

Mines Branch Information Circular IC 150

THE PHYSICS AND RADIOTRACER SUBDIVISION  
OF THE MINES BRANCH, 1959-1963

by

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ABSTRACT

The work of the Physics and Radiotracer Subdivision of the Mines Branch during 1959-1963 is reviewed. Particular attention is drawn to the close link between industry and government laboratories in this field, which is rapidly growing in importance and is inadequately served by private facilities. A survey is provided of the major projects in radiochemistry, tracer applications, activation analysis, and the development of specialized control instrumentation. A detailed bibliography is appended.

RÉSUMÉ

La présente circulaire donne une idée des travaux exécutés à la Subdivision de la physique et des indicateurs radioactifs de la Direction des mines, pendant les années 1959 à 1963. L'auteur apporte une attention particulière à la collaboration étroite qui existe entre l'industrie et les laboratoires de l'Etat dans ce domaine de plus en plus important où l'industrie privée n'est pas outillée pour répondre aux besoins de façon satisfaisante. La circulaire renseigne sur les principales entreprises dans le domaine de la radiochimie, des applications des indicateurs radioactifs, de l'analyse d'activation et de la mise au point d'instruments spécialisés de contrôle. L'auteur y a ajouté une bibliographie détaillée.

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Staff of Physics and Radiotracer Subdivision,  
July 1962

Left to right: 1st row standing: J.L. Horwood, Prof. H.M. Dutton,  
Dr. P.G. Manning, Miss B. Moore, Dr. C. Lapointe,  
Dr. H.P. Dibbs, G.E. Alexander, A.H. Bettens,  
Dr. G.G. Eichholz.  
2nd row: J.M. Lefebvre, I.I. Tingley, Dr. M. Donato,  
C.A. Josling, A.F. Seeley, H. Inhaber, F.W. Dampier,  
Dr. J.D. Keys.  
Kneeling: T.R. Flint, A. Halmy, C. McMahon,  
J.V. Krzyzewski.

## FOREWORD

When the Radioactivity Division of the Mines Branch was dissolved four years ago and its staff was dispersed from its historic Quonset Huts on Lydia Street into more modern and commodious quarters, the undersigned wrote a review of the activities of one of its sections, which was usually referred to as the Radiation Laboratory by remote analogy with other, bigger and more famous institutions. In the intervening years this section, under its more formal and resounding title of Physics and Radiotracer Subdivision of the Mineral Sciences Division of the Mines Branch of the Department of Mines and Technical Surveys, has considerably expanded its research and development work on applied radioisotopes, radiochemistry, and the provision of control instrumentation for the mining and metallurgical industries.

This diversification of activities occurred without significant change or increase in staff, indicating the adaptability and versatility of the individuals comprising the group. The new fields of activity reflect, to a large extent, the need and scope for research and development currently required to aid the mining and metallurgical industries in the solution of their problems.

The impending departure of the undersigned from his present post marks an appropriate occasion to review the advances and achievements of the past four years. In concentrating on the work of this small group one encounters what, I hope, is a good example of what can be achieved by government and industry working in close contact, each in its proper sphere, to improve the efficiency and the ultimate prosperity of Canadian industry in a field so vital to Canada's future development.

G.G. Eichholz

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## INTRODUCTION

The Mines Branch of the Department of Mines and Technical Surveys, Ottawa, is charged specifically with

"aiding the mineral industries by the development of new techniques and procedures and by the application of long-term research to improve the economics and utilization of the mineral resources of Canada".

This fairly broad mandate is operated under controls of a constitutional, financial and practical nature for the benefit of the country as a whole, in co-operation with the provinces and private industry. Both of the latter groups have their own areas of responsibility for development and exploitation of resources. The rapid technological developments that have taken place throughout the world during the past ten to fifteen years are finding applications in the mining and metallurgical industries. It is, therefore, important that this vital segment of the Canadian industrial economy take full advantage of such developments to retain and improve its competitive position in world markets.

One of the important functions of the Mines Branch is to develop, adapt and demonstrate scientific and technological improvements that are applicable to the mining industry. Although some of the larger mining companies have set up excellent facilities for research and development, many smaller firms lack the staff and financial resources to undertake effective work in this field. The rendering of scientific and technological assistance to both small and large industries constitutes an important function of the Mines Branch. Co-operative projects between the government laboratories and industry, right down to the working level, have been developed to a degree that is only rarely found in other technically advanced countries.

One of the most striking illustrations of such mutual assistance was the now historic development of the Canadian uranium industry based

largely on initial development done in the old Quonset Huts on Lydia Street\*, Ottawa, by the Radioactivity Division of the Mines Branch (1)\*\*. One of the smaller sections of that Division was the Radiation Laboratory, whose history the present writer has recounted in a previous Information Circular (D6), published in 1959. The present account is conceived as a continuation of the previous one and the listing of reports and publications in the bibliography in this report continues the numbering of the previous ones (H52, D6).

However, it will be seen that the subject matter covered has changed considerably in these four years, partly because of the decline of work required for the uranium mines and partly because of the increased emphasis that is being laid on better control instrumentation and methods of measuring process variables in milling and metallurgical operations. For this purpose several new instruments have been developed and introduced to the industry. At the same time, the use of radioactive tracers in exploring operating problems has continued and they have also been employed as an aid in many laboratory investigations where their use has often simplified measurements and may have been crucial to the feasibility of some projects. These are the fields that the present review proposes to explore.

In the earlier days of the Radioactivity Division, the work of the Physics and Electronics Section, our predecessor, was devoted entirely to the development and use of radiometric methods for the determination of uranium and thorium in ores. In time it became logical to develop other equipment that utilized this inherent radioactivity and thus a programme of electronic sorting and continuous assaying of radioactive ores and solutions was started, which was the forerunner of many current projects.

At the same time, the availability of suitable counting equipment led to an investigation of neutron activation analysis of ores; this work, too, has led to considerable developments in recent years, which will be discussed in detail later. Similarly, the use of radioactive isotopes as tracers in chemical processes and in the metallurgical industry is very slowly gaining acceptance, though rather more slowly than one would wish (A36). However, laboratory research using radiotracers is carried on in various directions and is yielding many useful results.

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\* Even the name Lydia Street is likely to be relegated to oblivion as a result of reconstruction of the area in connection with the Queensway.

\*\* Numbers in brackets refer to the bibliography at the end of this report.

## STAFF

The recent diversification of activity was made possible because of the range of disciplines existing in the professional background of the scientific staff. For most of the time the group has been divided almost entirely between physicists and chemists, a situation that is of mutual benefit when dealing with problems that are so much in the borderline area of the physical sciences. In addition, the presence of one or two postdoctorate fellows from various countries has infused new ideas and new approaches and has been most useful in maintaining momentum in fields of research which were sometimes only marginally related to the main programme of the Subdivision. It is to be hoped that it will be possible to attract research fellows of comparable calibre in future years.

The other aspect of fundamental importance to the widened research programme, with the increased work load it has inevitably produced, has been the presence of a stable and loyal technical staff. There has been no turnover in this group since the early 1950's, nor, unfortunately, any addition to spread the ever-growing work load. To cope with this situation, the Subdivision is fortunate in having a highly experienced group of men to whom responsibility for many of the established projects can be delegated with confidence. Many new skills have had to be mastered in pursuing the programme of the Subdivision.

Another group that should not be overlooked in discussing staffing are the summer students. The Subdivision has been fortunate in obtaining some very able summer assistants. It has been the practice to give each a project of his own so far as possible, and this has resulted in some very valuable work (e.g., A 31, A 60, H 60, H 61, H 62, H 101, H 102, H 108, H 121, H 122). The regular two student positions assigned to the Subdivision have usually been supplemented by one or two more summer assistants provided on loan by other sections or divisions that had projects of mutual interest. The benefits both to the Department, which in the past has often recruited permanent staff from among previous summer students, and to the students themselves are incalculable, since in many cases this work first introduces them to actual research laboratory conditions and confronts them with problems for which no ready textbook solutions may exist. At a time when it is vital to Canada to encourage students to enter the applied sciences, this training, if properly applied, can only be beneficial and it is to be hoped that it can be extended to a greater number of students, both undergraduates and graduates, in future.

Appendix A lists both permanent and temporary staff of the Subdivision since 1959.



## LABORATORY FACILITIES

When the previous survey was being written, in the spring of 1959 (D6), the Section still formed part of the Radioactivity Division. The radiometric and electronics laboratories were housed in congested bustle in the Quonset Huts at 30 Lydia Street (1), a laboratory that must have set a near-record for its density of scientists per cubic foot or its output in ultimate dollar value per unit of occupied floor area. The radioactive tracer work was carried on in only slightly less crowded quarters in the South wing of the Physical Metallurgy Division at 568 Booth Street. To illustrate the conditions there, it needs merely be stated that five scientists at times had to take turns at using the only available fume hood for radioactive work.

In the summer of 1959 the staff in the Quonset Huts was moved into vastly more spacious and convenient quarters in the new Mines Branch building at 555 Booth Street. The radiometric and electronics laboratories were housed on the second floor of the East (Lebreton Street) wing of that building and, for the first time in years, spare bench space became available for the rapidly growing list of new projects. Later in 1959, the tracer laboratories were moved into a suite of rooms on the third floor of the West (Booth Street) wing of the same building and for once the research workers could "luxuriate" in surroundings where there was one fume hood per man and where, at last, proper provisions could be made for separate, locked storage of bulk radioactive material (in the basement), for a "hot laboratory" properly fitted up to permit control of access, and for facilities for the handling of slightly higher levels of activity, up to 100 millicuries, than would normally be used in the other, more open laboratories (Figure 1). A separate lathe and polishing facilities for radioactive materials were also installed.

Consequent on the formation of the Mineral Sciences Division and in a move to consolidate all portions of the Division under one roof, the radiometric and electronic laboratories moved once more, in the summer of 1960, from Lebreton Street into the first floor on the Booth Street wing of the same building. This move permitted the present allocation of space and has benefitted particularly the semiconductor research programme.

The Subdivision now occupies four large laboratories on the first floor, 555 Booth Street, devoted to the physics and electronics section of the Subdivision. Here also, are the Subdivision office, a small workshop and a preparation room, as well as the Branch Electronics Stores, which are now run by a full-time storekeeper under the Branch Administration.

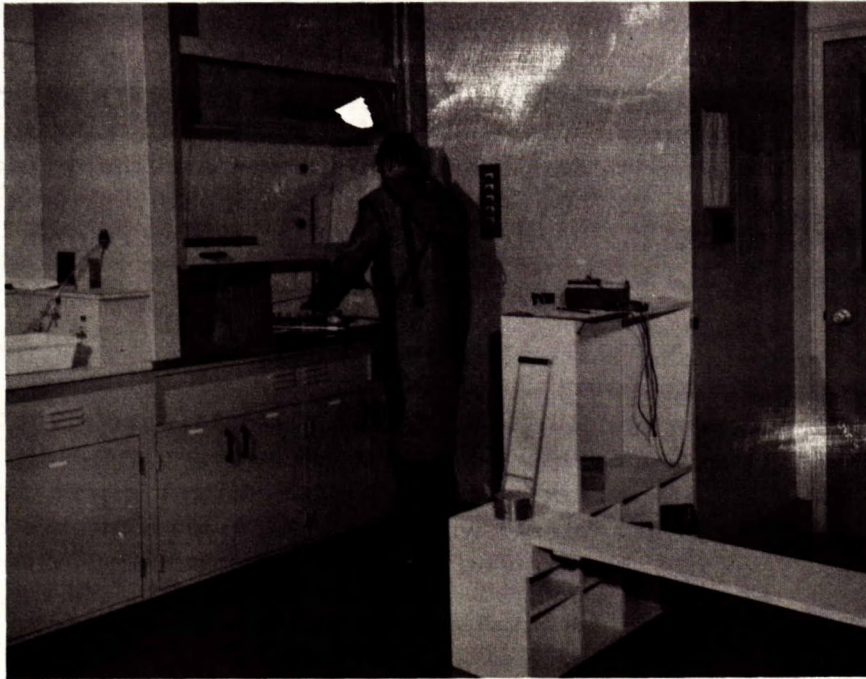


Figure 1. View of part of hot laboratory.

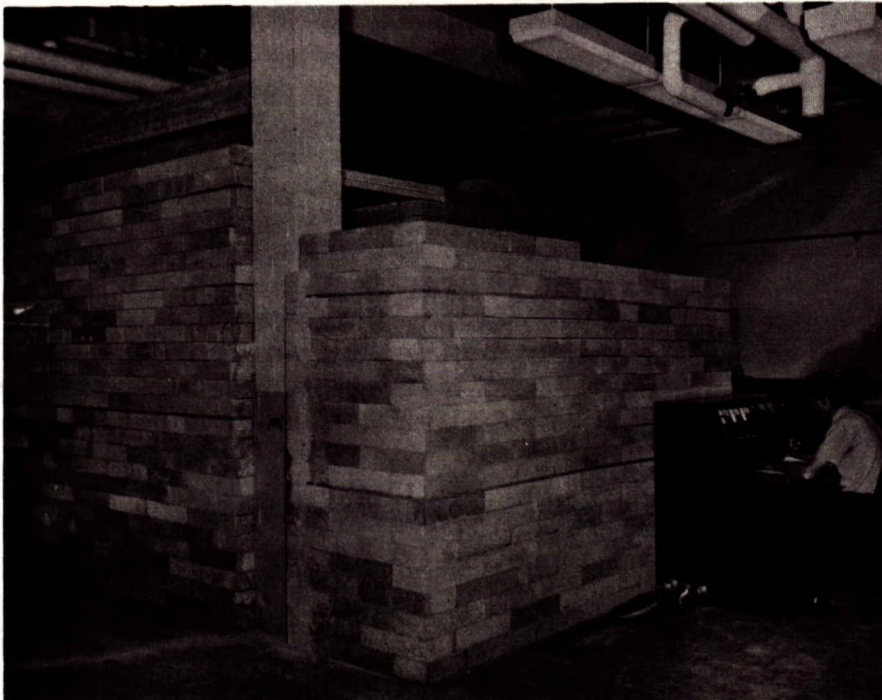


Figure 2. View of neutron generator console and shielding.

In the spring of 1961 a Texas-Nuclear neutron generator was acquired for a stepped-up development programme on neutron activation analysis. After initial attempts to install it on the top floor, it soon became evident that the concrete block shielding required would exceed the floor loading limit. The equipment was, therefore, moved into a large basement room and a massive concrete and lumber shield was erected by the staff of the Subdivision (Figure 2). Being almost entirely below ground level, the main radiation interaction, detected by very sensitive equipment, is in an upward direction.

The Tracer Laboratories on the third floor consist of a row of six large laboratories, two small offices and the small polishing room. Facilities for storing and handling radioactive materials have been improved steadily over the years. However, as far as possible, artificial radioactive isotopes, with the exception of alpha-emitters, are confined to the third floor and the basement, while the first floor radiometric laboratory is used for uranium and other natural radioactive materials.

## URANIUM

Since uranium formed the mainspring for the initial creation of the Section, its present position in the programme of the Subdivision is first considered. Although the demand for radiometric assays of uranium and thorium in ores has decreased sharply in the past few years, the facilities are maintained and a few assays are still run each year (e.g., G 35, 11, 12, 15, 118, 123). Contact is maintained, of course, with the old ore dressing section of the Radioactivity Division, now part of the Extraction Metallurgy Division, and some of the instrumentation development work was initially directed to assist uranium leaching (A 37, 2, A 57, G 23, G 24, G 25, H 93). Some pieces of equipment, such as those developed for electronic sorting ("picking") of uranium ore (3) and for control of acid concentration by conductimetric means at several uranium mines, are the direct outcome of the Subdivision's work.

A major project, which has demanded considerable time and effort, is the determination of ionium (Th-230) in uranium concentrates by alpha-ray spectrometry. This isotope of thorium is a uranium decay product occurring naturally in uranium ores. Since current uranium contracts specify an ionium content of not more than 1 part per million in concentrates, a specific method of detection is obviously useful.

However, its alpha-ray emission, the only specific identifiable property, is so close in energy to that of uranium (U-234), that resolution by alpha-ray spectrometry at the low intensities encountered poses considerable difficulties and demands extreme stability of the equipment and very thin, reproducible sources (Figure 3). A complete solution of this problem has always been "just around the corner", but it is seriously expected that a satisfactory solution will be reached now that a more stable pulse-height analyser with up to 100 channels has become available for this project. An earlier related project concerned the determination of Pa-231 in uranium solutions (G 25).

In the past few years several major programmes have been under way at the Mines Branch to find new uses for uranium outside the nuclear energy field. To this end, many tests have been run in the Physical Metallurgy Division to explore the properties and potential uses of ferrous and non-ferrous alloys containing varying amounts of uranium, usually as a deoxidant (F 16, 19). The Subdivision has been involved in this programme in three subsidiary, but important areas: uranium analysis, health monitoring, and trace impurity determinations.

The work on uranium analysis has been a natural outgrowth of earlier work on the gamma-ray spectra of uranium and thorium, particularly in the low-energy region. This led to the development of a graphical method for the rapid determination of uranium and thorium, mainly in ores, by means of a "kicksorter" (pulse-height analyser) (B 9, A 38). The high cost of the latter has restricted the wider use of this method, but from it has developed, gradually, a very rapid and convenient method for the determination of uranium in alloy samples (A 49, D 8, E 62). In this method the alloy sample, in the form of drillings compressed into a pellet, is counted with a scintillation detector and a single-channel pulse-height analyser, providing a rapid and relatively inexpensive means of obtaining control analyses. Such a unit has since been set up adjacent to the Mines Branch foundry and is in constant use. By careful calibration it has been made direct-reading.

Another convenient use of counting methods has been the application of a portable Geiger counter to check the uniformity of distribution of uranium in steel and alloy bars and billets, by measuring the local beta-ray activity over the length of the billet (H 91, I 3, G 25, H 95).





Figure 3. View of alpha-ray spectrometer.  
(Ionization chamber at extreme left, 18 channel  
kicksorter at right).



Figure 4. Air monitoring during experimental casting of uranium-steel heat.

The health and safety aspects of the uranium programme are absolutely vital to it, if the public is to accept the use of uranium-containing alloys for everyday use. For this reason it has been necessary to conduct tests for external radiation from finished and partly-finished uranium alloy materials and to obtain air samples in plants and melt shops during all stages of melting, pouring, rolling and cleaning of the metal, to confirm whether or not the concentration of airborne uranium was within permitted limits (Figure 4). This has entailed frequent sample runs and decontamination tests in the Mines Branch laboratories (I 3, H 70, H 97, H 98, H 100, H 105, H 111, H 116, I 9), as well as visits to all the Canadian plants that have participated in this programme (G 29, G 30, G 31, G 32, G 34, G 39, H 95). Coupled with this, there has been a certain amount of public relations work to overcome public misconceptions and apprehensions, and to clarify relevant technical and legal aspects of the use of uranium as an alloying material (A 44, A 55, B 15, B 21, D 7, F 16).

#### RADIOMETRIC RESEARCH AND DEVELOPMENT

It is convenient to group together under this heading some of the work that has been done on the use of neutron activation and gamma activation analysis, and of gamma-ray spectrometry on materials other than uranium.

Of these, the gamma-ray activation analysis of beryllium has been in use for some years following the initial suggestion of Senftle and Gaudin and the development of the "beryllometer" by Brownell and his associates. The beryllometer is intended essentially to be a portable, or, more correctly, a movable, unit employing a strong antimony-124 source. When the need arose at the Mines Branch to obtain accurate and rapid beryllium assays on beryl ores and concentrates, it was decided to set up a fixed laboratory facility. The outcome of this decision has been the development of two units, one for the screening of coarse ore samples (I 8) and one for more precise assays on powdered samples (E 64). This equipment has worked well, is easy to operate, and has found frequent use (I 6, I 7, I 8, I 10, I 11, I 22, I 30).

Neutron activation analysis as a possible industrial method is of relatively recent origin. In the past, many papers have been published of spectacular determinations of certain elements in sea water or high-purity metals, or on such exotic samples as Napoleon's hair or the liver of King Eric XIV of Sweden. In all these cases a nuclear reactor had to be used, which made the method too slow and expensive to be of genuine interest to industry. For this reason, work at the Mines Branch was



confined to the few possible applications in the mining industry where one could employ portable radium-beryllium neutron sources (A12, H3). Because of the low neutron flux available, such applications are too limited in scope; however, the recent advent of portable neutron generators of the accelerator type has changed that picture. The flux available is of the order of  $10^9$  fast neutrons/cm<sup>2</sup> or  $10^8$  thermal neutrons/cm<sup>2</sup>, high enough to open up a wide range of useful activation reactions that may be of interest to industry (E66, B16, H128). In addition, the availability of a flux of fast, highly energetic (14MeV) neutrons makes it possible to utilize some activation reactions not usually accessible to reactor users. Of these the most important is the  $O^{16}(n,p)N^{16}$  reaction, which permits the non-destructive, rapid determination of total oxygen in many metals and minerals (H122, I13, I15, I16, I17, I19, I20, I21, I29, I32, etc.).

The setting up of the neutron generator posed many problems, since at the time the generator was purchased, in the spring of 1961, only fragmentary information was available on the amount of shielding required, in spite of discussions with other Canadian users of such machines. As mentioned earlier, the equipment was finally set up in the basement and a concrete block shield erected around it with an entrance maze. The bricks were set up in staggered rows to avoid radiation leaks. The roof consists of a solid layer of heavy planks, assembled edge up, on which there is a further layer of concrete blocks, crowned finally by five nylon wading pools filled with water, an excellent neutron absorber. These pools were mentioned in a news release and rather dominated any mention of the equipment in the press, often to the exclusion of the real purpose of the equipment (e.g., 15, 16). This shielding lowers the neutron intensity around the outside of the shield to acceptable levels; however, there is still a sufficiently detectable background of radiation while the machine is running, so that it has been necessary to install an interlock switch to cut off sensitive counting equipment, three floors up, while the generator is running, to avoid spurious counts. Another interlock cuts off the generator beam, while the counter in the generator laboratory determines sample activity.

Because of the short half-life of some of the induced radioactivities, a rapid, pneumatic transfer system ("rabbit") has been installed, which permits the automatic transfer of samples into the irradiation position while the generator is operating, and their subsequent transfer into the counting assembly within a fixed short time interval. This has greatly improved assay accuracy, but the design of this facility has evoked considerable ingenuity in the staff concerned and it is now one of the show pieces of the laboratory. An extra coaxial cable has also been installed to permit feeding spectrum information from the scintillation detector in the basement laboratory to the gamma-ray spectrometer on the third floor. It is hoped to issue a detailed description of the whole facility shortly.

The main purpose of the neutron generator programme is not so much to do routine activation analyses by what may be a faster method, as to explore the feasibility of assaying certain elements of industrial interest where alternative methods at present are either too slow or too inaccurate. Since the cost of the equipment is comparable with that of an X-ray fluorescence system or an automatic direct-reading spectrometer, one can envisage an ultimate use of neutron generators in industry on a sizeable scale. Before this becomes possible, it is obviously necessary to explore such factors as sensitivity, accuracy, likely interferences from other elements, speed of determination, and so on. For this reason, a wide variety of samples has been studied and analysed, with the aim of presenting to industry, ultimately, a series of recommended procedures for analyses that may be of practical use and interest.

In the meantime, the gamma-ray spectra, half-lives and cross sections of irradiation are being studied to establish the reasons why many samples do or do not respond to the degree expected. Among the samples studied have been determinations of Si, Al and Mn in iron ore; Ag in bismuth telluride; Hg and Au in ore samples; Au in sands (G 40); O<sub>2</sub> in niobium salts (I 13, I 17), selenium (I 15), iron (I 16) and minerals (I 21, I 29); N<sub>2</sub> in graphite (I 33); Hf in zirconium (I 25); and other trace elements in barium titanate (I 24). Iron and aluminum alloys and products have been studied, as well as various semiconductor compounds. This is clearly a growing field and one of particular interest to industry in view of the rapidity and non-destructive nature of the analysis and the relative ease of operation of the equipment.

The proper evaluation of the activity induced in an unknown sample by neutron bombardment involves the intelligent use of the gamma-ray spectrometer. This equipment has been greatly improved over the years as a better detector assembly was built up and as improved kicksorters have become available (Figure 5). The most recent improvement has been the acquisition of a Nuclear Data Model ND 130 A pulse-height analyser, which can perform many of the computing functions directly and thus save a considerable amount of time and effort.

To a great extent the gamma-ray spectrometer has been pre-empted by the needs of the neutron activation project. However, alongside that project a specific gamma-ray spectrometry programme has been carried on. The most ambitious project here has been a co-operative research, shared with Dr. A.F. Gregory of the Geological Survey of Canada, on the degradation and changes observed in the gamma-ray spectra from natural uranium, thorium, and potassium in minerals and rocks on passage through long distances in air. This research has obvious applications in the aerial surveying for radioactive minerals. The field work to obtain data was done in the summer of 1959 at a radio



site with a 200 ft-high tower with an elevator, on which the detector and kicksorter could be raised or lowered (H62). The radiation sources included a truck load of bagged pitchblende concentrate and another of thorite concentrate. The results of a parallel laboratory study on synthetic ore sources, to obtain data on self-absorption effects and detector parameters, were published in 1961 (F12, B17). The results on the field experiments were reported recently (F19) and represent an exhaustive study of all factors involved in evaluating aerial survey results.

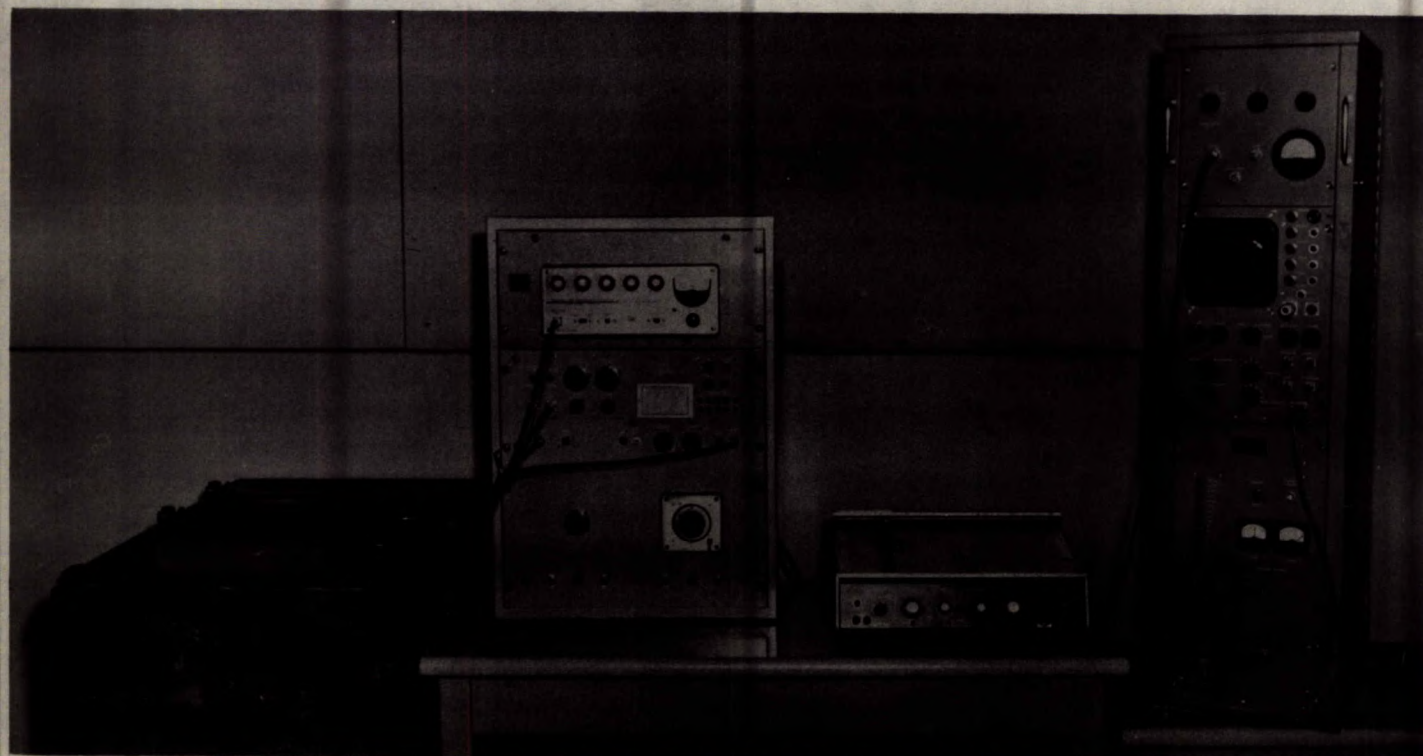


Figure 5. View of gamma-ray spectrometer (with CDS 100-channel kicksorter).

Other important gamma-ray studies of more restricted interest have been an extension of previous results on the composition of fall-out products (A 32), on ore samples (A 38, B 9), on flue dust samples (I 3), on West Coast plant samples (G 27), and on the identification of various radioactive isotopes in decontamination swabs.

In this connection it may be mentioned that the assay of air samples collected on the roof for gamma-ray fall-out activity has been continued, but, however, at a diminishing rate in more recent years as the amount of more systematic, published data has grown.

It is also worth noting that the earlier work in the Subdivision on the setting up of radiocarbon dating equipment, which was taken over by the Geological Survey of Canada, is now beginning to bear fruit (5).

#### INDUSTRIAL TRACER APPLICATIONS

The use of radioactive isotopes as a means of following the course of feed materials and solutions through industrial plant systems has been well established in the past few years. Nevertheless, it is not employed as widely as it deserves or would be desirable, in view of the almost complete absence of detailed operational data that seems to prevail at many metallurgical plants (A 51, A 36). The reasons for this situation are manifold, but the principal ones can be summarized easily. The first factor is an inherent reluctance to try a new and unfamiliar technique. This unfamiliarity is often coupled with many popular misconceptions fostered by the popular press, e.g., that radioactive isotopes render other materials radioactive, or that they invariably induce sterility. Added to this is a lack of knowledge about the handling of radioactive materials and the consequent problem of meeting legal requirements for an isotope licence.

To overcome this situation, members of the Subdivision have been available for several years to conduct or supervise the use of radioactive tracer tests in industry. Discussions have been held on possible uses of tracers (e.g., H 56, H 66, H 71, H 94, H 130), and the cost of such consulting work has been kept to a minimum by charging only for out-of-pocket travel expenses and, occasionally, for the supply of the radioisotope. In most tests two members of the Mines Branch staff have gone to the plant with all the necessary counting and monitoring equipment, have advised on sampling, and have operated the counting equipment. The staff of the host plant in most cases followed their usual operating routine



except for the collection of extra samples. Many useful tests have been run in this fashion and it is to be hoped that many more companies will avail themselves in future of this valuable service.

The largest number of tests run in the past four years concerned the measurement of residence time in kilns and furnaces both for industry (G 22, G 28, G 37) and for Mines Branch tests (H 87, G 21). Another important field was in the measurement of retention times, recirculation times, and the losses encountered in leach circuits (G 23, G 26). Particularly interesting, though laborious, was a detailed test to establish the source of underground water seepage at the Steep Rock mine, where tritium (hydrogen-3) was used as the tracer (G 36, A 50). A tremendous number of samples had to be treated and, because of the low concentrations involved, special methods of preconcentration and counting of the tracer had to be developed.

A project having both practical and theoretical implications was the tagging, with a tracer (E 58, E 60), of a heat of steel that was used for casting grinding balls. The purpose of this was to enable one to follow the relative wear properties of different types of balls under actual operating conditions (H 67). By sampling the ball mill at weekly intervals, it was also possible to obtain information on the wear rate of the balls and to confirm that equal thicknesses grind off in equal times (A 40a). This approach has many useful applications, and more general employment would yield much badly-needed information on ball-mills.

Another pilot plant operation in which tracers were used to advantage was in the extraction of cesium from pollucite (18). In this case the use of  $Cs^{137}$  made it possible to determine extraction and breakthrough conditions during the operation of the plant (4).

#### LABORATORY USES OF RADIOACTIVE TRACERS

Apart from the use of radioactive tracers in operating plants, it is obviously useful to employ them also on a smaller scale in a variety of engineering laboratory applications. The next section will deal with their use in radiochemistry.

The previous report has already mentioned earlier work on a viscometer for liquid metals by the falling-ball method (H 61), and its extension to slurries (A 31, A 31a, E 57). A related method has also been used to measure the fall time of iron ore particles in a reduction tower (H 90). A preliminary study of the kinetics of dephosphorization of iron with

$P^{32}$  as the tracer (H 82) has been taken up more recently as the basis for a larger research project at McMaster University.

The need to develop a flow gauge that could handle fast-flowing aircraft fuel at high pressures led to a design of an injector for the introduction of a tracer into the stream and its almost immediate precise detection (G 20).

At this stage one may mention the extensive research programme of Dr. F. Weinberg and his associates in the Physical Metallurgy Division, Mines Branch, on solidification and grain boundary segregation phenomena. Various radioactive tracers have been used, supplied through the Sub-division; by counting and autoradiography, this work has yielded much valuable information not otherwise obtainable (e.g., 6, 7, 11, 12).

## RADIOCHEMISTRY AND RADIATION CHEMISTRY

The radiochemical work in the Division is essentially of two kinds: surface chemistry, with radiotracer tracers used to indicate the strength and nature of adsorption phenomena; and analytical chemistry, where tracers are used essentially to provide quantitative information on the yield and extraction rates of separation processes. In principle these procedures are fairly straightforward and require only very small amounts of radioactive tracer material.

In the analytical field there have been a few special separations where the use of tracers simplified operations (e.g., H 103). The largest research programme in this field is being carried out by Mr. G.H. Faye, Analytical Chemistry Subdivision, who is housed adjacent to the Tracer Laboratories (8, 14) under the direction of Mr. W.R. Inman, Chief Chemist. He has studied in detail the assaying and separation of the precious metals, particularly the platinum series (9, 10). For this purpose, a fire-assay method using tin as collector has been developed and various isotopes have been used for quantitative tests.

Another programme of an essentially analytical nature has been that of Dr. Manning, an NRC Postdoctorate Fellow, who is interested in the complexing of the rare-earth elements and their separation by solvent extraction. Through the use of radioactive tracers the separation constants have been obtained for a few typical reagents and the effects of steric crowding have been studied (A 56, A 61, B 19). More recently he has investigated the formation of tartaric acid complexes and has shown that the measured stabilities require the formation of hydrogen bridges between some of the tartrate molecules (A 68, H 131), theoretically a rather important deduction.



The surface chemistry work, too, follows a number of different lines. Conceptually, the simplest have been the uses of tracers for the measurement of surface areas of fine metal and mineral particles, particularly for those ranges of surface area that are not readily accessible to the more widely used gas adsorption method (A 41). The method has been used for magnesium powders (A 35, F 7, F 10), uranium oxide powder (A 45, F 14), and various minerals (F 18, A 28). The adsorption of chromic acid on glass has been measured (A 48) and some studies have been made of anion adsorption on sulphide minerals (F 13).

Another illustration of the use of tracer methods in beneficiation tests has been a study of the adsorption of gold on carbon particles in certain gold ores, which interfered with proper cyanide dissolution of the gold (H 101). By the use of radioactive gold it was shown that this effect could be reduced most efficiently by roasting of the ore.

Closely linked with this work has been a continuing programme on the kinetics of adsorption of ions in solution on metal surfaces and their subsequent diffusion or re-dissolution. Initially, this research dealt with polycrystalline zinc (A 42, F 8) and silver (A 46, F 11) and their ions in solution. More recently, this work has been extended to single-crystal silver (H 119) and nickel, with interesting results. Some aspects of this work may have some bearing ultimately on plating and corrosion phenomena.

Another facet of surface chemistry research of special interest to the mining industry is the work on the adsorption of flotation reagents on minerals. Although flotation is widely used in mines throughout the country, the basic mechanism is only imperfectly understood and more economic operations may result from a closer study of the underlying phenomena. Because of difficulties encountered in the separation of hematite and quartz by flotation, much of the work has so far concentrated on the adsorption of oleic acid on those two minerals (F 18, H 127). This work has entailed the development of many specialized techniques and, more recently, a demonstration of the formation of iron oleate in oleic acid adsorption on hematite. Extension of this work to study the adsorption of xanthate is planned for the near future.

A very interesting project in surface chemistry, but not one involving tracers, was that chosen by Dr. Donato, an NRC Postdoctorate Fellow. He studied the effect of neutron irradiation of zinc oxide and chromium oxide catalysts used in the thermal decomposition of ethyl alcohol (H 115, F 17, A 53, A 63, B 24). Whereas usually irradiation merely reduces the catalytic efficiency of oxide catalysts, in the case of zinc oxide a change in catalytic behaviour occurred, which was shown to be related to the controlled introduction of a specific impurity (gallium) by neutron activation.

## CONTROL INSTRUMENTATION DEVELOPMENT

A more immediate impact on industry than that from tracer work may be expected from the efforts to develop instrumentation to improve the measurement and control of essential variables in plant operations. Here again a government laboratory finds itself in a slightly anomalous position. In theory it should be an easy matter for an instrument manufacturer to develop a device or some equipment and to sell it directly to the mining and metallurgical industry. In Canada things do not seem to work out quite so simply in practice. In contrast to the petroleum industry, which is highly instrument- and control-conscious, the mining industry in general is very conservative and has to be shown that instrumentation and closer control are profitable. Since few of the instrument manufacturers in Canada have the staff or facilities to run such tests under the appropriate conditions, the burden of this demonstration tends to fall on the Mines Branch. Not to undertake this task would mean a further slow-down in the long overdue modernization of many mills, which must face closer control of feed and operating conditions if they are to meet foreign competition (B 8).

Another aspect of this problem is the frequent lack of suitable instrumentation or of equipment adapted to the special needs of the mining industry. Here it is, in many cases, the equipment manufacturer who has held back, before committing production staff and facilities, until a ready and receptive market has been established. Again it has fallen to the Mines Branch to undertake much of the necessary missionary work and to provide the bridge between supplier and user.

The group that has attempted to fill some of these needs has been the Subdivision, working closely with the field officers of the Extraction Metallurgy Division. After establishing its objectivity and genuine desire to be of help, the Mines Branch staff has slowly been accepted by both sides as a valuable ally and interpreter.

Of the various instruments dealt with in the past few years, the one likely to have the greatest impact in Canadian mills is the conductimetric probe (B 11, E 63, A 39, A 52). The use of conductimetric methods in leach circuits is by no means new, but the usual small cells with platinum electrodes were ill adapted to mill solutions with dense slurries and abrasive particles. Immersion probes had been described in the literature, mainly for oceanographic work, but there were none on the market that seemed to meet the needs of the mining industry. An extensive effort was devoted to the development of a simple and rugged device, and pilot tests on many leach liquors were conducted at the Mines Branch (G 37, G 42, G 45) and in various plants (G 43, G 44, H 93, H 126),

using both acid and alkaline solutions. It was shown that conductimetric control is advantageous, in many cases, at very high and very low pH values, where most pH instruments are relatively insensitive, and for use under conditions where extreme ruggedness and low maintenance requirements are important. Figure 6 shows a conductimetric probe (suspended, centre) installed in the control demonstration circuit set up in the Extraction Metallurgy Division.

A very specialized device that has been developed for the gold mines is a gold leaf dissolution strength monitor (E 67). In this unit, which is shown in Figure 7, the test cell is filled with the solution under test, either statically or as a flow cell, and the dissolution strength is obtained from a graph of parallel lines produced as a gold foil sample is moved through a light beam in front of a photosensitive detector. As the foil is attacked by the cyanide solution, it becomes thinner and more transparent and the photocell voltage displayed on the recorder becomes progressively larger. The rate of growth of the lines provides an indication and record of dissolving strength.

In connection with the treatment of uranium waste solutions, a special monitor was devised to measure uranium content continuously (A 37, G 24). In this system the solution passes through a plastic cell containing several plastic scintillator sheets in parallel spaced layers. The fluorescence of the scintillators due to the uranium alpha-ray emission is detected and fed to a ratemeter circuit.

Another device, which originally grew from the uranium technology, arose from Gilmore's observations on the natural fluorescence of ion exchange resins (2). From this discovery derived a fairly detailed study of fluorescence phenomena of ion exchange resins and the quenching effects observed under loading and eluting conditions (F 15, A 57, A 60, H 121). It was shown that, for certain ions, observation of fluorescence emission is closely linked with loading and elution, and may provide a basis for rapid, automatic control, especially in the elution cycle.

Two instrument developments of interest to the iron ore producers may be mentioned here. The first concerns a simple magnetite assay unit (A 65, B 23) that has been used to control iron feed and kiln products (H 86, and later ones). In this case the magnetite content is related directly to the change in inductance of a standard coil. The other project is connected with attempts to develop a detector for large tramp metal pieces, such as steel bucket teeth and drill bits, in high-grade iron ore. Earlier tests based on a modified commercial metal detector were quite satisfactory (H 99, H 104); however, when the grade of the iron ore was increased appreciably, an electromagnetic detector proved inadequate. Test work is under way to attempt to solve

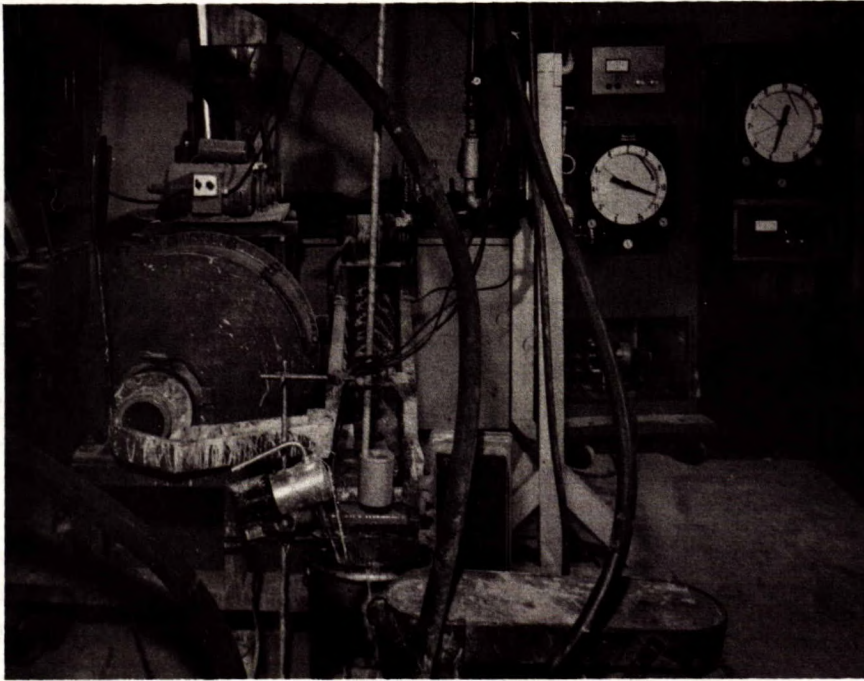


Figure 6. Demonstration grinding circuit with conductivity probe in the centre.

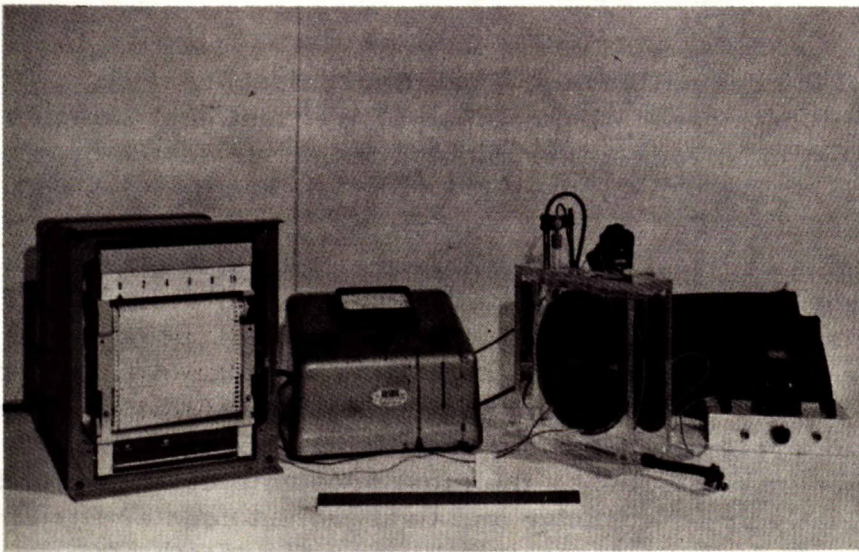


Figure 7. Gold leaf dissolving strength monitor.



this problem, which is of surprisingly widespread interest, by saturating the magnetic ore in a strong d-c magnetic field and then detecting the metal pieces by eddy current induction.

Other industrial monitor projects in hand at this time involve the development of a slime level detector for thickeners, an adaptation of a commercial slurry density gauge, and the development of a xanthate concentration monitor for flotation circuits.

A field of traditional interest to the Subdivision is the electronic sorting of ores, dating back to the early Lapointe picker (C1). Although the decreased activity in uranium mining has reduced demand for work on this subject, any development, notably the K and H picker at Bancroft, has been followed with interest (H49, H124). Following earlier pilot plant tests by Subdivision staff at Beaverlodge (H54), a complete picker system was finally installed there (3). Other sorting possibilities have also been explored, and a programme is under way at present to develop a method for sorting coarse molybdenite ore from waste, and possibly for tin ores (if they are not too friable); by the use of characteristic X-ray fluorescence excited by low-energy gamma-rays. Preliminary results look very encouraging for this process, which may have important consequences for the mining industry.

#### DEVELOPMENT OF LABORATORY INSTRUMENTATION

As part of the surveillance programme on dust levels in mines under the silicosis prevention campaign, frequent dust samples are collected at most hard rock mines, which are then evaluated by a dust count. In Ontario the "konimeter" has been adopted as the standard instrument for dust sampling. In this device a fixed volume of air is sampled and the contained dust is sprayed upon an adhesive glass disk. After heat treatment these disks are examined under a microscope for counting. Since visual counting is slow, fatiguing and inaccurate, the Mines Accident Prevention Association of Ontario has placed a contract with the Mines Branch for the development of an electronic counter for this purpose. After preliminary tests with a flying-spot scanner, a Vidicon camera tube was selected as the most economic device around which to build a relatively inexpensive and light-weight counter system (G41). Work is in progress at present to design and assemble a prototype unit.

Another electronic development in which the Subdivision is taking part is the design and assembly of an electron beam microprobe analyser. This project is under the direction of Dr. A.H. Gillieson, the chief spectrographer, with the Subdivision undertaking the design of the electronic power supplies and sweep circuits. Although several commercial units are available on the market (e.g., H113), the Mines Branch unit is expected to cost less and to have a number of different useful features. The Subdivision has also assisted the spectrographic laboratory with some electronic problems in conjunction with the redesign of the Mines Branch cathode-ray comparator-densitometer (17).

Other contributions of the electronic laboratories that may be mentioned here are the redesign of the circuit of the Thomas oxygen determinator (H 75) and a photoelectric counter unit for the timing and recording of galvanometer beam deflections in dilatometry (A 43). Development work has also been started on an electronic unit to measure the reflectivity of minerals (H108). In addition, from the early days of transistor development, transistor circuits have been used and developed for a variety of purposes, and the writer has participated in a responsible advisory capacity on developments in research and industry in the semiconductor field.

## SEMICONDUCTOR RESEARCH

This subject has been developed over the years by a union of the Subdivision's electronic and radiotracer interests and experience. Although the work was originally undertaken as a pure research programme, at the present state of the art much of the work done is of definite interest to industry (H125).

Research so far has concentrated on bismuth telluride, a semiconductor compound of particular interest for its thermoelectric properties. After much experimenting, procedures have been devised for the growing (Figure 8) and sectioning of  $\text{Bi}_2\text{Te}_3$  single crystals (e.g., H 77, H102, H120). The diffusion of gold and silver along the two major axes has been measured, as well as their solubilities in bismuth telluride (A 54, A 64, A 66, B 22, B 26). Work is under way to study the diffusion of tin. In addition, Dr. Keys is studying some properties of gallium arsenide while on an exchange visit to the Semiconductor Institute in Leningrad. During his absence a long-term ion drift experiment in bismuth telluride is being maintained.





Figure 8. Zone melting furnace for the growing of bismuth telluride crystals.

### CONCLUSIONS

The purpose of this report is to summarize the activities of a highly specialized laboratory in the field of physics and chemistry as applied in the metallurgical industries. In spite of the limited staff and funds available, it is evident that it is possible for even a small group to make useful contributions to the continued development of the Canadian mining industry, an industry greatly in need of intensive research and development work if it is to meet successfully the challenges of the immediate future. There is clearly room and need for such research at a central government facility to aid especially the smaller and undeveloped mines that would otherwise not have the means to engage outside consultants. Nevertheless, it is to be hoped that particularly the instrumentation and radiotracer fields will attract and maintain private consultants and manufacturers, who are willing and able to stake their resources on the continued development and expansion of this vital Canadian industry.

### ACKNOWLEDGEMENTS

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APPENDIX A

STAFF OF THE PHYSICS AND RADIOTRACER SUBDIVISION,  
1959-1963

A. Scientific Staff

Geoffrey G. Eichholz, B.Sc., Ph.D. (Leeds)	1951-
Christian M. Lapointe, B.Sc., D.Sc.(Laval)	1946-
John L. Horwood, B.A. (Toronto)	1947-
John D. Keys, B.A., Ph.D.(McGill)	1958-
Hugh P. Dibbs, B.Sc. (Manchester), Ph.D. (London)	1958-
José E. Sandor, D.Sc.(Buenos Aires) (NRC Fellow)	1958-1960
Pietro R. Gorla, Ph.D. (Turin) (NRC Fellow)	1959-1960
Matteo Donato, M.Sc. (Turin), Ph.D.(Milan Polytechnic) (NRC Fellow)	1960-1962
T. Richard Flint, B.Sc. (Alberta)	1960-1962
Irvine I. Tingley, B.A., M.Sc. (Dalhousie)	1961-
Philip G. Manning, B.Sc., Ph.D. (Wales)(NRC Fellow)	1961-
Ronald H. Goodman, B.A. (Sask.), Ph.D.(McMaster)	1963-

B. Technical Staff

Charles McMahan	1946-
Arsène H. Bettens	1951-
Jean M. Lefebvre	1951-
Gordon E. Alexander	1953-
Allan F. Seeley	1953-
Joseph V. Krzyzewski	1954-
Clifford A. Josling	1955-
Jan Kroon (on loan from Eldorado)	1962

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Miss Barbara Moore, Secretary	1960-
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C. Summer Assistants (to May 1, 1963)

J. Robert Barkley (Carleton)	1957, 1958, 1959
J. Claude Filion, B.Sc.(Montreal)	1959
Robert M. Hopwood (Carleton)	1959
Robert A. Wilkinson (Queen's)	1959
Laurie S. Wright (Queen's)	1960, 1961
T. Richard Flint, B.Sc.(Alberta)	1960, 1963

Peter M. Manning (Brit. Col.)	1960
David J.A. Johnson (Queen's)	1961
Louis G. Legall (Alberta)	1961
Henry M. Dutton, B.Sc.(London), M.Sc.(Western Ontario)	1962
Herbert Inhaber, B.Sc.(McGill)	1962
Frederick M. Dampier, B.Sc.(Manitoba)	1962
Akos Halmy (Toronto)	1962
Pranowo (Jogjakarta)	1963 (April)
Sandra Adams (McGill)	(1963)
Ray C. Klump (Manitoba)	(1963)
David Fisher (Carleton)	(1963)

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