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PAPER 67-23
Part I

GEOLOGICAL SURVEY OF CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

PROGRESS REPORT ON BIOGEOCHEMICAL RESEARCH AT THE GEOLOGICAL SURVEY OF CANADA 1963-1966

J.A.C. Fortescue and E.H.W. Hornbrook

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Frontispiece: Surficial material sampling operations at Timmins during March 1965. Note the winter road across the frozen muskeg in the foreground, the water truck to the left of the drill rig and the office trailer to the right (see section C).

FOREWORD

From time to time the Geological Survey of Canada initiates research in aspects of Earth Science which concern geology and biology. For example, some years ago Dr. Terasmae began a research program in the field of pollen geobotany and related studies. This report describes research in a similar field - biogeochemistry.

The establishment of landscape biogeochemistry, and particularly biogeochemical prospecting as a legitimate field of research in Canada has been due largely to the pioneer studies of Dr. H.V. Warren and his numerous co-workers at the University of British Columbia. The research described in this report follows logically from the lead given by these scientists.

A first progress report describing activities in a specialized field of research can be written either assuming that all readers are aware of the background and latest developments in the speciality or that the reader is not fully aware of the scientific foundations on which it is based. I have chosen the latter course, and in Section A have provided five short summaries of basic concepts which, taken together, form a theoretical background to the other nine sections in the three parts of the report. Some background information may be unfamiliar to a geologist (e.g., data on plant nutrition) and some unfamiliar to a biologist (e.g., data on mineral exploration). The reader not interested in the background information may turn directly to the list of objectives of the research program which concludes Section A.

A broad research program of this kind does not stand alone. In almost every phase of the program we have obtained advice or material assistance from individuals and organizations both within the government and in private industry. Consequently, it is particularly difficult to acknowledge in detail all who have in one way or another facilitated the research. For these reasons a list of acknowledgments is not included but instead specific reference is made to organizations and individuals in the text.

There are a small number of scientists who have provided encouragement during the past four years in every phase of our activities. These include Dr. H.V. Warren at the University of British Columbia, Mrs. H.L. Cannon of the United States Geological Survey, Dr. J.S. Rowe and Dr. P.J. Rennie of the Department of Forestry, Mr. W.K.W. Baldwin of the National Museum of Canada, and Mr. G. Pierepoint of the Ontario Department of Lands and Forests Research Station at Maple, Ontario. We would like also to acknowledge our debt to the members of the professional and technical staff of the Geological Survey of Canada who have from time to time provided help and constructive criticism.

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PROGRESS REPORT ON BIOGEOCHEMICAL RESEARCH AT THE GEOLOGICAL SURVEY OF CANADA 1963-1966

PART I

SECTION A BACKGROUND, SCOPE, AND OBJECTIVES

by J. A. C. Fortescue

A 1 Introduction

Although a geological survey provides a unique opportunity for setting up a research program in biogeochemical and geobotanical prospecting methods, research of this kind is complicated by three factors. First, there are usually numerous and varied mineral deposits within an area. Second, a large number of different kinds of landscapes, and associated vegetation cover types, are likely to exist within the area within any one of which a mineral deposit of any type may occur. Third, in order to relate the chemistry of the bedrock to that of the plants it may be necessary to study the chemistry of the surficial material and soil, which are relatively inaccessible components of a landscape. This factor is of extreme importance in Canada where most of the country is covered with transported surficial material and soils.

This report describes progress made by the biogeochemical research group of the Geological Survey of Canada towards the setting up of methods designed to study systematically the scope and limitations of plant prospecting for ores in Canada. The research described here was carried out as a part of a five year research program begun in January 1963 and aimed at achieving the five general objectives listed at the end of this section.

A 2 Layout of the report

The general layout of this report follows the flow sheet, Figure A 1. Part I is concerned largely with a description of background information, laboratory facilities, and field, greenhouse, and laboratory methods that have been evolved during the initial stages of the program. Part II presents the results of field investigations carried out near known mineral deposits, and Part III, the results of investigations carried out in specially selected control areas away from such deposits.

It should be stressed that although systematic methods of landscape study of the type described in this report are required to determine the scope

Manuscript received: January 3, 1967.

Section A BACKGROUND, SCOPE, AND OBJECTIVES

Background information summaries

General Geochemistry, Plant Nutrition

Landscape description, Mineral Exploration

Biogeochemical and Geobotanical Prospecting

Statement of objectives of research program 1963-66

1) Provision of laboratories and methods
2) Investigations near mineral deposits
3) Investigations in control areas
4) Greenhouse investigations
5) Systematic investigations near mineral deposits (none completed at the end of 1966)

Section B DESCRIPTION OF THE MOVABLE SPECTROGRAPH LABORATORY UNIT

Section C DESCRIPTION OF FIELD, LABORATORY, AND DATA PLOTTING METHODS

Section D DESCRIPTION OF FOUR FEASIBILITY GREENHOUSE EXPERIMENTS

Part II

Section E DESCRIPTION OF ELEVEN "VISITS"* TO UNDISTURBED MINERAL DEPOSITS

Section F DESCRIPTION OF TWO "QUICK PROJECTS" AT UNDISTURBED MINERAL DEPOSITS

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Section J A BIOGEOCHEMICAL INVESTIGATION IN PREPARATION FOR FIELD MINOR ELEMENT UPTAKE EXPERIMENTS

* The terms "Visit", "Quick Project" and "Pilot Project" are defined in Section C.

Figure A 1. Flow sheet showing the organization of the progress report on biogeochemical research 1963-66.

and limitations of plant prospecting methods, the eventual application of the prospecting methods (based on research of the type described here) should be relatively rapid, and less expensive in comparison to the cost of other methods of mineral exploration.

A 3 Background information

The background information on which the foundations of the research program were laid is conveniently summarized under the following five headings: General Geochemistry, Plant Nutrition, Landscape Description, Mineral Exploration, and Biogeochemical and Geobotanical Prospecting. A reader interested only in the statement of the objectives of the research program should turn directly to A 4.

(1) General geochemistry Geochemistry may be broadly defined as the study of the synthesis and decomposition of natural materials. The pioneer Norwegian geochemist V. M. Goldschmidt (1954) described the two major tasks of geochemistry as the study of the distribution and amount of chemical elements in nature and the study of the circulation of elements by natural processes and pointed out that the findings of geochemistry are frequently of practical as well as theoretical importance.

From the theoretical point of view geochemistry is divided into four major parts each of which involves the study of the chemistry of a different geosphere. The pioneer Russian geochemist V.I. Vernadski classified these as: lithogeochemistry (the study of the chemistry of rocks and minerals); hydrogeochemistry (the study of the chemistry of natural waters); atmogeochemistry (the study of the chemistry of the atmosphere), and biogeochemistry (the study of the chemistry and biochemistry of living and dead organic matter). Biogeochemical prospecting for ores is but a single practical application of a vast field of scientific knowledge.

The study of the scope of biogeochemical prospecting for ores involves relationships between the chemistry of plants and that of the landscape in which they occur. In order to relate data on the geochemistry of landscapes to the body of general geochemical data, it is convenient to consider the concept of the Geochemical Cycle described by Mason (1958), and illustrated in Figure A 2.

The geochemical cycle is a theoretical model set up to summarize the behaviour of particular chemical elements in nature. The cycle has two parts; a major cycle, which occurs in the Upper Lithosphere, and a minor cycle which involves the liberation of the chemical components of crystalline rocks, their transport from one part of the earth's crust to another, and finally, their incorporation into secondary rocks, which, after deep burial,

enter the major geochemical cycle. Whereas the major geochemical cycle occurs below the surface of the earth and is essentially geological, the minor geochemical cycle occurs at the surface of the earth, and at one time or another may involve almost all branches of the physical and biological sciences.

Polynov (1937), in a discussion of the fate of chemical elements during the "cycle of weathering" (minor geochemical cycle), pointed out that it is incorrect to express the cyclic processes followed by elements in the earth's crust as circles because the cycles have a progressive element at the same time. He suggested that the fate of an element involved in the synthesis of a secondary system, for example, a plant is more correctly represented by a curve traced by a point on the circumference of a circle rolling in a straight line. Such curves are called cycloids and are simple for annual plants and complex for perennials (Figs. A 3a, b).

Figure A 3a represents the element iron in three generations of an annual plant. (The minimum between the generations represents symbolically the iron in the seed during the winter months.) In Figure A 3b two chemical elements are involved, iron in the perennial organs and manganese in the annual organs of a hypothetical three year life cycle plant through three generations. It is convenient to call the cycles for specific elements subcycles of the minor geochemical cycle. The degree of development of the perennial subcycle (involving iron) and the annual subcycle (involving manganese) at the time of felling of a 7 1/2 year old deciduous tree is shown in Figure A 3c. The previous six completed annual subcycles for manganese are shown dotted because they represent the manganese removed by dead leaves at the end of the season. In this case the perennial subcycle has a duration (i.e. the life expectancy of the whole tree) of 100 years and the second (annual) subcycle has a duration of one year. At the time of sampling (vertical line on Fig. A 3c) both are interrupted and both partly completed. This example demonstrates the importance of the time factor in biogeochemical prospecting research, especially if results obtained from individuals of the same tree species obtained from different areas are to be compared with one another.

The time factor may be extremely important in either biogeochemical or geobotanical prospecting research. In order to obtain comparable samples of the same organ from individuals of the same species within a landscape (so that after chemical analysis the results can be compared in detail), it is essential that all the samples are collected at the same "instant in time". In the case of geobotanical methods (involving the observation of the presence or absence of indicator plants, or variations in the morphology of common plants) this principle is also important because the plants must be observed at the time in the growth cycle when the desired feature is present. For example, if the modification involves the flower colour this cannot be observed until there are open flowers on the plant. Hence, although the time of collection of data may be of vital importance during the study of the

behaviour of chemical elements during the minor geochemical cycle, it may be of little significance in studies involving the behaviour of the same elements in stable rocks during the major geochemical cycle.

The model of the geochemical cycle cannot be pressed too far. Atoms or ions of all elements do not follow all the different stages of the major or minor geochemical cycles. As Mason (1958) points out, the complete cycle for a given element may not be realized in practice; at some stage it may be indefinitely halted, or short circuited, or even have its direction reversed. But in spite of these limitations, the general concept of the geochemical cycle is valuable as a connecting thread for different kinds of chemical data obtained at or near the surface of the earth.

For example, suppose we consider the results of a number of similar, systematic studies of the migration of copper from weathering of mineral deposits up through surficial material into soil and vegetation. Taken together the results of these studies may be considered as a contribution to our knowledge of the behaviour of copper during the minor geochemical cycle under Canadian landscape weathering conditions. Generalizations drawn from systematic data of this kind may very well be used as a starting point for specialized geochemical research that is not connected directly with the development of prospecting methods; for example, in the fields of animal nutrition and human health, or in the study of pollution.

General information on geochemistry may be obtained from books by Mason (1958), Rankama and Sahama (1950), and Goldschmidt (1954). The geochemistry of the soil has been reviewed by Mitchell (1964); and with respect to specific elements, by Swaine (1955) and Vinogradov (1959). General information on geochemical prospecting can be obtained from Ginzberg (1960) and Hawkes and Webb (1962).

(2) Plant nutrition Biogeochemical prospecting research involves direct relationships between the chemical composition of plants and that of the soil in which they grow. With very few exceptions (which are not considered in this report) all plants are composed of ten macronutrient elements (C, H, O, N, P, S, K, Ca, Mg, Fe) and five micronutrient elements (B, Mn, Zn, Cu, Mo) all of which are essential for healthy plant growth. The macronutrient elements are present in relatively large quantities in the plant. They are mainly constituents of proteins, cell walls, or mechanical structures. They also play an important part in complex chemical reactions, but the bulk of each of these elements is present in protoplasm or in structural components. The role of the micronutrient elements in plants is primarily catalytic and they generally do not have structural functions. In addition to the essential elements there are other major elements (Na, Si, Al) and many minor elements (Ba, Sr, Ti, Ni, Pb, Ag, Co, Cr, V, As, Li, Cl, Rb, Se etc.) which may be found in significant amounts in plants although they appear not to be essential

for the healthy growth of the plant. The functions of many of the essential elements overlap, which often complicates the study of the role of a single element in plant nutrition. For example, Schütte (1964) shows that a boron deficiency is induced by adding over 2 per cent of potassium to soil of healthy soyabean plants to which no boron was added. But if a little boron was added to the plants, even 20 per cent of potassium in the soil did not induce the boron deficiency symptoms. Therefore it is desirable that systematic biogeochemical prospecting research should include not only the elements present in the mineral deposit (over which the samples were collected) but also other elements which provide general information on the health of the plants. More details of problems in plant nutrition may be obtained from textbooks by Steward (1963) or Schütte (1964).

It is evident that most chemical elements which are of interest in biogeochemical prospecting for mineral deposits occur as essential, or more commonly as non-essential minor elements in plants. Thus plant prospecting methods research is generally focussed upon minor elements and the effect that abnormally high amounts of a specific element (or elements) have on plant communities. This is in marked contrast to most studies of plant nutrition that are carried out in order to determine the effect of deficiencies of minor elements in plants (see Hewitt,1966).

The effect which abnormal amounts of a given element may have on a plant community are summarized in Figure A 4. It should be stressed that although there are three possible ways in which geobotanical prospecting can be carried out, these methods are at present less important in Canada than biogeochemical methods. This may be due to the relatively short time Canadian landscapes, with their characteristic plant communities, have had to develop since the last glacial retreat. In countries where no glaciation occurred, specific indicator plants or plant species with characteristic morphological changes due to the presence of mineral deposits, have been evolved (see Malyuga and Petrunina, 1961).

In the discussion of the plant subcycles of the minor geochemical cycle the effect of seasonal variation on the chemical composition of a plant, or plant organ was mentioned. Guha (1961) produced data showing the seasonal variation of a number of elements in leaves of deciduous trees located away from mineral deposits in Scotland (Fig. A 5). These data are of particular interest because they emphasize the fact that the amounts of some minor elements (Fe, Mn, B) tend to increase in concentration during the growing season whereas the amounts of other elements (Zn, Cu, Mo) decreases. It is interesting, especially from the point of view of the subcycles of the minor geochemical cycle discussed above, that the magnitude of the seasonal variation (expressed on an oven dry weight basis) may be as great as sevenfold (e.g., boron in sycamore leaves). These considerations pose two problems for biogeochemical prospecting research: (1) the need for simultaneous collection of samples, and (2) the degree of completion of

the current growth, and perennial subcycles of the minor goechemical cycle at the time samples are collected. It is clear that if biogeochemical data obtained from plant communities of the same type in different areas are to be compared, very careful attention must be given to problems of the "instant in time" in which the samples were collected.

It is common practice in biogeochemical prospecting, and in some studies of plant nutrition, to express analytical results on the basis of dry ash rather than on an oven dry weight basis. Dry ashing (for wet ashing, see Jackson, 1958) is a method of concentrating minor elements present in plant material, and at the same time destroying the organic compounds within a sample. At present, biogeochemical prospecting is usually based on the results of chemical analysis of samples of the same organ of a large number of individual plants of the same species collected from the area being surveyed. The difference between the chemical complexity of oven dry material and ash is evident from Table A 1. The determination of an element in the ash of a needle sample is a relatively simple task compared to the determination of the same element in an oven dry sample without ashing. An exception occurs in the case of samples which contain a significant amount of a radioactive tracer, for example, Co^{60}.

Table A 1.

The levels of organization within a plant.

	Level of organization	Example
	9) Plant individual	tree Pinus banksiana
	8) Organ	needle
A	7) Tissue	epidermis
	6) Cell	cuticle cell
	5) Cell microstructure	nucleus
	4) Macro molecule	DNA
	3) Micromolecule	H_2O
B	2) Element	Co
	1) Isotope	Co^{60}

Note Dry ashing reduces samples of the organ (A) to ash consisting of relatively simple inorganic chemical compounds (B).

In such a case the Co^{60} can be measured by counting the radiation given off by the sample (i.e., without ashing). Although current biogeochemical prospecting techniques are based on the analysis of ashed samples, one can expect that in the future separation of specific biochemicals from samples in the field (for example a chelate combined with a specific heavy element) will play an increasingly important part in prospecting.

The role that minor elements play in plant nutrition may be studied in books by Stiles (1961), Steward (1963), Schütte (1964), and Wallace (1961).

Details of biochemistry involving minor elements which could not be included in a review of this kind, may be obtained through general references; for example, Mallette, Althousa and Clagett (1960), White (1964), Stace (1964) and Haggis et al. (1964).

A convenient way to investigate the effect of abnormal amounts of particular minor elements in soil in which plants are growing is by greenhouse experiments. Experiments of this kind have been used extensively for many years by plant physiologists who studied in detail the mineral nutrition of plants (see Hewitt, 1966). It seems clear that greenhouses will become very important in biogeochemical and geobotanical prospecting research and for this reason greenhouse facilities have been provided for biogeochemical research on the roof of the Geological Survey of Canada Building in Ottawa.

(3) Landscape description Perhaps the most important stage in a biogeochemical prospecting research project is the interpretation of observations made on plants in terms of the chemistry and extent of a sub-outcropping mineral deposit in the landscape from which they were collected. In order to make an interpretation of this kind the different kinds of plant cover, soils, and surficial deposits in the area studied must be described systematically. The problem of landscape description becomes even more important if the results collected in one area are to be compared with those collected in other similar areas. For these reasons systematic studies of landscape description are of particular interest in biogeochemical prospecting methods research.

From the point of view of ecology, the environment in which a plant grows is called the "ecosystem". This term was proposed by Tansley (1935) to include the biome, which is the whole complex of living organisms found together in a single socialogical unit, as well as the habitat within which the biome lives. As Tansley (1946) pointed out, all parts of a given ecosystem interact resulting in a dynamic equilibrium in a mature ecosystem, and it is through these interactions that the whole system is maintained. The aim of biogeochemical prospecting research is to pinpoint within each ecosystem studied that part which is most suitable for practical prospecting.

Ecosystems are usually studied on an area basis (Ovington, 1962; Grieg-Smith, 1964), but in the case of plant prospecting methods research it is also essential to include the third dimension depth. Hence at any point within a landscape, which is studied, we are interested in the description of the morphology and chemistry of a volume of landscape extending from unweathered bedrock to the atmosphere. Such a unit volume of landscape may be referred to as a "prism of landscape" or, more conveniently in our case as a "prospecting prism". An expanded view of an idealized prospecting prism centred on a tree is shown in Figure A 6.

In this report it is not possible to describe, even in general terms, the movement of chemical elements from bedrock into surficial material, and from the surficial material into the soil. Information on the movement of water through surficial materials and soils may be obtained from the handbook by Ven Te Chow (1964), and on the formation and chemistry of soils from Jenny (1940) and Bear (1964). Information regarding the relationships between soils and plants may be obtained from Russell (1961) and Black (1957), and in general ecology textbooks (Daubenmire, 1959; Dansereau, 1957). For forest soils, Wilde (1958) and Romberger and Mikola (1964) should be consulted.

Theoretical descriptions of landscapes are, unfortunately, generally broader in scope than the more sharply focussed concept of the prospecting prism. Consequently, the relationship between a prospecting prism and landscape description is similar to the relationship between a geological hand specimen and an extensive outcrop area.

The Russian geochemist B. B. Polynov (1937) was one of the first to describe landscapes from the geochemical point of view in his classic book "The Cycle of Weathering". Glazovskaya (1963) developed these earlier ideas and summarized the current Russian approach to the systematic study of the geochemistry of landscapes. Briefly, he considered that from the geochemical point of view landscapes are three-dimensional natural bodies composed of genetically interrelated, or independent horizons that can be examined and discussed systematically as types, genera, species, and series in reference to their origin, evolution, interpretation and particularly, to their importance in geochemical prospecting for mineral deposits. He pointed out that the vertical geochemical profile is the most objective comprehensive index and expression of past and current chemical interactions between the different components of a landscape. However, the prospecting prism includes only a very small volume, whereas the classification of Glazovskaya begins with the broad concept of the Elementary Landscape Type which is later subdivided into smaller units. According to Glazovskaya, Elementary Landscape Types have the following characteristics. [1]

"a) They must contain the same bulk quantities of natural materials in their identically structured tiers of living substance (the total reserve, the ratio between the subaerial and subsurface parts and the fallout).

b) Vertical profiles of landscape of the same type must show the same ratio between the depth of their soils (determined by the depth of root penetration), the depth of the penetration of atmospheric waters, and the depth to the groundwater table.

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[1] The wording of this translation has been slightly altered for the sake of clarity. Any change in the sense is the responsibility of the present writer.

c) Landscapes belonging to the same type must be endowed with the same migrational velocities of chemical elements, tier to tier within the vertical profile, which depends in turn, upon the rate of combined physical, chemical and biological weathering processes."

The final conclusion of this interesting paper is worth quoting in full:

"Methodologically correct investigations of the material composition and of the forms in which the elements are found in every tier of the vertical geochemical profile of elementary landscapes is the problem for future research; this will serve to refine the classification. Examination and cross comparisons of the geochemical characteristics of the vertical profiles in a series of landscapes, from eluvial to superaqual and aqual will afford the basis for a rational geochemical classification of the various combinations of geochemically interrelated elementary entities."

The theoretical scheme of Glazovskaya would only be of academic interest if it had not been applied successfully to the solution of practical problems in the development of geochemical prospecting methods. For example, Perelman (1961) described in detail the relationships between the geochemical description of landscapes and the setting up of geochemical prospecting methods in the southern Urals of the U.S.S.R. He stressed the importance of the landscape description map as a guide to the choice of geochemical prospecting methods to be applied in a given part of the area, and pointed out that biogeochemical methods were most effective only under specific types of landscape conditions.

There is a pressing need for the development of a system of landscape description for use in organizing the vast amount of empirical geological, geophysical, and geochemical prospecting data which has so far been collected and published in Canada. Such data might be cross-referenced according to landscape type, exploration method (see Fig. A 6), and most important, to kind of mineral deposit involved.

Although there is at present no systematic system of landscape description set up specifically for use in mineral exploration in Canada, there are a number of systems which have been set up for other purposes. For example, Hills (1959) and his co-workers at the Ontario Department of Lands and Forests have set up a comprehensive and detailed system for the description of landscapes in Ontario, which has proven valuable in practice (see Hills et al., 1960). Similarly, on the basis of geology, climate, and particularly plant ecology, Krajina (1965) has described landscape types in British Columbia for seven biogeoclimatic regions. A third system of landscape description has been set up by Radforth (see summary by MacFarlane, 1961) for the delineation of landscape types in organic terrain. This classification was developed originally to aid in the solution of engineering problems in northern Canada.

(4) Mineral exploration Modern mineral exploration involves the application of one or more of the specialized geological, geochemical or geophysical exploration methods listed in Figure A 6 for locating ore within a given landscape. The particular methods selected for a given exploration program are chosen largely on the basis of the type of deposit being looked for and the kind of landscape conditions where the program will be carried out. The methods are applied by routine techniques each of which has been developed for surveying at a given level of intensity.

The general principles of a modern mineral exploration program are best illustrated by means of a simple hypothetical example. Suppose a greenstone belt is chosen for exploration. It is first delineated on a map of scale 1:250,000 and then surveyed by REGIONAL LEVEL[1] methods. These may include geological mapping, aeromagnetics, and stream sediments. The results from each survey are plotted on overlays at a scale of 1:50,000. After careful interpretation of all results a number of "areas of interest" are outlined on these maps. These areas of interest are then investigated more intensely by FOLLOWUP LEVEL[1] methods, such as detailed geological mapping, ground magnetics, and soil geochemistry. The results of these surveys are plotted on overlays at a scale of 1:10,000. Careful interpretation of these maps results in the delineation of a number of "anomalies" which in turn are investigated by DETAIL LEVEL[1] methods (for example, diamond drilling) and the results are eventually interpreted in relation to the presence or absence of ore. This process of successive approximations within a typical mineral exploration program is summarized in flow sheet form on Figure A 7.

It is with this background of the serial application of one or more methods of exploration at one or more levels of detail that the scope and eventual application of plant prospecting methods in Canada must be considered. Before this is done the three stages of evolution through which each mineral exploration method passes must be considered. Any mineral exploration technique passes, quickly or slowly, through these stages which may have to be repeated when a technique is applied under a new set of landscape conditions. During the experimental stage of the development of a new technique, it is tried out and shown to be a feasible principle on which prospecting methods can be based. The numerous investigations of biogeochemical prospecting described in Warren's early papers (see Table A 2) are examples of Canadian biogeochemical prospecting methods in the experimental stage of development. After being shown feasible both the scope and limitations of a prospecting method are then systematically evaluated during the development stage of the evolution of the method. The research described in this report is largely concerned with the development stage of biogeochemical prospecting in Canada. The third stage of evolution of an exploration method, the

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[1] The use of capital letters is explained in a previous paper (Fortescue, 1965).

Table A 2

Summary of published biogeochemical prospecting research in Canada 1947-1966.

Reference	Province	Landscape Materials Sampled						Elements												
		Rocks	Surficial Material	Soils	Vegetation			Cu	Zn	Pb	Ni	Mo	Fe	Mn	Ag	Au	As	Hg	Co	
					Ground	Under	Over													
Warren et al. (1947) (1) *	British Columbia					X	X	X	X											
Warren et al. (1948) (2)	British Columbia				X	X	X	X	X											
Warren et al. (1949) (3)	British Columbia					X	X	X	X											
Warren et al. (1949) (4)	British Columbia					X	X	X	X											
Starr (1949) (5)	British Columbia					X			X											a)
Warren et al. (1950) (6)	British Columbia																			b)
Warren et al. (1950) (7)	British Columbia				X	X	X								X	X				
White (1950) (8)	British Columbia						X	X	X											
Warren et al. (1951) (9)	British Columbia						X	X	X											
Warren et al. (1952) (10)	British Columbia					X	X	X	X											c)
Warren et al. (1952) (11)	British Columbia					X	X		X				X	X						
Riddell (1952) (12)	Quebec						X	X	X											d)
Warren et al. (1953) (13)	British Columbia					X	X					X								
Warren et al. (1954) (14)	British Columbia, Ontario						X				X									
Warren et al. (1955) (15-16)	Eastern Canada					X	X	X	X											
Warren et al. (1955) (17)	British Columbia					X	X	X	X					X						

Warren et al. (1955) (18)	Northwest Territories Yukon				X	X	X	X	X											
Warren et al. (1957) (19)	British Columbia			X		X	X	X	X											
Warren et al. (1957) (20)	Ontario				X	X	X												X	
Warren et al. (1960) (21)	British Columbia					X	X			X										
Warren (1962) (22)	British Columbia					X	X	X	X	X		X								
Warren et al. (1964) (23)	British Columbia			X			X										X			
Warren (1965) (24)	British Columbia				X	X	X	X				X								
Boyle (1965) (25)	Yukon	X	X	X	X	X	X	X	X	X										e)
Warren et al. (1966) (26)	British Columbia			X	X	X	X	X	X	X	X	X		X						
Warren et al. (1966) (27)	British Columbia			X	X	X	X	X	X	X	X	X		X						
Warren et al. (1966) (28)	British Columbia			X	X	X	X	X	X	X							X	X		

a) Brief note only
b) General summary, no results
c) Many analyses, samples of many species
d) Some water analyses
e) Brief note only

* Numbers refer to references at the end of the section.

established stage, is reached when a technique has been developed to the point where it can produce reliable results relatively rapidly and cheaply. Biogeochemical prospecting methods will be in the established stage in Canada when the best organic material and the most useful chemical element for prospecting for each type of mineral deposit in each type of landscape is known. Clearly much development-stage research will be needed before the true scope and limitations of biogeochemical and geobotanical prospecting methods in Canada is known.

Further information on modern mineral exploration may be obtained from the book of case history studies by Kelly and Westrick (1957) and from Neale (1965). Detailed information on the geology of mineral deposits appears in Bateman (1950). For geophysical prospecting methods, Grant and West (1965) and Parasnis (1966) should be consulted; and for geochemical prospecting (including plant prospecting), Ginzberg (1960), Hawkes and Webb (1962), and Cameron (in press).

(5) Biogeochemical and geobotanical prospecting The theoretical basis for different kinds of plant prospecting methods was summarized in flow sheet form in Figure A 4. The object of this short summary is merely to outline previous research in plant prospecting for ores with particular reference to Canada.

Perhaps the most concise introduction to plant prospecting methods research is contained in an article (with eighty references) by Cannon (1960). Mrs. H. L. Cannon is a pioneer biogeochemist and geobotanist in the United States Geological Survey, and in her paper she discusses progress made under both headings. She lists thirty-two indicator plant species which have been described from different parts of the world and that had been used to locate deposits of bitumen, gypsum, or mineral deposits containing one or more of the following elements: boron, copper, iron, lead, phosphorus, selenium, uranium, silver, and zinc. Under a second heading, a list of morphological changes that have been observed in plants growing in soils with abnormally high amounts of aluminium, boron, chromium, cobalt, copper, iron, manganese, molybdenum, nickel, uranium, or zinc is given. Only one reference is made to geobotanical prospecting in Canada.

In the same paper Mrs. Cannon mentions the results of fifty-three biogeochemical prospecting investigations of which forty-seven were partly (or completely) successful in locating mineral deposits. These investigations involved the determination of one or more of the following elements: Cu, Zn, Mn, Au, Ag, Ni, Mo, Sn, V, As, Cr, Pb, Ba, B, and Co in samples of individuals of a number of plant species collected from ground, understory, or overstory vegetation (see Fig. A 6). Twelve of these investigations were carried out in Canada; most by Warren and his co-workers (see Table A 2).

Malyuga (1964) describes biogeochemical methods of prospecting based largely on Russian experience. Systematic study of this book is recommended in order to obtain detailed information on the scope and limits of plant prospecting research. He lists eighteen ore occurrences in different parts of the world that were found by plant prospecting methods. None of these is in Canada. More information on Russian progress in biogeochemical prospecting for ores may be obtained from articles in the translation of the Russian journal "Geochemistry"; for example, Moissnko (1959) and Mararova (1960). Other Russian scientists have used plant prospecting for water (Victorov, 1960), and, more generally, for mapping rocks and soils (see Chikishev, 1965).

Only one published report on Canadian geobotanical prospecting was found. Shacklette (1964) noted variations in the colour and morphology of flowers of common fireweed Epilobium angustifolium near uranium deposits at Port Radium in the Northwest Territories. It is believed that a number of other unpublished geobotanical observations have been made in Canada.

The feasibility of biogeochemical prospecting in Canada has been demonstrated in Canada for twelve elements by H. V. Warren and R. E. Delavault and their numerous co-workers at the University of British Columbia. Data included in twenty-three papers by this group, and four by other Canadian workers, are summarized in Table A 2. It is interesting to note the scope of these papers, especially, with respect to the components of the landscape studied in each case and the elements determined in each suite of samples. For details of the results obtained and the organs and species included in each investigation, the reader is referred to the original papers. When one considers that the papers listed in Table A 2 contain almost all the published data on minor elements in Canadian forest plants collected near mineral deposits, the need for research of the type to be described in this report is evident.

A 4 General objectives of the plant prospecting methods research program 1963-1966

The overall aim of the research program is to examine the role of plant prospecting methods in the search for ores in Canada. During the first five year period the specific objectives of the research program were as follows:

1) To develop an integrated system of methods for
   (a) field area selection,
   (b) sample site layout,
   (c) rock, surficial material, soil, and vegetation sampling,
   (d) sample preparation,

(e) chemical analysis,
(f) mechanical plotting, and
(g) interpretation of results which can be used to produce uniform sets of descriptive and chemical data from observations and samples collected at an instant in time in any Canadian landscape.

2) To concentrate the major part of the field program at landscapes where known undisturbed mineral deposits occur which are suitable for the demonstration of the scope and limitations of biogeochemical and, possibly, geobotanical methods of prospecting.

3) To undertake a limited number of special investigations in landscapes which are particularly suited for plant prospecting methods research although distant from mineral deposits.

4) To establish the feasibility of using a greenhouse for plant prospecting methods research.

5) Where possible, to compile case histories of results of biogeochemical, geobotanical, and other geochemical methods, as well as those from parallel geological and geophysical investigations so that the relative effectiveness of plant prospecting methods can be directly related to the results obtained by other methods.

Progress made so far towards each of these objectives is described in the remaining sections of this three part report (see Fig. A 1). At the end of each part describing field investigations, conclusions are drawn in relation to the five aspects of the theoretical background described above.

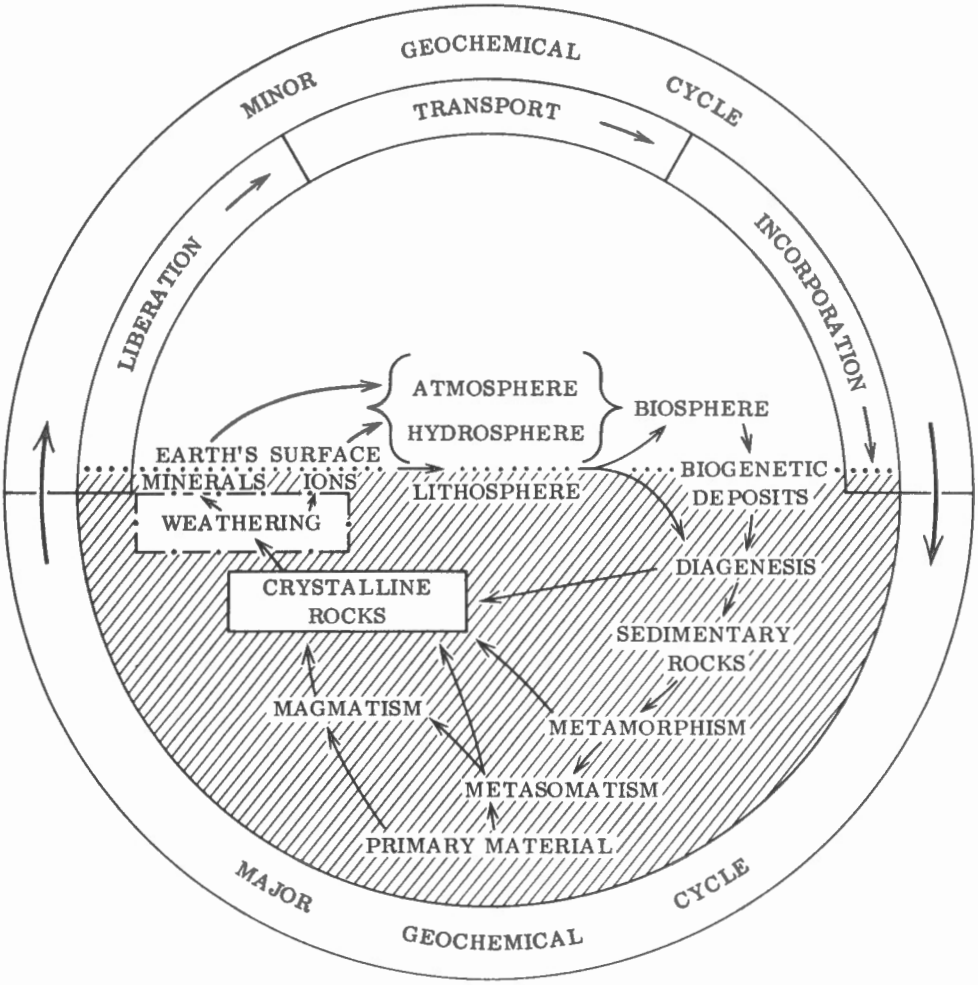
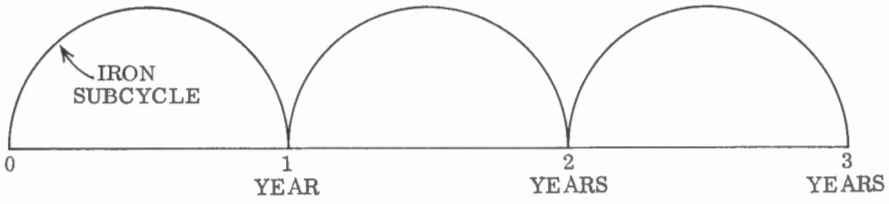
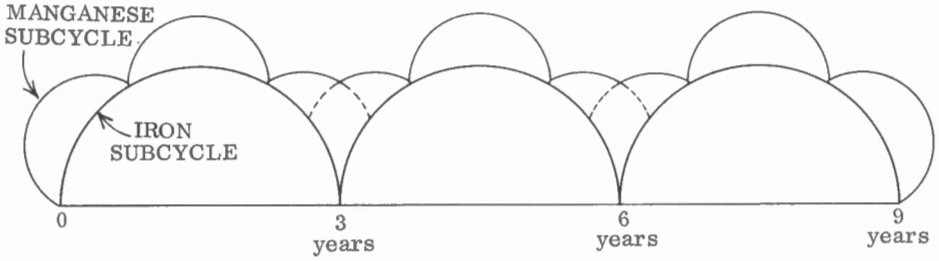


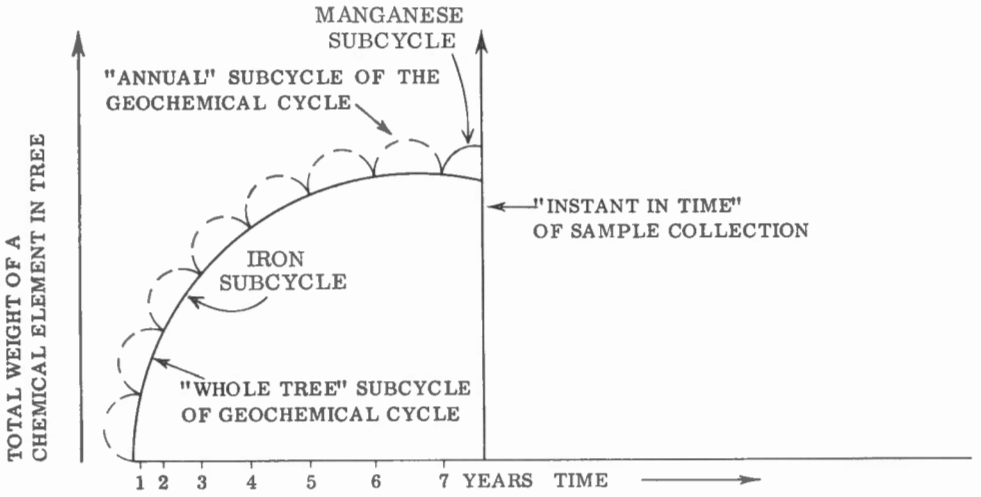
Figure A 2. The Geochemical Cycle (modified slightly after Mason, 1958).



a. CYCLOID



b. COMPLEX CYCLOID



c. THE APPLICATION OF THE COMPLEX CYCLOID MODEL
TO A SEVEN AND A HALF YEAR OLD DECIDUOUS TREE

Figure A 3. The Polynov concept of a complex cycloid in relation to subcycles of the minor geochemical cycle.

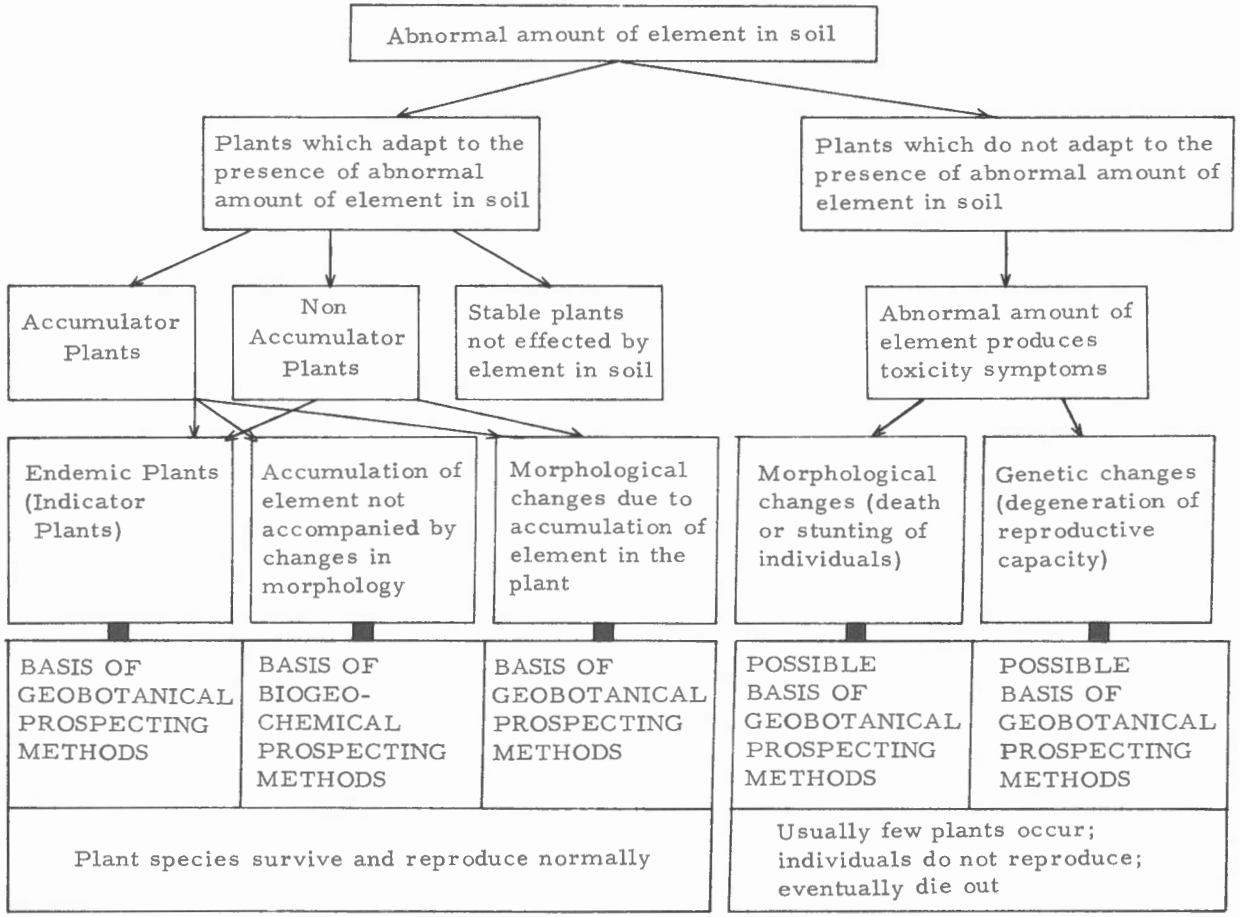
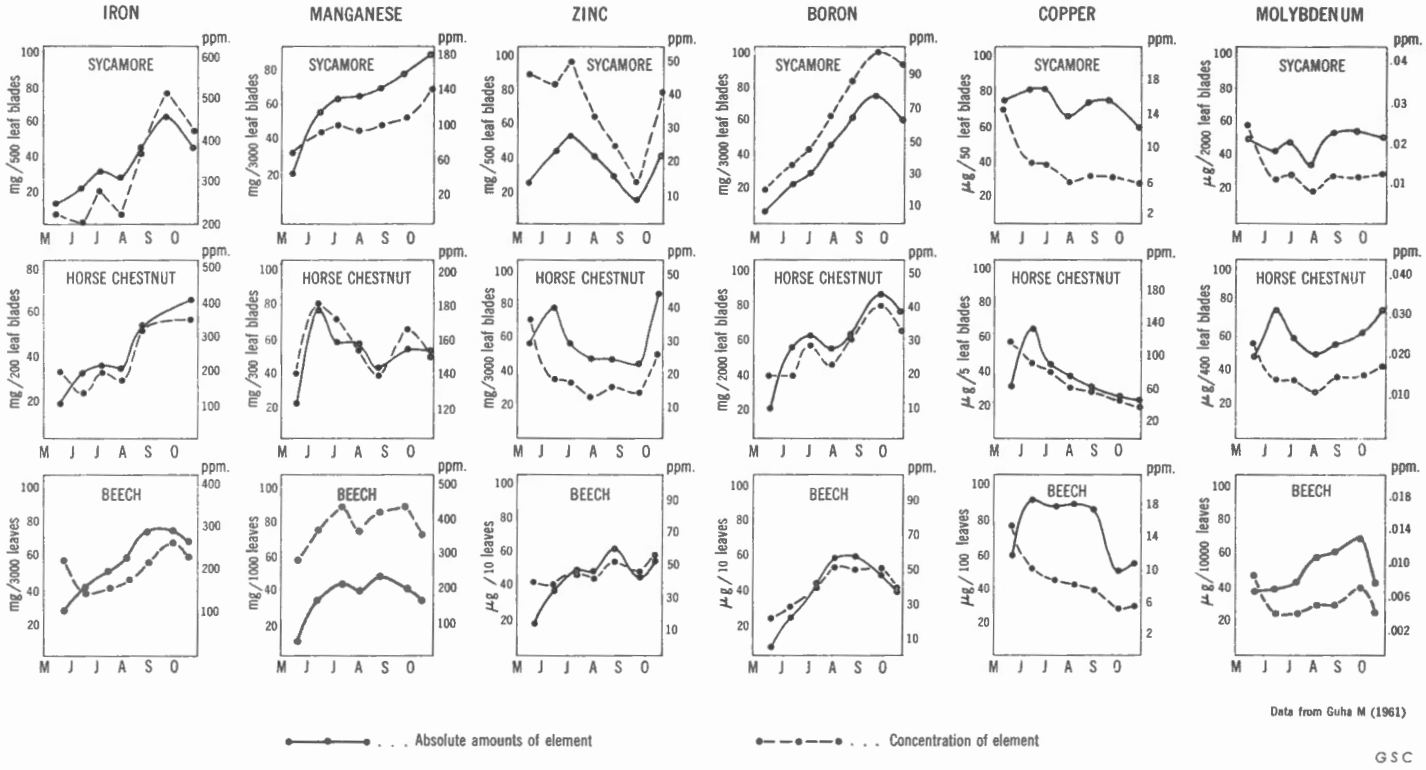


Figure A 4. Flow sheet showing the possible effect of abnormally high amounts of an element in soil on individuals of species of a plant community (after Korvalsky and Petrunina, 1965).



M J J A S O refer to the months of the year in which samples were collected

Figure A 5. Seasonal variation of the essential minor element content of leaves of three species of deciduous trees.

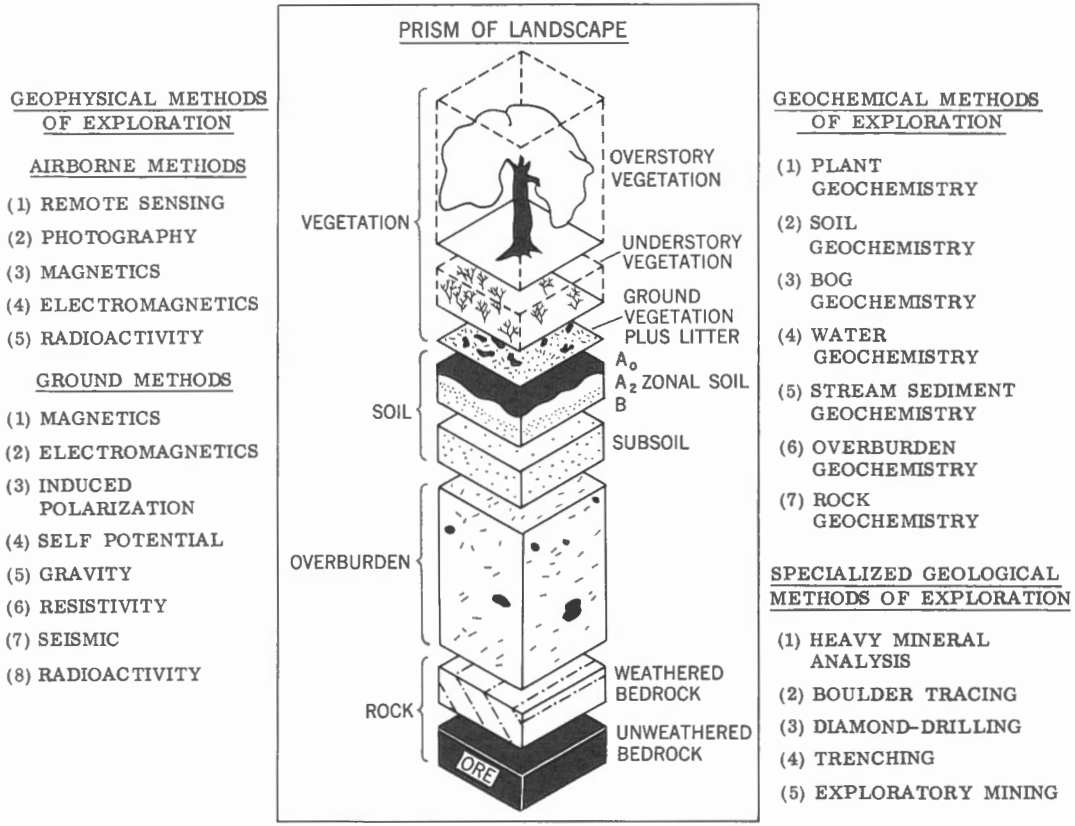


Figure A 6. An exploded isometric diagram of a generalized prospecting prism showing the different components of a landscape system that may be involved in prospecting methods research; and, the specialized geological, geophysical, and geochemical methods that are currently used for prospecting in Canada.

Stage I

Assessment of geological, geophysical, and geochemical data available for the region prior to exploration.

INTERPRETATION

Greenstone belt selected for prospecting.

Stage II

Application of selected geological, geophysical, and geochemical methods to the greenstone belt at the REGIONAL LEVEL of detail.

Overlay maps showing results on a scale of 1:50,000.

INTERPRETATION

Delineation of areas of interest.

Stage III

Application of selected geological, geophysical, and geochemical methods within the areas of interest at the FOLLOWUP LEVEL of detail.

Overlay maps showing results on a scale of 1:10,000.

INTERPRETATION

Delineation of anomalies.

Stage IV

Application of DETAIL LEVEL methods, including diamond drilling, to anomalies.

INTERPRETATION

Presence or absence of orebodies within the greenstone belt.

Figure A 7. Flow sheet showing the stages involved in an idealized exercise in mineral exploration.

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SECTION B DESCRIPTION OF THE MOVABLE SPECTROGRAPH LABORATORY UNIT

by J.A.C. Fortescue and E.H.W. Hornbrook

B 1 Introduction

In section A of this report a major objective of the biogeochemical research program within the Geological Survey of Canada during the period 1963-1966 was stated as follows:

To develop an integrated system of methods for
- a) field areas selection,
- b) sample site layout,
- c) rock surficial material, soil and vegetation sampling,
- d) sample preparation,
- e) chemical analysis,
- f) mechanical plotting, and
- g) interpretation of results which can be used to produce uniform sets of descriptive and chemical data from observations and samples collected at an instant in time in any Canadian landscape.

In this section the laboratory facilities that have been set up to carry out parts (d) and (e) of the above objective are described as an essential preliminary to a complete description of the whole methods scheme, involving all seven parts of the objective, which appears in Section C of this report.

B 2 Requirements for a laboratory for biogeochemical research

It is evident from the background information summarized in Section A that a laboratory which services a research program in biogeochemical prospecting must meet six exacting requirements. First, the laboratory must be capable of determining the chemical elements in various kinds of natural materials (i.e., rocks, surficial material, soil, bog material, and vegetation). Second, the scope of the analytical facilities (with respect to chemical elements determined) must be large enough to include both essential and, in many cases more important, non-essential minor elements, which occur in plants and mineral deposits. Third, because of the dynamic aspect of ecosystems the laboratory must be able to process samples rapidly (for example, at the rate of some 500 single element determinations per day) so that the results of the chemical analysis can be interpreted soon after collection. After interpretation, any further sampling of the same ecosystem can then take place at the same instant in time. Fourth, the precision of the combined sampling, subsampling, and chemical analysis methods system for

each material must be such that it will determine significant variations in the chemical composition of sets of similar samples that can be attributed to the presence of a mineral deposit in the landscape in which they were collected. Fifth, the subsampling and methods of chemical analysis must be simple and reliable enough to be taught to unskilled personnel who may be attached to the research program for the summer months only; and, sixth, because of the large numbers of chemical elements involved in the analytical program stringent precautions must be taken to prevent intercontamination of samples, and contamination of samples from outside sources during the analytical program.

Previous experience in biogeochemical prospecting in Canada (see Section A 3) has shown that at least twelve elements (Cu, Zn, Co, Ni, Ag, Au, Pb, Fe, Mn, As, Mo, Hg) are of particular interest, and in addition, experience obtained elsewhere indicates that the determination of other elements (K, Mg, P, B, Ba, Sr, Ti, etc.) is also desirable in research of this kind. It was clear at the beginning of the research program that results from the determination of more than one chemical element in all the different materials within a prospecting prism (see Fig. A 6) would result in a vast amount of digital information of the type which should be plotted mechanically. Therefore it was decided that account must be taken in the design of the laboratory for the inclusion of facilities for the preparation of results for input to a data processing centre, where neat accurate and uniform graphical and tabular arrays of results could be produced.

B 3 The evolution of the concept of the Movable Spectrograph Laboratory unit

At the time the laboratory facilities were in the design stage in 1963 and with the general considerations noted in Section B 2 in mind, four possible solutions to the problem of setting up laboratory facilities were considered:

1) A mobile field laboratory for sample processing and preparation and a permanent colorimetric laboratory in Ottawa.
2) A mobile field laboratory for sample processing and preparation and a permanent spectrograph laboratory in Ottawa.
3) A mobile field laboratory for sample processing and preparation and a mobile colorimetric laboratory.
4) A mobile field laboratory for sample processing and preparation and a mobile field spectrographic laboratory.

When the laboratory facilities were being planned, nearly all previous research in biogeochemical prospecting in Canada had relied upon colorimetric methods (see Section A 3 and Table A 2) that were generally used for the determination of a very small number of elements in each sample. For the purposes of the Geological Survey's program it was desirable to use an

optical spectrograph for the simultaneous determination of a relatively large number of minor elements in each sample. Fortunately, by early 1963, Dr. R.H.C. Holman had demonstrated the feasibility of mounting a routine spectrograph laboratory in a specially designed house trailer (see Holman and Durham, in press), and Mitchell (1948) had already shown that provided suitable methods were set up and applied systematically, an optical spectrograph could be used for routine determination of numerous elements in samples of plant and soil as well as rock materials.

At first it was hoped that the sample preparation and subsampling facilities and a spectrograph and associated equipment could all be housed within a single trailer. Preliminary design studies showed that if this were done the trailer would have to be over 40 feet long. This was considered to be too large for a single laboratory unit. Consequently, it was decided to house the laboratory in two 28-foot house trailers: one, for sample processing, and the other for the chemical laboratory and the spectrograph facilities. (see Figs. B 1, B 2, and Plates B 1-B 9).

Careful attention in the design of the laboratories to satisfy the six requirements described above resulted in the concept of a Movable Spectrograph Laboratory unit that could be used for full scale operation in the field in the summer and for full scale operation in a heated garage at headquarters during the winter. Because of the problems involved and the time required for packing and moving laboratories of this kind, it was planned to move the unit only twice a year, to and from the field. It was estimated that each move would take ten days, not including travelling time. In order to obviate the need for large towing vehicles to tow the trailers it was planned to move the trailers by rail flatcar, and to hire a tow truck to move them from the railhead to the summer location.

B 4 Description of the Movable Spectrograph Laboratory unit

General information on the design, layout, and construction of a mobile spectrograph laboratory very similar to that included in the Movable Laboratory unit is given in Holman and Durham (in press). The layout of the two trailers included in the Movable unit is shown in Figure B 2 which also shows details of the construction specifications. The latter may be of particular interest to readers who wish to design trailer laboratories to meet specific requirements. Details of the equipment installed in each trailer shell after delivery are given in Appendix BA for the sample preparation trailer, and in Appendix BB for the spectrograph trailer.

B 5 Operation of the Movable Spectrograph Laboratory unit

The different operations involved in the preparation of organic samples, ashing, and spectrochemical analysis are summarized in Figure B 1

and an indication of the room in which each operation is generally performed is also shown. At present, rock and mineral soils samples, are subsampled outside the trailers in order to avoid contamination. Later, when wet methods of analysis of these materials are developed, they will be processed inside the trailer. Details of all analytical methods developed so far for use in the trailers are given in Section C.

Both trailers have been tested at winter and summer locations. Experience has shown that air drying of samples can be completed in 72 hours or less. The collection of samples in the field is generally carried out in lots (one lot may include about 60 plant and 30 soil samples). Each lot is treated as a unit and dried in a single cabinet with all sample bags closed. In this way some 300 samples can be dried at the same time in the laboratory. In practice, however, one of the cabinets is usually used by the botanist for herbarium specimens. If the same three-man crew who collect the samples is responsible for subsampling and preparing samples, an average of 30 samples per day can be supplied to the spectrograph laboratory. In the spectrograph laboratory a second three-man crew, if fully trained, can ash, spectrograph and record results for thirteen elements in each of thirty samples per day on a routine basis.

The Sample Preparation Trailer was used in the field for the first time during the summer of 1965. It was taken on a rail flatcar to Fraserdale, northern Ontario and then towed a few miles by road and set up at Abitibi Canyon. During a seven week period, some 1500 samples of soils and plants were collected, dried, and subsampled by a four-man crew. In addition, the laboratory was used by the botanist for the preparation of some 500 mounted herbarium plant specimens. The routine operation of the Spectrograph Trailer Laboratory was tested out for the first time during the same summer in the winter location in Ottawa. In this case, during a 7 week period over 1000 samples of organic material were analyzed in the trailer. In the summer of 1966 both trailers were taken to the Petawawa Forest Experimental Station at Chalk River, Ontario. This experience showed that a fully trained three-man crew could pack, move, unpack, and ready for operation both laboratories within one week.

B 6 Summary and Conclusions

The exacting requirements for laboratory facilities needed to fulfil the objective of the biogeochemical research program listed at the beginning of this section were described under six headings. The philosophy behind the establishment of the Movable Spectrograph Laboratory unit was outlined, and brief descriptions of the layout and function of the two trailer laboratories were given. The experience gained so far with the headquarters and field operation of the Movable Spectrograph unit was also described.

We consider that the Movable Laboratory unit has well satisfied the requirements for analytical facilities for biogeochemical research. The unit has been shown to be fully mobile when required and well suited to both winter and summer routine operation. The complete processing of thirty samples of organic material per day for twelve elements plus a reference standard element is practical in the unit on a continuing basis. For these reasons we consider that the unit is adequate for the analysis of organic samples; however, in the case of mineral soil and rock samples, methods suitable for use in the trailer unit are still under development. Provision was made in the trailer unit design for the inclusion of an atomic absorption apparatus when it is acquired.

B 7 References

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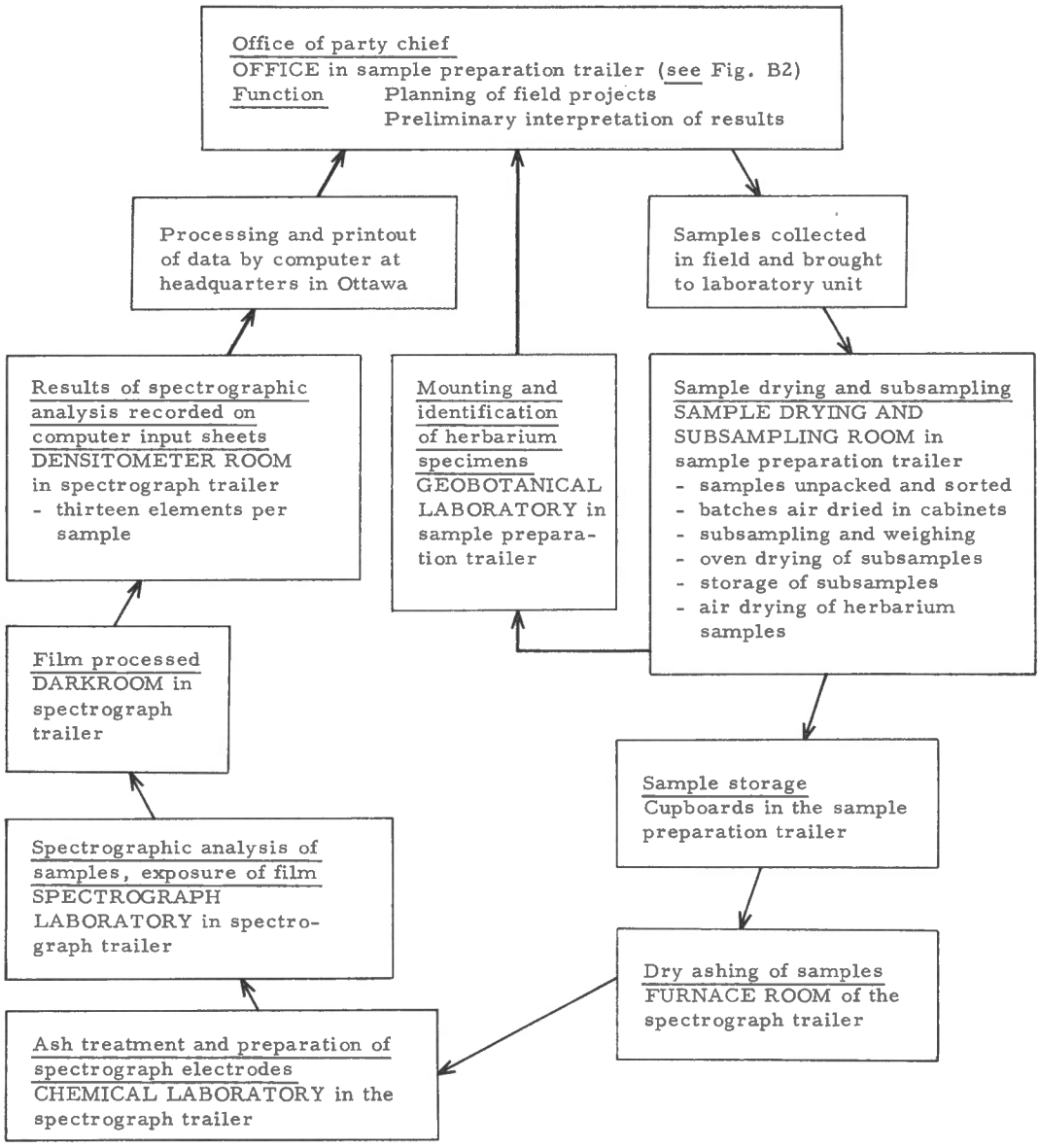

Figure B 1. Generalized flow sheet of operations carried out within the biogeochemical movable spectrograph laboratory.

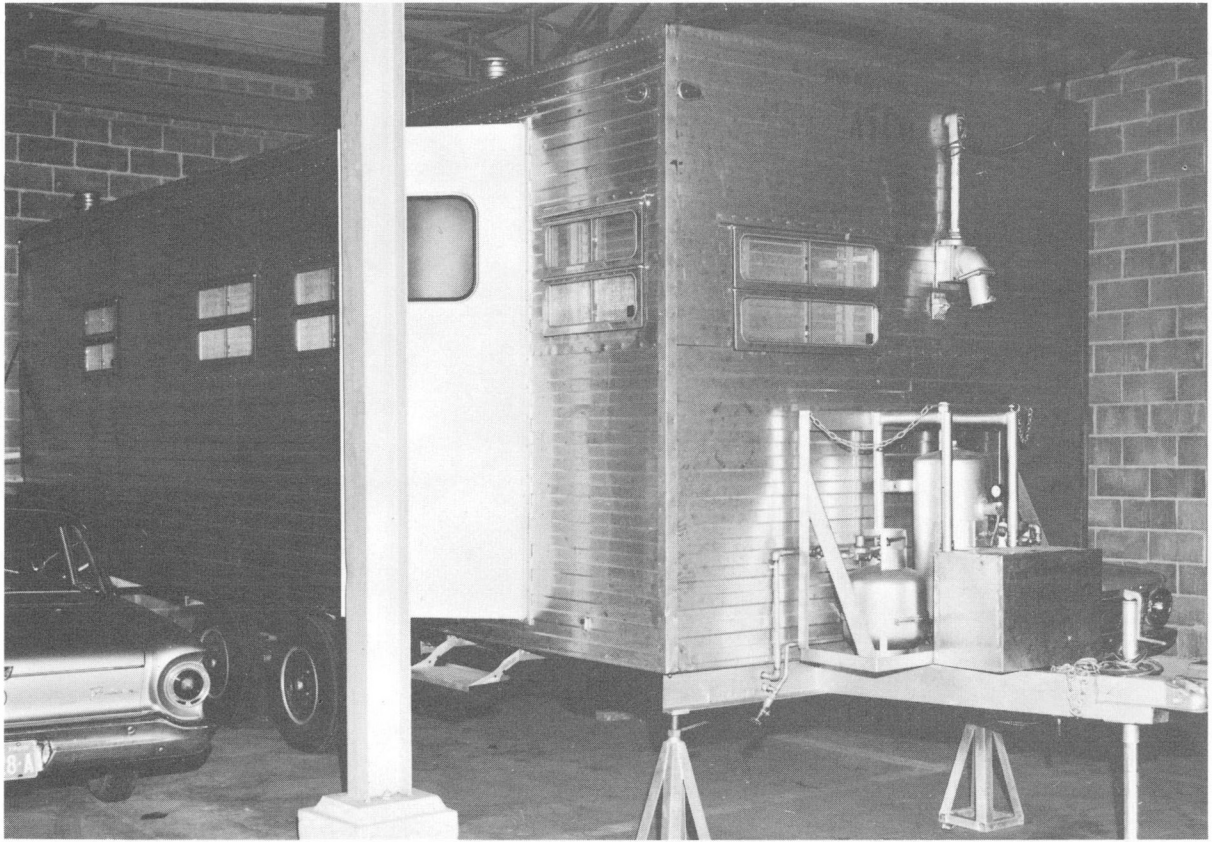

PLATE B 1 Exterior view of the Sample Preparation Trailer at the winter location.

PLATE B 2. Exterior view of the Spectrograph Laboratory Trailer at the winter location. Note the duct-work to the furnace room, the drainage pipe along the wall, the 100 amp service cable coiled under the trailer, and the eight point jack system.

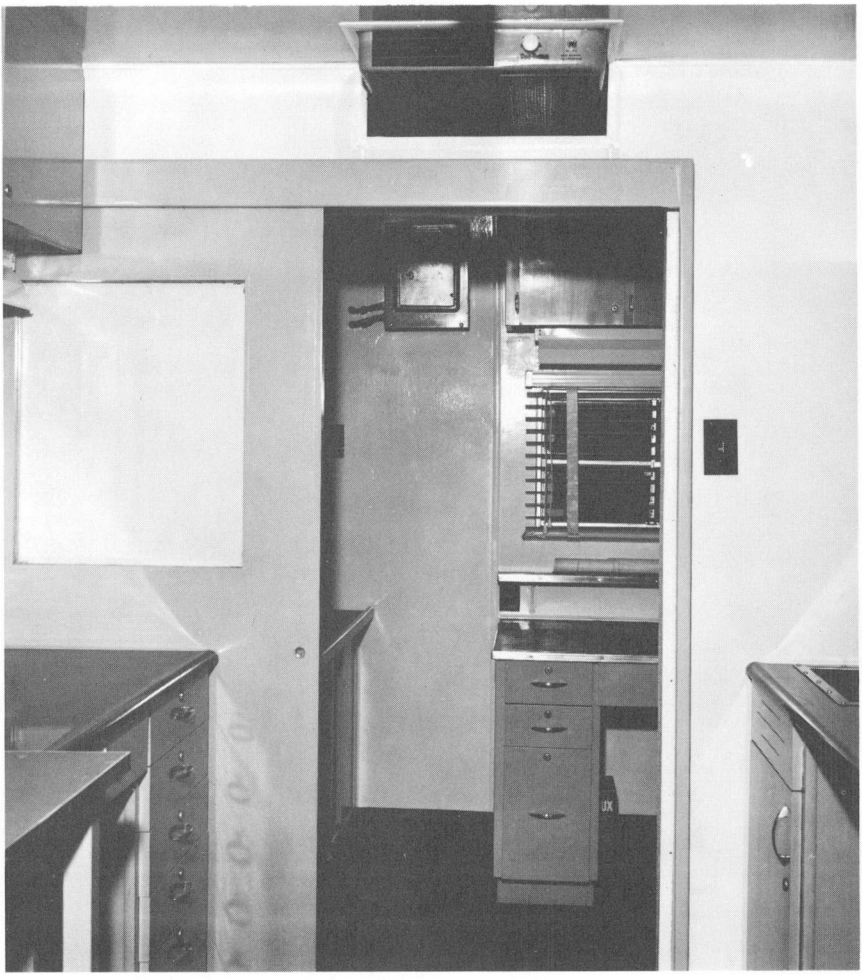

PLATE B 3. Interior of Sample Preparation Trailer showing office and the Geobotanical laboratory in the foreground. Note the air conditioner mounted in the roof and the sliding door between the two compartments.

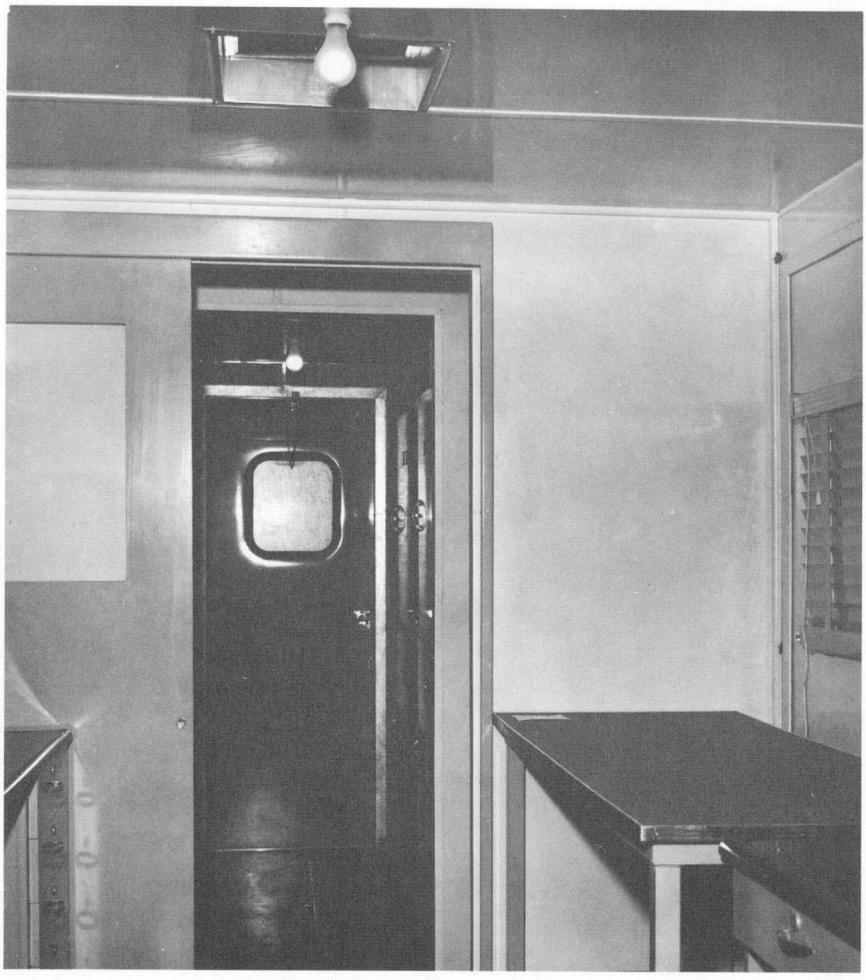

PLATE B 4. Interior of the Sample Preparation Trailer showing the Geobotanical laboratory in the foreground, and the storage space and air drying cabinets in the sample drying room in the background. The movable panel appears at the right of the picture in the Geobotanical laboratory.

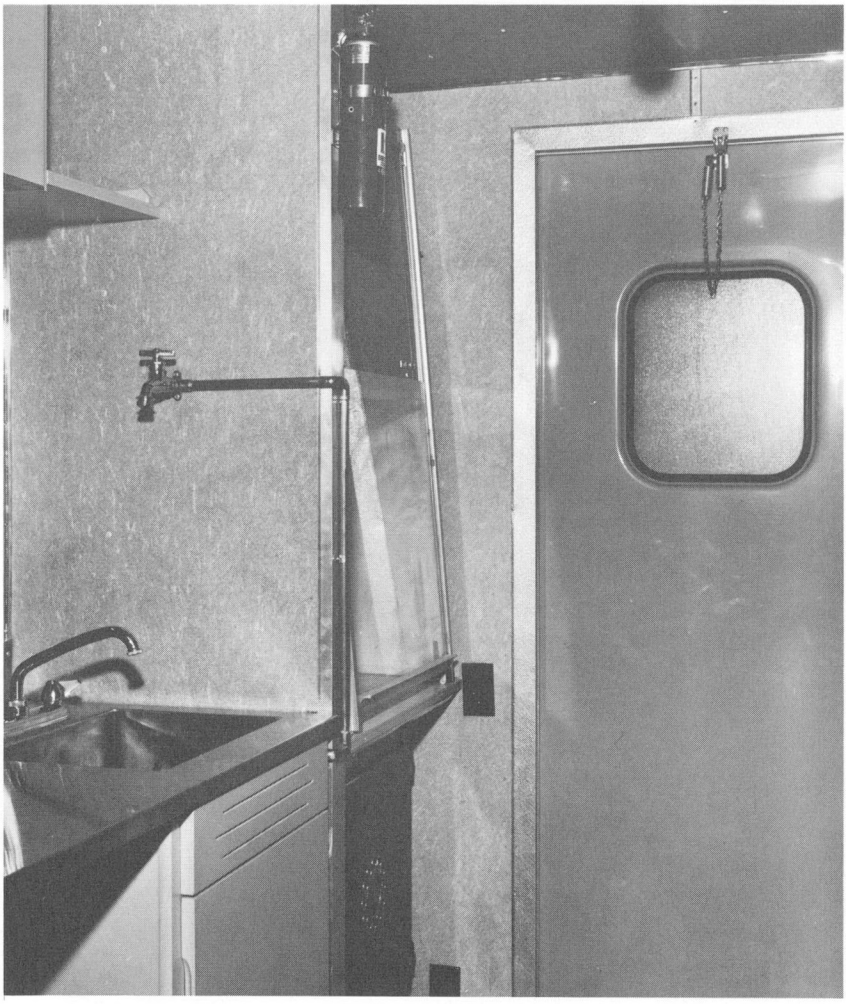

PLATE B 5. Interior of the Sample Preparation Trailer showing the fume hood and drying oven in the sample drying and subsampling room.

PLATE B 6. Interior of the Sample Preparation Trailer showing the drying racks within a drying cabinet.

PLATE B 7. Interior of the Spectrograph Trailer showing the front end of the Spectrograph Room; the darkroom at left, and the densitometer room at right. Note the Spectrograph in the left foreground and the refrigerator and spark source unit at the right. The cables carry the current from the source to the arc stand. The air conditioner is seen in the roof in the foreground, and the film processor and densitometer are shown in the two rooms in the background. The door to the outside is located at the left side between the source unit and the densitometer room.

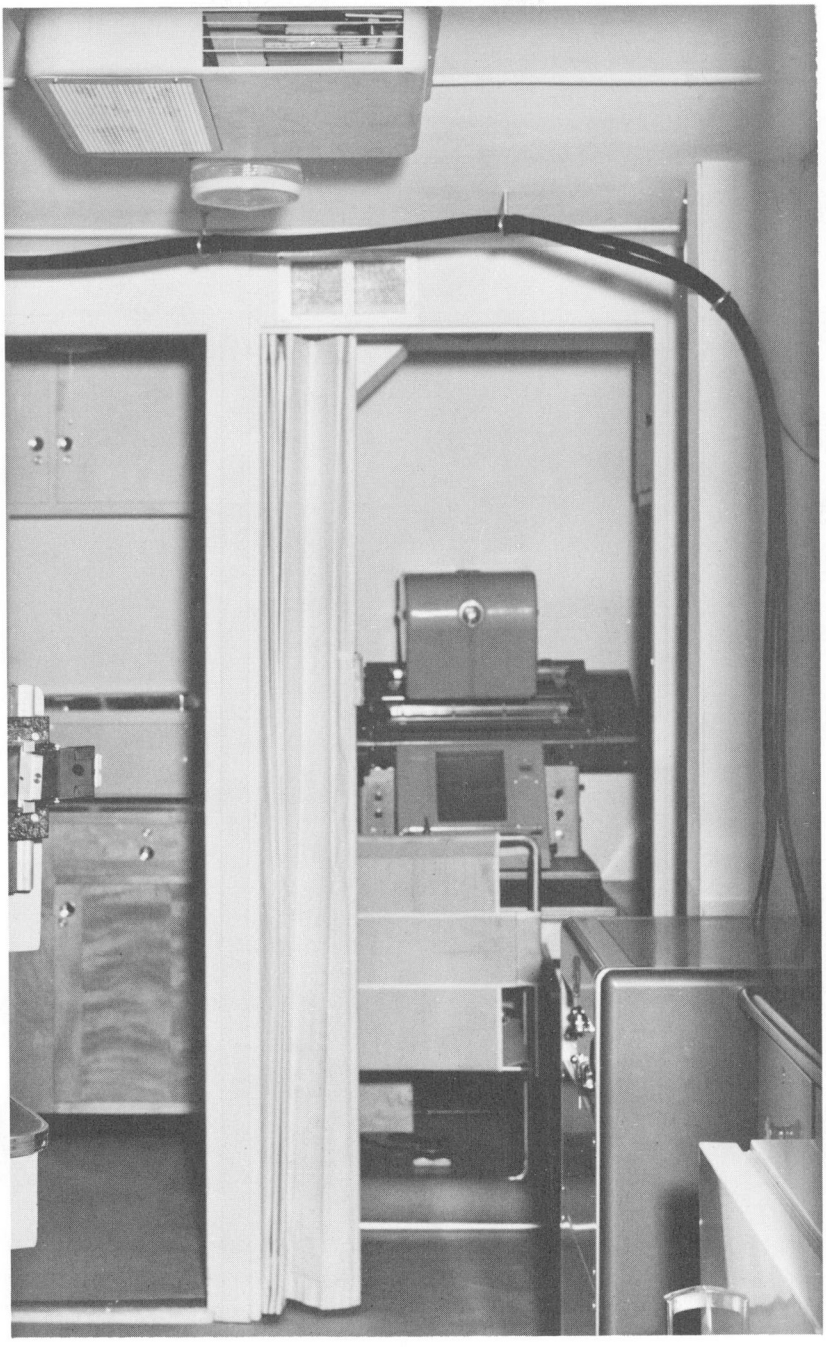

PLATE B 8. Interior of the Spectrograph Trailer.

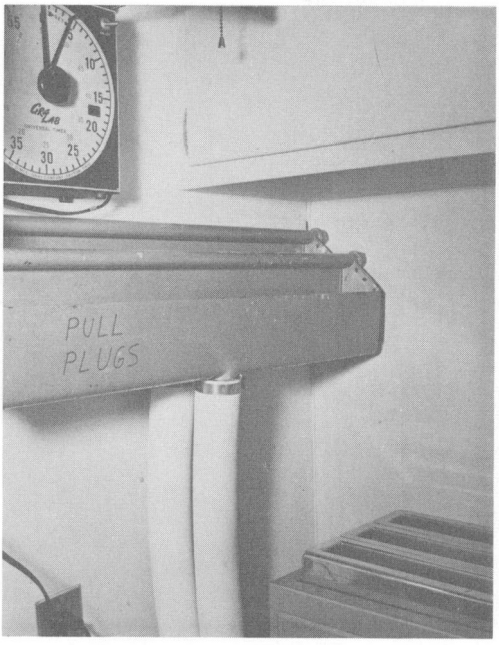

PLATE B 9a. Details of the installation of the film washing trays in the darkroom of the Spectrographic Trailer Laboratory.

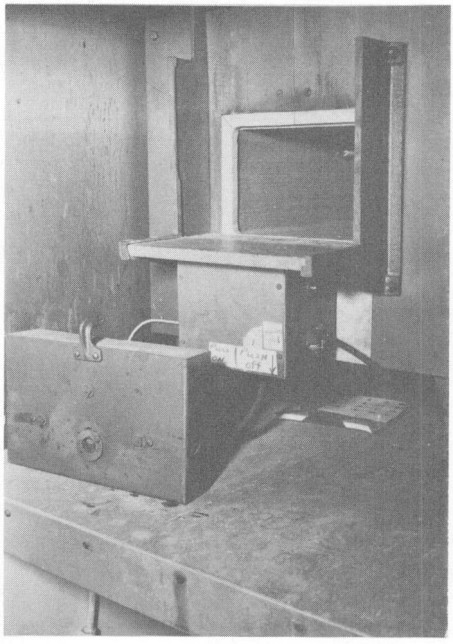

PLATE B 9b. The Hoskins type muffle furnace in the Spectrographic Trailer Laboratory. Note the lead from the controller at right and the thermocouple within the furnace.

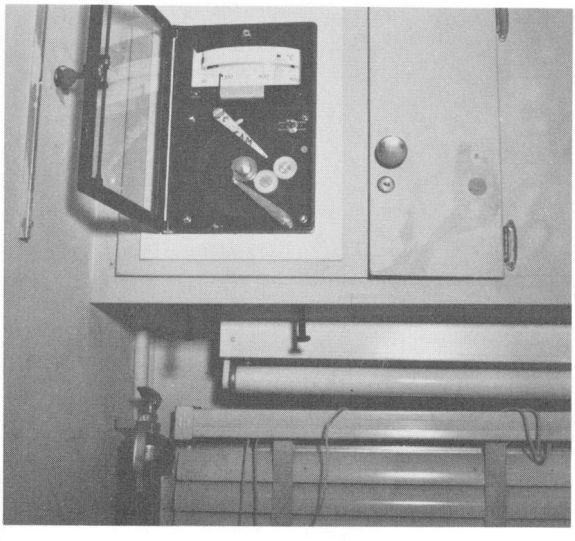

PLATE B 9c. The cam operated temperature controller for the muffle furnace as mounted in the chemical laboratory of the Spectrographic Trailer Laboratory.

APPENDIX BA

Equipment installed in Sample Preparation Trailer

1) Office No special equipment installed.
2) Geobotanical laboratory No special equipment installed.
3) Sample storage area No special equipment installed.
4) Subsampling and drying room Equipment installed as follows:

   a) Three full-sized rock cabinets (Office Specialty Co., Ottawa, Ont.): width 25 1/4", depth 24 1/4", height 75", with provision for 60 movable shelves 1" apart.
   b) Three heavy duty electric heaters (Markel Products Ltd. Fort Erie, Ont.): Model HIH 603T 3kv, Phase 1 240 volts.
   c) Drying oven (Fisher Senior Forced Draft Isotemp): 239 volts 60 cycle A.C., 1095 watts.
   d) Balance (Mettler Grammatic Balance Model K7T): Maximum load 800 g.

Remarks The standard rock cabinets were modified for use as drying cabinets. The modification involved the provision of two adjustable draft slides in the lower door of each cabinet, and, after 12 inch holes had been cut in the top of the cabinets, the fabrication of duct-work connecting the top of the cabinets with extractor fans built in the roof. For optimum drying conditions within each cabinet the fan heater was set at 3/4 full heat and the roof fans were turned on.

Originally, stainless steel shelves were used in the cabinets, but these proved unsatisfactory owing to rusting and were replaced by wooden trays of the type shown on Plate B 6. When the trailer was designed, provision was made for a water still and resin columns to be installed above the sink. At the time of writing these had not been installed.

APPENDIX BB

Equipment installed in the Spectrograph Laboratory Trailer

1) Furnace Room Equipment installed as follows:

   a) Muffle furnace (Hoskins Type FD 204, 220 volt): Inside box width 7.5", height 5.25", length 14.0".
   b) Hot Plate (Fisher Oscillating hotplate Type 11-429).
   c) Shaker (Eberbach 2 speed laboratory shaker and utility box).

Remarks The muffle furnace (Pl. B 9) was modified in two ways; a chimney, of 1" diameter iron pipe 14" long, was placed at the distal end of the furnace box just beyond the limit of the sleeve; and a 1/2" plate of asbestos was fixed inside the door in order to improve the insulation of the furnace. The temperature control of the furnace is maintained by a cam type program controller that is installed in the chemistry laboratory and is, thus, removed from the heat of the furnace (see Pl. B 9c). The shaking machine was placed on the floor under the fume hood, and when it is in operation it does not seriously interfere with the operation of the analytical balance in the chemistry laboratory. At the time the trailer was designed, it was not possible to obtain a ready made fume hood of the right size and weight for installation in the Trailer, and, consequently, a stainless steel fume hood was built into the trailer by the manufacturer. Experience showed that this hood was unsatisfactory for routine operation because of corrosion in the chimney and in the vicinity of the extractor fan which resulted in solid matter falling on to the hotplate at infrequent intervals. The installed fume hood is, therefore, being replaced by a Labcocco unit of the type installed in the Sample Preparation Trailer.

2) Chemistry Laboratory Equipment installed as follows:

a) Analytical balance (Sartorius Single Pan Balance Model 2623).
b) Torsion balance: capacity 50 mgm (Model "O" White Electrical Instrument Company).
c) Heat lamp ("Infra Rediator" with two 250 watt infra red heat lamps Fisher Scientific Co. No. 11-504-5v4).
d) Furnace controlled electronic "on" "off" controller (Thermovolt Instrument Ltd. Toronto 18, Ont.) Model PEC and MEC type 21.

Remarks The Sartorius balance was selected to enable the weighing of tarred objects up to 100 gm with an accuracy of 0.1 mg. This balance worked well in the trailer in the winter or summer location without any special precautions necessary to ensure a stable base. The form of the cam used in the program controller for the muffle furnace, as described above, was worked out experimentally using a standard thermocouple. No hot water system is provided in the trailer. A supply of hot water for washing apparatus is obtained from a two quart domestic electric kettle.

3) Spectrograph Laboratory Equipment installed as follows:

a) Jarrell Ash 15,000 line per inch replica grating spectrograph with ruled area 1 1/2" high by 2 1/4" wide. Wadsworth 1.5 metre stigmatic mounting. Linear reciprocal dispersion is

5.4 angstroms/millimetre in the second order. The entire region from 2100 Å to 9700 Å can be photographed. As the instrument is used, the wavelength range from 2100 Å to 4850 Å in the second order is photographed. Camera photographs 20 inches of spectrum on 35 mm film. Excitation of electrodes takes place in Jarrell Ash Arc/Spark stand model No. 19,000.

b) Excitation Unit (Applied Research Laboratories Modular Source Unit (H.V.S.), Model 28,000): Voltage 18,000 peak, Capacitance 0.007 microfarads, inductance 200 microhenries.

c) Voltage Regulator (Superior Electric Co. "Stabiline" automatic voltage regulator): Type ENT 4207, phase 1, KVA 7.5, normal output voltage 195-255, maximum line current 32.5 amps.

d) Timer in shutter circuit (Industrial Timer Corporation Parsippany, N.J.): Model P-3M, volts 115, cycles 60, 15 watts.

Remarks The spectrograph was specially shock mounted on the table. A small exhaust fan is provided above the arc stand and another inside the source unit to remove fumes. Small modifications were made to the arc stand to enable the platrode motor (see Section C) to be placed in the correct position. Special precautions are taken to ensure that the spectrograph table is rigid during transit. The large instruments are brought into the trailer through the side wall after taking off the movable panel (see Fig. B 1). Details of the preparation of the Spectrograph for transit are given in the paper by Holman and Durham (in press).

4) Dark Room Equipment installed as follows:

a) Film developing machine (Applied Research Laboratories): Model 2300.

b) Process Timer (Gralab Universal Timer): Model 171 (Dimco-Gray Co., Dayton, Ohio).

c) Film Washers: Two 35 mm film washers made by Applied Research Laboratories.

Remarks Experience has shown that a supplementary heater for the film processing solutions is required during winter operation of the trailers and a battery heater has been installed below the water bath in the body of the film processor. In order to save space the two film washers were installed one behind the other on the darkroom wall (see Pl. B 9a).

5) Densitometer Room Equipment installed as follows:

a) Comparator densitometer "Spectroline Scanner" (Applied Research Laboratories Model 22000).

Remarks In order to reduce operator fatigue, particularly with respect to the manual movement of film holders, the comparator densitometer was tilted upwards 30° about a horizontal axis coincident with the back of the instrument. This modification was found to facilitate the routine operation of the instrument as a comparator.

SECTION C DESCRIPTION OF METHODS OF SELECTION OF FIELD AREAS, COLLECTION OF ROCK, SURFICIAL MATERIAL, SOIL, AND VEGETATION SAMPLES; PROCESSING AND CHEMICAL ANALYSIS OF SAMPLES AND MECHANICAL METHODS FOR PLOTTING RESULTS

by J.A.C. Fortescue and E.H.W. Hornbrook

C 1 Introduction

One of the major objectives of the biogeochemical research program originally stated in Section A is as follows: to develop an integrated system of

a) field area selection,
b) sample site layout,
c) rock, surficial material, soil, and vegetation sampling,
d) sample preparation and processing,
e) chemical analysis,
f) mechanical plotting, and
g) interpretation of results which can be used to produce uniform sets of descriptive and chemical data from observations and samples collected at an instant in time in any Canadian landscape.

In Section B the Movable Spectrograph Laboratory, which was set up to enable sample preparation and processing and chemical analysis of subsamples to be carried out in the field during the summer, was described. In this section the methods system which has so far been evolved to achieve the above objective will be described under the following headings:

1) Methods of assignment of a type to an investigation to be carried out within a given landscape.
2) Methods of field description of landscapes and the general layout of sampling procedures within a given type of investigation.
3) Methods of description and collection of rock, surficial material, soil, and plant samples.
4) Methods of preparation of samples for chemical analysis.
5) Methods of chemical analysis of subsamples for minor elements.
6) Methods of mechanical data processing.
7) Methods of preparation of results for interpretation.

In order to maintain continuity in these descriptions, details of specific operational procedures (for example, the loading of spectrograph electrodes) are given in appendices.

C 2 Methods of assignment of a type to an investigation to be carried out within a given landscape

As a result of field experience gained during the summers of 1963, 1964, and 1965, it was decided to describe the different kinds of field investigations carried out in the biogeochemical research program under several headings: Visits, Pilot Projects, Main Projects, Quick Projects, and Special Projects. In the spring of 1966 a proposal describing three of these terms (Fortescue, 1967) was submitted for inspection by the Subcommittee on Mineral Deposits of the National Advisory Committee on Research in the Geological Sciences. This proposal was later published as an appendix to the annual report of the main committee. For completeness the three terms are described again with two others that have been established since.

(1) Visits It was evident from the background information given in Section A 3, that biogeochemical prospecting methods research involves the description of components of the landscape, as well as of plants, in an area selected for study. Experience gained at eleven undisturbed mineral deposits located in different parts of Canada has shown that much time can be saved if a two or three day Visit is made to a property before deciding whether or not further work should be done in the area. Although much important information about a mineral deposit and the landscape in which it occurs can be obtained orally from a company representative (or, perhaps, in writing in response to a set of questions), there is no substitute for a Visit to a property to bring into focus the particular problems of plant prospecting research within the area. For example, the extent and nature of the plant community types in relation to bedrock, surficial material, and soil conditions are of vital importance in our research, but at present, these parameters are seldom studied systematically during active exploration. Experience has shown that we cannot gain sufficient plant community data from air photographs alone without at least some ground control. A further advantage of a preliminary Visit to a property is that it provides an opportunity to collect a small, representative, suite of soil and herbarium specimens that can provide a firm foundation of detailed information on which to build any further project in the area. The location of ten Visits made by the group to date is listed in Figure C 1. These Visits will be described briefly in Section E (Part II).

(2) Pilot Projects The Visit was conceived originally as an essential preliminary to the selection of an area for a Pilot Project or a Special Project. A Pilot Project is designed to produce a uniform set of descriptive and chemical data from observations and samples collected along a cut line and at an instant in time, from a landscape in which an undisturbed mineral deposit occurs. During a Pilot Project information on lithology, structure, and particularly the nature of the bedrock surface, the overburden, and soil is collected in addition to information on plants. To date the only Pilot Project completed was at the Silvermine Property of Yava Mines Ltd., Cape

Breton Island, Nova Scotia (see Section G, Part II). This first Pilot Project is itself somewhat experimental and is not as comprehensive as those planned for the future.

(3) Main Projects After a number of Pilot Projects are completed and the results compared we hope that one area will be of particular interest. Consider a single mineral deposit system (e.g., a complex mineralized vein) sub-outcrops in adjacent landscapes of different types (e.g., alpine, forest, and organic terrain). In such a case the results of the Pilot Project might not be considered broad enough in scope to attain all plant prospecting methods objectives in the area, and a second more detailed Main Project would be required to tap the full research potential of the area. As planned at present, a Main Project will involve, in addition to biogeochemistry and geobotany, other parallel investigations (e.g., geophysics, surficial geology, surficial material drilling, and other types of geochemical prospecting). If required, a Main Project (unlike a Pilot Project which is carried out at the FOLLOWUP LEVEL of detail) (see Section A 3), could also be carried out at the REGIONAL LEVEL of detail.

(4) Quick Projects Occasionally, landscapes favourable for biogeochemical prospecting research have been brought to our attention a short time before a deposit is disturbed prior to mining. An example was the Texas Gulf Sulphur deposit near Timmins. The Geological Survey team was on the property within 48 hours after permission was granted to carry out the plant and soil sampling program. The sampling project was completed within 8 days during which over 500 samples of the two materials were collected. When the collecting party left the area the vegetation had already been stripped from the area from which samples were taken during the first few days! Such a project is called a Quick Project and is normally not so broad in scope as a Pilot Project carried out after a Visit. Two examples of Quick Projects will be described in Section F (Part II) (see Fig. C 1).

(5) Special Projects The four types of project already described were carried out near drilled, undisturbed mineral deposits. In addition we have carried out three field investigations in landscapes located away from mineral deposits. The results of these Special Projects are relevant to later investigations carried out near mineral deposits. For example, a method of collecting bog samples was set up at the Mer Bleue peat bog (see Section H, Part III) during the summer of 1963. Without this preliminary experience it would have been impossible to collect the samples of organic soil from the Timmins area at the speed dictated by events during the Quick Project the following year. In Part III the three Special Projects which have so far been completed (see Fig. C 1) will be described.

The relationships between the five different types of projects are shown on a flow sheet in Figure C 1 which also lists the examples of each project type completed under four of the headings. So far Visits before Special Projects have been somewhat informal and no written reports have been produced.

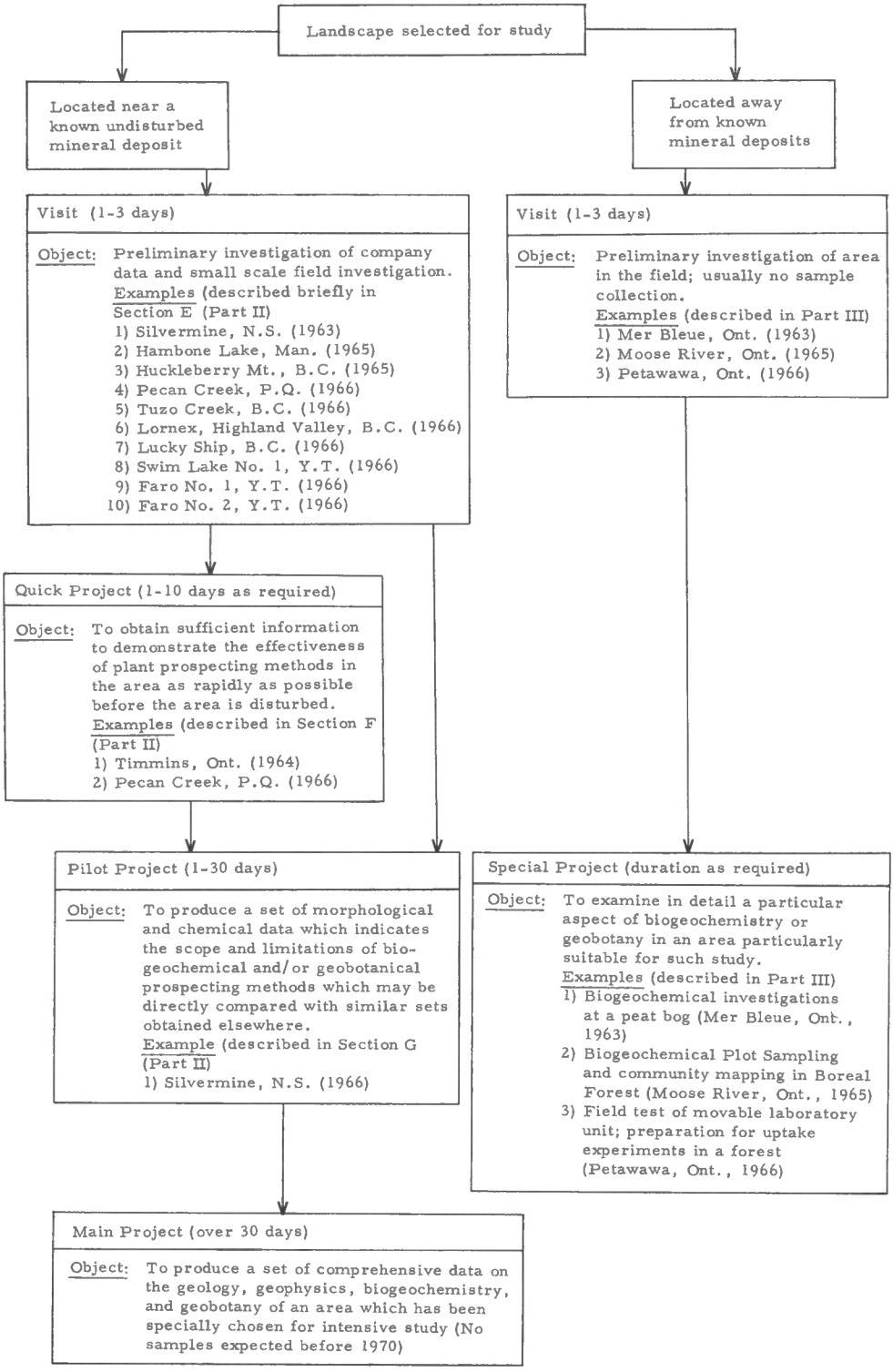

Figure C 1. Flow sheet showing the relationships between the five types of field investigations. The examples near mineral deposits will be described in Part II and those in other areas in Part III.

C 3 Methods of field program layout within a given type of investigation

The concept of the prospecting prism (Fig. A 6) is a convenient way of focussing attention on the scope of geobotanical and biogeochemical research conducted within a given landscape. The fundamental principle of biogeochemical prospecting involves a comparison of results obtained from chemical analysis of a number of samples of like material (for example, second year twigs) collected from individuals of the same plant species from sample sites distributed within the area being surveyed. The sites from which samples are collected may be located along a cut line, within a plot, or be based on some other consideration; for example, where individual plants of a given species are found. From the point of view of field collection, sample processing, chemical analysis and particularly the plotting of data, it is desirable to have groups of samples of the same material that can be treated throughout by routine methods.

These field laboratory and data recording requirements have been met by the evolution of a relatively simple system based on the Site Set. A Site Set is defined as a group of not more than 15 field stations which are related to each other in some way. For example, samples may be located at fixed intervals along a cut line, or at random within a plot, or at regular intervals down a hole drilled in a peat bog. All that is required is that a Site Set includes from one to fifteen stations. The number fifteen was chosen because there are sixteen positions on the spectrographic film thus permitting fifteen unknowns and a standard (see below).

The numbering system used for samples within a Site Set collected in the field persists until the results of the chemical analysis are plotted mechanically by the computer and is consequently of major importance. Each sample is given a six digit number plus a letter. The makeup of this code never varies, the first pair of digits refers to the project number (00-99) the second pair of digits (00-99) refers to the Site Set, the third pair of digits (00-99) refers to the type of material comprising the sample and the letter (A-$\emptyset$) refers to the sample point from which the sample was collected. Experience has shown that the station within a Site Set (for example, a picket on a cut line) must be distinguished from the point from which a sample is actually collected (for example, the nearest individual black spruce tree). When results are plotted on a map or plan, both the stations and the sample points are shown but during processing of samples only the point designation letters are used. When station locations are plotted on a map or section, they are referred to by a two digit number. This number begins with 01 in the first Site Set, 16 in the second Site Set, and so on. The station number is marked in the field at the time that the plant samples are collected, and may be used later for shallow seismic or other types of observations. Experience has shown that this numbering system works well in practice

although it becomes somewhat cumbersome if less than five stations are included within a Site Set. In order to take maximum advantage of the system, every effort has been made to use full Site Sets in more recent investigations.

Samples of the same material collected from the same Site Set are called Batches and, therefore, a full Batch from a given Site Set contains fifteen samples. The numbering system is flexible with regard to the number of samples of a given material within a given Site Set. For example, if 660160 denotes a Batch of lower bark (i.e., bark taken near the ground) samples which have been collected from nine stations, it is as easy to process and plot results from sample points A, B, C, D, G, I, K, M, & Ø as it is to plot results from adjacent points A, B, C, D, E, F, G, H, & I. This principle becomes particularly important when the vegetation of an area is complicated. For example, during the Timmins Quick Project one of the cut lines was located across a transition zone and within two distinct plant cover types. Within this Site Set eighteen Batches of separate materials were collected of which only two of the Batches contained the full fifteen samples. All these samples were processed without difficulty. Another advantage of this numbering system is that the same system can be used for Batches of samples collected in the field as well as for those which result from subsampling during sample preparation. For example, a foliage sample of a conifer (batch number 420170A) would, after drying and subsampling, generate the following six different Batches of plant organs:

Current needles	420166A	(15 samples)
Current shoots	420165A	(15 samples)
Second year needles	420164A	(15 samples)
Second year twigs	420163A	(15 samples)
Third year needles	420162A	(15 samples)
Third year twigs	420161A	(15 samples)

A further advantage of this system is that, while it facilitates the identification and sorting of samples by those familiar with the code system, the nature of the code makes casual interpretation of the results on the computer printout sheets difficult for those unfamiliar with the code.

This sample numbering system has been used for over two thousand samples involving several tens of different kinds of materials. It was used for sample points and stations located along cut lines (e.g., at Timmins), in plots (e.g., in the Moose River Special Project), for Bog samples (collected during the Mer Bleue Special Project), as well as for samples of surficial material (e.g , during the winter drilling project at Timmins). In all these cases the method was successful in facilitating the location and treatment of individual samples, and with very few exceptions, all samples were analyzed without difficulty.

C 4 Methods of description and collection of rock, surficial material, soil, and plant material

The operations involved in the collection, processing, and recording of results from chemical analysis of rock, surficial material, soils, bog material, and vegetation for biogeochemical research are summarized in Figure C 2. The location of the different operations carried out in the Movable Spectrograph Unit was described previously (see Fig. B 1). The different operations shown on the flow sheet (Fig. C 2) could be described in two ways according to material or according to type of operation (involving all materials). In order to avoid repetition, the latter method was chosen and each description begins with rocks (i.e., at the bottom of the prospecting prism) and ends with plant materials. Details of specific operational procedures are given in a series of Appendices.

(1) Rocks. Details of the geology of the landscapes in which undisturbed mineral deposits occur are obtained from the company engaged in prospecting and are based on the interpretation of geological, geophysical, and geochemical observations that had been made during prospecting before a Visit. In order to carry out field investigations data on tonnage or grade of mineral deposits is not needed, although a plan showing the known limits of the sub-outcropping mineralized zone is needed. Where a deposit has been mined by open pit methods soon after an investigation has been completed (for example the Quick Project at Timmins) detailed information on the geology of the bedrock and surficial material may be obtained from the rock exposures in the pit.

In areas where there is little outcrop of bedrock it is desirable to obtain small, typical hand specimens of the common rock types found within and near the deposit. So far we have obtained a large number of rock samples from only one property where a deposit is completely covered with overburden. This was during a visit to the Lornex Mining Corp. copper property in the Highland Valley of British Columbia, where percussion drill rock samples were obtained from fifteen holes located along parallel lines 200 feet on either side of the line from which soil samples were collected (see Section E, Part II).

Two drilling programs have been carried out under our direction. One followed the Timmins Quick Project in February and March 1965 where ten holes were drilled to bedrock and ten foot diamond drill cores were collected from the bottom of each hole (see below). The second drilling program was undertaken at the Mer Bleue where a single hole diamond drill core was collected.

Clearly, from the theoretical point of view, it is desirable to collect rock samples systematically; from the practical point of view, the high cost of drilling usually secludes systematic drilling to bedrock in our research.

(2) Surficial material. Investigations of surficial material have so far been of two kinds: those aimed at describing the thickness and composition of the material, and those aimed at collecting samples of surficial material for chemical analysis. The former investigations have involved the use of shallow seismic techniques and the latter drilling with a Failing Drill or a Pendrill. All the shallow seismic investigations carried out in connection with our research program have been made under the direction of G.D. Hobson of the Geophysics Division of the Geological Survey. The first seismic investigation was carried out at the Texas Gulf Sulphur property near Timmins during the summer of 1964 to obtain data on the thickness of the surficial deposits in the area. It was soon terminated when it was learned that data of the type required based on extensive Pendrilling of the surficial material by the company, would become available to us (see Fortescue and Hughes 1965). The seismic method was used again during the Moose River Special Project in the summer of 1965 when overburden thicknesses were obtained at each of twelve plots sampled and also along a traverse across a muskeg three miles long (see Section I, Part III). During 1966, the shallow seismic method was used at the Pilot Project at Silvermine (see Section G, Part II) and in the Quick Project at the Pecan Property of Terra Nova Mines Ltd. (see Section F, Part II). A brief description of the principles of the shallow seismic method used is given in Appendix CA. The results of the shallow seismic method have been so satisfactory that investigations of this kind will be included in all future Pilot Projects, and Main Projects, and when possible, in Quick Projects.

The most difficult part of the prospecting prism to sample for chemical analysis is the surficial material. Meaningful interpretation of results of biogeochemical prospecting methods research is often impossible without results from both physical and chemical investigations of the material which is found below the soil and above the fresh bedrock surface. Commonly, where local conditions permit, surficial material less than 10 feet thick, can be sampled from trenches dug with a tractor-mounted back hoe. Where the material is deeper than this samples must nearly always be collected by drilling, which often involves heavy equipment that can only be brought to the site when the ground is frozen. So far we have carried out a single drilling program of this type, at Timmins.

Sampling of surficial material during drilling can be done in several ways, for example, during February and March 1965 during the Timmins drilling project samples were collected at five foot depth intervals from each of ten holes. Varved clays were sampled with a 2-inch diameter Shelby Tube 24 inches long; and samples of Lower Till, below the clay layer, with a split spoon or a hardwall sampler (see Fortescue and Hughes 1965). A diamond drill core was collected below the till in order to prove bedrock.

As a result of the experience gained during the Mer Bleue Special Project in 1963 (see Section H, Part III), we became interested in obtaining core samples of surficial material between the bottom of the bog and the

bedrock surface. During the fall of 1965 it was arranged that two holes would be put down at the Mer Bleue using a Pendrill which was then being tested by the Department of Public Works.[1] In one of these a continuous 2-inch Shelby Tube core was taken of the clay material, and in the other, sampling was carried out at 5 foot intervals. Bedrock was proven in one hole.

(3) Bog material. Experience in Finland (Salmi,1955, 1956) and in Canada (Gleeson,1960) has shown that biogeochemical prospecting can sometimes be successfully carried out in organic terrain. In anticipation that some of the investigations of undisturbed mineral deposits would be carried out in peatland areas it was decided in the spring of 1963 to develop suitable routine methods for the collection of samples of organic material. Eventually, a Special Project was set up at the Mer Bleue peat bog near Ottawa (see Section H, Part III) where different types of peat sampling apparatus could be tested. Core samples of peat were obtained at 2-foot depth intervals in holes located at 200 foot intervals along lines traversing the bog. Three different kinds of peat samplers were tested none of which was entirely satisfactory. The first was a 1 1/2 inch diameter piston type sampler. This produced a core of suitable diameter but too short for sampling at the rate required. The second type was a Hiller which, although quick to use and easy to insert into deep (15- to 20-foot) holes, did not provide a large enough sample of the type required for chemical analysis by the methods to be described below. Best results were obtained with the third type which was loaned by Mr. I.C. MacFarlane of the Division of Building Research, National Research Council, Ottawa. This 3-inch Shelby Tube sampler produced an excellent core and was relatively simple to use in holes less than six feet deep. Because of the large diameter of the core, the sampler was difficult to manage at greater depths. This sampler was used to obtain shallow samples from the Mer Bleue and all the organic soil samples collected during the Timmins project the following year.

Eventually, as a result of this experience, a 2-inch piston type Shelby Tube sampler was designed and fabricated in the workshop of the Geological Survey by Mr. A.G. Meilleur (Pl. C 1). The operation of this sampler is described in detail in Appendix CB. This sampler was used successfully during the Moose River Special Project in 1965 (see Section I, Part III) and it was found that cores could be collected from as deep as ten feet without great difficulty.

(4) Soil material. Because of the extreme variability of the morphology of Canadian forest soils we have made it a rule to take soil samples from pits dug to expose the soil profile and not by hand auger. The profile inspection and sampling pit is dug with a trenching tool at each sample point within a Site Set. Pits usually extend to the transition zone below the B horizon and are often not more than eighteen inches deep.

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[1] By kind permission of Mr. N.E. Laycraft, Chief, Testing Laboratories Division, Department of Public Works, Ottawa.

To date our descriptions of soil profiles have been based on the following generalized terminology:

Material	Descriptive term
Litter	(Not sampled)
Organic soil layer	Humus (not further differentiated)
Visible leached layer	A_2
Transition zone	B_1
Zone of iron accumulation	B
Transition zone	B_2
Light coloured subsoil	C

Samples of mineral, or organic soil are collected in small (4 inch by 10 inch) paper sacks beginning at the bottom of the hole. As far as local conditions permit, our sampling policy has been to collect comparable material (for example, humus or B horizon) from each pit within a Site Set. Therefore, transition zones and the A_2 horizons are sometimes included in profile descriptions although samples are not collected from these (see Appendix CC). Beginning in 1967 representative soil profile monoliths (see Clarke, 1957) will be collected from typical soil types within each Site Set. Each monolith will be used as a source of detailed information about the morphology, the physical, and chemical properties of the soil, and possibly, for certain greenhouse experiments.

(5) Plant Material. So far plant cover types have been described using the system shown in Figure A 6 for three strata of vegetation:

(c) Overstory vegetation (tree).
(b) Understory vegetation (herb, shrub, tree).
(a) Ground vegetation (moss, lichen, herb, shrub).

These terms were selected for their practical value and are not designed to replace exact and detailed terminology set up by ecologists, foresters, and others who describe vegetation in detail.

The development of methods of plant cover type description specifically suited to the needs of our research program was commenced in the summer of 1965 by Miss L. Usik. As a result of preliminary investigations at Mer Bleue, and later experience in northern Ontario, Manitoba, and British Columbia, a provisional, twofold method of plant cover mapping was evolved based on (1) general maps prepared from air photographs (with sufficient ground control), and (2) detailed maps based on ground observation of plant species distribution within a given plot.

The object of the general maps was to delineate the easily recognizable static and transition plant cover types which occur within an area being studied (see Section E, Part II and Section I, Part III). The object of the

detailed maps was to produce a rapid method of description of relatively small areas within a general plant cover type that could be used as a basis for comparison with similar small units located in the same (or a similar) plant cover type (see Section I, Part III).

Experience showed that it was desirable to make herbarium collections during field observations prior to the preparation of general maps (see Section I, Part III) and particularly during intensive field investigations prior to the preparation of detailed maps of small areas (see also Section I, Part III). Herbarium specimen collections were also made during Visits in British Columbia and the Yukon during 1966 when samples of plants growing in the vicinity of each station within a Site Set were collected without the preparation of cover type maps.

Because the geobotanical investigations are still in the experimental stage of development and have not, as yet, been set up as Operational Procedures, descriptions of progress along these lines will be given in Parts II and III and not as appendices.

The sampling of vegetation for biogeochemical research has already been standardized. Using the Site Set and Batch system described above, plant material has been collected so far in two Quick Projects, one Pilot Project and two Special Projects. In all cases the same sampling procedure for bark, twigs, and leaves from trees has been used (see Appendix CD). In addition during investigations carried out in 1965 and 1966, disc samples have been cut from the trunk of each tree in order to determine the approximate age of the tree and, hence, to permit height-age relationships to be obtained.

During 1965 and 1966 particular attention was given to establishing a routine method for collecting samples from trees. The climax of this phase of the research was reached during the Silvermine Pilot Project (see Section G, Part II) when three samples of bark, three of twigs, and three of needles, collected from each of 45 trees, were analyzed for twelve minor elements and the analyses compared with results of analyses of different horizons of the soil from each location. We realize that a sampling procedure of this kind is not suitable for routine prospecting, however, the valuable experience gained using this elaborate method of sampling will be used to make tree sampling in later projects both simple and effective.

Some experience has been gained in the collection and chemical analysis of moss and shrub understory material (see Section F, Part II). Studies of this kind will become more important in the future and will be supplemented by biogeochemical investigations of herbs.

C 5 Methods of Preparation of samples for chemical analysis

Details of the apparatus installed for preparation of soil, bog, and plant samples for chemical analysis and the rooms in the trailer laboratories

of the Movable Laboratory unit in which subsampling and drying are carried out were given in Section B. The different drying, subsampling, and ashing procedures described below were shown in relation to one another in Figure C 2 and the operational procedures involved are described in Appendices CE and CF.

(1) Rocks. No systematic operational procedure has been set up for processing rock samples by the unit. The rocks collected during the Timmins Project were prepared by methods established in the Geological Survey and described by Lavergne (1965). The chip samples of rock obtained during the Visit to the Lornex Property were passed through a disc pulverizer to reduce grain size and to ensure good mixing before chemical analysis.

(2) Surficial Material. The Shelby tube, hardwall and split spoon samples of surficial material collected during the Timmins drilling project were treated as follows. On arrival in Ottawa the Shelby tube sample cores were extruded from the tubes by a hand extruder and described immediately by Dr. O.L. Hughes. A small channel sample was then cut along the length of the wet clay and placed in a paper bag. The cores were allowed to become air dry. Samples obtained by the hardwall sampler, or the split spoon were described in the field condition as they were obtained. Both the clay samples and the coarse materials were oven dried at 110°C, crushed, and then passed through an 80 mesh stainless steel sieve. The minus 80 mesh material was further mixed and pulverized in a ceramic ball mill before chemical analysis.

(3) Bog material. Peat cores collected during the Mer Bleue Special Project were allowed to become air dry before subsampling. A channel sample (weighing some 15 grams) of the dry material was taken as being representative of each core. These subsamples were oven dried overnight at 110°C, and after cooling, a 10 gram aliquot was weighed out ready for ashing (see Appendix CF).

After the Sample Preparation Trailer was available, experiments were carried out to discover the time required to air dry peat cores in the drying cabinets. Experience showed that 96 hours were needed if the cabinets were kept between 50°C and 60°C.

(4) Soil samples. After care had been taken to ensure that bags containing soil samples were clean on the outside, free from holes, and securely sealed, they were placed in the drying cabinets of the Sample Preparation Trailer for 48 to 96 hours (depending upon the material) until the sample became air dry. After this treatment the samples were removed from the trailer laboratory (still sealed) and opened in a place where dust generated by sieving would not contaminate other samples. The samples of mineral matter were crushed and passed through an 80 mesh stainless steel sieve. The organic material was first passed through a 10 mesh stainless steel sieve

to remove sticks and large stones and then through an 80 mesh sieve. The minus 80 mesh humus material was dry ashed before analysis by the operational procedure described in Appendix CF.

(5) Plant samples. Moss and woody shrub material collected during the Timmins Quick Project became air dry before subsampling. The air dry material was later oven dried overnight at 110°C and 10 gram portions of moss, stems, and leaves were weighed out as soon as cold. Samples of bark, twigs, needles, and leaves collected before 1966 were air dried and then oven dried at 110°C with the bags closed. Aliquots of 10 grams of the oven dry material were taken for ashing (see Appendix CF). During 1965 it was observed that 2 gram aliquots of oven dry material could be used without affecting the results of chemical analysis and, therefore, during 1966 smaller subsamples were used throughout which resulted in a significant speedup in the analytical program. Relatively large subsamples of plant material (i.e., 10 grams) were originally selected for ashing in order to obviate the need for grinding resinous material (for example, bark of balsam fir).

A different procedure was adopted for subsampling the material collected during the Moose River Special Project. In this case the Batches of 15 samples were first oven dried at 110°C and a 0.5 gram subsample taken from each sample. The subsamples were combined to make a single composite representing each Batch. In total, one hundred and twenty composite samples were included in this project representing 1800 samples collected in the field. It is believed that variations of this compositing procedure will become important in our research program as the scope of sampling, with respect to plant species and organs, increases.

All samples of oven dry bog material, humus, and plant material are dry ashed in a programmed muffle furnace. A cam activated thermostat controls the temperature of the furnace during the ten hour ashing cycle (see Appendix CF and Section B). The exact form of the cam was established by trial and error in such a way that a white ash from plant material was produced without charring. The maximum temperature within the furnace does not exceed 435°C during the ashing cycle.

After the samples have been ashed and allowed to cool, the beakers are weighed and the weight of ash recorded. A small plastic ball is then placed in each beaker and the ash mixed well before chemical analysis.

C 6 Methods of chemical analysis of samples for minor elements

From time to time a number of methods of chemical analysis have been used on samples collected during the biogeochemical prospecting research program of which two, the colorimetric method of determination of

total copper, lead, zinc and nickel in soils, rocks, surficial materials, and plant ashes, and the spectrograph method (as set up in the trailer laboratory) for the determination of copper, lead, zinc, cobalt, nickel, chromium, barium, strontium, silver, titanium, molybdenum, and manganese in plant ashes are the most important. These two methods are described first and are followed by brief mention of others.

(1) The colorimetric method. Standard methods for the determination of small amounts of zinc, copper, lead, and nickel in the above mentioned materials have been in operation at the Geological Survey of Canada for some years. The techniques used for the determination of these elements have been described by Gilbert (1959) for copper, lead, and zinc, and by Stanton and Coope (1958) for nickel. The extraction of minor elements from samples has been carried out by a simple procedure developed by J. J. Lynch in the laboratories of the Geochemical Section of the Geological Survey (see Appendix CG). Check standards are always included in batches of unknown samples. Experience has shown that the results from these methods are suitable for geochemical prospecting. For a given element the figure for concentration quoted is always within 25 per cent of the content of the element present and usually within 15 per cent. It should be noted that for reasons described below, the colorimetric results cannot be processed directly by the computer program which has been designed for use with the trailer spectrograph method.

The colorimetric method has been used extensively. It was used before the spectrographic method was set up to determine the minor element content of selected samples of soil and vegetation collected during the Timmins Quick Project in 1964. It was used again during the Timmins drilling project to analyze samples of overburden and rock. All samples of mineral soil collected before 1967 were analyzed by this method, which was also used for systematic checks on the performance of the scan spectrograph method (see below).

(2) The rapid scan spectrograph method. The analytical method used for routine determination of the chemical composition of plant ashes and soils is the most important part of the methods system. Some considerations in the choice of the analytical methods were discussed in connection with the description of the design of the trailer unit (see Section B). The need for systematic data on the minor element content of Canadian forest plants collected from landscapes in which undisturbed mineral deposits occur was stressed in Section A.

The choice of a spectrograph method for biogeochemical prospecting research was governed by the following considerations:

(a) The method must be sensitive enough to determine the content of a number of minor elements (some of which are important in prospecting

and others, in plant nutrition) in samples of ashes of a number of different kinds of plant, bog, and organic soil materials.

(b) The method must be precise enough to detect significant variations in the minor element content of ash samples of these materials in samples whose chemical composition is influenced by mineral deposits from other samples whose chemical composition is not so influenced.

(c) The procedure adopted must be so devised that all the elements required can be determined in a single spectrum (i.e., the method is sensitive enough to determine all elements during the same burn).

(d) The concentration range involved for all elements of interest should be between 1 and 10,000 parts per million and all standards should appear on one master film.

(e) A reference standard element should be included in each subsample analyzed in order to obtain information on the precision of the method.

(f) In order to facilitate the analysis of large numbers of samples, the same standard film should be used for all ash samples regardless of origin.

(g) In order to obtain results in the trailer laboratory rapidly, the method should include a visual comparison step to produce results directly as parts per million without the laborious calculations normally involved in quantitative spectrochemical analysis.

(h) The method should produce results on at least two batches (30 samples) per day on a routine basis using a permanent staff member and two part time assistants.

(i) The combined ashing, electrode preparation, and spectrographic procedure must be suitable for teaching to seasonal help in the minimum of time so that meaningful results can be obtained during the greater part of the short summer season.

(j) The method chosen should be suitable for adaption for use with a chemical preconcentration procedure with a minimum of modification to the basic spectrographic method.

After very careful consideration of the details of all these requirements, some of which are mutually exclusive, the procedure summarized in Figure C 3 was evolved. This method has now been used for the analysis of over 2000 samples of plant and vegetable ash and has proven suitable for the preparation of scan sheets showing the level of concentration of twelve elements in a number of different materials (see below and Parts II, III).

The operational procedures involved in the different stages of the spectrograph method are described in detail in Appendices CF, CH, CI, CJ, and CK. Briefly, spark excitation was chosen in order to improve sensitivity and at the same time preserve precision and reduce background on films which often impedes visual comparison of D.C. arc spectra. In a further attempt to reduce background aluminium electrodes were tried but it was found that this innovation decreased the sensitivity for some elements below the level required. The rotating platrode method of introducing the sample

Table C 1

Summary of the performance of the rapid scan spectrograph method applied to eight materials collected from fifteen trees during the Petawawa Special Project, 1966.

Material	Element							
	Indium***		Barium		Strontium		Manganese	
	Mean[x]	Coeff.*	Mean	Coeff.	Mean	Coeff.	Mean	Coeff.
Leaf Blades	200	18	2800	30	1650	42	691	35
Petioles	180	10	3833	8	2550	41	541	50
Twigs 1st yr	190	16	4750	26	2466	28	291	21
2nd yr	210	18	5416	26	3500	24	1125	55
3rd yr	180	10	5000	23	3166	20	503	50
Bark Upper	200	18	6166	21	2900	31	608	46
Middle	175	(all)	6750	20	2050	19	403	34
Lower	121	30	5357	17	1857	15	241	28
(Cannon 1964)[L]			1900		2080		6900	

	Element							
	Zinc		Lead		Copper		Nickel	
	Mean	Coeff.	Mean	Coeff.	Mean	Coeff.	Mean	Coeff.
Leaf Blades	1283	36	38	(2)	1360	48	96	10
Petioles	1600	32	38	(2)	2533	15	-	-
Twigs 1st yr	900	39	56	22	1933	35	-	-
2nd yr	2116	41	88	36	2833	37	-	-
3rd yr	1250	29	78	41	1950	27	-	-
Bark Upper	1700	35	46	40	433	38	-	-
Middle	1016	32	38	(2)	358	50	-	-
Lower	897	34	65	42	1303	67	-	-
(Cannon 1964)[L]	1130		75		250		55	

x parts per million metal in ash

* coefficient of variation

*** indium is added to unknowns as reference standard during electrode preparation

L average minor element content expressed as parts per million in ash of deciduous trees

(2) element detected in only two samples out of fifteen, note coefficient of variation not calculated

into the spark was selected to obtain some of the advantages of the extreme sensitivity of the copper spark method together with an increase in total weight of sample used. A sugar solution was used to bind the sample on to the electrode because it attached the sample to the electrode without flaking off in the discharge. Considerable development work was required before the optimum setting of all the different parameters involved in the excitation and recording of spectra was discovered.

Using this technique it is possible to determine the twelve elements in samples of ash between the quoted ranges in concentration as read off from a synthetic plant ash base master film (see Appendix CL).

Table C 2

The scope of the Scan Spectrochemical method

Element	Wavelength of line used Å	Concentration From To (ppm)	Normal level of element in plant ash (Cannon 1964) Deciduous (ppm)	Coniferous (ppm)
1) Lead	2833.1	37.5 - 10,000	50	75
2) Molybdenum	3170.3	7.5 - 10,000	7	5
3) Indium*	3256.1	5.0 - 10,000	(added level 750)	
4) Copper	3247.5	1.0 - 10,000	250	130
5) Zinc	3345.6	75.0 - 10,000	2000	1130
6) Titanium	3349.0	7.5 - 10,000	290	400
7) Silver	3382.9	1.0 - 10,000	1.6	Below 1
8) Nickel	3414.8	75.0 - 10,000	85	55
9) Cobalt	3453.5	7.5 - 10,000	8	7
10) Manganese	4034.5	17.5 - 10,000	6900	6100
11) Strontium	4077.7	1.0 - 10,000	2080	2500
12) Chromium	4254.3	7.5 - 10,000	6	9
13) Barium	4554.0	7.5 - 10,000	1900	1300

* Indium added in sugar solution as a reference standard.

The data in Table C 2 indicated that our scan spectrograph method should be good for titanium, copper, zinc, manganese, strontium, and barium; satisfactory for lead, silver, and nickel; and probably poor for molybdenum, chromium, and cobalt. Experience has shown that these predictions were accurate. For example, in the case of the Silvermine Pilot Project when ash of nine kinds of plant sample (i.e., 3 barks, 3 twigs and 3 needles from each tree) were involved it was possible to plot results for lead, zinc, copper, nickel, barium, strontium, manganese, titanium, and silver (see Section G, Part II). The results for molybdenum, cobalt, and chromium were unsatisfactory for large scale display.

A check on the precision of the spectrograph method is made by measurement of the apparent concentration of the indium reference standard, the same amount of which is added to each unknown. An example of this procedure is given in Table C 1 where the results of analysis of eight Batches of different materials, collected within the same Site Set during the Petawawa Special Project, are given (see also Section J, Part III). The mean and coefficient of variation of results for eight elements determined in each material are listed together with the results obtained for indium, which was added as the reference standard just before the analysis (and does not include errors involved in collection or processing of samples). The indium results for all Batches except the lower bark are considered satisfactory because the coefficient of variation is less than 20 per cent and the mean values are within 25 per cent of each other. In the case of the lower bark the mean for indium is distinctly low and the coefficient of variation above 20 per cent. Such results for the reference standard are unacceptable and would result in a duplicate film being made of the Batch in question.

A further example of the precision of the analytical method is shown in Table C 3 where the results for the coefficient of variation for the indium reference standard, in three Batches of each of nine different ash types, are given. With the exception of the results obtained for humus, ash, and the three exceptions explained at the end of the table, all coefficients of variation are below 20 per cent which is a reasonable limit for a semi-quantitative method of this kind.

Because the coefficient of variation for the indium reference standard is consistently less than that for other elements (see Table C 1) it is considered that the precision of the rapid scan method is adequate for research of the type described in Parts II and III. There is, however, a need for more precise analyses of samples for specific elements which will be fulfilled when an atomic absorption apparatus is included in the trailer laboratory equipment.

A third criterion for judging the performance of an analytical method is accuracy. In order to obtain maximum sensitivity from the spectrograph method no buffer was added to samples to reduce matrix effects during the preparation of the electrodes. This omission was justified because it was believed that variations in the relative proportions of the major elements (i.e., those which are responsible for matrix effects) in ashes of the same organ of the same Batch of samples would be relatively insignificant although variations in the major element content of ashes of different organs of the same plants might cause a consistent bias in results. Experience has shown that these suppositions are valid. For example, in Figure C 4 results obtained by the rapid scan spectrograph method and the colorimetric method are compared in the case of four elements determined in three different organs of the same plants. On a graph of this kind, points lie on the line at 45 degrees passing through the origin if values obtained by both analytical

Table C 3

The coefficient of variation for the indium reference standard in twenty-seven Batches of samples of vegetable ash included in the Silvermine Pilot Project, 1966.

Material	Batch Number*	Coefficient of variation for indium reference standard
Current growth	400169	15%
	400269	11%
	400369	9%
Second year needles	400165	15%
	400265	15%
	400365	9%
Third year needles	400163	29%[a]
	400263	13%
	400363	19%
Second year twigs	400166	13%
	400266	14%
	400366	15%
Third year twigs	400164	14%
	400264	15%
	400364	15%
Upper bark	400162	14%
	400262	14%
	400362	7%
Middle bark	400161	18%
	400261	33%[b]
	400361	11%
Lower bark	400160	9%
	400260	41%[c]
	400360	21%
Humus	400138	38%
	400238	40%
	400338	25%

* This is an example of the numbering system described in C-3 (Project = 40, Site Sets 01, 02, 03, and Batches 69, 65, 63, 66, 64, 62, 61, 60 and 38).

[a] One needle sample with a very high mean indium value (would be repeated).

[b] One very low value (single value would be repeated).

[c] One very high value (single value would be repeated).

methods are identical; if there is bias in one of the sets of results, the points are offset from the line. In Figure C 4 little bias is shown in the case of zinc; a slight positive bias occurred in the case of the lead spectrograph results which becomes more pronounced in the case of nickel and copper. It is of interest that the bias for a given element in the three plant organs is very similar. Clearly, it is desirable to control the bias of the spectrographic results by making a single accuracy check on a sample from each Batch of samples, although this is not needed for interpretation of the results in the field laboratory. The combination of atomic absorption and spectrograph methods mentioned above should permit this checking. This will allow both accurate and precise semi-quantitative results to appear on preliminary scan sheets (see below).

A second cause of bias in the spectrochemical results follows from the way in which the master film is prepared. The standards from which the film is prepared consist of a synthetic plant ash base to which known amounts of a mixture of a large number of elements at the same concentration level (i.e., element as parts per million) has been added. The base was prepared by a technique especially developed by Farmer (1955) to simulate plant ash material. In theory it would be desirable to prepare a master film for each type of ash analyzed but in practice this is not possible and one master film is used for all plant and humus ash analyses. Thus a bias in the results for specific elements may be caused by the fact that the relative proportions of the major elements in the standards is not the same as in the unknowns, or that the chemical compounds of the minor elements added to the standards may not be the same as that in the unknowns. In spite of these limitations it is believed that the rapid scan spectrograph method is adequate for the provision of a first approximation of the variations in minor element content in vegetable ash samples.

Bearing in mind the limitations just discussed, the rapid scan spectrograph method has adequately fulfilled the numerous requirements described above, and together with the atomic absorption methods which are planned should provide suitable analytical facilities for landscape biogeochemistry.

(3) Other methods. The rapid scan spectrograph method jus described was originally set up as a first stage towards a more effective analytical system involving a chemical preconcentration of minor elements in plant ash prior to the spectrochemical analysis. Experimental work along these lines by Carter (1965) showed that it was feasible to use the chemical preconcentration method described by Mitchell and Scott (1948) in the trailer laboratory and that if a full time qualified chemist was available, 30 samples could be passed through this procedure per day. The advantages of this stage in the analytical system are: (1) the preconcentration step increases the sensitivity of the spectrograph method so that more elements can be determined at one time, (2) the preconcentration procedure almost eliminates matrix problems because trace elements derived from any kind of ash type are concentrated in a matrix (iron and aluminium) of the same composition, (3) the

preconcentration step enables the standards which are used for the standard film to be prepared by exactly the same technique as the unknowns (i.e., they are in exactly the same matrix), and (4) this particular preconcentration method is suitable for use in a trailer laboratory because it does not involve the use of organic solvents or large amounts of strong acids or alkalis.

This method was not adopted because it is not possible to use it on a routine basis without supervision by a qualified chemist, and experience has shown that several months are required to train an operator to produce uniformly reliable results. This rules out the use of summer students to operate this method. With the advent of atomic absorption methods research with this method of preconcentration was discontinued.

C 7 Methods of mechanical data processing

The rapid scan spectrograph method will produce results for 30 samples per day, with 13 individual element results on each sample on a continuing basis throughout the summer. Thus 2000 individual element results may be obtained each week for perhaps 10 weeks during a summer when the laboratory is in full operation. One advantage of the Site Set and Batch numbering system is the listing of results of spectrochemical analysis in the order in which they are required for plotting and interpretation. The next problem was to design data record sheets which could be used both in the spectrograph laboratory and in the computer centre where the data would be processed. This problem was solved in two stages. The printed sheet expected from the computer was made in mockup form by using a large typewriter, and later a computer program was written to produce the required printout page for each element (see Appendix CL). After the program was written, special data input sheets were designed, which could be used for recording data in the trailer laboratory and which were suitable for punching the data on cards at the computer centre.

Examples of the data sheet and the output for different kinds of data are shown in Appendix CL. The advantages of this output for different kinds of data are numerous. The data need be recorded only once in the laboratory and need not be touched again until the results are printed. The output shows the data for a single element within a Batch arrayed on a page on the corner of which the Site Set and Batch number are printed for quick reference. The computer program includes calculations for individual results on an oven dry weight basis and calculations for the total, mean, sum of the squares, standard deviation, coefficient of variation, and median for results (on both an ash and an oven dry weight basis) that are printed below the data array. A list of the spectrograph standard intervals and a histogram showing the results for a maximum of fifteen unknowns within a Batch is also printed at the left side of the output sheet. The result for the standard sample appears to the right of the Batch on the histogram and is not included in the calculations. An advantage of the histogram is that it may be quickly traced and

Table C 4

Concentration intervals for results of the Scan Spectrochemical method used on the computer output.

Master film standard*		Intermediate value	
(1)	10,000 ppm		
		7,500	ppm
(2)	5,000 ppm		
		3,750	ppm
(3)	2,500 ppm		
		1,750	ppm
(4)	1,000 ppm		
		750	ppm
(5)	500 ppm		
		375	ppm
(6)	250 ppm		
		175	ppm
(7)	100 ppm		
		75	ppm
(8)	50 ppm		
		37.5	ppm
(9)	25 ppm		
		17.5	ppm
(10)	10 ppm		
		7.5	ppm
(11)	5 ppm		
		3.8	ppm
(12)	2.5 ppm		
		1.8	ppm
(13)	1 ppm		
(14)	Base		

* Standards and base without addition of minor elements appear in order (1) to (14) on spectrograph film.

included on a scan sheet showing all results obtained from a Site Set (see below). A limitation of the method at present is that only specific intervals for the content of an element within a sample can be used (see Table C 4). The use of this simple mechanical method for plotting results has made a significant contribution to the effectiveness of the biogeochemical research program. The method is rapid; results from the 540 samples included in the 1966 field program were processed by the computer in 47 1/2 minutes!

C 8 Methods of preparation of results for interpretation

Originally, the objective of the scan spectrograph method was to produce rapidly multi-element data on many different kinds of soil and plant material collected from Site Sets located in landscapes of special interest. Experience showed that it is desirable to plot data on an oven dry rather than on an ash weight basis. To display data to best advantage for purposes of comparison Scan Sheets of the type shown on Table C 5 were designed.

The advantage of a Scan Sheet of this type is that it brings together results from all elements determined in all materials examined in a form that facilitates interpretation.

Table C 5

Layout of a Scan Sheet for vegetation and humus data

Material sampled	Elements determined												
	Pb	Mo	Cu	Zn	Ti	Ag	Ni	Co	Mn	Sr	Cr	Ba	(In)[1]
Current needles	X[2]	X	X	X	X	X	X	X	X	X	X	X	X
Second year needles	X	X	X	X	X	X	X	X	X	X	X	X	X
Third year needles	X	X	X	X	X	X	X	X	X	X	X	X	X
Current twigs	X	X	X	X	X	X	X	X	X	X	X	X	X
Second year twigs	X	X	X	X	X	X	X	X	X	X	X	X	X
Third year twigs	X	X	X	X	X	X	X	X	X	X	X	X	X
Upper bark	X	X	X	X	X	X	X	X	X	X	X	X	X
Middle bark	X	X	X	X	X	X	X	X	X	X	X	X	X
Lower bark	X	X	X	X	X	X	X	X	X	X	X	X	X
Humus	X	X	X	X	X	X	X	X	X	X	X	X	X

[1] Indium reference standard.

[2] X = a small histogram showing the results for 15 (or less) samples for a single element on an oven dry weight or an ash weight basis.

Many of the results reported in Parts II and III are laid out on scan sheets of various kinds. For example, in Section E, Part II the scan sheet was used to display the results obtained for four elements determined in a single Batch of mineral soil samples collected from each of nine undisturbed mineral deposits which were included in Visits. A similar approach was adopted for the Mer Bleue Project with the exception that the scan sheet involved results from samples obtained from drill holes within the peat.

C 9 Summary and Conclusions

The progress which has been made towards setting up an integrated methods system for collecting sets of morphological and chemical data on the geochemistry of Canadian landscapes at an "instant in time" has been described under seven headings.

Although a good foundation has been laid, several problems still remain to be solved before the methods system is completed; for example, an atomic absorption apparatus is needed to complete the analytical facility. As more experience is gained with all parts of the methods system, improvements to details of techniques will occasionally be made. For this reason

most of the operational procedures (described in Appendices CB to CK) are somewhat provisional; however the remaining seven sections of this report are based on the techniques described here.

It is both feasible and practical to obtain similar sets of morphological and chemical data from prospecting prisms located in different kinds of Canadian landscapes on the scale dictated by Pilot Projects at an instant in time by the methods described.

C 10 References

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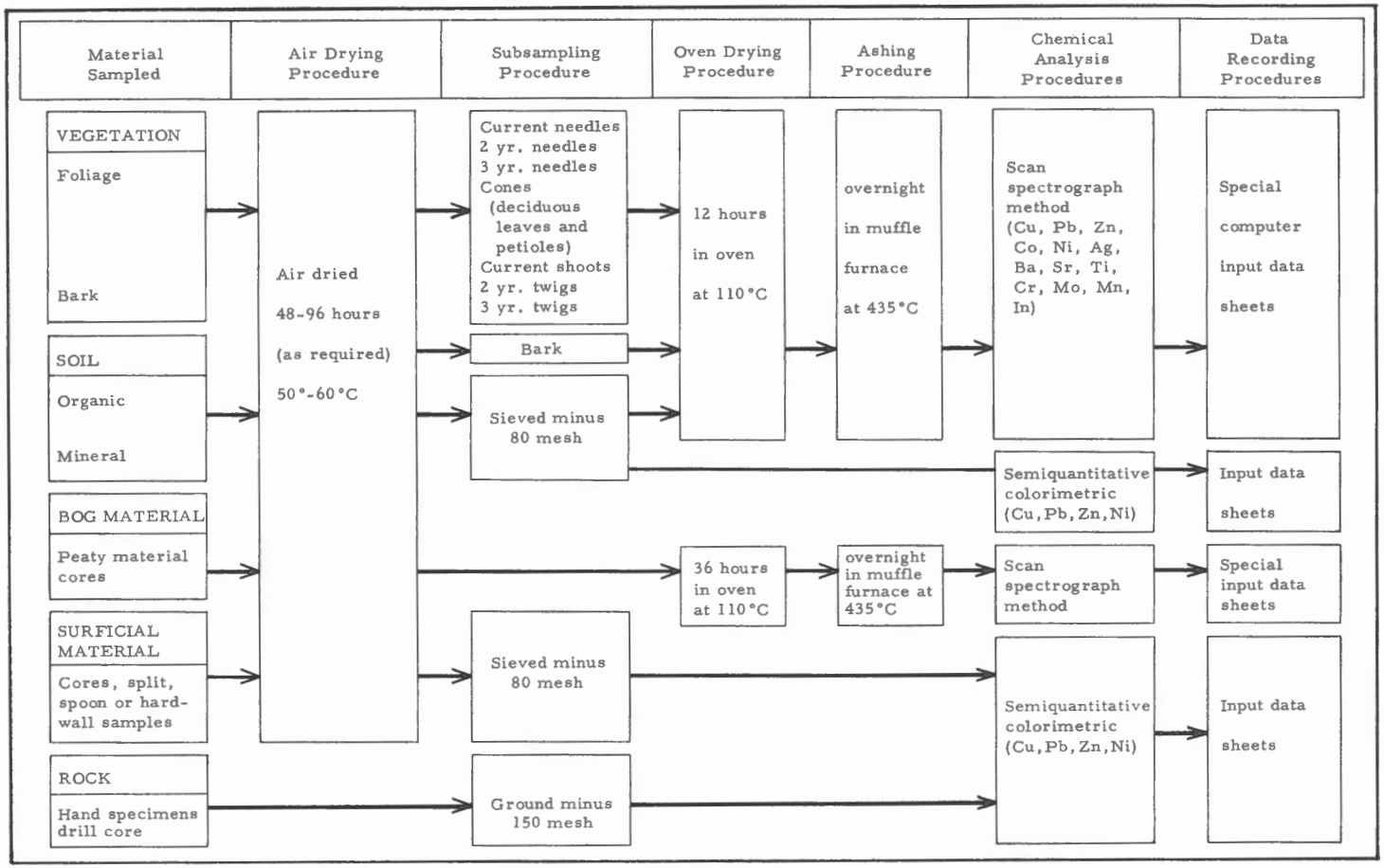


Figure C 2. Flow sheet showing the general procedure for collection and treatment of samples in biogeochemical research.

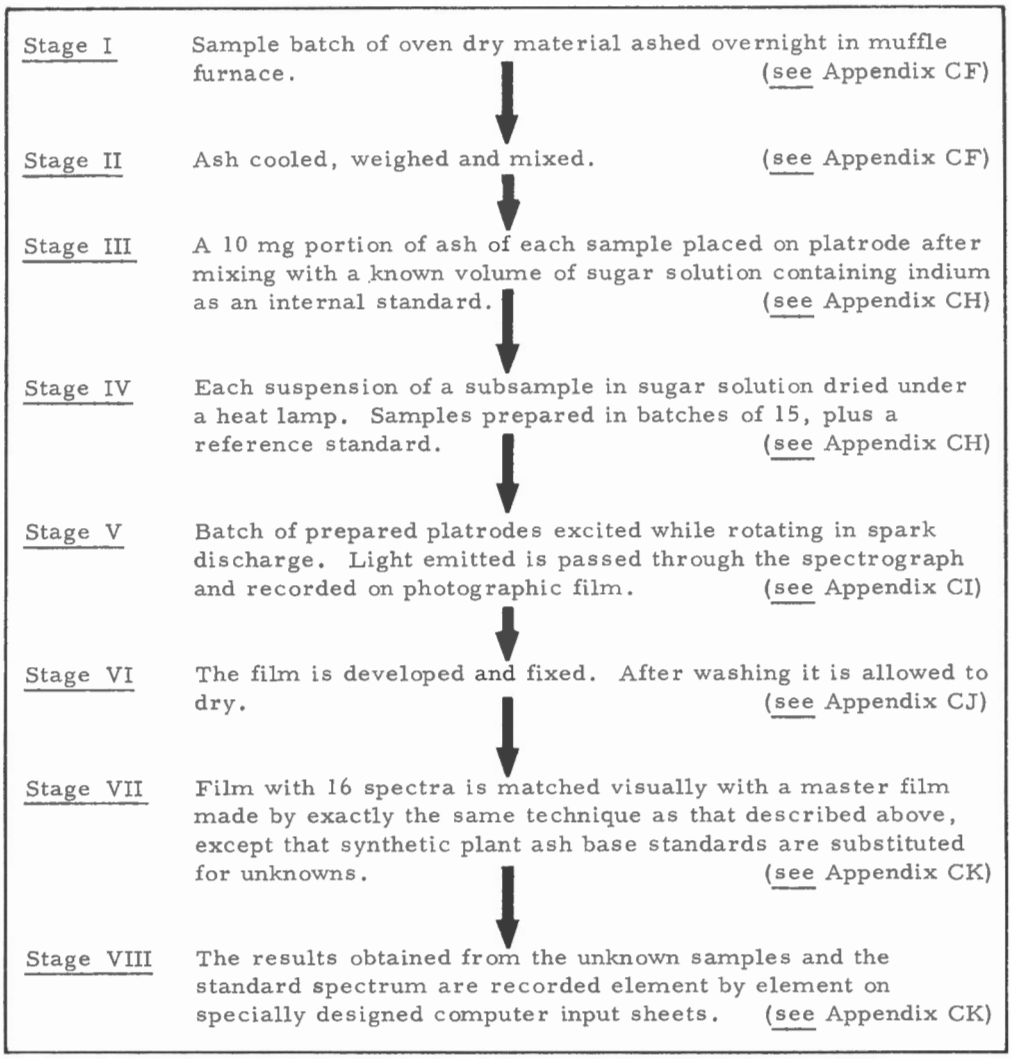
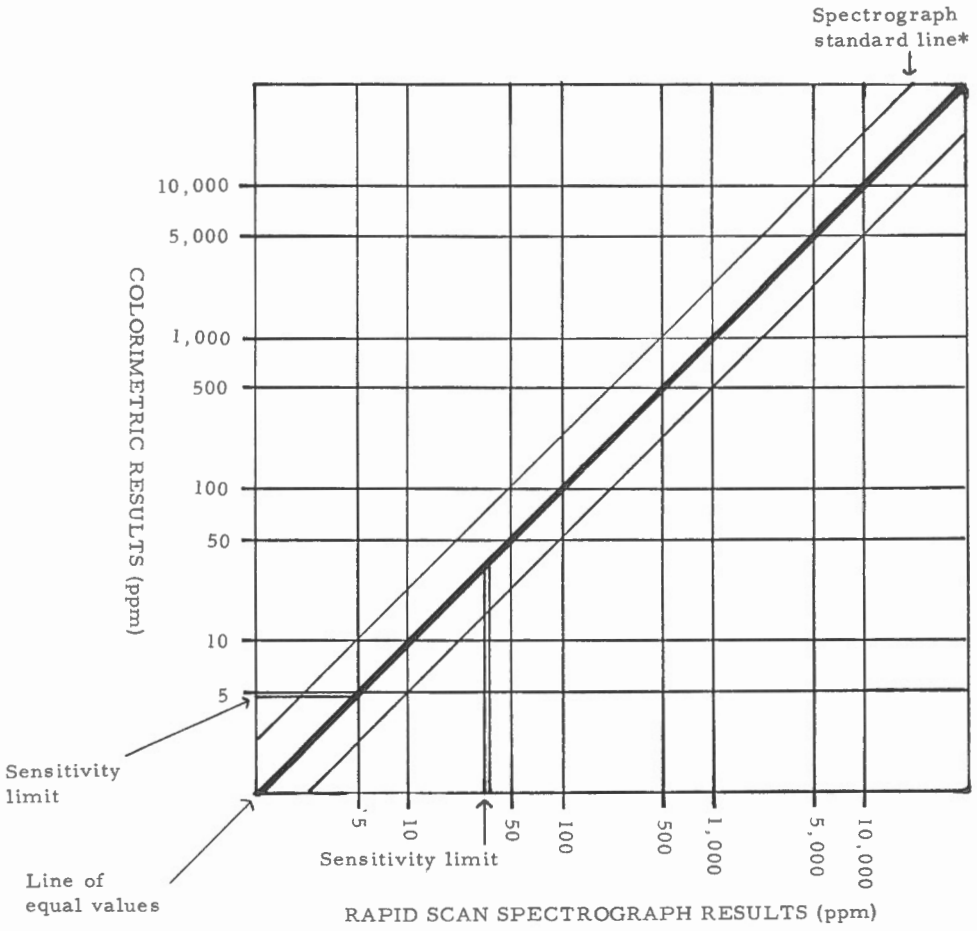


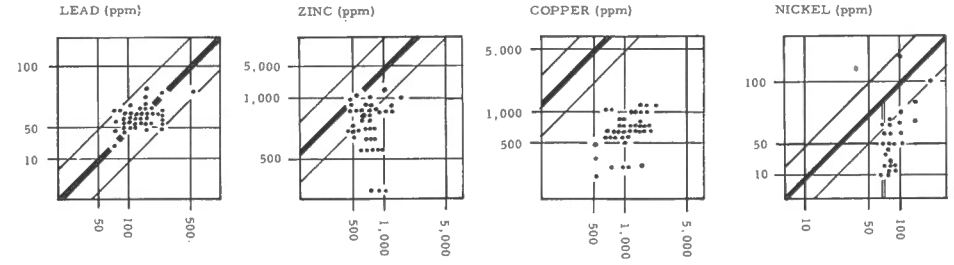
Figure C 3. Flow sheet showing the sequence of operations involved in the analysis of a batch of samples of plant material by the Scan Spectrochemical method as set up in the Spectrograph Trailer Laboratory unit.



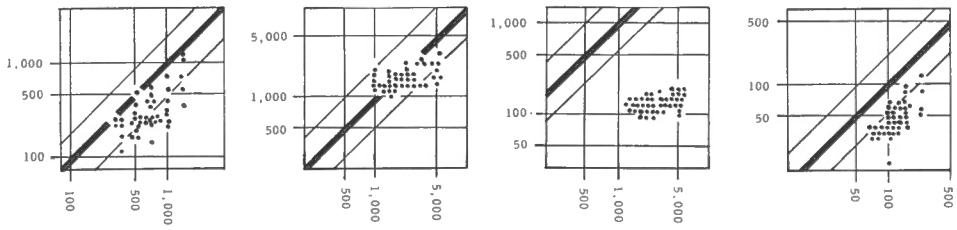
* If the central line represents the content of an element in the spectrograph master film the parallel lines represent the next highest and next lowest standard values. During analysis of unknowns concentration levels halfway between standards may be estimated and recorded.

Figure C4. Data on the accuracy of the Rapid Scan Spectrochemical method.

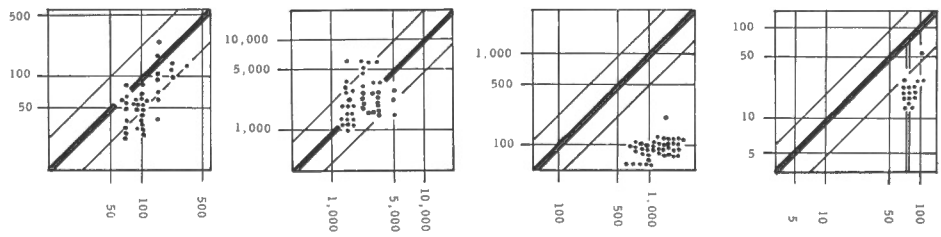
NEEDLES

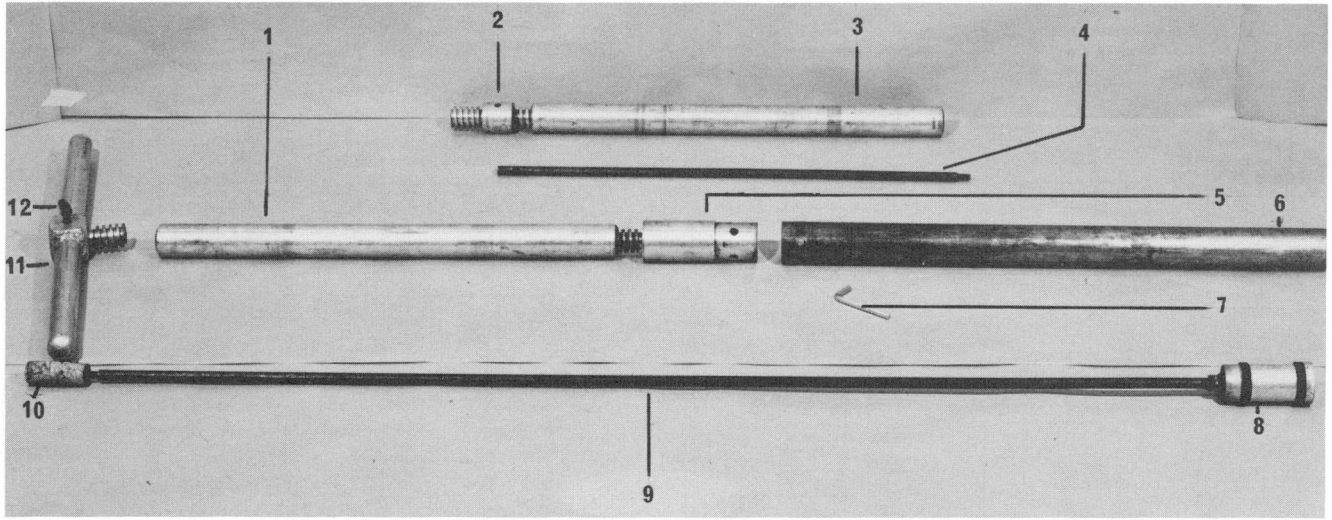


TWIGS



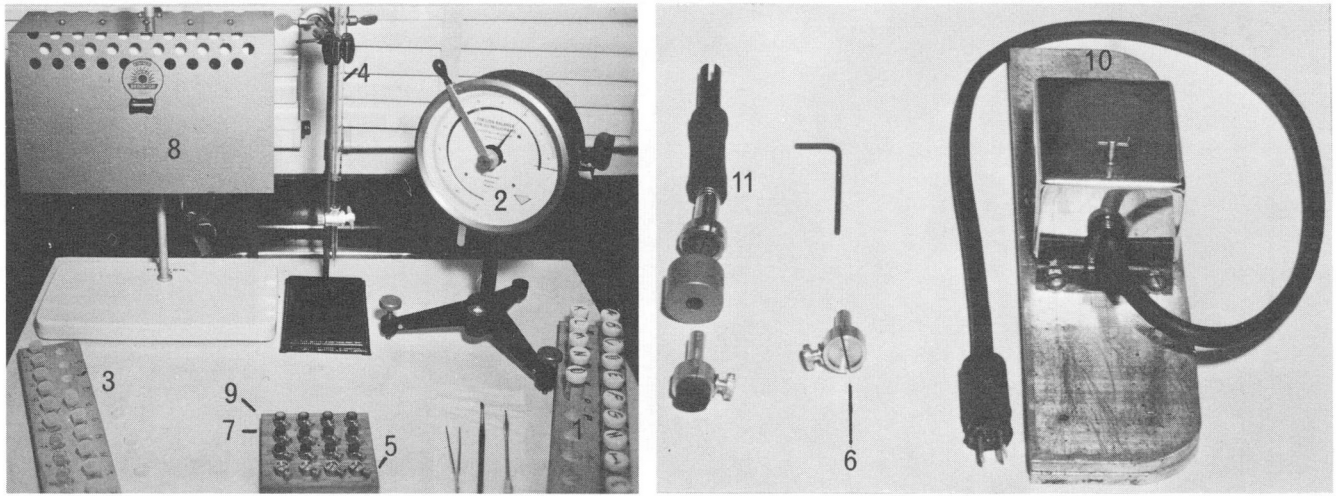
BARK





1) Shelby tube body
2) Extension connector
3) Body extension
4) Piston rod extension
5) Adaptor
6) Shelby tube
7) A Key wrench
8) Piston with leather rings
9) Piston rod
10) Piston rod handle
11) Shelby Tube body handle
12) Thumb screw

Plate C 1 A 2 inch Shelby Tube Piston Type Sampler for Bog material



1) 10 ml plastic vials; containing mixed ash
2) 50 mg torsion balance
3) 2 ml plastic vials: weighed ash subsample
4) Burette for dispensing sugar solution containing internal standard
5) Wooden plate
6) Aluminium platrode holder
7) Impregnated platrodes
8) Heat lamp
9) Platrodes ready for excitation
10) Platrode rotation electric motor (10 r.p.m.)
11) Platrode holder drive assembly

(Note The platrode holder stem fits into a small metal heat sink which is firmly attached to the drive assembly by means of an allen set screw).

Plate C 2 Apparatus required for spectrograph electrode preparation and excitation

APPENDIX CA

Shallow seismic investigations used to determine depth of surficial material

by G. D. Hobson

Seismic exploration depends fundamentally upon the propagation of seismic waves within elastic media. Elastic waves generated by explosions or man made sources travel downward in all directions following the physical laws of optical theory. These waves are reflected and refracted at interfaces at depth and return to the surface of the earth. The interpretation of recorded seismic data consists of determining the velocity of the propagation of these elastic waves and analyzing the refraction and reflection phenomena at the interface or boundaries between rock layers that are characterized by different acoustic properties. The refraction phenomena have been of principal interest in all investigations associated with biogeochemical studies over mineralized areas.

The quantity observed and recorded at each location is the time interval between the initiation of the elastic wave by an explosion or hammer blow and the first disturbance of the ground as detected by a seismometer at a known distance from the source of energy. The proportion of the energy refracted is dependent on the difference in propagation velocities on the opposite sides of the acoustic boundary. The basis of the refraction technique is Snell's Law and Hugen's Principle, and its successful application is dependent upon the increase in velocity with depth.

A graph of the time interval required for the energy to travel from source to detector plotted against the distance of the source from the detector permits a determination of the velocity of the energy wave through the various media penetrated. Standard formulae are then used to determine the thickness of and, therefore, the depth to the various refractors.

A Model F.S.-2 portable hammer seismograph made by Huntec Limited has been used throughout all seismic surveys associated with the biogeochemical investigations. All seismic events are recorded on dry electro-sensitive paper by a sweeping electric stylus.

The purpose of conducting the seismic survey over a landscape being investigated is to determine the thickness of the different Pleistocene strata and the total depth to bedrock. Because most materials exhibit a seismic velocity, or range of velocities that are peculiar or characteristic it is generally possible to identify the various overburden materials by means of the seismic velocity associated with them. For instance, dry and wet materials

can generally be distinguished as can sand, clay and till. Sometimes seismic velocities can be correlated with bedrock lithology to distinguish contacts in the bedrock. The seismic method is generally considered to yield depth determinations with an accuracy of plus or minus 10 per cent of the true value.

Reference

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1964: Geotechnical investigations Red River floodway, Winnipeg, Manitoba; Geol. Surv. Can., Paper 64-18.

APPENDIX CB

Operational procedure for the collection of bog material

Apparatus:
- Two inch Shelby tube sampler (illustrated on Plate C 1)
- Sheets of polyethylene 30" x 30"
- Strong string
- Magic markers
- Allen wrench to remove Shelby tube as required

Procedure:
(1) Sample site marked with coloured plastic tape and the sampler is assembled before sampling.
(2) Sample cores of peaty material are collected by inserting the sampler into the ground, with the thumb screw loose on the body handle, until the Shelby tube is full. The thumb screw is then locked and the sampler withdrawn from the ground.
(3) The sampler is laid horizontally on the ground, the thumb screw is released, and the sample core extruded by pushing on the piston rod handle. (It may be necessary to clean out the tube between samples.)
(4) The core sample is extruded onto a plastic sheet which is then rolled up and tied at each end to form a sausage.
(5) The sample number is written on the outside of the sausage with a magic marker.

Remarks:
(1) The sampler works well except in very wet peat or in bog material which contains large woody fragments.
(2) When the hole exceeds 4 1/2 feet in depth, it is necessary to add on the extension section.

APPENDIX CC

Operational procedure for the collection of soil material

Apparatus:
- Mattock, or trenching tool
- Shovel
- Double thickness lined brown paper bags, 5 lb size sealed with metal strip
- Magic marker

Procedure:
(1) A sample site for the pit is located close to the tree being sampled.
(2) Litter is removed from the humus material.
(3) Enough humus material is removed with the shovel or by hand to fill the sample bag.
(4) The bag is sealed, and the sample number written on the outside on both sides with magic marker.
(5) A small pit is dug with the mattock to the lowest soil horizon to be sampled, and the soil profile exposed as a vertical face.
(6) Successive horizons are sampled from the bottom of the vertical face to the top. Extreme care is taken not to mix material from the different horizons.
(7) The samples are put in bags and sealed as described above.

Remarks:
(1) When the humus is less than 1 inch thick it is difficult to avoid contamination of the organic material with mineral matter.
(2) The soil pit is usually dug after the tree is felled to avoid interference between the soil and plant sampling operations.

APPENDIX CD

Operational procedure for collection of plant material

Apparatus:
- Chain saw (power) and accessories
- Axe
- Pruning shears (good quality steel)
- Pocket knife
- Magic marker
- Cloth tape (100-foot)
- Double thickness lined brown paper sample bags, 5 lb size and 10 lb size
- Triple thickness 50 lb paper sacs for transport of samples to laboratory

Procedure:

(1) Trees to be sampled within a Site Set are selected for uniformity in age, height, foliage, drainage conditions, and in relation to other components of the plant community.
(2) Selected trees are blazed and the Site Set sample point letter marked on the blaze with grease pencil.
(3) Trees are felled and the maximum height measured.
(4) Tree trunks are marked at one-third and two-third levels of the total tree height.
(5) The trunk is cut at breast height (4 feet 6 inches above ground level).
(6) Several narrow discs are cut from the trunk at breast height, and at each of the two marked levels.
(7) One of the discs from each level is trimmed with an axe to provide a cross-section of the tree for growth ring dating. Where possible, discs are selected that are free from knots or rot.
(8) The bark is stripped from the remaining discs by knife or axe and placed in bags to provide bark samples from the three levels of the trunk.
(9) Samples of current growth, second, and third year twigs of branches and needles of conifers are taken from the lead shoot and top whorl (or whorls) and put into separate bags. For deciduous trees, all foliage is put into the same bags and subsampled after drying.

Remarks:

(1) During the collection of all types of plant material all long twigs are broken to prevent ripping of the sample bag.
(2) At each station the samples taken are checked against a master list before putting them into a 50 lb paper sack for transport to the trailer laboratory.

APPENDIX CE

Operational procedure for sample drying and subsampling
(For details of the trailer laboratories see Section B)

Apparatus:

Drying cabinets (in the sample preparation trailer)
Forced draught drying oven
Pruning shears
Stainless steel sieves (10 mesh and 80 mesh) with cleaning equipment
Plastic vials: 10 ml and 60 ml size
Single pan Mettler balance

Procedure: Bog Material

(1) Peat samples are opened and laid out in trays in drying cabinet and dried overnight, at approximately 15°C. The material of highest moisture content is placed on trays at the bottom of the cabinet because it is cooler at the top of the cabinet than at the bottom.

(2) Dry peat samples are subsampled by taking a channel sample from the length of each core. The subsample is dried overnight in the oven at 110°C.

(3) Ten grams of the oven dry material when cold is weighed, and stored in 60 ml plastic vials.

Soil Samples

(1) Sealed bags of humus and soil samples are laid out on the drying cabinet trays until dry.

(2) After drying humus material the bags are opened outside the trailer and after removal of large sticks and stones, the samples are passed through a 10 mesh followed by an 80 mesh sieve. The minus 10 plus 80 mesh material and the minus 80 mesh material are retained and stored in 60 ml plastic vials.

(3) Mineral soil samples are crushed in the sealed bags before opening them outside the trailer. The crushed material is passed through an 80 mesh sieve and the minus 80 mesh fraction retained and stored in 60 ml plastic vials.

Plant samples

(1) All sealed bags containing freshly collected material are cabinet dried as required.

(2) Subsamples of coniferous foliage samples (see Appendix CD), and samples of deciduous trees, and all bark samples are dried in the oven at 80°C overnight.

(3) Needles are separated from coniferous twigs and a subsample of each material is weighed and stored in 10 ml vials. The remainder of the dried material is stored in 60 ml vials.

(4) Oven dry bark material is broken up by hand or cut with shears, then weighed out into 10 ml vials. The remainder is retained for storage.

Remarks: (1) To facilitate dry ashing and subsampling all plant material is broken up into fragments 1/4" by 1/2" after oven drying. Subsamples are carefully selected to be representative of the whole sample from which they were drawn.

APPENDIX CF

Operational procedure for dry ashing vegetable material

Apparatus: Pyrex beakers: various sizes
Plastic vials

Muffle furnace (see Appendix BB) with controller
Analytical balance
Plastic beads
Weighing accessories

Procedure:
(1) The weighed subsamples of oven dry material are placed in a weighed Pyrex beaker of suitable size.
(2) Beakers containing samples of like material (i.e., Batches obtained from the same Site Set) are placed in the furnace.
(3) The furnace and controller are switched on and set for a time temperature controlled ashing cycle (i.e., overnight).
(4) The beakers are withdrawn from the furnace and the ash is allowed to cool to room temperature.
(5) Weight of the ash is determined and the percentage of ash in the samples is calculated.
(6) The ash is transferred to a 10 ml plastic vial mixed by adding a 3/8" plastic bead to the vial and shaking by hand.

Remarks:
(1) After operation (6) the ash is almost directly used for electrode preparation. Experience showed that delay results in poor results from the spectrograph method.
(2) The muffle furnace is too small for routine ashing of samples in 50 ml beakers. To speed this up 2 gram samples are at present ashed in 10 ml beakers. This is satisfactory, but does not yield enough ash for a repeat spectrograph analysis on a sample of ash.

APPENDIX CG

Operational procedure for colorimetric analysis of soils and vegetable ashes

These analyses were carried out in the Chemical Laboratory of the Geochemical Section of the Geological Survey of Canada under the direction of J. J. Lynch.

Procedure: (Summary only)
(1) One hundred mg of minus 80 mesh soil material (or vegetable ash) is placed in a platinum dish and treated with 5 ml of 48% $HClO_4$ and allowed to digest overnight.
(2) The mixture is evaporated to fumes of $HClO_4$ and then the sides of the dish washed with metal free water. Fuming and washing are repeated four more times, before evaporating to dryness.
(3) Residue dissolved in 5 ml of 1 N HCl and diluted to 10 ml with water.

(4) Aliquots of this solution are removed as required for the zinc, lead, copper and nickel tests. These elements were determined by methods due to Gilbert (1959) in the case of copper, lead and zinc and Stanton and Coope (1962) in the case of nickel.

(5) The performance of these methods in the normal working range (20-1000 ppm) is within 25 per cent of the total amount of metal present.

(6) In some samples molybdenum was determined exactly according to North (1956). This method involves a fusion with modified Na_2CO_3 flux, leaching with water, and a final determination of molybdenum with zinc dithiol. The performance of this method is similar to that of the other four.

References

Gilbert, M. A.
1959: Field and Laboratory methods used by the Geological Survey of Canada in Geochemical Surveys No. 1 Laboratory methods for determining copper, zinc and lead; Geol. Surv. Can., Paper 59-3.

North, A. A.
1956: Geochemical field methods for the determination of tungsten and molybdenum in soils; The analyst, vol. 81, p. 660.

Stanton, R. E. and Coope, J. A.
1958: Modified field test for determination of small amounts of nickel in soils and rocks; Bull. Inst. Mining Met., London, vol. 623, p. 9.

APPENDIX CH

Operational procedure for spectrograph electrode preparation

Apparatus: (The apparatus described here is illustrated in Pl. C 2)
Graphite platrodes (Grade AGKSP - (L - 4078)
Aluminium platrode holders. These were specially fabricated to our specifications. The upper surface is recessed and receives the platrode which is locked in place by means of a thumb screw. On the bottom is a one inch stem that fits into a socket on the rotating drive mechanism in the arc stand. The stems also fit into drilled holes in

wooden plates so that 16 platrodes can be handled at one time.
50 mg torsion balance
Small mohair brush for removing ash from weighing paper
Tweezers
Square wooden plates to hold 16 platrode holders
Infra-red heat lamp
5% sugar solution
5% sugar solution containing 13.3 micrograms indium (as nitrate) per ml.
50 ml burette with three way stop cock, connected to reservoir of the indium sugar solution.
2 ml plastic vials

Procedure:

(1) A Batch of sixteen platrodes are inserted into their holders and the stem of the holder inserted into the appropriate hole in the wooden plate.

(2) Three drops of 5 per cent sugar solution is placed on the top of each platrode.

(3) The plate containing the platrodes is put under the infra-red heat lamp.

(4) As soon as the solution is dry on the surface of the platrode it is removed from the heat lamp as the impregnation of the porous upper surface of the platrode is complete. The platrodes are not impregnated properly if they remain under the heat lamps after the point of initial dryness. Care is needed in judging the time of removal of the electrodes from under the heat lamp because they do not all dry at the same rate.

(5) The platrode and holder are allowed to cool to room temperature.

(6) A 10 mg portion of ash is weighed out into a 2 ml vial.

(7) Three drops of the sugar indium solution are dropped into the vial.

(8) The ash/sugar solution mixture is rolled carefully around in the vial to mix well and take up all the ash in the liquid phase which does not wet the inside wall of the vial. The mixture is then dumped out onto the surface of a sugar impregnated graphite platrode.

(9) The loaded platrode is placed under the heat lamp and dried. When dry, the ash and sugar form a thin layer completely covering the platrode surface much like the icing on a cake.

(10) Steps (6), (7), (8) and (9) are repeated until all 16 platrodes are dried. These operations are carried out concurrently on a Batch of samples.

Remarks: (1) The first fifteen platrode holders are numbered A to Ø (this refers to the sample point numbers within a Site Set in which the Batch was collected). The P position is occupied by a known standard sample or a replicate of one of the unknowns.

(2) The temperature of the surface of the electrode is about 150°C when drying.

APPENDIX CI

Operational procedure for excitation of electrodes

Apparatus:

1.5 metre Wadsworth grating Jarrell-Ash spectrograph (see Appendix BB)
High voltage A.C. spark source unit
Voltage controller
Small 10 r.p.m. platform motor to rotate the platform and holder assembly (see Pl. C 2)
Two step optical filter 100% and 40% transmission
Graphite counter electrodes (Grade AGKSP)
Tweezers
Auxiliary extractor fan for high voltage source unit
Shutter timer circuit

Procedure: (1) The apparatus is prepared for exposure of a film by turning on the extractor fans in the source unit, and in the arc stand, by turning on the arc stand projection lamp and platform rotation motor and the timer circuit voltage controller and source unit. After the camera is racked into the required position, a platrode is placed in the holder and a flat ended graphite counter electrode is positioned 2 mm from the surface of the platrode. Excitation for 20 seconds with no prespark time commences under the following conditions:

Slit width: 15 microns
Slit height: 1.5 mm
Filter two steps used 100% and 40% transmission
Film 35 mm Kodak Spectrum Analysis No. 3 safety film

APPENDIX CJ

Operational procedure for processing spectrograph film

The processing of the spectrograph film follows the normal procedure; development (in Kodak D-19) for 4 minutes at 68°F; followed by 2 minutes in a stop bath; fixing for 30 seconds; washing and drying.

Some difficulty was found with the adjustment of the film tray water bath temperature. The solution to this problem is discussed in the text.

APPENDIX CK

Operational procedure for recording of chemical results

Apparatus: "Spectroline scanner" comparator densitometer
Standard Master Film (see text)
Input sheets for recording results (see Appendix CL)

Procedure:
(1) The film is removed from the drying spool and inserted into the upper holder of the densitometer. The Master Film is placed in the lower holder.
(2) Analysis lines as marked on the Master Film (see Table C 2) are matched with those on the unknown film by lining them up on the split frosted viewing plate of the comparator.
(3) The amount of an element in the unknown sample is estimated from comparison with the intensity of the lines on the Master Film. The result is recorded in parts per million as one of the concentration intervals selected (see Table C 4), and then recorded on a data sheet (see Appendix CL)

Remarks:
(1) No duplicate of the data sheet is made; but, the film with unknown spectra recorded upon it is stored in a sealed plastic vial for future use.

APPENDIX CL

Description of the method for printing biogeochemical results

To obtain the printed sheets shown in Figures CL 3 to CL 7 the following procedure is used:

(1) The data are recorded in the trailer laboratory (see Appendix CK).
(2) The data are punched on I. B. M. cards.
(3) The cards are processed by an electronic computer.

The data sheet used for recording results of spectrochemical analysis in the trailer laboratory is shown in Figure CL 1. This sheet, specially designed for the project, is printed partly in green and partly in black. Only the names of the chemical elements in each of the fifteen samples comprising a Batch of unknowns plus a standard (see Appendix CH), and the decimal points are printed in black. The operator of the comparator in the trailer laboratory annotates the chemical results in the appropriate columns. When the Batch identification (shown at the top of Fig. CL 1) and the chemical results are completed, the sheets are sent to the Computer Science Division of the Department of Energy, Mines and Resources at 588 Booth Street, Ottawa. Here, the key punch operators punch only the data in black thus all letters in green on the original data sheet are not punched. Finally, the cards are processed by Program C40901 (shown in Fig. CL 2) on the CDC 3100 computer. As illustrated in Figure CL 3, each set of data for a Batch has (1) an ash percentage card for samples A-Ø and for P., (2) any number of element cards, and (3) a synopsis card to indicate the end of the Batch. The last card of any input deck whether for one or more Batches is always a blank card to stop the processing of the entire job.

Each element card has the Batch identification with codes for operator, method, data of analysis, film number, year, project, Site Set and Batch in columns 1-16; for the element name, in columns 21-30; and either of the two type codes, in columns 39-40. If the element is analyzed, its type code is left blank in columns 39-40. This element card is always followed by two analysis cards which have PPM values for samples A-Ø and for the standard P. If the element is not analyzed, its type code is NR for NO RESULTS, and an arrow is drawn on the data sheet to inform the key punch operator that the two subsequent lines are to be omitted.

In the processing of an element which has a blank type code, the computer prints a histogram, an array of data, and a statistics table of the results on an ash as well as on an oven dry weight basis (Fig. CL 3) provided one or more of the samples A-Ø are not missing. If any of these samples is missing, the computer prints a histogram for P only (Fig. CL 4). If the element has the type code NR the program stores the element in the NO RESULT list to be printed after the computer reads the synopsis card

(Fig. CL 7). The synopsis card differs from the element card in that only the batch identification is punched.

In the case of the reference standard indium a different method of printing the histogram is used (Fig. CL 5). Here the letters are printed only once across the histogram and are not repeated below (Fig. CL 5).

In this program the histogram comprises forty-one ppm values ranging from 1,000,000.0 to 0.1. No other values can appear on the data sheets (see Section C and Table C 4). The data sheet in Figure CL 1 that is used to obtain the printed sheets shown in Figures CL 3 to CL 7 illustrates in detail the scope of the program.

C 40901

DR. J. A. FORTESCUE
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RESULT SHEET FROM BIOGEOCHEMICAL SPECTROGRAPHIC LABORATORY

Operator 02 | Method 05 | Date 38 | Film Number 01 | Year 66 | Project 50 | Site Set 01 | Batch 72

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
ASH %	3.98	5.01	4.85	4.96	5.23	4.78	4.79	4.50	5.01	4.75	4.92	5.09	5.21	4.83	4.78

Master/Metal	Card							
Master/Metal	0205380166500172		LEAD					
	A 50.	B 50.	C 175.	D 750.	E 5000.	F 3750.	G 5000.	H 5000.
	I 2500.	J 1000.	K 500.	L 500.	M 250.	N 100.	O 50.	P 50.
Master/Metal	0205380166500172		MØLYBDENUM					
	A 0.	B 0.	C 0.	D 0.	E 0.	F 0.	G 0.	H 0.
	I 0.	J 0.	K 0.	L 0.	M 0.	N 0.	O 0.	P 500.
Master/Metal	0205380166500172		INDIUM					
	A 750.	B 750.	C 750.	D 750.	E 750.	F 750.	G 750.	H 750.
	I 750.	J 750.	K 500.	L 750.	M 750.	N 750.	O 750.	P 750.
Master/Metal NEXT CARD	0205380166500172		CØPPER	NR				
	A 0.	B 0.	C 0.	D 0.	E 0.	F 0.	G 0.	H 0.
	I 0.	J 0.	K 0.	L 0.	M 0.	N 0.	O 0.	P 0.
Master/Metal	0205380166500172		ZINC					
	A 0.	B 0.	C 0.	D 0.	E 0.	F 0.	G 0.	H 0.
	I 0.	J 0.	K 0.	L 175.	M 0.	N 0.	O 0.	P 0.
Master/Metal NEXT CARD	0205380166500172		TITANIUM	NR				
	A 0.	B 0.	C 0.	D 0.	E 0.	F 0.	G 0.	H 0.
	I 0.	J 0.	K 0.	L 0.	M 0.	N 0.	O 0.	P 0.
Master/Metal	0205380166500172		SILVER					
	A 0.	B 0.	C 3.8	D 0.	E 0.	F 10.	G 0.	H 0.
	I 0.	J 0.	K 0.	L 0.	M 0.	N 0.	O 0.	P 0.
Master/Metal	0205380166500172		NICKEL					
	A 0.	B 0.	C 37.5	D 0.	E 0.	F 0.	G 175.	H 0.
	I 175.	J 0.	K 0.	L 0.	M 0.	N 0.	O 0.	P 0.
Master/Metal NEXT CARD	0205380166500172		CØBALT	NR				
	A 0.	B 0.	C 0.	D 0.	E 0.	F 0.	G 0.	H 0.
	I 0.	J 0.	K 0.	L 0.	M 0.	N 0.	O 0.	P 0.
Master/Metal	0205380166500172		~~MANGANESE~~					
	A .	B .	C .	D .	E .	F .	G .	H .
	I BLANK CARD	J .	K .	L .	M .	N .	O .	P .
Master/Metal			STRØNTIUM					
	A .	B .	C .	D .	E .	F .	G .	H .
	I .	J .	K .	L .	M .	N .	O .	P .
Master/Metal			CHRØMIUM					
	A .	B .	C .	D .	E .	F .	G .	H .
	I .	J .	K .	L .	M .	N .	O .	P .
Master/Metal			BARIUM					
	A .	B .	C .	D .	E .	F .	G .	H .
	I .	J .	K .	L .	M .	N .	O .	P .

Please insert blank card

Figure CL 1 Data Sheet used for recording results of Scan Spectrograph method.

Figure CL 2. Computer Program for processing data recorded on Scan Spectrograph Data Sheets.

```
                                    3200  FORTRAN    (2.1)            / /

      PROGRAM C40901
C  STATISTICS OF SPECTROGRAPHIC LAB RESULTS
C  PROGRAMMED BY MISS M.W. GRIEVE. DEPT. OF ENERGY, MINES AND RESOURCES.
C  COMPUTER SCIENCE DIVISION, OTTAWA.
C  LAST CARD OF INPUT MUST BE BLANK
C
      DIMENSION MET(5),ASH(15),P(16),VP(15),OD(15),VOD(15),OBS(15),
     1          ODOBS(15),S(41),IAY(42,16),NAME(30,5),IQ(30)
      COMMON/DATA/L(16)
      DATA(L=1RA,1RB,1RC,1RD,1RE,1RF,1RG,1RH,1RI,1RJ,1RK,1RL,1RM,1RN,1RO
     1,1RP)
      IRL=1R
      IRL2=2R
      NORSTS=2RNR
      IND=2RIN
C        STORE PPM NUMBERS FOR HISTOGRAM
C
      S(1)=1000000.
      S(2)= 750000.
      S(3)= 500000.
      S(4)= 375000.
      S(5)= 250000.
      S(6)= 175000.
      S(7)= 100000.
      S(8)=  75000.
      S(9)=  50000.
      S(10)= 37500.
      S(11)= 25000.
      S(12)= 17500.
      S(13)= 10000.
      S(14)=  7500.
      S(15)=  5000.
      S(16)=  3750.
      S(17)=  2500.
      S(18)=  1750.
      S(19)=  1000.
      S(20)=   750.
      S(21)=   500.
      S(22)=   375.
      S(23)=   250.
      S(24)=   175.
      S(25)=   100.
      S(26)=    75.
      S(27)=    50.
      S(28)=    37.5
      S(29)=    25.
      S(30)=    17.5
      S(31)=    10.0
      S(32)=     7.5
      S(33)=     5.0
      S(34)=     3.8
      S(35)=     2.5
      S(36)=     1.8
      S(37)=     1.0
      S(38)=      .8
      S(39)=      .5
      S(40)=      .3
      S(41)=      .1
C        READ ASH PERCENTAGE CARD FOR BATCH
C
```

```
      READ(60,1) (ASH(I),I=1,15)
    1 FORMAT(15F5.2)
C        INITIALIZE NO OF ELEMENTS, NO OF NO RESULTS FOR BATCH,
C                   SUM, SUM OF SQUARES FOR ASH AND OVEN DRY, AND
C                   ARRAY FOR EACH ELEMENT
C
      NOM=1
      IRN=1
   20 NOP=0
      ITABLE=2
      SP=0.
      SVSP=0.
      SOD=0.
      SVSOD=0.
      DO 21 M=1,42
      DO 21 I=1,16
   21 IAY(M,I)=IBL
C        READ ELEMENT CARD AND CHECK FOR LAST CARD OF BATCH
C
   19 READ(60,2) IOP,ME,ID,JFN,IYR,IPR,ISS,IB,MET,IQ(IRN)
    2 FORMAT(8I2,4X,5R2,8X,R2)
      IF (MET(2).EQ.IBL2) 62,61
   61 DO 999 I=1,5
  999 NAME(NOM,I)=MET(I)
      IF (IQ(IRN).EQ.NORSTS) 63,22
   63 NOM=NOM+1
      IRN=IRN+1
      GO TO 19
   22 WRITE(61,3) IOP,ME,ID,JFN,IYR,ISS
    3 FORMAT(9H1OPERATOR,I4,8X,6HMETHOD,I4,8X,16HDATE OF ANALYSIS,I4,8X,
     111HFILM NUMBER,I4,8X,4HYEAR,I7,7X,8HSITE SET,I4)
      WRITE(61,4) IPR,IB
    4 FORMAT(1H0,89X,7HPROJECT,I4,7X,5HBATCH,I7)
      WRITE(61,55)
   55 FORMAT(1H0)
      WRITE(61,5) MET
    5 FORMAT(1H0,16HPPM METAL IN ASH,6X,5R2)
      WRITE(61,55)
C        READ PPM OF SAMPLES A - O AND OF P
C
      READ(60,6) (P(I),I=1,16)
    6 FORMAT(8(1X,F9.1))
C        CHECK IF SAMPLES A - O ARE MISSING TO PRINT HISTOGRAM ONLY
C
      DO 998 I=1,15
      IF (P(I)) 997,998
  998 CONTINUE
      DO 996 I=1,15
  996 IAY(42,I)=L(I)
      IF (P(16)-S(11)) 993,992,989
  992 M=11
      GO TO 991
  993 DO 990 M=12,41
      IF (P(16)-S(M)) 990,991,990
  990 CONTINUE
  989 DO 988 M=1,10
      IF (P(16)-S(M)) 988,991,988
  988 CONTINUE
  991 IAY(M,16)=L(16)
      ITABLE=1
      GO TO 994
  997 DO 31 I=1,16
      IF (P(I)-S(11)) 23,24,25
   24 M=11
      GO TO 29
   23 IF (P(I)) 26,26,27
```

```
   26 IAY(42,I)=L(I)
      GO TO 31
   27 DO 28 M=12,41
      IF (P(I)-S(M)) 28,29,28
   28 CONTINUE
   25 DO 30 M=1,10
      IF (P(I)-S(M)) 30,29,30
   30 CONTINUE
   29 IAY(M,I)=L(I)
   31 CONTINUE
C        CALCULATE STATISTICS FOR P(I) FROM 1 TO 15
C
      DO 32 I=1,15
      IF (ASH(I)) 932,832
  932 OD(I)=P(I)*ASH(I)*0.01
      GO TO 32
  832 OD(I)=0.0
   32 CONTINUE
      J=1
      DO 100 I=1,15
      IF (P(I)) 101,100
  101 OBS(J)=P(I)
      ODOBS(J)=OD(I)
      SP=SP+OBS(J)
      SOD=SOD+ODOBS(J)
      NOP=NOP+1
      J=J+1
  100 CONTINUE
C        TABULATE STATISTICS FOR ONLY 1 SAMPLE
C
      J=J-1
      PNO=NOP
      IF (NOP.EQ.1) 933,934
  933 PM=SP
      ODM=SOD
      PMED=PM
      ODMED=ODM
      SD=0.0
      ODSD=0.0
      CV=0.0
      ODCV=0.0
      GO TO 994
C        DETERMINE IF NO OF OBS IS EVEN OR ODD FOR MEDIAN
C
  934 PM=SP/PNO
      ODM=SOD/PNO
      IF (NOP-3) 33,34,35
   33 PMED=PM
      SD=0.
      CV=0.
      ODMED=ODM
      ODSD=0.
      ODCV=0.
      GO TO 35
   34 SD=0.
      CV=0.
      PMED=OBS(2)
      ODSD=0.
      ODCV=0.
      ODMED=ODOBS(2)
   35 DO 38 I=1,15
      IF (P(I)) 36,36,37
   36 VP(I)=0.
      VOD(I)=0.
      GO TO 38
   37 VP(I)=PM-P(I)
```

```
      SVSP=SVSP+VP(I)*VP(I)
      VOD(I)=ODM-OD(I)
      SVSOD=SVSOD+VOD(I)*VOD(I)
   38 CONTINUE
      IF (NOP-3) 994,994,75
   75 REC=1./(PNO-1.0)
      SD=SQRTF(SVSP*REC)
      CV=SD*100.0/PM
      ODSD=SQRTF(SVSOD*REC)
      ODCV=ODSD*100.0/ODM
      IF (NOP-14) 40,40,39
   39 PMED=OBS(8)
      ODMED=ODOBS(8)
      GO TO 994
   40 ODD=NOP/2
      IF (ODD-(PNO/2.)) 41,42,42
   41 NODD=(PNO+1.)*.5
      PMED=OBS(NODD)
      ODMED=ODOBS(NODD)
      GO TO 994
   42 ND2=NOP/2
      N2=ND2+1
      PMED=(OBS(ND2)+OBS(N2))*.5
      ODMED=(ODOBS(ND2)+ODOBS(N2))*.5
C        COMPLETE ARRAY FOR PRINTING HISTOGRAM
C
  994 IF (MET(2).EQ.IND) 68,67
   67 DO 45 I=1,16
      DO 45 M=1,40
      IF (IAY(M,I).EQ.IBL) 45,44
   44 M=M+1
      DO 46 K=M,41
   46 IAY(K,I)=L(I)
   45 CONTINUE
   68 DO 49 M=1,12
   49 WRITE(61,7) S(M),(IAY(M,I),I=1,16)
    7 FORMAT(1H ,F13.1,6X,16R1)
      IF (ITABLE.EQ.1) 987,986
  987 DO 985 M=13,41
  985 WRITE(61,7) S(M),(IAY(M,I),I=1,16)
      WRITE(61,14) (IAY(42,I),I=1,16)
      WRITE(61,74)
   74 FORMAT(1H )
      WRITE(61,16) (L(I),I=1,16)
      WRITE(61,76) IOP,ME,ID,JFN,IYR,IPR,ISS,IB
   76 FORMAT(7H MASTER,13X,8I2)
      GO TO 995
  986 WRITE(61,8) S(13),(IAY(13,I),I=1,16),MET
    8 FORMAT(1H ,F13.1,6X,16R1,41X,5R2)
      WRITE(61,7) S(14),(IAY(14,I),I=1,16)
      WRITE(61,9) S(15),(IAY(15,I),I=1,16)
    9 FORMAT(1H ,F13.1,6X,16R1,26X,15HASH P.C.     ASH,8X,8HOVEN DRY)
      WRITE(61,7) S(16),(IAY(16,I),I=1,16)
      N=1
      DO 50 M=17,31
      WRITE(61,10) S(M),(IAY(M,I),I=1,16),L(N),ASH(N),P(N),OD(N)
   10 FORMAT(1H ,F13.1,6X,16R1,24X,R1,F6.2,F14.2,F12.4)
   50 N=N+1
      WRITE(61,7) S(32),(IAY(32,I),I=1,16)
      WRITE(61,70)S(33),(IAY(33,I),I=1,16),SP,SOD
   70 FORMAT(1H ,F13.1,6X,16R1,24X,5HTOTAL,F16.2,F12.2)
      WRITE(61,7) S(34),(IAY(34,I),I=1,16)
      WRITE(61,11)S(35),(IAY(35,I),I=1,16),PM,ODM
   11 FORMAT(1H ,F13.1,6X,16R1,24X,4HMEAN,F17.2,F12.2)
      WRITE(61,7) S(36),(IAY(36,I),I=1,16)
      WRITE(61,72)S(37),(IAY(37,I),I=1,16),SVSP,SVSOD
```

```
   72 FORMAT(1H ,F13.1,6X,16R1,24X,6HSUM SQ,F15.2,F12.2)
      WRITE(61,7) S(38),(IAY(38,I),I=1,16)
      WRITE(61,12)S(39),(IAY(39,I),I=1,16),SD,ODSD
   12 FORMAT(1H ,F13.1,6X,16R1,24X,4HS.D.,F17.2,F12.2)
      WRITE(61,7) S(40),(IAY(40,I),I=1,16)
      WRITE(61,13)S(41),(IAY(41,I),I=1,16),CV,ODCV
   13 FORMAT(1H ,F13.1,6X,16R1,24X,9HCOEFF VAR,F12.2,F12.2)
      WRITE(61,14)(IAY(42,I),I=1,16)
   14 FORMAT(13H NOT DETECTED,7X,16R1)
      WRITE(61,15) PMED,ODMED
   15 FORMAT(1H ,59X,6HMEDIAN,F15.2,F12.2)
      WRITE(61,16) (L(I),I=1,16)
   16 FORMAT(14H SAMPLE NUMBER,6X,16R1)
      WRITE(61,17)IOP,ME,ID,JFN,IYR,IPR,ISS,IB,IOP,ME,ID,JFN,IYR,IPR,ISS
     1,IB
   17 FORMAT(7H MASTER,13X,8I2,24X,6HMASTER,6X,8I2)
  995 NOM=NOM+1
      IRN=IRN+1
      GO TO 20
C        PRINT THE NO RESULT LIST FOR BATCH
C
   62 WRITE(61,3) IOP,ME,ID,JFN,IYR,ISS
      WRITE(61,4) IPR,IB
      NOM=NOM-1
      DO 69 I=1,NOM
      IF (IO(I).EQ.IBL2) 66,64
   66 WRITE(61,99) (NAME(I,K),K=1,5)
   99 FORMAT(1H0,5R2)
      GO TO 69
   64 WRITE(61,18) (NAME(I,K),K=1,5)
   18 FORMAT(1H0,5R2,10X,10HNO RESULTS)
   69 CONTINUE
C        READ ASH PERCENTAGE CARD AND TEST FOR BLANK CARD
C
      READ(60,1)(ASH(I),I=1,15)
      DO 108 I=1,15
      IF (ASH(I)) 109,108,109
  108 CONTINUE
      STOP
  109 NOM=1
      IRN=1
      GO TO 19
      END
```

Figure CL 3. Computer output from program.

OPERATOR 2 METHOD 5 DATE OF ANALYSIS 38 FILM NUMBER 1 YEAR 66 SITE SET 1
PROJECT 50 BATCH 72

PPM METAL IN ASH LEAD

1000000.0	
750000.0	
500000.0	
375000.0	
250000.0	
175000.0	
100000.0	
75000.0	
50000.0	
37500.0	
25000.0	
17500.0	
10000.0	
7500.0	
5000.0	E GH
3750.0	EFGH
2500.0	EFGHI
1750.0	FFGHI
1000.0	EFGHIJ
750.0	DEFGHIJ
500.0	DEFGHIJKL
375.0	DEFGHIJKL
250.0	DEFGHIJKLM
175.0	CDEFGHIJKLM
100.0	CDEFGHIJKLMN
75.0	CDEFGHIJKLMN
50.0	ABCDEFGHIJKLMNOP
37.5	ABCDEFGHIJKLMNOP
25.0	ABCDEFGHIJKLMNOP
17.5	ABCDEFGHIJKLMNOP
10.0	ABCDEFGHIJKLMNOP
7.5	ABCDEFGHIJKLMNOP
5.0	ABCDEFGHIJKLMNOP
3.8	ABCDEFGHIJKLMNOP
2.5	ABCDEFGHIJKLMNOP
1.8	ABCDEFGHIJKLMNOP
1.0	ABCDEFGHIJKLMNOP
.8	ABCDEFGHIJKLMNOP
.5	ABCDEFGHIJKLMNOP
.3	ABCDEFGHIJKLMNOP
.1	ABCDEFGHIJKLMNOP

NOT DETECTED

SAMPLE NUMBER ABCDEFGHIJKLMNOP
MASTER 2 538 16650 172

LEAD

	ASH P.C.	ASH	OVEN DRY
A	3.98	50.00	1.9900
B	5.01	50.00	2.5050
C	4.85	175.00	8.4875
D	4.96	750.00	37.2000
E	5.23	5000.00	261.5000
F	4.78	3750.00	179.2500
G	4.79	5000.00	239.5000
H	4.50	5000.00	225.0000
I	5.01	2500.00	125.2500
J	4.75	1000.00	47.5000
K	4.92	500.00	24.6000
L	5.09	500.00	25.4500
M	5.21	250.00	13.0250
N	4.83	100.00	4.8300
O	4.78	50.00	2.3900
TOTAL		24675.00	1198.48
MEAN		1645.00	79.90
SUM SQ		56895250.00	133602.99
S.D.		2015.92	97.69
COEFF VAR		122.55	122.27
MEDIAN		5000.00	225.00

MASTER 2 538 16650 172

OPERATOR 2 METHOD 5 DATE OF ANALYSIS 38 FILM NUMBER 1 YEAR 66 SITE SET 1

PROJECT 50 BATCH 72

PPM METAL IN ASH MOLYBDENUM

```
1000000.0
 750000.0
 500000.0
 375000.0
 250000.0
 175000.0
 100000.0
  75000.0
  50000.0
  37500.0
  25000.0
  17500.0
  10000.0
   7500.0
   5000.0
   3750.0
   2500.0
   1750.0
   1000.0
    750.0
    500.0                    P
    375.0                    P
    250.0                    P
    175.0                    P
    100.0                    P
     75.0                    P
     50.0                    P
     37.5                    P
     25.0                    P
     17.5                    P
     10.0                    P
      7.5                    P
      5.0                    P
      3.8                    P
      2.5                    P
      1.8                    P
      1.0                    P
       .8                    P
       .5                    P
       .3                    P
       .1                    P
NOT DETECTED       ABCDEFGHIJKLMNO

SAMPLE NUMBER      ABCDEFGHIJKLMNOP
MASTER              2 538 16650 172
```

OPERATOR 2 METHOD 5 DATE OF ANALYSIS 38 FILM NUMBER 1 YEAR 66 SITE SET 1

PROJECT 50 BATCH 72

PPM METAL IN ASH	INDIUM
1000000.0	
750000.0	
500000.0	
375000.0	
250000.0	
175000.0	
100000.0	
75000.0	
50000.0	
37500.0	
25000.0	
17500.0	
10000.0	
7500.0	
5000.0	
3750.0	
2500.0	
1750.0	
1000.0	
750.0	ABCDEFGHIJ LMNOP
500.0	K
375.0	
250.0	
175.0	
100.0	
75.0	
50.0	
37.5	
25.0	
17.5	
10.0	
7.5	
5.0	
3.8	
2.5	
1.8	
1.0	
.8	
.5	
.3	
.1	
NOT DETECTED	
SAMPLE NUMBER	ABCDEFGHIJKLMNOP
MASTER	2 538 16650 172

	INDIUM	
	ASH P.C.	ASH	OVEN DRY
A	3.98	750.00	29.8500
B	5.01	750.00	37.5750
C	4.85	750.00	36.3750
D	4.96	750.00	37.2000
E	5.23	750.00	39.2250
F	4.78	750.00	35.8500
G	4.79	750.00	35.9250
H	4.50	750.00	33.7500
I	5.01	750.00	37.5750
J	4.75	750.00	35.6250
K	4.92	500.00	24.6000
L	5.09	750.00	38.1750
M	5.21	750.00	39.0750
N	4.83	750.00	36.2250
O	4.78	750.00	35.8500
TOTAL		11000.00	532.88
MEAN		733.33	35.53
SUM SQ		58333.33	200.83
S.D.		64.55	3.79
COEFF VAR		8.80	10.66
MEDIAN		750.00	33.75
MASTER	2 538 16650 172		

'OPERATOR 2 METHOD 5 DATE OF ANALYSIS 38 FILM NUMBER 1 YEAR 66 SITE SET 1
PROJECT 50 BATCH 72

PPM METAL IN ASH ZINC

1000000.0	
750000.0	
500000.0	
375000.0	
250000.0	
175000.0	
100000.0	
75000.0	
50000.0	
37500.0	
25000.0	
17500.0	
10000.0	
7500.0	
5000.0	
3750.0	
2500.0	
1750.0	
1000.0	
750.0	
500.0	
375.0	
250.0	
175.0	L
100.0	L
75.0	L
50.0	L
37.5	L
25.0	L
17.5	L
10.0	L
7.5	L
5.0	L
3.8	L
2.5	L
1.8	L
1.0	L
.8	L
.5	L
.3	L
.1	L
NOT DETECTED	ABCDEFGHIJK MNOP
SAMPLE NUMBER	ABCDEFGHIJKLMNOP
MASTER	2 538 16650 172

ZINC

	ASH P.C.	ASH	OVEN DRY
A	3.98	0	0
B	5.01	0	0
C	4.85	0	0
D	4.96	0	0
E	5.23	0	0
F	4.78	0	0
G	4.79	0	0
H	4.50	0	0
I	5.01	0	0
J	4.75	0	0
K	4.92	0	0
L	5.09	175.00	8.9075
M	5.21	0	0
N	4.83	0	0
O	4.78	0	0
TOTAL		175.00	8.91
MEAN		175.00	8.91
SUM SQ		0	0
S.D.		0	0
COEFF VAR		0	0
MEDIAN		175.00	8.91
MASTER		2 538 16650 172	

OPERATOR 2 METHOD 5 DATE OF ANALYSIS 38 FILM NUMBER 1 YEAR 66 STTF SET 1
PROJECT 50 BATCH 72

PPM METAL IN ASH SILVER

1000000.0	
750000.0	
500000.0	
375000.0	
250000.0	
175000.0	
100000.0	
75000.0	
50000.0	
37500.0	
25000.0	
17500.0	
10000.0	
7500.0	
5000.0	
3750.0	
2500.0	
1750.0	
1000.0	
750.0	
500.0	
375.0	
250.0	
175.0	
100.0	
75.0	
50.0	
37.5	
25.0	
17.5	
10.0	F
7.5	F
5.0	F
3.8	C F
2.5	C F
1.8	C F
1.0	C F
.8	C F
.5	C F
.3	C F
.1	C F
NOT DETECTED	AB DE GHIJKLMNOP
SAMPLE NUMBER	ABCDEFGHIJKLMNOP
MASTER	2 538 16650 172

SILVER

	ASH P.C.	ASH	OVEN DRY
A	3.98	0	0
B	5.01	0	0
C	4.85	3.80	.1843
D	4.96	0	0
E	5.23	0	0
F	4.78	10.00	.4780
G	4.79	0	0
H	4.50	0	0
I	5.01	0	0
J	4.75	0	0
K	4.92	0	0
L	5.09	0	0
M	5.21	0	0
N	4.83	0	0
O	4.78	0	0
TOTAL		13.80	.66
MEAN		6.90	.33
SUM SQ		19.22	.04
S.D.		0	0
COEFF VAR		0	0
MEDIAN		6.90	.33
MASTER		2 538 16650 172	

OPERATOR 2 METHOD 5 DATE OF ANALYSIS 38 FILM NUMBER 1 YEAR 66 SITE SET 1

PROJECT 50 BATCH 72

PPM METAL IN ASH NICKEL

1000000.0				
750000.0				
500000.0				
375000.0				
250000.0				
175000.0				
100000.0				
75000.0				
50000.0				
37500.0				
25000.0				
17500.0				
10000.0				
7500.0				
5000.0				
3750.0				
2500.0				
1750.0				
1000.0				
750.0				
500.0				
375.0				
250.0				
175.0		G	I	
100.0		G	I	
75.0		G	I	
50.0		G	I	
37.5	C	G	I	
25.0	C	G	I	
17.5	C	G	I	
10.0	C	G	I	
7.5	C	G	I	
5.0	C	G	I	
3.8	C	G	I	
2.5	C	G	I	
1.8	C	G	I	
1.0	C	G	I	
.8	C	G	I	
.5	C	G	I	
.3	C	G	I	
.1	C	G	I	
NOT DETECTED	AB	DEF	H	JKLMNOP
SAMPLE NUMBER	ABCDEFGHIJKLMNOP			
MASTER	2 538 16650 172			

NICKEL

	ASH P.C.	ASH	OVEN DRY
A	3.98	0	0
B	5.01	0	0
C	4.85	37.50	1.8187
D	4.96	0	0
E	5.23	0	0
F	4.78	0	0
G	4.79	175.00	8.3825
H	4.50	0	0
I	5.01	175.00	8.7675
J	4.75	0	0
K	4.92	0	0
L	5.09	0	0
M	5.21	0	0
N	4.83	0	0
O	4.78	0	0
TOTAL		387.50	18.97
MEAN		129.17	6.32
SUM SQ		12604.17	30.51
S.D.		0	0
COEFF VAR		0	0
MEDIAN		175.00	8.38
MASTER	2 538 16650 172		

OPERATOR 2 METHOD 5 DATE OF ANALYSIS 38 FILM NUMBER 1 YEAR 66 SITE SET 1
PROJECT 50 BATCH 72

LEAD	
MOLYBDENUM	
INDIUM	
COPPER	NO RESULTS
ZINC	
TITANIUM	NO RESULTS
SILVER	
NICKEL	
COBALT	NO RESULTS

SECTION D DESCRIPTION OF FOUR FEASIBILITY EXPERIMENTS CARRIED OUT IN A BIOGEOCHEMICAL RESEARCH GREENHOUSE

by J.A.C. Fortescue (with a contribution by R.K. Wanless)

D 1 Introduction

Greenhouse experiments can be carried out in support of either geobotanical or biogeochemical investigations and one of the main objectives of the research program was to establish the feasibility of using the Geological Survey greenhouse for plant prospecting methods research.

For geobotanical investigations experiments in the greenhouse may be designed to examine the effect of adding known amounts of minor elements to the soil on the morphology of the plants, or to make detailed studies of induced toxicity symptoms in experimental plants (see Fig. A 4). Biogeochemical greenhouse experiments can be designed to solve problems arising from establishing sampling techniques, or to examine in detail accumulator plants under controlled conditions. Generally, the type of greenhouse experiments likely to yield information in either method of plant prospecting research can be carried out by less sophisticated techniques than those described by plant physiologists for observing the role of minor elements in plant nutrition (see Hewitt, 1966). It must be remembered that previous experience in plant prospecting (e.g., of the type reviewed in Section A 3) is often based on plants where the substrate in which they are growing has a five, ten, or even a hundredfold increase in the content of a minor element in the soil due to the presence of a mineral deposit. Consequently, in order to introduce variations in the minor element content of soil of this order of magnitude relatively crude methods can be used to add the metal to the soil - at least in preliminary experiments.

Four feasibility experiments were carried out in the greenhouse: one in the summer of 1963, and three during the summer of 1964. The spectrochemical analyses of samples derived from these experiments were made in the trailer laboratory by the rapid scan spectrograph method (see Section C) during the summer of 1965. Other methods of analysis that were tried out in connection with specific experiments are described below.

The detailed aims of the four experiments were:

(1) To investigate the feasibility of making short term minor element uptake experiments by growing cuttings in an artificial soil to which known amounts of elements have been added.
(2) To investigate the feasibility of using non-radioactive isotopic tracers in small scale experiments using cuttings.

(3) To investigate the feasibility of using a bottom watering technique for introducing minor elements to small trees and to gain experience in the use of Co^{60} as a tracer in greenhouse studies.
(4) To investigate the feasibility of using small tree phytometers in the greenhouse.

D 2 Description of the greenhouse

The greenhouse is at the south end of the main corridor on the eighth floor of the Geological Survey Building. Access is through a door at the end of the corridor that opens into a narrow preparation room oriented east and west. The main greenhouse, 34 feet long and 18 feet wide extends from the east end of the preparation room. There are slatted benches along each side of the main house (see Pls. D 1, D 2) and a large slatted bench 28 feet by 6 feet in the centre. The main house is heated by hot water pipes located under the benches. The temperature of the hot water system and the natural ventilation system are controlled thermostatically. The preparation room is air heated. During summer operation, the whole house is slat shaded; during the winter, the slats are rolled up.

Experience showed that it is necessary to reduce further the light in the main house during the summer months by pinning three thicknesses of white signal cloth inside the glass. No provision has yet been made to control the humidity of the house automatically. During very hot weather frequent spraying of the concrete floor of the house is necessary to avoid heat damage to plants.

D 3 Choice of substrate

Greenhouse experiments may be conducted by growing plants in one of four kinds of substrate:

(1) Solutions (containing nutrient elements and treatments).
(2) Inert substrates (for example, plastic balls or pure silica sand) to which nutrients and treatments are added as solid, or in solution.
(3) Mineral soils that have been specially prepared and mixed for greenhouse use (to which nutrients and treatments are added as solids, or in solution).
(4) Natural soils that have been taken intact from the field with plants growing in them (to which treatments are added in solution).

Three of the four experiments described are concerned with plants growing in substrates of type (3). Mixtures were of "sterilized soil" (obtained from the greenhouse of the Central Experimental Farm, by kind permission of Dr. A. P. Chan), river sand and garden peat. In experiments

where substrates of this kind were used, a layer of pea gravel was placed at the bottom of each container before the soil was put in. All the willow cutting experiments (i.e., Experiments I and II) were carried out in containers without drainage holes. The phytometers of "natural soil" (type 4) in Experiment IV were obtained from the Forest Experimental Station at Petawawa (by kind permission of the director, Dr. I.C.M. Place).

D 4 Selection of experimental plant species

The experimental plants belong to fast growing hardy forest species which occur in many parts of Canada. For the tree experiments, white birch Betula papyrifera Marsh was chosen, and for experiments with cuttings a common willow, Salix sp. All the willow cuttings were obtained from the same tree by Mr. J. Santon of the Forest Experimental Station at Petawawa.

D 5 General harvesting procedure

The methods which were used to grow the plants and to add treatments to them are described below. After growth of the willow cuttings was completed each plant was cut two inches above the original stem obtained from Petawawa. The leaves were separated from the stems and each organ weighed in the field condition. The samples were weighed again after they had been oven dried overnight at 110°C. For the birch trees, samples of stems and leaves were collected at intervals of one fifth of the total height of the stem at the end of the experiment. Each sample was divided into current leaves and stems, and second year growth of stems. These subsamples were weighed in the field condition and later, oven dry. The samples were stored until being dry ashed by the procedure described in Appendix CF.

D 6 Experiment I: The uptake of nickel, cobalt, lead, and silver by willow cuttings

This experiment was aimed at investigating the feasibility of making short term minor element uptake experiments by adding the elements in known quantities in treatment solutions to cuttings established in an artificial soil. Four non-essential, minor elements, which may occur in high concentrations in soil near mineral deposits, were included in the treatments. The treatments were made from known amounts of nitrates of nickel, cobalt, lead, and silver dissolved in tap water. In the case of silver solutions a very slight cloudiness, due to precipitation of silver chloride, was present in some treatments.

The plants were prepared as follows: In May 1964, eighty willow cuttings (between 0.4 and 0.8 inches in diameter and 6 to 8 inches long) were

Table D 1

Experiment I General data on treatments, volume of water added, height of plants, and field oven dry, and ash weights of single plants from each pan of cuttings.

Pan	Number of Treatments	Final concentration and elements added	Total volume of water added	Average height of new growth (4 plants) in inches	1964 TWIGS Sampling weight			1964 LEAVES Sample weight			Percent ash	
					Field g	Oven dry g	Ash g	Field g	Oven dry g	Ash g	Twigs	Leaves
A	---------- water only ----------		14.1	45	6.6	1.6	.472	11.0	2.8	.377	3.0	14.0
B	---------- water only ----------		16.0	34	1.8	0.7	.011	8.1	1.5	.212	1.6	14.1
C	10	100ppm Co	16.31	49	11.0	3.7	.099	21.0	5.5	.493	2.7	9.0
D	1	10ppm Co	19.3	51	7.7	5.3	.188	25.4	6.2	.765	3.6	10.7
E	1	1ppm Co	16.8	47	11.5	3.9	.129	18.4	4.9	.692	3.3	14.1
F	10	100ppm Ni	19.8	55	13.6	4.6	.119	22.6	5.7	.571	2.6	10.0
G	1	10ppm Ni	15.3	51	7.1	2.3	.067	15.2	3.4	.432	2.9	12.7
H	1	1ppm Ni	16.3	53	8.9	2.8	.080	19.1	4.3	.535	2.8	12.4
I	10	100ppm Pb	17.5	54	10.5	4.7	.154	11.0	5.5	.541	3.3	9.8
J	1	10ppm Pb	18.0	50	12.8	4.1	.120	22.9	5.5	.689	2.9	12.5
K	1	1ppm Pb	18.5	57	17.2	5.4	.189	23.6	5.6	.785	3.5	14.0
L	10	100ppm Ag	14.0	41	4.9	1.5	.034	18.0	4.1	.382	2.3	7.4
M	1	10ppm Ag	14.0	40	5.8	2.0	.064	14.4	3.2	.409	3.2	12.8
N	1	1ppm Ag	15.5	48	5.6	1.8	.068	15.0	3.5	.452	3.8	12.9
O	1	100ppm Co, Ni, Pb, Ag	-	Plants died after three days								
P	10	100ppm Co, Ni, Pb, Ag	14.5	43	6.3	2.6	.061	14.0	4.3	.361	2.3	8.4
Q	1	10ppm Co, Ni, Pb, Ag	19.0	48	7.0	2.7	.087	19.0	5.3	.492	3.2	9.3
R	10	10ppm Co, Ni, Pb, Ag	15.5	47	9.4	2.8	.102	24.0	5.4	.629	3.6	11.6
S	1	1ppm Co, Ni, Pb, Ag	17.5	46	10.8	3.5	.107	23.1	4.9	.621	3.1	12.7
T	10	1ppm Co, Ni, Pb, Ag	18.0	53	10.7	3.5	.105	11.0	2.8	.501	3.0	11.9

planted in 3-inch square peat pots containing a 1:1 mixture of sterile soil and peat (see Pl. D 3). Groups of six peat pots were placed in plastic dish pans (approximately 12" x 14" x 8") containing a 1-inch layer of pea gravel at the bottom for drainage. A centrally placed 1-inch diameter plastic pipe stood upright on the bottom of the pan and extended one inch above its rim. Additional peat was placed around the peat pots to support them and to keep the roots of the plants cool. Prior to the application of the treatments the cuttings were allowed to grow six weeks in this condition during which they all became well established in the peat pots (Pl. D 4).

After this preliminary period the peat pots were removed from the plastic pans which were then emptied, washed, and dried. A new layer of gravel was placed at the bottom of each pan followed by four peat pots containing the plants and enough dry river sand to bring up the total weight of the pan, gravel, peat pots, and plants to 35 pounds. The twenty plastic pans used in the experiment are shown in Plate D 1 at the time of planting on June 16th, and in Plate D 2 ten weeks later just before harvesting.

Different treatment solutions were added to each pan. The concentration of treatment solutions was based on the 35-pound weight of the pan and plants at the commencement of the experiments. (The weight of water needed to bring the river sand to the field condition was considered to compensate for the weight of the inert plastic container and the pipe.) It was calculated that in order to obtain a 1 ppm level of concentration of a metal in a pan it would require the addition of 0.0159 gm of metal in the treatment solution. On this basis treatment solutions were made containing each of the four elements to give 1 ppm, 10 ppm, and 100 ppm levels of concentration. Other solutions were prepared containing all four elements at each of these three levels of concentration. No attempt was made to vary the levels of concentration of different elements within the same treatment. Details of the treatment solutions added to the twenty pans included in the experiment are given in Table D 1. It will be noted that in some cases (for example, in C and F) the treatment was made in ten equal weekly instalments, whereas, in the case of the 10 ppm and the 1 ppm concentration levels the treatment was all included in the first instalment. An exception to this rule occurs in the case of pans R and T containing all four elements at the 10 ppm and 1 ppm levels, where the treatments were also added in ten weekly instalments.

Table D 1 also shows the total volume of water added to each pan during the ten week period, the average height of the four plants grown in each pan at the end of the experiment and the field, oven dry, and ash weights of the stem and leaf material collected from plants selected for analysis at the end of the experiment. The percentage ash for the two organs of each plant is also given.

Discussion of results:

(1) Growth of plants. The growth of the plants in all pans (see Pl. D 2) was vigorous and uniform regardless of treatments. The relatively low

average height of plants in A and B (Table D 1) may be the result of an anion effect (i.e., the nitrate added may have promoted growth of the treated plants). One exception to the healthy growth of all plants in a pan was in the case of pan O to which all four elements were added at the 100 ppm level in one treatment. In this case the plants died from unknown causes after three days. Death may have resulted from the anion effect, or the cation effect, or a combination of the two. It should be noted that when the same amount of the four elements was added in ten instalments (pan P) the treatment did not kill the plants.

While the plants were growing they were occasionally attacked by aphids. The aphids were removed by the application of a Cygon -2e spray six times during the summer. On the advice of Mr. A. Buckley of the Central Experimental Farm, Rapid Grow fertilizer solution was added to the plants several times during the summer as soon as a slight yellowing of the bottom leaves was observed. The fertilizer was added at a strength of four teaspoons per gallon of tap water. Because the same volume of this solution was added to each pan, and the elements within the treatments were all non-essential minor elements, it was assumed that these additional, and occasional treatments would not have a significant effect on the results of the experiment.

Tap water was used for making up the treatment solutions and for watering and spraying the trees in the greenhouse. This was done partly to find out if significant results could be obtained from experiments under these conditions and partly because large volumes of distilled, or metal free, water were not available at the time the experiments were carried out. Experience showed that the temperature and humidity of the greenhouse could be kept within workable limits during very hot weather only by keeping all doors and windows open and by frequent spraying of the inside of the house with water. The volume of water that reached the substrate in which the plants were growing because of spraying was not measured.

(2) Chemical analysis of plant ash. After harvesting the plants from each pan at the end of the experiment, one plant was selected for ashing and chemical analysis. (Because of the similar growth of the four plants in each of the pans and the need for reduction of scale of the analytical program to a minimum, it was considered that chemical results from a single plant would be sufficient in this case to demonstrate the feasibility of the experimental approach.) The samples of stem and leaf material were oven dried and ashed by the procedure described in Appendix CF and analyzed by the rapid scan spectrograph technique described in Section C. The results obtained from the spectrograph method for eight elements, four not included in the treatments and four included in the treatments, are given in Tables D 2 and D 3. All these results are on an oven dry weight basis and include combined sampling, subsampling and analytical error of the rapid scan spectrograph method. The results in Table D 2 indicate that the total error and variation in concentration level due to variations from plant to plant for the four elements not included in the treatments, is less than fourfold. It is of interest

Table D. 2

Experiment I The content of four elements (zinc, barium, strontium, and titanium), which were not included in the treatments, in the stems and leaves of 1964 growth of willow cuttings (oven dry weight basis).

Cutting	Ash %		Zinc		Barium		Strontium		Titanium	
	Stems	Leaves	Stems	Leaves	Stems	Leaves	Stems	Leaves	Stems	Leaves
A	3.0		29.5	244.	7.4	34.9	7.4	52.3	.7	2.4
B	1.6	14.1	15.7	247.	2.7	35.3	2.9	70.7	.3	5.3
C	2.7	9.0	26.8	156.	10.1	33.6	6.7	33.6	.5	1.6
D	3.6	10.7	17.8	188.	3.6	40.2	6.2	18.7	.4	.8
E	3.3	14.1	33.2	353.	8.3	70.7	8.3	70.7	.3	2.5
F	2.6	10.0	45.2	100.	12.9	25.1	9.7	25.1	.3	1.7
G	2.9	12.7	09.1	318.	7.3	63.5	10.9	47.7	.5	2.2
H	2.8	12.4	21.0	217.	5.0	31.1	7.1	46.6	.3	2.2
I	3.3	9.8	32.8	98.	8.2	24.6	12.3	24.6	.6	1.7
J	2.9	12.5	22.0	125.	5.1	31.4	7.3	31.4	.5	1.3
K	3.5	14.0	35.0	105.	6.1	24.5	8.8	52.6	.6	1.4
L	2.3	7.4	40.1	184.	5.7	27.6	5.7	27.6	.4	2.8
M	3.2	12.8	31.8	223.	8.0	48.0	8.0	48.0	.3	2.2
N	3.8	12.9	37.8	322.	6.6	64.6	9.5	64.6	.7	3.2
Ø	-	-	-	-	-	-	-	-	-	-
P	2.3	8.4	23.2	84.	8.7	21.0	11.6	31.5	.6	1.5
Q	3.2	9.3	56.3	93.	8.1	23.2	12.1	23.2	.8	1.6
R	3.6	11.6	36.2	116.	9.1	29.0	9.1	43.6	.6	2.0
S	3.1	12.7	30.1	222.	5.4	63.4	7.7	63.4	.5	2.2
T	3.0	11.9	30.0	209.	5.3	44.7	7.5	59.7	.5	2.1

Table D 3

Experiment I The content of four elements (nickel, cobalt, lead and silver), which were included in the treatments, in the stems and leaves of 1964 growth of willow cuttings (oven dry weight basis).

Cutting	Number of Treatments	Final conc. of elements added	Lead		Nickel		Cobalt		Silver	
			stems	leaves	stems	leaves	stems	leaves	stems	leaves
A	--------- water only ---------		1.0	17.5	1.8	10.0	n.d.	1.0	n.d.	n.d.
B	--------- water only ---------		0.5	17.5	1.8	17.5	0.1	1.8	0.01	0.50
C	10	100ppm Co	0.3	2.5	n.d.	n.d.	3.8	10.0	0.03	0.08
D	1	10ppm Co	n.d.	25.0	n.d.	17.5	0.5	17.5	n.d.	0.30
E	1	1ppm Co	0.5	25.0	1.8	10.0	0.3	1.8	0.25	0.50
F	10	100ppm Ni	0.5	2.5	17.5	37.5	n.d.	n.d.	0.08	0.10
G	1	10ppm Ni	0.5	17.5	10.0	37.5	n.d.	n.d.	0.05	0.80
H	1	1ppm Ni	n.d.	17.5	2.5	25.0	0.1	1.0	n.d.	0.50
I	10	100ppm Pb	7.5	7.5	n.d.	n.d.	n.d.	0.80	0.03	0.08
J	1	10ppm Pb	0.5	17.5	1.8	17.5	n.d.	1.0	n.d.	0.10
K	1	1ppm Pb	0.5	n.d.	5.0	n.d.	n.d.	1.0	0.03	0.10
L	10	100ppm Ag	0.1	17.5	1.8	17.5	n.d.	1.0	0.08	1.00
M	1	10ppm Ag	n.d.	5.0	n.d.	n.d.	n.d.	1.0	0.30	0.30
N	1	1ppm Ag	n.d.	50.0	2.5	10.0	n.d.	n.d.	0.03	0.30
Ø	1	100ppm Co, Ni, Pb, Ag	---------- plants died after three days ----------							
P	10	100ppm Co, Ni, Pb, Ag	0.5	3.8	10.0	25.0	3.8	7.5	0.10	0.80
Q	1	10ppm Co, Ni, Pb, Ag	0.3	5.0	5.0	7.5	0.08	1.8	0.10	0.30
R	10	10ppm Co, Ni, Pb, Ag	0.3	17.5	2.5	17.5	0.30	3.8	0.10	2.50
S	1	1ppm Co, Ni, Pb, Ag	0.5	17.5	2.5	17.5	n.d.	1.8	0.10	0.50
T	10	1ppm Co, Ni, Pb, Ag	1.0	17.5	2.5	10.0	0.3	1.8	0.05	0.50

n.d. = not detected

that the per cent ash of the leaf material is almost always three times that of the stems. The essential element zinc and the non-essential elements barium and strontium are consistently higher in concentration in the leaves than in the corresponding stems. In the case of titanium the concentration levels are near the detection limit of the method and may not be as significant.

The results for the elements included in the treatments are given in Table D 3 and may be summarized as follows: The addition of lead or silver did not produce positive results in the plants. In the case of nickel only the addition of 100 ppm of the metal produced positive results in both stems and leaves, although it appears that the addition of 10 ppm nickel in one treatment did affect the content of this element in stems only (pan G). The best results were obtained with cobalt. In this case the 100 ppm treatment resulted in a significant increase in the metal content of both stems (less than 0.2 ppm to 3.8 ppm) and leaves (less than 0.2 ppm to 7.5 ppm). Less consistent, but positive, results were obtained for stems and leaves of plants to which 10 ppm cobalt had been added in the treatments (see Table D 3).

Summary and conclusions

The experience gained during this experiment showed that:

(a) It is practical to grow willow cuttings by the technique described and to obtain positive results from simple uptake experiments from plants treated for a short ten week period.
(b) Experiments of this kind produce enough ash from both current stem and leaf growth for analysis of individual plants by the rapid scan spectrograph method described in Section C.
(c) Willow leaves contain a higher percentage of ash and minor elements than do the stems.
(d) Experiments of the kind described require about 20 litres of water to be added to a pan containing four plants over the ten week period. (This does not include the volume of water added during spraying of house on hot days.)
(e) Results from the application of the growing, sampling, processing, and scan spectrograph methods indicate that not more than a fourfold variation of minor element content can be expected from plants taken from different pans. Experience with the spectrograph method described in Section C 6 suggests that error from the analytical method is a relatively small part of the total error.
(f) Of the four elements included in the treatments, cobalt gave the best response, although a tenfold increase in the content of this element in the treatment (i.e. from 10 ppm to 100 ppm) did not produce a corresponding tenfold increase in the level of concentration of the element in the plant.

This experiment has shown that it is feasible to make short term minor element uptake experiments by growing willow cuttings in an artificial

substrate. Because of the preliminary nature of this experiment the results were discussed somewhat briefly. A conclusion to be drawn from the experiment is that in future experiments of this type treatments should be added at a concentration of not less than 100 ppm in a single application at the commencement of the experiment. The writer would like to acknowledge the painstaking work carried out by Mrs. T. Dawes during the summer of 1964 when she set up the treatment solutions for the willow plants and was responsible for keeping day to day greenhouse reports of the progress of Experiments I and II described here. This account is prepared partly from an internal report which she submitted in August 1964.

D 7 Experiment II. The uptake of non-radioactive isotopes of strontium and lead by willow cuttings

by R.K. Wanless and J.A.C. Fortescue

Introduction The object of this experiment was to explore the feasibility of using non-radioactive isotopic tracers in small scale biogeochemical investigations designed to provide information about the movement of certain elements through the soil and their uptake by plants. This study embodies one aspect of research into plant prospecting methods and is of a preliminary nature. Stable isotopes were employed because the use and hazards of radioactive materials in the greenhouse area are avoided, and because the side effects on forest plants due to the application of relatively large amounts of radioactive isotopes are unknown.

Six willow plants, with established rooting systems, were treated with solutions of strontium, or lead nitrate. Some plants received solutions prepared with normal isotopic compositions while others were treated with solutions enriched in strontium^{86} and still others with solutions enriched in lead^{206}. These two elements were chosen because stocks of isotopically enriched material were on hand and because the requisite mass spectrometric analytical techniques were available in the Isotope Geology Laboratory. Fortescue was responsible for originating the idea and for supervising the growing of the plants; Wanless prepared the tracer solutions, carried out the mass spectrometric analysis, and wrote up the results obtained.

Greenhouse methods The isotope experiments involved six of the same batch of willow cuttings as used in the previous experiment (Pl. D 4). Because of the small amounts of enriched isotopes available, single plants were employed for each treatment, and the size of the containers in which the peat pots containing the sprouting cuttings were placed, was reduced. Small circular plastic containers were used instead of plastic pans (Pl. D 5). The peat pots were placed in empty containers and the space between the peat pot wall and the container was filled with dry river sand so that the total weight of the container and plant was 1000 grams at the beginning of the experiment. Each container was always bottom watered by means of a plastic funnel (Pl. D 5).

Table D 4

Experiment II Details of the results of the strontium isotope determinations.

Date	Sample No.	Material	Sr^{84}	Sr^{86}	Sr^{87}	Sr^{88}	Sr^{86}/Sr^{88}	Sr^{87}/Sr^{86}	Sr^{87}/Sr^{86} X	Sample weight (g)
25-3-65	X-77	Peat Moss	0.577	9.866	7.064	82.492	.1196	.7160	.7166	0.0503
			0.563	9.887	7.072	82.479	.1199	.7153	.7168	
28-4-65	X-73	Pine Needles	0.578	9.917	7.026	82.479	.1202	.7085	.7109	0.0519
29-4-65			0.562	9.795	7.017	82.625	.1185	.7164	.7137	
3-5-65	X-73-II	Pine Needles	0.597	9.837	6.952	82.615	.1191	.7067	.7058	0.0878
			0.562	9.851	6.974	82.612	.1192	.7079	.7072	
10-6-65	Sol. No. 1	$Sr(NO_3)_2$	0.559	9.836	7.017	82.588	.1191	.7134	.7125	
9-6-65	Sol. No. 2*	$Sr(NO_3)_2$	0.187	88.744	4.522	6.547	13.55	.051		
10-5-65	W-1	Leaves	0.568	10.010	7.014	82.407	.1215	.7007	.7069	0.5944
10-5-65	W-1	Stems	0.553	9.962	7.028	82.458	.1208	.7055	.7096	0.1838
12-5-65	W-2*	Leaves	0.302	76.629	4.904	18.165	4.218	.0640		0.3077
			0.246	76.625	4.912	18.217	4.206	.0641		
13-5-65	W-2*	Stems	0.258	78.565	4.846	16.332	4.810	.0617		0.1878
31-5-65	W-2*	Soil	0.569	10.492	7.019	81.920	.1281	.6690		0.5165
20-5-65	W-4	Leaves	3.527	14.419	6.479	75.575	.1908	.4493		0.2493
14-6-65	W-5	Soil	0.569	9.788	7.024	82.619	.1185	.7176	.7149	0.4298
2-6-65	W-6	Soil	3.232	14.246	6.497	76.024	.1874	.4561		0.5875
			3.227	14.299	6.479	75.994	.1882	.4531		
3-6-65			3.252	14.346	6.510	75.892	.1890	.4538		
14-6-65	W-6-II	Soil	5.453	17.948	6.086	70.513	.2545	.3391		0.2582

* These analyses are of enriched material.

X Sr^{87}/Sr^{86} refers to ratio corrected for mass descrimination based on $Sr^{86}/Sr^{88} = 0.1194$.

At the end of a week after transplanting, all six plants began to droop and loose colour despite the addition of more water. Only the plants of this experiment were affected and it was suspected that the roots of the plants in the small containers were becoming too warm and that this was adversely affecting growth. In order to cool the roots the small plastic containers were placed in clean foam acid containers (Pl. D 6), the annular space was filled with peat for insulation, and a layer of paper was placed above the sand in each peat pot. After these changes the plants recovered and remained healthy for the duration of the experiment. Another problem associated with growing these plants was that, owing to the relatively small volume of the substrate, frequent watering was required. It was found that if the plants were left for more than 48 hours during very hot weather serious wilting occurred.

The experiments were commenced on June 19th, 1964, and the plants were harvested on August 18th.

Results:

(1) Strontium preliminary experiments. As an essential preliminary to experiments with growing plants strontium was extracted from samples of peat, moss, and pine needles. The extractive techniques and the purification procedures used were similar to those set up to process material for age determination studied. In this way sufficient strontium was recovered to permit satisfactory isotope ratio determinations (see Table D 4). From an examination of the data it is evident that these plant materials contain strontium of normal isotopic composition (compare $Sr(NO_3)$, solution 1 Table D 4).

Plant Experiments: Solutions containing known quantities of strontium nitrate with normal and enriched isotopic compositions were prepared and added to the soil in which willow plants were grown (see Table D 5). In each case a total of 1000 ml of treatment solution was added throughout the growing period of ten weeks. The plant materials were harvested and the leaves and stems were ashed separately.

The following analyses were carried out:

1) Strontium from leaves and stems of plant W1 treated with solution containing strontium of normal isotopic composition.
2) Strontium from leaves and stems of plant W2 treated with solution containing strontium enriched in Sr^{86}.
3) Strontium from soil in which plant W2 grew (enriched in Sr^{86}) in order to establish the isotopic ratio in soil after the experiment.
4) Strontium extracted from the leaf ash of plant W4 (i.e., not treated with a strontium solution). This extracted material was spiked with a strontium tracer solution to permit calculation of the normal strontium in a plant.

5) Strontium extracted from the soil that did not have strontium solution added W5. This analysis revealed the ratio of the normal strontium originally in the soil (compare with $Sr(NO_3)_2$ of Table D 4).
6) Strontium extracted from a second soil sample that had not been contaminated with treatments including strontium W6. This material was spiked with a strontium tracer solution and the concentration of normal strontium in the soil was determined.

Analyses were also carried out on aliquots of the normal strontium solution and the enriched strontium solutions used in the experiments.

Table D 5

Experiment II Details of the treatments added to each plant and the ash yield during preparation of samples for isotopic analysis

Plant	Treatment solution	Isotope Type	Solution Concentration	Weight of ash recovered		Weight % of soil ash recovered
				Stems (mg)	Leaves (mg)	
W 1	$Sr(NO_3)_2$	Normal	13.3 ugm/ml	180	580	12.7
W 2	$Sr(NO_3)_2$	Enriched	13.6 ugm/ml	180	620	13.1
W 3	$Pb(NO_3)_2$	Enriched	9.3 ugm/ml	130	740	14.5
W 4	$Pb(NO_3)_2$	Enriched	120 ugm/ml	140	730	13.0
W 5	$Pb(NO_3)_2$	Normal	8.6 ugm/ml	200	540	12.0
W 6	$Pb(NO_3)_2$	Normal	109 ugm/ml	180	540	12.1

Results from plant W1: (See Pls. D 5, D 6, D 7) (13,300 ugm normal strontium).

Analyses from leaves and stems are essentially identical to the results obtained from solution No. 1 (Table D 4) and to the strontium extracted from the peat moss and pine needle samples.

Results from plant W4: Strontium was extracted from leaves and spiked with enriched Sr^{86} in order to determine the concentration of strontium in an untreated plant. The value determined for the leaves was assumed to be the same for the stems. It has been determined that there is 694.6 ugm of strontium per gram of plant ash.

For W4:

Number of ugms strontium in leaves	= 694.6 x .730 ugms	=	507 ugms
Number of ugms strontium in stems	= 694.6 x .140 ugms	=	97 ugms
	Total		604 ugms

For W2:

Number of ugms strontium in leaves	= 694.6 x .620 ugms	=	431 ugms
Number of ugms strontium in stems	= 694.6 x .180 ugms	=	125 ugms
	Total		566 ugms

These results show very good agreement.

Results from plant W6: Strontium was extracted from soil ash and spiked with enriched Sr^{86} in order to determine the concentration of normal strontium originally in the soil. In this case the soil ash was found to contain 361.6 ugms/gm of ash. For the soil from plant W6 the ash recovery amounted to 12.2 ugms/gm of the soil by weight. The soil strontium content is, therefore, 361.6 x 0.1219 = 44 ugms/gm of soil. Each plant was grown in some 900 grams of soil having a total strontium content of 900 x 44 = 39,600 ugms of the element.

Results from plant W5: Strontium was extracted from an uncontaminated soil sample so that the isotopic ratio of the original strontium could be established. The values obtained from this test as shown in Table D 4 are in excellent agreement with the values obtained for the peat moss, pine needle, and solution No. 1 analyses.

Results from plant W2: In this experiment isotopic analyses were made for leaves, stems, and soil. Calculations for the partition of normal and enriched strontium in the three phases were made in an attempt to arrive at a material balance as follows:

At start of experiment

Soil contained	39,600 ugms normal strontium
Solution added	13,600 ugms enriched Sr^{86}
Total	53,200 ugms strontium

Leaves contained	431 ugms normal strontium
Stems contained	125 ugms normal strontium

Note The above listed figures for leaves and stems were derived from results obtained from plant W4 after growth for ten weeks. It is assumed that plant W2 took up the same amount of common strontium in addition to its increment of enriched strontium.

At the end of the experiment

Soil ratio indicates	1% enriched strontium	and 99% normal strontium
Leaves contain	85% enriched strontium	and 15% normal strontium
Stems contain	87% enriched strontium	and 13% normal strontium

The stems originally contained 125 ugms of normal strontium, and assuming no loss, this represents 13% of the strontium now found in the stems. That is, of a total of 962 ugms of strontium in the stems at the end of the experiment, 837 were enriched and 125 normal. Similarly, in the case of leaves, the total content is 2873 ugms of which 2442 ugm were enriched and 431 ugm normal.

The total enriched strontium content of leaves and stems = 837 + 2442 = 3279 ugms or 24% of the enriched strontium added to the soil. The remaining enriched strontium (13,600 - 3280 = 10,320 ugms) should have produced a greater change in the soil ratio because it amounts to about 26% of the quantity of normal strontium originally present. Calculations indicate that the soil only contained 1% of enriched strontium at the end of the experiment. In this experiment the roots and lower stems of the plant were not harvested and consequently it is not possible to complete the material balance. It is, therefore, likely that the remaining enriched strontium was retained in the lower portions of the plant.

Results:

(2) Lead. A similar group of experiments were carried out using lead nitrate solutions of normal and enriched isotopic composition. In this instance the plants were also grown for ten weeks and harvested in the same way as for the strontium experiments. Sufficient lead was recovered from approximately 150 mgm of plant ash to handle with extractive procedures set up to prepare samples for mass spectrographic analysis in connection with lead age determinations.

The following analyses were made:

(1) Lead from leaves and stems of a plant treated with solution containing lead of normal isotopic composition (W 6).
(2) Lead from leaves of plant treated with solution containing 9.3 ugms/ml of enriched Pb^{206} (W 3).
(3) Lead from leaves, stems, and soil of experiment W 4. In this instance the treatment solution contained 120 ugm/ml of lead enriched in Pb^{206}.
(4) Lead from leaves taken from a strontium experiment (W 2) to which some Pb^{206} spike had been added to permit the determination of the lead content in an uncontaminated plant.
(5) Lead from soil of strontium experiment W 1. Enriched Pb^{206} was added to obtain the lead concentration of the original soil.

(6) Lead from soil of strontium experiment W2 to provide isotopic ratio of lead in untreated soil.
(7) Blank run carried out on the reagents used in order to establish the extent to which lead contamination might influence the analytical results.

Results from plant W6: A total of 109,000 ugms of lead of normal isotopic composition was added. The analysis of leaf and stem ash are in good agreement with the value determined for Solution No. 6 (see Table D 6), and also for the soil of W2.

Results from plant W2: A portion of the ash of the leaves was used to determine the lead concentration in an untreated plant. The ash contained 57.6 ugms/gm.

For W2:
Number of ugms of lead in leaves = 57.6 x .620 = 35.7 ugms
Number of ugms of lead in stems = 57.6 x .180 = 10.4 ugms
Total 46.1 ugms

For W4:
Number of ugms of lead in leaves = 57.6 x .730 = 42.0 ugms
Number of ugms of lead in stems = 57.6 x .140 = 8.1 ugms
Total 50.1 ugms

Results from plant W1: Only strontium was added to W1; therefore, an isotope dilution analysis using enriched Pb^{206} permits calculation of the lead in an untreated soil sample. The result here was 17 ugms of lead/gram of ash. Since the ash represents 12.7%, the lead content of the soil is 17 x .127 = 2.1 ugms/gm of soil. Each plant was grown in 900 grams of soil containing a total of approximately 1900 ugms of lead.

Results from plant W4: A total of 120,000 ugms of Pb^{206} were added to plant W4. Analyses were carried out on the leaves, stems and soil after the experiment.

At the beginning

Soil contained	1,900	ugms of normal lead
Solution contained	120,000	ugms of enriched Pb^{206}
Total	121,900	ugms of lead

Leaves contained	42.0 ugms of normal lead
Stems contained	8.1 ugms of normal lead
Total	50.1 ugms of normal lead

Table D 6

Experiment II Details of the results of the lead isotope determinations

Date	Sample No.	Material	Pb^{204}	Pb^{206}	Pb^{207}	Pb^{208}	Pb^{206}/Pb^{204}	Pb^{207}/Pb^{204}	Pb^{208}/Pb^{204}	Pb^{208}/Pb^{206}	Pb^{207}/Pb^{206}
16-6-65	Soln No. 6	$Pb(NO_3)_2$	1.291	26.384	20.628	51.697	20.44	15.98	40.04	1.959	.782
15-6-65	Soln No. 4*	$Pb(NO_3)_2$	0.010	91.168	8.311	0.511	9117	831.1	51.1	.006	.091
8-5-65	W-6	Leaves	1.351	25.634	21.226	51.789	18.97	15.71	38.33	2.020	.828
26-7-65	W-6	Stems	1.294	26.318	20.728	51.660	20.34	16.02	39.92	1.963	.787
31-5-65	W-3*	Leaves	1.287	29.170	20.844	48.730	22.67	16.20	37.86	1.670	.715
21-5-65	W-4*	Leaves	0.930	47.056	17.234	34.779	50.60	18.53	37.40	.739	.366
26-5-65	W-4*	Stems	0.185	82.540	10.044	7.231	446.2	54.29	39.09	.088	.122
7-5-65	W-4*	Soil	0.214	79.808	10.592	9.386	372.9	49.50	43.86	.118	.133
8-5-65	W-2	Leaves	0.392	73.206	12.085	14.905	186.7	30.83	38.02	.204	.165
1-6-65	W-1	Soil	0.823	51.498	16.196	31.482	62.57	19.68	38.25	.611	.314
7-6-65	W-2	Soil	1.381	24.900	21.527	52.191	18.03	15.59	37.79	2.096	.865
20-5-65	Blank	Reagents	0.018	90.308	8.493	1.180	5017.1	471.8	65.56	.013	.095

* These analyses are of enriched material.

Note As in the case of the strontium experiments, the figures for leaves and stems were obtained from another plant grown for the same period of time. It was assumed that plant W4 took up the same amount of normal lead and the enriched lead component also.

At the end

Soil ratio indicates	83% enriched lead 17% normal lead
Leaves contain	33% enriched lead 67% normal lead
Stems contain	87% enriched lead 13% normal lead

The stems originally contained 8 ugms of normal lead; therefore, the total lead now in the stems = 62 ugms (8 ugms normal and 54 ugms spike). Originally the leaves contained 42 ugms of normal lead. The total leaf component is now 63 ugms (42 ugms normal, 21 ugms spike).

The total enriched lead in the plant = 54 + 21 = 75 ugms.

The enriched lead remaining in the soil = 120,000 x .83 = 99,600 ugms.

Only a very small percentage (approximately .06%) of the lead was taken up by the plant; and, it will be noted that 83% of the enriched lead was to be found in the soil after the experiment. This is in marked contrast to the strontium experiment where only 1% of the enriched material was found in the soil after the experiment.

When one tries to establish a material balance one finds a very large discrepancy. In this instance the plant stems and leaves account for 75 ugms of lead, whereas, a total of 20,400 ugms appears to have been removed from the soil. This experiment clearly suffers from the same limitation as the corresponding strontium one because no analyses were carried out on the roots and lower stem of the plant. It is very probable that a major portion of this "missing" lead may be localized in these parts of the plant.

Another interesting comparison may be made with the strontium experiment (W2). In the latter case both leaves and stems were found to contain strontium with essentially the same ratio, whereas, in the lead experiment the stems contained much more enriched lead. It would appear that the normal lead originally localized in the stem was eventually deposited in the leaves. This assumption may not be entirely valid because the leaves are assumed to have contained more normal lead initially.

Results from plant W3: Analyses were made only on leaves of W3. In this case a spike solution containing only 9.3 ugm/ml of Pb^{206} was added (total lead added = 9300 ugms). It was found that the leaves contained only 4.8%

spike lead (as opposed to 32.5% spike for the leaves of experiment W 4). The per cent take up is approximately in the same proportion as the ratio of the concentrations of the solutions used, indicating that, at these levels, the concentration of the lead does not influence the rate of uptake by the plant.

Results from the reagent blank: The lead content of reagents used in this work was found to be approximately 0.2 ugms per experiment and, therefore, constitutes a negligible contribution to the lead extracted from the plant and soil materials.

The results for both strontium and lead in treated plants may be summarized as follows:

Table D 7

Experiment II Summary of results showing the enriched isotope partition in the soil stems and leaves of treated plants.

Element	Soil		Stems		Leaves	
	Normal	Enriched	Normal	Enriched	Normal	Enriched
STRONTIUM	99% (W 2)	1% (W 2)	13% (W 2)	87% (W 2)	15% (W 2)	85% (W 2)
LEAD	17% (W 4)	83% (W 4)	13% (W 4)	87% (W 4)	67% (W 4)	33% (W 4)

Conclusions: It has been demonstrated that it is feasible to use Sr^{86} and Pb^{206} as tracers in short duration minor element uptake experiments using willow cuttings. A marked difference in the behaviour of strontium and lead has been observed: the former is much more mobile than the latter. The lower uptake of lead does not appear, however, to be due to toxic effects because the response was found to be identical for plants treated with solutions differing in concentration by a factor of ten. Attempts to establish a complete material balance were limited by a lack of experimental data for the root and lower stem systems in these preliminary experiments. It was found practical to use plants treated with one element as controls for plants treated with another element. This procedure reduces the number of plants required and indicates that the study of interelement effects of relatively large amounts of enriched elements may be satisfactorily undertaken by the techniques described here.

D 8 Experiment III The uptake of normal cobalt and cobalt^{60} by birch trees growing in large containers

Introduction This experiment was undertaken to explore the feasibility of a bottom watering technique for the introduction of a minor element (cobalt) into the rooting systems of small birch trees growing in relatively large containers in the greenhouse. This experiment is considered an essential preliminary to Experiment IV which involves the use of phytometers of the same size as the containers used in this experiment. A secondary object of the experiment was to gain experience in the use of radioactive tracers in the greenhouse. This was done by adding the same amount of a Co^{60} tracer to each of the four treatment solutions. In addition, the treatments contained 10, 100, 1000, or 10000 units of normal cobalt. One of these treatments was given to a single birch tree growing in a large container in order to discover how much metal was needed to produce a significant response in the cobalt level in the different organs of the tree. Another objective of the experiment was to gain information on the distribution and amount of cobalt in different organs of the same plant collected at intervals up the stem.

Similar to the two experiments previously described, this experiment was set up as a feasibility study and no rigorous precautions were taken to exclude contamination of the soils or plants by cobalt compounds in the tap water used to make up the treatments, or from dust in the atmosphere.

Method Four three year old birch trees were purchased in the balled condition and planted in sterile soil in ten gallon plastic garbage cans each containing a 2-inch layer of pea gravel at the bottom. Small holes were made at the base of each can at intervals round the circumference to allow easy access of the solution into the bottom of the can. Each plastic container complete with plant was placed in a plastic "saucer" for the duration of the experiment. Bottom watering with the treatment solutions was made by adding the solution to the saucer for a set time until the soil was completely saturated with the solution. The remainder of the treatment was then siphoned back into a storage bottle. Sufficient water was then added to the storage bottle to provide the same volume of treatment at the next application. In this way the same volume of water was added during each cycle of watering and as the experiment progressed more and more of the original treatment was added to the plants.

The plants were bottom watered with water only for the first month after planting. The first treatment was applied on July 17th, 1963, and the experiment was terminated during the second week of September 1963. Occasionally, during the summer a Malathion solution was sprayed on to the foliage of the plants to kill leaf boring insects.

Table D 8

Experiment III Details of the treatment solutions applied to the four birch trees

Treatment Solutions

Tree	Amount of carrier	Amount of Co^{60}	Final volume of each treatment
A	10 mgm	0.2 microcuries	1 litre plus 2 gallons
B	100 mgm	0.2 microcuries	1 litre plus 2 gallons
C	1000 mgm	0.2 microcuries	1 litre plus 2 gallons
D	10000 mgm	0.2 microcuries	1 litre plus 2 gallons

The application of the treatment was carried out as described above. To determine the approximate amount of each treatment added during the first application the count rate of the treatment was taken before and after the solution had been added to the plant.

The heights of the four trees at the start and at the end of the experiment are given in Table D 9. At the end of the experiment, the 1963 growth of twigs and leaves was sampled together with 1962 twig material. Before sampling the total height of each tree was measured and the main stem marked off at 1/5 intervals (these intervals were numbered I-V from below). The samples of the three organs obtained from each of the five levels up the main stem of each of the four trees were analyzed by the scan spectrograph method. All the results from this method are reported on an oven dry basis. Details of the method used, for measuring the radioactivity of the samples of solutions, soils, and plant ash are given in Table D 12.

Discussion of the results: In addition to cobalt, six other elements were determined in the samples of leaves and twigs taken from the four birch plants. The results for the elements not included within the treatments have been summarized in Table D 11. There is good general agreement between the results obtained for the 1962 and the 1963 growth of twigs, and in all cases the levels of concentration of the elements in the leaves is higher than in the twig material. One object of presenting this data was to show that the minor element content of all four trees is not exactly the same. In the case of trees A, B and D, the results are in fairly good agreement, but in the case of tree C consistently higher values were obtained for strontium, titanium and lead in many of the twig subsamples as well as in the case of all six elements in the leaves. The cause of this anomaly is not known. However, such background information appears to be of importance in assessing the general homogeneity of the minor element content of experimental plants quite apart from their physical characteristics. These results substantiate the observation made in the willow cutting experiment that higher values are found in leaf as opposed to stem material.

Table D 9

Experiment III Decrease in count rate and amount of treatment absorbed during the first application of cobalt solutions to four birch trees

Plant	Volume of treatment	Units of normal Co in solution	Count rate before application of treatment	Volume taken up by the soil	Count rate after application of treatment
A	1 litre + 2 gallons	10	2230 counts/min.	5.61	633 counts/min.
B	1 litre + 2 gallons	100	2257 counts/min.	6.01	788 counts/min.
C	1 litre + 2 gallons	1000	2240 counts/min.	6.01	850 counts/min.
D	1 litre + 2 gallons	10000	2170 counts/min.	5.61	975 counts/min.

Table D 10

Experiment III Heights of the four birch trees at the start and end of the treatment period

Tree	Height (June 7th)	Height (September 7th)
A	56"	61"
B	58"	66"
C	57"	64"
D	58 1/2"	66"

Table D 11

Experiment III The content of zinc, manganese, barium, strontium, titanium and lead in three organs of each of four birch trees

ELEMENT	TREE	Oven dry weight base		
		1962 twigs ppm	1963 twigs ppm	1963 leaves ppm
Zinc	A	31.4	34.9	77.9
	B	31.4	41.0	49.9
	C	46.3	35.4	192.3
	D	46.2	32.8	63.0
Manganese	A	4.2	6.3	33.1
	B	3.7	6.6	37.1
	C	6.2	7.8	61.4
	D	3.6	6.3	27.8
Barium	A	3.8	5.7	20.5
	B	1.8	3.1	15.0
	C	7.5	7.8	30.7
	D	3.7	4.3	17.5
Strontium	A	4.8	8.6	22.3
	B	4.8	10.2	24.7
	C	15.3	16.4	38.6
	D	8.9	14.7	31.5
Titanium	A	.46	.63	7.8
	B	.30	.48	7.3
	C	2.10	1.30	11.5
	D	.57	.75	7.0
Lead	A	1.90	1.25	31.2
	B	1.40	.63	19.7
	C	4.70	.85	57.7
	D	3.40	.38	24.3

Note These figures are arithmetic means for results obtained for the same organ taken at 1/5th intervals up the tree. All results are on an oven dry weight basis.

Table D 12

Experiment III Results from the addition of Co^{60} and normal cobalt to three year old birch trees

Position of the tree	TREE A			TREE B			TREE C			TREE D			Observation
	1962 twigs	1963 twigs	1963 leaves	1962 twigs	1963 twigs	1963 leaves	1962 twigs	1963 twigs	1963 leaves	1962 twigs	1963 twigs	1963 leaves	
V	x	318	220	x	80	68	x	52	46	x	31	39	Counts/minute/ash (1)
IV	267	337	212	85	99	67	0	50	43	54	31	32	Counts/minute/ash (1)
III	269	341	198	103	68	51	62	54	45	56	33	33	Counts/minute/ash (1)
II	246	364	197	86	77	42	75	47	50	58	31	32	Counts/minute/ash (1)
I	x	x	x	x	x	x	x	x	x	x	x	x	Counts/minute/ash (1)
V	x	ND	ND	x	10.0	ND	x	100	75	x	375	175	Total cobalt in ash ppm (2)
IV	10	10	ND	10	10.0	ND	50	100	50	375	375	175	Total cobalt in ash ppm (2)
III	10	ND	ND	ND	17.5	ND	25	50	50	375	375	100	Total cobalt in ash ppm (2)
II	ND	10	ND	ND	17.5	ND	25	25	25	375	375	100	Total cobalt in ash ppm (2)
I	x	x	x	x	x	x	x	x	x	x	x	x	Total cobalt in ash ppm (2)
Average	6.3	5.0		3.3	13.8		33	68	50	375	375	138	Total cobalt in ash ppm (2)
V	x	2.86	9.61	x	3.54	12.12	x	3.83	12.33	x	3.39	12.20	Ash percent
IV	1.47	2.80	9.29	2.24	3.26	11.73	2.33	3.50	12.68	2.07	3.70	12.27	Ash percent
III	1.88	3.01	9.74	1.80	3.85	12.87	2.79	4.13	12.44	2.04	3.65	12.84	Ash percent
II	2.46	3.93	9.88	2.36	4.40	2.82	3.17	4.75	11.66	2.31	3.75	13.29	Ash percent
I	x	x	x	x	x	x	x	x	x	x	x	x	Ash percent
Average	1.93	3.15	9.63	2.13	3.76	9.88	2.76	4.05	12.27	2.14	3.62	12.60	Ash percent

Position in Container		TREE A					TREE B					TREE C					TREE D					Observation
		N.	S.	C.	E	W	N	S	C	E	W	N	S	C	E	W	N	S	C	E	W	(See note 4)
Upper [5]		.6	5.8	3.9	5.0	.5	6.1	2.8	7.0	11.0	3.4	38	.6	15	1.1	2.3	4.4	15	2.6	7.9	11	Counts/minute/soil (1)
Middle		5.3	4.8	3.5	3.2	6.0	5.3	6.6	5.4	0.0	6.1	4.6	9.0	.6	.7	.7	53	30	2.1	33	98	Counts/minute/soil (1)
Lower		264	190	21	186	263	191	115	16	138	175	158	111	11	109	160	144	141	78	210	154	Counts/minute/soil (1)
	AV																					[3]
V	.35	.32	.27	.22	.25	.27	.34	.34	.25	.38	.33	.38	.35	.28	.37	.37	.43	.50	.38	.37	.35	Counts/minute/outside container (6)
IV	.68	.42	.35	x	.37	.35	.80	.70	x	.80	.60	.80	.70	x	.80	.80	.80	.90	x	.80	.70	Counts/minute/outside container (6)
III	.82	.50	.48	x	.48	.45	.90	.90	x	.90	.80	.90	.90	x	1.00	.90	1.20	1.10	x	.90	.90	Counts/minute/outside container (6)
II	1.27	1.10	1.10	x	1.00	1.00	1.40	1.10	x	1.30	1.20	1.4	1.60	x	1.60	1.30	1.30	1.40	x	1.30	1.30	Counts/minute/outside container (6)
I	1.38	1.40	1.20	1.5	1.50	1.40	1.40	1.30	1.9	1.40	1.30	1.6	1.60	1.90	1.70	1.40	1.10	1.30	1.6	1.30	1.20	Counts/minute/outside container (6)

Notes (1) Counts were made as follows: 100 mgm of plant ash (or 1.5 gm of soil) were placed in an aluminium plauchet at room temperature and humidity. Radioactivity was counted for ten minutes using an end window proportional counter. These results are relative to each other and semi-quantitative giving only a general indication of the variation of radioactivity in samples.

(2) Spectrochemical analyses made by the Scan Method described in Section C.

(3) Counting made with an atomic ace portable Geiger counter. (Background was 0.03 mr/hr)

(4) Letters refer to location at a given level in container N = North, S = South, E = East, W = West, C = Centre.

(5) "Upper" refers to samples taken in top 2" of soil. "Middle" refers to samples collected in middle 2" layer of soil. "Lower" refers to samples taken in lower 2" of soil just above the gravel layer.

(6) At positions N, S, E, W, five readings (I-V) were taken at equally spaced intervals from bottom to top of the container. Additional readings were taken at the centre of the top and the bottom.

No sample = x.

No cobalt detected in sample = N.D.

Four kinds of measurements were made to determine the level of concentration of the cobalt in the plant organ samples and the soils in which the plants were grown.

1) The radioactivity of the plant organ ash was measured and recorded.
2) The scan spectrograph results for the cobalt content in ash of plant samples was recorded.
3) The distribution of the radioactivity measured on the outside of the containers at the end of the experiment was recorded.
4) After drying, the soils were sampled and the radioactivity of each sample of soil was determined.

The results have been tabulated in Table D 12. Because of the preliminary nature of these experiments the interpretation of the results cannot be pressed too far. Briefly, as expected, there was a significant decrease in the count rate (of the Co^{60}) in the order of increasing total cobalt added (i.e., trees A-B-C-D); but, in the case of each organ taken at intervals up the tree the results are in excellent agreement. The results for the total cobalt concentration in the ash of the different organs of the four trees shows, as expected, an increase in concentration of the metal with increase in concentration in the treatment solution (i.e., A-B-C-D).

For these reasons the experiment was considered successful for the plants. It is of interest that a tenfold increase in the cobalt content of the treatment between tree C and D only resulted in an approximate threefold increase in the total cobalt content in the leaves; fivefold in the 1963 twigs; and tenfold in the 1962 twigs. This variation is almost certainly due to the relatively short period of time the trees were treated compared with the total time the trees had grown. An important result in relation to the phytometer experiment described below is that either 0.2 microcuries of Co^{60} or 1 gm of normal cobalt in some 5 litres of treatment solution was sufficient to give a good signal in the plant when applied under the conditions described above.

After the plants had been harvested the soils were left in the warm greenhouse until completely air dry. It was assumed that this drying out period would not affect the location of the Co^{60} tracer in the soil. When the soils were dry, count rates were recorded from the outside of the container first and then from fifteen soil samples collected as the sod was broken up (see explanation to Table D 12). The object of these observations was to determine how far the Co^{60} tracer had penetrated into the soil and if there was a significant creep of the tracer solution up the side of the container. The results in Table D 12 indicate that the greater part of the radioactivity was concentrated at the bottom of the container. The measurements made on the outside of the container suggest that there is an appreciable creep of the solution up the inside of the container. The measurements made on the soil samples are considered to give a more realistic picture of the distribution of

the tracer in the sod. In this case the relatively low values found in the centre sample taken at the bottom of the sod are particularly interesting, suggesting that the greater part of the treatment is concentrated at the outside edge of the gravel layer near the holes on the container wall.

Summary 1) The results of this experiment showed that it is feasible to bottom water quite large volumes of soil in the manner described and in this way introduce significant amounts of cobalt into a plant during a three month period.

2) If cobalt is used as a tracer, either normal cobalt, or Co^{60} tracer can be used.

3) The experiment showed that it is feasible to grow relatively large trees in the biogeochemical greenhouse without attention, except for watering and occasional spraying to kill insect pests.

Conclusions If small birch trees are grown in the greenhouse under the conditions of the experiment it is feasible to use either radioactive Co^{60} or normal cobalt as a tracer provided the metal is introduced as the nitrate by the bottom watering technique as described. Positive results may be obtained from the tracers in as short a period as three months.

D 9 Experiment IV The establishment of tree phytometers in the greenhouse

In Section A (Fig. A 5) it was stressed that biogeochemical or geobotanical research involves the relationship between the chemistry of soil and that of plants growing in the soil. Because of the extreme complexity of natural soil/plant systems it is desirable to have some means of bringing such systems intact into the greenhouse where they can be studied in detail. Transplanting methods of this kind were developed many years ago and described in detail by Clements and Goldsmith (1924) who called such transplanted systems phytometers. Our experiments with phytometers began in the summer of 1964 and were based on the experience gained with growing birch trees in the greenhouse the previous year.

Methods The birch trees selected for inclusion in the experiment were growing wild in a forest at the Petawawa Forest Experimental Station. Plastic garbage cans, of the same type as those used in the previous experiment, were used to collect the plants. Prior to going to the field the bottom was cut out of one of the cans and a vertical cut made from the bottom of the can to the rim at the top. The bottomless can was designed as a template and used in the collection of each tree.

After the trees had been selected in the field, a circular trench was dug around each individual leaving a central sod (see Pl. D 9). Using a large butcher knife and the template the sod was very carefully trimmed to fit inside a garbage can (Pl. D 9) the template was then placed around the sod and strapped in position while the sod was separated from the subsoil. The sod, held in the template for support, was then transferred to a garbage can (in which a three inch layer of gravel had been placed). The template was then used again to collect the next tree.

The phytometers were taken to the greenhouse and set up in saucers as described in the previous experiment. For a period of nearly four months the trees in the phytometers grew well in the greenhouse in spite of the variation in climate between the greenhouse and the forest from which they were collected.

Summary and Conclusions This experiment showed that it is feasible to extract small birch trees together with soil in which they are growing and transpose them to the greenhouse in containers in which they then grew successfully.

D 10 General summary and conclusions

The four preliminary experiments described here have provided us with valuable experience in the development of greenhouse technique for biogeochemistry. As a result of the first experiment a technique has been established for growing cuttings under standard conditions suitable for use in short term minor element uptake experiments. This approach could be used using treatments added as liquids or as solids. The second experiment, which was probably the most important, demonstrated that it is feasible to use Sr^{86} and Pb^{206} as tracers in short duration, minor element, uptake experiments using cuttings. The third experiment provided important information on the use of tracers in larger scale experiments involving trees to which treatments were added in solution from below. In this case also, the results were positive and the weight of element in the treatment required to produce a response in the different organs of the plants was determined. In the fourth experiment the feasibility of a method for bringing soil/plant systems from forest into the greenhouse was demonstrated.

D 11 Future greenhouse investigations

Throughout this section the emphasis has been on the establishment of the feasibility of the different greenhouse techniques which are described. It is fitting at the end of this section to answer briefly the question - Feasibility for what? - which must often have occurred to the reader.

Greenhouse investigations based on those described here may be divided into three main types:

Type A Experiments where the experimental component is the soil and the same plants are used to compare the availability of chemical elements in different substrates.

Type B Experiments where the experimental component is the plant (which may be a whole plant or a cutting) growing in one of a series of standard substrates.

Type C Experiments where both the plant and the soil are experimental components in which the relative mobility and effect of externally applied treatments on small "prisms of landscape" are studied.

Experiments of Type A would be carried out along the lines of Experiments I and II where plants of the same kind (e.g., willow cuttings, perhaps plus cuttings of other trees and herbs) would be grown in artificial substrates of the type described here, or in soil containing relatively large concentrations of minor elements collected in the vicinity of mineral deposits of the types described in Sections E, F and G of this report. These experiments would lead naturally to the establishment of "standard substrates" which would have given positive results in experiments of Type A. These standard substrates would then be used to investigate the uptake of chemical elements by plants of species found near known undisturbed mineral deposits, and these experiments would be of Type B. Type C experiments would follow the experience gained from the Experiments II, III and IV in which the use of tracers was shown to be practical in biogeochemical greenhouse experiments. Experiments of Type C would be based on phytometers taken in the field in a manner similar to that described in Experiment IV.

It seems clear that there is a broad and interesting field of research in biogeochemistry which can be based on greenhouse experiments. The results of such experiments would not only be of interest to those engaged in prospecting but also in the fields of forest nutrition, pollution and even theoretical geochemistry. The reason why such experiments are of particular interest is because they throw light on the details of relationships between the content of minor elements, particularly non essential minor elements, in soils and in plants growing upon the soils in closed, artificial, or natural systems. Hence, the emphasis here is on geochemistry, in the sense described in Section A 3 and not on plant physiology or nutrition.

References

Clements, F.E. and Goldsmith, G.W.
1924: Carnegie Inst. of Washington, Pub. 356.

Hewitt, E.J.
1966: Sand and water culture methods used in the study of plant nutrition; Technical Communication No. 22 (Revised); Commonwealth Bureau of Horticulture and Plantation Crops, East Malling, Maidstone Kent.

Plate D 1
The south side of the main greenhouse showing the willow cutting experiment set up on June 16th, 1964. Note the plants included in Experiment II beside the measuring cylinders. The signal cloth tacked to the roof of the greenhouse supplements the shading provided by the slats on the outside.

Plate D 2
South side of the main greenhouse showing the willow cuttings just before sampling in September 1964. Note the preparation room in the background.

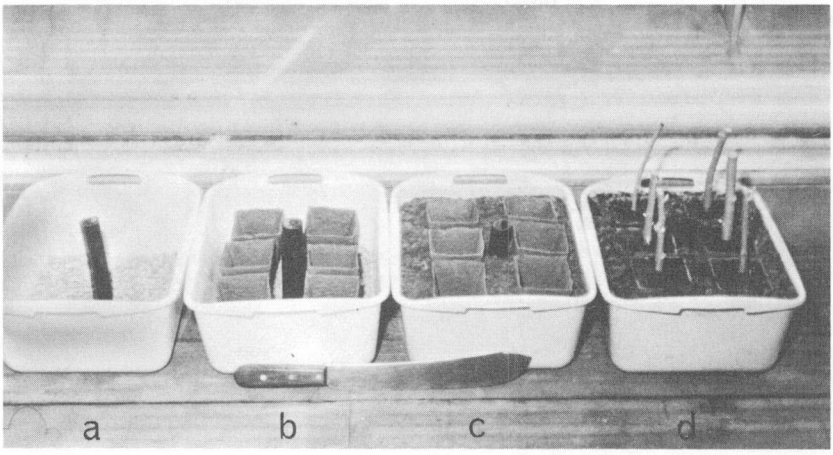


Plate D 3 Experiment I Stages in the preparation of a pan for growing willow plants.

a Plastic pipe for watering in place, on layer of gravel.
b Peat pots placed on layer of gravel.
c Peat placed around pots for support and insulation.
d Cuttings in place in artificial sterile soil/peat mixture.

Plate D 4 Experiment I A pan of willow cuttings after the six week preliminary growth period.

Plate D 5 Experiment II Willow cuttings used in isotope uptake experiments at the commencement of the experiment June 19th 1964. Note the small plastic containers and the funnels for bottom watering.

Plate D 6 Experiment II Willow cuttings used in isotope uptake experiments four weeks after commencement of the experiment. Note the additional insulation of the plastic containers.

Plant W 1
Normal
13.3 µgm/ml

Plant W 2
Enriched
13.6 µgm/ml

STRONTIUM EXPERIMENT

Plant W 3
Enriched
9.3 µgm/ml

Plant W 4
Enriched
120 µgm/ml

LEAD EXPERIMENT

Plant W 5
Normal
8.6 µgm/ml

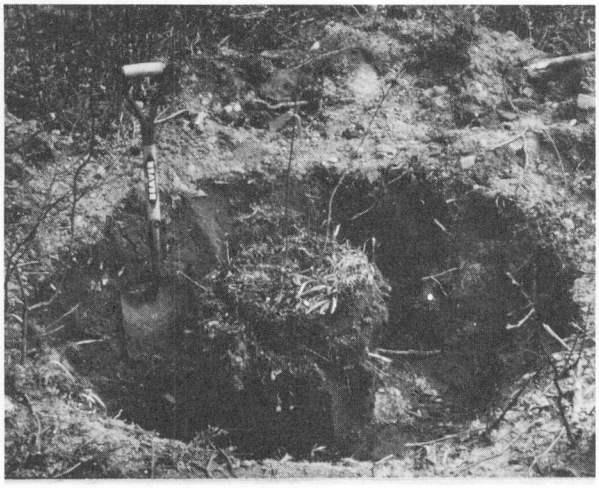
Plant W 6
Normal
109 µgm/ml

LEAD-STRONTIUM EXPERIMENT

Plate D 7 Experiment II Plants involved in isotope experiment just prior to sampling in September 1964.

Plate D 8 a, b Experiment III The growth of the three year old birch tree (Plant D). The 10,000 refers to the concentration of cobalt in the treatment solution.

a-At the commencement of the experiment June 1963
b-At the end of the experiment September 1963



Stage I. A circular trench is dug round the tree.

Stage II. Tree and sod are brought intact to greenhouse.

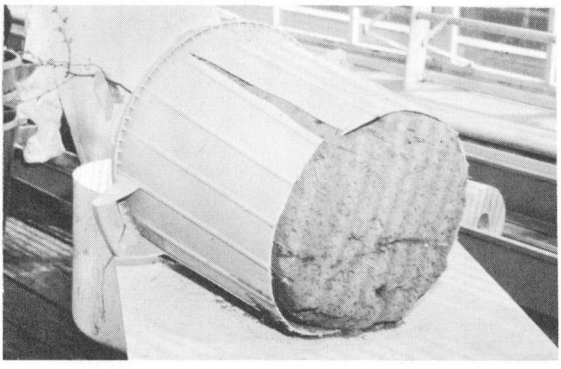
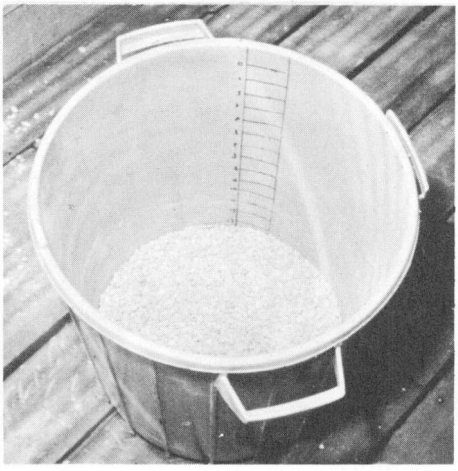
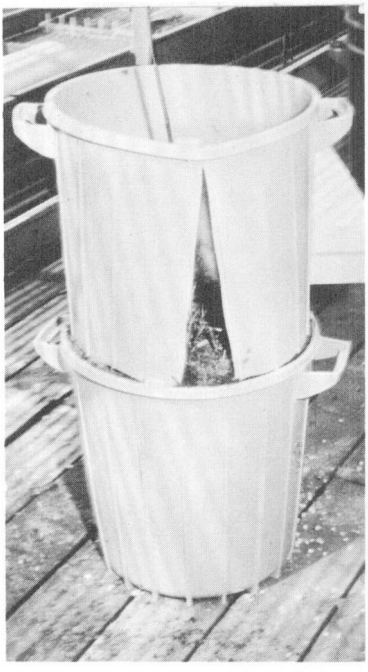


Plate D 9 Experiment IV Four stages in the preparation of a tree phytometer for greenhouse use. Stages II, III and IV are usually carried out in the field as soon as Stage I is completed.



Stage III. Plastic container prepared to receive tree and sod. Note gravel layer.



Stage IV. Sod is placed in container.