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"A Study of the Variation in Trace Element Content of Hill Pastures."

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CONTENTS

CHAPTER I

INTRODUCTION

For many years it has been recognised that a certain limited number of chemical elements are essential for plant and animal nutrition. In the last thirty years in particular, considerable attention has been directed by agronomists to investigations dealing with the mineral requirements of plants and animals, the disease conditions associated with deficiencies, and in some cases, excesses of particular elements, and the remedial measures required for the control of these diseases.

Apart from the elements carbon, hydrogen, oxygen and nitrogen, which together with phosphorus and sulphur form the organic constituents of living plant and animal tissues, it has been known that the mineral elements calcium, potassium, magnesium and iron are likewise essential for normal nutrition, while in addition the animal requires sodium and chlorine. This has been shown by the invariable presence of these elements in the living organisms, and by culture experiments, which have demonstrated that without them, normal development cannot take place.

It was further known that certain other mineral elements were present in plant and animal tissues, but in very minute quantities of the order of several parts per million. Initially, however, their occurrence was regarded as accidental. Animals depend for their food on plants, which in turn derive/

derive their nourishment from the soil. This food is taken up by the plant roots in the form of a solution, and it was considered reasonable to suppose that in addition to absorbing the essential mineral elements calcium, potassium, magnesium, iron, sodium and chlorine, the plant simultaneously absorbed smaller amounts of these other minerals, which happened to be in the soil. They were, however, present in such small amounts, and did not apparently have any fundamental role in the metabolic processes, that they were ignored.

But comparatively recently, considerable advances in analytical techniques together with more intensive studies of the factors responsible for normal growth, have established certain disorders of plants and animals, as diseases due to deficiencies, and in some cases excesses, of one or more of these particular elements. These occur in minute amounts or traces and are accordingly termed trace elements.

These trace element deficiencies or excesses may affect the health of the plant, or the animal consuming the plant, or both. For example, boron is an element, shortage of which in the soil creates disease conditions in the plant, without producing an effect in the stock grazing thereon. On the other hand, cobalt would appear not to be essential for plant growth and development, but if present in inadequate amounts in pastures, is associated with pining in stock. In comparison, there are one or two elements e.g. molybdenum, selenium, which can cause disease in the animal, if present in the soil or growth medium in excessive amounts.

Many of these trace element deficiency diseases are so widespread as to be of the utmost economic importance, and as such, to warrant further investigations in relation to the elements concerned.

There are two main types of problem in connection with the study of trace element status in plants and animals, namely the pathological and the physiological. The pathological problem is concerned with the abnormal conditions produced in the plant or animal, resulting from deficiencies, and the methods to be adopted in correcting such conditions. The physiological problem concerns the function of the particular element in the living organism. With regard to the latter problem, recent research would appear to indicate that the function of some trace elements is catalytic in nature, and concerned with the regulation of the normal growth processes. There are many trace elements distributed throughout plant and animal tissue, however, which apparently have no particular function, in that no disorders have yet been found associated with deficiencies of these elements.

On the other hand, our knowledge of the pathological conditions caused by certain deficiencies is more advanced than our physiological knowledge, and certain well-recognised diseases have been attributed to particular trace element shortages.

In recent years, for example, collaborative work between the Macaulay Institute for Soil Research, Aberdeen, and the Animal Diseases Research Association, Edinburgh, has shown that/

that cobalt deficiency in soil and pasture is fairly widespread throughout Scotland, particularly in the Border uplands, where it is associated with the disease of "pining" in sheep. This disorder has been remedied by the application to the areas in question of cobalt in a suitable form e.g. cobaltised fertiliser, and this has considerably reduced the disease in lamb flocks, thereby effecting an annual saving of approximately £40,000.

It has, therefore, become highly desirable to carry out a survey of cobalt, and in general trace element content of upland pastures, in order to obtain a picture of trace element status throughout the upland regions of Scotland. Further, the story of any particular deficiency disease is usually one of the deterioration in the health of the animal over a long period, and, in the case of cobalt, there would appear to be a number of steps between the stage where there is not enough of the element to the stage where the deficiency is obvious. There may, therefore, be many areas where the condition is sub-clinical and as yet unrecognised. When these areas have been surveyed and preventive measures of control applied, greater carrying capacity of these areas for healthy stock will eventuate.

In connection with a survey of this type there are three essential requirements:-

(a) A method of analysis capable of assessing the trace element content of pastures and soil extracts.

(b)/

(b) A method of sampling pasture from hill areas, so that the results obtained are not only representative of the hillside or area chosen, but are accurate to within certain definite limits.

(c) Animal experiments to obtain (i) the necessary relationship between the trace element content of pasture and the incidence of disease, and (ii) the simultaneous accumulation of standard data providing ranges of normal contents with which the results obtained from abnormal samples may be compared.

With regard to (a) the spectrographic methods of analysis evolved at the Macaulay Institute for Soil Research, Aberdeen, details of which are given later in a separate chapter, have been accepted as satisfactory for the elements concerned.

Regarding (b) no tried sampling method is available and the problem then exists of the representative sampling of pastures from large tracts of hillside consisting of different plant associations growing on different soil types. Without an investigation of the errors involved in the sampling of hillside pastures, doubt must always be attached to the results obtained for particular samples. In this connection the following statement by Wood and Stratton (1910) is appropriate:-

> "The interpretation of experimental results of any kind requires considerable caution, and this is more than ordinarily necessary in the case of agricultural experiments./

> experiments. The chemist deals, for the most part, with chemical compounds, for which there are well-recognised criteria of purity. He can make use of absolute methods of analysis, and is usually free from worries about sampling. But the agriculturist must often use methods which are at the best only conventional: he deals with substances of variable composition such as soils, manures, and crops, where errors of sampling are always apt to occur: he must at times trust to weighings or measurements of animals whose individuality is even more marked than that of plants. Everyone who has carried out experiments in the field or in the farmyard, must be well aware that the result of a single experiment is very often entirely misleading."

A later statement by Yates and Cochran (1938) indicates further the difficulties encountered in the sampling of agricultural material:-

> "Agricultural experiments on the same factor or group of factors are usually carried out at a number of places and repeated over a number of years. There are two reasons for this. First, the effect of most factors (fertilisers, varieties, etc.) varies considerably from place to place and from year to year, owing to differences of soil, agronomic practices, climatic conditions and other variations in environment. Consequently the results obtained at a single place and in a single year, however accurate in themselves, are of limited utility either/

> either for the immediate practical end of determining the most profitable variety, level of manuring, etc., or for the more fundamental task of elucidating the underlying scientific laws.......
>
> Agronomic experiments are undertaken with two different aims in view, which may roughly be termed the technical and the scientific. Their aim may be regarded as scientific in so far as the elucidation of the underlying laws is attempted, and as technical in so far as empirical rules for the conduct of practical agriculture are sought. The two aims are, of course, not in any sense mutually exclusive, and the results of most well-conducted experiments on technique serve to add to the structure of general scientific law, or at least to indicate places where the existing structure is inadequate, while experiments on questions of a more fundamental type will themselves provide the foundation of further technical advances."

The Investigation, Its Nature and Scope.

The investigation described in this thesis is concerned with the question of the representative sampling of hill pastures for trace element analysis. Such an investigation involves a study of the normal variation in trace element composition of such pastures, and of the factors which may contribute to the variation and their effects, with the ultimate establishment of a method of sampling, so that it will/

will be possible to apply to the results obtained a reasonable estimate of error.

The application of this method in the survey contemplated will permit a more accurate assessment of the trace element status of hillsides, and further, the work which must follow, namely, the animal experiments given under (c) above, can then be satisfactorily undertaken.

A tract of hillside representative of the uplands of South-Eastern Scotland was therefore chosen. From this site 678 pasture samples and 455 soil samples were taken according to certain procedures. Analyses have been made of the trace elements manganese, cobalt, nickel and iron in the pasture herbage, and of acetic acid extracts of the soil for cobalt and nickel, and the results obtained have been examined statistically. The pasture was also analysed for calcium, potassium and magnesium, in order to compare the variations in trace element content with those in major element content and to obtain a picture of the major element content of such pastures.

CHAPTER II

THE TRACE ELEMENTS IN PLANT AND ANIMAL NUTRITION.

It is proposed in this chapter to discuss in some detail the trace elements in plant and animal nutrition, with regard to the general problems involved in a study of these elements, and to indicate, with appropriate references, some of the diseases found in plants and animals associated with particular trace element deficiencies.

In connection with the general study of trace elements, two main types of problem are involved, namely, (a) the demonstration of the essentiality of particular elements, and the part played by them in the plant and animal, and (b) methods of diagnosis and control of those diseases of plants and livestock associated with deficiencies of these elements.

With regard to the first problem, the essential nature of the trace elements was for many years overlooked on account of the difficulty of constructing diets for plants and animals entirely free from the elements. In the case of plant development these were introduced into the culture experiments partly from the seed from which the plant developed, and were further derived from impurities in the water and nutrient salts used in preparing the culture solution, and by solution from the containing vessels. There also existed the possibility of atmospheric contamination. The same general problem was presented in the study of animal nutrition, the main difficulty in obtaining proof of the essential nature of an element depending upon the/

the availability of suitably constructed animal foods free from the elements concerned. For example, in the original investigations in Australia into the condition of ruminants, known as "pining" (referred to later in this chapter), the crude iron compound administered to the sick animals was effective in preventing and curing the disease, which was accordingly associated with iron deficiency.

In order then to investigate the essentiality of a particular trace element, means had to be devised of preventing the introduction of impurities from all the possible contaminating sources. Even so-called pure salts sold as analytical reagents may and often do contain amounts of trace contaminants, sufficient in themselves to supply the needs of the plant and animal. It is therefore imperative to stress the need for the purification of all material used in this type of work, and although various diseases in plants and animals have been attributed to particular deficiencies, the possibility of interaction between two or more elements must not be excluded.

The problem of obtaining water and nutrient salts free from trace metal impurities for work on fungi nutrition has been studied by Steinberg (1919, 1935) among others. Steinberg's original method for obtaining a nutrient solution free from metal contaminants consisted in heating the complete nutrient solution with pure calcium carbonate under pressure, thereby increasing the alkalinity of the solution/

solution so that traces of heavy metal are precipitated as carbonate, hydroxide and phosphate and adsorbed on calcium salts precipitated in bulk. In this way traces of manganese, copper, zinc and iron were removed.

For work with higher plants, it is more difficult to purify the complete culture solution, owing to the larger quantities required. In the purification of water it is essential to avoid the use of metal parts in the distillation apparatus. Stout and Arnon (1939) have given an account of methods suitable for the purification of water and various salts used in the preparation of nutrient solutions.
A full account of the use of sand and water culture methods in the study of plant nutrition is given by Hewitt (1952).

In order to investigate the part played in plant and animal nutrition by trace elements it is essential that methods for their quantitative determination be available. The concentrations of these elements in plant and animal material are so low that the ordinary methods of chemical analysis are not sufficiently accurate.

During the last twenty-five years a number of physical instruments have been developed for the measurement of small quantities of material, and it is possible by means of these to estimate with sufficient accuracy most of the trace elements in plant and animal matter.

The instruments are the absorptiometer and spectrograph. The absorptiometer is a form of colorimeter, incorporating photo-electric cells. By means of these cells the depth of colour/

colour of a solution is matched against that of a standard solution, and thereby measured.

In the spectrograph, a small sample of the material to be analysed is placed in an electric arc, flame, or other suitable source of excitation in such a way that the molecules of the sample are dissociated into their constituent atoms, which are then stimulated to emit radiation. This radiation is limited to certain wavelengths which are characteristic of the elements in the substance and of the conditions of excitation used. The radiation is passed through a prism and recorded as spectrum lines on a photographic plate. Each line possesses a density or intensity of blackening which depends, within the limits of under-exposure and over-exposure of the plate, on the intensity of radiation of the particular wavelength, and the quantity of the element emitting that radiation. The spectrograph affords the most sensitive method of measuring small quantities of a large number of elements simultaneously, and provides a permanent record, so that results are readily available for future reference.

Stiles (1951) refers to the use of the polarograph for the determination of potassium, copper, manganese, aluminium, iron, zinc and barium.

In the analysis of trace elements in plants, soils and related materials, attention must be drawn to the need for freedom from contamination at all stages of the procedure. All reagents used in any chemical treatment preceding/

preceding the analysis must be purified, including those reagents used in making up standard mixtures for calibration purposes. (See next Chapter.)

The second problem in trace element studies generally is concerned with methods of diagnosis and control of plant and animal diseases associated with particular deficiencies. It is obvious that such methods are of immense economic importance.

There are three methods of diagnosis. In the first method deficiencies are recognised by specific symptoms, exhibited by the plant or animal. In the plant, foliage symptoms are usually displayed, as for example, characteristic grey spots in the lower half of the oat leaf associated with manganese deficiency. In the animal definite symptoms may be recognised, such as deformity of the leg bones in chicks fed on manganese deficient diets. Good colour photographs of symptoms displayed by plants suffering from various mineral deficiencies are given by Wallace (1951).

The second method by which deficiencies may be determined is provided by the method of analysis referred to earlier. The results obtained would provide evidence of the adequacy or otherwise of the quantity of various elements in the plant or animal, but at the present time such information is extremely limited. With regard to the plant, it would be necessary to establish the minimum quantities of the respective mineral elements in the different plant organs of each species at different stages of development. At the present/

present time there are few available data on the trace element contents of the different animal organs and mixed herbage analyses are taken as an index of trace element status in the animal: before such analyses can have any value, however, sampling errors, particularly on hill pastures, must be investigated.

A third method of diagnosis consists in introducing the element, in a suitable form, e.g. as a solution of the element, or as a soluble salt, into the plant or animal and observing the reaction. The principle underlying these methods is that the introduction of the element into the plant or animal in which the element is deficient will produce a response in the direction of a cure of the disease. These methods, in fact, while useful for diagnostic purposes, may also serve as methods of prevention and control of disease.

The plant is allowed to absorb the solution of the element through a cut surface or through an intact leaf. The usual response is a colour change in the leaf which becomes greener; this is often accompanied by an increased rate of growth.

The animal may be "drenched" with a suitable soluble salt of the element or the salt may be added to the diet and the reaction of the animal noted.

Methods of control in the plant involve:-

(a) the application to the soil of a soluble salt of the element, e.g. copper sulphate in the control of copper deficiency, or

(b)/

(b) spraying of the plants or crop, e.g. manganese sulphate spray for the correction of manganese deficiency in oats and sugar beet, or

(c) injection of a salt of the element into the plant, e.g. the injection of solid ferrous sulphate into the trunks of fruit trees affected by iron deficiency.

Effective measures of control in the animal are:-

(a) dosage with pills or tablets containing the appropriate element, e.g. worm tablets containing cobalt in the control of cobalt deficiency in ruminants,

(b) the use of licks and mixtures containing the necessary element, e.g. cobalt "licks" in the control of cobalt deficiency,

(c) the inclusion in the diet of a salt of the element concerned, e.g. the addition of manganese sulphate to the diet of chicks in order to prevent manganese deficiency,

(d) the spreading on the land of appropriate salts, e.g. the application of cobalt sulphate, in instances of "pining" disease of sheep, at the rate of 2 lb. of the salt per acre will raise the cobalt level of the pasture to a value suitable for carrying healthy stock.

Roach (1939) discusses, in detail, the different methods of injection of plants. Methods suitable for the control of "pining" disease in sheep and ruminants suffering from cobalt deficiency are given by Hopkirk and Patterson (undated, about 1950).

Reference/

Reference has already been made in the first chapter to the elements carbon, hydrogen, oxygen, nitrogen and sulphur, which form the organic constituents of plant and animal tissues, and in addition calcium, potassium, magnesium and iron. Further, the animal requires sodium and chlorine. Silicon is included among those elements which produce beneficial effects on the growth of certain plants, but have not been shewn so far to be absolutely essential to growth. Some plants, particularly cereals and grasses, grown in the absence of silicon, seem to be susceptible to attack by fungi. The general functions of these major elements in plant and animal nutrition are reasonably well understood. In the plant, carbon, hydrogen and oxygen are derived from the air and water and used to build up fats and carbohydrates; nitrogen, phosphorus and sulphur are obtained from the soil and combined with the first three to form proteins. The power of the plant to fix carbon lies in its green pigment chlorophyll which contains magnesium, while iron, although not a constituent of chlorophyll, is required for its formation. Both calcium and potassium fulfil important functions in the leaves and cell sap, and are recognised as essential elements.

In the animal body, carbon, hydrogen, oxygen and nitrogen are required combined in the form of fats or oils and proteins, which form the major part of the body tissues. It must also be noted that water is the most abundant substance in the animal body. Calcium and phosphorus are required/

required for skeleton formation and are necessary constituents of other tissues. Sulphur is found in the tissues, and is an essential part of sheep wool. Sodium and chlorine are required in relatively larger quantities. The exact role of sodium is not clear, although it is known to be essential to life, while chlorine is present in, and necessary for the formation of acid in gastric juice. Potassium, though it must be present, is not so important an element to the animal as it is to the plant. Its functions, which are not yet completely understood, are concerned with the regulations of muscular movements. Magnesium is required by the body in comparatively small amounts and is very important physiologically, being intimately associated with calcium metabolism. Iron exercises functions of fundamental importance; its association with anaemia and its concentration in the red corpuscles of the blood are well known.

The other mineral constituents or trace elements in plant and animal matter occur in minute quantities, and those which have been shown to be essential are listed below in a general classification of the mineral composition of plants and animals, including those elements which form the organic constituents. Iron occurs in the animal body in such amounts that it is not generally considered as a trace element; its action is bound up with them to such an extent, however, that it may be regarded in the following classification, as occupying an intermediate position.

Category	Source	Constituents	
MAJOR	Plant	C.H.O. and N.P.S. (organic) Ca.K.Mg. (Si)(inorganic)	↑
	Animal	C.H.O. and N.P.S. (organic) Ca.K.Mg. (Si) Na.Cl. (inorganic)	Fe.
TRACE	Plant	Mn.Cu.Zn.B. (Mo)(Se),(F),(Ni)	
	Animal	Mn.Cu.Zn.Co.I (Ni)	↓

In the above table, several elements have been put in brackets, e.g. silicon among the major elements, and molybdenum, selenium, fluorine and nickel among the trace elements. While silicon produces beneficial effects on the growth of certain plants, there is no evidence to shew that it is absolutely essential in either plant or animal nutrition. In the case of the trace elements molybdenum, fluorine and selenium, these occur in plants. Molybdenum has been shewn to be essential in plant nutrition, but there is no definite evidence for the elements fluorine and selenium. On the other hand, excesses of molybdenum and selenium in plants are associated with diseases of livestock feeding on them, while if fluorine be ingested by animals in amounts much larger than the usual traces it interferes seriously with bone metabolism. With regard to nickel, while it occurs in plants there is no evidence to shew that it is essential for either plant or animal; nevertheless comparatively recent research has suggested that nickel may be of some importance in increasing particular crop yields, and that it could partly replace cobalt in the treatment of cobalt deficiency disease.

In/

In the following general review of the essential trace constituents in plant and animal nutrition, the more important deficiency diseases attributable to particular trace element shortages are discussed, with particular reference to the elements investigated in this thesis, namely, manganese, cobalt, nickel and iron.

With regard to the major plant constituents calcium, potassium and magnesium which have also been examined, accounts of these elements in plants and animals are given by Corrie (1948) and in plants by Wallace (1951).

Manganese

It has been known since the second half of the nineteenth century that manganese was a common constituent of soils and plants. Its agricultural significance was not realised however until it was shewn that the well-known disease of oats called "Grey Speck" could be induced by depriving the plant of manganese, and controlled by the addition of manganese compounds to the growing crop. Two other well-recognised pathological conditions in plants are associated with manganese deficiency and are of sufficient importance economically to be designated by common names. These are "Speckled Yellows" of sugar beet and "Marsh Spot" of peas.

Manganese deficiency is not related to the amount of manganese in the soil but to the form in which it exists, and to the amount which can become available to the plant.

Among the hypotheses put forward to explain the circumstances are (i) the low availability and oxidised state of manganese under alkaline conditions, and (ii) the occurrence of bacteria which render manganese compounds insoluble under slightly acid or alkaline conditions, thereby reducing the resistance of the plant to micro-organisms.

With regard to the functions of manganese in the plant, Wallace (1951) writes "Manganese is regarded as having the functions of a catalyst, its activities being specially concerned with oxidation and reduction reactions within the plant tissues. Iron and manganese together may well be the main oxidation-reduction regulators in plants".

The disease of grey speck in oats, sometimes termed "grey spot" or "grey stripe", is characterised by the appearance in the leaves of small chlorotic areas, which gradually expand, becoming brown at the margins and grey in the centre, eventually coalescing to form brown streaks. The leaf often breaks due to the necrosis of the tissue in these "specks". Colour photographs of oats badly effected by grey speck are given by Wallace (1951, pls. 26-29). Badly affected plants may die early, whereas in less severe cases, flowers may be produced but little grain is obtained. Grey speck of oats occurs in different parts of Europe, as well as in Britain and America. The disease can be controlled by the application of a soluble manganese salt, in the form of a soil dressing, or by spraying the foliage. The proof that grey speck was related to manganese deficiency however/

however was supplied by Samuel and Piper (1928, 1929) from the results of their experiments with Algerian oats, which they grew in controlled water cultures. Different amounts of manganese sulphate were added to the culture solutions, which contained the usual nutrients. The concentrations of manganese in the cultures varied from 0 to 1 p.p.m. Cultures grown in manganese free solutions developed grey speck symptoms in about four weeks, and only in culture solutions containing 1 part or more of manganese in 10 million were the plants normal and no symptoms displayed. Gerretsen (1937) has however argued that it is necessary to discriminate between the actual physiological effect of manganese which is a retardation of growth, and the symptoms of grey speck disease which are related to the activity of micro-organisms. Somewhat similar symptoms are obtained for manganese deficient wheat, barley and rye (see Wallace, 1951, pls. 35, 15, 30 resp.).

The disease of sugar beet known as "Speckled Yellows" involves intravenal chlorosis of the leaves, (see Wallace, 1951, pl. 129). The name is derived from the general appearance of the plant, and the disease is remedied by the application of a soluble manganese salt. Smith, (1952, pl. 14b) records analyses of affected and unaffected sugar beet. In the former the manganese content of leaves was 7 p.p.m., as compared with 112 p.p.m. in unaffected leaves sampled at the same time.

The/

The disease of peas known as "Marsh Spot", a necrosis of seed cotyledons (see Wallace, 1951, pls. 203, 204) named from its occurrence on Romney Marsh, was described and investigated by Pethybridge (1934, 1936) who suggested a connection with manganese deficiency. Affected plants may appear quite healthy or show a chlorotic condition of the foliage. Separation of the two seed cotyledons reveals a small brown speck or large circular brown area on the flat surfaces. The association of this disease with manganese deficiency was confirmed by Piper (1941) by means of controlled water culture experiments.

With regard to animal nutrition, as a result of experimental work with rats and more recently with poultry, manganese has been established as an element essential to animal life. It has been demonstrated in laboratory experiments with rats that manganese starvation interferes with reproduction: however, most foods contain sufficient manganese so that deficiency conditions should not arise. On the other hand, the effect of overliming with consequent reduction of the availability of soil manganese to the plant must not be overlooked.

The effect of manganese deficiency in bringing about a disease of young chickens known as "perosis" or "slipped tendon" was first indicated by Wilgus, Norris and Heuser (1936).

This disease is characterised by a deformity of the

leg/

leg bones and to some extent of the wing bones. These bones, in the diseased fowl, are shorter than the normal. The relationship of manganese deficiency to perosis was confirmed by different groups of workers, including Gallup and Norris (1937). Heller and Penquite (1937) found that traces of manganese added to a perosis-inducing diet practically prevented the disease occurring. Lyons and Insko (1937) shewed that the embryos produced in eggs of hens fed on a manganese deficient diet were characterised by abnormally short legs and wings.

Cobalt

Cobalt is not known to be necessary for normal plant metabolism, but the plant acts as carrier of the elements to the animal for which it is essential.

The function of cobalt is closely associated with the formation of blood and with the bacterial flora of the ruminant stomach. It is required in the synthesis of vitamin B 12. Sheep and cattle with their essentially vegetable diet, apparently depend upon this synthesis for their animal protein factor which contains vitamin B 12, and without it they cannot thrive. One distinct feature of cobalt deficiency in the ruminant is a marked anaemia, and it is possible that the two factors operating are (i) interference with the bacterial flora and with the consequent synthesis of vitamin B 12 which is associated with the animal protein factor, and (ii) the effect of blood formation.

A/

A disease of sheep and cattle in which the animal looses condition and becomes emaciated, scours badly and shews a bloodlessness and running from the eyes, which is most characteristic, has been attributed to cobalt deficiency, and is known under a variety of names such as "Pine", "Vinquish" and "Daising" in Scotland; "Moor Sickness" and "Pining" in South West England; "Nakuruitus" in Kenya; "Morton Mains" disease, "Bush Sickness" and "Mairoa Dopiness" in New Zealand. Sometimes a deficiency of cobalt is accompanied by a deficiency of copper, as for example in "Coast Disease" in Australia, and "Salt Sick" in Florida, in the United States of America.

Pining disease was recorded in Scotland as long ago as 1831 by James Hogg, the Ettrick Shepherd. Generally the disease does not appear on newly broken land but only after several years sheep farming on the same land, and deep ploughing seems to lessen the disease for a few years. These facts in themselves suggest a deficiency of some element as the cause of the disease. In addition, affected stock recover quickly on being transferred to healthy country. The story of the discovery that cobalt is essential in animal nutrition is perhaps the most fascinating one in the whole field of trace element research. An excellent account of cobalt deficiency in relation to animal health is given by Hopkirk and Patterson (undated, about 1950).

In New Zealand at the beginning of the century "bush sickness" had been successfully treated by top dressing of the/

the pasture with mixtures containing iron compounds leading to the suggestion that the trouble was caused by a deficiency of iron. During this time Aston (1924 (a) and 1924 (b)) insisted that the disease was related to iron deficiency in the soil. He advised dosing with ferrous ammonium citrate and this procedure was found effective. Later he suggested the use of bog-iron (limonite) as a material for licks. This material was cheaper than the double salt and was found on application to be a useful preventive agent for "bush sickness". Later experiments, however, indicated that some limonite samples were not effective. Australian observers suspected that the curative batches of limonite contained some trace element not present in the other specimens. Filmer (1933) and Filmer and Underwood (1934) investigated a disease in Western Australia known as "Enzootic Marasmus" identical in appearance with "Bush Sickness" in New Zealand. They showed that iron was not the causative factor by feeding extracts of limonite from which the iron had been removed, and that the curative extract contained nickel, manganese, zinc and cobalt. They then tried the effect on stock of administering each element in turn. They were partially successful in their first attempt, using nickel, but later found that the nickel used was contaminated with cobalt. Marston (1938) and his collaborators in their investigation of "Coast Disease" found that administration of cobalt was successful, although they later found that copper was also required to give complete health.

Discussion/

Discussion between both sets of workers in West and South Australia established the beneficial effect of the feeding of small doses of cobalt in both areas. Sheep given 0.1 mg. and cattle 0.3 to 1.0 mg. cobalt daily were kept in good health. Askew and Dixon (1936) confirmed the findings of the Australian workers with regard to the New Zealand deficiency areas.

In Great Britain McGowan and Smith (1922) investigated pining on the slopes of the Cheviots and discussed the movement of affected stock to healthy pastures as a cure. Russell Greig, and others (1933) described the pining of sheep and cattle on the Island of Tiree in the Inner Hebrides and its remedy with iron oxide supplements.

Russell Greig, Dryerre, Corner and Smith (1938) found that ferrous ammonium citrate would cure the condition, while Corner and Smith (1938) following on the work of Filmer and Underwood (1937) shewed that Border Pine could be cured by the administration of 1 mg. of cobalt per day. Experience has also shewn that a top dressing of the order of 2 lb. of cobalt sulphate per acre is most successful. A description of the history and distribution of pining disease in Scotland with a discussion of the methods of control is given by Stewart (1946). The occurrence of pining in the North of England and its treatment is discussed by Lyle Stewart (1944). Many figures are available for the cobalt content of pastures from countries where cobalt deficiency affects grazing animals. Mitchell (1944-~~45~~) gives/

gives 0.03 to 0.6 p.p.m. for the cobalt content of pastures in north-eastern Scotland. The same authority (1945) writes: "It now appears that herbage contents of about 0.08 p.p.m. Co are necessary to ensure complete animal health. Below this there is a transition range from about 0.05 to 0.08 p.p.m. where trouble may or may not occur, depending possibly on factors such as amount eaten and its digestibility. Below 0.04 p.p.m. it is unlikely that a healthy permanent sheep stock can be carried. These values refer to samples taken during the spring and summer period, when the herbage is growing, and not to samples taken in autumn and winter". The figures given relate to dry matter basis. A report of the U.S. National Research Council (1945) deals with the nutrient requirements of beef cattle and, after reviewing the cobalt contents of healthy and "affected" pastures, concludes "Judging from these data, the minimum cobalt requirement is met by about 0.1 p.p.m. of cobalt in the dry matter of the feed, an intake of about 1 mg. daily by adult cattle".

Within recent years cobalt has been shown to be significant in the treatment of "Ketosis" or "Slow Fever" disease of cattle. The disease occurs in Sweden, Great Britain and Ireland. The disorder is quite definite in its symptoms and comes on two or three weeks after calving. It is characterised by a loss of appetite; a sweetish odour which is exhaled in the breath and in the milk of affected animals. The disease has an effect upon the milk yield of affected/

affected animals, depressing it, and making the milk unpalatable. O'Moore (1952) has discussed the successful treatment of cases of Ketosis in Ireland by means of cobalt drenching.

Another disease of cattle, which responds to cobalt medication as reported by Hunter (1952) is "malignant catarrh". This disease does not occur in any particular season, nor as an epidemic. Only two or three cases occur at one time, and the animals affected are usually 18 months of age and upwards. The disease rarely occurs in younger animals. In acute cases there may be fever and the animals are depressed. The membranes of the nose, mouth, eye and vagina are congested. There is a discharge from the nose of mucus which may become bloodstained and later dries around the orifice. The horns become hot and painful, and the muzzle is dry and hot. In extreme cases the animal may become excited, stagger about, and have convulsions. Urination is frequent and the faeces vary from normal to bloodstained diarrhoea. Rumination may stop, thirst is increased and the milk yield falls. Some cases have responded to the administration of cobalt with phenothiazine. 2 fluid ounces of a weak solution of cobalt sulphate is given twice daily for two or three days until cudding recommences.

Nickel

Nickel is not yet included in the list of elements essential to plant or animal life, and any importance attached to it is due rather to its poisonous effects on crops./

crops.

Mitchell (1945) has given data for the acetic-acid soluble nickel from soils in Aberdeenshire. Cereals on these soils display symptoms typical of heavy metal chlorosis and crops grown on the infertile sections, after liming, contain up to 50 p.p.m. nickel. Haselhoff (1893) has reported the ill-effects of waste water from a nickel-ore crushing plant. On the other hand, Roach and Barclay (1946), in experiments in the Romney Marsh with wheat, potatoes and broad beans, recorded statistically significant increases in crop yields following the application of nickel sprays.

There is no conclusive evidence of any nickel deficiency in stock, although Dixon (1937) has found a slight response to nickel, as well as cobalt, at Morton Mains, New Zealand.

Iron

Iron is generally described as a major element. Wallace (1951) however, lists it among trace elements, with a footnote to the effect that in dealing with field problems it is more convenient to group it with the trace elements. It has long been known that iron is essential to the plant.

Although iron is one of the most abundant elements in the soil it is often unavailable in sufficient quantity to satisfy plant requirements. While not a constituent of chlorophyll, it is essential to its formation, so that the symptom of iron deficiency is a chlorosis due to lack of chlorophyll./

chlorophyll. Iron deficiency is one of the most serious nutritional problems in fruit culture, but certain legumes and timber trees, especially conifers, may also suffer injury from lack of iron. The soil conditions responsible are fairly well understood. The presence of excess calcium carbonate in the soil, except when it is rich in organic matter, or a high manganese content of the soil renders the iron unavailable. The deficiency is difficult to control, the soil conditions being such as to convert any iron compounds into the insoluble ferric state. Addition of ferrous salts are generally unsatisfactory or of limited application. The spraying of foliage with iron solution is only partially effective and is often damaging to the foliage and fruit. Greater success has attended the injection of solid ferrous sulphate, ferric citrate or ferric tartrate into the trunk of the tree, and this has become the usual practice. Recently investigations have been carried out on the use of chelating agents as a means of supplying iron to plants. Leonard and Stewart (1953) found that iron chelated with ethylene diamine tetra-acetic acid was readily available to plants growing in soil. Wallace and his associates (1953) have described soil treatment with the three chelating agents ethylene diamine tetra-acetic acid, hydroxyethyl-ethylene diamine tri-acetic acid and diethylene triamine penta-acetic acid combined with iron: all gave an improvement of the chlorotic condition in eighteen different woody species. An account of the application/

application of metal chelates in plant nutrition is given by Stewart and Leonard (1954). The availability of iron seems to be affected by the same factors as affect manganese availability, namely, an alkaline reaction and oxidising conditions in the soil.

In the animal iron deficiency is associated with various anaemic disorders. Cow's milk is normally low in iron and a nutritional anaemia develops in rats which are fed on it. This cannot be cured by the addition of a pure iron salt alone, and copper must also be fed, if the formation of haemoglobin is to become normal again.

A similar anaemia is sometimes developed in sucking pigs. McGowan and Crichton (1923, 1924) have dealt with iron deficiency in pigs or "piglet anaemia". They attributed the condition to lack of iron in the sow's milk and concluded from their experiments that it could be prevented by the introduction of ferric oxide into the sow's pen. The efficacy of this treatment was considered due to the provision of an accessible store of iron for the young pigs and not to an increase of iron in the milk through consumption by the sow of the enriched food.

The condition rarely occurs in litters farrowed out of doors and reared on grass. The soil offers opportunities to pick up additional iron. It is generally agreed that most effective control is obtained by early and individual dosing of the piglets with a solution of iron and copper salts, the copper being added because milk is poor in it and

copper/

copper functions in the mobilisation of iron for haemoglobin formation. The early dosage should furnish 20 to 30 mg. of iron and 2 to 3 mg. of copper per head.

Anaemia occurs in calves and other young animals in the event of there being kept too long on an exclusive diet of milk. Iron deficiency is rarely found in poultry, although it may be induced experimentally.

Zinc

Zinc is essential in both plant and animal nutrition.

As to its functions in the plant, Wallace (1951) states that the evidence points to a role as catalyst and regulator. Deficiency of zinc in British crops has not been recorded, although Roach and Barclay (1946) obtained in their Romney Marsh experiments large increases in crop yields following the application of zinc sprays. Abroad zinc deficiency is associated with a number of serious disorders e.g. "Little Leaf" or "Rosette" of apples, pecans and stone fruits; "Mottle Leaf" of citrus; "Bronzing" of Tung trees; "White Bud" of maize. Descriptions of these diseases and the remedial measures for their control are given in Stiles (1951).

With regard to the animal, Green (1944) has pointed out that an ordinary diet for animals may well supply 0.1 mg. zinc per kilogram of body weight, suggesting that no natural occurrence of zinc deficiency disease can be expected. Deficiency symptoms can, however, be induced in laboratory animals using synthetic diets.

Boron

Boron is essential to plant nutrition, and although it is present in the animal body, there is no proof that boron performs any useful function in the animal body. Wallace (1951) describes the functions of boron in the plant as those of a catalyst or reaction regulator. The same authority summarises the effects which have been attributed to the element:- "It can delay the onset of calcium deficiency effects but cannot replace calcium; it tends to keep calcium soluble; it may act as a regulator of potassium/calcium ratios, and of the absorption of nitrogen; it may be concerned with the oxidation-reduction equilibria in cells."

The availability of boron to the plant is decreased by liming and by dry conditions. Overliming is especially harmful. Apart from this fact, deficiency of the element is probably mainly determined by the natural supplies in the parent rocks. The deficiency frequently occurs on sandy soils. When boron deficiency is found on acid soils the presumption is that there is an actual deficiency of boron in the soil.

Deficiency of boron with serious results is widespread among fruits and vegetables throughout the world. Boron deficiency conditions are associated with the following disorders:-

"Heart Rot" of sugar beet (see Wallace, 1951, pls. 133, 134) and mangold (see Wallace, 1951, pls. 143-145); "Canker and Internal Black Spot" of red or garden beet; "Brown Heart" or/

or "Raan" of swede (see Wallace, 1951, pls. 106-108) and turnip; "Browning" and "Hollow Stem" of cauliflower (see Wallace, 1951, pls. 73-77); "Cracked Stem" of celery (see Wallace, 1951, pl. 240); "Drought Spot" or "Cork" of apples. These disorders are all discussed by Stiles (1951 pp. 80-89).

The deficiency condition is readily controlled by the application of borax to the soil, usually in amounts ranging between 20 and 50 lb. per acre. Spraying is suitable for fruit trees using a solution of 3 lbs. borax in 100 gallons of water.

Copper

Copper as a trace element essential to the nutrition and health of animals, has assumed considerable importance during the last twenty years.

Although no specific functions have been attributed to copper in the plant, the evidence according to Wallace (1951) is that it serves as a catalyst and regulator. It has been shown that copper protein compounds, haemocuprein and hepatocuprein occur in the blood and liver respectively of mammals. Although the actual part played by the compounds in the physiology of the animal is not clearly understood, it would appear to be established that the functions of copper are to be found to some extent in the role of these copper protein compounds.

The two diseases in plants, so far reported, attributable to copper deficiency are (a) "exanthema" or "dieback" or chlorosis of fruit trees, and (b) a disease of various plants known/

known as "reclamation disease", exanthema or "dieback" of citrus, pears and other fruits has been reported in the United States and Australia. Wallace (1951) states that in exanthema, shoots die back, the foliage may show chlorosis, and rosetting and bud formation may occur. Characteristic symptoms of "reclamation disease" in cereals are withering and marginal chlorosis (yellowish grey) of the tip of the younger leaves, and a tendency for the foliage to wilt. Grain formation is more severely restricted than vegetative growth.

Dunne (1938) gives figures for the copper content of healthy apple leaves in Western Australia at 5.5 - 12 p.p.m. and of others from trees affected by exanthema at 1 - 3.6 p.p.m., all measurements referring to dry weight. These values agree with those obtained by other investigators. The diseases may be controlled by the application of copper sulphate to the soil, although there are indications that, where practicable, spray treatment is most effective.

The animal body contains a minute trace of copper, but a deficiency of the element in the food affects animal health. One disorder associated with copper deficiency is "piglet anaemia" (referred to under the discussion on Iron).

A disease which occurs in Australia is described by the term "enzootic ataxia" which most commonly affects lambs from one to two months old. They become unthrifty and growth is retarded. In 1937 in Australia, research workers/

workers shewed that the condition could be prevented by drenching ewes with copper sulphate. The condition was later correlated with a low copper status of the animal and herbage. At the same time British workers were investigating a disease known as "swayback" which is widely distributed through Great Britain. The results of extensive experiments in North Derbyshire shewed that the incidence of the disease was greatly reduced amongst the offspring of ewes receiving licks with copper supplements. Dunlop and his collaborators (1939) confirmed the effectiveness of copper administered to pregnant ewes in preventing the disease. Later work by Hunter and associates (1945) demonstrated the importance of ensuring ingestion by the ewe of the necessary amount of copper, and indicated that some factor in the natural herbage, other than copper, is responsible for the low blood copper status of grazing ewes. It would therefore seem that while pathologically the enzootic ataxia of Australia and swayback in Britain are identical conditions, the disorder is not correlated in Britain with a low copper content of the herbage, and it remains to be determined what conditions prevent the grazing ewe from utilising the natural copper of "swayback" herbage.

"Coast Disease" of grazing animals in Australia (referred to under Cobalt) and "Salt Sick" disease of Florida are both associated with a dual deficiency of copper and cobalt.

Iodine/

Iodine

Iodine is vitally essential in animal nutrition, but not generally regarded as necessary for the plant.

Wallace does not include iodine among those elements, such as sodium, chlorine and silicon, which though not regarded as essential to growth, have been found to produce beneficial effects on the growth of certain plants.

On the other hand, certain iodine compounds, including tri-iodobenzoic acid and iodophenoxyacetic acid have been investigated and found to be of value as plant growth substances. The preliminary researches on the use of organic compounds of iodine as plant growth substances were carried out in the United States. The results of these researches to date suggest that such iodine compounds may prove very useful as plant growth substances. Several investigations have claimed beneficial results following the application of iodine to deficient crops, e.g. increases in yield of millet and hops. Whatever part may be played by the element in plant nutrition, considerable importance must be attached to the maintenance of a supply of iodine to crop plants, as carrier of the element to man and animal.

In the animal the iodine exchanges within the body are controlled by the thyroid gland. In this gland the iodine derived from the food is used in the formation of thyroxine. Thyroxine is an amino-acid which forms part of the protein thyreoglobulin. The functions of iodine in the form in which it occurs in thyroxine is concerned with physical and mental/

mental development, adequate reproductive capacity, the growth of wool and hair, and the regulation of body temperature.

Iodine deficiency conditions may be induced experimentally by thyroidectomy, when, in the case of sheep, the wool becomes coarse and easily pulled out. The removal of the gland from young fowls causes considerable reduction in growth rate, while feather structure is altered. In the milking cow thyroidectomy has been shewn to reduce the lactation period and yield of milk.

In many parts of the world in what are commonly classed as "iodine deficient" or "goitre" areas, goitre and hairlessness are of common occurrence among farm livestock fed on unsupplemented rations.

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Practically any trace element may produce poisoning, if presented to the plant or animal in too great excess. For example, a high concentration of manganese within the plant may lead to iron deficiency and chlorosis; the reported poisonous effects of nickel have been discussed earlier; the addition of large quantities of iron compounds to the rations of young animals may cause rickets, by forming an insoluble phosphate, and interfering with normal phosphorus absorption.

There are two diseases of animals, however, with definite and characteristic symptoms, which have been attributed to the effects of trace elements present in excess in the animal food. The trace elements concerned in the two diseases are respectively molybdenum and selenium.

Molybdenum

As stated previously molybdenum has been shewn to be necessary for plant nutrition. The symptoms shewn by tomato plants and ascribed to molybdenum deficiency have been described by Arnon and Stout (1939). The symptoms of molybdenum deficiency of oats grown in water culture are given by Piper (1940). There are no reports of molybdenum deficiency for any crop in this country, although recently in Australia a deficiency occurs in certain acid soils, which affects plant growth; it has been remedied by the application of small dressings of molybdenum.

There is no evidence that molybdenum is essential to animal health. Healthy plants however can take up molybdenum in amounts injurious to animals. In certain regions in Wyoming the element is believed to be a factor in the poisoning of range livestock.

In this country one example of the injurious effect of molybdenum is provided by the "teart" pastures of Somerset, where a comparatively high molybdenum content of the herbage causes scouring of grazing ruminants. The subject is discussed by Lewis and Watson (1942). The molybdenum content of teart herbage is about 3-20 p.p.m. of fresh material. This malady can be prevented and cured by the feeding of copper sulphate. For cows 2 gms. and for young stock 1 gm. per head daily are sufficient, and less may be required on mild "teart" pastures.

On/

On certain Scottish pastures comparatively rich in molybdenum, Mitchell and his associates (1941) have found that the application of cobaltised fertiliser, in the control of the pining of sheep, may increase the molybdenum content of the herbage to a "teart" level.

Selenium

There is no substantial evidence that selenium is essential to plant nutrition. It would appear, however, to accumulate in relatively large amounts in certain species.

The element, while tolerated by most plants, is highly toxic to the animals consuming the plants. Two different types of selenium poisoning in livestock are known, namely, the "alkali" disease of Western South Dakota and Northern Nebraska, characterised by sloughing of the hoofs, and "blind staggers", the main form of "selenosis" in cattle and sheep on the ranges of many western States. Selenosis has been recorded in Eire, but not in the United Kingdom. A good account of the early work on selenium poisoning is given by Trelease and Martin (1936).

CHAPTER III

THE SPECTROGRAPHIC METHODS OF ANALYSIS USED FOR DETERMINATIONS IN PLANT AND SOIL MATERIAL

The following analytical procedures have been adopted in this investigation; they are based on the techniques evolved and used in the Macaulay Institute for Soil Research, Aberdeen.

These are summarised below for convenience.

i) The Determination of Calcium, Potassium, Magnesium, and Manganese in Plant Ash.

a) Chemical Extraction

5g. of air dry, finely ground pasture are ashed, in a silica basin, in a silica-lined furnace at 450°C. The ash is weighed, evaporated to dryness twice with concentrated hydrochloric acid, then dissolved in 25ml. of 2 N hydrochloric acid, covered with a watch glass and digested on the water bath for 30 minutes. The resultant extract is filtered into a 250 ml. **graduated** flask, washed with hot water and the filtrate diluted to 250 ml. The residue is ignited and weighed. From the weighings the figure for silica-free-ash is obtained. For the estimation of manganese and magnesium, the original extract is used, whilst a 1:4 dilution is required for potassium, and a 1:49 to 1:99 dilution for the determination of calcium,

b) Spectrographic Procedure

The Lundegardh emission method of flame analysis, as described/

described and modified by Mitchell (1936, 1948) has been used.

The method involves the introduction into an air acetylene flame, by means of an air supply, of the hydrochloric acid extract of the pasture sample being analysed. The sprayer unit comprises an ebonite sprayer nozzle fitting into a glass sprayer vessel which uses 2-10 ml. solution. The solution is atomised as a fine spray by the compressed air and carried into a glass burner tube, where it is intimately mixed with the acetylene and burnt at a slit-type platinum-iridium aperture. The burner aggregate which includes the sprayer unit and burner tube is used in conjunction with a Hilger Medium Quartz Spectrograph. A 5" by 4" Ilford Zenith plate is carried in the long wavelength half of the plate-holder covering approximately the 2800Å-5000 Å range, in which region the analysis lines of the elements concerned occur. The slit length is 1.5 mm. enabling duplicate spectra of 6 standard solutions and 16 solutions for analysis to be recorded on one plate. The gas pressures and exposure time (60 secs.) are strictly controlled, thereby yielding a high degree of reproducibility.

A concentrated standard solution is prepared containing the following molar concentration of the chlorides of the elements;/

elements:

Ca: M/4000

K : M/200

Mg: M/200

Mn: M/4000

Fe: M/200

The solution is in dilute hydrochloric acid of about N/20 to prevent hydrolysis of manganese and iron. From this solution five dilutions are made of one half, one quarter, three twentieths, one tenth and one twentieth thereby providing a convenient dilution series for each element and giving densities suitable for measurement. The plate is developed for $5\frac{1}{2}$ minutes in 1:2 1.D.2. Ilford metol-hydroquinone developer.

c) Photometry

The low energy associated with the flame source yields a relatively simple spectrum showing few lines. The spectrographic slit width employed of 0.045 mm. gives a satisfactory background density and resolves sufficiently the potassium doublet at 4044/47°A and the manganese triplet at 4031/34°A. The background density is used as a form of internal standard correcting for emulsion sensitivity and exposure time. As standard spectra are recorded on each plate, errors due to differences between plates are eliminated. Measurements are made on a Hilger/

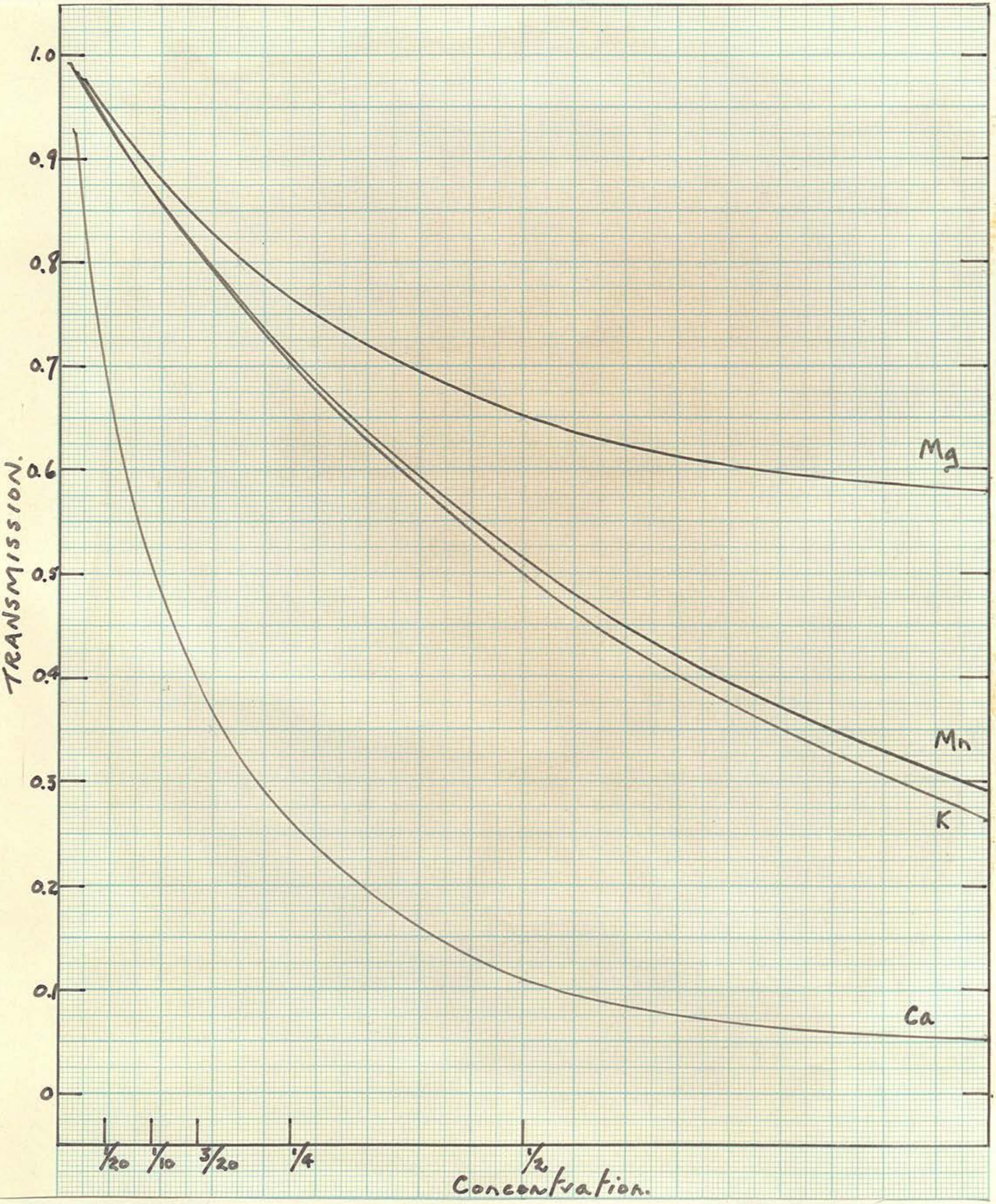


Fig. 1. Typical Lundegardh working curves (calcium, potassium, magnesium, manganese).

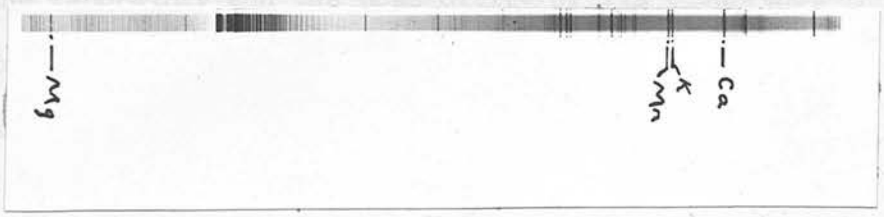
a Hilger Non-recording microphotometer of the galvanometer deflections for the analysis line concerned (calcium, potassium and manganese) and the background on which it is superimposed, the galvanometer being previously adjusted to give a total blackness reading of zero. The magnesium line lies in a banded background on a band of intensity equal to that immediately below it in wavelength. In this case, measurements are made of the deflections for the magnesium line, and the first band of shorter wavelength treating this latter as background.

The ratio Deflection (analysis line + background) / Deflection (background) is plotted against the concentration for the standard solution spectrograms for each element. A series of the typical curves is shewn opposite in Fig. 1. From these curves the concentrations of the solutions for analysis may be determined, and thereby the contents of the particular elements in the pasture samples. In this investigation the results for calcium, potassium and magnesium have been reported as percentages of the element in each case, while the results for manganese and the other trace elements are expressed as parts per million. The wavelengths of the lines are as follows:

Ca : 4227°A
K : 4044°A
Mg : 2852°A
Mn : 4031°A

d)/

(d) The photograph (Fig. 2.) below shows a typical Lundegårdh flame spectrogram with the analysis lines marked:



(ii) The Determination of Cobalt and Nickel in Plant and Soil Material.

(a) General Remarks

Cobalt, nickel and iron, in common with other trace elements in plant material and soil extracts, are not present in sufficient amounts to be estimated directly by spectrographic methods, and accordingly a chemical pretreatment or concentration procedure is required. This pretreatment requires more time than a direct spectrographic analysis and increases the risk of contamination, but serves simultaneously (a) to remove variables such as phosphates, alkaline earths and alkalies in plant material and alkaline earths and alkalies in soil extracts, (b) to eliminate effects/

effects of composition variation in the sample for analysis compared with the standard samples and (c) to permit the incorporation in the method of the variable internal standard technique.

In connection with the problem of contamination in the laboratory and the possibility of its occurrence during the chemical process the following notes are inserted.

Laboratory Equipment

All ignitions are carried out in platinum basins in a silica-lined muffle furnace. Water baths are of stainless steel and are periodically covered, as are all metal parts in the laboratory, with a coat of special plastic resin paint. The baths are fitted with unplasticised perspex tops with porcelain rings. Pyrex glass ware only is used and spatulas are platinum or non-metallic.

Laboratory Reagents

Distilled water is obtained from a Manesty Still incorporating a pyrex glass condenser tube. All reagents used in the chemical process are tested for trace element impurity and subsequently purified if required, with the exception of the precipitants, 8.hydroxyquinoline, tannic acid and thionalide, which are normally suitable. Hydrochloric and acetic acids are all glass distilled and ammonia is purified by reabsorption in distilled water, and/

and stored in waxed bottles. Methods for the purification of sodium carbonate, alumina and ferric oxide are given by Mitchell (1948). In the course of this investigation, "blank" estimations have been made regularly to ensure freedom of the samples from impurity at every stage of the process. In the event of high "blanks" being obtained, repurification of reagents is carried out and the samples analysed in the interval are reanalysed.

(b) Chemical Pretreatment

The concentration methods for the recovery of trace elements from plant materials and soil extracts are those of Scott and Mitchell (1943) and Mitchell and Scott (1947). Different methods are used for obtaining the trace elements in solution from pasture and soil samples prior to their precipitation in an alumina matrix. Thereafter the procedure is essentially the same. The methods for obtaining solution of the trace elements from plant and soil material are given below, followed by the precipitation procedure.

(1) Solution of the samples

Plant Materials

20 g. of air dry finely ground pasture are ashed overnight in a platinum basin, covered with a silica-ware watch glass in a silica-lined muffle furnace at 450°C. A gentle current of air is drawn through the furnace for the first three hours to remove the volatile carbonaceous matter. On cooling, the ash is transferred, by means of an aluminium/

aluminium spatula and camel-hair brush, into a platinum crucible. 4 g. of purified Na_2CO_3 are added, mixed with a glass rod, covered with a platinum lid, and fused (at first gently) for some 15 minutes. After cooling, the crucible and lid are transferred to a 4 inch silica-ware basin containing a little distilled water. 25 ml. of 6 N HCl are added, and after the melt has dissolved, the crucible and lid are washed and a little HCl boiled up in the crucible and added to the main solution. The solution is then evaporated to dryness on a water-bath and dried for about 30 minutes. A solution containing 25 ml. of 6 N HCl and 25 ml. of distilled water is added and after five minutes a further 30 ml. of water. After a further 15 minutes heating the solution is filtered through a 9 cm. Whatman No. 41 paper into a 250 ml. pyrex beaker and washed with hot water. After cooling the equivalent of 30 mg. of AL_2O_3 and 0.4 mg. of Cd. as $CdCl_2$ are added. The solution is now diluted to 150 ml. and is ready for precipitation.

Acetic acid extracts of soils

20 g. of air-dry soil (sieved through a 2 mm. screen) are weighed into a shaking bottle (quart milk bottle). 800 ml. of 0.5 N purified acetic acid are added and shaken overnight. The solution is filtered through an 18.5 cm. No. 530 Whatman paper into a litre pyrex beaker, and the bottle washed twice with distilled water and the filter paper

paper once. This extract is evaporated to low bulk on the hot plate and finally to dryness on the steam bath. Two to three 10 ml. portions of A.R. hydrogen peroxide are added to destroy any organic matter present, giving a colourless residue. The residue is dissolved in dilute HCl (25 ml. of 6 N HCl and 25 ml. water) and allowed to stand covered on a steam bath for about 15 minutes. It is then filtered into a 250 ml. pyrex beaker through a 9 cm. No. 41 paper and washed.. The equivalent of 2 mg. of Fe_2O_3 are added and 0.4 mg. of Cd as $CdCl_2$. The solution is now diluted to 150 ml., and is ready for precipitation.

(2) 8-Hydroxyquinoline-tannic-acid-thionalide precipitation for the determination of Cobalt and Nickel.

To the sample solution (containing Cd as the internal standard for Zn, in the event of such determination being made or required), are added 10 ml. of a 5% solution of 8-hydroxyquinoline in 2 N acetic acid for pasture material and 15 ml. for soil extracts. Purified 1:1 NH_4OH is added drop by drop until the solution turns emerald green at a pH of 1.8. 30 ml. of 2 N ammonium acetate are then added to bring the pH to approximately 5.1, and the solution stirred to coagulate the precipitate. 2 ml. of freshly prepared 10% tannic acid in 2 N ammonium acetate are added, and after stirring, 2 ml. of freshly prepared 1% thionalide in glacial acetic acid, followed immediately by an equivalent quantity of NH_4OH with stirring. After standing overnight, the precipitate is filtered through/

through an 11 cm. No. 540 Whatman filter paper, washed with cold water, allowed to drain and partially dry in the funnel, transferred to a porcelain crucible and ignited in the silica-lined muffle at 450°C.

The ignited precipitate is allowed to reach moisture equilibrium in the balance room, weighed (the weight should be between 30 and 50 mg.) on glazed paper on a torsion balance, mixed by grinding in an agate mortar and transferred to a glazed paper boat.

The iron content of the residue or concentrate is determined colorimetrically, as described below under (iii).

A portion (about 20 mg.) of the final residue derived in the above process is weighed out on a 500 mg. torsion balance on an approximately 2 x 2.5 cm. glazed paper carrier, and mixed in a small agate mortar with twice its weight of carbon powder. The carbon powder is obtained by grinding purified carbon rods in an agate mortar. The carbon electrode is maintained in a small plastic block, and a small paper collar slipped over the narrow portion projecting some 2 mm. above the electrode top. The carbon-concentrate mixture is poured into the collar, and introduced into the cavity by tapping the block gently. The mixture is compressed in the cavity by means of a stainless steel needle. Every precaution is taken to ensure freedom from contamination. After each sample the agate mortar is cleaned, first with cotton wool and then by grinding

out/

out with powered alumina. After each electrode has been filled the mortar is washed with hydrochloric acid. The mortars used are retained for this purpose only.

(c) Spectrographic Procedure

The method used is the variable internal standard method applied to the cathode layer arc as described previously by Davidson and Mitchell (1940).

The quantitative analysis of trace element content in concentrates derived from plant material and soil extracts involves the excitation of the concentrate material in the cathode layer D.C. arc, operated in conjunction with the Hilger Large Quartz Spectrograph.

This effect is obtained by focussing the image of that portion of the arc column adjacent to the cathode at the prism of the Hilger Large Quartz Spectrograph, by means of a spherical lens yielding even illumination of the slit and placed 2 cm. in front of it. Only light from the cathode tip and about one third of the adjacent arc column (i.e. the cathode layer) is permitted to reach the prism by means of a mask placed at the collimator lens. A step sector carried on the lens mount and driven by a constant speed motor rotates between the lens and slit (of width 0.015 mm. and length 8 mm.). In this way the slit is illuminated for each exposure, by the 6 step sector/

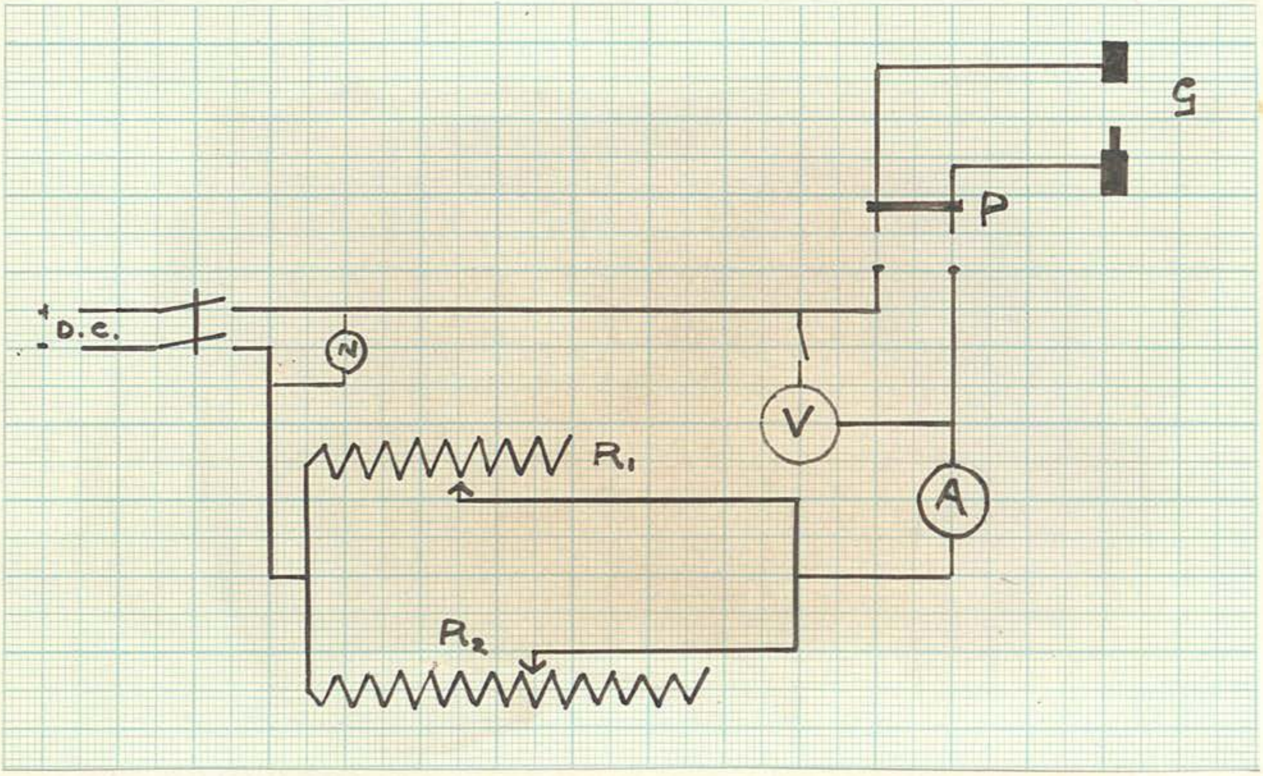


Fig. 3. Circuit for control of d.c. arc.

A : Ammeter 0-15 amp.
V : Voltmeter 0-120 volt
N : Neon indicator
R_1: Variable resistance
R_2: Tapped resistance
P : Polarity reversal plug
G : Arc gap

sector, in 7 steps corresponding to relative exposures of 64 (uninterrupted light), 32, 16, 8, 4, 2 and 1. The working position of the arc is controlled optically, by a lens and screen placed at the rear of the arc stand. The arc stand carried on the accessory bar permits quick and efficient manipulation of the electrodes. The stand is fitted above with a hood to remove fumes and material burnt in the arc, which would otherwise contaminate the laboratory. In addition the slight draught caused stabilises the arc.

The electrode containing the sample for analysis and the counter electrode (a flat-ended 5 mm. carbon rod) are fixed in the stand holders. The sample electrode is made the cathode of a 9 amp. 10 mm. D.C. arc the preliminary alignment of the electrodes being effected by reference to the control screen. The diagram (see fig. 3 opposite page) gives the circuit employed for control of the D.C. arc. An exposure of 3 minutes is sufficient for complete volatilisation of the sample, and the spectra are recorded on an Ilford chromatic 10" x 4" plate in the spectral range 2750 - 4750°A. The plates are developed in 1:2 I.D.2 metol-hydroquinone developer for $2\frac{1}{2}$ mins. at 18°C.

(d) Photometry

In this method, as described in the previous section, stepped spectrograms are obtained for each exposure. All measurements are obtained using a Hilger Non-Recording Microphotometer, giving a full scale deflection of 50 cms.

for/

for developed unexposed emulsion. For both the analysis and internal standard lines of a particular line pair, deflections are measured for 3 steps, the densities of which, derived from the deflections (from tables by Mitchell and Scott, 1942-43) lie on the straight line portion of the characteristic curve for each line. By means of these characteristic curves, obtained by plotting density against the appropriate log. relative exposure, measurements can be made on the basis of one photographic density.

In the case of nickel the separation is measured between the analysis and internal standard line curves at a density of 0.4. This separation which is estimated in arbitrary units, assumes positive and negative values according to the relative strengths of the lines and is a measure of their log. relative intensities, as log. $\frac{I_A + I_B}{I_S + I_B}$, where I_A, I_S, I_B are the intensities of the analysis line, internal standard line and background respectively. The separation is plotted against the concentration of nickel as log. p.p.m. in the mixture concerned.

In the case of cobalt, a background correction at constant density is made, according to the method given by Scott (1944, 1946), for each spectrum, involving additional measurements of as many steps of the background as possible in the neighbourhood of the lines concerned. This procedure gives a value for the intensity ratio, as log I_A/I_S, free from background effects. The calculations involved are simplified by reference to Tables by Mitchell and his associates (1943-44). This/

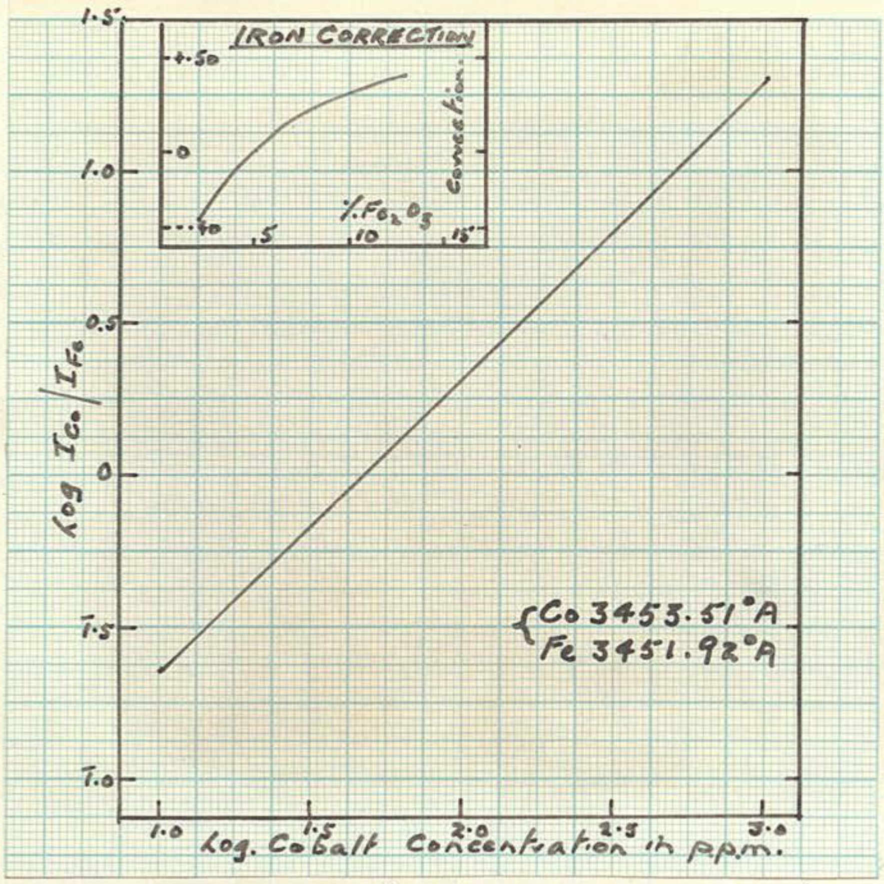


Fig. 4a. Working curve (and iron correction curve) for the determination of cobalt in an alumina matrix, with correction for spectral background.

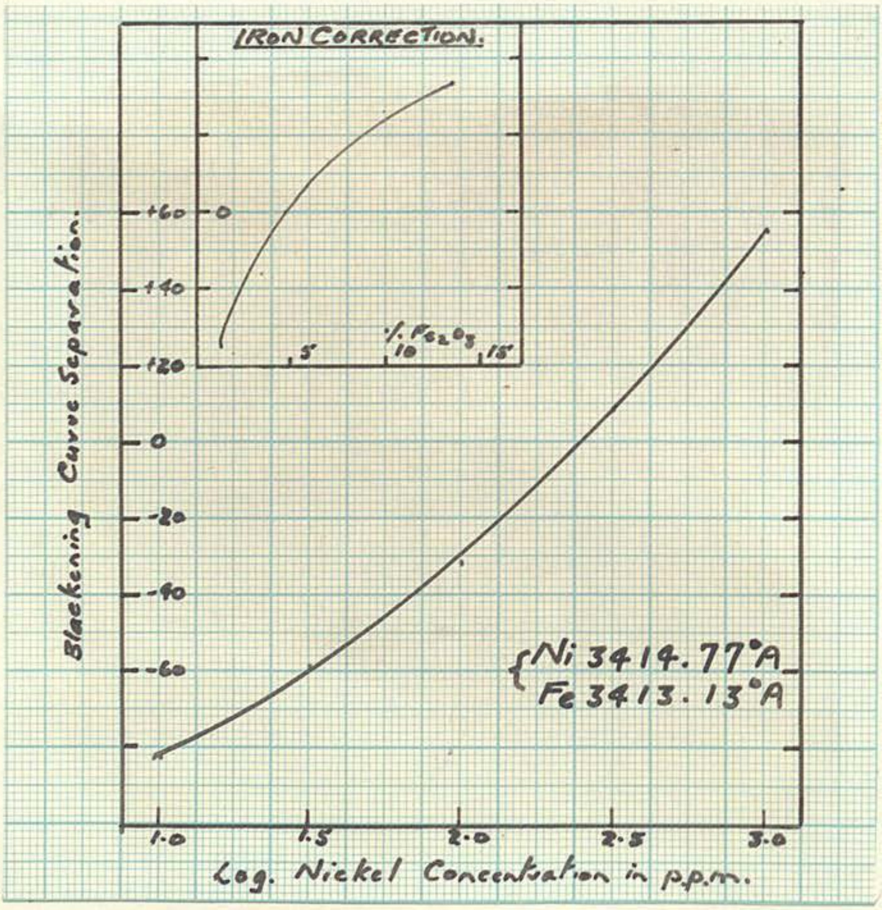


Fig. 4b. Working curve (and iron correction curve) for the determination of nickel in an alumina matrix, without correction for the effect of spectral background.

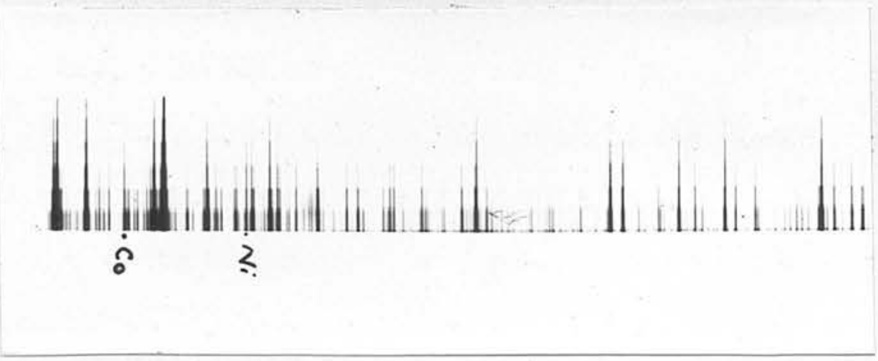
This method of analysis incorporating a stepped sector and internal standard enables standard spectra and the spectra of samples for analysis to be recorded on separate plates, compensating for variations in the source, plate development and exposure time. In the initial calibration of the instrument, a series of working curves is obtained from standard mixtures, containing increments of the elements cobalt and nickel in matrices similar in composition to that of the concentrates, and having increments of the internal standard element, iron in this case, from set to set. Each standard set covers a range in steps of $\sqrt{10}$ from 10,000 to 10 p.p.m. for the elements cobalt and nickel, and the six sets employed cover an Fe_2O_3 range of from 2.0 to 13.0%. The series of working curves obtained from the sets for the same trace element concentration range, but different Fe_2O_3 contents, are parallel, and from their relative displacements, an iron (as % Fe_2O_3 in concentrate) correction curve is obtained, permitting all curves with the exception of one (in this case the curve for the 5.0% Fe_2O_3 set) to be discarded. The line pairs used in the investigation were:-

Co: 3453.51^{o}A Fe: 3451.92^{o}A
Ni: 3414.77^{o}A Fe: 3413.13^{o}A
Ni: 3101.55^{o}A Fe: 3099.90^{o}A

Working curves for cobalt and nickel together with the iron correction curves are shewn in Fig. 4a and 4b opposite.

(e) The photograph (Fig.5) below shews part of a typical arc spectrogram/

spectrogram with the cobalt (3453.51°A) and nickel (3414.77°A) lines marked.



(iii) The Determination of Iron in Plant and Soil Concentrates.

The method adopted has been described by Scott (1941) and Scott and Mitchell (1943) and employs the amethyst colour produced by iron in the presence of sodium salicylate in acetic acid solution.

5 to 10 mg. of the finely ground concentrate are weighed in a small platinum crucible on a micro-balance. To the sample are added about 200 mg. of anhydrous A.R.Na_2Co_3. The contents are stirred with a platinum wire, fused for some 15 minutes, and cooled when the melt is dissolved in 10 ml. 1:1 HCl. The resultant solution is transferred to a 50 ml. graduated flask and diluted to 50 ml. with water. A 20 ml. aliquot is transferred to another 50 ml. graduated flask and 5 ml. of a 1% sodium salicylate solution added. 1:1 NH_4OH is added drop by drop until the reddish colour just obtained turns yellow, then two drops in excess. 1:1 acetic acid is now added until a reddish colour reappears, then a further 5 ml. are added. The volume is made up to 50 ml. with distilled water, and the colour density immediately determined on a Hilger Spekker Absorptiometer, using 4 cm. cells, and No. 5 green filters, and initially adjusted to a distilled water calibration value of 1.00. The iron values (as Fe_2O_3) are obtained by reference to a curve (see Fig. 6 opposite) obtained from a standard solution treated in the same way and prepared from ferric ammonium sulphate and covering a range of 0.05 to 0.50 mg. in the final 50 ml. solution.

All/

All pasture results are finally calculated on an (80°C) oven-dry basis, the soil determinations referring to air-dry samples.

(iv) General Note relating to the Precision of the Methods.

The following tables give the derived values of the errors (expressed as coefficients of variation and based on sets of 20) associated with the analysis for each element. These errors are discussed in greater detail in the next chapter. The silica-free-ash figures for the pasture samples (as percentages) have been included in the results.

TABLE 1. Lundegardh Technique (Ca, K, Mg, Mn).

PROCEDURE	ERROR			
	Coefficient of Variation (%)			
	Ca	K	Mg	Mn
Spectrographic analysis	5	3	10	5
Complete analytical technique; including laboratory sub-sampling, extraction and spectrographic analysis.	6	6	12	7

TABLE/

TABLE 2. Technique for Co and Ni Determinations.

PROCEDURE	ERROR	
	Coefficient of Variation (%)	
	Co	Ni
Spectrographic analysis	4	4
Concentration and spectrographic analysis	6	6
Complete analytical technique; including laboratory sub-sampling, fusion, concentration and spectrographic analysis (HERBAGE)	10	12
Complete analytical technique; including laboratory sub-sampling, extraction, concentration and spectrographic analysis (SOIL)	10	12

TABLE 3/

TABLE 3. Iron Estimation.

PROCEDURE	ERROR
	Coefficient of Variation (%)
'Spekker' determination	2
Concentration and 'Spekker' determination.	4
Complete analytical technique; incl. laboratory sub-sampling, fusion, concentration and 'Spekker' determination (HERBAGE)	10

Silica-Free-Ash Estimation

The error (as coefficient of variation) associated with this estimation is of the order of 6%.

CHAPTER IV

EXPERIMENTAL

The assessment of the variation in trace element content of upland pastures involves measuring the variations for different plant associations growing on different soil types. Accordingly the experimental site chosen was divided into smaller areas, as far as possible uniform within themselves with regard to vegetation and soil type. These small uniform areas have been termed for convenience "sub-areas".

In order to obtain a measure of the variation with regard to any element in pasture or soil within a particular sub-area, observations must be made at a number of points within that area. In the initial experiments, to be described later, these points were selected according to a systematic procedure, in which a circular area was chosen, and points were taken round the circumference, corresponding to the "points of the compass". In the later experiments points were taken at random within the sub-area: for example, random co-ordinates were used within a rectangle in the centre of the sub-area, these co-ordinates being referred to particular reference points within the rectangle.

Irrespective of the procedure adopted, the herbage or soil sampler, on locating the points, took the appropriate sample (corresponding to one observation) from an area of 1 square yard (1 yard by 1 yard) immediately in front of the point. This unit of area has been termed a "point-area".

These two terms "sub-area" and "point-area" have been introduced/

introduced to avoid confusion with more general terms.

(i) General Notes

There are certain fundamental precautions which must be taken in the sampling of plant and soil material for trace element analysis. These precautions are outlined in the following notes.

(a) Collection of a Pasture Sample.

A sample is cut from the point-area concerned, the pasture herbage being cut with ordinary shears 1 inch above soil level, every precaution being taken to avoid soil contamination (treated more fully under (c)) and the inclusion of decaying vegetation. No differentiation with regard to species is made except where precisely stated.

(b) Collection of a Soil Sample.

A sample is obtained from the point-area by taking two or three slices with a spade, the mat being excluded, but the humified layer being included with soil down to the rock or to about 8 inches, whichever is smaller.

(c) Problem of Contamination.

As mentioned previously in the description of the laboratory analytical technique, every precaution is taken to ensure freedom from contamination throughout the complete analytical process. The possibility of contamination however is as great, if not greater, in the field, and, in consequence, the following points merit strict attention.

(1) Cut grass only, as opposed to plucked grass, is considered suitable for analysis. Samples of the latter type/

type are invariably "spoiled" with soil-bearing roots extracted in the process.

(2) Herbage and soil samples are each collected in paper-lined bags, and not in metal containers.

(3) Herbage samples are taken when the herbage is as free as possible from soil contamination, which may occur either in the form of dust after a long dry spell, or as "soil splutterings" immediately after heavy rain.

(4) Although not always strictly practicable (in the case of hill land) the sampling of selected areas is performed wherever possible when the areas have been free from grazing for several days.

In any event, the herbage operator while taking the sample, immediately rejects any "cutting" which appears "trampled down" or messed with contaminating material, e.g. soil, faeces.

(d) Method of Sampling Pasture and Soil Material with two operators.

The sampling of pasture and soil material is done in pairs, one operator attending to the pasture samples and the other to the soils. With one operator only, the risk of soil contamination is high, as the operator must handle herbage and soil alternately. The herbage sample is therefore taken first from the point-area irrespective of whether single or composite samples are required. After the herbage sample has been taken, and only then, is the soil sample taken by the soil operator from the same point-area./

area.

(e) Transportation of Material to the Laboratory.

Pasture samples are collected in 14lb. clean, paper-lined, waterproof bags. A card marked with the particulars of the sample is enclosed for subsequent identification.

Soil samples are collected in a lined paper bag (2 lb. capacity) with an enclosed identification card. The soil is firmly packed to avoid bursting of the bags in transit.

Pasture and soil samples are packed separately in clean cardboard boxes.

(f) Laboratory Manipulation of Material prior to analysis.

Upon receipt at the laboratory the soils are air-dried on paper-covered aluminium trays in a warm room at 30°C. The paper lining is renewed for each sample. The soil aggregates are broken up with a wooden roller and passed through a 2 mm. aluminium sieve, stones being rejected. The 2 mm. sample is then quartered to give a 20 g. aliquot for the laboratory analysis.

The pasture samples are dried on clean paper boats at 80°C in a stainless steel oven, and milled in a Christie Norris mill. Every precaution is taken to ensure freedom of the sample from dust during the process of milling.

(ii) Description and Particulars of Site chosen for Sampling Experiment.

(a) Location of Site.

Sourhope experimental hill farm, of some 2,340 acres, is situated near the head of the Bowmont valley in the Cheviot/

Cheviot Hills at the southern tip of Roxburghshire, the land ranging in altitude between 700 and 2,000 feet above sea level. Auchope farm, of 450 acres, adjoins Sourhope and is worked in conjunction with it. The majority of hill tops are between 1,000 and 1,500 feet high. The Schil hirsel stocked with Blackface sheep reaches the highest altitude. Auchope, the next highest, is stocked with North Country cheviots. The remaining hirsels are stocked with cheviots. The average sheep population for the farm of Sourhope is approximately 0.7 head per acre. Fasset Hill and Hairney Law were selected for the present investigation as being reasonably representative of the uplands of south-eastern Scotland.

(b) Recent and Geological History of Site.

There is ample evidence that the present rough grass vegetation, with small areas of heather, has dominated the region for at least five hundred years. The original forest cover of the lower slopes was probably destroyed by the action of man in the period 1000-1500 A.D. Attack from birds and the grazing of rodents and stock have since prevented almost completely the natural regeneration of woodland, which would be the natural vegetation of the region.

The underlying rock is porphyritic lava of a fairly rich andesitic nature. The region has however been glaciated, and it is possible that even the highest hills were completely covered by the ice. The distribution of the present areas of/

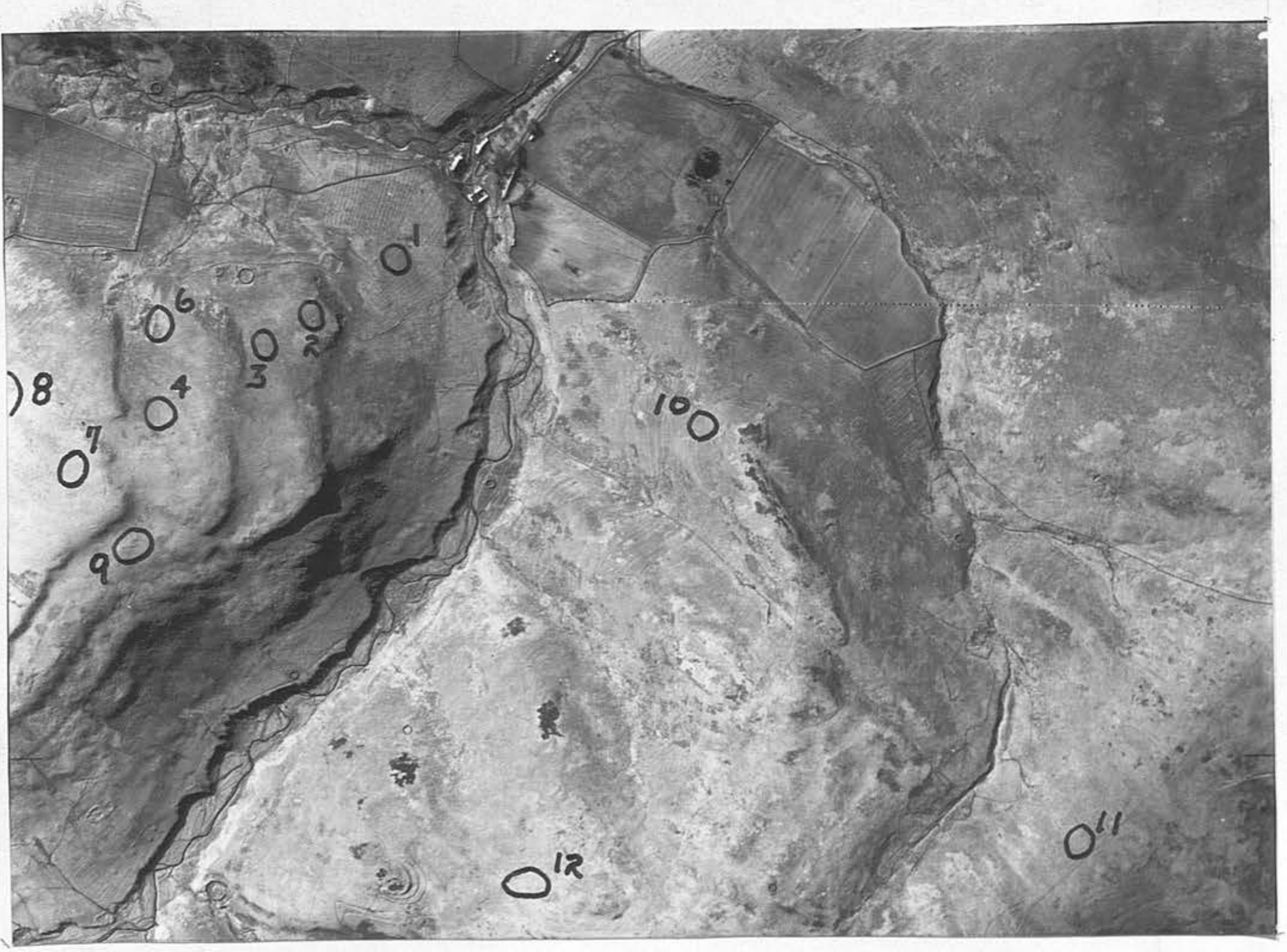


Fig. 7. Aerial photograph of experimental site, shewing sub-areas (1-4, 6-12) marked. (Photograph by Air Ministry.)

Fig. 8. Sourhope Experimental Farm.

of glacial material depends on the steepness of slope, and on the outcrops of lava.

In general, the lower valley slopes are covered with boulder clay of a clay or clay-loam texture. Some of the steeper slopes are covered by a strong loam type of glacial till, as are some of the flat tops. The higher flat tops consist of residual soils formed from the lavas, and there is a certain amount of screeing on the steeper slopes. Some podsolisation has occurred on the wetter slopes, and there is a cap of climatic peat, as much as 8 feet deep or more on the higher hills.

(c) Division of Site into Sub-areas.

Two factors which must be considered in connection with the variation in trace element content of hill pastures and soils are (a) vegetation and (b) soil type. Accordingly 11 sub-areas were selected each of approximate area 1-1½ acres, and as far as could be ascertained, of uniform soil type and plant association. Eight of these sub-areas, numbers 1-4 and 6-9, were selected on Fasset Hill, while sub-areas numbers 10-12 were taken on the slopes of Hairney Law, across the valley. An aerial photograph is shewn in Fig. 7 with the sub-areas marked. Fig. 8 is a photograph of Sourhope experimental farm, shewing the surrounding country.

Originally a sub-area 5 was listed among the areas to be examined. It was so similar in plant association and soil type to sub-area 1, however, that no sampling was actually/

actually carried out on it. The other sub-area numbers have been retained for convenience.

The profile descriptions (below) may be taken as typical of the sub-areas concerned, and they cover all the types to be found on Sourhope, with the exception of some complex valley soils and blanket peat found on the tops of the higher hills. The chemical status of the soils is poor, but not as poor as might be expected. It is problematical why heather has not invaded the hills to a greater extent, and podsolisation has not been more widespread. The residual soils in particular and the heavy clays may be considered as fairly fertile for hill soils, and could easily be improved by the application of lime and phosphatic fertilisers. Ploughing could undoubtedly be carried out successfully on sub-areas 1, 3, 4, 7 and 8, and sub-area 10 has now actually been ploughed. On areas 1, 4 and 6-9 cattle have recently been introduced, and it is yet to be seen what result this will have.

(d) <u>Description of the sub-areas chosen.</u>

<u>Sub-areas 3, 4, 7 and 8.</u>

Vegetation:		Agrostis, Fescue (mainly) with Nardus and Molinia.
Topography:		sub-areas 3, 4, 7 Flat " 8. E. to S.E. slope.
Profile:	0"- 2"	undecomposed grass litter with $\frac{1}{2}$" mull humus at bottom.
	2"- 8"	very dark-brown gritty sandy loam merging into
	8"-13"/	

8"-13" Dark-brown gritty sandy loam, rich in humus, and more compact than above. Roots. Stones mainly angular.

13"-20" Yellow brown, very indurated, very gritty sandy loam. Very few roots.

20"-31" Stony fine sandy clay loam, grey-brown. No roots. Slightly ochreous staining merging into

at 31" Red-brown gritty stony loam with yellow staining.

Drainage: Moderate.

Sub-areas 1 and 6.

Vegetation: Agrostis, Fescue (mainly) with Nardus and Deschampsia caespitosa.

Topography: sub-area 1: Flat
" 6: Steep southern slope.

Profile: 0"-1" undecomposed grass litter sharply changing to

1"-13" dark-brown clay loam. Roots. Crumb to loose cloddy structure. Worms. Moderate amount of stones, merging into

13"-21" dark-brown very gritty clay loam. Roots and stones.

at 21" dark-brown red gritty heavy clay. Stones. Few roots, ochreous mottling increasing with depth.

Drainage: Good.

Sub-areas 2, 10 and 11.

Vegetation: Agrostis, Fescue, Nardus, Deschampsia flexuosa, Festuca ovina and Bracken.

Topography: sub-areas 2; 10: N.W. slope
11: N.E. slope.

Profile: 0"-2" undecomposed grass litter.

2"-3" well decomposed mor humus.

3"-9"/

3"-9" medium, "muffy" sandy loam. Very dry. Roots plentiful, merging into

9"-14" fine medium brown sandy loam with many small angular stones, becoming larger with depth.

at 14" shattered rock.

Drainage: Good to very good.

<u>Sub-area 9</u>.

Vegetation: Molinia (dominant), Nardus, Festuca ovina.

Topography: N. exposure. Flat.

A small area of basin peat, based on heavy boulder clay.

Profile: 0"-12" wet black greasy peat. Roots. Sharp division at

12"-18" grey with ochre-staining heavy clay loam, merging into

at 18" grey-brown, very gravelly clay loam, with dark-brown staining at depth.

Drainage: Poor.

<u>Sub-area 12</u>.

Vegetation: Nardus, Molinia, Agrostis and Fescue.

Topography: Flat.

Profile: 0"-2" undecomposed grass litter.

2"-8" black, well-decomposed granular humus, merging into

8"-12" dark-brown stony gritty loam with high organic matter. Numerous roots.

at 12" iron pan 1/16th"

12"-24" stony gritty heavy loam with ochreous mottling, yellow brown colour.

at 24" strongly indurated brown-grey stony gritty sandy loam.

Drainage: Good.

(iii)/

(iii) Sampling Technique

Introductory Note.

Previous reference has been made to the difficulty encountered in interpreting the result of a single observation. For example, on a large tract of hillside, on which different plant associations are found growing, on different soils, doubt must always be attached to the results obtained from one "cutting" or "sample" of herbage. Even in the case of particular so-called "representative" samples gathered from one area, different ideas exist among samplers as to what constitutes a "representative" sample. Even if such samples be defined regarding the number of "cuttings" made, and their method of selection (i.e. random, systematic, etc.), the results obtained cannot be compared with any degree of certainty.

The present investigation is concerned essentially with the variation in trace element content of hill pastures and following upon such a study, with the establishment of a method of sampling, such that if the sampling be carried out in accordance with certain recommendations, the results obtained will be accurate within certain definite limits.

The following sections A and B describe the methods adopted in the collection of samples from the sub-areas, and the results obtained together with their statistical presentation.

In the statistical analysis all variations and errors have been expressed throughout as coefficients of variation, the/

the coefficient of variation being derived from the standard deviation according to the relationship

$$\text{Coefficient of variation} = \frac{100\sigma}{\bar{x}}$$

where σ is the standard deviation and $\bar{x}$ the mean of the observational data. The standard deviation, σ, is given by

$$\sigma = \sqrt{\frac{S(x-\bar{x})^2}{N-1}}$$

with the usual notation.

In tables giving distributions of variance, the variance, being equivalent to the square of the standard deviation, has been expressed as the square of the coefficient of variation.

(A) Normal Variation in Mixed Pasture.

In the analysis of a particular element in pasture herbage there are two main factors which may contribute to the normal variation (V), or variation of results about their mean value for samples taken within an area, namely (a) the variation contributed by the laboratory process of analysis (L), i.e. the error inherent in the analytical technique, and (b) the variation involved in the field (F), i.e. the sampling error. These three variations are connected by the following statistical relation:

$$V^2 = L^2 + F^2$$

If no error were involved in sampling (i.e. $F = 0$) the normal variation would be entirely due to the error inherent in the analytical technique, and could be reduced, if required, by replication of the analysis.

On/

On the other hand if a sampling variation does exist (i.e. $F \neq 0$) the total error involved in the result from a single observation (i.e. V) may be reduced either by increasing the number of samples taken, and analysing each individually, when the error of the mean V_1 is given by

$$V_1{}^2 = \frac{L^2}{n} + \frac{F^2}{n} = \frac{V^2}{n}$$

n being the number of observations; or, if L be small in comparison with F, by making a composite sample of n sub-samples and analysing the composite sample, when the error of estimation, V_2, is given by the relation

$$V_2{}^2 = L^2 + \frac{F^2}{n}$$

It is therefore necessary to carry out experiments to assess the normal variation, and to determine the relative magnitudes of the analytical and sampling errors.

Examination of the results obtained will indicate any replication necessary in the sampling or analytical technique in order to reduce the error of estimation. In this connection any replication contemplated must necessarily involve the following considerations:

(i) the permissible error to be attached to the result,

(ii) the relative magnitudes of the analytical and sampling errors, and (iii) the obvious unpracticality of taking large numbers of samples.

The conditions laid down for the collection of uncontaminated pasture and soil samples have been noted previously under "General Notes" at the beginning of this chapter/

chapter and these recommendations have been observed throughout all sampling experiments.

It is now proposed to consider and discuss the experiments conducted and the results obtained.

(a) Analytical Laboratory Error.

Each analytical method (as described in Chapter III) consists essentially of a chemical pretreatment, involving either an extraction (calcium, potassium, magnesium and manganese) or a fusion and concentration (cobalt, nickel and iron in pasture) or an extraction and concentration (cobalt and nickel in soil) followed with the exception of iron by a spectrographic method of analysis.

There are therefore two stages in the analytical procedure and errors associated with each stage contribute to the analytical error.

In addition, when the complete laboratory analytical process is considered, there is one further source of error, namely, sub-sampling of material in the laboratory.

The three stages then in the complete laboratory analytical process are:-

(i) Sub-sampling in the laboratory.

(ii) Chemical pretreatment.

(iii) Spectrographic analysis.

The determination of the total analytical error, i.e. the error involved in the complete laboratory analytical process for the elements investigated was obtained in the following/

following manner.

For the Lundegardh technique (calcium, potassium, magnesium and manganese) 20 aliquots (5g. each) were taken from a milled finely-ground typical pasture sample, and analysed according to the method previously described.

For the determination of the error involved in the estimation of cobalt and nickel (in pasture and soil) 20 aliquots of 20g. each were taken from typical pasture and soil samples respectively, and analysed.

From the results in each case estimates of the errors were derived.

The appropriate spectrographic errors were calculated from the results of replicate analyses (20 times) of a single sample, e.g. a solution in the case of the Lundegardh technique, and a concentrate in the case of cobalt and nickel.

An estimate was made of the errors associated with the combined chemical and spectrographic procedure for cobalt and nickel determinations again by replication of analysis (20 times) for a solution of known concentration.

The errors involved in the estimation of iron in pasture were also investigated.

In each case several sets of analyses were carried out, and the errors, expressed as coefficients of variation, and based on these sets, have been given previously in tables 1-3. A note is included in Appendix I giving the results from a typical set of pasture and soil analyses and the total analytical errors derived from the observations. From tables/

tables 1-3 it is seen that the total analytical errors for calcium, **pot**assium and manganese, are of the order of 6-7% while the error for magnesium is 12%.

For cobalt and iron in pasture the total analytical errors are 10% and the corresponding error for nickel is 12%; for cobalt and nickel estimations in soil extracts the total analytical errors are 10 and 12% respectively.

Table 4 below shews for each variate the distribution of the analytical errors (as variance) between the different treatments involved in the complete laboratory analytical procedure, and gives the approximate relation between the error of the spectrographic technique and the total analytical error.

TABLE 4. Distribution of Analytical Errors.

Variate	Variance			Spectrographic Error as percentage of Total analytical Error.
	Total	Spectro-graphic	Chemical (incl. sub-sampling in laboratory).	
Ca	36	25	11	69
K	36	9	27	25
Mg	144	100	44	69
Mn	49	25	24	51
Co	100	16	84	16
Ni	144	16	128	11

From the above table it is seen that the error inherent in the/

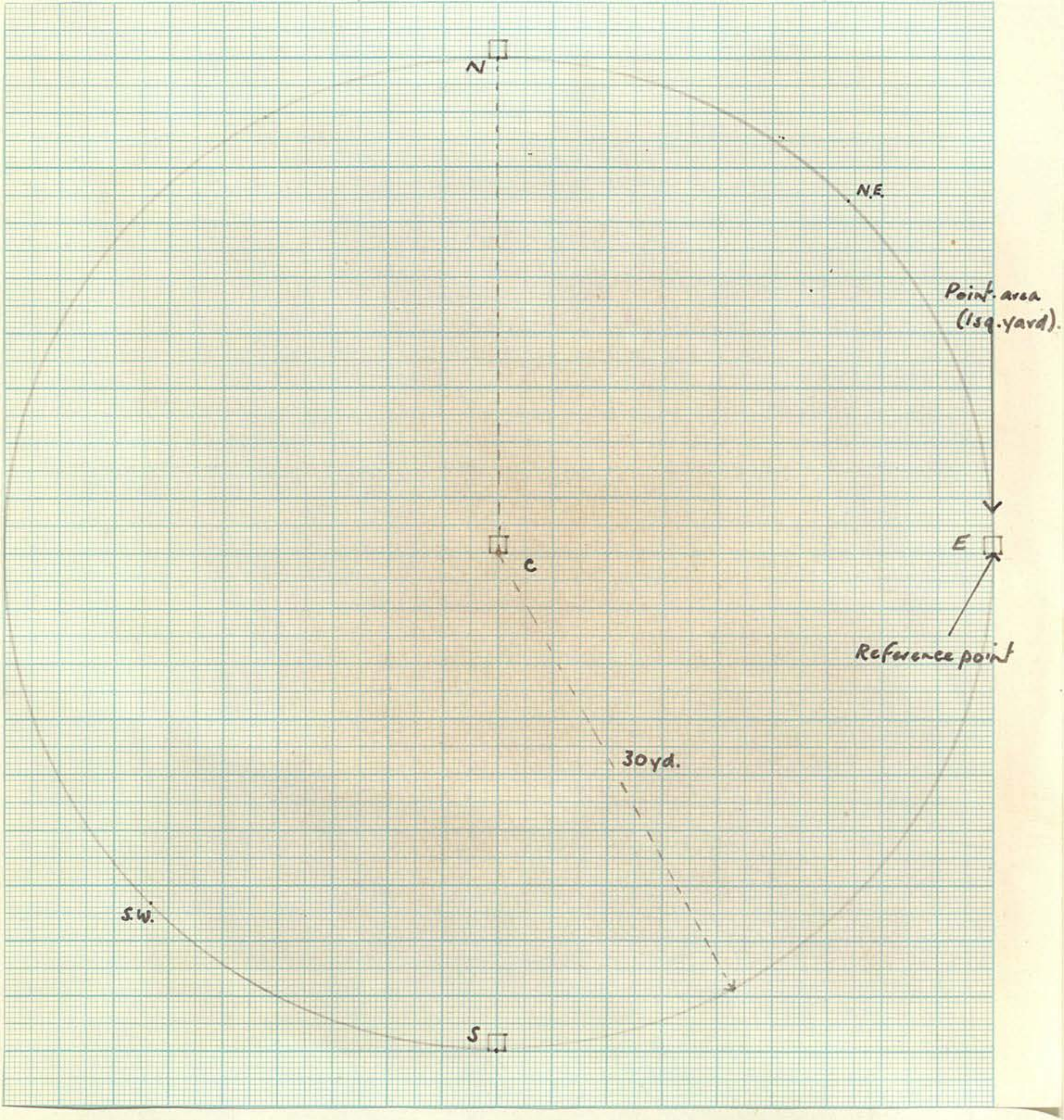


Fig. 9. Pictorial presentation of sampling scheme (September, 1949).

the spectrographic technique accounts for less than 50% of the total analytical error in the case of cobalt, nickel and potassium, and exceeds 50% of the total analytical error in the case of calcium, magnesium and manganese.

(b) Field Sampling Error.

The factors which are liable to affect and therefore contribute to the sampling error involved in the trace element analyses of hill pastures are:-

(i) Soil Type and Pasture Association,

(ii) Personal Error in Sampling, and

(iii) Seasonal Variation.

It is therefore necessary to determine the normal variation, and hence the relative magnitudes of the total analytical and sampling errors. The sampling error must also be considered in relation to the above factors.

Factors contributing to sampling error.

(i) Soil Type and Plant Association.

Preliminary sampling experiment, September, 1949.

In the initial experiment conducted in September, 1949, the sampling technique adopted was to select for each sub-area a central spot C, (see Fig. 9 opposite) and at each of 16 points corresponding to "points of the compass" and radiating 30 yards from this spot, a pasture herbage sample was taken from the point-area immediately in front of the point. No differentiation was made with regard to species and bracken was included. The samples were collected in accordance/

accordance with the recommendations, noted previously, except that in this case soil samples were taken only from alternate points, e.g. N, N.E., E, S.E., S, S.W., W, and N.W. Both pasture and soil samples were taken from the central spot C, thus yielding in all 17 herbage and 9 soil samples from each sub-area, for sub-areas numbers 2-4 and 6-9. In sub-area number 1, 8 pasture herbage samples were taken from point-areas corresponding to the compass points N, N.E., E, S.E., S, S.W., W, N.W. A sample was also taken from the central point C. The same point-areas were sampled by a different operator who obtained the samples by "plucking" as opposed to "clipping". Single soil samples were taken from the point-areas concerned.

A pictorial diagram of the sampling plan is indicated in Fig. 9.

Discussion of Results.

The analytical results are given in Appendix IIa. With regard to the pasture herbage, one sample collected from sub-area 1 (W, clipped) and one taken from sub-area 4 (N.N.W.) were rejected as unsuitable for analysis. In the case of soils, one sample from sub-area 7 was rejected. In the statistical treatment of the soil results the cobalt result for sample N. in sub-area 8 was not included in the analysis.

Pasture.

In general, the means for the different sub-areas, for a given variate, were found not to differ significantly; the/

the variation within sub-areas was, on the whole, sufficient to account for the variation between them. There were in most cases, however, two or three sub-areas which gave results markedly different from the others. In these exceptional areas, the variation tended to be very small and peculiar distributions were obtained. For example, the nickel results were found to have a very skew distribution. This was very largely on account of the large numbers of results below 1.00 in sub-areas 7, 8 and 9; in the other sub-areas results were spread out between 0 and 13 p.p.m. (with three very large values of 14.16, 17.48 and 29.92 p.p.m.) Regarding the results from sub-area 1, no significant differences were found between the means of results for the "plucked" and "clipped" samples. These results were therefore pooled.

In analysing the results, the best treatment appeared to consist in rejecting for each variate those sub-areas, in which the variation was unduly low, or in which the sample was suspected of being unrepresentative, and to base all calculations on the remaining sub-areas. These sub-areas were treated as though they had been sampled randomly, and, in each case they were treated as a homogeneous group, as the variation between them did not differ significantly from the variation within them.

With regard to the nickel results, it was found more convenient to multiply all results by 10, and take the logarithms. After taking logarithms the distributions approximated/

TABLE 5. Statistical Presentation of Results
of Pasture Analyses, September 1949.

Variate	Range of Concentration	Mean	Coefficient of Variation (%) One Observation	Sub-areas Chosen
s.f.a.	1.42-5.81	3.82%	23.2	2,3,4,6,7,8.
Ca	.14- .37	.23%	22.8	1,3,4,6,7.
K	.36-2.51	1.35%	30.0	2,3,4,6,7,8.
Mg	.06- .24	.14%	26.6	all sub-areas
Mn	333 - 944	581 ppm	25.0	3,4,6,7,8.
Co	.02- .92	0.30ppm	58.0	all sub-areas
Ni	.44-29.92	*2.64ppm	94.0	1,2,3,4,6.
Fe	52- 206	115 ppm	25.3	1,3,4,6,7,8,9.

* Median

approximated more to normality. Sub-areas 7, 8 and 9 were still markedly different from the rest in that they gave on the whole lower and less variable results. Taking sub-areas 1, 2, 3, 4, and 6 as a group, however, it was found that the mean, based on 78 cases, for the value of the logarithm was 0.515 with a standard deviation of 0.374. This indicated a median value for the nickel results in sub-areas 1-6 of 2.64 with a coefficient of variation of the order of 90%.

Table 5 (opposite) gives for each variate, the range of concentration, the mean (or median) and the coefficient of variation for one observation. In each case the sub-areas, on which the statistics are based, are indicated.

From the table opposite, and in consideration of the results obtained, it may be seen that:

(1) No significant differences were found between the means of results for the "plucked" and "clipped" samples. There is, however, always an increased risk of contamination in "plucking", and this practice is therefore not to be recommended.

(2) Very high coefficients of variation are found for cobalt and nickel of the order of 60% and 90% respectively.

(3) With the exception of cobalt and nickel, the variates, all have coefficients of variation in the neighbourhood of 25%.

(4) The errors inherent in the complete analytical process in the case of cobalt and nickel are negligible in comparison with the errors of sampling. (Cf. Tables 2 and 5).

(5)/

(5) Mention has already been made of discrepancies in the results (particularly nickel) which may be explained partly by the systematic method of sampling adopted. With regard to the nickel results there is one further possible explanation.

In September, 1949, additional samples were taken of certain species from different patches on the hillside in connection with an investigation into the composition of particular species, to be discussed later. The analytical results for the species collected in September, 1949, are given below for convenience.

Extract from Table 20.

Composition of Different Species sampled from Selected Patches.

Species	Variate							
	% s.f.a.	% Ca	% K	% Mg	p.p.m. Mn	p.p.m. Co	p.p.m. Ni	p.p.m. Fe
Bracken (sub-area 2)	7.09	.21	2.76	.41	70	.06	17.14	90
Bracken (sub-area 4)	7.17	.30	3.17	.44	158	.09	15.25	114

From the above extract from table 20 the bracken is seen to be relatively high in nickel content. In the sampling experiment carried out, no differentiation was made with regard to species, but bracken was included in the samples taken from point-areas on which it grew. The inclusion of bracken, in this case, in pasture samples, by virtue of its greater/

greater relative density would tend to increase the nickel content of the samples. Further there was a reasonable growth of bracken on sub-area 2, and on sub-areas 1, 3 and 4 there were patches of bracken although sub-area 6 was practically free, and the pasture samples from areas 1, 2, 3 and 4 are characterised by high nickel contents of the order of 10 p.p.m. or more. It is therefore suggested that while the discrepancies in the nickel results referred to earlier may be partly explained by the systematic method of sampling adopted, the relatively high nickel contents and consequently higher variation obtained are attributable to the inclusion of bracken in the samples.

Soil.

In the case of cobalt the figures for the mean and coefficient of variation were not very different from those obtained from the pasture analyses (the mean is a little lower). Generally the sub-areas did not differ very markedly, and the results for all areas were therefore pooled.

The nickel results were on the other hand, different from those based on the pasture analyses. Both the mean and coefficient of variation were much lower. In order to compare satisfactorily the pasture and soil results, the mean and coefficient of variation for nickel were calculated solely from the figures for sub-areas 1, 2, 3, 4 and 6 (see table 5).

Table 6 (next page) gives for each variate, the range of acetic-acid soluble cobalt and nickel, the mean and the coefficient/

coefficient of variation for one observation.

TABLE 6. Statistical Presentation of Results of Soil Analyses, September, 1949.

Variate	Range of Concentration (p.p.m.)	Mean	Coefficient of Variation (%) One Observation	Sub-areas chosen
Co	0.03-1.09	0.24 p.p.m.	60.6	All sub-areas
Ni	0.28-2.62	0.58 p.p.m.	69.5	1, 2, 3, 4,6.

From the results in the above table it is seen that:

(1) High variations are found for cobalt and nickel in acetic-acid extracts of the order of 60 to 70%.

(2) The errors inherent in the analytical process are negligible in comparison with the errors of sampling (Cf. tables 2 and 6).

(3) The variation of cobalt in acetic-acid extracts is of the same order as that in pasture; with regard to nickel the variation is significantly less in soil extracts than in pasture.

Combining both sets of results (i.e. tables 5 and 6) the indications are that considerable fluctuations are to be found in the cobalt and nickel content of pastures over comparatively small areas. The variation in cobalt content of pasture has been found to be of the same order as that obtained for soil (acetic-acid extracts) while the variation in nickel content of pasture is greater than that of the corresponding/

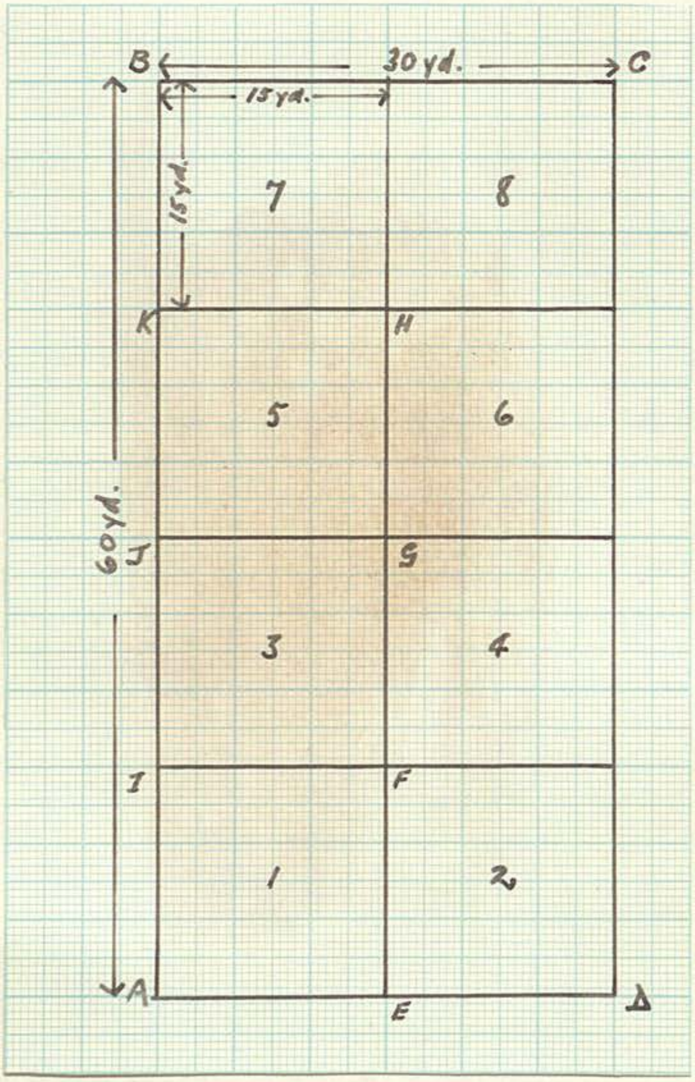


Fig. 10. Dimensions of rectangular sections chosen for sampling scheme in June and September of 1950.

corresponding soil extracts.

The variations in manganese and iron contents in pasture are far less than those for cobalt and nickel and are approximately of the same order as those obtaining for the major elements, i.e. approximately 25%.

Sampling Experiments in June and September, 1950.

Subsequent experiments were carried out in June and September of 1950, in order to assess any seasonal variation (to be discussed later), and to permit the investigation of additional sub-areas of differing soil type, situated on the Hairney Law across the valley from Fasset Hill. These three new areas were sampled in place of sub-areas 1, 2 and 4, which with the exception of sub-area 2 were very similar to sub-areas 3, 7 and 8.

In consequence of the variations obtained for the cobalt and nickel results in the preliminary sampling experiments the method of sampling was changed from a systematic to a randomised one for all the areas examined, and the number of pasture and soil samples taken from each sub-area increased. No differentiation was made with regard to species, except that bracken was excluded. Apart from these modifications, the usual procedure was adopted in the collection of pasture and soil samples.

The sampling scheme adopted (see Fig. 10) consisted in selecting a rectangle ABCD, measuring 60 yd. by 30 yd. in the centre of each sub-area. This rectangle was sub-divided into 8 smaller rectangles, numbered 1 to 8, each measuring 15 yd./

15 yd. by 15 yd. Wooden posts were used to locate the points A, B, C, D, E, F, G, H, I, J and K of the larger rectangle.

Three pairs of operators were engaged in the sampling scheme, each pair being allocated one random point in each of the smaller rectangles. This was achieved by allowing the herbage operator of each pair to draw numbered cards from three boxes designated respectively "Rectangle", "X" and "Y". The box designated "Rectangle" contained 8 cards numbered 1 to 8, while the "X" and "Y" boxes each contained cards numbered 1 to 15. Herbage operator A, for example, would select a particular small rectangle by drawing a card from the "Rectangle" box, and the corresponding co-ordinates for a point within that rectangle by drawing cards from each of the "X" and "Y" boxes.

Only the "X" and "Y" cards were returned to their respective boxes, and these boxes reshuffled. The same operator would than obtain co-ordinates for a point in another small rectangle by drawing a card from the remaining seven in the "Rectangle" box, an "X" and "Y" card and so on for the remainder of the small rectangles. On completion of the process, herbage operator A (and therefore the A pair of operators) had been allocated one point per small rectangle, the co-ordinates of each point being referred to the bottom left-hand corner of each small rectangle. Prior to the actual sampling, each pair of operators was given a small chart, giving the co-ordinates for each small rectangle.

With/

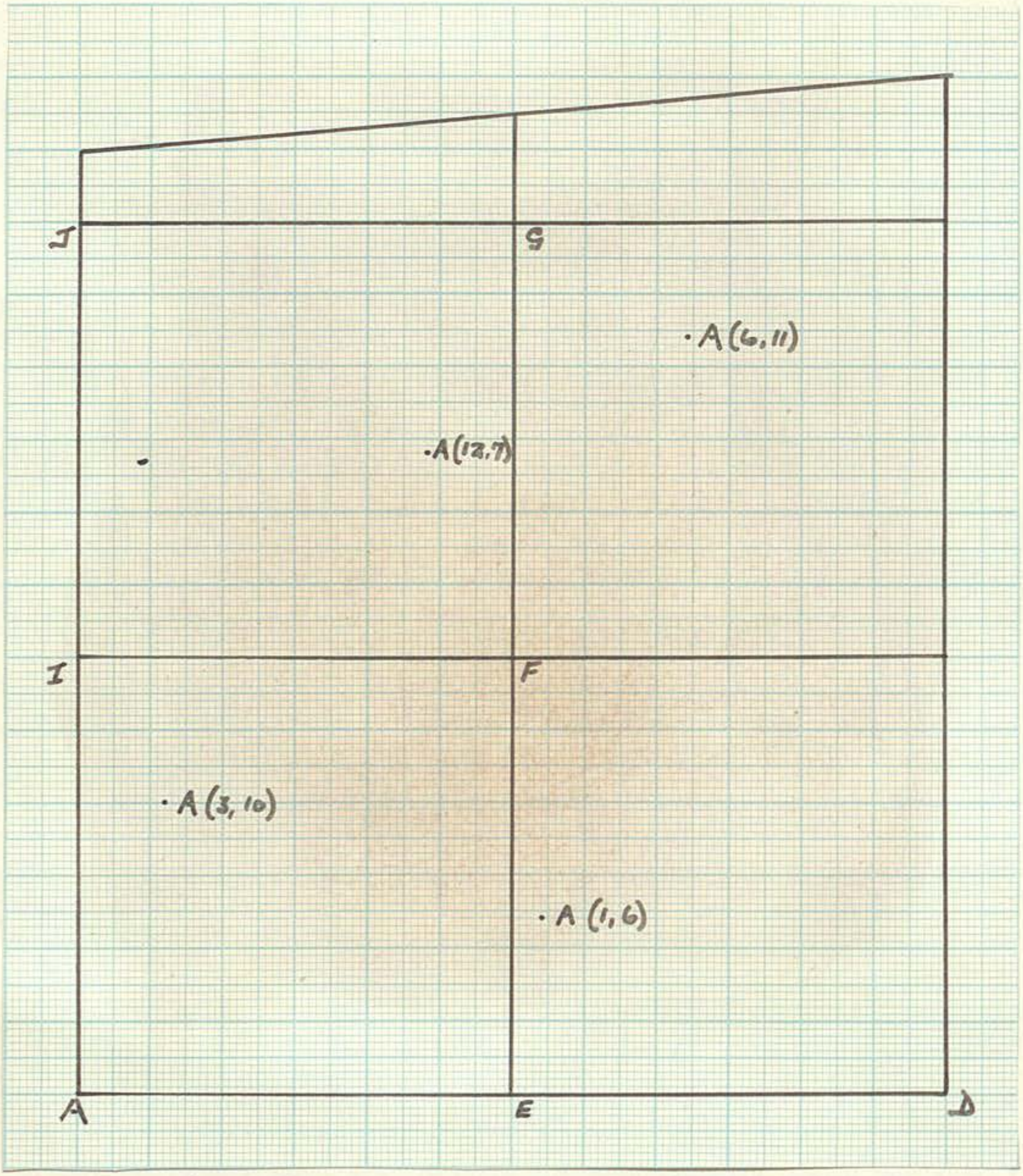


Fig. 11. Part of typical sampling chart (Operator A).

With three pairs of operators 24 random points were chosen, thereby yielding 24 pasture and 24 soil samples per sub-area. When only two pairs of operators were available, co-ordinates were obtained in the same way for three points in each small rectangle. One pair of operators then sampled the left half, and the other pair the right half of the large rectangle.

A measuring tape was used to locate each point, and the sampling confined to the point-area immediately in front of the point. Fig. 11 shews part of a typical sampling chart.

Discussion of Results.

The analytical results are given in Appendix II (b and c).

Sampling in June, 1950.

Of the pasture samples taken, one sample (B7 in sub-area 9) was rejected as being unsuitable for analysis, and for the same reason, the following soil samples were also rejected; C4 (sub-area 8), A7 and 8 (sub-area 9), and A2, B4 and 7, and C4 (sub-area 10).

An examination of the pasture and soil results indicated considerable differences between sub-areas 10-12 on Hairney Law, and the sub-areas 3 and 6-9 on Fasset Hill. In the former areas the pasture analyses were characterised by much higher means for the trace elements cobalt, nickel and iron, than the latter, but by much lower means for the silica-free-ash, calcium, potassium, magnesium and manganese contents. Similarly the means for the acetic-acid soluble cobalt and nickel were higher in sub-areas 10-12. The variation between sub-areas 3 and 6-9 was not very great. It was noticeable that/

TABLE 7. Statistical Presentation of Pasture and Soil Results, June 1950.

Variate	Range of Concentration	Mean	Coefficient of Variation (%)
			One Observation
s.f.a.	2.56-5.28 (1.84-3.37)	4.08 (2.61)%	12 (14)
Ca	.10- .29 (.09-.18)	.16 (.11)%	13 (13)
K	.69-2.08 (.58-1.22)	1.38 (.81)%	13 (18)
Mg	.05- .22 (.05-.16)	.13 (.09)%	15 (19)
Mn	179-756 (149 - 463)	487 (300) p.p.m.	22 (30)
Co	.02- .46 (.04-.30)	.09 (.15) p.p.m.	37 (28)
Ni	.40-4.38 (.52-9.34)	1.06 (2.35) p.p.m.	42 (42)
Fe	45- 421 (100 - 434)	167 (249) p.p.m.	22 (22)
Co (Soil)	.05- .71 (.10-1.14)	.21 (.39) p.p.m.	41 (37)
Ni (Soil)	.20-2.97 (.32-7.06)	.66 (1.19) p.p.m.	44 (46)

that in each of the sub-areas 10, 11 and 12 very high values for iron were recorded.

The main difference between the figures for June, 1950, and those for September, 1949, is that the coefficients of variation for the 1950 results are lower, and in some cases considerably lower. This may be partly explained by the different method of sampling employed.

As in the results for September, 1949, cobalt and nickel are by far the most variable elements, with coefficients of variation around 40%. It was also noticeable that no exceptionally high nickel contents were recorded for pasture growing on sub-area 3, in comparison with the corresponding results in September, 1949.

Table 7 (opposite) gives for each variate the range of concentration, mean, and coefficient of variation for one observation. The unbracketed figures refer to sub-areas 3 and 6-9, while those in brackets refer to sub-areas 10-12, and are given for comparison. The above coefficients of variation give a measure of the variation within sub-areas and not between them, although in the case of sub-areas 3, and 6-9, this latter is not very large.

Sampling in September, 1950.

Of the pasture samples taken the three samples A2 (sub-area 3), A11 (sub-area 10) and A7 (sub-area 11) were rejected as being unsuitable for analysis. For samples B5 and 6 (sub-area 11) sufficient material was not available for the determination of cobalt, nickel and iron. Of the soil samples/

samples taken B5 (sub-area 6), and samples A1, B3 and 8, C1, 5, 6, 7 and 8 (all from sub-area 9) were rejected as being unsuitable for analysis.[*] Soil samples were not available on this occasion from sub-areas 11 and 12. (Table 8)

The results are much the same as those for June, 1950. In this case the median of each variable was found separately for each sub-area. These are given below in table 8. The trace element results are expressed in p.p.m. while the results for the silica-free-ash and major elements are expressed in percentages.

TABLE 8.

Sub-area	Variate									
	Pasture								Soil	
	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe	Co	Ni
3	5.26	.16	1.99	.22	719	.08	1.32	88	.11	.42
6	4.67	.20	1.75	.23	649	.05	1.22	95	.48	.43
7	4.34	.16	1.62	.19	593	.05	.77	87	.13	.56
8	4.18	.15	1.57	.19	802	.17	1.07	130	.32	.53
9	2.75	.16	.87	.16	273	.16	.62	92	.22	.67
10	2.48	.16	.75	.11	404	.24	2.70	269	.45	.99
11	2.53	.12	.89	.10	286	.16	1.18	196	-	-[*]
12	2.80	.07	.97	.14	220	.13	.84	158	-	-[*]
Average	3.63	.15	1.30	.17	493	.13	1.22	139	.28	.60

As/

As in June, 1950, there were clearly very large differences between sub-areas. In the main for the pasture results, the differences were chiefly between sub-areas 3, 6, 7 and 8 on the one hand and 9, 10, 11 and 12 on the other. Taking the sub-areas 3, 6 and 7 as a standard, the figures underlined (see table 8) are those which are either very low in the case of silica-free-ash, calcium, potassium, magnesium and manganese, or very high in the case of cobalt, nickel and iron.

Table 9 below gives the range of concentration and the coefficient of variation for one observation within sub-areas (averaged over all sub-areas) for each variable. It is seen that cobalt, nickel and iron give the most variable results.

TABLE 9. Statistical Presentation of Results of Pasture and Soil Analyses, September, 1950.

Variate	Range of Concentration	Coefficient of Variation (%) One Observation
s.f.a.	1.18 - 6.56 %	15
Ca	.05 - .26 %	14
K	.11 - 2.37 %	17
Mg	.07 - .41 %	23
Mn	45 - 1206 p.p.m.	24
Co	.02 - .58 p.p.m.	42
Ni	.16 - 5.69 p.p.m.	38
Fe	13 - 432 p.p.m.	32
Soil Co	.02 - 2.40 p.p.m.	44
Soil Ni	.21 - 1.78 p.p.m.	24

General/

General Discussion of Results for sampling experiments in 1949 and 1950.

When the results from the sampling experiments in September, 1949, and in June and September of 1950 are considered together, the following facts emerge:-

(1) Cobalt and nickel have been found to be the most variable of the elements investigated, and the variations in the content of these elements in mixed pasture have been shewn to be as great as those in acetic-acid extracts of soil samples taken at the points where the pasture was cut. The analytical results for cobalt and nickel for both pasture herbage and soil have coefficients of variation of the order of approximately 25 to 60% and 25 to 90% respectively.

Manganese and iron are not so variable as cobalt and nickel, and the results give coefficients of variation approximately intermediate in value between those obtained for the major elements calcium, potassium and magnesium, and those for the trace elements cobalt and nickel, i.e. about 20 to 30%. The results for the major elements and silica-free-ash, with the exception of magnesium, give relatively low coefficients of variation of the order of 13 to 20%. The normal variation for magnesium is slightly greater. The variations for all variates are generally of the same order of magnitude for all the areas examined, irrespective of soil type or pasture association.

(2) The coefficients of variation obtained in September, 1949, were higher than the corresponding values in June and September/

September of 1950. These initial larger values may be partly explained by the systematic method of sampling adopted at that time. The greatest fall in the coefficient of variation between the 1949 and 1950 results is observed for nickel in pasture herbage, the coefficient of variation falling from 94% to less than 50%. It has been suggested that the very high nickel contents of the pasture samples taken in September, 1949, and the correspondingly higher variation obtained, may be attributed to the inclusion in the samples of bracken from the sub-areas concerned, the bracken being characterised by nickel contents of the order of 15 - 17 p.p.m.

(3) Omitting the variations obtained in September, 1949, for reasons given under (2) above, it is seen that for mixed pasture herbage and soil samples for June and September of 1950, the coefficients of variation for one observation or normal variations for each variate fall within the following range:-

	Variate	Range of Normal Variation (%)	Accepted upper limit of normal variation (%)
Pasture	s.f.a.	12 - 15	20
	Ca	13 - 14	20
	K	13 - 18	20
	Mg	15 - 23	30
	Mn	22 - 30	35
	Co	28 - 42	50
	Ni	38 - 42	50
	Fe	22 - 32	35
Soil	Co	37 - 44	50
	Ni	24 - 46	50

Taking the values in the above right-hand column as the upper limits of the normal variation for small uniform areas, table/

table 10 below shews the approximate distribution of the sampling and analytical errors (as variance).

TABLE 10. Distribution of Sampling and Analytical Errors.

Variate	Normal Variation (%)	Variance		
		Total	Sampling	Total Analytical
s.f.a.	20	400	364	36
Ca	20	400	364	36
K	20	400	364	36
Mg	30	900	756	144
Mn	35	1,225	1,176	49
Pasture and soil Co	50	2,500	2,400	100
Pasture and soil Ni	50	2,500	2,356	144
Fe	35	1,225	1,125	100

It is obvious from the above table that the total analytical errors are very small in comparison with sampling errors in each case, and especially for the variates cobalt and nickel.

(4) Considerable differences have been found in the mineral composition of pasture samples and in the acetic-acid soluble cobalt and nickel in soil samples taken from Hairney Law (sub-areas 10, 11, 12) compared with the corresponding results obtained/

obtained from Fasset Hill (sub-areas 3, 6, 7, 8, 9). Comparing the results from the sub-areas on Hairney Law with those obtained from the sub-areas on Fasset Hill, the former are characterised by much higher means for cobalt, nickel and iron in pasture and for the extractable cobalt and nickel in soils, but by much lower means in pasture for the major elements and manganese.

It would be reasonable to expect differences between the results for Hairney Law and Fasset Hill, for the soils on Hairney Law (sub-areas 10, 11, 12) can be classed together as a soil type distinct from the immature brown earth type (sub-areas 3, 6, 7, 8) and peat formation (sub-area 9) on Fasset Hill. Sub-areas 10 and 11, and especially 12, are characterised by podsolisation and this would appear to be indicated by the high pasture iron values recorded for these areas.

It was noticeable, however, that in September, the pasture results for sub-area 9 in the case of potassium, manganese and cobalt, approximated more to those obtained on the Hairney Law, although the soil results were comparable, as in June, with the other soil results from Fasset Hill. It is suggested that the increase in cobalt content and the decreases in potassium and manganese contents are connected with the stages of growth of the dominant species growing on that sub-area.

(ii) Personal Error in Sampling.

With regard to the sampling of pasture herbage and soil from the different sub-areas in 1949 and 1950, it must be noted/

noted that different operators were engaged for this purpose during the experiments. In September, 1949, one pair of operators took the pasture and soil samples from the point-areas where the soils were sampled, while an additional operator was required for the collection of pasture samples at intermediate points. In June and September of 1950 three pairs and, in some cases, two pairs of operators were required for each sub-area.

Consideration must therefore be given to the possible introduction of a personal error in sampling, and its effect on the variations obtained in previous experiments. It was therefore decided to determine what differences, if any, in the results found for mixed pasture samples obtained by different operators from the same point-areas, could be attributed to slight variations in individual sampling technique. The sampling of soils was also tested for this possible error.

In an attempt to assess any significant differences between individual herbage and soil operators respectively, each operator, in either case, was given an equal chance of selecting the same sample from 16 point-areas within a sub-area and the following procedure was adopted:-

In June, 1951, in each of the sub-areas 3, 9 and 12, rectangular areas were chosen and 16 random points taken within each. The rectangles conformed to the same relative dimensions as those described in the earlier experiments but were somewhat larger in area, as follows:-

Sub-area 3: 2 acres
" 9: 2 acres
" 12: $3\frac{1}{2}$ acres.

Each/

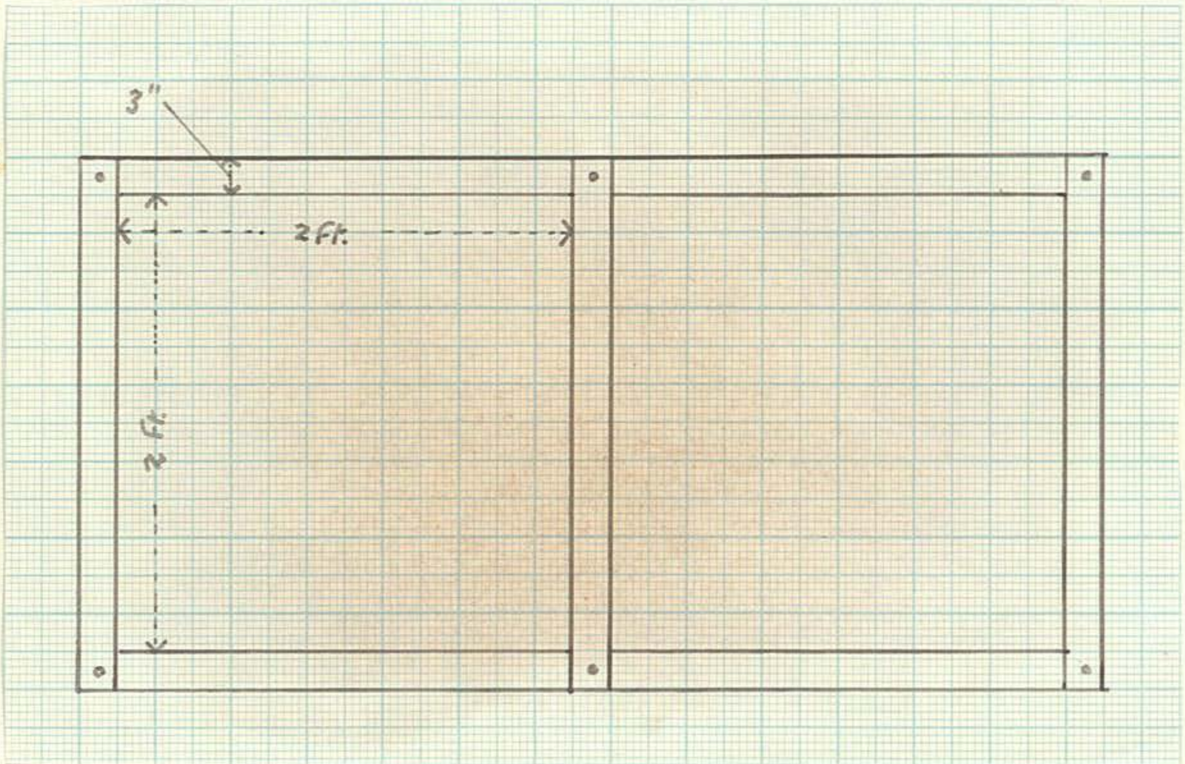


Fig. 12. Dimensions of wooden frame used in sampling for assessment of personal error.

Each rectangle was subdivided into 8 smaller rectangles as in the previous experiments in June and September of 1950, and 2 random points were taken in each smaller rectangle. Two strips each measuring 2 feet by 2 feet were taken around each point with the aid of a special but simply constructed wooden frame (see Fig. 12). The frame was placed lengthwise in the same direction as the shorter side of the large rectangle about each point. The strips were designated "l" and "r" (for left and right respectively). By random selection one operator was given the "L" strip and the other operator the "r" strip. Different operators A, B and C sampled the sub-areas; for example, in sub-area 12 operators A and B, in sub-area 9 operators B and C, and in sub-area 3 operators A and C. In the same way, again by random selection, soil samples were drawn from the strips, using the same random points by operators D and E in sub-area 12 and operators F and G in sub-area 9.

Discussion of Results

The analytical results are given in Appendix IId.

With regard to pasture, in sub-area 3, only 31 observations were available in the case of cobalt, nickel and iron, while in sub-area 9, 4 samples were discarded as being unsuitable for analysis. In the case of soils, 10 samples from sub-area 9 were discarded.

Pasture

Table 11 below gives for each variate the totals for the individual operators for the three sub-areas. There are clearly/

TABLE 11. Pasture Herbage.

Sub-area	Statistics	Variate							
		%				p.p.m.			
		s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
12	Total (A)	54.08	1.52	18.81	2.00	5615	2.74	40.30	3763
	Total (B)	51.22	1.55	17.59	1.83	4987	2.83	45.80	3839
	Mean of sub-area	3.29	.10	1.14	.12	331	.17	2.69	238
9	Total (B)	34.81	1.01	12.63	2.58	3806	2.63	25.59	3299
	Total (C)	36.09	1.07	12.16	2.70	3376	2.27	28.73	2789
	Mean of sub-area	2.53	.07	.89	.19	257	.18	1.94	217
3	Total (A)	68.96	2.02	27.44	2.48	7308	2.13	19.79	2796
	Total (C)	67.32	2.06	26.30	2.64	6517	1.98	25.68	2656
	Mean of sub-area	4.26	.13	1.68	.16	432	.13	1.47	176

clearly no significant differences and the totals are surprisingly close. The results have been pooled and the means are included in the table.

Soil

Table 12 gives for each variate the totals for the individual operators for the two sub-areas sampled. There are no significant differences: the results were therefore pooled and the means are included in the table.

TABLE 12. (Soil)

Sub-area	Statistics	Variate p.p.m. Co	 p.p.m. Ni
12	Total (D)	2.24	12.90
	Total (E)	2.69	13.90
	Mean of sub-area	.15	.84
9	Total (F)	1.70	9.26
	Total (G)	1.78	11.50
	Mean of sub-area	.16	.94

With regard to the above results for both pasture and soil, the coefficients of variation have not been given but these are of the same order of magnitude as those obtained previously, the results for cobalt and nickel again being the most variable.

Tests/

Tests of significance in relation to the above results.

The t test of significance, as given by Chambers (1948), has been applied to the above results, where, for the comparison of small numbers of observations (<50)

$$t = \frac{(\bar{x}_1 - \bar{x}_2)\sqrt{\frac{1}{N^1}}}{\sqrt{S({x_1}^2) - \frac{(S(x_1))^2}{N_1} + S({x_2}^2) - \frac{(S(x_2))^2}{N_2}}}$$

The above expression for t, with the usual notation, applies to N_1 observations of the variable x_1 and N_2 observations of the variable x_2, and

$$N^1 = \frac{N_1 + N_2}{N_1 \,.\, N_2 \,.\, (N_1 + N_2 - 2)}$$

R.A. Fisher (1936) in Table IV gives values of t corresponding to a probability $P = 0.05$ for different values of n, the number of degrees of freedom, in this case, $N_1 + N_2 - 2$. If the calculated value of t is greater than that given in the table for the appropriate value of n, then the means are significantly different.

Table 13 below gives in the comparison of results for the different operators the observed or calculated values of t, and those obtained from R.A. Fisher's table.

TABLE 13./

TABLE 13.

Sub-area and Operators.	Variate	t observed	t theoretical
Sub-area 12	s.f.a.	.74	2.042
Herbage	Ca	.138	"
operators	K	1.165	"
A and B.	Mg	.91	"
	Mn	1.69	"
	Co	.516	"
	Ni	.65	"
	Fe	.27	"
Soil operators	Co	.94	2.042
D and E.	Ni	.67	"
Sub-area 9	s.f.a.	.38	2.056
Herbage	Ca	.99	"
operators	K	.50	"
B and C.	Mg	.31	"
	Mn	.78	"
	Co	1.80	"
	Ni	.70	"
	Fe	.47	"
Soil operators	Co	.17	2.086
F and G.	Ni	1.17	"
Sub-area 3	s.f.a.	.48	2.042
Herbage	Ca	.22	"
operators	K	.91	"
A and C.	Mg	.81	"
	Mn	1.82	"
	Co	.06	2.045
	Ni	1.73	"
	Fe	.15	"

It is seen from the above table that in every case the observed 't' value is considerably less than the theoretical 't' value, indicating that no significant differences exist between the/

the results of pasture and soil samples collected by different operators sampling from the same point-areas.

It may therefore be concluded that any personal error may be ignored.

(iii) Seasonal Variation.

In the investigation of seasonal variation, sub-areas 3, 6, 7, 8 and 9 have been grouped together, the variations within these sub-areas being sufficient on each occasion to account for the variation between them. In the same way sub-areas 10, 11 and 12 have been grouped together, except that in this case only soil results for sub-area 10 are available for comparison.

The following tables (14 - 17) give for pasture and soil, for each variate examined, the means, coefficients of variation (to the nearest whole number), the number of samples on which the statistical calculations are based, and the errors of the means. The coefficients of variation are those given previously in tables 5, 6, 7 and 9.

TABLE 14./

TABLE 14. Pasture Herbage.

(Statistics based on results from sub-areas 3, 6, 7, 8 and 9.)

Season	Variate	Mean	Coefft. of Variation	No. of samples	Error of Estimation	
					% of mean	actual
Sept. 1949	s.f.a.	3.52	23	85	2.5	± .09%
June 1950	%	4.08	12	119	1.1	.04%
Sept. 1950		4.24	15	119	1.4	.05%
Sept. 1949	Ca	.21	23	85	2.5	± .01%
June 1950	%	.16	13	119	1.2	.00%
Sept. 1950		.16	14	119	1.3	.00%
Sept. 1949	K	1.21	30	85	3.3	± .04%
June 1950	%	1.38	13	119	1.2	.02%
Sept. 1950		1.56	17	119	1.5	.02%
Sept. 1949	Mg	.14	27	85	2.9	± .00%
June 1950	%	.13	15	119	1.4	.00%
Sept. 1950		.20	23	119	2.1	.00%
Sept. 1949	Mn	532	25	85	2.7	±14ppm.
June 1950	p.p.m.	487	22	119	2.0	10 "
Sept. 1950		607	24	119	2.2	13 "
Sept. 1949	Co	.28	58	85	6.3	± .02 "
June 1950	p.p.m.	.09	37	119	3.4	.00 "
Sept. 1950		.10	42	119	3.9	.00 "
Sept. 1949	Ni	1.94	94	85	10.2	± .19 "
June 1950	p.p.m.	1.06	42	119	3.9	.04 "
Sept. 1950		1.00	38	119	3.5	.04 "
Sept. 1949	Fe	112	25	85	2.7	± 3 "
June 1950	p.p.m.	167	22	119	2.0	3 "
Sept. 1950		98	32	119	2.9	2 "

TABLE 15. Pasture Herbage.

(Statistics based on results from sub-areas 10, 11 and 12.)

Season	Variate	Mean	Coefft. of Variation	No. of samples	Error of Estimation	
					% of mean	actual
June 1950	s.f.a.	2.61	14	72	1.7	±.04%
Sept. 1950	%	2.60	15	70	1.8	.05%
June 1950	Ca	.11	13	72	1.5	±.00%
Sept. 1950	%	.12	14	70	1.7	.00%
June 1950	K	.81	18	72	2.1	±.02%
Sept. 1950	%	.87	17	70	2.0	.02%
June 1950	Mg	.09	19	72	2.2	±.00%
Sept. 1950	%	.11	23	70	2.8	.00%
June 1950	Mn	300	30	72	3.5	±11p.p.m.
Sept. 1950	p.p.m.	303	24	70	2.9	9 "
June 1950	Co	.15	28	72	3.3	±.01 "
Sept. 1950	p.p.m.	.18	42	68	5.1	.01 "
June 1950	Ni	2.35	42	72	4.9	±.12 "
Sept. 1950	p.p.m.	1.57	38	68	4.6	.07 "
June 1950	Fe	249	22	72	2.6	± 7 "
Sept. 1950	p.p.m.	208	32	68	3.9	7 "

TABLE 16. Soil.

(Statistics based on results from sub-areas 3, 6 7, 8 and 9.)

Season	Variate	Mean	Coefft. of Variation	No. of samples	Error of Estimation	
					% of mean	actual
Sept. 1949	Co	.24	61	70	7.4	±.02 p.p.m.
June 1950	p.p.m.	.21	41	117	3.8	.01 "
Sept. 1950		.25	44	111	4.1	.01 "
Sept. 1949	Ni	.58	70	44	10.4	±.06 "
June 1950	p.p.m.	.66	44	117	4.1	.03 "
Sept. 1950		.52	24	111	2.3	.01 "

TABLE 17. Soil.

(Statistics based on results from sub-area 10.)

Season	Variate	Mean	Coefft. of Variation	No. of samples	Error of Estimation	
					% of mean	actual
June 1950	Co	.39	23	20	5.1	±.02 p.p.m.
Sept. 1950	p.p.m.	.45	44	24	9.0	.04 "
June 1950	Ni	.96	12	20	2.7	.03 "
Sept. 1950	p.p.m.	.99	24	24	4.9	.04 "

Discussion of Results

Coefficients of Variation.

It was noted earlier that the coefficients of variation obtained in the experiments in June and September of 1950 for both pasture herbage and soil results were generally of the same order, but lower for all variates, than those obtained in September 1949. This effect was attributed to the method of sampling adopted in 1949, and also in the case of the pasture nickel results for that period to the inclusion of bracken in the sampling.

Mean Contents.

Regarding the analytical results for pasture herbage there are differences in the mean content for all variates between the times of sampling, with the exception of calcium and nickel (table 14) and silica-free-ash and manganese (table 15) for June and September, of 1950. (These means have been underlined in the tables.) The corresponding differences for silica-free-ash (table 14) and calcium (table 15) are very small.

There are significant differences in the analytical figures for the September periods of 1949 and 1950; among the major elements the potassium and magnesium contents are higher in September, 1950, this rise being reflected in the figures for silica-free-ash, while the calcium content is lower; among the trace elements manganese has increased, while the cobalt, nickel and iron contents have fallen.

If/

If the results for the summer period of 1950 (i.e. June, 1950 and September, 1950) are compared, for both tables 14 and 15, it is seen that of the major elements, the potassium and magnesium contents have risen, while the calcium content remains reasonably constant. Of the trace elements, manganese and cobalt have risen, while the iron content has fallen. The nickel results are not consistent shewing no significant difference in table 14 and a decrease in content in table 15. The above differences are however relatively small with the possible exception of magnesium, iron and nickel.

With regard to the soil results (table 16), there are small but significant differences between the mean contents of both cobalt and nickel for the June and September results of 1950 only. Regarding the results from sub-area 10 (table 17) the differences are obviously not significant.

The above statistics have been calculated from a large number of individual observations (e.g. 85, 119, in table 14 and 44, 70, 117, 111, in table 16), each involving a complete laboratory analysis.

Such replication in analytical technique and sampling procedure however could not be envisaged in normal practice, on account of (a) the relative magnitude of the analytical laboratory error and the undesirability of replicating analyses, and (b) the obvious necessity of keeping the sampling within practicable limits.

Accordingly the final formulation of any sampling technique/

technique must be concerned with the bulking of smaller numbers of sub-samples to give composite samples, each such composite sample being subjected to a single analysis. The errors attached to the results for a single composite sample will therefore be greater than those obtaining for the errors of the means in tables 14, 15 and 16, in each case.

For example, if the normal variation in respect of a particular element in mixed pasture herbage be V% (as coefficient of variation) and the laboratory analytical error (as coefficient of variation) be L%, the error to be attached to the result of an individual analysis of a composite sample of n sub-samples is given by V_1 where

$$V_1^2 = L^2 + \frac{V^2 - L^2}{n}$$

Obviously with no replication in the analytical technique V_1 will be greater than L and its value will depend upon n and the relative magnitudes of V and L.

For the trace elements, where V is large compared with L, if n is relatively small (e.g. of the order of 25), V_1 will be in reasonable excess of L. In the case of cobalt, for example, V is of the order of 50% and L = 10% (Ch. III, sec. (iv).) whence for n = 25, V_1 = 14%.

For the major elements V does not so greatly exceed L, and therefore V_1 approximates to L. In the case of potassium V is of the order of 20% and L = 6% (Ch. III, sec. (iv).) whence for n = 25, V_1 = 7%.

Referring again to Ch. III, sec. (iv), the values of L for/

for the variates concerned are as follows:-

Major Elements: Ca $L = 6\%$
K $L = 6\%$
Mg $L = 12\%$

Trace Elements: Mn $L = 7\%$
Co $L = 10\%$
Ni $L = 12\%$
Fe $L = 10\%$

When these values are considered with regard to the above formula for V_1, and the differences between the mean contents for June and September of 1950, it is apparent that such differences will assume less, if any importance, in relation to the sampling errors, with the exception of the differences in iron, and possibly magnesium and nickel content. The differences however would appear to be relatively small.

(B) Normal Variation within Particular Species and Differences in Composition of Some Species.

The assessment of trace element status in relation to animal health in upland pastures is complicated by the present lack of knowledge of "what the animal eats". It is generally assumed that the animal has preferences for certain species; the possibility exists however, and it is quite reasonable to assume, that these preferences may differ at different times.

In the previous experiments mixed pasture samples have been considered. Only in the preliminary experiment in September, 1949, was bracken included in the sampling of the pasture herbage, and it was excluded in the subsequent sampling on account of its comparatively high nickel content.

It was therefore decided to direct attention to a few typical/

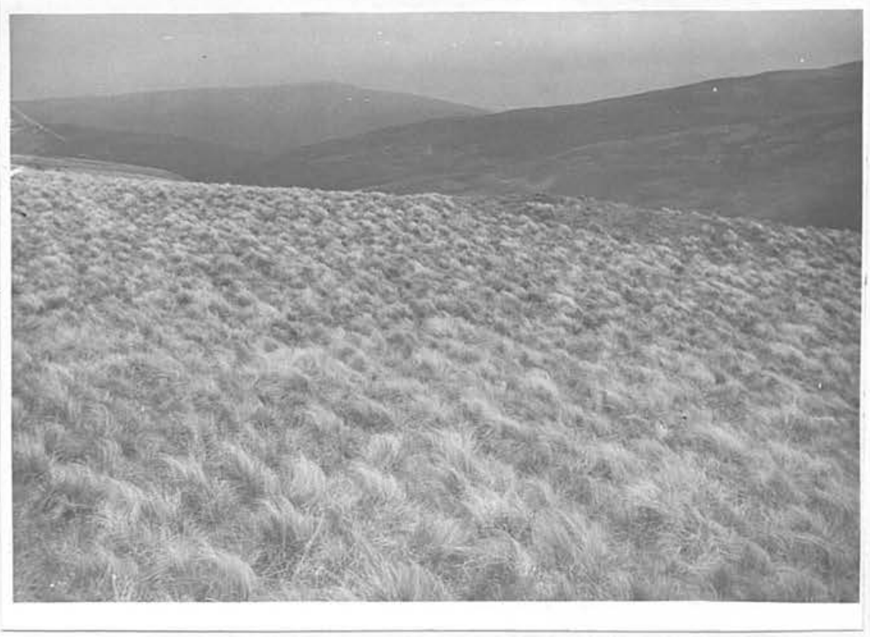


Fig. 13. Close up of a Molinia-Nardus association (sub-area 9)

typical hill pasture species, and in June, 1951, experiments were conducted to determine the variations in the mineral content of these species (as opposed to mixed pastures), and further to investigate the general composition of a few types.

Normal Variation

The first experiment was carried out to determine the normal variation within two hill pasture species, Molinia coerulea (flying bent) and Nardus stricta (white bent).

These two types were chosen for the following reasons:-

(a) They both grow well above ground level and the risk of soil contamination involved in their sampling is low;

(b) They are easily differentiated in the field;

(c) Molinia is found mainly on peats, whereas Nardus cannot be assigned to any one particular habitat, occurring on soils of very different character and covering large areas of hill land;

(d) Both species are characteristic in that they tend to dominate and occupy the areas on which they grow, often to the virtual exclusion of almost all others. With regard then to Molinia and Nardus associations, herbage samples (taken in accordance with the usual recommendations) are not so completely representative of the general plant association and their analysis may therefore tend to approximate to that of pure Molinia and Nardus samples. The composition of these two species therefore merits close consideration. In Fig. 13 opposite, is shewn a close-up of a Molinia - Nardus association (sub-area 9). Sub-area 9 was chosen for/

TABLE 18.

Statistical Presentation of Results for Molinia and Nardus pasture species.

Variate	Species	Range of Concentration	Mean	Coefficient of Variation	Error of Mean %
% s.f.a.	Molinia	2.93 - 3.77	3.34	6	1.1
	Nardus	2.36 - 3.03	2.72	8	2.5
% Ca	Molinia	~~.06~~ .04 - .16	.08	~~10~~ 24	~~1.8~~ 4.3
	Nardus	.06 - .09	.07	16	5.1
% K	Molinia	.91 - 2.13	1.10	21	3.8
	Nardus	.58 - .82	.75	10	3.2
% Mg	Molinia	.08 - .15	.13	13	2.4
	Nardus	.08 - .18	.12	25	8.0
p.p.m. Mn	Molinia	188 - 398	273	19	3.5
	Nardus	147 - 272	216	18	5.7
p.p.m. Co	Molinia	.04 - .16	.07	35	6.4
	Nardus	.08 - .17	.12	24	7.6
p.p.m. Ni	Molinia	.47 - 3.22	1.32	46	8.4
	Nardus	.69 - 3.15	1.41	47	14.9
p.p.m. Fe	Molinia	51 - 269	125	51	9.3
	Nardus	102 - 339	185	34	10.8

for the experiment, being a Molinia (dominant) - Nardus - Festuca association. A rectangle measuring 75 yards by 30 yards was selected in the centre of the sub-area and divided into 10 smaller rectangles each 15 yards by 15 yards. Three random points were taken in each small rectangle and three samplers were employed, each one taking one sample from an appropriate point-area in each rectangle. The procedure was essentially the same and was carried out in the same way as in the sampling experiments of June and September, 1950, except that 30 Molinia samples were obtained. In addition one further random point was taken in each small rectangle and a Nardus sample obtained from each yielding in all 10 Nardus samples.

Results

The analytical results are given in Appendix II e.

The ranges of concentration, means, coefficients of variation (to the nearest whole number), and errors of the means for all variates are given in table 18 (opposite) for both species.

From the table it is seen that:-

(1) With regard to both species, the respective coefficients of variation for all variates are of the same order of magnitude as those obtained for mixed grass samplings, i.e. for silica-free-ash and the major elements generally $\leqslant 20\%$, and for the trace elements cobalt, nickel and iron 35 to 50%. The coefficient of variation for manganese in each case is slightly less than that obtained for mixed pastures.

(2)/

(2) As in the case of mixed grass samplings, the most variable results are those obtained for cobalt, nickel and iron.

In addition it may be noted that:-

(3) There would appear to be significant differences in trace element content between the species with the exception of nickel, and in the major element content with respect to potassium. This difference in potassium content is reflected in the figures for silica-free-ash.

(4) With regard to these differences, Molinia is richer than Nardus in potassium and manganese content, but poorer in cobalt and iron.

Differences in Composition of Some Species.

The second experiment involved the collection of a number of composite samples of dominant species only, taken from (a) small prescribed areas in order to reduce the effects of soil variation and (b) selected patches on extensive areas.

In the first experiment (just described) it was noted that there were differences in trace element content between the species Molinia coerulea and Nardus stricta and the second experiment is an extension of this study.

(a) It was decided to investigate the composition of Nardus stricta (white bent) and Festuca ovina (sheeps' fescue) from different areas at different times of the year. The selection of these particular species for examination was governed by the following considerations:

(1)/

(1) Both species are easily differentiated in the field, although Festuca is a grass of lower habit.

(2) The existing general opinion of herds and those more familiar with grazing habits, is that, of the two species, Festuca ovina as the name implies is the more favourable sheep-diet, and

(3) Bearing on (2) above, the general opinion of contemporary authorities that diseases of hill sheep have their highest incidence on "Nardus" or "white bent" land.

Two areas of uniform soil and uniform Nardus - Festuca association were chosen in September, 1951, and composite samples of each species taken from each area. Each composite sample was obtained by bulking "sub-samples" or "cuttings" taken from 30 random points within an area of 75 yards by 30 yards, the procedure being essentially the same as that adopted under "Normal Variation" (above) for Molinia, except in this case, for the bulking of the sub-samples to form composite samples. Two operators were employed, one taking the Nardus and the other the Festuca sample. The usual sampling precautions were observed at all stages, and prior to transportation to the laboratory, each sample was tested in the field laboratory for "botanical" purity.

The description of the areas chosen is as follows:-

(1) Sub-area 1: Festuca ovina (main), Agrostis, Nardus stricta, Deschampsia caespitosa (on mineral soil).

(2) Sub-area 9: Molinia (main), Nardus stricta, Festuca ovina (on peat).

The/

TABLE 19. Composition of Nardus stricta and Festuca ovina.

Area	Season	Species	Variate							
			%				p.p.m.			
			s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
Sub-area 1	Sept. 1951	Nardus	1.59	.16	.52	.07	346	.21	3.06	131
		Festuca	3.11	.18	1.12	.10	421	.09	2.43	126
	June, 1952	Nardus	2.40	.13	.71	.12	639	.10	.82	150
		Festuca	4.50	.16	1.49	.14	722	.08	.80	109
	Sept. 1952	Nardus	1.29	.15	.46	.12	328	.18	2.17	261
		Festuca	3.59	.16	1.54	.13	572	.07	1.62	182
Sub-area 9	Sept. 1951	Nardus	1.61	.11	.54	.08	161	.10	1.99	167
		Festuca	2.78	.13	.94	.06	350	.07	.68	115
	June, 1952	Nardus	1.97	.11	.72	.14	297	.13	1.73	252
		Festuca	3.23	.13	1.24	.12	619	.09	1.07	140
	Sept. 1952	Nardus	1.24	.09	.46	.10	216	.18	2.50	219
		Festuca	3.17	.12	1.17	.07	351	.08	1.14	154

TABLE 20.

Composition of Different Species sampled from selected patches.

Date	Species and Location	Variate							
		%				p.p.m.			
		s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
Sept. 1949	Bracken (sub-area 2)	7.09	.21	2.76	.41	70	.06	17.14	90
	Bracken (sub-area 4)	7.17	.30	3.17	.44	158	.09	15.25	114
	Juncus communis	4.71	.18	1.55	.18	281	.29	1.14	136
	Nardus stricta	2.37	.26	.86	.11	202	.12	2.05	159
	Agrostis	3.19	.18	1.44	.10	343	.05	2.47	101
June 1951	Calluna vulgaris (N. Fasset)	2.29	.23	.59	.16	923	.41	3.87	228
	Nardus stricta (N.E.Fasset)	2.55	.07	.92	.09	330	.09	1.49	145
	Agrostis (sub-area 7)	4.45	.11	1.76	.15	296	.10	.86	151
	Molinia (N.Fasset)	3.81	.08	1.53	.14	242	.04	.79	90

The sampling was repeated for both areas in June, 1952, and September, 1952. The results are shown in table 19 opposite.

(b) Various samples of other typical hill pasture species were collected during this investigation from selected patches at different times. Each sample was secured from a particular patch by bulking random cuttings taken from an area of about 100 square yards.

The results are tabulated opposite (table 20).

From table 19 it is seen that:-

(1) There are obvious differences in the mineral contents of Nardus and Festuca pastures.

(a) With regard to the major element content, assuming variations of the same order of magnitude as those obtaining for mixed pasture samples (see table 10) and taking into consideration the respective laboratory analytical errors (see table 1) and method of sampling, it is apparent that Festuca is significantly higher in potassium content than Nardus, these differences being reflected in the figures for silica-free-ash. The differences in calcium and magnesium contents between the two species are relatively small.

(b) With regard to the trace elements, and with the same assumption as in (a) above, (see tables 2 and 10) Nardus is significantly greater in cobalt, nickel and iron content than Festuca, although the differences in nickel content for sub-area 1 are not very large. Festuca is richer in manganese.

(2)/

(2) There are seasonal changes in the mineral content of both species. Referring in particular to the summer period of 1952, the major element composition for both Nardus and Festuca is reasonably constant, within the error of sampling, for both June and September, with the exception of potassium which in the case of Nardus is significantly higher in June. In the case of both species for sub-area 9, the magnesium contents are higher in June. With regard to the trace elements for both species manganese is significantly higher in June: the nickel and iron contents are higher in September, although on the peaty soil (sub-area 9) these differences are mostly insignificant. The cobalt content of Festuca is reasonably constant for June and September, whereas the Nardus cobalt content is significantly higher in September.

From table 20 it may be noted that:-

The species differ from each other in their range of values for the several variates.

(a) Calluna (heather) is comparatively rich in manganese, cobalt and iron. This is in agreement with the results obtained by Thomas and his associates (1945) in their investigation into "the minor elements of common heather". Their results suggested that "the heather fell provides iron, manganese, copper and cobalt in relative abundance". The ranges given by Thomas are as follows:- .073 - .111% Mn, .108 - .224 p.p.m., Co and .0213 - .0415% Fe. These results are calculated on a dry-matter basis and refer, as do the results in the present investigation, to the edible fractions.

(b)/

(b) It is often held that members of the rush family are of negligible feeding value; yet Juncus communis (common rush), with regard to the trace elements, cobalt and iron, compares favourably with the other species examined (e.g. 0.29 p.p.m. Co and 136 p.p.m. Fe).

(c) The bracken collected from Fasset in September, 1949, is characterised by comparatively high major element content, low cobalt and manganese, but abnormally high nickel content. These high nickel results have been referred to earlier in the investigation.

When the results from tables 18, 19 and 20 are considered together the following facts emerge:-

(1) The variations in mineral content of the species Molinia coerulea and Nardus stricta, with regard to the elements investigated, approximate to those obtained for mixed pasture samples.

(2) It is generally recognised that "pining" of sheep occurs mainly on "white" land, i.e. land carrying predominantly Nardus species and that, in general, diseases of hill sheep have their highest incidence on this type of land. The assumption is sometimes made that the increased incidence of sheep diseases on "white" land may be due entirely to mineral deficiency. Thomas and Trinder (1947) investigated the mineral constituents of some moorland plants including Nardus, at different stages of growth. With regard to Nardus they found comparatively low calcium (.085 - .126% as Cao) and copper. The cobalt content (.08 - .117 p.p.m.) was however not so poor as had been expected in view of the dominance/

dominance of this species on swards associated with "pine". Theirresults however (calculated on a dry-matter basis) give no indication of the errors of sampling.

The results obtained in this investigation indicate that Nardus is comparatively richer than Molinia and Festuca in trace element content with regard to cobalt and iron, although poorer in manganese.

With regard to the manganese status of Nardus species, it might be expected that its manganese content would compare favourably with those of Molinia and Festuca, for Nardus is very tolerant to acid conditions. There may be, on this account, however, some mechanism in the Nardus metabolism which regulates the uptake of manganese thereby preventing toxicity.

(3) With regard to the results for cobalt in Nardus and Festuca it has been noted that the Nardus cobalt content is significantly and consistently higher than that of Festuca, and in September especially, is almost twice that of Festuca. Festuca is a species of lower habit than Nardus, and therefore a herbage sample drawn from a Nardus-Festuca association (in accordance with the usual recommendations) will contain a larger proportion of Nardus grass. It is evident that the cobalt result obtained from such a sample will be liable to misinterpretation from the point of view of advisory work, if it be assumed that the animal prefers Festuca grass.

In the present state of knowledge, however, no particular ruling can be given regarding the preference of sheep for particular/

particular species, until a fuller study of this problem has been undertaken. On the other hand, the results of further study in this direction might permit modification in due course of the final sampling scheme adopted, involving the omission of particular species during sampling in certain cases. Consideration must also be given to the question of possible differences in the availability to the animal of cobalt, and trace elements in general, in the different pasture species.

For example, in advisory work there have been known responses in animal experiments at pasture cobalt levels of the order of 0.12 p.p.m., and slightly above, despite the general findings in the literature that such a level is adequate. While the results quoted in the literature do not take sampling errors into account, it is suggested that the above discrepancies may find a possible explanation in the foregoing discussion.

(4) Under the discussion on "Seasonal Variation" with regard to mixed pasture samples (see present chapter, section A iii) it was noted that for the results of the sampling carried out in June and September of 1950, the greatest and most consistent differences (for all the variates examined) were found for the iron contents, which fell in September (see tables 14 and 15). In Nardus and Festuca species for mineral soil (see table 19) the iron contents rose about 60% above the corresponding June values. It would appear then, some other pasture/

pasture type or types must experience a reasonable fall in iron content between June and September, to account for the seasonal variation in mean iron content in mixed pasture samples.

(5) Under the general discussion of the results for the sampling experiments in 1949 and 1950, it was noted that in the case of sub-area 9 in June, 1950, the results for mixed pasture samples were similar to those obtained as a whole for the other areas on Fasset Hill, whereas in September the corresponding results with regard to potassium, manganese and cobalt, approximated more to the results obtained on the Hairney Law. It was suggested that this effect was probably connected with the stage of growth of the dominant species on that area, namely Molinia, Nardus and Festuca. This suggestion is confirmed from the results in table 19, concerning the seasonal changes in the mineral contents of Nardus and Festuca species. The mineral composition of both species differ in June and September. Festuca is characterised by lower manganese content and Nardus by lower potassium and manganese contents and higher cobalt content in September. These seasonal changes are reflected in the results of the mixed pasture samples taken from sub-area 9 in June and September of 1950.

(6) The results obtained in table 20 for various species selected from patches on the hillside would suggest that Calluna vulgaris (common heather) is a comparatively valuable source of trace elements, manganese, cobalt and iron.

Experienced herds attach much importance to good heather land, and the scientific basis for this belief may be found in the efficiency of Calluna as a source of these trace elements, and particularly those deficiencies of which are associated with diseases of livestock.

CHAPTER V.

DISCUSSION OF RESULTS.

a) Sampling Experiments

The results of the investigation of the variation in trace element content of upland pastures and soils demonstrate clearly that considerable fluctuations are liable to be found over relatively small areas, and it is exceedingly difficult to obtain a sample which may be regarded as representative of a large tract of hillside. In the case of the major element content of pasture, the variations are smaller.

Various factors may contribute to the variation, namely, (a) analytical technique, (b) soil type, (c) pasture association, (d) personal error in sampling and (e) season, and these have all been examined. If the sampling and analysis be carried out in accordance with well established practice, the personal and analytical errors have been shown to be relatively unimportant. It has also been demonstrated from the results that for sampling carried out in June and September, the seasonal effect is not large.

Attention may therefore be directed to the different soil types and pasture species which are commonly related to each other. The soils examined have varied from shallow mineral soils with about 10% organic matter to peats consisting of about 70% and more organic matter, and considerable and significant differences have been found between classes of soils. The interpretation of the results /

results obtained, however, is necessarily complicated when soils are compared for which the moisture content in the field may vary on an air-dry basis from 20% to more than 100%. The main difficulty, however, is presented by the variations occurring within types which are reasonably uniform in chemical and physical characteristics; it is reasonable to expect differences between types that are easily distinguishable in the field.

The problem of differentiating between soils also applies to the vegetation growing on them. The pasture herbage analyses have been carried out on mixed species that are found growing together. In the case of cobalt and nickel, the variations in content of these trace elements in mixed pasture have been found to be as great as those of the acetic-acid available cobalt and nickel in the soil samples taken at the points where the herbage was cut, and of the order of 50% Lesser variations have been found for the trace elements manganese and iron in pasture herbage (i.e. about 35%), while the variations obtained for the major elements have been found to be relatively smaller (about 20%).

In addition, the sampling of dominant typical hill pasture species (e.g. Molinia, Nardus and Festuca), have been carried out from small prescribed areas to reduce the error from soil variation, and several species, (e.g. Bracken, Callunavulgaris, and Juncus communis) have been sampled from selected patches on the hillside. The

results/

results have indicated that the variations in mineral content of the species examined (e.g. Molinia, Nardus) are of the same order as those found for mixed pastures, and that certain species may differ from others in their range of values (e.g. Nardus, Festuca, Calluna, Bracken), and that seasonal variations in the mean contents may also occur.

While this investigation is concerned essentially with the sampling of mixed pastures, attention has been drawn to the difficulty, from the point of view of advisory work, of interpreting the results obtained for mixed pasture samples (e.g. the cobalt content of Nardus-Festuca pasture association). The difficulty is concerned with the representative sampling of particular associations, the different trace element contents of the species comprising such associations, and finally the problem of knowing "what the sheep eats".

Correlations between the trace elements in mixed pasture herbage, and between the cobalt content of pasture and the soil-extractable cobalt, are discussed in the remainder of this chapter.

The results, as a whole, demonstrate clearly the futility of carrying out elaborate analyses for "undefined" or "particular" samples drawn from large tracts of hillside. They provide an excellent confirmation of the points set forth by Wood and Stratton (1910) and Yates and Cochran (1938).

TABLE 21.

Relationship	June 1950		September 1950	All Results	X^2
	Sub-areas 3, 6-9	Sub-areas 10-12	Sub-areas 3, 6-12		
a. Cobalt/Manganese	Co <.09 ≥.09 27 29 <487 Mn 35 28 ≥487	Co <.15 ≥.15 20 16 <300 Mn 17 19 ≥300	Co Lesser Greater or Equal 40 53 Lesser Mn 42 52 Greater or Equal	Lesser Greater or Equal 87 98 Lesser Mn 94 99 Greater or Equal Co	Not Significant
b. Manganese/Iron	Mn <487 ≥487 30 24 <167 Fe 38 27 ≥167	Mn <300 ≥300 24 12 <249 Fe 23 13 ≥249	Mn Lesser Greater or Equal 36 52 Lesser Fe 55 48 Greater or Equal	Lesser Greater or Equal 90 88 Lesser Fe 116 88 Greater or Equal Mn	Not Significant
c. Cobalt/Iron	Co <.09 ≥.09 51 17 <167 Fe 10 41 ≥167	Co <.15 ≥.15 29 17 <249 Fe 8 18 ≥249	Co Lesser Greater or Equal 58 30 Lesser Fe 25 74 Greater or Equal	Lesser Greater or Equal 138 64 Lesser Fe 43 133 Greater or Equal Co	$X^2 = 74.23$ (highly significant)

b). Trace Element Relationships in Pasture Herbage

It was decided to examine the results of the pasture analyses for possible correlations between the trace elements investigated, and attention was directed to the three elements manganese, cobalt and iron, which at the present moment are of considerable importance in plant and animal nutrition.

In determining these relationships, the results obtained in June and September of 1950 for mixed pastures were considered. The procedure adopted was to divide the values for each variate into two classes, according as to whether they are less or greater than their mean for the sub-area or sub-areas concerned. As in the previous statistical treatment, sub-areas 3, and 6-9 were grouped together, as also sub-areas 10-12,for the results for June 1950. In the case of the results for September 1950, the above procedure was applied to each sub-area individually. There were no obvious differences between the sub-areas for any of the relationships examined in this case, however, and the statistical results were therefore pooled.

These results have been recorded in the fourfold tables 21 a. b. and c. given opposite. It is obvious by reference to these tables, that there are no striking differences, in any case, between the results for June and September of 1950. The statistical results for both seasons were therefore pooled, and are shewn under the heading/

heading "All Results".

It is seen that there are no significant associations between either cobalt and manganese or manganese and iron. In the case of cobalt and iron however, there is a significant positive correlation between these variables and $X^2 = 74.23$ (highly significant). The value of X^2 has been calculated according to the method given in Chambers (1948, p. 85). Visual inspection of the results however suggests that this association does not necessarily apply at low or moderate values of iron, e.g. $\leqslant 150$ p.p.m. This effect may be due to contamination.

c). Relationship between soil-extractable cobalt and pasture herbage cobalt

The dilute acetic-acid extractable cobalt was determined on the soils, as this fraction has been generally found to be closely related in the case of acid mineral soils to herbage levels, and more particularly to the occurrence of cobalt deficiency disease in ruminants, as discussed by Stewart, Mitchell and Stewart (1941).

Two factors must however be considered in the assessment of soil cobalt, namely a) the effect of soil pH on the availability of cobalt to the plant, and b). the organic matter of the soil. With regard to a). it is well known that the availability of cobalt to the plant decreases with increasing pH., and lime–induced deficiences have been referred to throughout the literature. For the soils examined, however, i.e. the samples obtained in June and September, 1950, the pH values lie in the range 3.6-5.0, and would not therefore greatly influence the result. With regard to the second consideration under b). the organic matter content may influence the assessment in the following way. The density of the soil decreases with increasing organic matter content, and therefore for a given concentration of cobalt per unit weight of soil, the concentration per unit volume will likewise decrease with increasing organic matter content, and the weight-volume relationship may reduce the amount of available cobalt, assuming the root habit remains unchanged. Soil organic matter may also conceivably be associated with some type of trace/

TABLE 22.

Soil-extractable cobalt/pasture cobalt relationship.

Results for June 1950

Sub-area 3

Soil Co \ Pasture Co.	<.08	≥.08
<.16	8	7
≥.16	5	4

Sub-area 6

Soil Co \ Pasture Co	<.08	≥.08
<.16	6	7
≥.16	4	7

Sub-area 7

Soil Co \ Pasture Co	<.12	≥.12
<.09	7	7
≥.09	4	6

Sub-area 8

Soil Co \ Pasture Co	<.10	≥.10
<.14	7	6
≥.14	7	3

Sub-area 9

Soil Co \ Pasture Co	<.07	≥.07
<.20	8	3
≥.20	3	7

Sub-area 10

Soil Co \ Pasture Co	<.16	≥.16
<.28	5	4
≥.28	5	6

Sub-area 11

Soil Co \ Pasture Co	<.15	≥.15
<.29	6	5
≥.29	5	8

Sub-area 12

Soil Co \ Pasture Co	<.13	≥.13
<.23	5	9
≥.23	6	4

Results for September 1950

Sub-area 3

Soil Co \ Pasture Co	<.09	≥.09
<.14	8	7
≥.14	4	4

Sub-area 6

Soil Co \ Pasture Co	<.06	≥.06
<.46	5	5
≥.46	8	5

Sub-area 7

Soil Co \ Pasture Co	<.06	≥.06
<.10	8	4
≥.10	5	6

Sub-area 8

Soil Co \ Pasture Co	<.19	≥.19
<.26	4	7
≥.26	8	4

Sub-area 9

Soil Co \ Pasture Co	<.18	≥18
<.13	4	6
≥.13	4	2

Sub-area 10

Soil Co \ Pasture Co	<.25	≥.25
<.48	8	5
≥.48	5	5

All Results

Soil Co \ Pasture Co	Lesser	Equal or Greater
Lesser	89	82
Equal or greater	73	71

type of trace element fixation, but this effect has not been considered.

A correction was therefore applied to the soil cobalt results for June and September of 1950, and was derived from the following arbitrarily graded weight-volume correction table;

Loss on Ignition	Correction Factor
0 - 12%	X 1
12 - 25%	X $\frac{3}{4}$
25 - 50%	X 2/3rds.
$\geqslant$50%	X $\frac{1}{2}$.

The relationship between the "corrected" soil cobalt and pasture cobalt results was then examined by the same procedure as that adopted for the pasture correlations, the results in either case being classed in two categories, according as to whether they were less than, or greater than their respective means. Abnormally high results in either case (in September 1950 results, pasture sample B 1, sub-area 7, and soil sample B 6 sub-area 8). were excluded, and this procedure was adopted for the results obtained from each sub-area individually. There were no obvious differences in the relationship between sub-areas or for the two times of sampling. This is brought out in the fourfold tables in the complete table 22, given opposite. The statistical results were therefore pooled, and are given under "All Results" at the foot of the table.

It is seen fomthe results examined, that there is no association between these two variables.

Bearing in mind the arbitary mature of the correction applied to the soil cobalt figures, it cannot be stated that the variation in soil-available cobalt is more than a probable contributary factor to the variation in pasture cobalt content; and further, until animal experiments have been carried out to establish the critical levels for cobalt in pasture and soil extracts, the dilute acetic-acid extractable cobalt cannot be taken as an index of cobalt status in the pasture.

CHAPTER VI.

CONCLUSIONS AND RECOMMENDATIONS

a). Introductory Note

It is appropriate at this stage to restate the aim of the present investigation, which has been to study the variation in trace element content of hill pasture, with a view to establishing ultimately a sampling scheme for the determination of these elements, such that the results obtained are accurate to within certain definite limits.

As discussed previously, the normal variation (V) in the content of any particular element in hill pastures and soils is derived essentially from two main sources, namely a). the analytical variation (L) i.e. the error inherent in the analytical technique and b). the sampling variation (F) i.e. the error involved in the sampling in the field, and these variations are corrected by the following statistical relation:-

$$V^2 = L^2 + F^2.$$

The results obtained have shewn that for the variates examined and especially the trace elements, L is relatively small in comparison with F, and that the normal variation is almost wholly accounted for by the sampling variation.

Examination of the factors which may contribute to the sampling error has shewn that the personal error is relatively unimportant and that there is no significant seasonal/

seasonal effect between June and September. The variations for all variates have been shewn to be generally of the same order of magnitude for areas, uniform within themselves, but differing in vegetation and soil type.

Referring to the results of June and September of 1950 (i.e. the summer period), it was seen that the variations fell within the limits shewn in table 23 below.

Table 23

Variate	Range of normal variation (%)	Accepted upper limit of variation (%)
s.f.a.	12 - 15	20
Ca.	13 - 14	20
K	13 - 18	20
Mg.	15 - 23	30
Mn.	22 - 30	35
Co.	28 - 42	50
Ni	38 - 42	50
Fe	22 - 32	35
Soil Co	37 - 44	50
Soil Ni.	24 - 46	50

Taking the respective values on the right hand column of the above table as the upper limits for the normal variations obtained from uniform areas, it is obvious that errors may be reduced by taking very large numbers of samples. In this respect, however, it is necessary to take/

take into account a) the relative magnitude of the total analytical error, and b) the necessity of keeping the sampling within practicable limits.

Bearing the above points in mind, the following procedure is recommended for the sampling of hill pastures and soils for trace element analysis.

b). Method Recommended for the Sampling of Hill Pastures and Soils

There are three stages in the procedure, viz.; a) the sub-division of the area or tract of hillside into sections, b). the collection of the samples, and c). the subsequent transportation of the samples to the laboratory.

The extent of the area from which a sample should be taken must rest inevitably with the judgment of the sampler concerning its uniformity. In the case of a large area or tract of hillside, it would be better to divide it into three or four sections. From each section 25 samples of pasture and soil should be taken at random to form composites representative of that section. In practice it is difficult to ensure pure randomisation in the sampling technique, without recourse to the time-consuming operations of dividing the area into rectangles and using a system of co-ordinates : a reasonable approximation to random sampling may be obtained by allowing the samplers to traverse the area in a zig-zag direction, sub-samples being/

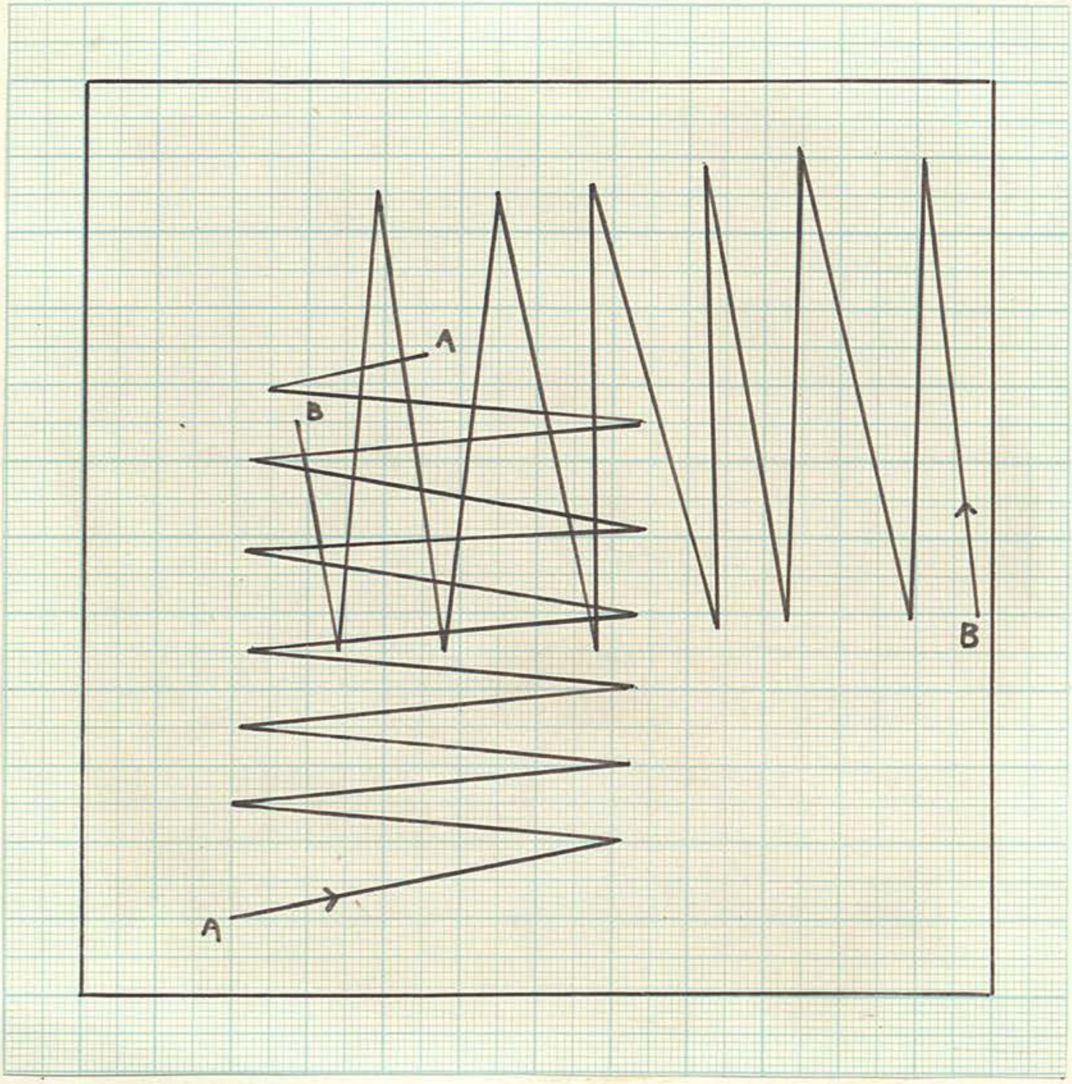


Fig. 14. Typical paths traversed by operators A and B sampling in an area according to the method recommended.

being taken at every 15 yards. (see Fig. 14). The actual sampling must be carried out in accordance with the recommendations given earlier (under "General Notes" in Chapter IV) no differentiation being made with regard to species, except that bracken be excluded. In the case of composite pasture samples, these should amount to a total of about 6 or 7 lb. and must be placed in a clean paper or linen bag. With regard to soil samples, the trowel will be found most convenient as a sampling instrument . The total amount of soil taken according to this procedure will probably be about 10lb. This should be collected in a linen bag, then thoroughly mixed on a clean sheet of cloth or stout paper, and a composite of about 3 lb. secured and placed in clean paper or linen bag.

Pasture and soil samples must be packed in clean separate cardboard boxes prior to transportation to the laboratory.

In connection with the sampling of pasture in the field, as with the analytical process, strict attention must be given at all stages to the avoidance of contamination (e.g. soil and faeces in the field; metal parts in the laboratory).

Provided the above instructions are followed, definite limits of accuracy may be assigned to the results obtained, and these are given in table 24 below for all variates.

Table 24

Variate	Normal variation, V(%)	Total Analytical Error, L(%)	Accuracy of Method, V_1(%)
s.f.a.	20	6	7
C.a.	20	6	7
K	20	6	7
Mg.	30	12	13
Mn	35	7	10
Co	50	10	14
Ni	50	12	15
Fe	35	10	12
Soil Co	50	10	14
Ni	50	12	15

In the above table, the maximum error (V_1), associated with each variate for the results obtained from a single composite sample, has been calculated from the normal variation (V), the total analytical error (L) and the number of samples **n** (= 25) taken **to form the composite** sample, in accordance with the equation:-

$$V_1^2 = L^2 + \frac{V^2 - L^2}{n}$$

The values for V_1 have been given to the nearest whole number (c.f. total analytical errors in tables 1 - 3).

From table 24 , it is seen that with the exception of magnesium/

magnesium, the errors associated with the method for silica-free-ash and major element determinations are of the order of $<10\%$. With a more refined spectrographic analytical technique for magnesium having a precision comparable to those associated with calcuim and potassium determinations, the error for a magnesium estimation would then also be of the order of $<10\%$. With regard to trace element determinations, the corresponding errors lie between 10 and 15%.

c) Application of the Method

The method was tested in the following manner.

In June 1953, a uniform area, carrying an Agrostis-Fescue-Nardus pasture association and of about 4 - 5 acres was selected on the Park Law at Sourhope, south of Fasset Hill. Five pasture samples were taken by each of two different samplers from this area, according to the recommended procedure.

At the same time and in the same way, sub-area 10 (previously described in Chapter IV.) was sampled by two soil samplers each sampler taking five composite soil samples.

In each case the samples were taken, as near as was practically possible, at random from the areas, the samplers traversing the areas in zig-zag fashion, and selecting the sub-samples at every 15 yards. Each sampler was instructed to take sub-samples at the appropriate points, and not to select/

select "convenient" neighbouring points. The samples were numbered and submitted for analysis.

The analytical results are given in tables 25a. and b. below.

Table 25a.

PASTURE

Sample	% s.f.a.	%Ca.	%K	%Mg.	ppm.Mn.	ppm.Co.	ppm.Ni.	ppm.Fe.
1	2.92	.16	1.03	.13	565	.09	1.77	115
2	2.57	.14	.89	.14	468	.10	1.57	115
3	2.36	.15	.89	.12	499	.10	1.80	116
4	2.76	.15	1.05	.14	533	.11	1.52	163
5	2.65	.15	.99	.14	508	.11	1.64	138
6	2.49	.15	.89	.14	471	.11	1.52	123
7	2.37	.14	.93	.10	493	.09	1.45	130
8	2.53	.14	.93	.11	476	.12	1.02	114
9	2.63	.13	.91	.11	426	.13	1.36	131
10	2.55	.13	.90	.10	427	.11	1.60	123

Table 25b.

SOIL

Sample	ppm. Co.	ppm. Ni.
1	.35	1.00
2	.34	.93
3	.27	.74
4	.26	.72
5	.35	.75
6	.31	.74
7	.29	.75
8	.33	.83
9	.36	.79
10	.29	.69

The errors associated with the results obtained from a single composite sample have been calculated from the above analytical figures, and are compared in table 26 below with the corresponding respective estimates derived for the sampling procedure recommended.

Table 26

Variate	Error (Coefficient of Variation, %).	
	Observed Error	Derived Error
s.f.a.	6.6	7
Ca	6.7	7
K	6.4	7
Mg.	14.1	13
Mn	8.9	10
Co	11.7	14
Ni	14.6	15
Fe	11.9	12
Soil Co	11.4	14
Soil Ni.	12.5	15

The results given in the above table are in very good agreement when consideration is given to the haphazard as opposed to purely random method of selection of the samples, and also to the small number of samples taken.

d). Concluding Remarks and Suggested Further Work

The results obtained in this investigation have clearly demonstrated the futility of carrying out elaborate analyses without taking sampling errors into account, and following upon such a practice, the degree of uncertainity which must result in making a survey of the trace element status of upland pastures, or in providing information to advisory services. In this respect the sampling scheme recommended marks a definite advance in work of this type, although the method is meantime limited in being applicable only in the case of relatively small areas.

In consequence of the results obtained, there are two main lines of research along which further work may be directed. The first problem concerns the application of the present recommended method of sampling to larger areas, while the second involves a fuller investigation of the trace element content of typical hill pasture species, consequent upon a study of animal behaviour in relation to animal preferences for particular species. This latter problem must also take into account the availability to the animal of the trace elements in the different pasture species./

species.

1. The critical values of trace elements in pastures (and soils) will not be defined until until such animal experiments have been conducted, and sufficient evidence relating to the incidence and control of diseases is secured. Meanwhile, only arbitrary limits can be tentatively adopted, in order to differentiate cases of serious deficiency on the one hand and adequacy on the other.

SUMMARY

1. The normal variation in mineral content of mixed hill pastures has been investigated with particular reference to the trace elements manganese, cobalt, nickel and iron. The variations in the cobalt and nickel contents of mixed pastures have been found to be as great as those of acetic-acid extracts of soil samples taken at the points where the pasture was sampled, and range from approximately 30% to 50%. The variations in manganese and iron contents are somewhat less (20% to 30%), while those in major element and silica-free-ash contents are less than 20%, with the exception of magnesium for which the variation is slightly higher.

2. Factors which may contribute to the normal variation in each case, viz. (a) analytical error, (b) soil type and plant association, (c) seasonal effect and (d) personal error in sampling have been examined. The analytical and personal errors have been shewn to be relatively unimportant, and there is no significant seasonal effect. The variations have been found to be generally of the same order of magnitude for areas uniform within themselves, but differing in vegetation and soil type.

3. /

3. The mineral composition of some typical hill pasture species has also been investigated.

4. The results for mixed pastures have been examined for possible trace element relationships, and a highly significant positive correlation has been found between cobalt and iron. No correlation has been found between pasture and soil-extractable cobalt, the latter values having been reassessed according to soil characteristics.

5. A method has been recommended for the sampling of hill pastures for trace element analysis, and the errors associated with the method have been given. The method has been tested and found satisfactory.

The substance of the results of this investigation is in process of publication as a Bulletin to be issued by the Department of Agriculture for Scotland.

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APPENDIX I

PRECISION OF ANALYTICAL METHODS

The following is a typical set of pasture and soil analytical results obtained according to the methods described in Chapter III for the estimate of the total analytical error associated with each of the variates examined. In this particular set the statistics are based on a 10 times replication in the estimation of the errors of the Lundegardh technique (Ca, K, Mg, Mn) and those for soil determinations (Co, Ni). For the errors associated with pasture determinations (Co, Ni) the statistics are based on a 20 times replication.

a. Lundegårdh Technique (Ca. K. Mg. Mn.)

The concentrations for the different variates have been expressed as the readings (R) on the concentration scales of the Lundegårdh working curves

Sample	R(Ca)	R(K)	R(Mg)	R(Mn)
1	194	130	133	40
2	194	130	161	40
3	183	120	128	40
4	179	130	159	40
5	175	128	161	38
6	167	119	128	34
7	169	112	128	35
8	169	125	165	38
9	185	115	161	35
10	176	110	154	35
Mean	179.1	121.9	147.8	37.5
Error (as coefficient of variation)	5.5%	6.3%	11.0%	6.7%

b. Pasture Determinations (Co, Ni, Fe).

Sample	Variate		
	p.p.m. Co	p.p.m. Ni	p.p.m. Fe
1	.25	3.14	199
2	.20	3.05	175
3	.25	3.36	216
4	.22	3.95	176
5	.24	3.34	182
6	.23	3.04	181
7	.23	3.36	180
8	.28	2.81	179
9	.26	3.33	169
10	.23	3.43	208
11	.24	3.64	199
12	.24	3.36	240
13	.25	2.95	203
14	.22	3.00	172
15	.23	3.40	162
16	.21	3.78	196
17	.23	3.97	186
18	.22	3.43	199
19	.21	3.36	186
20	.20	3.48	200
Mean	.23	3.36	190
Error (as coefficient of variation)	8.8%	9.3%	9.6%

c. Soil Determinations (Co, Ni).

Sample	Variate	
	p.p.m. Co.	p.p.m. Ni
1	.40	.67
2	.33	.61
3	.32	.59
4	.32	.61
5	.34	.68
6	.33	.67
7	.40	.79
8	.41	.79
9	.36	.59
10	.38	.66
Mean	.36	.67
Error (as coefficient of variation)	9.9%	11.0%

APPENDIX II

RESULTS

The pasture and soil analytical results for all the sampling experiments have been included in this Appendix with the exception of some results for pasture species and the results of the test experiment (Ch. VI) when it was found more convenient to include these in the text. The loss of ignition (% L.I.) and pH estimations included in the results for the soils taken in September, 1949, and June and September of 1950, were obtained from the appropriate Soils Laboratory reports.

a). i) Pasture Analytical Results (September 1949).

SUB-AREA 1.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
Plucked								
C	2.98	.28	.89	.13	583	.10	2.59	133
N	3.07	.21	.95	.10	635	.18	2.81	136
NE	2.74	.21	.80	.10	463	.09	2.21	113
E	2.22	.27	.83	.14	412	.16	8.57	143
SE	3.18	.16	.86	.10	755	.31	2.34	150
S	2.27	.21	.62	.09	429	.07	10.26	94
SW	2.27	.19	.67	.14	652	.72	29.92	178
W	2.15	.19	.61	.08	506	.22	5.00	92
NW	2.27	.21	.62	.09	695	.27	2.14	206
Clipped								
C	3.38	.28	1.20	.16	435	.22	4.67	129
N	2.50	.21	.86	.10	539	.45	4.67	129
NE	2.50	.26	.94	.14	484	.08	1.35	97
E	3.22	.29	.94	.15	340	.32	7.57	132
SE	2.12	.19	.76	.08	539	.22	11.30	151
S	2.62	.21	.78	.10	367	.07	4.61	102
SW	2.74	.25	.81	.10	607	.35	7.57	148
W	Rejected							
NW	2.70	.21	.86	.14	532	.17	17.48	139

SUB-AREA 2.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
C	2.45	.38	.83	.09	292	.11	10.72	86
N	4.15	.37	1.39	.15	233	.54	10.03	91
NE	2.97	.24	.89	.11	322	.76	9.20	113
E	4.25	.26	1.51	.15	479	.36	2.05	88
SE	4.31	.31	1.51	.16	388	.43	12.62	89
S	3.12	.28	1.05	.12	436	.08	2.10	118
SW	4.79	.33	1.89	.14	635	.33	11.40	67
W	4.29	.31	1.59	.14	564	.12	1.37	65
NW	2.57	.28	.77	.10	503	.63	2.98	120
WSW	3.76	.33	1.25	.13	482	.58	1.93	112
SSW	3.14	.28	.93	.13	511	.10	5.26	112
WNW	4.77	.34	1.67	.16	394	.18	.52	54
NNW	4.32	.35	1.61	.17	557	.87	.94	77
ENE	4.66	.33	1.59	.16	463	.34	1.91	64
NNE	4.28	.42	1.24	.15	597	.12	2.52	172
ESE	5.28	.32	1.99	.17	353	.82	25.28	84
SSE	5.23	.33	1.87	.19	348	.15	3.15	48

SUB-AREA 3.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
C	4.21	.23	1.50	.18	764	.07	5.86	125
N	2.89	.20	.89	.11	506	.10	14.16	110
NE	2.85	.17	1.34	.13	772	.36	6.67	91
E	4.28	.22	1.43	.16	738	.24	1.64	83
SE	4.77	.21	1.76	.18	644	.04	1.62	72
S	4.95	.20	1.84	.20	695	.21	5.61	85
SW	3.79	.26	1.21	.17	764	.39	1.50	158
W	2.65	.17	.71	.09	755	.08	1.72	108
NW	2.21	.16	.62	.07	575	.49	2.62	118
WSW	2.41	.17	.75	.08	592	.39	1.70	141
SSW	3.84	.21	1.37	.16	420	.23	2.13	83
WNW	1.94	.15	.55	.07	386	.24	2.58	121
NNW	2.64	.17	.86	.08	944	.19	2.83	83
ENE	4.13	.20	1.48	.16	644	.48	12.71	86
NNE	3.54	.19	1.19	.12	695	.10	8.89	107
ESE	4.13	.21	1.44	.17	429	.07	1.86	93
SSE	3.69	.28	1.33	.15	429	.13	1.94	88

SUB-AREA 4.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
C	3.20	.16	1.07	.09	532	.49	3.73	124
N	2.26	.19	1.10	.10	815	.16	2.34	110
NE	3.05	.16	.95	.09	463	.25	.61	91
E	3.37	.27	.95	.11	403	.40	2.84	125
SE	4.44	.22	1.61	.15	686	.29	1.21	95
S	4.60	.22	1.59	.14	472	.30	2.88	104
SW	4.39	.19	1.66	.15	798	.27	3.82	112
W	4.23	.20	1.53	.14	695	.32	2.20	152
NW	2.78	.19	.83	.08	592	.53	5.90	199
WSW	3.11	.18	1.00	.11	592	.54	7.46	185
SSW	4.13	.22	1.59	.17	549	.44	6.78	89
WNW	4.37	.28	1.50	.16	609	.62	2.61	112
NNW				Rejected				
ENE	2.43	.20	.89	.10	360	.46	.44	180
NNE	3.07	.21	.90	.16	352	.43	11.48	95
ESE	3.72	.17	1.33	.14	721	.13	1.83	111
SSE	4.37	.23	1.63	.16	746	.35	8.09	92

SUB-AREA 6.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
C	4.86	.34	1.94	.17	541	.44	.89	91
N	4.45	.25	1.69	.17	463	.92	1.21	113
NE	4.19	.22	1.51	.17	583	.21	1.77	108
E	4.97	.28	2.00	.24	463	.65	2.19	151
SE	4.33	.31	1.60	.19	429	.41	1.54	122
S	2.59	.20	.93	.10	412	.31	.91	91
SW	4.28	.30	1.39	.17	365	.11	6.84	107
W	4.67	.36	1.50	.16	374	.14	2.16	86
NW	4.28	.23	1.42	.17	600	.32	.98	143
WSW	3.96	.24	1.33	.15	494	.30	2.40	107
SSW	4.26	.28	1.46	.14	458	.65	4.72	121
WNW	3.92	.22	1.30	.16	713	.13	1.30	106
NNW	3.45	.16	1.33	.14	597	.15	1.75	101
ENE	4.96	.24	1.82	.23	549	.17	2.66	99
NNE	4.16	.21	1.46	.16	746	.49	1.82	132
ESE	4.69	.28	1.90	.17	403	.42	5.16	328
SSE	4.21	.28	1.44	.16	333	.87	2.65	144

SUB-AREA 7.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
C	4.08	.23	1.71	.13	696	.30	.21	105
N	2.27	.17	.57	.08	599	.29	1.56	158
NE	5.81	.37	2.51	.14	566	.30	1.01	81
E	4.16	.14	1.46	.17	360	.22	1.36	103
SE	3.46	.26	1.28	.10	455	.59	2.15	157
S	4.06	.32	1.50	.13	707	.29	1.55	143
SW	3.61	.23	1.39	.13	762	.32	1.11	112
W	4.62	.31	1.79	.17	843	.27	.87	79
NW	4.05	.19	1.60	.18	741	.27	.97	97
WSW	3.79	.27	1.32	.17	678	.44	.71	120
SSW	4.18	.31	1.40	.14	583	.34	1.07	52
WNW	3.69	.17	1.27	.16	403	.02	.58	77
NNW	3.23	.17	1.09	.17	523	.17	.81	124
ENE	2.55	.19	.93	.11	352	.21	.71	90
NNE	4.26	.27	1.14	.17	669	.19	1.18	107
ESE	4.66	.23	1.69	.19	643	.05	1.00	172
SSE	5.17	.32	1.78	.19	695	.30	1.07	114

SUB-AREA 8.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
C	2.91	.16	.94	.09	472	.36	.90	114
N	3.32	.17	1.05	.12	686	.22	.85	87
NE	5.03	.22	1.85	.20	798	.32	1.02	145
E	1.42	.10	.36	.13	360	.23	.72	86
SE	2.11	.16	.56	.09	395	.30	.71	113
S	3.24	.18	.82	.12	601	.28	1.36	111
SW	2.72	.19	.91	.08	559	.19	.52	108
W	2.48	.15	.77	.06	463	.28	1.19	172
NW	3.45	.14	1.23	.08	576	.44	1.07	136
WSW	4.27	.19	1.42	.16	884	.47	.169	160
SSW	4.22	.19	1.54	.13	591	.28	.81	136
WNW	3.65	.16	.81	.08	412	.34	3.24	110
NNW	4.04	.17	1.33	.15	635	.25	.63	108
ENE	2.86	.17	.93	.15	498	.28	1.21	78
NNE	4.18	.19	1.43	.14	739	.29	1.10	109
ESE	2.05	.15	.60	.08	378	.13	.49	64
SSE	4.35	.19	1.97	.09	772	.23	.84	108

SUB-AREA 9.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
C	2.36	.23	.77	.10	261	.16	.62	94
N	1.84	.16	.65	.07	233	.10	.65	171
NE	2.01	.20	.68	.11	204	.29	.66	113
E	2.03	.19	.63	.10	199	.04	.51	62
SE	2.36	.19	.80	.10	185	.14	.81	124
S	2.86	.17	.96	.15	549	.36	.84	136
SW	2.93	.21	.90	.13	395	.25	.86	109
W	3.04	.22	.96	.13	403	.03	.50	74
NW	3.08	.18	1.04	.11	463	.27	.71	103
WSW	2.93	.21	.88	.11	472	.17	.82	85
SSW	2.91	.19	.84	.14	566	.54	.86	142
WNW	3.07	.21	.88	.13	420	.35	.95	114
NNW	3.17	.21	1.01	.16	463	.21	.53	101
ENE	2.18	.16	.71	.11	264	.24	.45	93
NNE	2.25	.23	.71	.10	319	.16	.56	90
ESE	2.29	.19	.78	.09	214	.19	.47	101
SSE	2.38	.14	.68	.11	309	.23	.86	101

ii). Soil Analytical Results (September 1949)

SUB-AREA 1.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
C	.15	.47	4.7	12.2
N	.16	.58	4.8	14.1
NE	.33	.62	4.7	15.8
E	.29	.66	4.6	16.2
SE	.25	.53	5.2	14.8
S	.26	.40	5.4	12.1
SW	.10	.45	5.4	18.0
W	.34	1.13	4.7	12.0
NW	.18	2.23	4.7	16.2

SUB-AREA 2.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
C	.13	.38	4.4	12.0
N	.29	.48	4.8	12.0
NE	.30	1.72	4.3	13.2
E	.31	1.37	4.4	12.2
SE	.21	1.12	4.5	12.8
S	.36	1.65	4.9	14.5
SW	.20	.47	4.4	16.1
W	.22	.70	4.3	10.8
NW	.21	2.62	4.5	11.8

SUB-AREA 3.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
C	.24	.88	4.1	10.1
N	.19	.41	4.5	9.8
NE	.11	.41	4.0	9.0
E	.10	.36	4.5	10.2
SE	.05	.43	4.1	11.4
S	.19	.30	4.2	12.2
SW	.15	.35	4.4	10.0
W	.17	.40	4.5	9.6
NW	.30	.80	4.5	8.2

SUB-AREA 4.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
C	.17	.31	4.4	9.9
N	.21	.38	4.1	10.1
NE	.22	.34	4.2	11.2
E	.10	.48	4.5	9.2
SE	.14	.38	4.3	12.6
S	.10	.46	3.9	11.1
SW	.03	.50	3.9	9.0
W	.18	.69	4.0	9.6
NW	.24	.61	4.2	9.6

SUB-AREA 6.

Sample	p.p.m.			
	Co	Ni	pH	% L.I.
C	.35	.64	4.0	20.0
N	.16	1.45	4.2	19.6
NE	.22	1.70	4.2	17.2
E	.20	1.42	3.9	15.0
SE	.17	1.40	4.2	19.0
S	.14	1.38	4.1	15.2
SW	.28	1.83	4.1	12.8
W	.42	1.36	4.3	19.0
NW	.35	.28	4.2	19.0

SUB-AREA 7.

Sample	p.p.m.			
	Co	Ni	pH	% L.I.
C	.22	1.89	4.6	25.1
N	.68	.49	4.3	28.2
NE	1.09	.33	4.3	20.0
E	Rejected			
SE	.18	.58	4.1	20.1
S	.26	.43	4.3	30.1
SW	.05	.29	4.3	22.8
W	.38	.37	4.3	21.2
NW	.20	.34	4.4	19.8

SUB-AREA 8.

Sample	p.p.m.			
	Co	Ni	pH	% L.I.
C	.20	.22	4.3	17.2
N	2.77	.39	4.3	19.6
NE	.12	.32	4.2	17.8
E	.12	.38	4.1	17.1
SE	.14	.28	4.2	19.6
S	.17	.36	4.5	19.0
SW	.44	.31	4.5	20.5
W	.16	.33	4.1	21.2
NW	.10	.18	4.2	20.0

SUB-AREA 9.

Sample	p.p.m.			
	Co	Ni	pH	% L.I.
C	.21	.51	3.9	73.1
N	.42	.50	4.2	80.6
NE	.14	.53	4.0	78.9
E	.10	.38	4.0	72.6
SE	.15	.48	3.9	72.8
S	.25	.48	3.9	72.1
SW	.19	.54	3.9	84.1
W	.58	.80	4.1	82.4
NW	.40	.24	4.1	82.0

b). i). Pasture Analytical Results (June 1950).

SUB-AREA 3.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	3.78	.17	1.44	.11	664	.08	.40	139
2	4.91	.19	1.82	.16	731	.05	1.18	108
3	4.36	.19	1.44	.12	358	.09	1.51	146
4	4.71	.20	1.78	.11	478	.10	1.17	113
5	4.44	.24	1.52	.14	615	.05	.54	116
6	3.61	.17	1.28	.10	424	.05	.89	161
7	4.68	.18	1.61	.17	433	.05	.65	136
8	4.88	.23	1.68	.17	570	.02	.78	111
B1	4.47	.15	1.69	.10	490	.04	.76	119
2	5.28	.23	1.78	.14	524	.03	.86	116
3	4.64	.17	1.74	.11	484	.04	.77	137
4	4.80	.17	1.70	.14	604	.08	.85	163
5	4.65	.19	1.76	.15	570	.08	.78	116
6	4.33	.19	1.51	.13	686	.12	2.27	184
7	4.38	.18	1.59	.14	632	.12	1.26	162
8	4.89	.18	1.77	.14	559	.09	1.57	110
C1	5.07	.19	2.08	.15	662	.06	3.02	164
2	4.03	.17	1.53	.12	567	.10	1.42	189
3	3.96	.26	1.69	.13	628	.23	1.29	193
4	4.87	.16	1.82	.13	584	.06	1.66	144
5	4.89	.19	1.81	.13	561	.06	1.66	143
6	4.51	.19	1.67	.17	608	.08	1.65	155
7	4.33	.20	1.55	.12	649	.06	.67	148
8	4.65	.17	1.69	.11	641	.06	1.16	140

SUB-AREA 6.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	4.80	.29	1.82	.16	635	.09	1.68	170
2	4.51	.25	1.74	.15	616	.08	.76	148
3	4.70	.20	1.81	.15	555	.08	.96	132
4	4.47	.20	1.69	.16	512	.06	1.18	125
5	4.38	.16	1.69	.17	524	.08	.85	136
6	4.19	.19	1.69	.15	473	.06	1.00	111
7	4.28	.19	1.71	.16	464	.06	.83	121
8	4.49	.18	1.70	.16	379	.09	.90	139
9	4.49	.20	1.66	.22	489	.04	1.06	128
10	4.44	.21	1.59	.13	467	.04	.99	139
11	4.24	.20	1.54	.14	496	.05	1.13	145
12	4.12	.26	1.40	.17	566	.09	1.22	178
C1	3.90	.21	1.50	.14	733	.04	1.01	144
2	4.50	.28	1.48	.12	660	.09	1.09	159
3	4.36	.20	1.40	.13	604	.12	1.25	172
4	4.45	.21	1.48	.13	700	.09	1.14	152
5	4.33	.22	1.52	.13	652	.15	1.32	145
6	4.51	.26	1.61	.11	610	.15	.85	131
7	4.85	.21	1.61	.15	648	.12	1.19	146
8	4.53	.21	1.51	.13	657	.09	1.08	160
9	4.45	.13	1.54	.13	627	.07	1.25	151
10	4.35	.20	1.49	.11	633	.06	1.24	120
11	4.38	.20	1.49	.13	642	.07	.67	140
12	4.07	.17	1.53	.14	655	.12	1.71	207

SUB-AREA 7.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	2.79	.25	.86	.08	350	.13	.74	246
2	2.56	.17	.81	.08	343	.12	.82	260
3	3.06	.15	.96	.09	339	.13	.74	267
4	3.38	.13	1.18	.11	329	.12	.88	228
5	4.45	.17	1.51	.13	618	.03	1.06	190
6	4.27	.18	1.47	.14	545	.14	.71	237
7	4.76	.19	1.66	.13	694	.09	.81	197
8	3.01	.16	1.01	.09	320	.15	.84	236
B1	2.71	.13	.85	.08	347	.13	.78	237
2	3.58	.12	1.54	.11	470	.06	1.02	152
3	4.04	.14	1.54	.12	649	.09	1.03	161
4	5.16	.18	1.72	.14	741	.07	1.01	168
5	3.46	.17	1.25	.10	500	.09	1.03	170
6	4.28	.13	1.29	.11	409	.13	.99	223
7	3.41	.14	1.08	.11	293	.04	1.13	99
8	4.25	.17	1.27	.15	585	.12	1.03	224
C1	4.47	.19	1.33	.13	504	.12	1.25	268
2	3.66	.15	1.19	.11	416	.11	1.19	173
3	3.22	.15	1.06	.10	354	.11	.56	294
4	4.43	.21	1.49	.13	725	.03	1.21	210
5	3.81	.14	.94	.11	503	.08	1.30	228
6	4.41	.18	1.36	.14	642	.46	1.39	246
7	3.08	.14	1.06	.10	352	.23	1.23	248
8	4.60	.18	1.39	.13	756	.14	1.12	210

SUB-AREA 8.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	4.00	.15	1.28	.10	506	.10	1.43	190
2	3.35	.12	1.05	.09	400	.11	1.15	195
3	3.50	.16	1.26	.10	325	.29	3.20	421
4	4.13	.14	1.45	.12	545	.06	.88	157
5	4.55	.16	1.53	.11	346	.14	1.56	197
6	3.77	.15	1.28	.10	396	.08	1.34	172
7	2.90	.13	.88	.10	288	.07	1.18	217
8	3.63	.14	1.09	.11	381	.12	1.41	242
B1	4.54	.13	1.53	.12	477	.05	.94	112
2	3.86	.15	1.21	.11	572	.06	1.12	129
3	4.39	.17	1.45	.11	570	.08	1.59	168
4	3.67	.17	1.09	.10	395	.05	1.00	194
5	4.89	.15	1.54	.14	417	.07	.77	133
6	4.68	.21	1.38	.17	616	.06	2.00	160
7	4.57	.11	1.45	.13	523	.17	3.44	211
8	3.38	.14	1.10	.10	369	.09	1.30	243
C1	3.40	.13	.96	.10	394	.11	1.08	201
2	3.97	.13	1.30	.11	440	.12	1.23	228
3	2.92	.11	.93	.08	360	.09	.88	161
4	4.36	.14	1.37	.13	496	.06	.86	131
5	3.99	.13	1.05	.10	571	.08	.76	197
6	4.62	.14	1.25	.11	550	.09	.75	193
7	4.32	.15	1.22	.11	548	.10	1.73	225
8	4.10	.14	1.32	.13	604	.09	1.07	186

SUB-AREA 9.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	3.61	.14	1.29	.14	413	.06	1.73	103
2	3.23	.10	.69	.05	179	.05	2.55	133
3	3.14	.13	1.15	.11	374	.03	4.38	45
4	3.21	.15	1.07	.12	399	.10	1.04	159
5	4.49	.14	1.18	.11	336	.10	1.56	225
6	3.41	.11	1.20	.11	326	.06	1.74	265
7	4.22	.13	1.30	.11	260	.14	2.68	234
8	4.21	.13	1.13	.11	321	.14	3.38	290
B1	3.88	.14	1.16	.11	346	.13	1.53	200
2	3.19	.12	1.11	.10	208	.12	2.21	202
3	4.50	.14	1.18	.14	285	.07	1.22	119
4	3.40	.11	1.19	.11	299	.10	2.61	166
5	3.57	.14	1.40	.11	356	.03	.90	74
6	4.09	.14	1.32	.15	315	.06	1.03	103
7	Rejected							
8	3.06	.14	1.25	.13	364	.06	1.32	105
C1	3.55	.14	1.26	.13	279	.05	1.23	105
2	3.04	.17	1.05	.09	538	.06	1.18	125
3	3.23	.16	.99	.14	457	.10	1.47	120
4	3.66	.17	1.25	.14	389	.08	1.24	147
5	3.27	.15	1.37	.16	274	.10	1.96	157
6	3.24	.12	1.14	.10	209	.06	.98	116
7	3.35	.13	1.08	.11	292	.04	.98	154
8	3.45	.14	1.03	.13	238	.07	1.62	156

SUB-AREA 10.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	2.28	.13	.69	.07	275	.16	2.08	234
2	2.40	.14	.66	.08	282	.22	2.05	262
3	3.36	.12	.61	.07	290	.16	2.54	239
4	3.18	.14	.85	.11	359	.16	2.77	228
5	3.33	.17	1.10	.10	379	.11	1.89	163
6	2.33	.14	.64	.07	193	.10	1.81	145
7	2.31	.12	.66	.08	239	.11	1.43	147
8	2.49	.15	.71	.08	218	.12	2.07	184
B1	2.62	.13	.77	.07	343	.21	3.01	332
2	2.63	.14	.81	.07	357	.08	2.56	310
3	2.09	.17	.61	.05	356	.17	3.09	278
4	2.51	.14	.77	.07	208	.22	3.73	330
5	2.37	.18	.83	.09	374	.28	4.43	299
6	2.07	.11	.62	.07	265	.27	3.46	434
7	2.28	.15	.66	.10	293	.25	3.24	355
8	2.13	.12	.58	.08	265	.16	3.67	233
C1	3.37	.15	1.05	.11	442	.14	2.20	212
2	2.98	.16	.91	.08	396	.19	2.58	277
3	2.77	.14	.86	.09	356	.20	3.07	264
4	2.48	.12	.81	.07	283	.13	4.84	249
5	3.06	.14	.94	.11	357	.12	3.53	242
6	2.35	.13	.66	.07	283	.15	9.34	222
7	2.49	.14	.79	.09	300	.15	2.88	244
8	2.84	.14	.88	.12	313	.13	4.50	326

SUB-AREA 11.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	2.83	.11	.83	.07	414	.05	1.82	189
2	2.40	.11	.71	.07	376	.11	1.93	254
3	2.61	.10	.77	.08	298	.13	3.00	242
4	2.24	.09	.58	.07	349	.06	1.15	259
5	2.84	.09	.76	.07	400	.13	1.84	241
6	2.43	.10	.70	.07	328	.21	4.02	219
7	2.27	.11	.58	.08	272	.14	.81	276
8	3.00	.10	.86	.07	319	.19	1.50	195
B1	2.79	.09	.79	.07	420	.14	2.34	202
2	2.71	.09	.81	.07	352	.18	2.26	240
3	2.44	.09	.63	.08	331	.11	1.97	234
4	2.74	.10	.73	.06	456	.17	1.51	302
5	3.00	.13	1.11	.08	402	.15	.88	198
6	2.28	.12	.82	.06	274	.16	1.62	329
7	3.33	.11	1.10	.11	439	.17	2.10	210
8	2.63	.11	.83	.08	376	.17	2.11	208
C1	2.72	.11	.94	.08	373	.11	2.74	258
2	2.32	.10	.86	.08	316	.14	3.19	246
3	2.77	.13	.96	.08	438	.17	3.85	278
4	2.56	.14	.98	.07	393	.16	1.25	190
5	3.23	.10	.92	.07	393	.18	2.64	243
6	2.46	.11	.79	.06	374	.16	2.92	228
7	2.71	.13	.75	.11	284	.12	1.25	180
8	2.70	.11	.87	.08	463	.18	2.75	282

SUB-AREA 12.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	2.86	.10	.96	.13	282	.12	1.13	210
2	2.69	.09	.83	.13	303	.11	1.12	216
3	2.69	.10	.88	.14	224	.14	1.31	190
4	3.37	.13	1.22	.14	326	.10	3.80	137
5	2.17	.09	.69	.09	220	.11	.71	204
6	2.91	.15	.99	.11	198	.14	1.33	195
7	2.58	.10	.86	.10	193	.13	1.37	241
8	2.23	.11	.66	.10	191	.13	1.84	222
B1	2.87	.10	.96	.13	307	.10	.94	200
2	2.44	.09	.61	.11	229	.17	2.97	315
3	3.01	.10	.96	.09	275	.30	1.13	241
4	2.49	.09	.58	.10	220	.10	1.34	180
5	2.45	.11	.82	.10	155	.13	1.48	222
6	2.08	.10	.63	.08	162	.15	2.39	280
7	2.78	.17	.62	.08	156	.25	3.23	315
8	2.16	.09	.66	.08	158	.26	2.25	300
C1	3.20	.11	1.06	.16	323	.10	4.37	198
2	1.84	.10	.70	.10	178	.16	1.72	233
3	2.72	.09	.87	.10	222	.06	1.02	180
4	2.42	.11	.78	.10	177	.13	1.67	220
5	2.65	.11	.96	.10	231	.04	5.10	100
6	2.16	.11	.73	.09	154	.16	2.05	291
7	2.12	.10	.72	.10	149	.05	.52	355
8	2.50	.12	.80	.10	158	.08	1.36	224

ii) Soil Analytical Results
(June 1950)

SUB-AREA 3.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.29	.42	4.5	9.9
2	.19	.34	4.2	12.1
3	.20	.34	4.4	10.8
4	.15	.27	4.4	10.0
5	.16	.36	4.0	11.1
6	.17	.41	4.1	11.9
7	.14	.32	4.4	10.0
8	.15	.32	4.0	9.8
B1	.24	.36	4.0	11.6
2	.29	.40	4.6	11.9
3	.20	.34	4.2	10.2
4	.14	.32	4.3	11.0
5	.12	.37	4.4	11.1
6	.14	.31	4.0	11.0
7	.31	.41	4.1	11.0
8	.12	.80	4.3	11.8
C1	.09	.67	4.0	11.1
2	.05	.70	4.0	11.0
3	.10	.71	4.1	11.9
4	.08	1.03	4.0	11.2
5	.11	.65	4.6	11.6
6	.18	.78	4.3	11.0
7	.14	.66	4.6	11.0
8	.09	.61	4.4	11.6

SUB-AREA 6.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.27	.45	4.3	22.1
2	.23	.36	4.3	28.2
3	.26	.56	4.1	20.0
4	.22	.43	4.2	25.2
5	.24	.32	4.2	26.2
6	.24	.20	4.3	22.9
7	.24	.33	4.1	30.1
8	.33	.32	4.4	30.0
9	.21	.36	4.0	30.0
10	.27	.35	4.0	29.0
11	.22	.25	4.0	30.0
12	.25	.42	4.6	30.0
C1	.19	.37	4.3	22.0
2	.16	.28	4.1	26.0
3	.08	.28	4.1	21.2
4	.16	.26	4.1	20.0
5	.21	.36	4.2	22.8
6	.13	.41	4.1	21.9
7	.32	.36	4.1	20.2
8	.18	.41	4.0	22.2
9	.35	.30	4.0	21.2
10	.22	.32	4.2	28.1
11	.19	.48	4.4	26.0
12	.20	.34	4.0	24.2

SUB-AREA 7.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.11	.48	4.4	25.8
2	.22	.59	4.2	22.1
3	.11	.75	4.0	28.6
4	.08	.68	4.0	26.4
5	.12	.57	4.0	22.1
6	.10	.62	4.2	26.4
7	.16	.60	4.1	27.9
8	.09	1.19	4.4	26.2
B1	.06	1.30	4.0	28.2
2	.05	.92	3.9	29.8
3	.08	.35	4.2	31.0
4	.10	.68	4.0	20.1
5	.10	.40	4.3	20.0
6	.13	.49	4.0	20.0
7	.15	.34	4.4	22.8
8	.13	.62	4.4	24.6
C1	.05	.59	4.2	21.8
2	.08	.53	4.6	24.9
3	.07	.37	4.0	26.1
4	.07	.37	4.0	20.0
5	.15	.95	4.0	20.9
6	.14	.97	4.3	19.2
7	.42	.35	4.3	18.7
8	.19	.52	4.3	19.6

SUB-AREA 8.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.09	1.36	4.3	24.0
2	.10	1.09	4.3	22.0
3	.11	.57	4.2	26.8
4	.06	.57	4.1	23.5
5	.18	.76	4.2	23.5
6	.24	.99	4.5	29.1
7	.15	.59	4.5	20.8
8	.41	.93	4.2	20.8
B1	.49	.64	4.3	18.6
2	.14	.70	4.2	21.6
3	.22	.52	4.2	20.2
4	.37	.59	4.2	19.9
5	.08	.55	4.2	19.9
6	.09	.54	4.2	23.4
7	.11	.72	4.1	16.8
8	.14	.63	4.3	22.1
C1	.16	.42	3.9	31.2
2	.07	1.32	4.0	20.6
3	.09	.45	4.1	21.2
4	Rejected			
5	.26	.31	4.1	22.1
6	.25	.38	4.3	21.1
7	.27	.40	4.3	21.1
8	.21	.46	4.4	30.6

SUB-AREA 9.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.10	1.44	3.8	74.1
2	.20	2.46	3.8	76.2
3	.18	1.17	3.8	78.1
4	.28	1.45	3.9	81.2
5	.43	1.17	4.0	80.6
6	.71	2.97	3.9	84.1
7	Rejected			
8	Rejected			
B1	.39	1.81	3.9	75.7
2	.41	1.49	3.9	62.1
3	.48	.47	3.9	72.8
4	.55	2.19	3.9	76.1
5	.38	.82	3.9	80.8
6	.54	1.14	4.0	84.1
7	.35	1.62	4.0	80.8
8	.19	1.59	3.9	81.4
C1	.25	.92	4.0	76.2
2	.39	.60	4.1	78.9
3	.68	.82	4.2	78.8
4	1.01	.57	3.9	72.1
5	.25	.33	3.9	80.4
6	.37	.29	3.9	78.2
7	.33	.74	3.9	74.0
8	.31	.41	3.9	75.0

SUB-AREA 10.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.48	1.11	4.8	24.8
2	Rejected			
3	.43	1.06	4.3	21.0
4	.55	1.20	4.5	20.8
5	.51	1.09	4.8	26.2
6	.54	.94	4.8	26.0
7	.45	1.02	4.6	28.0
8	.33	1.00	4.5	27.8
B1	.40	.87	4.2	24.6
2	.32	.98	4.6	22.6
3	.39	.85	4.7	24.7
4	Rejected			
5	.43	.83	4.7	22.8
6	.37	.98	4.8	29.6
7	Rejected			
8	.34	.96	4.6	29.0
C1	.46	1.00	4.6	21.2
2	.35	.89	4.6	18.6
3	.30	.93	4.6	21.8
4	Rejected			
5	.38	.77	4.7	15.6
6	.19	.82	4.4	21.2
7	.28	.75	4.7	20.0
8	.28	1.07	4.4	16.2

SUB-AREA 11.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.45	.73	3.9	29.9
2	.35	.76	4.0	38.1
3	.47	.82	4.0	42.1
4	.25	.48	3.9	32.1
5	.28	.48	4.1	46.2
6	.47	.47	4.1	38.2
7	.12	.33	4.1	41.2
8	.48	.61	3.9	43.0
B1	.73	.65	3.9	28.1
2	1.05	.78	3.9	38.7
3	.56	.80	4.0	38.0
4	.42	.80	4.0	32.1
5	.30	3.03	4.2	40.8
6	.20	.32	4.0	32.6
7	.38	1.41	4.0	40.0
8	.54	3.31	4.1	40.8
C1	.34	1.02	5.0	42.0
2	.46	2.11	4.2	42.0
3	.56	1.40	4.1	47.6
4	.58	1.53	4.0	46.2
5	.53	1.35	4.1	40.8
6	.52	3.26	4.4	41.6
7	.10	1.00	4.0	48.2
8	.22	.93	4.4	49.1

SUB-AREA 12.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.61	4.03	4.2	50.1
2	.53	3.90	4.1	62.0
3	.55	1.36	4.3	40.7
4	1.14	.90	4.0	46.0
5	.41	1.28	4.2	42.8
6	.45	1.27	4.0	56.4
7	.35	7.06	3.9	58.2
8	.38	6.18	4.0	60.0
B1	.71	1.32	4.0	52.8
2	.54	1.35	4.1	62.0
3	.29	1.37	4.1	41.2
4	.27	2.31	4.1	50.8
5	.29	1.69	4.1	60.3
6	.35	2.07	4.4	52.8
7	.39	1.41	4.3	58.6
8	.24	2.03	4.1	52.7
C1	.52	2.78	4.6	56.1
2	.22	1.16	4.1	60.0
3	.31	1.32	4.1	40.8
4	.38	1.10	4.1	42.1
5	.32	1.21	4.3	66.0
6	.22	1.22	4.1	52.6
7	.11	1.04	3.9	58.5
8	.12	.92	4.3	56.7

c). i). Pasture Analytical Results (September 1950).

SUB-AREA 3.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	5.08	.17	1.73	.19	785	.12	.56	88
2	Rejected							
3	5.64	.16	2.03	.25	720	.03	.53	76
4	5.21	.18	1.81	.26	756	.04	.89	85
5	4.08	.19	1.40	.19	719	.08	1.11	125
6	3.46	.14	1.13	.12	575	.14	1.13	200
7	3.83	.14	1.34	.13	802	.13	1.44	154
8	4.59	.17	1.73	.18	646	.10	1.43	157
9	5.87	.17	2.04	.26	760	.08	1.79	106
10	5.26	.17	1.99	.22	707	.12	.73	56
11	3.79	.16	1.54	.13	713	.08	2.01	141
12	4.83	.16	1.49	.17	705	.13	1.23	43
B1	5.65	.17	2.13	.21	932	.09	4.59	130
2	5.21	.15	1.94	.16	1,149	.19	3.60	164
3	5.84	.16	1.99	.22	711	.06	5.45	108
4	5.86	.15	2.10	.24	714	.05	2.70	59
5	6.02	.15	2.13	.25	903	.03	1.80	60
6	5.26	.16	1.99	.23	727	.21	.56	48
7	6.05	.18	2.20	.23	950	.11	1.32	88
8	6.11	.16	2.18	.23	679	.08	.72	62
9	5.35	.17	2.00	.20	1206	.03	5.39	50
10	4.93	.15	1.76	.20	508	.07	2.06	99
11	6.56	.15	2.27	.25	416	.12	1.11	89
12	5.79	.17	2.11	.24	699	.02	1.66	71

SUB-AREA 6.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	3.30	.20	1.49	.18	550	.08	1.59	95
2	4.42	.19	1.55	.20	527	.06	1.20	138
3	3.97	.19	1.24	.20	548	.08	1.69	109
4	4.72	.17	1.49	.19	480	.05	1.38	82
5	4.52	.21	1.64	.23	612	.04	.87	85
6	4.12	.17	1.36	.23	574	.05	.86	102
7	5.06	.21	1.61	.26	646	.06	1.30	200
8	4.45	.17	1.66	.26	617	.04	.92	92
B1	4.66	.17	1.73	.22	735	.10	.95	88
2	4.87	.20	1.78	.22	652	.04	1.03	86
3	4.58	.19	1.74	.20	781	.04	.61	78
4	4.31	.19	1.55	.20	687	.06	1.59	90
5	4.85	.24	1.82	.26	780	.03	1.16	41
6	4.52	.21	1.78	.24	758	.02	1.16	105
7	4.65	.26	1.78	.24	831	.05	3.75	108
8	5.21	.18	2.15	.23	782	.07	2.70	125
C1	5.53	.21	2.08	.24	691	.04	1.00	71
2	3.77	.26	1.94	.23	611	.04	1.23	95
3	4.67	.23	1.75	.20	700	.06	1.12	95
4	4.98	.23	1.80	.25	739	.05	1.62	114
5	4.88	.20	1.72	.23	614	.08	5.45	91
6	5.20	.21	2.03	.25	617	.05	2.24	95
7	5.32	.26	1.88	.28	652	.05	1.56	75
8	5.11	.21	1.95	.25	601	.06	2.18	132

SUB-AREA 7.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	3.03	.19	1.08	.15	486	.09	2.68	180
2	2.93	.17	1.01	.16	583	.16	.97	147
3	4.55	.18	1.63	.23	741	.02	.81	63
4	3.97	.18	1.41	.16	531	.19	2.45	276
5	3.73	.17	1.62	.17	697	.08	1.07	98
6	4.01	.15	1.50	.19	669	.03	.54	109
7	4.44	.15	1.69	.19	552	.05	.59	90
8	4.08	.19	1.64	.19	513	.03	.51	94
B1	3.68	.16	1.35	.16	626	3.17	.78	117
2	5.03	.17	1.88	.22	710	.06	.84	83
3	3.88	.21	1.34	.17	721	.09	.84	151
4	5.08	.19	1.98	.25	735	.09	.76	109
5	4.23	.14	1.51	.18	551	.05	.55	83
6	5.16	.14	1.83	.23	603	.04	.42	44
7	4.48	.14	1.53	.23	719	.08	.83	109
8	4.39	.17	1.60	.25	551	.04	.32	36
C1	5.13	.17	1.78	.23	702	.04	.24	29
2	3.78	.14	1.67	.19	509	.03	.30	42
3	4.93	.15	1.84	.24	700	.05	.94	73
4	4.70	.14	1.78	.23	808	.02	.87	81
5	3.97	.13	1.54	.14	436	.05	.43	13
6	4.62	.14	1.75	.21	435	.06	.57	162
7	4.29	.14	1.61	.17	484	.03	1.06	90
8	4.75	.14	1.61	.22	829	.08	.52	66

SUB-AREA 8.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	3.26	.14	1.36	.22	1073	.12	1.26	121
2	4.15	.14	1.41	.17	754	.11	.65	93
3	4.89	.15	1.54	.19	1089	.10	.89	108
4	3.76	.16	1.32	.16	602	.07	.39	91
5	4.20	.15	1.39	.20	891	.13	1.24	128
6	4.29	.16	1.55	.23	1153	.40	1.70	203
7	3.52	.15	1.25	.14	465	.15	2.10	131
8	3.05	.15	1.05	.14	398	.21	1.18	179
B1	4.61	.15	1.69	.23	1160	.25	1.13	155
2	4.36	.14	1.72	.16	674	.19	1.10	133
3	5.04	.16	1.88	.25	989	.11	1.22	118
4	3.73	.14	1.43	.16	499	.30	1.74	218
5	3.58	.14	1.34	.14	467	.19	1.11	157
6	5.23	.16	2.11	.20	810	.18	.98	127
7	5.51	.14	2.25	.22	860	.24	.70	195
8	3.36	.17	1.29	.14	355	.23	1.22	273
C1	4.72	.16	1.81	.19	838	.14	.41	109
2	4.69	.14	1.84	.18	870	.16	.92	99
3	4.67	.16	1.84	.19	852	.13	.88	98
4	4.13	.15	1.59	.19	725	.33	.84	135
5	4.47	.14	1.67	.23	849	.19	1.21	133
6	4.13	.16	2.37	.24	795	.13	.73	86
7	4.78	.14	1.92	.19	714	.16	.97	129
8	3.13	.14	.99	.16	397	.26	1.04	157

SUB-AREA 9.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	1.95	.12	.65	.12	234	.15	.61	112
2	3.05	.12	.63	.12	226	.14	.31	74
3	2.05	.13	.66	.14	239	.21	.44	91
4	2.20	.14	.61	.14	277	.13	.58	142
5	2.51	.17	.69	.17	338	.13	.53	79
6	2.15	.11	.62	.19	170	.20	.66	153
7	2.63	.14	.87	.15	352	.20	.72	123
8	2.17	.11	.61	.14	220	.19	.86	134
B1	2.73	.14	.82	.13	331	.15	.63	102
2	2.94	.12	.93	.13	269	.11	.91	85
3	2.81	.15	.87	.15	299	.31	.67	102
4	2.47	.12	.86	.13	228	.11	.67	183
5	3.73	.19	.97	.27	199	.18	1.07	86
6	2.98	.17	.90	.14	371	.35	.97	103
7	2.86	.17	.88	.12	348	.27	1.36	104
8	2.74	.17	1.13	.19	224	.43	2.29	104
C1	2.90	.18	.87	.17	284	.16	.64	81
2	2.75	.17	.91	.17	280	.17	.41	39
3	2.66	.17	.74	.17	301	.11	.25	48
4	2.60	.17	.86	.17	278	.22	.53	71
5	2.87	.19	.87	.21	255	.10	.39	57
6	3.18	.20	.94	.20	283	.04	.30	43
7	3.26	.21	.95	.23	464	.11	.55	77
8	2.85	.17	.93	.20	164	.04	.54	92

SUB-AREA 10.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	2.76	.16	1.01	.11	370	.18	3.04	200
2	2.48	.17	.99	.11	386	.30	2.16	232
3	3.05	.18	1.01	.12	575	.29	2.31	198
4	2.36	.16	.85	.11	386	.13	2.19	238
5	1.69	.13	.71	.10	404	.27	2.29	277
6	2.32	.14	.90	.11	367	.22	2.97	218
7	2.90	.18	.83	.08	386	.28	2.70	321
8	2.47	.15	.85	.10	402	.25	2.68	271
9	2.96	.18	1.08	.14	453	.26	2.32	269
10	2.99	.15	1.05	.13	345	.26	2.99	298
11	Rejected							
12	2.89	.12	.81	.08	344	.19	2.13	266
B1	2.28	.17	.78	.10	367	.24	3.72	337
2	2.68	.17	.64	.13	487	.27	3.65	307
3	2.40	.14	.81	.09	279	.23	3.21	315
4	2.04	.17	.52	.08	518	.17	1.53	255
5	1.89	.15	.42	.08	621	.23	1.88	281
6	1.92	.17	.54	.07	782	.31	2.72	351
7	1.90	.16	.56	.09	589	.58	2.79	432
8	1.83	.12	.56	.07	318	.14	5.69	310
9	3.39	.23	.13	.19	590	.17	3.33	194
10	3.96	.21	.13	.16	632	.22	3.45	198
11	3.52	.25	.13	.19	623	.24	1.08	155
12	3.53	.19	.12	.16	583	.24	2.69	218

SUB-AREA 11.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	2.53	.22	.95	.11	286	.15	1.17	169
2	2.61	.21	1.01	.11	360	.17	1.15	196
3	2.95	.17	1.08	.11	328	.13	1.73	188
4	2.40	.17	.88	.10	354	.22	1.97	283
5	2.32	.15	.89	.10	298	.14	1.07	205
6	1.18	.14	.95	.12	301	.13	.95	174
7	Rejected							
8	2.20	.09	.81	.10	239	.14	1.52	228
9	2.28	.11	.87	.13	315	.19	2.11	248
10	2.40	.11	.95	.12	295	.16	1.26	180
11	2.77	.12	1.07	.11	326	.26	.74	152
12	2.59	.11	1.01	.10	320	.10	.68	149
B1	3.40	.11	1.00	.09	203	.12	2.16	208
2	2.63	.11	.85	.10	252	.36	1.97	211
3	2.50	.11	.76	.08	213	.21	1.87	288
4	2.54	.10	.88	.08	198	.18	1.15	225
5	2.71	.11	.95	.08	360	-	-	-
6	2.45	.11	.75	.08	320	-	-	-
7	2.39	.09	.83	.07	199	.15	1.18	220
8	3.02	.11	1.05	.10	269	.13	.73	161
9	3.05	.11	1.04	.11	252	.13	1.00	135
10	2.42	.11	.75	.08	207	.17	1.18	196
11	2.51	.11	.81	.08	211	.19	1.41	241
12	2.58	.11	.86	.08	204	.11	.125	156

SUB-AREA 12.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	2.80	.08	.97	.14	239	1.57	1.50	176
2	2.33	.07	.83	.17	227	.13	.75	146
3	2.88	.07	1.05	.19	213	.04	.67	109
4	3.13	.06	1.05	.41	119	.23	.68	109
5	2.81	.09	1.00	.14	219	.07	.45	93
6	3.52	.09	1.15	.14	297	.08	.68	102
7	2.83	.09	.99	.13	297	.08	.67	102
8	2.92	.07	.97	.14	188	.10	.65	168
9	2.70	.06	.97	.10	222	.11	.95	176
10	2.76	.06	1.14	.14	173	.20	1.58	262
11	2.51	.07	.99	.21	173	.16	1.07	137
12	2.89	.07	.99	.19	253	.10	1.71	128
B1	2.59	.07	1.06	.15	267	.29	1.24	198
2	2.54	.08	.92	.18	252	.24	1.00	155
3	3.36	.08	1.05	.18	326	.03	.16	40
4	2.67	.07	.88	.15	254	.15	.98	199
5	3.95	.06	.91	.19	230	.22	.97	193
6	2.06	.06	.37	.07	85	.26	1.12	173
7	2.52	.06	.61	.10	128	.11	.98	209
8	2.02	.09	.11	.09	45	.10	.70	140
9	2.93	.05	.45	.10	78	.09	.68	138
10	3.31	.07	.91	.19	161	.30	.79	242
11	2.14	.07	.95	.12	218	.14	.79	243
12	2.82	.07	.93	.14	242	.11	.76	162

ii). Soil Analytical Results (September 1950)

SUB-AREA 3

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.12	.48	4.0	8.2
2	.22	.38	3.9	7.6
3	.11	.30	4.5	6.9
4	.08	.31	4.2	10.1
5	.14	.44	4.5	7.8
6	.18	.50	4.5	7.9
7	.05	.33	4.6	8.1
8	.03	.31	4.5	10.1
9	.10	.28	4.8	6.8
10	.18	.30	5.0	6.9
11	.09	.28	4.2	7.9
12	.17	.31	4.2	8.6
B1	.09	.60	4.5	9.2
2	.15	.40	4.2	8.4
3	.11	.50	4.3	7.6
4	.08	.47	4.1	7.8
5	.05	.44	4.1	6.2
6	.10	.41	4.1	7.8
7	.09	.40	4.2	9.2
8	.29	.67	4.3	8.2
9	.28	.47	4.2	8.8
10	.13	.46	4.1	8.6
11	.08	.50	4.1	9.1
12	.47	.50	4.2	9.3

SUB-AREA 6.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.38	.46	4.5	8.9
2	.51	.36	4.2	7.5
3	.30	1.78	4.8	7.6
4	.39	.27	4.8	7.5
5	.49	.44	4.7	8.2
6	.60	.42	4.2	8.8
7	.47	.43	4.2	9.2
8	.55	.52	4.8	6.9
B1	.30	.21	4.6	6.8
2	.53	.39	4.5	9.2
3	.42	.33	4.5	9.8
4	.59	.37	4.8	9.5
5	——— Rejected ———			
6	.46	.37	4.2	10.2
7	.43	.47	4.2	10.1
8	.48	.40	4.1	10.6
C1	.20	.44	4.8	11.1
2	.52	.57	4.7	8.6
3	.58	.58	4.4	9.2
4	.48	.36	4.4	11.1
5	.42	.48	4.4	8.6
6	.70	.45	4.4	8.9
7	.41	.47	4.2	10.1
8	.27	.37	4.8	11.0

SUB-AREA 7.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.11	.68	4.2	18.1
2	.15	.74	4.2	16.2
3	.27	.49	4.2	12.0
4	.12	.54	4.2	12.8
5	.23	.75	4.3	12.9
6	.21	.57	4.1	11.6
7	.12	.64	4.5	15.2
8	.11	.59	4.5	15.0
B1	.14	.63	4.2	12.8
2	.19	.69	4.2	11.6
3	.09	.52	4.2	11.8
4	.15	.58	4.5	14.0
5	.07	.52	5.0	18.2
6	.02	.28	4.2	18.1
7	.10	.55	4.3	12.6
8	.09	.48	4.2	12.9
C1	.13	.65	4.4	13.5
2	.14	.50	4.2	13.5
3	.10	.58	4.2	12.6
4	.23	.39	4.2	18.0
5	.09	.52	4.2	13.6
6	.15	.73	4.3	13.2
7	.12	.48	4.2	14.6
8	.19	.37	4.1	12.8

SUB-AREA 8.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.20	.51	4.0	9.9
2	.23	.50	4.0	13.6
3	.51	.61	3.9	12.5
4	.18	.50	3.8	12.1
5	.40	.66	3.9	9.9
6	.24	.36	4.9	12.6
7	.16	.72	4.2	13.2
8	.08	.48	4.0	12.8
B1	.34	.51	4.0	10.2
2	.14	.39	4.2	12.8
3	.38	.41	4.0	12.0
4	.13	.45	4.0	11.6
5	.47	.53	4.0	9.2
6	2.40	.57	4.2	9.8
7	.31	.56	3.9	11.6
8	.13	.51	4.1	13.2
C1	.44	.55	4.0	12.1
2	.41	.52	4.0	12.9
3	.55	.58	4.0	13.2
4	.18	.62	4.2	10.9
5	.36	.64	4.1	11.1
6	.41	.54	4.4	12.6
7	.65	.53	4.4	14.6
8	.12	.67	4.0	12.9

SUB-AREA 9.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	Rejected			
2	.22	.48	3.7	72.1
3	.17	.52	3.6	56.2
4	.46	.64	3.8	68.1
5	.27	.82	3.8	72.1
6	.22	.69	3.9	73.6
7	.12	.74	3.8	59.2
8	.41	.69	3.8	61.8
B1	.15	.66	3.9	61.9
2	.30	.62	3.8	75.2
3	Rejected			
4	.24	.72	3.7	60.1
5	.44	1.45	3.7	71.8
6	.20	.96	3.8	72.6
7	.17	.51	4.0	78.1
8	Rejected			
C1	Rejected			
2	.13	.42	3.9	60.8
3	.33	.75	4.2	66.1
4	.11	.47	3.0	72.8
5	Rejected			
6	Rejected			
7	Rejected			
8	Rejected			

SUB-AREA 10.

	p.p.m.			
Sample	Co	Ni	pH	% L.I.
A1	.30	1.30	4.1	12.2
2	.45	1.24	3.9	11.9
3	.29	1.01	4.0	10.1
4	.50	.85	3.8	8.6
5	.24	.92	4.0	9.2
6	.46	.87	3.7	10.2
7	.38	.52	3.7	9.1
8	.68	1.16	3.8	8.6
9	.42	1.39	3.6	7.6
10	.48	.99	4.2	8.2
11	.42	.77	4.2	9.6
12	.39	1.02	4.2	9.2
B1	.89	1.61	4.0	9.8
2	.69	.93	3.9	7.6
3	.64	.99	3.8	6.8
4	.33	.48	3.8	6.8
5	.44	.79	3.9	9.2
6	.55	.89	4.0	9.4
7	.86	.80	3.9	10.2
8	.36	.93	4.0	10.1
9	.54	1.37	4.1	6.9
10	.56	1.30	4.2	7.8
11	.38	1.02	4.1	7.8
12	.34	1.14	3.8	27.6

d). i) Pasture Analytical Results.
(June 1951)

SUB-AREA 12

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
1A	2.71	.09	1.05	.13	316	.14	2.85	261
B	2.45	.07	.79	.11	231	.14	1.83	177
2A	3.94	.10	1.58	.19	448	.09	2.28	185
B	3.20	.10	1.12	.11	362	.25	4.62	318
3A	3.42	.10	1.27	.11	406	.18	3.68	204
B	2.97	.15	1.16	.11	390	.26	5.02	325
4A	3.15	.10	1.12	.13	334	.16	4.45	208
B	2.68	.08	1.08	.09	306	.23	4.60	250
5A	3.28	.11	1.25	.11	320	.15	1.77	200
B	3.15	.10	1.09	.09	362	.08	1.64	137
6A	2.65	.09	.95	.08	317	.15	2.14	197
B	3.18	.10	1.19	.11	329	.11	2.08	186
7A	4.39	.10	1.30	.20	488	.30	1.75	323
B	4.10	.10	1.19	.19	409	.12	1.75	178
8A	3.03	.09	1.01	.11	353	.17	1.85	237
B	2.46	.07	.86	.10	234	.24	2.67	288
9A	2.85	.07	.95	.08	292	.19	3.33	273
B	2.55	.08	.95	.11	283	.09	1.39	167
10A	3.85	.09	1.19	.19	307	.18	2.27	270
B	3.61	.09	1.12	.11	295	.18	1.77	235
11A	3.44	.09	1.17	.10	357	.19	1.94	266
B	2.89	.08	1.00	.13	317	.20	1.76	246
12A	4.33	.09	1.27	.17	440	.14	3.19	185
B	4.82	.11	1.35	.14	403	.19	6.83	236
13A	2.84	.11	1.11	.09	274	.23	2.43	279
B	2.57	.11	1.00	.06	201	.14	2.15	252
14A	2.46	.08	.86	.07	234	.15	1.70	220
B	2.40	.09	.82	.06	202	.22	2.76	269
15A	3.80	.13	1.38	.11	332	.14	1.48	202
B	4.46	.11	1.61	.14	311	.23	2.91	308
16A	3.94	.08	1.35	.13	397	.18	3.19	253
B	3.73	.11	1.26	.17	352	.15	2.02	267

SUB-AREA 9.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
1B	1.16	.07	.88	.11	252	.20	2.25	256
C	2.11	.09	.88	.07	236	.16	1.78	196
2B	2.34	.06	.81	.08	209	.19	1.49	168
C	2.61	.07	.95	.11	274	.07	2.18	92
3B	1.59	.06	.62	.11	196	.20	2.06	222
C	1.68	.06	.70	.11	123	.17	1.94	222
4B	3.33	.06	1.18	.14	326	.10	1.49	144
C	2.50	.06	.87	.10	299	.13	1.59	182
5B	2.79	.07	.87	.34	244	.23	2.51	247
C	2.57	.09	.86	.25	254	.17	2.13	202
6B	3.51	.07	1.10	.26	437	.22	3.01	257
C	2.95	.06	.84	.28	294	.20	4.46	183
7B	2.14	.06	.66	.29	135	.22	2.34	280
C	2.56	.08	.82	.40	162	.21	2.36	239
8B	3.18	.12	1.04	.26	509	.21	1.41	223
C	3.43	.10	1.26	.29	372	.19	.71	231
9B	3.24	.08	1.07	.32	454	.16	1.22	161
C	3.46	.07	1.01	.31	292	.15	1.83	207
10B	3.04	.09	.92	.22	306	.09	.91	140
C	2.42	.07	.74	.29	115	.16	1.44	210
11B	1.58	.06	.99	.07	135	.24	2.08	476
C	2.85	.07	1.01	.08	274	.10	1.39	166
12B	2.42	.06	.92	.08	95	.17	1.47	252
C	2.03	.09	.69	.19	206	.16	1.50	160
13B	2.74	.07	1.01	.16	291	.24	1.56	231
C	2.73	.09	.91	.14	297	.23	1.41	262
14B	1.75	.08	.56	.14	217	.16	1.79	242
C	2.19	.07	.62	.08	178	.17	4.01	237
15B	Rejected							
C	Rejected							
16B	Rejected							
C	Rejected							

SUB-AREA 3.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
1A	4.86	.14	1.99	.16	540	.13	1.54	203
C	4.35	.13	1.76	.13	501	.14	1.45	176
2A	4.12	.12	1.67	.13	542	.11	.91	126
C	4.61	.12	1.66	.16	480	.09	.96	212
3A	4.46	.14	1.74	.14	528	.23	1.07	155
C	5.00	.15	1.87	.20	507	.09	.97	140
4A	5.06	.16	1.90	.22	662	.09	.78	174
C	4.40	.13	1.84	.19	431	.15	1.19	149
5A	3.87	.10	1.51	.15	492	.19	.81	193
C	2.43	.09	.89	.13	302	.16	1.52	192
6A	4.20	.14	1.63	.15	533	.14	1.56	169
C	3.88	.13	1.58	.15	450	.11	2.10	135
7A	4.25	.15	1.93	.24	257	.14	.51	158
C	4.54	.15	2.00	.25	412	.20	4.58	275
8A	4.47	.14	1.89	.21	420	.10	1.27	192
C	4.37	.14	1.82	.19	326	-	-	-
9A	4.15	.18	1.60	.12	352	.16	.83	220
C	4.49	.14	1.76	.23	352	.12	1.31	150
10A	4.28	.11	1.60	.15	420	.18	2.12	239
C	3.76	.14	1.55	.15	378	.14	1.69	192
11A	4.63	.10	1.76	.15	481	.10	.86	137
C	4.31	.10	1.60	.13	395	.24	2.74	269
12A	4.25	.10	1.55	.08	360	.10	1.25	145
C	4.15	.11	1.45	.14	403	.08	1.49	168
13A	4.02	.11	1.68	.14	412	.06	2.33	207
C	3.78	.13	1.33	.14	391	.12	1.78	173
14A	4.00	.14	1.49	.15	472	.15	1.43	169
C	4.31	.17	1.53	.14	391	.19	2.18	200
15A	3.86	.09	1.63	.13	438	.14	1.33	171
C	4.26	.11	1.71	.16	429	.09	.87	137
16A	4.48	.10	1.87	.16	399	.11	1.19	138
C	4.68	.12	1.95	.15	369	.06	.85	88

ii). Soil Analytical Results
(June 1951)

SUB-AREA 12.

	p.p.m.	
Sample	Co	Ni
1D	.21	.67
E	.34	.66
2D	.13	.61
E	.10	.63
3D	.25	.53
E	.14	.77
4D	.07	.63
E	.09	.90
5D	.10	.88
E	.20	1.66
6D	.12	.63
E	.07	.93
7D	.24	1.07
E	.24	.57
8D	.09	1.55
E	.18	.87
9D	.07	.76
E	.14	1.21
10D	.12	.85
E	.11	1.04
11D	.07	.81
E	.08	.74
12D	.06	..76
E	.09	.76
13D	.11	.99
E	.10	1.01
14D	.10	.95
E	.37	.63
15D	.31	.62
E	.27	.77
16D	.19	.59
E	.17	.75

SUB-AREA 9.

	p.p.m.	
Sample	Co	Ni
1F	——— Rejected ———	
G	——— Rejected ———	
2F	——— Rejected ———	
G	——— Rejected ———	
3F	——— Rejected ———	
G	——— Rejected ———	
4F	.16	.76
G	.14	1.78
5F	.38	1.23
G	.24	1.98
6F	.15	.72
G	.15	.78
7F	.26	.90
G	.33	1.17
8F	.05	.78
G	.26	.87
9F	.15	.94
G	.06	.91
10F	.15	.82
G	.24	1.15
11F	.10	.76
G	.08	.62
12F	.12	.99
G	.11	.81
13F	.10	.72
G	.07	.77
14F	.08	.64
G	.10	.66
15F	——— Rejected ———	
G	——— Rejected ———	
16F	——— Rejected ———	
G	——— Rejected ———	

e) Pasture Species Analytical Results
(June 1951).

SUB-AREA 9.

i). Molinia Pasture

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
A1	3.34	.08	1.21	.11	289	.07	1.54	269
2	3.77	.09	1.00	.14	344	.05	1.19	160
3	3.61	.08	1.29	.13	294	.06	1.71	175
4	3.57	.09	1.10	.14	253	.06	2.22	196
5	3.42	.07	.98	.15	225	.06	1.39	156
6	3.47	.07	1.01	.11	228	.16	2.52	261
7	3.39	.07	1.12	.11	268	.07	1.22	119
8	3.43	.06	1.08	.12	233	.04	1.34	122
9	3.50	.06	1.04	.13	246	.06	.69	76
10	3.47	.07	.98	.14	188	.07	3.22	51
B1	3.67	.08	1.23	.13	256	.04	.67	86
2	3.50	.07	1.05	.11	259	.06	.84	84
3	3.00	.08	1.04	.14	370	.04	1.44	93
4	3.24	.08	1.04	.13	398	.09	1.74	120
5	3.36	.07	.97	.13	340	.05	.71	101
6	3.24	.08	1.04	.14	290	.05	1.35	116
7	3.20	.07	1.00	.14	215	.06	2.07	126
8	3.02	.08	.91	.13	254	.07	1.17	162
9	3.33	.08	.92	.14	236	.06	1.56	140
10	3.24	.07	1.01	.15	255	.05	1.40	137
C1	3.22	.07	1.05	.15	220	.09	2.24	123
2	3.14	.07	.93	.14	304	.06	1.68	133
3	3.13	.07	1.00	.13	364	.06	.53	74
4	2.93	.07	.95	.13	315	.10	.85	114
5	3.31	.08	1.16	.14	256	.09	.64	91
6	3.31	.16	1.57	.12	220	.08	.89	125
7	3.23	.07	1.50	.08	234	.15	1.18	139
8	3.43	.09	2.13	.12	259	.08	.47	65
9	3.59	.04	1.15	.13	291	.08	.50	67
10	3.26	.09	1.11	.09	293	.06	.60	79

SUB-AREA 9.

ii). Nardus Pasture.

	Per cent				p.p.m.			
Sample	s.f.a.	Ca	K	Mg	Mn	Co	Ni	Fe
D1	2.58	.07	.74	.09	245	.17	3.15	339
2	2.70	.07	.78	.13	227	.12	.69	102
3	2.47	.06	.76	.10	215	.16	1.26	150
4	2.59	.07	.82	.11	205	.13	1.02	136
5	2.87	.06	.80	.11	260	.08	.96	225
6	2.36	.09	.70	.11	147	.11	1.43	193
7	2.96	.09	.72	.18	272	.12	1.46	166
8	3.03	.06	.81	.14	230	.10	1.35	162
9	2.60	.07	.58	.13	169	.13	1.49	201
10	3.01	.06	.72	.08	192	.09	1.27	181