2	3-30-45	102	5-10-45	104	8- 3-47	99	4- 6-48	145
3	5-23-45	156	6-15-45	140	9-15-47	142	5-14-48	183
4	6-20-45	184	7-17-45	172	11- 1-47	189	6-18-48	218
5	7-25-45	219	8-22-45	208				
6	9- 4-45	260	9-28-45	245				
7	10-22-45	308	11- 2-45	280				
8	12- 6-45	353	12-21-45	329				
9	2- 4-46	413	2- 6-46	376				
10	4-23-46	491	4-26-46	455				
11	7- 3-46	562	7- 3-46	523				
12	8-29-46	619	8-29-46	580				
13	10-18-46	669	10-18-46	630				
14	11-29-46	711	11-29-46	672				
15	2- 5-47	779	2- 5-47	740				

[1] Discussion of experiment 5 begins on p. 24.
[2] Discussion of experiment 7 begins on p. 28.

The first cuttings were made when the plants were in about half-bloom, but the time of subsequent harvests was based more on the height of the plants. After harvests 4, 7, and 9, small additions of K_2SO_4 were made in an attempt to maintain better growth and a consequently greater extractive force on the nonexchangeable potassium. This added potassium, however, was absorbed rapidly with no evidence of improved growth.

The clover yields of complete Latin squares and significant differences for each harvest are listed in table 10.

Both the yields and the potash values listed later are calculated on the basis of pounds per 2,000,000 pounds of soil (pounds per acre). The yields of all harvests after the second are those of the first Latin square only.

In experiment 2 significant differences between yields on some soils occurred at every harvest. With a few exceptions the Sable soil far outyielded the others at the various harvests. The Herrick soil quite consistently was second in yield, while the relative positions of the re-

TABLE 10.—*Yields of Ladino clover in greenhouse experiments 2 and 3*

EXPERIMENT 2

Soil series or accession No.	Mean yield of dry matter in pounds per acre at harvest No.—															
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	Total
Wooster	7,018	6,432	6,345	6,982	3,880	2,078	3,286	2,403	1,782	4,626	5,540	4,524	3,987	2,562	2,506	65,550
Hagerstown	6,307	[illegible]	6,773	[illegible]	4,822	3,427	3,596	2,702	1,950	4,673	5,800	4,687	2,634	1,829	2,013	64,408
Decatur	5,727	[illegible]	5,775	6,155	3,654	[illegible]	3,425	2,723	2,059	4,965	5,693	3,458	2,910	2,132	1,817	58,908
Sable	9,750	7,870	7,09	7,177	4,268	3,208	5,290	4,378	3,309	[illegible]	10,731	7,717	6,091	3,497	4,087	93,989
Herrick	8,128	6,570	5,817	6,814	3,968	3,586	4,044	3,865	2,896	6,011	8,138	6,220	5,521	3,105	3,079	78,677
Caribou	4,212	4,547	4,775	5,717	4,272	3,081	3,565	2,080	2,089	4,582	5,465	4,734	3,247	2,347	2,523	57,936
Least significant difference, $P=0.05$	763	498	488	673	580	543	850	728	635	1,150	1,370	835	1,103	585	1,240	6,617
Least significant difference, $P=0.01$	1,025	665	669	918	790	(1)	1,163	990	868	1,570	1,870	1,138	1,500	795	(1)	9,035

EXPERIMENT 3

Soil series or accession No.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	Total
Caribou:																
E1548	2,390	2,170	3,517	2,244	2,108	2,985	1,913	2,281	1,578	4,855	5,827	6,190	4,393	3,886	3,036	48,713
E1552	5,098	5,882	7,004	4,428	3,949	3,583	2,727	3,102	2,041	5,396	6,508	5,817	4,545	4,216	4,348	69,482
E1664	9,402	8,613	8,191	5,104	4,399	3,280	2,527	2,762	1,946	5,478	[illegible]	6,009	4,394	3,665	3,202	76,865
E1666	9,486	10,473	[illegible]	6,988	6,682	5,585	4,167	3,606	2,534	6,612	8,257	6,205	4,689	3,803	3,341	95,416
E1667	10,204	11,422	16,152	7,905	9,054	6,560	7,059	4,096	3,227	7,644	10,957	7,559	5,242	4,775	3,343	117,299
E1558	8,239	8,779	10,416	5,065	4,743	4,263	2,815	3,247	2,365	6,214	8,525	6,337	4,700	3,562	3,767	83,037
Least significant difference, $P=0.05$	620	550	937	631	869	871	806	683	443	1,097	1,689	997	(1)	(1)	(1)	3,944
Least significant difference, $P=0.01$	828	735	1,280	860	1,183	1,189	1,097	931	603	1,497	2,203	1,360	(1)	(1)	(1)	5,379

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[1] No significant difference.

maining four fluctuated between cuttings. The total yields on the Decatur and Caribou were appreciably less than those on the Wooster and Hagerstown and about 60 percent below the yield on the Sable. The ability of the Sable and Herrick soils to outlast the others became obvious during the second half of the experiment.

In experiment 3 the superior ability of the straw and manured samples (E1666 and E1667) to produce clover was apparent from the start and continued to be evident until about the twelfth harvest. After this the superiority of even the manured sample was not significant, according to analysis of variance. The no-potash sample (E1548) was the lowest producer until the twelfth harvest. All differences between adjacent pairs of total yield values are significant beyond the 1-percent level of probability. The order of yields follows that of increasing applications of potash; however, the quantities added in the straw and manure can only be estimated. The uptake of potash by the clover is shown in table 11.

These values were determined from compositing the dry matter of all pots of each soil. A large fraction of the uptake in the first several harvests is derived from soluble and exchangeable potassium, so that early differences between soils chiefly reflect differences in exchange levels. Later the exchangeable form becomes reduced to a minimum level and the uptake then represents a rough index to the relative availability of the nonexchangeable reserve. A sharp decline in uptake during the course of experiment 2, regardless of the soil, is readily apparent. Throughout the experiment, the Sable far exceeded the other soils, which were grouped relatively close, although the Herrick was second quite consistently.

In experiment 3 the pattern of potash absorption by the clover closely paralleled that of the yields. The enormous uptake from the manured sample in the first three cuttings is due to its high initial content of exchangeable potassium. This sample provided a higher uptake even at the end of the experiment, but at this stage its superiority was slight. A slight advantage was maintained by E1558 and E1666 over the remaining samples.

An indication of the percent of potash content of the clover may be obtained by dividing the values of table 11 by the corresponding values of table 10. In experiment 2 luxury consumption occurred in the first harvest, and throughout the experiment the Sable and Herrick soils produced clover of higher potash content than did the other soils. In the later harvests the general average for the group of four lower potash soils was about 0.3 percent, which is considerably below the value of 0.96 percent in leaves, which has been said to be the critical level for deficiency symptoms in Ladino clover (*22*). In experiment 3 the initially high values were abruptly reduced in subsequent harvests, and after harvest 9 all percentages ranged between 0.2 and 0.4. At the first harvests of the experiments, the size of leaves and stems was about normal for Ladino clover, but later declined to about that of white clover.

At alternate harvests the roots of the discontinued pots were removed while moist by screening; and were washed, dried, weighed, and analyzed for potassium. The soil was air-dried and its exchangeable potassium content was determined. Declines in root weights occurred near the finish of the experiments because of death of plants and runners.

TABLE 11.—*Uptake of potash by Ladino clover in greenhouse experiments 2 and 3*

EXPERIMENT 2

Soil series or accession No.	Potash uptake by clover tops in pounds K_2O per acre for harvest No.—															
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	Total
Wooster	77	44	39	27	39	22	14	17	11	10	21	15	13	9	7	365
Hagerstown	98	53	37	28	40	22	13	23	11	19	17	18	7	6	6	398
Decatur	68	44	20	29	38	14	13	19	13	18	16	9	6	6	5	318
Sable	239	132	110	75	69	49	58	51	41	51	59	37	27	18	20	1,036
Herrick	94	52	47	28	37	26	24	27	15	21	24	24	19	15	12	465
Caribou	39	26	28	27	39	16	13	19	8	17	12	9	6	7	6	272

EXPERIMENT 3

Soil series or accession No.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	Total
Caribou:																
E1548	10	10	18	11	9	10	5	10	6	13	18	14	11	9	7	161
E1552	60	63	46	25	20	17	7	14	7	13	18	14	11	10	10	335
E1664	81	69	43	21	22	13	7	14	8	16	25	15	12	9	11	366
E1666	249	190	142	60	42	29	14	20	12	21	24	15	13	12	11	854
E1667	391	384	287	115	105	82	39	24	16	28	32	24	17	17	14	1,575
E1558	165	144	90	40	31	29	11	18	11	22	23	18	15	12	13	642

This effect is reflected in the total potash content of the roots (table 12).

TABLE 12.—*Amount of potash in Ladino clover roots in greenhouse experiments 2 and 3*

EXPERIMENT 2

Soil series or accession No.	Potash in pounds K_2O per acre for harvest No.— 2	4	6	8[1]	10	12	14	15[2]
Wooster	26	14	24	25	26	10	9	3
Hagerstown	29	20	17	20	22	16	9	2
Decatur	12	17	18	19	22	13	10	1
Sable	67	65	34	37	40	17	20	6
Herrick	30	18	19	21	22	13	11	5
Caribou	22	19	15	15	15	9	8	3

EXPERIMENT 3

Soil series or accession No.	2	4	6	8[1]	10	12	14	15[2]
Caribou:								
E1548	5	7	7	9	10	12	9	1
E1552	24	12	11	12	12	13	17	3
E1664	29	14	11	11	11	10	16	4
E1666	55	41	14	15	15	15	16	5
E1667	105	85	46	34	22	19	17	6
E1558	64	29	17	19	21	14	15	5

[1] Samples lost; values estimated as means of harvests 6 and 10.
[2] Values low because of leaching of potash from dead roots on washing.

The roots in the Sable soil contained about twice as much potassium as those in any of the other five soils throughout the experiment. In experiment 3 the potash content of the roots in the manured sample, E1667, was high at the earlier harvests, but at the conclusion of the experiment was virtually no higher than those of the other samples. Part of this decrease can be attributed to translocation from the roots to aerial tissues.

The exchangeable potassium values are tabulated in table 13 and shown graphically in figures 1 and 2.

TABLE 13.—*Exchangeable potash contents of soils in greenhouse experiments 2 and 3*

EXPERIMENT 2

Soil series or accession No.	Initial	Exchangeable potash in pounds K_2O per acre after harvest No.— 2	4	6	8	10	12	14	15[1]
Wooster	200	90	88	70	84	70	89	64	84
Hagerstown	309	122	116	108	89	99	85	80	87
Decatur	263	148	112	116	120	138	129	107	114
Sable	500	298	296	260	275	240	253	237	265
Herrick	290	186	156	172	169	174	188	145	182
Caribou	168	84	76	71	83	82	58	61	77

EXPERIMENT 3

Soil series or accession No.	Initial	2	4	6	8	10	12	14	15[1]
Caribou:									
E1548	55	78	84	52	68	77	66	55	74
E1552	220	98	99	92	85	86	65	68	78
E1664	244	100	74	78	83	70	78	80	70
E1666	584	129	114	94	98	89	94	69	77
E1667	1,100	287	110	96	104	92	80	77	90
E1558	170	129	82	89	68	60	72	80	74

[1] Mean of 3 replicates.

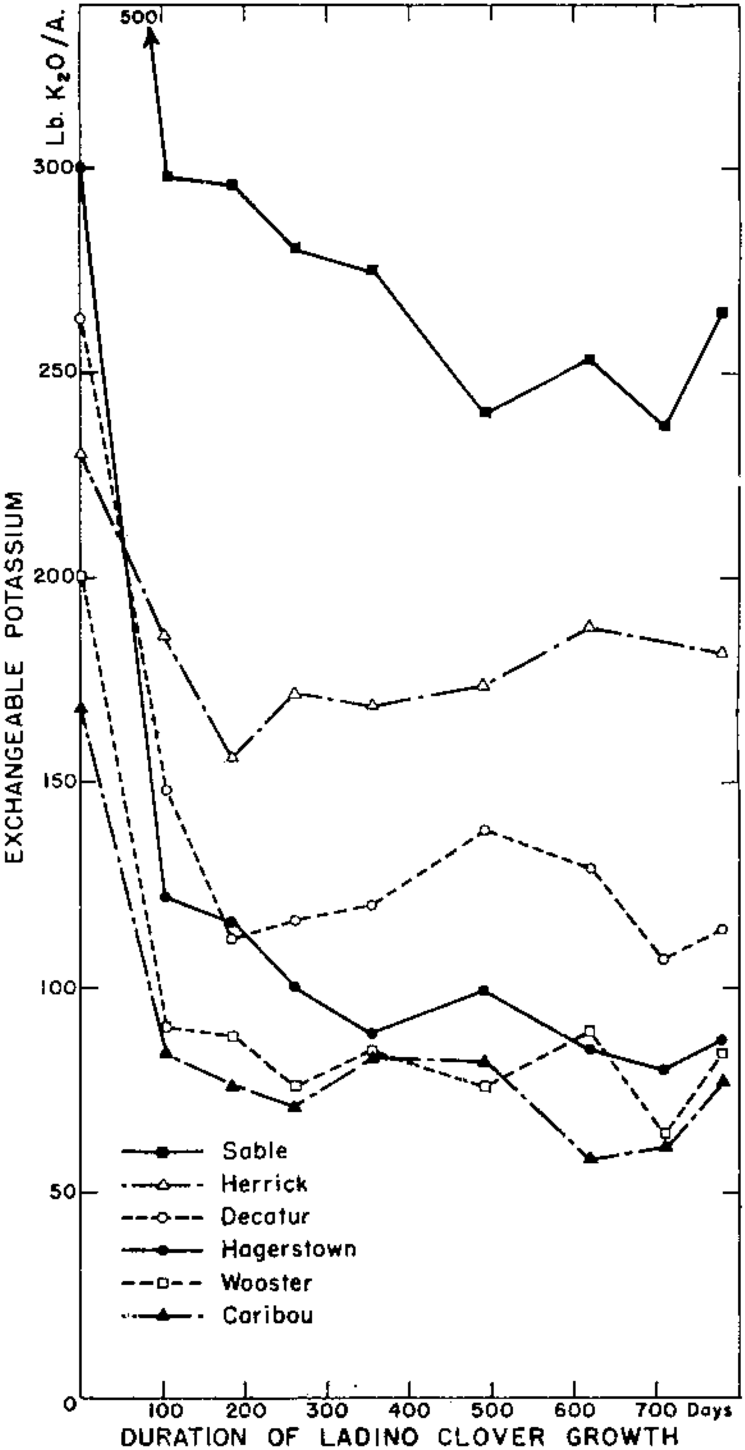

FIGURE 1.—Exchangeable potassium of soils in greenhouse experiment 2.

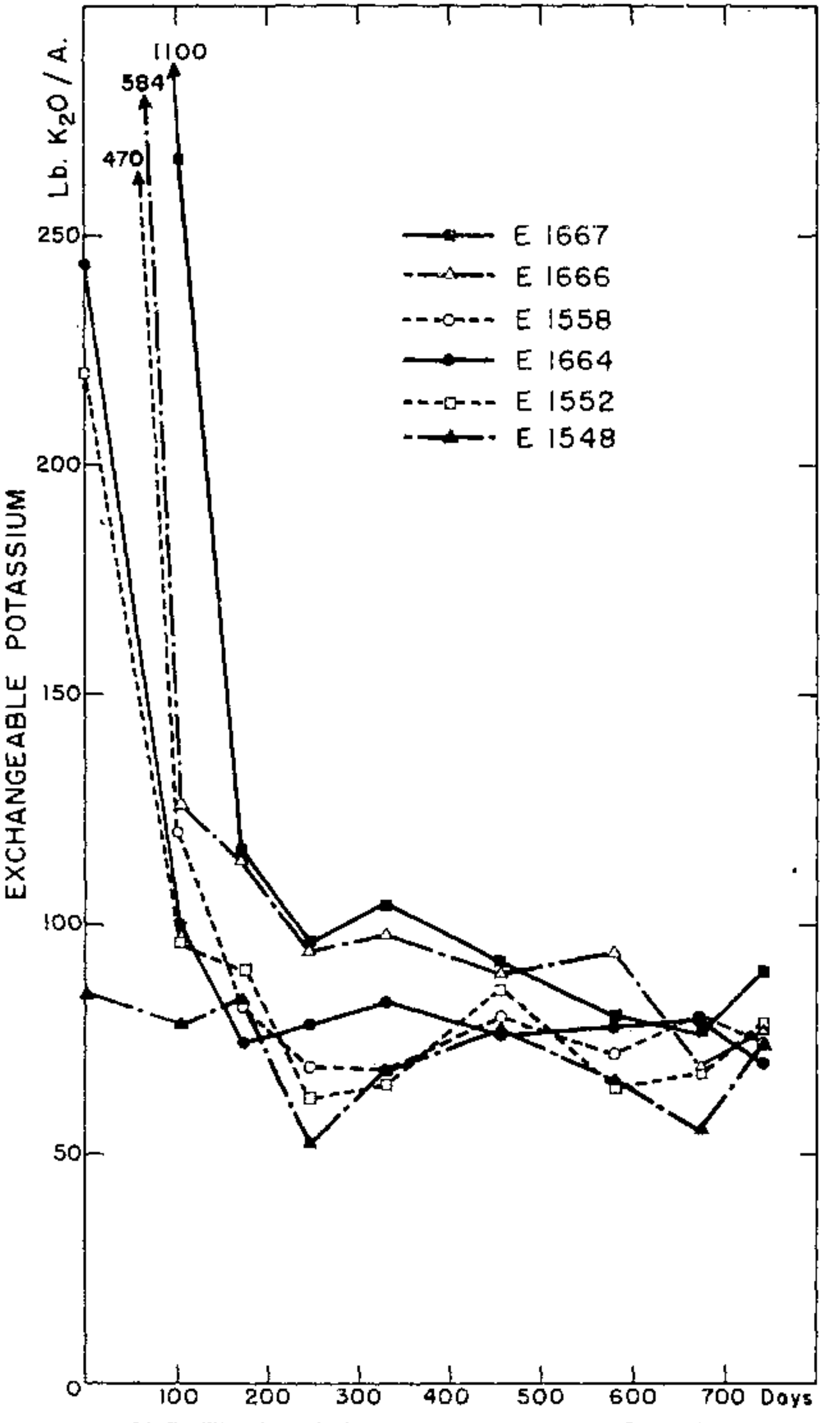

FIGURE 2.—Exchangeable potassium of soils in greenhouse experiment 3.

In experiment 2 the initial exchange values ranged from 168 to 500 pounds of K_2O per acre. By the second harvest all values had been drastically reduced. Disregarding the fluctuations that occurred subsequently and which are attributed to other causes, the values found at harvest 2 were reduced no further during the rest of the experiment. This statement perhaps does not apply to the Sable soil, which exhibited a decline until harvest 10; on a percentage basis, however, this decrease for the Sable is not so great as the range of fluctuation for some of the other soils. With respect to minimum exchange levels, the Wooster, Hagerstown, and Caribou are grouped closely, while the Decatur is somewhat higher, followed by the Herrick and Sable at ever-widening intervals.

In experiment 3 the initial exchange values covered a wide range, and on this basis the six samples can be grouped in four levels, namely (a) E1548, (b) E1552 and E1664, (c) E1666 and E1558, and (d) E1667. The potash in excess of 85 pounds per acre, the E1548 value, represents that accumulated from fertilizer, straw, manure, or green manure which was not absorbed by crops, fixed, or leached from the surface soil The initial level for E1548 was scarcely reduced during the entire experiment, as it is but slightly higher than the minimum value for this soil type. The values for the other samples were severely reduced by harvest 2, and several were at the minimum level by harvest 4. Since all the samples reached the same minimum range of values, the general pattern differs from that of experiment 2, in which there occurred a considerable range of minimum values.

The existence of this characteristic of intensively cropped soils to retain a minimum level of unavailable exchangeable potassium against absorption has been observed by a number of workers; for example, Ayres (*7*), Bear, Prince, and Malcolm (*12*), Chandler, Peech, and Chang (*22*), and Gholston and Hoover (*35*). An explanation was advanced by Ayres (*7*) that this minimum level represents an average for a mass of soil and that the concentrations at root-soil contacts are considerably lower than the average and maximum values.

The calculation of cumulative release of nonexchangeable potash during the period from the start of an experiment to any particular harvest is as follows:

Potash release = cumulative uptake by tops +
current content of roots + current exchangeable content −
initial exchangeable content −
quantity added to the soil during the growth period (exclusive of that added in water or other extraneous sources).

Cumulative values for experiments 2 and 3 are included in table 14 and shown graphically in figures 3 and 4.

Release occurred in all soils but its extent varied considerably. In general it did not proceed by equal increments between alternate harvests; this may be caused by a number of factors; for example, an initial high exchange level, varying intervals of time between harvests, seasonal and random changes in weather factors affecting growth, infestations by insects, and applications of soluble potash. For all soils the highest rate of release occurred in the early stages, but large proportions of the total release occurred while the soil was in a minimum exchange status.

In experiment 2 the total release varied from 82 pounds per acre for the Decatur to 763 for the Sable, a range of almost tenfold. The average rate for the Sable soil was almost 2 pounds per acre per day. Herrick and Wooster soils were intermediate. It is presumed from the history of these six soil samples that no appreciable fixation of potassium had occurred in the field and that consequently the magnitude of release is an index of the availability of the native potash. The fraction of the clover potash originating in the nonexchangeable reserve is obtained by dividing the total release by the sum of the uptake by the tops and the final root content. For the six soils, in the order of table 14, these fractions are 0.45, 0.25, 0.26, 0.73, 0.72, and 0.35. Although the magnitudes of these values are, of course, dependent upon the initial exchange levels, their grouping is quite similar to that of the release values.

TABLE 14.—*Cumulative release of nonexchangeable potash in greenhouse experiments 2, 3, 5, and 7*

Soil series	Release of potash in pounds K_2O per acre											
	In experiment 2 after harvest No.—								In experiment 5 after harvest No.—		In experiment 7 after harvest No.—	
	2	4	6	8	10	12	14	15	2	4	2	4
Wooster	37	60	80	101	112	145	141	186	250	206	441	523
Hagerstown	2	58	45	48	75	30	91	99	137	168	245	209
Decatur	0	27	32	40	84	90	77	82	110	143	213	294
Sable	236	417	438	515	605	691	728	763	920	1,045	1,359	1,477
Herrick	132	165	195	210	247	200	289	327	413	503	688	800
Caribou	3	47	43	62	71	82	77	97				
Caribou accession No.	In experiment 3								In experiment 5		In experiment 7	
E1548	18	55	42	50	64	87	93	118	155	182	264	382
E1552	28	70	84	84	110	122	150	159				
E1664	35	56	44	95	97	147	165	163				
E1666	36	212	230	250	250	308	304	317				
E1667	347	278	406	461	470	511	540	582	617	[illegible]	753	920
E1558	21	71	117	130	152	178	214	215	286	298	379	531

In experiment 3, release was manifested by the second harvest, even though E1667 had an initial exchange content of 1,100 pounds per acre. Release continued to increase during the rest of the experiment, but only very slowly near its termination. Among the six Caribou samples the qualitative orders with regard to initial exchange level, yield, uptake, and release present a consistent picture. Without exception, the order, from low to high, is E1548, E1552, E1664, E1558, E1666, and E1667. This is probably the same order as for the total additions of potash to the plots. If the release from E1548, 118 pounds, is assumed to be the result entirely from native reserves, the release from fixed supplies in the other samples ranges from 40 to 440 pounds per acre. For the six samples in the order of table 14, the fractions of clover potash derived from nonexchangeable forms are 0.73, 0.47, 0.44, 0.37, 0.36, and 0.33. With respect to order of availability of potash reserves, this pattern is virtually opposite that of experiment 2.

Following the discontinuance of experiments 2 and 3 because of severe decline and low potash uptake by the clover, 8 of the 12 soils that had produced 15 cuttings of clover were replanted to Kobe lespedeza in a study designated experiment 5. These were the soils of experiment 2,

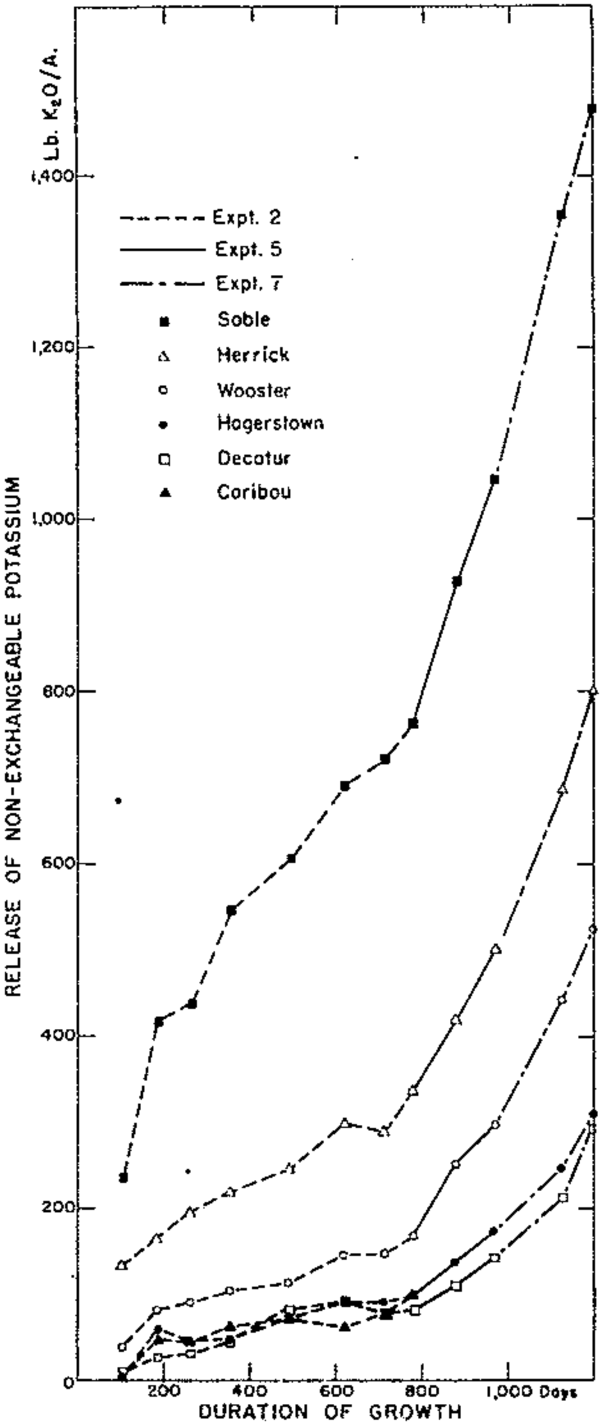

FIGURE 3.—Cumulative release of nonexchangeable potassium in greenhouse experiments 2, 5, and 7.

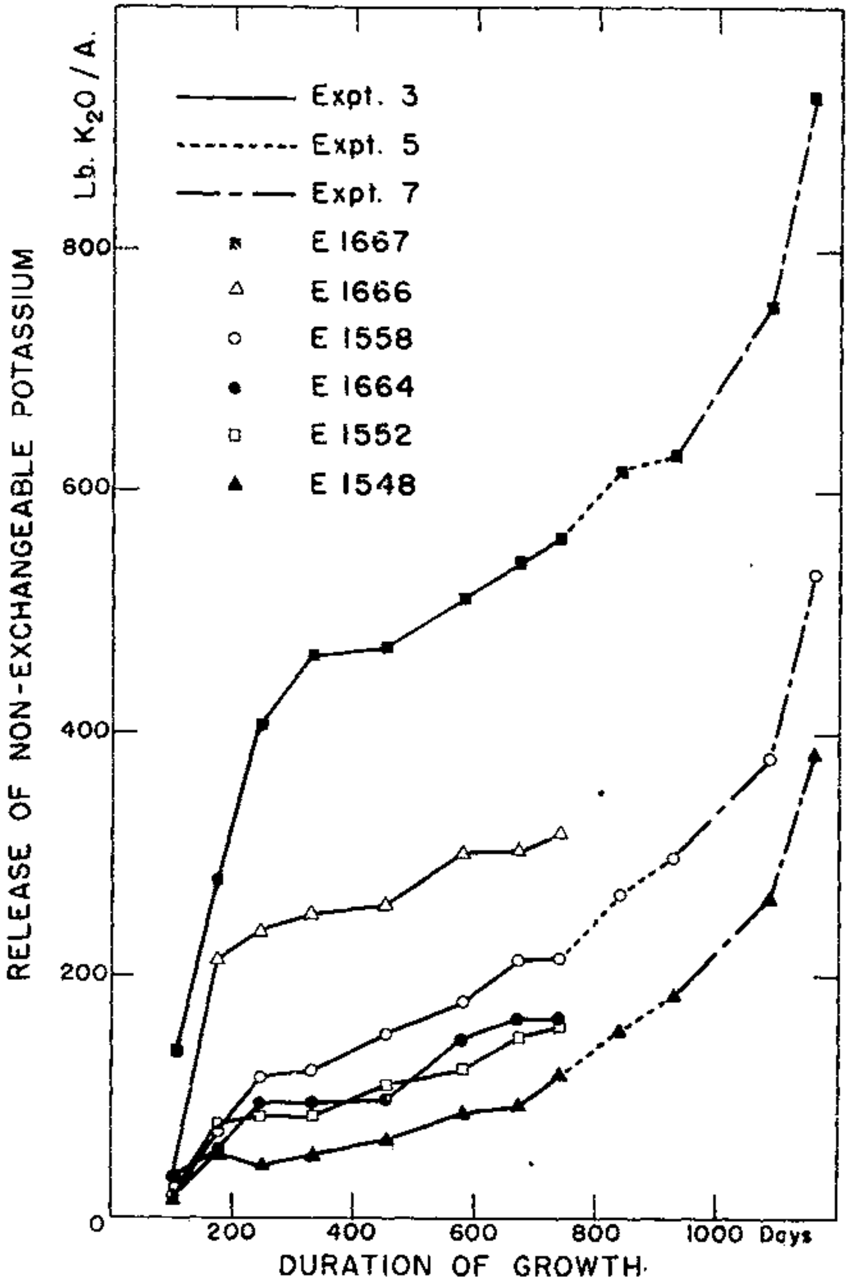

FIGURE 4.—Cumulative release of nonexchangeable potassium in greenhouse experiments 3, 5, and 7.

except the Caribou, and 3 Caribou samples from experiment 3, E1548, E1558, and E1667. Experimental arrangements included the following: 700 gm. of soil in small ceramic pots; 4 randomized blocks, each having 1 replicate of each soil; seeds planted after contact with 75 percent H_2SO_4 for 30 minutes; inoculated with rhizobia for lespedeza; and thinned to 20 plants 19 days after planting. Seeding and the 4 harvest dates are listed in table 9. One block was discontinued after harvest 2 for root and exchange determinations.

The yields and potash uptake of the lespedeza are presented in table 15. Significant differences in growth occurred throughout the experiment, the Wooster, Sable, and Herrick soils showing superiority from the start. As a group, the three Caribou samples performed quite poorly as compared with the other soil samples, but E1558 and E1667 outproduced E1548. The plants on most of the soils declined abruptly during the fourth harvest period, and the experiment was terminated after this cutting. Appreciable uptake of potash from some soils occurred, especially in the first two growth periods. Except for the potash uptake that might originate from a further decrease in exchange level from the minimum of the clover experiments, the source of this is the nonexchangeable reserve. The uptake from the Wooster, Sable, and Herrick soils therefore represents a substantial liberation of potassium. The remaining five samples are grouped in a much lower range, which suggests a drastic reduction in the moderate availability of the fixed potassium of the Caribou samples.

TABLE 15.—*Yields and potash uptake of Kobe lespedeza in greenhouse experiment 5*

Soil series	Accession No.	Mean yield of dry matter in pounds per acre at harvest No.—					Potash uptake in pounds K_2O per acre for harvest No.—				
		1	2	3	4	Total	1	2	3	4	Total
Wooster	E1640	8,892	6,582	5,564	3,510	24,548	59	36	28	18	141
Hagerstown	E1670	5,589	5,337	4,407	1,910	17,243	21	27	22	7	77
Decatur	E1583	3,841	4,451	2,504	1,723	12,519	17	27	12	6	62
Sable	E1618	9,269	11,198	14,760	5,020	40,247	116	109	84	30	339
Herrick	E1610	9,738	6,504	10,463	6,366	33,071	88	67	44	13	212
Caribou	E1548	3,185	3,702	1,375	1,545	9,807	16	21	8	6	51
Do	E1667	5,009	7,830	2,745	1,247	16,831	23	35	12	4	74
Do	E1558	3,007	5,528	3,965	1,987	14,487	15	29	21	8	73
Least significant difference, $P=0.05$	....	1,380	1,580	2,348	971	3,517	.....	....	......	.....	....
Least significant difference, $P=0.01$	....	1,880	2,151	3,248	1,346	4,860	.....	....	......	.....	......

The potash contents of the lespedeza roots at harvests 2 and 4 (table 16) emphasize even better the rapid decline of the plants at the finish of the experiment.

The death of roots seriously reduced the recoverable proportion and permitted the return of potassium to the soil.

Exchangeable potassium values of experiment 5 are listed in table 16. The initial values shown are the final values of experiments 2 and 3. The drop in exchange level of the soils at harvest 2 below the minimum levels of those experiments is not sufficiently great to be accepted as significant except in the cases of the Sable and Herrick soils. For these

TABLE 16.—*Amount of potash in roots of Kobe lespedeza and exchangeable potash contents of soils in greenhouse experiment 5*

Soil series	Accession No.	Root potash in pounds K_2O per acre for harvest No.—		Exchangeable potash in pounds K_2O per acre after harvest No.—		
		2	4	Initial	2	4
Wooster	E1649	12	1	84	61	72
Hagerstown	E1670	8	1	87	70	78
Decatur	E1563	6	1	114	92	112
Sable	E1648	24	2	205	179	206
Herrick	E1640	15	1	182	94	135
Caribou	E1548	8	1	74	66	86
Do	E1667	13	2	90	74	82
Do	E1558	10	2	74	71	82

two soils, at first glance the minimum level appears to vary with the plant. The higher values obtained for the fourth harvest are attributed to potash of dead roots that was either returned to the soil or contained in small pieces of roots remaining with the soil during screening.

The extent of release during experiment 5 is not listed separately but is added to that of the preceding experiments, as indicated by the cumulative values of table 14 and figures 3 and 4. Appreciable release occurred during both halves of the experiment. The Wooster, Sable, and Herrick soils released considerably more potash than did the other five soils, whose total release values were grouped closely between 61 and 83 pounds per acre. The average rates of release for all soils were higher than in the clover experiments; that of the Sable was close to 1.5 pounds per acre per day.

Samples of the same 8 soils, which had undergone 15 clover harvests and 4 lespedeza cuttings, were next planted to perennial ryegrass in an experiment designated No. 7. Experimental arrangements included the following: 700 gm. of air-dry soil in small ceramic pots; 3 randomized blocks, each having 1 pot of each soil; addition of Ca-Mg lime, micronutrients, and 200 pounds of NH_4NO_3 per acre. Seeding and harvest dates are recorded in table 9. Subsequent nitrogen additions were as

TABLE 17.—*Yields and potash uptake of perennial ryegrass in greenhouse experiment 7*

Soil series	Accession No.	Mean yield of dry matter in pounds per acre at harvest No.—					Potash uptake in pounds K_2O per acre for harvest No.—				
		1	2	3	4	Total	1	2	3	4	Total
Wooster	E1649	2,294	4,834	6,360	2,928	16,416	16	58	90	40	204
Hagerstown	E1670	2,044	4,482	4,820	2,920	14,272	13	32	46	27	118
Decatur	E1563	1,844	4,410	5,386	2,460	14,100	16	38	51	24	129
Sable	E1648	2,418	4,416	6,643	2,937	16,414	84	117	151	70	422
Herrick	E1640	2,880	4,716	6,595	2,820	17,011	67	72	92	56	280
Caribou	E1548	2,178	4,671	6,780	3,308	16,937	15	40	79	47	181
Do	E1667	2,575	5,706	8,591	3,077	20,840	16	61	111	63	251
Do	E1558	2,210	4,427	5,714	2,826	15,177	15	49	92	46	202
Least significant difference, $P=0.05$		300	591	1,231	(1)	2,257					
Least significant difference, $P=0.01$		423	817	1,823	(1)	3,384					

[1] No significant difference.

follows: April 6, 1948, 100 pounds of N per acre as NH_4NO_3; June 11, 1948, 50 pounds of N per acre as $Ca(NO_3)_2 \cdot 4H_2O$.

The results of experiment 7 are included in tables 17 and 18. The yields did not show the magnitude of range obtained in the preceding experiments. Nevertheless, the precision was sufficient to establish highly significant differences for the first three harvests and for the totals. The previous superiority of the Herrick and Sable soils with respect to growth was not evident here, and after the first cutting the manured Caribou sample, E1667, produced the greatest yields. The potash uptake, however, was highest in the Herrick and Sable, the latter being far in the lead at each harvest; on this basis the manured Caribou was third. It is of interest that the potash contents of the plants on the six soils of lowest uptake, expressed in percent, increased considerably during the experiment, which is directly opposite the trend in the preceding experiments. The range for the eight soils at harvest 4 was 0.93 to 2.38 percent. This increase is ascribed partly to the development of large root systems during the first half of the experiment. After the third cutting, the grass underwent rapid decline and large quantities of roots died. When addition of calcium nitrate effected no noticeable recovery the experiment was concluded. At harvest 4 the roots could not be separated satisfactorily and the portion screened out was pulverized and returned to the soil. The roots of ryegrass had accumulated far more potash than those of lespedeza at corresponding stages of experiments 5 and 7.

TABLE 18.—*Amount of potash in roots of perennial ryegrass and exchangeable potash contents of soils in greenhouse experiment 7*

Soil series	Accession No.	Root potash in pounds K_2O per acre for harvest No. 2[2]	Exchangeable potash in pounds K_2O per acre after harvest No.—	
			2	4[1]
Wooster	E1649	67	76	95
Hagerstown	E1670	34	76	101
Decatur	E1563	34	94	134
Sable	E1648	136	177	216
Herrick	E1610	69	113	146
Caribou	E1548	30	74	105
Do	E1667	55	73	127
Do	E1558	37	62	113

[1] Roots of harvest 4 not harvested.
[2] Includes all roots; mean of two replicates.

This return of root potash to the soil increased the exchangeable potassium values for harvest 4 considerably over those for harvest 2, which was previously observed in experiment 5. Therefore, only the value for harvest 2 of each experiment can be regarded as representative of the minimum exchange level. For most soils the agreement for harvest 2 of experiments 5 and 7 is good and particularly so for Decatur, Sable, and Caribou E1667.

Cumulative release values are indicated in table 14 and figures 3 and 4. All soils released potash at both the second and fourth cuttings. The total release of potash in experiment 7 was considerable, greatly exceeding that of the lespedeza experiment. The Sable released the most potash by far, and the Herrick and manured Caribou were at the second level. The Hagerstown and Decatur were least effective. The Sable and Herrick, and to a lesser extent the Wooster, liberated an excessive fraction of the total during the first two harvest periods. The disintegration of the roots in the later stages must be partly responsible for this, because at harvest 2 the roots contained as much potash as the tops.

Figure 3 indicates that both lespedeza and ryegrass can utilize nonexchangeable potassium more effectively than Ladino clover, even though they are grown subsequent to the clover. This can be considered a parallel to the observation of Evans and Attoe (*27*) that Ladino clover extracted more nonexchangeable potassium from soils of relatively high exchange level than did oats, but less on soils of low exchange level. This was attributed to the ability of plants such as oats to grow well at low levels of available potassium. The relative order of the soils with respect to potassium-supplying capacity, however, was not greatly altered by the change in plants except in the case of ryegrass on Caribou soils. In the lespedeza experiment the three Caribou samples were grouped together with Hagerstown and Decatur at the low level; however, ryegrass absorbed considerably more potash from the Caribou samples, and the manured soil released as much as the Herrick.

Air- and Oven-Drying

After harvest 14 of experiments 2 and 3, exchangeable potassium was determined on a part of the soil of the discontinued replicates (1) while it retained the moisture present after screening out the roots, (2) after being air-dried in the usual manner for 7 days, and (3) after being heated in an oven at 105° C. for 1 day. The results (table 19) suggest that there was a general increase in exchangeable potassium by both air-drying and oven-drying.

With only one exception, all values increased successively by the two consecutive dryings. However, many of the changes due to air-drying amounted to only about 10 pounds; for example, E1649, E1670, E1550, E1548, E1552, and E1667. The outstanding increases occurred in the Decatur, Herrick, and especially the Sable, which showed an increase of 132 percent.

At harvest 15, the soil from duplicate pots was subjected to similar treatments (table 19). Storage while moist for 6 and 46 days produced no consistent or substantial change in the exchange level. After 4 days of air-drying only the Herrick and Sable exhibited large increases; the others showed a small increase or decrease. Oven-drying brought about further appreciable increases for the Sable and Herrick and about 10-pound increases in the Decatur and E1667. Air-drying for 10 days after moist storage for 46 days resulted in significant increases only for the Sable and Herrick. On the basis of this and later evidence it is considered that the determined moist values for harvest 14 were low, and that only the Sable and Herrick, and to a lesser degree the Decatur, release appreciable amounts of potassium upon drying.

This conclusion is supported by results from harvest 2 of experiments 5 and 7. Only the Sable, Herrick, and Decatur consistently released significant amounts of potassium by air-drying; the value of 94 for Herrick appears far too low, for unknown reasons. For harvest 4 of

TABLE 19.—*Exchangeable potash as affected by air- and oven-drying, in pounds K_2O per acre*

Soil series	Accession No.	Experiments 2 and 3, harvest 14			Experiments 2 and 3, harvest 15[1]						Experiment 5, harvest 2		Experiment 5, harvest 4[2]		Experiment 7, harvest 2	
		Wet, 0 day	Air-dry, 7 days	Oven-dry, 4 day	Wet, 0 day	Wet, 6 days	Air-dry, 4 days	Oven-dry, 1 day	Wet, 40 days	Air-dry, 10 days	Wet, 0 day	Air-dry	Wet, 0 day	Air-dry	Wet, 0 day	Air-dry, 7 days
Wooster......	E1019......	57	64	80	80	70	85	88	92	86	55	61	87	72	80	78
Hagerstown...	E1070......	88	80	82	88	91	90	91	101	84	60	70	95	78	77	70
Decatur......	E1503..	64	107	121	108	110	114	122	116	110	87	92	125	112	75	94
Sable........	E1018.....	102	237	217	137	146	206	209	140	233	123	179	170	207	128	177
Herrick.....	E1046.....	79	145	197	80	104	181	190	109	151	80	94	109	125	88	113
Caribou......	E1550......	50	61	71	102	77	83	78	76	68	.....	.....	.....	.....	.....	.....
Do.......	E1548......	42	55	68	72	64	78	78	75	70	60	66	110	86	87	74
Do.......	E1552......	50	68	64	86	80	77	85	77	68	.....	.....	.....	.....	.....	.....
Do.......	E1604......	50	88	78	80	80	75	77	70	68	.....	.....	.....	.....	.....	.....
Do.......	E1666......	41	60	92	88	84	80	84	80	70	.....	.....	.....	.....	.....	.....
Do.......	E1667......	68	77	89	99	100	95	105	104	87	71	74	110	82	109	79
Do.......	E1558......	50	80	81	77	70	75	77	60	60	80	71	114	82	69	92

[1] Mean of 2 replicates.
[2] Mean of 3 replicates.

experiment 5 the results indicate, except for the Sable and Herrick, fixation on air-drying of some of the additional potash from dead roots.

Later experiments on particle-size separates of these soils, not reported here, have demonstrated that this specific ability to release potassium is a property of clay minerals of these soils. A rough averaging of acceptable data leads to these approximate values of the release of potash by air-drying of Decatur, Herrick, and Sable, respectively; 10, 60, and 120 pounds per acre in experiment 2; 10, 25, and 50 pounds in experiments 5 and 7. As soils of experiments 2 and 3 were air-dried prior to planting experiment 5, and again before planting experiment 7, sizable fractions of the release by the Herrick and Sable soils during the lespedeza and ryegrass growth periods must be assigned to the respective release on air-drying that occurred just prior to the start of the latter two experiments. Thus, of the 166 pounds apparently released by the Herrick soil during lespedeza growth, 60 pounds was liberated by air-drying; similarly for the Sable, 120 of the 282 pounds indicated. In the ryegrass experiment, 25 pounds of the 297 released by Herrick is attributed to air-drying, as is 50 of the 432 pounds released by the Sable. Adjustments to account for this effect would reduce the rates of release of the two soils during experiments 5 and 7.

Neubauer

The 14 soil samples were subjected to the Neubauer potassium test by a procedure in general accordance with the usual technique (*82*). Registered Rosen rye seedlings were grown in quadruplicate cylindrical Pyrex glass dishes arranged in four randomized blocks for 17 days in a room maintained at 20° ± 1° C. The fluorescent-light intensity at plant level for 16 out of every 24 hours averaged 90 foot-candles. The potassium content of the seedlings was determined by a gravimetric cobaltinitrite method (*92*).

A close agreement between many of the mean Neubauer values and the corresponding exchangeable potassium values was noted, which might suggest that in these cases all of the exchangeable potassium was absorbed by the rye seedlings. As the elimination of exchangeable potassium by cropping was considered improbable, a second run was made, using the same procedure as in the first; however, the mixture of soil, sand, and root washings was not discarded as before but was evaporated and air-dried at room temperature and analyzed for the residual content of exchangeable potassium. The Neubauer and exchange values and the release values calculated from them are listed in table 20.

Very close agreement between exchange and Neubauer values is illustrated by samples E1646, E1552, and E1665. Moderate agreement is indicated for a number of other samples. However, it is to be noted that after removal of the rye seedlings all soils contained residual exchangeable potassium, and that the residual quantities are in general agreement with the minimum exchange values of the prolonged cropping experiments, except for the manured Caribou sample E1667. The high residual value for this sample is attributed to the fact that its Neubauer value is the maximum possible with 100 gm. of soil under the particular growth conditions.

Release of potash is indicated for all soils except the Hagerstown in amounts ranging from 12 to 313 pounds per acre. Some of the differences between the lesser values are not significant. The higher values represent a considerable release when it is considered that the growth period was only 17 days. In the cases of the Herrick and Sable about 50 and 100 pounds, respectively, of the release should be assigned to the air-drying operation necessary to prepare the soils for determination of residual potassium. In this Neubauer procedure the release by the Sable was exceeded by that of E1667. Coupled with the results of the ryegrass experiment, this suggests that the nonexchangeable potassium of the Caribou soil is relatively available to grasses. In a later experiment, reduction of the amount of E1667 to 25 gm. did not substantially increase the release expressed as pounds per acre. The correlation coefficient between Neubauer release and that during 740 days of Ladino clover growth was 0.897 (*72*).

TABLE 20.—*Release of nonexchangeable potash in Neubauer study, in pounds K_2O per acre*

Soil series	Accession No.	Neubauer value	Residual exchangeable potash [1]	Sum of Neubauer and residual potash	Initial exchangeable potash	Nonexchangeable potash released
Wooster	E1639	164	80	244	200	44
Hagerstown	E1670	167	83	250	270	−20
Decatur	E1563	234	113	347	263	84
Sable	E1648	561	199	760	500	260
Herrick	E1646	236	115	351	230	121
Caribou	E1570	122	72	194	168	26
Do.	E1548	45	61	106	85	21
Do.	E1752	222	67	289	220	69
Do.	E1663	164	58	222	210	12
Do.	E1664	221	62	283	244	39
Do.	E1665	319	69	388	310	78
Do.	E1666	690	97	787	584	203
Do.	E1667	1,271	142	1,413	1,100	313
Do.	E1558	512	79	591	470	121
Least significant difference, $P=0.05$		85	15	37		97
Coefficient of variation [2]		6.9	11.1	5.8		26.0

[1] Remaining after seedlings were removed from soil.
[2] Expressed as percent.

Electrodialysis

One hundred-gm. portions of the 14 soil samples were continuously electrodialyzed in 2 Mattson-type cells having platinum-gauze electrodes and cellophane membranes under a potential difference of 100 to 110 volts for periods of 30 to 90 days. The open cross-sectional dimensions of the cells were 5.3 by 15 cm., and the thickness of the center soil compartment 3.6 cm. The direct-current source was at first a battery of large wet cells; later, a direct-current motor-generator. The electrode chambers were drained at frequent intervals, especially near the start of a run. After drainings the current direction was reversed, and potassium in each catholyte was determined separately. The magnitude of the current is affected at first by initial electrolyte content and quantity of exchangeable bases and later by the extent of decomposition and liberation of nonexchangeable bases.

Curves representing the cumulative extraction of exchangeable and nonexchangeable potassium for periods up to 60 days are shown in figures 5 and 6.

All curves present a generally similar appearance. A quantity of potash equivalent to the initial exchangeable is liberated within a period of 1 to 3 days. This is followed first by a period of decreasing rates and later by a period of virtually linear release, of slope characteristic of the particular soil. The curves for electrodialysis over 30-day periods, found by Ayres and coworkers (*7*, *8*), are very similar to those shown here.

The correlation and regression coefficients for the relationship between release during 740 days of Ladino clover growth and that during 30 days of electrodialysis were previously reported to be 0.964 and 1,063, respectively (*72*). The rates of release for the constant-rate parts of the dialysis curves, for the 14 soil samples in the order listed in table 20 are as follows: 3.0, 3.3, 0.88, 10.3 (mean of two runs), 8.0, 1.9, 1.6, 2.5, 1.5, 2.2, 2.1, 3.6, 3.8, and 3.0 pounds of K_2O per acre per day. For the 6 soils of experiment 2 the range of values is more than tenfold, that is, from 0.88 for the Decatur to 10.3 for the Sable. Among the Caribou samples the range is only about twofold, from 1.5 to 3.8, but the qualitative order is about the same as for other criteria of release. During dialysis, an appreciable part of the potash fixed in the Caribou soils is released during the first few days (nonlinear section of the graph), while the subsequent linear rate appears more representative of the availability of the native reserves. The nonlinear sections of the release curves of Caribou samples E1558 and E1667 are equivalent to about 100 and 400 pounds of K_2O per acre, which agrees roughly with their superiority over E1548 in experiments 3, 5, and 7.

The samples dialyzed for 30 to 90 days were air-dried and analyzed for residual exchangeable potassium, with the following results for the soils in the order of listing in table 20: 59, 57, 48, 157, 131, 50, 25, 48, 31, 32, 26, 28, 67, and 48 pounds of K_2O per acre; values for E1648, E1552, and E1664 are means of two runs. The residual levels for many of the soils are almost as high as the minimum levels existing after prolonged intensive cropping. The finding of residual exchangeable potassium in substantial quantities was not anticipated. It has not been considered in the calculations of release. Later studies indicate that the effect of the air-drying on the dialyzed soils is similar to the effect on cropped samples.

The dialysis method can be considered an acid extraction, but one in which the hydrogen ion concentration in the intermicellar phase is low and in which the adsorbed hydrogen is the agent of release and decomposition. Jenny (*49*) reported that contact with exchangeable H, Na, and NH_4 ions of cation-exchange resins was much more effective in releasing nonexchangeable potassium from Ramona soil than was leaching with soluble acids and salts. In prolonged dialysis appreciable decomposition of the soil is observed, and a part of the liberated potassium must originate in the disintegration of the crystal structure of surfaces of particles. According to Gieseking (*36*) electrodialysis for several months eliminated the characteristic basal spacings for montmorillonite from a young loessial clay. It is not presumed that extensive similar decomposition occurs with the release of potassium during plant growth.

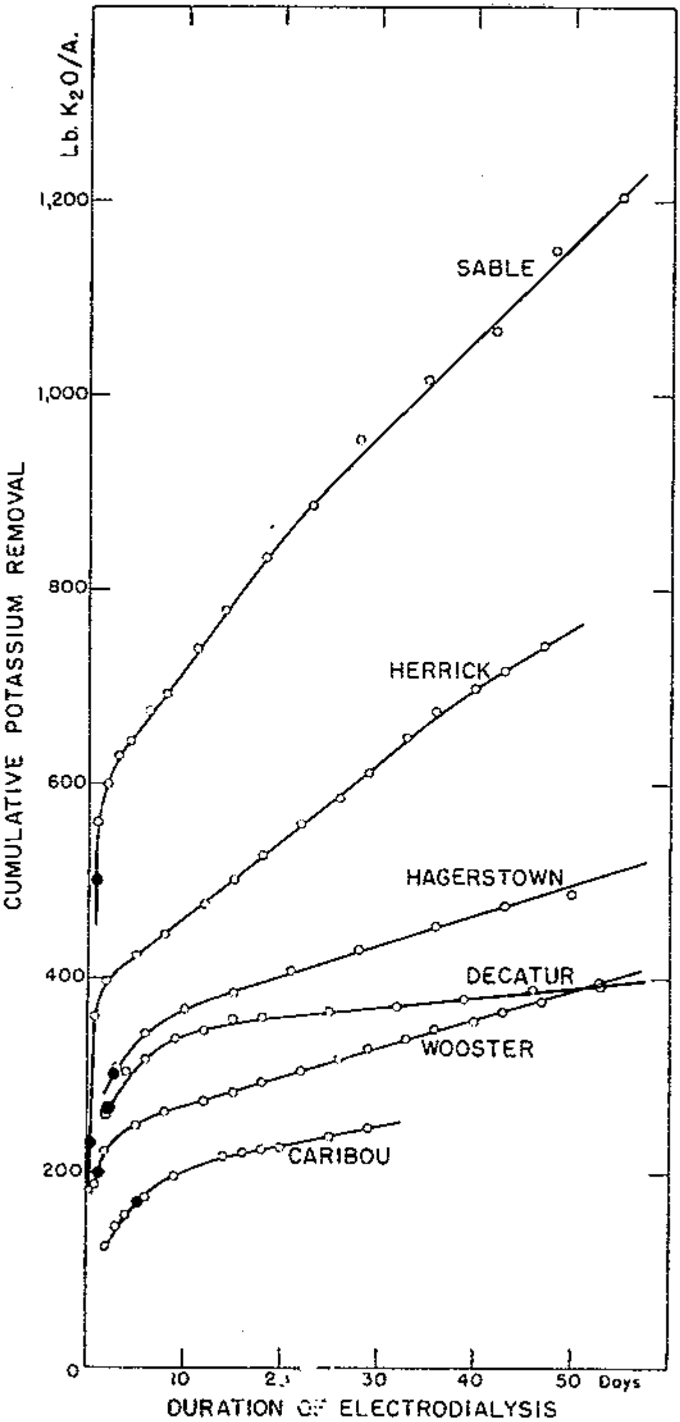

FIGURE 5.—Extraction of potassium from soils of greenhouse experiment 2 by prolonged electrodialysis (exchangeable potassium indicated by black dot).

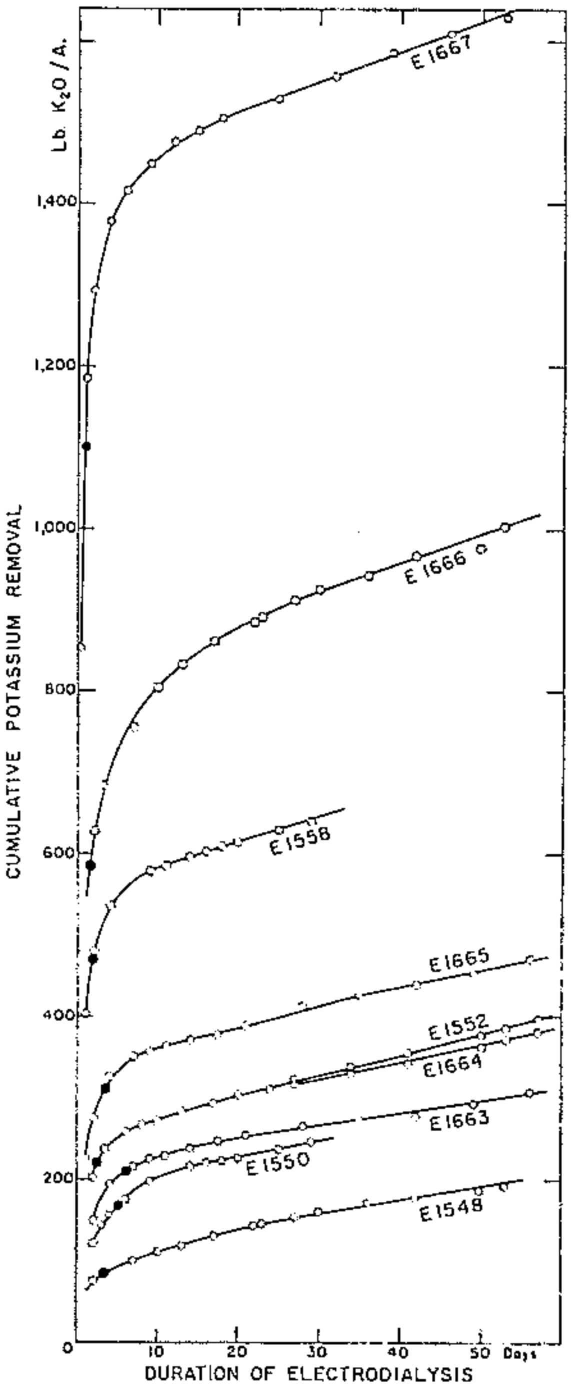

FIGURE 6.—Extraction of potassium from Caribou loam samples by prolonged electrodialysis (exchangeable potassium indicated by black dot).

Acid Digestion

Preliminary studies with strong mineral acids indicated that augmented release resulted from an increase in concentration, temperature, or time. The 14 soil samples were extracted under a variety of conditions, and the results of some procedures are presented in table 21.

In all the treatments, 10 gm. of soil was digested with 100 ml. of acid. The total extracted potassium, determined after filtration and washing of the soil, was corrected for exchangeable potash to obtain the values reported. During 2 days at room temperature, 1 N HNO_3 extracted relatively small amounts of nonexchangeable potassium from the soils. From the Decatur it actually extracted less than that exchangeable by ammonium acetate. The samples showing highest release by other methods liberated the most potassium, but the range was narrow.

Boiling HCl, H_2SO_4, and HNO_3 removed considerably more potassium than did the cold acid. Many differences in the results by the three acids appear significant on the basis of agreement of duplicates, but no systematic difference for the soils in general has been noted. In most cases the release values are much higher than those of experiments 2 and 3, but are comparatively close to the cumulative values of experiment 7. The correlation coefficient between the boiling HNO_3 values and release by Ladino clover in 740 days has been reported to be 0.938 (*72*).

TABLE 21.—*Release of nonexchangeable potash by various acid-digestion procedures, in pounds K_2O per acre* [1, 2]

Soil series	Accession No.	Boiling for 10 minutes in					48 hours at room temperature, 1 N HNO_3
		1 N HCl	1 N H_2SO_4	1 N HNO_3	5 N HNO_3	60 percent $HClO_4$	
Wooster	E1849	696	684	184	1,192	2,543	24
Hagerstown	E1870	484	788	516	808	2,292	20
Decatur	E1508	861	123	425	703	3,609	-17
Sable	E1848	1,590	1,996	1,228	1,804	3,212	162
Herrick	E1646	874	730	650	1,066	2,200	122
Caribou	E1550	424	290	344	770	3,032	40
Do	E1548	443	171	251	607	3,403	19
Do	E1552	436	340	484	816	4,132	56
Do	E1863	286	270	350	574	3,822	14
Do	E1604	380	478	348	608	3,308	12
Do	E1865	394	650	362	810	3,400	34
Do	F1866	552	552	520	824	3,004	98
Do	E1867	516	692	788	1,476	4,532	390
Do	E1558	458	362	522	998	3,914	74

[1] Exchangeable cations not removed previously.
[2] Mean of two replicates.

The use of boiling 5 N HNO_3 raised the values for most of the soils far beyond the magnitudes of release obtained by the cropping procedures. Boiling $HClO_4$ removed large fractions of the total potash contents of the soils, 5 to 35 percent, and thereby obliterated any distinctions among the soils that could be related to other indices of release.

Moist Storage and Freezing and Thawing

Descriptions of procedures involving (1) moist storage and (2) monthly freezing and thawing cycles during a period of 210 days, and the cumulative results, were given previously (*72*). Both the absolute release values and the precision of correlation with other methods were low. For 10 of 13 soils, release by storage equaled or exceeded that by freezing cycles. Although the Sable soil released the most potassium by the two methods, 152 and 109 pounds of K_2O per acre, respectively, the range for the remaining soils was very narrow, 41 to 100 pounds. No systematic relationship as to order of soils or relative effectiveness of the two methods has been found.

These soils were ammonium-saturated for the duration of the treatments. It is currently presumed that the low release values of most of the soils and the general lack of relationship to other methods resulted from the property of ammonium to be fixed and thereby to tend to block release of potassium.[11]

DISCUSSION

Previous investigations of this nature have compared soils of relatively narrow geographical distribution, usually from within State borders. The soil samples of the present study originated from six locations in five States in the eastern half of the United States. They are classified under five great soil groups, namely, Podzol, Gray-Brown Podzolic, Red Podzolic, Humic Gley, and Planosol soils. The parent materials from which they were derived include limestone, loess, and glacial till.

Within the group of samples of six soil types that had undergone low-potash treatments, the outstanding consistency of potassium-supplying capacities, when compared by a variety of methods, lies in the superiority of the Sable soil and the intermediate performance of the Herrick, both from Illinois. The remaining four soils are clustered at the lower end of the range, and their relative order with regard to the release of nonexchangeable potassium varies with the method.

In the Ladino clover, lespedeza, and ryegrass experiments, the Wooster was the highest of the four; in total release of potassium by electrodialysis for 30 days it was about equal to the Hagerstown and Decatur, but its constant rate of release was about four times that of the Decatur. By the Neubauer procedure the Hagerstown released less than the other three soils, and the results actually indicated a negative release. On the basis of acid-digestion values, no consistent order of release appears among the four soils. Relatively slight release from a sample of Decatur clay by continuous cropping, of about the same magnitude as for nine other Alabama soils, has been reported (*81*).

With respect to the extent to which they made available nonexchangeable potassium to plants in continuous-cropping procedures, the six soils can be divided into four levels, namely, (1) Sable, (2) Herrick, (3) Wooster, and (4) Caribou, Decatur, and Hagerstown. Field experiments on these soils at the sample sites, from which the response to potash can be gaged, afford general confirmation of this grouping. The Sable soil, it is reported, shows no response. The Herrick requires small potash applications for maximum crop production. The Wooster is said to be intermediate of Ohio soils in regard to potassium supply. Maximum production of potatoes on Caribou loam of low exchangeable potassium requires large additions of potash. The rela-

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[11] See footnote 4, p. 4.

tively low response on the Decatur soil is attributed to the low annual removal of potash in the harvested parts of crops. The Hagerstown soil shows moderate response to small applications of potash.

No explanatory relationship has been established between the general order of potassium-supplying capacity and other properties that have been determined, such as initial exchangeable potassium, total potash content of soil, clay, or silt, abundance of silt, clay, or organic matter, content of potassium-bearing minerals in the silt fraction, and hydrous mica content of the < 2-micron clay fraction. The Herrick and Sable soils have the highest montmorillonite content, cation-exchange capacity, and silt-plus-clay content. Only the Decatur has a relatively low total potash content.

The absence of any obvious relationship between the extent of potassium release and the content of hydrous mica or of potassium-bearing minerals of silt size does not preclude the assumption that the potassium behavior of these soils must be related to their mineralogical characteristics. The proper assessment of the role of these minerals must depend not only on their total abundance but on their present potash contents and stages of weathering or formation. Merwin[12] observed no relationship between release by several New York soils and the mineralogical composition of their silt fractions. The primary micas in soils may contain much less potassium than unweathered specimens (*24*), and the hydrous mica group comprises minerals of varying potash content and properties related thereto (*48*).

Soils having appreciable contents of potassium in both the silt and clay fractions probably release it by both silt weathering and release from clay lattices. The part of the soil that supplies the larger fraction of the potassium can be expected to differ between soils. Illinois workers (*19, 25*) appear to consider the clay-size fraction of soils of the State the mor important source. Of the two Illinois soils in this study, the younger Sable far surpasses the older Herrick in potassium availability, although little difference can be detected between their patterns of mineralogical and chemical composition; a higher content of potash feldspars is indicated for the Sable. Of two Mississippi soils, the greater release by Grenada soil, as compared to that by Savannah soil, was attributed to the occurrence of a substantial amount of potassium-bearing clay minerals of the 2:1 lattice type in the Grenada, and predominance of 1:1 type clays in the Savannah (*45*).

As the fixation mechanism involves clay minerals and release and fixation appear to be inverse processes, clays containing potassium would be expected to contribute to release. The potash availability of the samples within the Caribou group of differential fertilizer and organic matter treatments, as shown by the various methods, conclusively demonstrates the accumulation of fixed potassium in plots receiving excess applications. The mechanism whereby this soil fixes potassium has not been established. As it contains both montmorillonite and hydrous mica, it is possible that both minerals participate in fixation. The necessity of having soil dry enough that montmorillonite will thus function does not exclude this mineral, because surface layers may be desiccated, even by freezing.

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[12] See footnote 4, p. 4.

Some of the fixed potassium is highly available compared with the native potassium of this soil, as is shown by the rapid early release, especially from the straw and manured samples in cropping and electrodialysis procedures. In the prolonged growth experiments, the manured sample acted as a soil of potassium-supplying capacity between that of Sable and that of Herrick, whereas the low-potassium Caribou samples resembled Decatur and Hagerstown. Constant dialysis rates did not show such a great range, however, which may be due to exhaustion of fixed potassium before the constant-rate section is reached or to differing release mechanisms in cropping and dialysis.

No especial affinity of organic matter for potassium beyond that of the usual cation-exchange relationships has been established. Neither has an augmentation of release of mineral potassium by decomposing organic matter been acceptably demonstrated. The increased fixation and the consequent increased availability of nonexchangeable potassium in the organic-treated plots, therefore, are attributed solely to the addition of the potash contained in the organic materials.

It is inferred from the results of Attoe (*3*) and Lee[13] that all soils tested by them increased in exchangeable potassium by air-drying, or would have if the exchange level in the moist condition were sufficiently low. Most of the Hawaiian soil samples studied similarly by Ayres (*7*), however, did not change during air-drying. In the present investigation, after prolonged intensive cropping, only the two Illinois soils Herrick and Sable consistently increased appreciably in exchangeable potassium by air- and oven-drying. The Herrick sample is from the same experiment field as one sample of Lee's. Some soils apparently do not possess the minerals or the potassium status required for release by air-drying. This qualitative separation of soils does not extend to the other methods employed in this study, which almost always indicate release in some measurable amounts.

SUMMARY

Fourteen soil samples representing surface soil of six soil types from five States were subjected to chemical and mineralogical examinations and to measurements of their comparative abilities to release nonexchangeable potassium by a variety of methods. Wooster silt loam from Ohio, Hagerstown silt loam from Pennsylvania, Decatur clay loam from Alabama, Sable silty clay loam and Herrick silt loam from Illinois, and Caribou loam from Maine were represented by single samples that had received low-potash applications in field experiments. In addition, the group included eight other samples of Caribou loam that had received differential applications of potash and potassium-containing organic matter in the field.

All soils had large total contents of potash, about 1.5 to 2.5 percent, except Decatur, which contained 0.5 percent. This was also true of the clay and silt fractions of the soils. The exchangeable potash content ranged from 85 to 1,100 pounds per acre. In the group of Caribou soils the exchange level tended to increase with the extent of the previous potash applications.

The content of potash micas and potash feldspars in the silt fractions

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[13] See footnote 6, p. 5.

varied considerably. Only the Caribou was high in micas, with an estimated content of about 25 percent. The highest estimated feldspar contents were about 20 and 30 percent for the Sable and Hagerstown soils, respectively. It is emphasized that the potassium contents of these soil minerals may be substantially lower than those of unweathered specimens.

The clay mineral composition of the < 2-micron clay fraction was determined by a combination of methods, namely, X-ray diffraction spectrogram, differential thermal analysis, cation-exchange capacity, and potassium content. The hydrous mica content, disregarding the occurrence of quartz and amorphous materials, was estimated to range up to 90 percent, with only the Decatur containing less than 60 percent. The Sable and Herrick had the highest montmorillonite content, 30 percent, while none was indicated for the Decatur and Hagerstown. The Decatur clay contained 50 percent of kaolinite.

Twelve of the samples, including 7 Caribou treatments, were cropped by 15 cuttings of Ladino clover over a period of 2 years. In the soil-type comparison, the release of potash by Sable was outstanding, followed by Herrick, Wooster, and the remaining 3, in that order. In the Caribou experiment the manured soil sample released the most by far, followed next in order by the straw-treated soil and continuous-potatoes sample. Eight of these 12 cropped soils—4 Caribou samples were omitted—subsequently were successively cropped to 4 cuttings each of Kobe lespedeza and perennial ryegrass. The results were similar to those with clover, except that the rates of release were generally higher than in the clover experiments and that the potassium of the Caribou samples was comparatively highly available to ryegrass.

Other methods by which potash was released included air-drying and oven-drying, a Neubauer procedure, 30- to 90-day electrodialysis, various acid digestions, moist storage, and freezing and thawing. Important release during air-drying of cropped soil occurred only in the Sable and Herrick. The electrodialysis curves were similar in shape for all samples but differed in magnitude of release and in the constant rate of release. In general, all these release methods rated the soils in about the same order as the prolonged cropping technique. With regard to native potassium, the Herrick and Sable soils had much higher supplying capacities than the others. The prior fixation of field-applied potash in the manured Caribou had increased its potassium availabilty to a level between those of the Herrick and Sable.

Significant relationships have not been found between the comparative potassium-supplying capacities of these soils and the other properties that were determined in this study. Since differences between soils with respect to their availability of nonexchangeable potassium must lie in their mineralogical characteristics, it is concluded that the usual methods of estimating mineral contents must be supplemented by determination of the actual potassium content and behavior of minerals as they exist in soils.

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