

USE OF THE BRAY SOIL TEST IN FORESTRY.

2. DETERMINATION OF CATION STATUS

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ABSTRACT

A comparison was made between amounts of exchangeable (extracted by $1_M \text{NH}_4\text{OAc}$, pH7) and Bray ($0.1_M \text{HCl} + 0.03_M \text{NH}_4\text{F}$) extractable K, Mg and Ca in the topsoils of 128 forest soils and their relationships to levels in the foliage of *Pinus radiata* growing on these soils.

Amounts of K, Mg and Ca removed by a 1-minute extraction with the Bray solution were quantitatively similar to and closely correlated with amounts extracted by NH_4OAc ($r = 0.914, 0.899$ and 0.762 respectively). Increasing the extraction period with the Bray solution to 30 minutes had negligible effects on amounts of K but increased the amounts of Mg and Ca extracted, particularly from less weathered soils.

Potassium extracted by all three methods was significantly correlated with foliar K but explained only 9 to 12% of its variation. Correlations between foliar Mg and Ca and amounts of these cations extracted by the three methods were non significant although significant relationships were found within a few specific soil groups. It was concluded that, until such time as soil tests are developed and calibrated which give a good indication of tree-available K, Mg and Ca, the Bray 2 (1-minute) extraction can be used just as effectively, and more rapidly, for characterising exchangeable cations in forest soils as the NH_4OAc extraction.

INTRODUCTION

The availability of cations in soils is usually estimated by determining their exchangeable fractions (Doll and Lucas, 1973). The standard method for extracting exchangeable cations is with neutral normal ammonium acetate (Blakemore, Searle and Daly, 1972). However, cations extracted by NH_4OAc have generally proved to be poorly correlated with cation levels in the foliage of conifers both in New Zealand (Ballard, Jackson and Will, 1971; Adams, 1973; Adams and Walker, 1975) and overseas (Leaf, 1968). Where alternative methods have been evaluated, better correlations with foliar K and Mg have been obtained using stronger extractants which give a measure of non-exchangeable reserves of these cations in soils (Adams, 1973; Leaf, 1968). Nevertheless in most forest soil investigations requiring soil characterisation, or monitoring the effects of trees or management practices on soil properties, cation status is still determined using the NH_4OAc method.

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The Bray 2 soil test method which uses 0.1M HCl + 0.03M NH₄F as an extractant is routinely used for determining the P status of forest soils in New Zealand (Ballard, 1974). The Bray soil test has also been shown to be of value for predicting the P-retention capacity of forest soils using the levels of aluminium in the extract (Ballard, 1978). Since most soils coming into this laboratory are being tested by the Bray 2 method for one purpose or another, it would save considerable time and effort if the cation status of these soils could also be determined using cation levels in the same Bray extract. Several studies have indicated that levels of both K and Mg in Bray and NH₄OAc extracts are often closely correlated (Doll and Lucas, 1973; Mehlich, 1974; Ballard and Pritchett, 1975). In the Southeastern U.S.A., Ballard and Pritchett (1975) also observed that levels of K and Mg extracted by the Bray 2 method were more closely correlated with levels of these cations in the foliage of *P. elliotii* than those extracted by NH₄OAc.

This paper reports a study designed to examine the inter-relationships between Bray and NH₄OAc extractable cations and foliar cation levels in radiata pine for a wide range of forest soils in New Zealand.

MATERIALS AND METHODS

Soil and Foliage Samples

Soil and foliage samples were those collected from 128 plots (0.04 ha) established in 10- to 20-year-old *P. radiata* stands by Dr D. S. Jackson during the course of a nationwide study of environmental variables influencing growth of radiata pine. The distribution of these plots throughout New Zealand is shown in Jackson and Gifford's Fig. 2 (1974).

The soil sample from each plot was a composite of seven random sub-samples collected from the 0 to 7.5-cm depth of mineral soil. All soil samples were air-dried and ground to pass a 2-mm sieve prior to chemical analysis. For interpretative purposes soils were assigned to groups using the New Zealand genetic classification (N.Z. Soil Bureau, 1968a). There were 11 recent soils (Recent), 14 yellow-brown sands (YBS), 5 brown-grey earths (BGE) 12 yellow-grey earths (YGE), 23 yellow-grey earth/yellow-brown earth intergrades (YG/YBE), 14 southern yellow-brown earths (SYBE), 26 central and northern yellow-brown earths (YBE), 6 podzolised northern yellow-brown earths and podzols (PYBE), 7 yellow-brown pumice soils (YBP) and 10 yellow-brown loams, brown loams and brown-granular loams (all denoted BL). These same soil samples were used in a study relating Bray extractable Al to P retention reported in a companion paper (Ballard, 1978).

Foliage samples were collected from each of the seven tallest dominant trees in each plot. One-year-old needles were collected from secondary branches in the mid-crown. Because of the magnitude of the collection task, it was not possible to collect the foliage samples at any one specific time of the year and sampling was done over the entire spring-summer period. All foliage samples were oven-dried at 70°C and finely ground prior to chemical analysis.

Analytical Methods

Exchangeable cations were extracted by leaching 5-g soil samples with 230 ml of 1.0M NH₄OAc at pH 7.0 (Blakemore, *et al.*, 1972). Bray extractable cations were

obtained by shaking 2.5-g soil samples with 25 ml of Bray solution (0.1M HCl + 0.03M NH₄F) for 1-minute (Bray 2) and 30-minute (Bray 3) extraction periods. Potassium, Mg and Ca in the NH₄OAc and Bray extracts were determined by atomic absorption spectrophotometry. While strontium chloride was used as the releasing agent in the NH₄OAc extracts, preliminary studies showed lanthanum chloride was more effective in the Bray extracts. However, even lanthanum chloride did not completely override the interference of P in the K determination and a correction was made for the amount of P in the Bray extracts (Nicholson, 1977).

Other soil properties were determined by standard New Zealand Soil Bureau procedures (Blakemore, *et al.*, 1972).

Foliage samples were dry-ashed at 480°C for 4 hours and the ash dissolved in HCl. Cations in the acid solution were determined by atomic absorption spectrophotometry using strontium as a releasing agent.

RESULTS AND DISCUSSION

Potassium

Mean foliar K values for each soil group (Table 1) are all well above the critical level (0.40%) for optimal growth of radiata pine. The lowest value recorded in an individual plot was 0.4% which is consistent with the general lack of response to K fertilisers in New Zealand forests (Mead and Gadgil, 1978).

The mean amounts of K extracted from the various soil groups by the NH₄OAc and both Bray methods are very similar. In all cases, except for the YBS and BL groups, the amounts extracted by the three methods from soils within groups are also closely correlated (Table 1). Amounts of NH₄OAc and Bray extractable K are also closely correlated when all soils are grouped together. Over all soils the relationships between NH₄OAc extractable K and amounts extracted by the two Bray methods are very similar (Fig. 1). Below 0.15 me/100 g of NH₄OAc extractable K, the Bray methods tend to extract slightly more K than NH₄OAc, but above this level the trend is reversed. The similarity in mean amounts extracted and the close correlations between amounts extracted from individual soils suggests that, for the majority of the soils used in this study, the Bray methods extract essentially the same fraction of K as NH₄OAc. It is only for soils in the YBS and BL groups that this is apparently not true and in this regard it is interesting that only in the BL group did increasing the extraction period with the Bray solution markedly increase the amounts of K extracted and to the point where much more was extracted than by NH₄OAc (Table 1).

There are highly significant correlations between foliar K concentrations and K extracted by all three methods over all soils (Table 1). However despite the significance of the correlations extractable soil K values account for only 9 to 12% of the variation in foliar K concentrations. Thus at best these soil tests could only be used to give a very coarse indication of foliar K levels, and so presumably K status of radiata pine stands. Within soil groups there are generally very poor relationships between foliar K and extractable soil K. Only within the SYBE group are all correlations significant while within YBS group correlations between foliar K and Bray extractable K are significant. Thus stratification of soils into the broad units of the New Zealand genetic classification

TABLE 1—Mean foliage and extractable soil potassium values by soil groups and simple linear correlations between the variables

Soil Group	No.	Foliage (%)	Potassium			Correlation Coefficients				
			NH ₄ OAc	Bray 2	Bray 3	Fol/NH ₄	Fol/Bray 2	Fol/Bray 3	NH ₄ /Bray 2	NH ₄ /Bray 3
			— — — (me/100 g) — — —							
Recent	11	0.701	0.17	0.17	0.19	0.263	0.200	-0.170	0.817**	0.727**
YBS	14	0.641	0.15	0.13	0.16	0.276	0.592*	0.757**	0.082	0.283
BGE	5	0.608	0.24	0.27	0.31	-0.241	-0.196	-0.418	0.975**	0.982**
YGE	12	0.778	0.54	0.45	0.46	-0.133	-0.353	-0.163	0.843**	0.837**
YG/YBE	23	0.653	0.28	0.22	0.24	-0.122	-0.069	-0.310	0.959**	0.948**
SYBE	14	0.859	0.40	0.31	0.34	0.665**	0.594*	0.530*	0.979**	0.966**
YBE	26	0.739	0.33	0.24	0.28	0.191	0.142	0.158	0.956**	0.910**
PYBE	6	0.701	0.26	0.17	0.20	0.684	0.494	0.565	0.944**	0.869*
YBP	7	0.846	0.35	0.30	0.39	0.444	0.357	0.335	0.976**	0.836**
BL	10	0.758	0.27	0.26	0.37	-0.165	0.207	0.100	0.325	0.037

All	128	0.727	0.30	0.25	0.29	0.340**	0.312**	0.297**	0.914**	0.839**

* Significant at the 5% level

** Significant at the 1% level

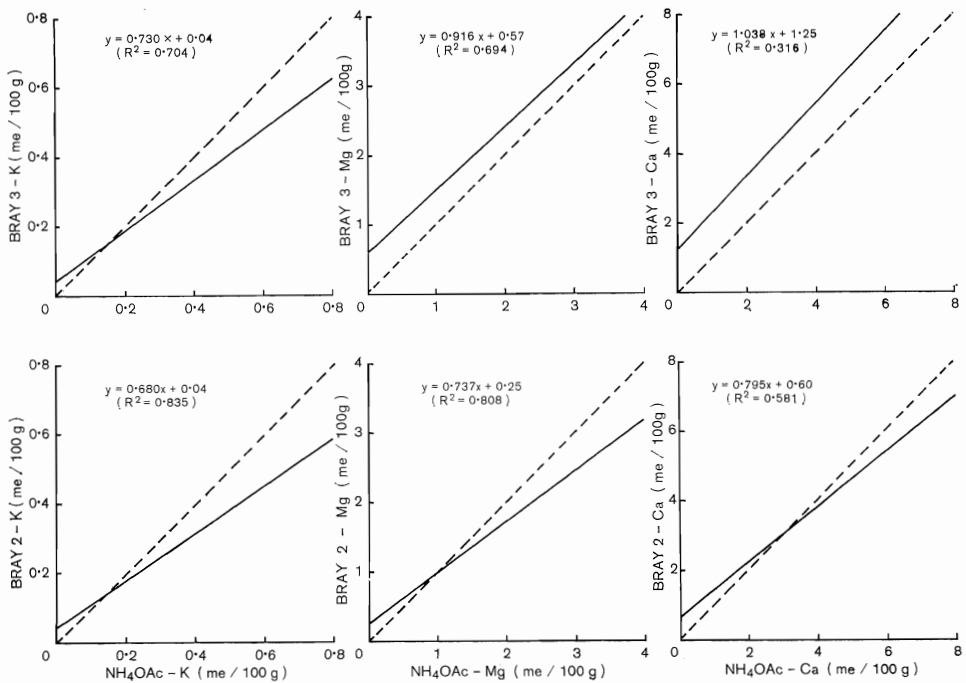


FIG. 1—Relationships between soil cations extracted by three methods. Dotted lines indicate equivalent extraction.

system apparently offers little chance of improved prediction of K status using these soil tests. It is noteworthy that for one of the groups (YBS) within which the NH₄OAc and Bray methods apparently extract different forms of K, foliar K levels are significantly related to the Bray and not NH₄OAc extractable K.

There may be several explanations for the generally weak relationships between foliar K and extractable K. (1) Close correlations between soil and foliar nutrient levels are most likely to be obtained when using sites which range from deficient to sufficient in the nutrient in question and all other soil and site factors are non-limiting to growth and fairly constant (van den Driessche, 1974). According to the foliar analysis results none of the sites used in this study are K deficient. Furthermore there is a wide range in productivity between sites associated mainly with differences in amounts and distribution of rainfall, effective soil depth, soil N and P and seasonal departures of ambient temperature from postulated optima (Jackson and Gifford, 1974). (2) Foliage samples were collected throughout the growing season and since K in radiata pine foliage shows marked seasonal variation (Mead and Will, 1976), this would act to mask the relationship between soil and foliar K. Additionally there is no strong evidence to indicate that K concentrations in the age class of needles in the crown position sampled give the most sensitive indication of the K status of the tree. In *P. resinosa* there is some evidence that the K concentrations in buds provides a better index of K status than that in foliage (Marion, Berglund and Leaf, 1968). (3) The soil tests used do not extract the fraction of soil K utilised by trees. Nearly all comparisons of soil testing procedures

on K deficient forest soils have shown that strong extractants, which remove the potentially available fraction as well as exchangeable fraction of soil K, give a better separation of K-deficient and non-deficient soils than extractants which remove only soluble and exchangeable K (Leaf, 1968; Phu, 1975). Data from the current study indicates that for most soils the Bray method extracts the same exchangeable K fraction that NH_4OAc does. (4) Concentrations of extractable K in the topsoil are unlikely to adequately reflect the supply available to deep-rooted tree species unless there is a fairly constant relationship between topsoil and subsoil supplies and the depth of exploitable soil is similar for the soils being examined. Other studies relating soil and foliar K have shown the relationship improves if the soil K is expressed as the amount within the rooting volume (White and Leaf, 1964; Ballard and Pritchett, 1975). In a somewhat similar range of New Zealand soils Metson (N.Z. Soil Bureau, 1968b) showed no constant relationship between topsoil and subsoil levels of exchangeable or reserve K. Also Jackson and Gifford (1974) reported considerable variation in the effective soil depth at the 128 sites from which the soils for this present study were collected: this soil variable was important in accounting for variation in the productivity of the radiata pine stands growing on the sites.

Improved predictions from soil test results have sometimes been achieved by expressing the amount of extracted cation as a ratio to other extracted cations or as a per cent of the CEC (Doll and Lucas, 1973). However expressing NH_4OAc extractable K in this manner reduced the level of correlation with foliar K (Table 2). Both CEC and pH are significantly correlated with foliar K but neither as closely as NH_4OAc or Bray extractable K (Tables 1 and 2).

Magnesium

Mean foliar Mg values for each soil group are well above Will's (1966) proposed critical range of 0.06 to 0.08% (Table 3). The only soil groups with individual observations in or below this critical range were the SYBE and YBP soil groups. Adams

TABLE 2—Simple correlations between foliar cations and soil variables

Soil variable	Foliar K	Foliar Mg	Foliar Ca
NH_4OAc K	0.340**		
" Mg		0.015	
" Ca			0.007
NH_4OAc K/Ca+Mg	0.267**		
" Mg/K+Ca		0.018	
" Ca/Mg+K			0.036
NH_4OAc K/CEC	-0.089		
" Mg/CEC		0.135	
" Ca/CEC			-0.012
CEC	0.288**	-0.222*	-0.042
pH	-0.181*	0.063	-0.012

* Significant at the 5% level

** Significant at the 1% level

TABLE 3—Mean foliage and extractable soil magnesium values by soil groups and simple linear correlations between the variables

Soil Group	No.	Foliage (%)	Magnesium			Correlation Coefficients				
			NH ₄ OAc	Bray 2	Bray 3	Fol/NH ₄	Fol/Bray 2	Fol/Bray 3	NH ₄ /Bray 2	NH ₄ /Bray 3
			— — — (me/100 g) — — —							
Recent	11	0.175	0.86	0.95	1.68	—0.453	—0.347	—0.027	0.835**	0.727**
YBS	14	0.186	1.24	1.37	2.20	—0.688**	—0.537*	—0.247	0.853**	0.283
BGE	5	0.190	1.04	0.62	1.30	—0.597	0.457	0.597	0.089	0.982**
YGE	12	0.139	1.98	1.85	2.48	—0.263	—0.327	—0.253	0.971**	0.837**
YG/YBE	23	0.156	1.47	1.25	1.77	0.352	0.365	0.402	0.990**	0.947**
SYBE	14	0.129	1.09	0.97	1.45	0.355	0.477	0.552*	0.975**	0.966**
YBE	26	0.133	1.40	1.23	1.69	—0.044	—0.022	—0.077	0.987**	0.910**
PYBE	6	0.153	1.22	1.11	1.39	—0.734	—0.707	—0.767*	0.993**	0.869*
YBP	7	0.103	0.90	1.09	1.35	0.718*	0.307	0.196	0.556	0.836**
BL	10	0.132	0.96	1.00	1.52	0.090	0.033	0.254	0.873**	0.037
All	128	0.155	1.29	1.20	1.75	0.015	0.029	0.096	0.899**	0.839**

* Significant at the 5% level

** Significant at the 1% level

(1973) has reported Mg deficiency in radiata on a SYBE soil and Will (1966) has reported the same on YBP soils. For all the other soil groups the ranges in foliar Mg were fairly similar, being of the order of 0.10 to 0.21%.

The mean NH_4OAc extractable Mg (exchangeable) values for the various soil groups are within the range reported for these groups by Metson and Brooks (1975). Mean amounts of Mg extracted from the soil groups by the NH_4OAc and Bray 2 methods are quantitatively very similar except for the BGE group in which the Bray 2 extracts markedly less (Table 3). As with K, at low levels of extractable Mg the Bray method tends to extract slightly more Mg than NH_4OAc with the reverse being true above about 1 me/100 g NH_4OAc extractable Mg (Fig. 1). However as opposed to K, which was little affected by increasing the extraction period with the Bray solution, the increase in extraction period increased the removal of Mg quite noticeably over the whole range of soils (Fig. 1). The mean amount extracted from each group by the Bray 3 method is consistently greater than that extracted by both the Bray 2 and NH_4OAc methods. The biggest differences between the Bray 2 and Bray 3 extractions occur in the weakly weathered groups (Recent, BGE, YBS, YGE) which probably reflects the greater amounts of Mg-bearing primary minerals in these relatively unweathered soils (Metson and Brooks, 1975).

Over all soils, levels of Mg extracted by NH_4OAc are closely correlated with those extracted by both Bray methods, with the correlation being slightly higher with the Bray 2 method which extracted quantitatively more similar amounts (Table 3). Within most soil groups the same close relationships exist, with the Bray 2 method again correlating most closely with the NH_4OAc method in all groups except BGE and YBP. Within the BGE group the mean amount extracted by the Bray 3 method is closer to that for NH_4OAc than is that for the Bray 2 method. Over the range of soils used in this study the Bray 2 method could be used just as effectively as the NH_4OAc method to give a measure of exchangeable Mg.

Within soil groups there are few significant correlations between foliar Mg and soil Mg extracted by the three methods (Table 3). Of those that are significant those within the YBS and PYBE groups are negative. The explanation for this is unclear, but within the PYBE group it may be associated with variable nature of the cation exchange sites within this group. The six soils in this group ranged from podzolised clays, in which clays probably contribute most of the cation exchange sites, to sand podzols in which organic matter undoubtedly contributes most of the cation exchange sites in the topsoil. Organic exchange sites do not show the marked specificity for divalent ions that inorganic sites do (Wiklander, 1964) and thus the lower levels of exchangeable Mg in the podzols are probably more available than the higher levels in the podzolised clays. It is noteworthy that the only significant positive correlations between foliar Mg and extractable soil Mg occurs within the two groups (SYBE and YBP) which include Mg deficient sites. The reason for the NH_4OAc method giving the best prediction within the YBP group and the Bray 3 method the best prediction within the SYBE group is not clear.

Correlations between foliar Mg and soil Mg over all soils are non significant for all 3 methods (Table 3). Expressing exchangeable Mg as a ratio to other cations or as a per cent of CEC does not significantly improve the correlation although the magnitude

of the correlation coefficient does increase with the CEC correction (Table 2). CEC itself is the only variable examined which is significantly correlated with foliar Mg over all soils. The negative correlation is a reflection of either the increasing strength of retention of the divalent Mg with increasing CEC (Wiklander, 1964) or an association of low CEC with weakly weathered soils which have higher levels of reserve (tree available?) Mg (Metson and Brooks, 1975). However none of the relationships observed, either within or over all soil groups, could be used satisfactorily as the basis for predicting the Mg status of radiata pine sites. The use of stronger extractants which remove reserve forms of Mg, such as used by Adams (1973), will obviously have to be looked at for this purpose. However, until such time as methods using stronger extractants have been assessed and calibrated over a range of soil conditions the Bray 2 method can be used as effectively as the NH_4OAc method to characterise absolute or relative levels of exchangeable Mg in New Zealand forest soils.

Calcium

Calcium deficiency has not been definitely identified in radiata pine in New Zealand but it is suspected to occur in conjunction with severe phosphorus deficiency on strongly weathered clay soils in the North of the North Island (Will, 1978). During selection of the sites used in this study Jackson and Gifford (1974) deliberately excluded sites which showed overt P-deficiency symptoms, which shows in the foliar Ca results (Table 4): mean values for the soil groups are well above the rather ill-defined critical Ca level of 0.10% (Flinn, 1975) and the lowest level recorded for an individual site was 0.11%.

Differences between amounts of Ca extracted by the three methods are similar to those observed for Mg (Fig. 1, Table 4). Mean amounts extracted by the NH_4OAc and Bray 2 methods within and over all soil groups are similar. Increasing the extraction period with the Bray solution markedly increases the amount of Ca removed from all soil groups with the effect being greatest for the less weathered soils (Recent, BGE and YBS) and soils formed from the more basic volcanic material (BL).

Within most soil groups the correlations between NH_4OAc and Bray 2 extractable Ca are highly significant. Within these soil groups from which the Bray 3 method extracts considerably more Ca than the Bray 2 method the correlations between NH_4OAc and Bray 3 extractable Ca are clearly lower than those between NH_4OAc and Bray 2 extractable Ca. This poorer correlation also shows for the overall relationship (Table 4). This indicates that the extra amounts removed by the longer extraction (from dissolution of primary minerals) are unrelated to levels of exchangeable Ca for this range of soils.

Correlations between foliar Ca and soil Ca extracted by any of the methods are poor both within soil groups and over all soils (Table 4). The possible reasons for the poor relationships are undoubtedly the same as those discussed under potassium. Other soil properties and expressions of exchangeable Ca are also essentially unrelated to foliar Ca (Table 2). However as pines are acidiphiles, Ca in forest soils is seldom determined for the purpose of providing an indication of levels of tree-available Ca, but rather because it is commonly the dominant exchangeable cation in soils and as such has a strong influence on many other soil properties and soil reactions. When being determined for this purpose the Bray 2 method could be used just as effectively as the NH_4OAc method for extracting exchangeable Ca.

TABLE 4—Mean foliage and extractable soil calcium values by soil groups and simple linear correlations between the variables

Soil Group	No.	Foliage (%)	Calcium			Correlation Coefficients				
			NH ₄ OAc	Bray 2	Bray 3	Fol/NH ₄	Fol/Bray 2	Fol/Bray 3	NH ₄ /Bray 2	NH ₄ /Bray 3
			— — — (me/100 g) — — —							
Recent	11	0.216	2.45	3.30	7.54	-0.123	-0.167	-0.087	0.959**	0.692*
YBS	14	0.266	2.41	2.94	4.19	0.079	-0.356	-0.249	0.267	0.291
BGE	5	0.208	1.62	1.80	4.31	0.832*	0.779	-0.401	0.983**	-0.038
YGE	12	0.229	3.52	3.45	4.44	-0.165	-0.347	-0.442	0.909**	0.787**
YG/YBE	23	0.328	2.14	1.82	2.32	0.375	0.286	0.179	0.971**	0.942**
SYBE	14	0.227	1.95	1.71	2.37	-0.291	-0.431	-0.484	0.925**	0.863**
YBE	26	0.225	2.53	2.32	3.16	-0.115	-0.052	-0.106	0.961**	0.964**
PYBE	6	0.245	2.20	2.15	3.06	0.679	0.741	0.713	0.983**	0.992**
YBP	7	0.217	2.96	3.90	4.59	0.466	0.511	0.534	0.747*	0.801*
BL	10	0.221	2.53	3.10	4.92	-0.165	-0.164	-0.091	0.757**	0.364
All	128	0.247	2.44	2.54	3.79	0.007	-0.123	-0.163	0.762**	0.561**

* Significant at the 5% level

** Significant at the 1% level

DISCUSSION

There is considerable evidence in the literature indicating that tree crops are capable of utilising non-exchangeable as well as the exchangeable and water soluble forms of soil K and Mg. The fraction of these non-exchangeable forms available to trees has been effectively estimated by using strong acid extractants (Leaf, 1968; Adams, 1973). It had been hoped that the Bray extraction solution, which contains both an acid (HCl) and a strong complexing agent (F^-) capable of causing dissolution of primary and secondary minerals, would extract the non-exchangeable forms, or a fraction related to them, which are available to trees.

Evidence in this paper indicates that extraction of soils for 30 minutes with the Bray solution apparently resulted in removal of some non-exchangeable Mg and Ca, but not K. However amounts of Mg and Ca removed by the Bray solution using a 30-minute extraction period did not significantly improve on the poor correlations obtained between foliar levels of these two cations and exchangeable (NH_4OAc extractable) soil levels. There appear to be two basic reasons for this result. (1) The non-exchangeable forms extracted are unrelated to those available to trees. The actual amounts of non-exchangeable Mg and Ca removed from the soils are very small (both HCl and F^- are very dilute in the Bray solution); on average less than a twofold increase over exchangeable levels. The successful acid extractants used by Leaf (1968) and Adams (1973) extracted on average 10 times as much Mg as was present in exchangeable form, from soils most of which contained exchangeable Mg in the same range as the soils dealt with here. (2) The range of soil conditions and the foliage sampling procedure used in this study were not conducive to an effective comparison of the suitability of different extraction techniques for indicating levels of tree-available nutrients (see earlier discussion).

The Bray 2 solution with an extraction period of 1 minute is currently used for determining levels of tree-available P in New Zealand forest soils (Ballard, 1974) and it has also been shown that levels of aluminium in the same extract can be used to give a good indication of the soil's P-retention capacity (Ballard, 1978). The current study has shown that this method also removes very similar amounts of K, Mg and Ca to the NH_4OAc method which is traditionally used to determine levels of exchangeable cations in soils. Thus it is possible to determine available P, P-retention capacity and exchangeable cations using a single, rapidly obtained soil-extract. This time-saving procedure should only be used until such time as techniques have been developed and calibrated which give a good indication of soil cation status with respect to tree growth over a wide range of soil conditions.

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