PREFACE

Seven papers were prepared by the Department of Mines and Technical Surveys for the United Nations Conference on the Application of Science and Technology for the Benefit of the Less Developed Areas, which was held at Geneva, February 4-20, 1963. The titles of these papers are listed on the last page of this report.

Since the United Nations does not plan to publish the papers from this conference, the department decided that its contributions should be reprinted in the usual departmental reports series. Four of the Mines Branch papers are published collectively in this Information Circular. A fifth paper, Mineral Information Bulletin MR 66, is a joint contribution by the Mineral Resources Division and the Mines Branch.

John Convey, Director, Mines Branch.

May 9, 1963

PRÉFACE

Sept études ont été rédigées par le ministère des Mines et des Relevés techniques à l'intention de la Conférence des Nations Unies sur l'application de la science et de la technologie au profit des régions insuffisamment évoluées. La Conférence en question s'est tenue à Genève du 4 au 20 février 1963. Les titres des études apparaissent à la dernière page du présent rapport.

Étant donné que les Nations Unies n'ont pas l'intention de publier les études présentées à la Conférence, notre Ministère a décidé de réimprimer le texte des travaux de ses fonctionnaires dans le cadre des séries régulières des rapports du Ministère. Quatre des études de la Direction des mines ont été réunies et forment la présente circulaire d'information. Une cinquième étude, savoir le Bulletin d'information minière MR 66, représente une contribution conjointe de la Division des ressources minérales et de la Direction des mines.

John Convey, directeur des Mines.

le 9 mai, 1963

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Mines Branch Information Circular IC 149

PAPER 1

ANALYTICAL CHEMISTRY AND SPECTROGRAPHY AS ESSENTIAL CONTROLS FOR DEVELOPMENT AND PRODUCTION IN THE MINERAL AND METAL INDUSTRIES

by

W.R. Inman* and A.H. Gillieson**

ABSTRACT

The paper reviews the trends in the past twenty years and the present status of analytical chemistry in the mineral and metallurgical industries. Analytical requirements for prospecting, mining, process development and production are outlined. Modern instrumentation is discussed in some detail, with particular reference to requirements for analysing high purity compounds and complex mineral aggregations.

Plans for training personnel in analytical chemistry are considered.

RÉSUMÉ

Les auteurs passent en revue les tendances des vingt dernières années et indiquent la situation présente de la chimie analytique dans les industries des mines et de la métallurgie. Ils indiquent les analyses auxquelles il faut recourir dans les domaines de la prospection, de l'extraction, de la mise au point des procédés et de la production. Ils analysent en détail les instruments modernes, en insistant tout particulièrement sur les exigences que comporte l'analyse de composés très purs et d'agrégats minéraux complexes.

Les auteurs envisagent la possibilité de dresser des plans afin de pouvoir former le personnel voulu pour travailler dans le domaine de la chimie analytique.

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SUMMARY

1. The development of analytical chemistry as related to the mining and metallurgical industries over the last two decades is reviewed briefly.

2. The development of a mineral and metal industry falls into three phases:

- (a) discovery and mining of ore deposits;
- (b) development of processes for extracting the valuable minerals and metals; and
- (c) their economic production based on the work done in (b).
- 3. All phases require analytical control, with the highest skills and most sophisticated instrumentation required for phases (a) and (b). Obviously, it would be a vital part of the development to select the analytical methods that could be performed with the simpler, more rugged instruments whenever possible for the production phase. In fact modern plants should be designed, whenever possible to include in-stream analytical systems so as to have a continuous record of the quality of the ingredients and products.
- 4. Modern instrumentation utilizes a wide variety of methods for performing both simple and complex analysis; and instrumentation frequently offers the only feasible approach to difficult analytical problems. Among the analytical instrumental techniques used in a modern mineralogical and metallurgical research institution are emission spectrography, X-ray spectrometry, electron probe microanalysis, flame emission and absorption photometry, spectrophotometry and polarography. Radiochemical techniques are useful in certain problems and can be done with relatively simple and inexpensive equipment.
- 5. In underdeveloped areas the availability of trained and skilled personnel may be a limiting factor in the efficiency of the analytical laboratories. It is considered that the preferable approach to this problem is, firstly, to have an expert work in the country, setting up the laboratory and training those who will be engaged in the analytical work; secondly, the most promising of these should be sent to the country of the foreign expert for advanced study and training.

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INTRODUCTION

1. Development of Analytical Chemistry Instrumentation and Spectrographic Techniques and Apparatus in the Past Two Decades

(a) Prior to World War II the mining and metallurgical industries were able to cope with their analytical problems by means of the slow methods of classical analytical chemistry. There were, however, a number of laboratories in universities and government research institutes where instrumental methods were being investigated. This is true even of emission spectrography and polarography which, before the war, were practically unknown in industrial laboratories.

(b) The enormous demands on production of both well-established and new materials during the war brought sharply to notice the importance of analytical chemistry and the need for analytical control. It is true to say that one major wartime enterprise, involving the production of fissionable material, nearly foundered through initial lack of appreciation of the analytical effort and problem involved.

.(c) The tremendous wartime and post-war increase in the number of chemical analyses required, found the western world acutely short of skilled analysts. In order to make up for this shortage, the development of instrumental methods, which were capable of operation by the semi-skilled technician, was greatly accelerated as the only possible solution to this urgent problem.

(d) Novel analytical techniques previously only practised by research workers in universities and research institutes soon became commonplace necessities in the industrial laboratories. In the past two decades widespread adoption has occurred of the polarograph, the spectrophotometer, the flame photometer, the infra-red spectrometer, the direct-recording spectrograph and the X-ray fluorescence spectrograph. Recently the vacuum spectrograph, the gas chromatograph, the electron probe microanalyser and mass spectrometer have entered industrial research laboratories.

(e) In emission spectrographic analysis alone, the novel techniques of atomic absorption, carrier distillation, arc stabilization, the plasma jet and the application of the hollow cathode have been put to increasing use during the last twenty years.

(f) Analytical chemistry has been extended and enriched by applications and analytical developments of solvent extraction and ion-exchange methods and by the techniques of radiochemistry and isotope analysis. (g) These new analytical tools have resulted in progress in the mineral and metallurgical industries. In the petroleum industry the advent of the infra-red spectrometer and the gas chromatograph have made possible analyses that previously were well-nigh impossible. New purifying techniques such as zone-refining have produced metals of hitherto unobtainable purity and the need for their analysis has resulted in many refinements of analytical techniques. The metallurgist has thus been provided with alloying constituents of a purity such that the effect of very small traces of impurities on the properties of alloys could be studied for the first time. The opportunity for the microanalysis of minerals and metals has been greatly increased by the introduction in recent years of the electron probe and its application to the identification of microstructure in alloys and minerals, and to the analysis of inclusions in metals and of corrosion products.

(h) It is obvious from the foregoing that the development of mineral resources and metallurgical industries by underdeveloped nations should not be hindered by any great difficulties in the field of analytical chemistry, provided that money is available for the purchase of modern equipment and that trained staff can be obtained. It is beyond the terms of reference of this paper to discuss the financial aspects, but staff training will be considered in a later section.

2. Present Status of Analytical Chemistry in Support of Mineral and Metallurgical Industries

(a) In the economic development of mineral resources from initial prospecting through process development to successful production, analytical chemistry plays an indispensable part. Through the years the improvement in methods of prospecting, the development of new and more efficient processes for winning metals from their ores and the production of high quality metals and alloys of constant composition, have been paced by advances in analytical chemistry.

(b) Prospecting

(i) The first phase of prospecting is the detection of valuable minerals. The modern prospector has available a number of physical and chemical aids. For example, small portable chemical kits have been designed which use simple methods for the estimation of valuable elements in water, vegetation and rocks. In the geochemical laboratory semi-quantitative results by spectrographic and chemical techniques permit the examination of a large number of samples in a relatively short time. Mobile laboratories have been developed that can be fitted with modern chemical instruments including the emission spectrograph, flame photometers and spectrophotometers. With such equipment available it is possible to investigate an area much more rapidly than in the past. All these facilities enable modern prospecting teams to select more readily the particular mineralized locations that hold promise of becoming mines.

When the potentially valuable areas are localized, and more in-(ii)tensive exploratory work is undertaken, more accurate analyses become necessary. Economically valuable metals, such as gold and platinum, rarely occur as high grade ores and very accurate trace analysis is necessary in the evaluation of their occurrence. When it is realized that one ounce of gold per ton of ore, or three one-thousandth of one per cent constitutes a rich ore, and one-fifth of that amount may be an economic ore, the necessity for accurate assays in the prospecting phase can be appreciated. Other elements such as platinum and rhodium normally occur in ores in even lower concentrations. Similar requirements arise with a number of strategic metals such as molybdenum, tungsten, niobium and uranium, which normally occur in the range of 0.1-2.0%. The list of elements of economic interest and value is steadily increasing; recently lithium, caesium, beryllium, germanium, niobium, zirconium, uranium and rare earths have all advanced from the status of purely academic interest to being materials of economic importance. Due to their chemical properties, these metals are difficult and tedious to determine in their ores by chemical methods. Instrumental methods are therefore necessary to make rapid and accurate determinations of these elements.

(c) Process Development

(i) When it has been established that ore occurs in sufficient quantity and grade for establishing a mine, the selection of the most efficient methods of beneficiating and otherwise processing the ore is undertaken.

(ii) In process development, particularly for complex ores, the knowledge and skill of the analytical chemist are at a premium. He is presented with compounds where the elements may be in unusual associations and in ratios that make separations difficult. Furthermore, due to the associations, segregation, resulting from gravity differences, may be a complicating factor.

(iii) All techniques and instruments in the field of inorganic analytical chemistry are required to support research in mineral processing, mineralogy and metallurgy. Emission spectrography, X-ray diffraction, X-ray fluorescence spectrometry and electron probe microanalysis are employed to reveal the distribution of the minerals contained in an ore and of the elements within the mineral grains. Wet chemical methods, electrometric techniques, flame photometry, spectrophotometry and fire assaying methods, in addition to those above, are used for the specific accurate assays required.

(iv) Although beneficiation of the ore is usually a physical treatment such as flotation or magnetic separation, the assessment of the efficiency of the operation depends upon the analytical laboratory. Not only should the analyst be competent and ingenious, but he should have an appreciation of the problems of the mining and metallurgical engineer. In this way he will not only provide the assays requested, but be in a position to suggest and perform other assays that might assist with the elucidation of the problem. In effect, when the ore-dressing specialist and analytical chemist are working as an understanding and integrated team, the best conditions for research are achieved.

(v) Pyrometallurgical processes for the reduction to metal, purification by liquid-liquid extraction in the uranium industry, pressure leaching, ion-exchange as used in rare earth separations, and distillation procedures for the separation and purification of volatile metals, all require analytical control to ensure efficient operation. Observing the increasing number of processes involved in modern metallurgy, and the wide variety of products, it is readily seen that the analytical services must be versatile. This is particularly true in the development stage. The premium on high-quality products makes it imperative that the process development be directed to this end. Conversely the "penalty metal" clause can render uneconomic a process that fails to remove the undesired impurity. Control of these aspects of process development is a function of the analytical chemist.

(d) Production

(i) Once an economic process has been developed its application to production requires continued analytical control. The production analyst may not have to contend with the wide variations in composition of materials that face his colleague in the development field, but the crude ores or concentrates for the production process may still, unavoidably, be far from constant in composition and require careful, accurate analysis to guide the process engineers in the modifications necessary to treat raw materials of varying composition.

The product is normally held to close quality tolerances and it is (ii)the analyst's responsibility to demonstrate that these requirements are being maintained by the plant. The fact that very little compositional variation is permitted in the product, permits the application of automation in analytical control. For the production of materials such as ferrous and non-ferrous alloys, control is increasingly exercised by automatic instruments such as the direct recording emission- and X-ray spectrograph, and radiochemical methods are used in some analyses of very high purity materials. Normal methods of analysis are time-consuming and the application of rapid automatic methods of analysis can result in appreciable saving of time, and hence considerable savings in production costs. Process control concerned with changes in chemical concentration or acidity is clearly an extension of analysis from the laboratory into the plant, and in many plants such controls are indeed manned by staff from the analytical laboratory. Here also there is scope for automation to produce savings in the labour force and to result in less compositional variation in process streams than under manual operation. Although in-stream analysis has been developed largely by the chemical industries, particularly by those producing petroleum, heavy chemicals

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and foods, some instruments have been designed to do similar jobs, for the processing of ores. Siggia¹, in his recent book, reviews the field of continuous analysis applied to chemical processes and illustrates the use of X-ray spectrometry to monitor the quality of filter cake from ore processing. In setting up new plants in less developed countries, it should be worthwhile to consider the integrating of continuous in-stream analytical procedures. The advantages of instantaneous data are obvious and such systems are much cheaper when installed concurrently with the fabrication of the plant.

(iii) The general trend toward higher purity requirements for many materials, notably semiconductor ingredients, has made highly accurate analytical control of paramount importance. Even with common materials such as steel, cement and the like, it has been found that minute amounts of certain elements can profoundly affect the properties of the material, and the analyst is increasingly called on for routine analysis in the parts per million or even parts per billion range.

(iv) For such work, many ingenious refinements of emission spectrographic techniques have been developed, for the standard of purity demanded is so high that ordinary chemical reagents cannot be used because of their relative impurity. Recourse must therefore be taken to a method, such as emission spectrography, where little other than very high purity graphite is brought in contact with the material under examination. The spectrographic technique is, apart from the still uncommon methods of radioactivation analysis and solid-source mass spectrometry, the only means of determining impurities at these very low levels.

(v) The production analytical staff together with the production engineers are concerned with trouble shooting. Considerable analytical research may be required to discover causes for alterations in the grade, colour and other properties of the product. It is the function of the production staff to investigate immediate possibilities before referring the problem to the research laboratory.

(vi) The analytical laboratory in the plant is commonly required to provide services for the health and safety of employees. This usually involves determination of levels of toxic substances such as fumes, dust, etc., which result from the different stages of the process.

(vii) From the foregoing it is evident that the analytical laboratory may be required to perform a wide variety of analyses in the mineral and metal processing fields. In less well-developed areas, consulting and referee laboratories will probably not be readily available. Therefore, when planning analytical services, self-sufficiency must be emphasized so that production will not suffer from inadequacy of facilities to meet most contingencies.

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3. Staff Requirements for Analytical Laboratories

(a) The staffing of a large or medium sized analytical laboratory in a mining and metallurgical industry in Canada will be described. Suggestions will be advanced as to how a similar laboratory in a less developed area might obtain the necessary personnel.

(b) In analytical chemistry laboratories serving development and production in Canada, the chief chemist usually is a university graduate with five or more years of experience following graduation, or has equivalent qualifications. It is desirable that the development analyst have, in addition, a higher degree, for originality and scientific leadership are needed to ensure that the analytical contribution to the overall research and development problem is adequate in all respects. The chief analyst should be the severest critic of the work put out by his laboratory and should instil in his staff those standards of scientific integrity without which confidence in the results of the analyst is quickly lost.

(c) In addition to a highly qualified chief analyst a number of university graduates, usually acting as project or group leaders, are also required. Most of the routine analytical work is performed by technicians whose training has been gained in one of two ways.

(i) One group completes secondary school and then attends a technical institute for a two or three year course in chemistry. These are well trained and, with experience, become competent analysts. The theoretical subjects included in their curriculum are usually sufficient to allow them to proceed to semi-professional status and in fact be classed as technologists.

(ii) The other category enters the laboratory directly from secondary school, in some cases without completing the course. Many of these, with several years training, become good routine analysts, capable of doing a wide variety of procedures. They are often most useful in instrumental analytical techniques. It must be realized, however, that certain of these, due to lack of ability, never become more than semi-skilled, tied to simple repetitive functions.

(iii) It, perhaps, should be again emphasized that automation and instream analyses allow a plant to function with a greatly reduced number of analysts. However, the analytical chemist, not the production engineer, is considered to be responsible for such equipment. This aspect is discussed thoroughly by Siggia¹.

(iv) In the underdeveloped areas, there are then, broadly, the two situations. Firstly, the provision of the highly qualified and experienced leaders of the analytical teams, and secondly, the training of unskilled locally recruited junior staff to provide the technical support. a If the educational system of the countries concerned includes secondary schools, technical institutes and universities with adequate science faculties, no serious problem exists. Presumably these schools and universities could provide the basic training, and the necessary experience could be obtained in laboratories at home or abroad, as under the present Colombo plan.

b However many of these underdeveloped areas are deficient in centres of higher scientific learning. Persons desiring a career in science or engineering must go abroad to gain the academic qualifications and necessary experience to fit them for senior positions in analytical chemistry. Although this means of providing well-qualified chemists has some advantages, particularly with respect to gaining experience, it does not keep up with the demands of the growing industries. Rather exceptional talents and adaptability are required on the part of the individual, since he must become fluent in the tongue used in the country in which he studies, as well as devote his efforts to his scientific training.

С The alternative is that, for an initial period, highly qualified and experienced expert analysts be loaned to the underdeveloped areas, each expert serving for about three years. Their duty would be to guide the setting up of suitable analytical laboratories. and to advise on the appropriate analytical methods with respect to operation by locally recruited staff. In many ways the most important duty of all, is to start a training scheme for such staff. In this way, the particular industrial development would be supplied quite rapidly with a nucleus of reasonably skilled analytical workers. At the end of his term the foreign expert would select, from the staff he has trained, those whom he considers would benefit from a two- or three-year period of advanced training in similar laboratories of his own land. It is recommended that the majority of the advanced training be in the land of the tutor, for inevitably he will have given his trainees their preliminary instruction in the methods and on the equipment with which he is most familiar. In addition he can also ensure that they will get their further experience under the most advantageous conditions.

d From our observation, the practice of individuals being sent by the authorities of underdeveloped areas to gain further experience abroad does not, in all cases, result in a choice of the laboratories best suited to give the particular training required. In contrast, the foreign expert, who has seen and appreciated the peculiar problems of the particular underdeveloped area, is in a position to know which of the training grounds in his own country will give the trainee the experience of greatest value.

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PAPER 2

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DEVELOPMENTS IN THE PRODUCTION, CASTING AND FABRICATION OF IRON AND STEEL IN RELATION TO SMALL TONNAGE STEEL PLANTS

by

G.P. Contractor* and W.A. Morgan**

ABSTRACT

The paper discusses recent developments in the production of steel under circumstances where market demand is not sufficient to maintain a modern blast furnace integrated with a steel mill. "Direct-irons" are discussed in relationship to their suitability as feed for electric furnaces, hotblast cupolas and basic oxygen furnaces.

The importance of hot-blast cupolas in the dual function of providing hot metal for duplex steel processes and of providing pig-iron for foundry requirements is included. Canadian experience with the injection of carbon into iron is discussed.

Developments in the continuous casting of steel are discussed along with the economic advantages which are possible when continuous casting can be substituted for conventional ingot practice.

résumé

Dans la présente étude, les auteurs examinent les progrès récents dans la production d'acier lorsque la demande n'est pas suffisante pour maintenir en service un haut fourneau moderne intégré à une aciérie. Les auteurs examinent les "fers d'utilisation directe" au regard de la possibilité de les utiliser pour les introduire dans les fours électriques, les cubilots à vent chaud et les fours basiques à oxygène.

Les auteurs parlent également de l'importance des cubilots à vent chaud qui jouent un double rôle en fournissant du métal chaud pour les procédés duplex de fabrication de l'acier et en fournissant également de la fonte en gueuses pour les besoins de la fonderie. Ils traitent de l'expérience obtenue au Canada en matière d'injection de carbone au sein du fer.

On étudie les progrès réalisés dans la coulée continue de l'acier, de même que les avantages économiques qui en résultent lorsque la coulée continue peut être substituée à la pratique courante de coulée en lingots.

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SUMMARY

- 1. The developments in the production of steel, under circumstances where market demand is not sufficient to maintain a modern blast furnace integrated with a steel mill, are discussed briefly.
- 2. One of the obstacles to the development of an integrated steel industry in under-developed countries is the general shortage of capital, which is of considerable consequence since a steel industry requires large investments.
- 3. Installation of an integrated steel plant based on classical production processes, including the reduction of iron ores by coke in blast furnaces, can only be justified on economic grounds if the market demand of steel products is above a certain minimum.
- 4. In addition to the excessive initial investment involved, a blast furnace unit lacks the flexibility of operation that must be reckoned as one of the requirements in less-developed areas where the market is small. It appears that consideration should therefore be given to the many direct reduction methods of making iron other than conventional blast furnace since, in general, they need smaller investments, have greater flexibility of operation, use low-grade fuel and employ a large proportion of man power.
- 5. There are a large number of so-called direct reduction processes in various stages of development, which produce, in some instances, a high grade product equivalent to scrap and, in others, some variety of pig iron. The choice of the process will depend on the types and costs of fuel and energy available, capital costs/annual ton capacity, market size, quality, quantity and type of ore, etc.
- 6. Steelmaking on a small or medium scale does not present the same problems as ironmaking. The basic oxygen or L-D process appears to be suitable for small scale conversion of pig iron to steel. One of the advantages of the L-D process is the lower capital investment as compared with conventional open-hearth melting shop.
- 7. Under favourable conditions of electric power and scrap supplies, electric furnaces produce steel at costs comparable with open-hearth furnaces. At the same time, capital investment is appreciably lower, about 60 per cent of the open-hearth. It must, however, be mentioned that in most of the less-industrialized regions the situation regarding power and scrap supplies is far from favourable.

The use of direct reduced iron as a substitute for scrap in steelmaking has been studied extensively in recent years. Although investigators have reported promising results on the utilization of sponge iron as melting stock for electric furnaces, open-hearths, basic oxygen converters and hot-blast cupolas, there are still a number of problems. Suggestions have been made that concentration and briquetting of the metallic fraction of the sponge would eliminate or reduce many of the problems but would add to both capital and operating costs.

A brief mention is made of hot-blast cupolas for steel plants and foundries. It appears that basic hot-blast cupolas may be one means of providing hot metal for steelmaking without the construction of additional blast furnaces, particularly for smaller plants. The combination of hotblast cupola and oxygen converter plant seems to rank among the lowest in capital cost and production cost for ingot producing facilities.

Attention is drawn to the use of carbon injection technique to raise the carbon level of grey cast iron melted in a cold-blast cupola. It seems that a substantial saving in the cost of metal charged can be realized by this technique.

Application of the method of continuous casting is mentioned briefly. It is pointed out that in works with outputs ranging from 50,000 tons to 300,000 tons per annum, continuous casting may replace conventional ingot casting, stripping, soaking pit and primary mill practice, with an appreciable saving in investment and operational costs.

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1. Introduction

(a) A close link between the economic development of a country and its steel consumption is well known¹. Consequently, it is believed that the first efforts of less-industrialized countries should be aimed at developing iron and steel production for industrialization, an indispensable factor for social and human progress.

(b) It is known that the majority of the under-industrialized countries are not able to obtain the steel tonnages which they require, but rather only the quantity which their capacity to import permits. One of the obstacles to the development of the steel industry in these countries is the general shortage of capital, which is of considerable consequence since development of a steel industry requires large investments. Capital is not the only scarce factor for industrially emerging regions. In many countries, the availability of foreign exchange to pay for imports represents a similar or even worse problem. It is, however, important to bear in mind that domestic production, even on a small scale, could well save a substantial percentage of the foreign exchange necessary to cover the delivered cost of imported steel.

2. Market-Size and Steel Works Capacity

(a) One of the most important factors in determining the capacity of a new steel industry is knowledge of the size of the market. It has been a common practice to estimate market demand on data, in most cases scanty, for the immediately preceding years. This method may lead to underestimation of the market in cases where unsatisfied demand existed because of limitations in the capacity to import. The main objective is to adjust the steel mill capacity to the dimensions of the market leaving room for future expansions. The capacity of a steel works has a prime influence upon production costs. In general, steel works with lower assembly costs (i.e., mining cost of raw materials plus haulage costs to the plant) and wage rates are in a more favourable position to face competition; that is, the minimum size will be smaller. The size of operation is also governed by other factors which influence costs. Among those of major importance may be mentioned the composition of raw materials, the distance of the plant from local markets, and so forth.

(b) Installation of an integrated steel plant based on classical production processes, including the reduction of iron ores by coke in blast furnaces, can be justified on economic grounds if the market demand of steel products is above a certain minimum. Although the actual capital cost figures are difficult to give, it appears that blast furnace plants, including all auxiliaries, may cost from \$60 to \$90 per ton of pig iron annually², and a fully integrated steel works may cost in the order of \$350 to \$600 per ingot-ton of annual capacity, depending on the product mix, the capacity of the plant and its location. (c) In addition to the excessive initial investment involved a blast furnace unit lacks the flexibility of operation that must be reckoned as one of the requirements in less-industrialized countries where the market is small, say, less than 300,000 tpy. It appears that consideration should therefore be given to the many methods of iron ore reduction other than classical blast furnace since, in general, they need smaller investments, have greater flexibility of operation and employ a larger proportion of man power. In addition, they do not require good metallurgical coking coal. Among these methods, the following are the most important: electric iron smelting, low-shaft smelting, and numerous so-called direct reduction or direct iron methods, which produce in some instances a high grade product equivalent to scrap and in others some variety of pig iron with special characteristics.

(d) Figure I gives a general classification³ of the various methods of making iron. It will be seen that there are a large number of direct reduction methods in various stages of development. There is no doubt that large coke-fired blast furnaces will continue to be the major producers of pig iron in areas where markets are large and metallurgical coke is available. The purpose of this paper is to examine briefly the scope of direct reduction and other non-conventional processes for the establishment of smaller steel plants in regions where the potential market is limited and the coking coals are not easily available. These small plants could meet a part of the regional demands and open the door to a variety of processing industries, which in turn would further accelerate the industrial development of the region.

(e) As stated earlier the direct iron processes are in different stages of development. Some are in commercial operation while others are in an advanced planning stage and are ready for immediate installation. Others are in the experimental stage, e.g., the SL direct reduction process²⁵ recently developed in Canada. Since a voluminous literature on these processes is available, only the few that have operated successfully as production processes will be described. There is no intention to imply superiority of one process over another. The choice, as stated earlier, will be made on the basis of the types and costs of fuel and energy available, capital costs/ annual ton of capacity, market size, quality, quantity and type of ore, etc.

3. The Wilberg-Soderfors Process

(a) The Wilberg method of manufacturing sponge iron has a long record of successful operation in several Swedish steel plants, the first industrial size unit being built in 1932.

(b) The ore must be in lump or pelletized form but the reducing atmosphere may be generated from coke or coal, or by cracking natural gas. Usually, the gas is produced from coke or coal in an electrically heated carburetor. When coke is used as fuel, its sulphur content is largely gasified and occurs mainly as hydrogen sulphide in the gas. This sulphur is removed in a filter interposed between the carburetor and the furnace.

(c) According to one source⁴ the estimated production cost in 1958, in a Swedish plant of 27,500 net tons annual capacity, was 280 Sw Cr per net ton of sponge iron (86% Fe), or about U.S. \$53.70 per net ton. This is equivalent to about \$62.46 per net ton iron in the sponge. The estimated cost was based on a total investment cost (excluding pelletizing plant) of \$1.6 million.

4. HyL Process

(a) This direct iron ore reduction process has proven to be a commercial success. The process can be employed where scrap is either in short supply or expensive and where high grade ores (or concentrates) and cheap natural gas are available.

(b) The HyL process is a batch process in which rich lump ore is reduced in a retort by desulphurized hot reformed natural gas. This gas is passed through a fixed ore bed in the reactors for a period up to 4 hr. The ore, preheated to 850-1000°C (1500-1830°F), is 90% reduced to form sponge and is then stored hot or discharged directly to an electric melting furnace.

(c) Capital cost for a plant of the HyL type will vary according to local conditions. However, according to one estimate⁵, HyL installations require about half of the capital investment required for a conventional blast furnace, including coke ovens, of the same capacity. The reported ⁶ capital investment for a 500 ton/day plant was estimated in 1958 at \$30 per annual ton iron, and the operating cost was placed at about \$10 per ton.

5. Krupp-Renn Process

(a) This process was developed over 25 years ago in Germany, and has probably enjoyed wider commercial application than any other direct reduction process. Basically, the Krupp-Renn process involves the continuous reduction of iron ore in a rotary kiln using coke breeze as a reducing agent and a pulverized fuel flame for heating. Gas or oil can also be used for heating. Reduction is not dependent on the use of lumpy metallurgical coke. A low-quality solid carbonaceous fuel can be employed to reduce a wider range of ores. This includes both low grade ores and ore fines. Furthermore, the process is amenable to siliceous ores. The mixture of gangue and metallic iron is discharged in a pastry condition and is known as "luppen" (nodules). Iron and slag are then separated by milling and magnetic separation. Sulphur pick-up from reducing material may present production problems.

I. Smalling Conventional blast furnace; low shaft furnace (Demag-Humboit)*** Shalt Furnaces Tysland Hole*; Lubatti*; deSy Ghent*** **Electric Furnaces** Strategic-Udy***;Orcarb***: Dwight-Lioyd-McWane*** Electric Furnaces with Pre-reduction Rotary kiln Basset* II. Flash Smelting Flame Smelting (Cyclosteel)***; Jet Smelting***; Diamond Alkali III. Direct Reduction Shaft Furnace Wiherg-Soderfore; Lurg! Galluser*** igas reducant? Madaras***; Hyl* Retort Esso Little***; Nu-Iron***; Armco H-Iron***; Stelling***; Magrigai Fluid ited Tunnel kiln (Hoganas-Sieurin)*; Krupp-Renn; Bureau of Mines; R-N**; Bruckner*; Urqushart*** IV. Direct Reduction (solid reducant)

* Commercial. ** Semi Works. *** Pilot. .

Process Nama	Principals	Description and Status
Flama Smelting (Cyclosteel)	Brilish Iron & steel Heacarch Assn.	Pilot plant; makes iron, not steel.
Jet Smaiting	Ontario Research	Natural gas and nowdered ore mingled to produce iron; early pilot stage.
Diamond Aikali	Diamond Alkali Co.	Gas reactor for desulphurization of pic iron has been mentioned for possible application to iron making

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Class	Process Name	Principals	Description and Status
Shaft Furnacas	Conventional Blast Furnece		, -
	Demag-Humholt		Lew shalt furnace employ briquatts that include fue ore and limastone. Pik plant 10-15 lpd has hean run new plant due with capacit of 100 lpd.
Electric Furnace	Tysiand Hola	Elektre- kemisk	Conventional electric smelling employs tump are and electr arc; 100 furnaces in oper tion; 3-4 million tona capacij
	Lubatti	Demag	Employselectric currant throug slag; commercial, but not i wide usa.
	daSy Ghent	U. of Ghant	Electric furnace with two com partmants; pilot plant built
Electric Furnece with pre-reduction	Elkem (with pra-reduction klin)	Eiskira- kemisk	Submarged arc furnace; pro reduces and pre-bsata ora i kiln.
· · ·	\$irat og ic-Urly	Siratmat- Koppero	Electric resistance end arc hes ing system; uses killn for pri reduction and pre-heating 1000 kw semi-works plant operation; letters of intant (two plants. One latter covas 300 ions day plant for Quabe South Shara Iron & Steel C
	Orearb ·	Swindeli- Dressier	Employs bails of fine ors an coking cost. This is heate- pre-reduced and passes through electric furnace. Pik plant at Harmarville. Pa.
·	Dwijhl-Lloyd- McWane	McDewell Co.	Pellats of fine ceel and ora an passed into sintering machine product goes in electric fu neca.
Batery Kiln	Baneel .	F. L. Smith Co.	Maka liquid Iron in klin; con mercial plants in Europe; a old procass.

(solid reducant - soli	•	
Precess Name	Principals	Description and Status
Tunnei kiin	Hoganas Sleurin	Ors and coke heatsd in tunnei klin process long used in Sweden to produce high grade powdered from Ons plant in Riverton, N. J.
Krupp-Renn	Fried Krupp Southwestarn Engineering Co.	Kiln used to produce partially fused iro ahot; applied to special orea. Thirly five kilns have been built; many nov inoperative.
Bureau of Mines	U. S. Bursau of Mines	Rotary kiln used to reduce coal, ore an limestone; semi-warks plant was bui Project not active.
R-N	Republic Steel Corp., National Lead Co.	Kiln used to reduce ore and produc briquettes; can handle high phos phorous ores or high grade ores. Plan near Birmingham has capacity of 30 t 75 tons a day.
Brucknèr	Kennecolt Copper	Horizontat rotary kiln used to mak sponge iron in connection with coppe recovery; commercial.
Urquahart Othera: Freeman; Kalling Domnarfvet	Steet Precessing Co. Fort Pitt Bridge Co.)	Ore fines and carbon changed into in clined rolating tube: Iron balls up afte reduction; small test furnace.

General classification of the various methods Figure I. of making iron 3 .

Process Name Class Principats Wiberg-Soderfors Stora-Kopparberg

Shaft Furnaca Lurgi Galiuser Pilot plant; no details. Lump ore or peilets reduced by gas; pilot stage. Madaras Madaras Lump or aggiomerated ora with reducing gas; 200 Ion unit now operating; 500 Ion plant coming. HyL (Hojaiata y Lamina) M. W. Keilogg Esso Development Co., A. D. Little Co., Texas Co. Esso Little Pilot plant. Original 2 ton capacity pilot plant being refined. Nu-fron U. S. Steel Pilot plant built; second plant being built at Los Angeles with a capacity of 110 tons a day. Hydrocarbon Research, Inc., Bethlohem Steel H-iron Stora-Kopparborg Pilot plant.

Description and Status

Coke or natural gas used as reduc-ing agent; carbon fully oxidized to give high fust sfficiancy.

Shafi Furnace

III. DIRECT REDUCTION (gas reductant -- solid product)

Stelling

Retort

Fluid bed

Othars: Armco Pilot plant.

(b) On the basis of published estimates⁷,⁸, it appears that the investment cost of Krupp-Renn plant in 1958 was about \$42 to \$50 per annual ton of "luppen" containing 92-95 per cent, and the cost of production was at DM200 (\$45.75) per ton "luppen". In a more recent estimate Astier⁹ gave a figure of 180 new French francs (\$39.80) per ton of "luppen" made from siliceous ore of 30-33 per cent Fe content.

6. R-N Process

(a) The Republic Steel-National Lead process employs a rotary kiln operating below fusion temperatures. Crushed and sized ore of a wide variety or balled fines are fed into the kiln along with the carbonaceous reducing agent. Limestone or dolomite is also fed to control the sulphur content of the ore and the reductant material. Coke breeze or char is used for reduction and for heating gas (methane) or oil. As the ore travels through the kiln, countercurrent to the flow of heat, it is dried, preheated and reduced. The discharged materials are separated by screening and by gravity and/or magnetic separation.

(b) Although more recent figures are not available, it may be interesting to point out that in 1958 the estimated capital cost of an R-N unit, with an annual capacity of 400,000 gross tons, was reported¹⁰ at about \$19.5 million, and the production cost was estimated at \$43.20 per net ton of R-N briquettes containing approximately 80-90 per cent metallic iron.

7. Submerged Arc Electric Smelter

(a) One of the most outstanding methods for reducing the requirement of metallurgical coking coal - at the same time making small scale ironmaking possible - is the electric smelting furnace¹¹. Iron ore and carbon are reacted in an arc furnace to produce molten iron, slag and waste gas containing carbon monoxide and carbon dioxide in the ratio of about 5:1. Electric energy serves the purpose of heating while low grade carbonaceous material acts as a reducing agent. The electric furnace requires only onehalf as much fuel/reductant per ton of iron as a blast furnace. However, electric smelting requires about 1850-2400 kWh of energy per ton of iron, and its commercial feasibility, therefore, depends on the availability of cheap power.

(b) In order to decrease power consumption considerable progress has been made in recent years in preheating and prereducing the burden in rotary or shaft kilns utilizing the by-product gas from the furnace, thus reducing the power requirement by almost 50 per cent. In addition to some of the advantages outlined above, electric smelters operate efficiently in units with outputs as low as 100 tons per day. (c) Estimated capital investment for an electric smelter (without preheating or prereduction auxiliaries) with a rated daily capacity of 800 tons of pig iron has been reported⁴ at 60-75 per annual ton.

8. Steelmaking and Remelting of Sponge Iron

Steelmaking on a small or medium scale does not present the (a) same problems as ironmaking. The basic oxygen or L-D converter process appears to be suitable for small scale conversion of pig iron to steel. Basically, the L-D process consists of blowing high purity oxygen (99-99.5 per cent), vertically downward, through a water cooled lance, on the surface of molten iron in a closed bottom vessel lined with basic refractories. Scrap. iron ore or cold pig iron are added to regulate the heat. One of the advantages of the L-D process is the lower capital investment as compared with a conventional open-hearth melting shop. In a recent study¹² in India it was shown that the capital cost of an L-D melt shop (including oxygen plant) was about 60 per cent of the cost of an open-hearth plant of equivalent capacity, i.e., two-million ingot tons per year. In terms of Canadian dollars the capital cost amounted to about \$23,50 and \$39,00 per ingot ton annually for L-D and open-hearth melt shops, respectively. These figures will be substantially higher for a smaller melt shop. It was also reported that the production cost of the L-D process was about Rs. 20 (\$4,40) less per ingot ton than that of the open-hearth shop.

A passing mention may also be made of the Savard-Lee bottom-(b)blown converter process¹³ developed recently in Canada. Instead of top lancing as in the L-D process, oxygen is injected at a high pressure into the bottom of the metal through a specially designed injector. It is claimed that the metal can be converted to steel without further refining in an arc furnace. The importance of electric arc furnaces for steelmaking, especially in countries with low cost electric power and adequate scrap supply, is known all over the world. Under favourable conditions of electric power and scrap supplies, electric furnaces produce steel at costs comparable with openhearth furnaces. At the same time capital investment is appreciably lower, about 60 per cent of the open-hearth. It must, however, be mentioned that in most of the under-industrialized regions the situation regarding power and scrap supplies is far from favourable. Till recently, one of the major drawbacks of electric furnaces for the production of plain carbon steels was that they are not effectively adapted to molten iron charges. However, recent studies have shown the feasibility of refining molten pig iron in the electric arc furnace, thus improving its economics and increasing its scope.

(c) This brings up the question of using direct reduced iron or sponge iron as a substitute for cold scrap. In fact, extensive work on the utilization of sponge iron for steelmaking has been carried out in the last ten years and very promising results have been reported. It is no secret that Sweden uses a greater tonnage of sponge iron per year than possibly any other country at the present time. Similarly, HyL sponge⁵ has been in use for electric steelmaking in Mexico since 1958. Weaver¹⁴, Farley¹⁵, and Babcock¹⁶ investigated the melting of R-N briquettes in electric furnaces and more recently Masi and Cannizzo¹⁷ reported that direct reduced iron is a good component for the cold charge of electric furnaces and that it can be tolerated in open-hearth melting.

(d) Although considerable progress has been achieved in the technique of remelting sponge iron in electric furnaces, there are still a number of problems. For instance, it is generally known that the power consumption may be increased by 10-25 per cent when sponge is substituted for steel scrap. In addition, the fluxing of a large percentage of gangue results in a very large slag volume in the electric arc furnace. According to Armstrong⁴ the concentration and briquetting of the metallic fraction of the sponge would eliminate or reduce many of these problems but would add to both capital and operating costs.

(e) In addition to the use of direct reduced iron in electric steelmaking, investigations have also been carried out to utilize it as a melting stock in basic hot-blast cupolas. In a recent study¹⁸ in Germany it was shown that direct iron briquettes containing 88-91 per cent metallic iron and 0.3-0.4 per cent carbon can be successfully remelted in a hot-blast cupola with a capacity of 20 tons per hr. The liquid metal produced was suitable for open-hearth and L-D practice, and analysed 3.80-4.20 per cent carbon and 0.02-0.04 per cent sulphur. The conversion costs (excluding the cost of briquettes) compared favourably with 100 per cent steel scrap melting, and approximated the attractive figure of DM 40.00 (U.S. \$10.00) per metric ton of the liquid metal produced. It was, however, reported that coke consumption for the remelting of briquettes was slightly higher than the all scrap melting.

9. Basic Hot-Blast Cupola

(a) Suggestions have been made to integrate some of the direct reduction processes into foundries with a view to reducing their cost of iron and their dependence on outside source of pig iron and scrap. It appears that hot-blast cupolas for steel plants and for foundries have acquired enormous interest in recent years. It has been said that basic hot-blast cupolas may be one means of providing hot metal for steelmaking without the construction of additional blast furnaces, particularly for smaller plants when the relatively small investment cost for added capacity is considered.

(b) According to Harman¹⁹ the combination of basic hot-blast cupola and oxygen converter or L-D plant ranks among the lowest in capital cost and production cost for ingot producing facilities. On the basis of published information the capital cost of a hot-blast cupola plant is placed at about \$7 to \$10 per ton of annual capacity, and the net cost of production is estimated in the order of \$40 to \$50 per net ton of liquid metal, depending on the cost of metallic and fuel charges.

(c)The cold-blast acid-lined cupola is still the most widely used melting furnace for cast iron and it seems that this situation will remain for many years yet, although the economic advantages of using heated blast will cause an increase in the number of such installations. One of the most widely used methods of regulating the carbon content of grey iron melted in a cold-blast cupola is to adjust the pig iron in the charge. Since this material is the most expensive component of the charge, newer techniques have been suggested²⁰ of controlling the final carbon content, particularly at the higher levels. The most successful answer to this problem is the carbon injection process. Briefly, the process consists of injecting fine particles of graphite or other carbonaceous material by a stream of relatively inert gas, such as nitrogen, through a graphite lance immersed in the liquid metal. Generally, the carbon pick-up depends on the feed rate and carbon recovery. Therefore, the major operating factor that affects carbon pickup is the immersion depth, which acts on both components. One of the factors that tends to limit the application of the process is the increase in sulphur content as the pig iron charge is decreased. However, it has been suggested that simultaneous desulphurization and carbon injection may offer a solution to the problem.

(d) On the basis of long term tests in Canada it was estimated²⁰ that a saving of about \$6.15 per net ton of metal charged can be realized.

10. Continuous Casting

(a) As a result of the great technical progress that has been made in recent years, it is now considered practicable to replace the orthodox stages of ingot mould casting, stripping, soaking pits, and primary rolling in small steel plants by the adoption of continuous casting²¹. By eliminating these production stages the investment cost of a steel plant can be appreciably reduced. There are a number of machines in various parts of the world that cast billets, blooms and slabs in continuous lengths on a commercial scale and several more are in the experimental stage or under construction.

(b) Advantages of the continuous casting process become apparent in making comparisons with conventional casting. The yield of metal is greatly increased. For instance, improvement of 10 to 12 per cent in the overall yield of the primary rolled product from liquid metal has been reported ²². Characteristic high surface quality of the continuous cast product has practically eliminated the need for surface conditioning. The other main advantage of the process is that the low capital investment makes it quite practical to locate plants to suit the market and to take advantage of favourable freight rates. These are some of the features that will be particularly attractive to less-industrialized regions where the production rates contemplated are not very high.

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(c) The economics of investment and operating costs are difficult to give. However, the figures published ²³ in 1957 indicate investment cost of approximately \$17 per ton and about \$7 per ton for machines having an annual capacity of 50,000 tons and 300,000 tons, respectively. These costs and the corresponding amortization are believed to be considerably lower than those prevailing in integrated steel works based on cogging and other primary mills. The cost increment would be in the order of 50-70 per cent or more for a conventional plant, depending upon the location.

(d) According to one source²⁴ the net saving per ton of low carbon steel billets produced by continuous casting is estimated at about \$8 - \$10. This is equivalent to reducing billet costs by about 12 per cent. On the basis of published information it appears that in works with outputs ranging from 50,000 tons to 300,000 tons per annum, continuous casting may replace conventional ingot casting, soaking pit and primary mill practice with an appreciable saving in investment and operational costs.

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PAPER 3

ORE MICROSCOPY AS A GUIDE TO ORE DRESSING PROCEDURE

by

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SUMMARY

1. A brief outline is presented of the use of ore microscopy as a guide to ore dressing procedure. Practical advice is given as to the apparatus and the technique of preparing and examining polished sections of ores. Photomicrographs illustrate some of the features of metallic ores. The works listed in the bibliography cover the field, and contain numerous references to the detailed literature.

RÉSUMÉ

1. La présente étude donne un bref aperçu de l'examen au microscope du minerai aux fins de servir de guide aux procédés de préparation mécanique du minerai. L'auteur donne des conseils utiles au sujet des appareils et des moyens employés pour préparer et examiner des lamelles polies de minerais. Des photomicrographies indiquent certaines des caractéristiques des minerais métalliques. Les ouvrages mentionnés dans la bibliographie traitent de la technique à l'étude et contiennent de nombreuses références aux documents plus détaillés.

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INTRODUCTION

1. Definition of Ore Microscopy

Since its first modern application more than fifty years ago¹. ore microscopy has become not only an important branch of mineralogy, but has also proven itself indispensable in the fields of mining geology, mineral processing and extractive metallurgy. In a strict sense, the term "ore microscopy" refers only to the examination of opaque minerals in polished sections under the reflecting microscope. In modern practice, however, it implies much more. Now the importance of the transparent gangue minerals in metallic ores is recognized, and the growing significance of such elements as niobium and beryllium requires that the transparent minerals in which they occur be studied in detail. Then too, we are faced with diminishing mineral resources and consequently with the necessity of exploiting deposits that are mineralogically complex and hence difficult to treat. This has resulted in the use of several other techniques supplementing the microscope. For instance, X-ray powder diffraction methods are used for the identification of the minerals and the recently developed electron probe micro-analyser is most helpful in determining the distribution of the elements among the mineral grains. However, in spite of these modern trends, the microscope itself remains the most useful basic means of learning the character of mineral assemblages. In this very brief summary of its use in the field of ore dressing, the strict meaning of the term "ore microscopy" will be adhered to closely.

2. Apparatus

(a) General statement: The apparatus required for ore microscopy will vary widely in complexity and cost, according to the need and the funds available. The references include many excellent descriptions of the apparatus and its uses. In general it is true that a minimum of apparatus essential for the microscopical examination of metallic ores will yield the greatest return on the investment, and that elegant and costly accessories should be avoided unless they are needed for special cases.

(b) Polishing apparatus: The preparation of polished sections may be either a simple or a complicated procedure. Solid sections of ores can be prepared very simply by hand-polishing the unmounted specimens on relatively inexpensive machines², ³. Fragmented samples, such as mill products, require mounting before polishing, and since their study cannot be avoided in connection with ore dressing, a means of mounting must be provided. Some ores are soft and friable and their preparation is extremely difficult and involves impregnation with special resins. In fact, the preparation of polished sections is a highly skilled craft and minerals vary so greatly in physical character that it is not surprising that the methods for polishing ores also vary in detail², ³, ⁴, ⁵, ⁶. Basically however, the minimum of equipment required includes a rough grinding lap for shaping ore specimens, facilities for mounting ores and mill products in plastic^{7, 8}, and polishing machines fitted with either metal or fabric-covered laps^{2, 3}, 4, 5. More elaborate installations would include a precision diamond saw, semi-auto-matic polishing machines and many other accessory devices.

(c) Microscopical apparatus: The most important requirement for ore microscopy is a modern reflecting microscope of highest quality. Accessories can be added as required. A microscope that can be converted to a petrographic microscope by using an additional tube and sub-stage is highly desirable. There should be at least three air objectives for low, medium and moderately high powers of magnification, and a good high power oil immersion objective. Many additional accessories may be required for special purposes such as photomicrography, statistical grain analysis and measurement of reflectivity, but a good microscope equipped with objectives covering a wide range of magnifications and having a micrometer ocular and traversing stage for grain analyses, will be found to be adequate for many purposes.

3. The Microscopical Examination of Polished Sections

(a) General statement: The microscopical examination of polished sections of ores and mill products must be done by trained personnel. It will be apparent, after referring to any of the standard works on the subject, that especially the interpretation of mineral textures and their correlation in terms of milling behavior require training and experience.

(b) Identification of minerals: The identification of the minerals in polished sections under the reflecting microscope is based upon their optical, physical and chemical properties 4, 9, 10, 11, 12, 13. Optical properties include colour, brightness (reflectivity), anisotropic effect and the character of any internal reflections. Physical properties are hardness, sectility, crystal habit, cleavage or parting, and zoning as reflected in systematic fracturing and pitting. Chemical properties that can be observed under the microscope are normally restricted to surface reactions brought about through the application of etching reagents 12; this also tends to display any zoning due to changes in chemical composition. Microchemical tests¹² are sometimes very useful and although most of the common minerals can be recognized readily in polished sections after some experience, positive identification of minerals in complex and fine-textured ores is frequently difficult. If such ores are encountered, it may be necessary to employ some of the supplementary techniques mentioned earlier in paragraph 1, for the identification and characterization of the minerals.

(c) Form and texture: The form and texture shown by minerals and the manner of their mutual association⁴, 14, 15, 16 are the basic guides to ore dressing procedure. The minerals are the units, and the ore dressing engineer has to separate the valuable constituents from those that are worthless. Therefore, their grain sizes, physical characteristics and

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the degree to which they are combined with one another will determine the extent to which grinding must be carried to effect their separation.

(d) The examination of mill products: The examination of mill products in conjunction with ore dressing procedures, either in testing laboratories or in operating mills, is indispensable, particularly when difficulties arise in separating the minerals. When the significant products are studied microscopically, the cause of the trouble is usually quite apparent. It is advisable to study the solid ore sections before the examination of mill products is attempted, because the minerals can be more easily identified in the larger grains of the solid ore than in the smaller fragments. In fact the study of ore prior to its test treatment can frequently save the engineer many hours of testing, and in some instances the behavior of the ore can be forecast with remarkable accuracy. In operating mills it is advisable to use a binocular microscope, because the experienced engineer can tell a great deal about the functioning of the circuits by examining the products.

(e) Grain analyses: Grain analyses can be very useful in ore dressing operations. Several methods have been used for ascertaining the relative quantities of the minerals in ores and mill products², ⁴, ¹⁷. Sometimes very complete statistical studies have been made to determine the degrees of freedom and the combinations of the various minerals of a complex ore. Such studies, however, are ordinarily too fatiguing and time-consuming to be justified, and some thought is now being given to find a method of performing these analyses automatically through the use of modern electronics applied to the varying reflecting characteristics of the ore minerals.

4. Interpretation of Microscopical Data

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(a) Gold Ores: The interpretation of microscopic features in terms of ore dressing procedures is based on their correlation with the behavior of the ore under milling conditions. The relationship between mill practice and the manner in which gold occurs illustrates this rather clearly. If the gold occurs as comparatively coarse grains and veinlets in quartz (See Fig. I) or in sulphides (See Fig. II), satisfactory recoveries can be expected to result from comparatively coarse grinding and gravity methods of separation. If its average grain size is less (Fig. III), the ore may require finer grinding followed by cyanidation to recover the finer gold. If sulphides are present, as they frequently are in the form of pyrite and arsenopyrite, some of the gold may be enclosed within the sulphides (See Fig. IV). If the inclusions are in very dense sulphide (See Fig. V), the sulphides may have to be concentrated by flotation, re-ground and cyanided. When the average grain size of the gold in the sulphides is very small (See Figs. V and VI), recovery by cyanidation of the flotation concentrate may be negligible; in such cases the concentrates must be roasted before re-grinding and cyanidation. Fig. VI shows tiny gold particles in arsenopyrite; in this ore the arsenopyrite itself is very finely disseminated, and presents a problem in concentration. In fact, for this reason, the operation of the mine was on the economic

borderline. It is probable that in some ores the gold is largely in submicroscopic form, and possibly also in solid solution. The presence of interfering minerals, such as talc or other soft flakey minerals, may cause trouble. Chalcopyrite (See Fig. VII), as well as pyrrhotite, can contaminate cyanide solutions and cause trouble in the circuits. Acute complications are introduced when gold and silver tellurides, graphite, et cetera are present in appreciable quantities as shown in Fig. VIII.

(b) Iron ores: The beneficiation of iron ores requires treatment of very large tonnages by very economical methods. The coarse-textured ore seen in Fig. IX is easily treated by gravity and magnetic separators. On the other hand the ore shown in Fig. X is practically impossible to treat economically. A titanium-bearing ore, in which ilmenite, hematite and magnetite occur as separate, individual, relatively coarse grains, can be treated satisfactorily, but no amount of grinding can free the intimately admixed ilmenite-magnetite or ilmenite-hematite intergrowths (See Fig. XI) frequently found in titaniferous iron deposits.

(c) Base metal ores: Base metal ores show great variation in their characteristics and, hence, present a great variety of problems to the ore dressing engineer. Some are coarse textured and the minerals are comparatively easy to separate (See Fig. XIII), while others are fine textured and the minerals are so intimately associated that their separation is impractical, if not impossible (See Figs. XII and XIV). The reason for poor recoveries or incomplete separation of the minerals is usually apparent when the products are examined microscopically. Two examples are given in Figures XV and XVI, but numerous others have been recorded.

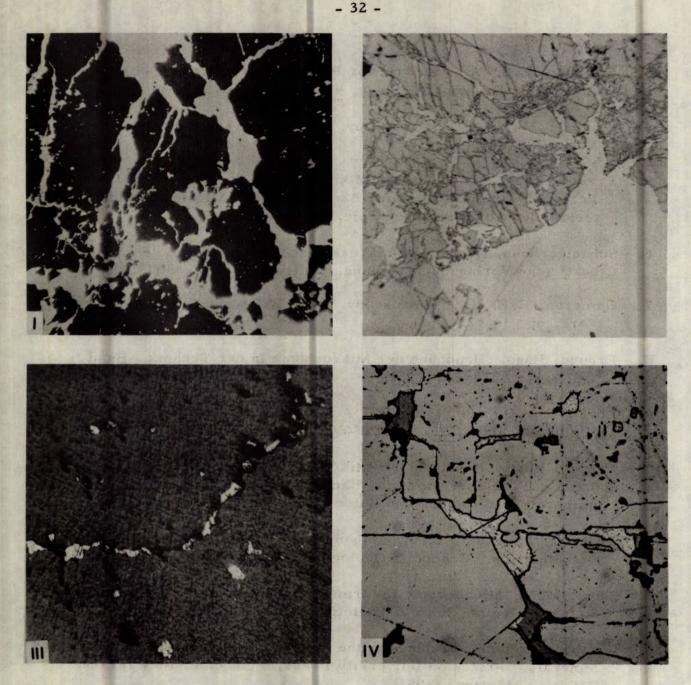
5. Conclusion

It must be remembered that metallic ores seldom even approach homogeneity in the distribution of the minerals. In fact ores commonly show very wide variations throughout a deposit, and each type presents a unique set of problems that must be solved before it can be treated satisfactorily. Ore microscopy is one of the most effective weapons in solving these problems and for guiding and improving ore dressing practice.

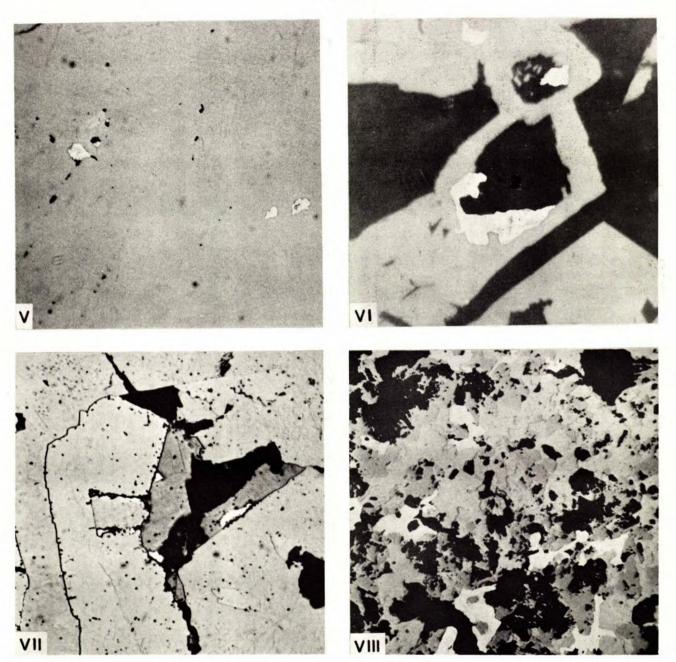
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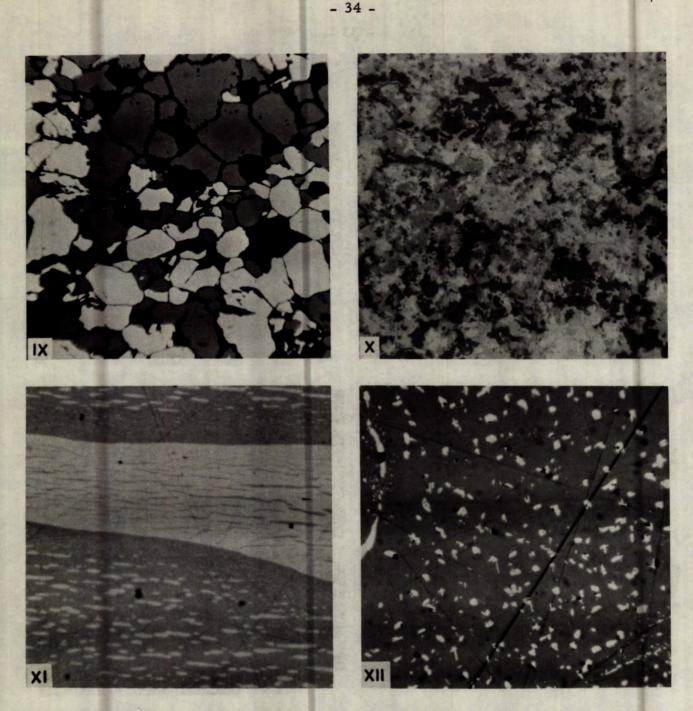
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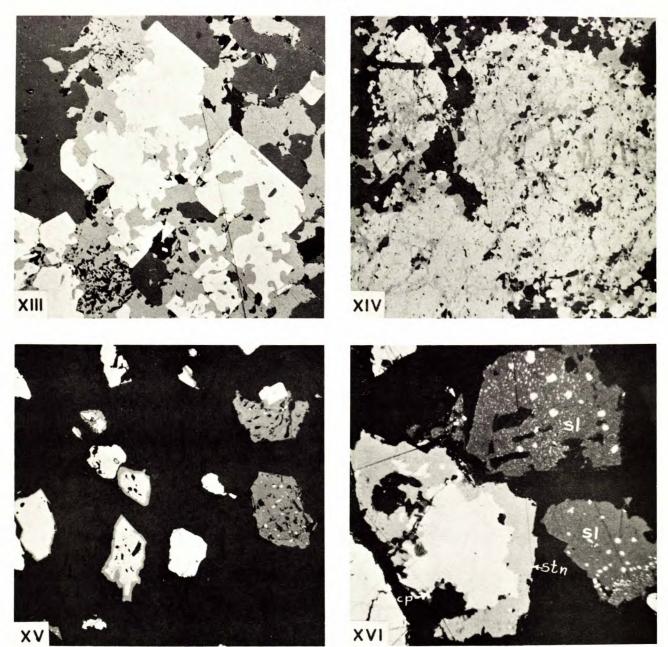
- Fig. I Polished section of rich gold ore from Lake Shore Mine, Ontario. The veinlets of gold (white) in the quartz (black) are very easily freed and recovered by gravity methods. Magnification 50x.
- Fig. II A very rich section of gold ore from the Bralorne Mine, British Columbia. Here the coarse gold is associated with arsenopyrite (grey). This type of gold occurrence presents no treatment problem. 50x.
- Fig. III Gold in quartz from the Porcupine District, Ontario. This gold (white) occurs as much smaller grains, and treatment involves finer grinding and cyanidation. 100x.
- Fig.IV Moderately coarse to fine gold (white) in pyrite (light grey) and quartz gangue (dark grey) from northern Ontario. The coarse gold is recovered by gravity methods, the finer must be recovered by cyanidation. 100x.



- Fig. V Tiny grains of gold (white) in dense pyrite (grey) from northern Ontario. The sulphides are concentrated by flotation and cyanided after re-grinding. 1000x.
- Fig. VI Tiny grains of gold (white) in skeletal crystals of arsenopyrite from Beattie Gold Mine, Quebec. The arsenopyrite must be concentrated and the concentrate roasted and re-ground before cyanidation. 1500x.
- Fig. VII Tiny grains of gold (white) along the boundaries of chalcopyrite (dark grey), pyrite (light grey) and arsenopyrite (slightly darker grey than the pyrite). The black areas are pits. 1000x.
- Fig. VIII A rich section of gold from Kirkland Lake, Ontario. Here the gold occurs with an assemblage of gold and silver tellurides, pyrite (varying shades of grey), graphite (not shown in this field) and quartz (black). This requires complex treatment. 500x.



- Fig. IX Iron ore from central Labrador, Newfoundland. Coarse magnetite and hematite (both white) in quartz, and carbonate gangue (grey). Pits are black. The iron minerals are freed from the gangue minerals very easily. 100x.
- Fig. X Heavily oxidized iron ore from central Labrador, Newfoundland. The ore is soft and friable, and the minerals include goethite, hematite and silicates. Beneficiation is very difficult, if not indeed impossible by gravity methods. 1000x.
- Fig. XI Hematite-ilmenite intergrowth in an iron ore from Ontario. The hematite is white, the magnetite is grey. These cannot be separated mechanically. 1000x.
- Fig. XII Numerous tiny exsolved inclusions of chalcopyrite (white) in sphalerite (grey) in a base metal ore from New Brunswick. It is obvious that the copper and zinc of this assemblage cannot be separated mechanically. 1000x.



- Fig. XIII Coarse-textured intergrowth of sphalerite (light grey) and pyrite (white, with cubic habit) in quartz (dark grey) from British Columbia. 50x.
- Fig. XIV Pyrite (white) intricately veined by chalcopyrite (light grey) and associated with sphalerite (dark grey) and quartz (black). Copperzinc ore from northern Quebec. 200x.
- Fig. XV Combined grains in a flotation concentrate from a British Columbia mill. Chalcopyrite (light grey) rimmed by chalcocite and covellite (grey and indistinguishable from one another in the photomicrograph), and sphalerite (dark grey) containing numerous tiny dots of exsolved chalcopyrite, and in one instance still attached to pyrite (white). The mounting medium is black. 200x.
- Fig. XVI Combined grains in a concentrate from a copper-tin ore from New Brunswick. Chalcopyrite (white - cp), stannite (light grey - stn) and sphalerite (dark grey - sl). The mounting medium is black. 1000x.

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PAPER 4

THE USE OF RADIOISOTOPES AND SPECIAL INSTRUMENTATION FOR PROCESS CONTROL AND DEVELOPMENT IN THE MINERAL AND METAL INDUSTRIES

by

G.G. Eichholz*

ABSTRACT AND SUMMARY

1. The possible applications of electronic process controls and instrumentation are considered in their relation to development conditions for the metallurgical and mining industries in less-developed areas. In the three phases of resource development--exploration, process development and routine operations--different levels of technical competence are required as well as different degrees of individual responsibility on the part of the operating staff. The greatest field for semi-automatic process control lies in the area of routine plant operations, where it is superior to the alternative of breaking down operations into a large number of simple steps capable of being handled by low-level unskilled labour.

2. The use of radioactive markers and gauges is reviewed and it is seen that, when properly installed, such devices do not differ inherently from other types of process equipment in either complexity or maintenance requirements.

3. The use of radioactive tracers for process development and for the adjustment of operating conditions is valuable in many fields of technology and should be used to the fullest extent wherever experienced supervision is available. Tracer applications are not recommended on a continuous basis in production circuits, unless conditions of recovery of the tracer and total expected radiation levels are exceptional.

4. A brief review is given of recent developments in the use of electronic control equipment in the mineral industries.

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1. Les applications possibles d'instruments et de vérifications électroniques des procédés sont examinées dans la présente étude en fonction des conditions qui favorisent la mise en valeur dans les industries des mines et de la métallurgie des régions moins évoluées. Pour ce qui est des trois stades des travaux de mise en valeur des ressources, savoir l'exploration, la mise au point des procédés et les opérations régulières, les hommes qui sont chargés de ces travaux doivent avoir une haute compétence technique et assumer leurs propres responsabilités. La vérification semi-automatique des procédés ne saurait mieux se faire que quand l'usine fonctionne normalement: cela vaut bien mieux que de fragmenter les opérations en un grand nombre de tâches simples confiées à des ouvriers non spécialisés.

2. L'emploi d'indicateurs et de barêmes radioactifs y est également examiné, et l'on peut voir que, une fois installés de façon appropriée, ces auxiliaires ne diffèrent pas essentiellement des autres genres d'appareils utilisés pour les procédés, que ce soit en matière de complexité ou d'entretien.

3. L'emploi de radioindicateurs pour la mise au point des procédés et des conditions de fonctionnement est utile dans de nombreux domaines de la technologie, et l'on devrait y recourir au maximum lorsqu'on peut compter sur des surveillants expérimentés. Il n'est pas recommandé d'utiliser les indicateurs de façon continue dans les circuits de production, sauf si les conditions de récupération des indicateurs et si le niveau prévu des radiations totales sont exceptionnels.

4. L'auteur examine brièvement les récents progrès réalisés dans l'utilisation d'appareils électroniques de vérification dans les industries minérales.

RÉSUMÉ

INTRODUCTION

1. The present climate of international cooperation and technical aid extended to many of the less-developed areas provides them with an opportunity to benefit from the experience gained and advances made in other countries to obtain the highest possible productive efficiency. The extent to which electronic and radioactive instrumentation can be employed in the mineral and metallurgical industries depends greatly on the level of operations. The opening up of the mineral resources of less-developed areas can be broken down into the usual three phases through which any industrial development must pass: exploration, process development, and routine processing. To these should perhaps be added long-term research as a fourth phase usually associated only with more advanced technologies. Each of these phases has its own specialized requirements for equipment, some of which depend heavily on the available personnel and their relevant skills. It is this interplay of improved process efficiency by semi-automatic controls and its requirements for skilled personnel that will be discussed here as it applies to resources development in underdeveloped areas.

OPERATION OF ELECTRONIC PROCESS EQUIPMENT

2. In the three development phases mentioned, there is a steady increase in complexity in equipment requirements as one passes from exploration to routine processing. Opposed to this, there is a reverse process of dilution in the average level of skill and specialized knowledge required as the scope of operations grows.

3. The exploration phase usually requires a highly knowledgeable person directing a team capable of using a range of instruments and techniques under constantly varying conditions. These techniques include various methods of aerial surveying, ground prospecting and borehole logging, supported by geochemical and mineralogical analyses. The lessdeveloped areas may well depend on technical aid from more advanced countries to press ahead in this field for many years to come.

4. The process development phase involves small-scale mill tests, beaker-scale laboratory extraction work and, finally, pilot plant operations. All these operations require an experienced staff and well-equipped laboratories. Much of the equipment must be properly instrumented to provide all necessary data on process variables and laboratory results, such as analytical determinations, feed densities, flow rates, pH values, gas pressures and many others. Instruments for such measurments have been available commercially for many years and it should not pose too big a problem to train operators to collect and record instrument readings. The actual direction of development test must always devolve on a trained professional with considerable theoretical and practical experience, who in many cases may have to devote a fair share of his time to training his operating staff. 5. The routine processing or mill operating phase will in general depend to the greatest extent on local resources. The design of the plant must take into account both economic and labour problems in addition to the usual requirements for a high degree of plant efficiency and reliability. In the mineral and metal industries this often implies that an early decision has to be made whether to adopt more modern, semi-automatic means of process control and operation, or whether to retain a more traditional approach involving a great number of relatively unskilled workers performing routine operations as well as their training and capabilities permit.

6. It has often been argued that in many underdeveloped areas unskilled manpower is the chief readily available resource and that the latter approach is more suitable, particularly since skilled technicians may be scarce and breakdowns of expensive control equipment may be hard to remedy due to lack of spares and of experienced repair men. However plausible such an argument may appear, particularly as a means of ensuring wider employment of unskilled labour, it suffers from a number of obvious contradictions under present-day conditions.

7. One is the inherent waste of valuable resources in mining and metallurgical operations conducted without proper control of process variables. Experience in Canada and in many other countries has hown that spectacular improvements can often be made in the yield and overall production of extraction plants when important chemical or physical factors are properly controlled; this in most cases leads to a substantial lowering of costs of production. Many of these factors cannot be judged by the operator by experience alone, but require instead the use of properly designed, often highly specialized, process instrumentation.

8. The second weakness in the argument for the use of large numbers of unskilled labour is its inherent inefficiency. In those countries where this approach has been practised widely, it has been found that almost invariably the labour force suffered from excessive turnover. This required a large proportion of the work force to be tied up in training courses, lasting from a few days to several weeks in some cases, and an often longer period of adjustment and learning on the job, with a low level of average efficiency and relatively high cost of production per man.

9. This practice contrasts with a semi-automatic plant installation operated by a smaller number of more highly trained men who are less likely to find competitive posts elsewhere at similar remuneration. By careful selection, a sufficient number of men can be chosen to attend training classes similar to those given by several North American instrumentation companies to acquaint technicians thoroughly with the operation and maintenance of selected electronic equipment. Such courses may last two to three weeks and do not demand any theoretical knowledge of the process being controlled or of circuit theory as such. Extension of such schemes to many of the less-developed countries, together with mechanical and electrical design to suit the climatic conditions of the area, would go a long way to ensure efficient and economic operation of plants.

10. It is an obvious corollary that probably more care has to be given then hitherto to engineering design of process instrumentation intended for use in less-developed areas, where often very adverse climatic conditions and uncertain electrical power supply conditions will be encountered. To minimize repair problems, more equipment should be designed in modular sub-assemblies so that defective sub-units can be readily replaced by spare units of easy accessibility and moderate cost. This is achieved to an increasing extent by means of printed circuit techniques and transistor circuits. Such an approach to servicing is likely to overcome the common tube replacement problem and may be more adaptable to regions with difficult communications.

11. Another vital matter, which can only be mentioned in passing, is the need for detailed instruction manuals with complete circuit and layout diagrams, and careful operating and trouble-shooting instructions in the language of the prospective user. This may well prove to be a major problem to the average instrument supplier and may deserve some special government attention and support.

THE USE OF RADIOACTIVE SOURCES IN CONTROL EQUIPMENT

12. Radioactive materials are used to an increasing extent in the metallurgical industry. Because of their relative novelty and the special legislation covering their use in most countries, many potential users still feel mildly uneasy when the use of radioactive materials is suggested in routine operations. For a sensible discussion it is essential to classify such uses according to whether the radioactive material is enclosed or sealed permanently, or whether it is dissolved or entrained in other substances, and according to its mounting, either in a fixed location or in a mobile installation.

13. Mobile, sealed radioactive sources are used primarily in exploration work and for radiographic inspection. Typical applications include gamma-ray and neutron borehole logging¹, ², gamma-activation analysis of beryllium³, ⁴, and other neutron sources for mine analyses⁵. The need for mobility limits the amount of shielding that can conveniently be carried, although truck installations containing several curies of activity have been used. Needless to say, careful handling in accordance with instructions is required to avoid overexposure of the operator or any other bystanders.

14. Similar considerations apply to the use of fixed, sealed sources in control equipment. While the question of the usefulness and desirability of reactor construction in underdeveloped areas is still a contentious subject, there can be no question as to the usefulness and economic advantage of utilizing radioactive sources in certain industrial applications. A properly shielded radiation source, installed in flow or density gauges, in itself presents no problem of handling or maintenance; only its associated electronic detector and control circuits will require periodic inspection and maintenance, like any other electronic gear. In general, no great difficulty has been encountered in designing such industrial gauges in such a way that any operator is well shielded from any radiation from the radioactive source, thus avoiding the need for classifying him as an "occupationally exposed person". If the source is limited in use by the half-life of its effective nuclide, replacement for such radioactive sources can be obtained quite readily from any of the many countries now exporting radioisotopes.

The main advantages of radioisotope radiation sources lie in their 15. ready availability, relatively low cost, absence of moving parts or highvoltage circuits (which might require frequent maintenance), silent operation, and simplicity of installation. The latter point becomes evident when one compares the installation of a radioactive density gauge or level indicator outside the pipe or vessel containing the medium being controlled with any alternative non-radioactive method. All these advantages matter when maintenance and staff training considerations are paramount. By choosing a radioisotope of the appropriate character as to type of radiation and energy emitted, suitable conditions can be established to meet a wide variety of requirements⁶. Most of the gauging systems in industrial use utilize changes in the intensity of radiation transmitted or backscattered from the medium under observation. This radiation level is detected by means of a Geiger counter, scintillation counter or ionization chamber, integrated, and then fed to a conventional electronic recorder or controller.

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Industrial uses of sealed sources can be classified as follows:

- a) High-level radiation sources for purposes of sterilization, pasteurization and food preservation, as well as a processing technique in the plastics industry⁷, and the petroleum industry⁸;
- b) Static eliminators and similar ionizing devices;
- c) Thickness gauges, density gauges and level indicators;
- d) Markers and indicators, such as are used for emergency lighting and labels or for the identification of selected materials;
- e) Flow meters and radioactively tagged "go-devils" in pipe lines⁹.

17. Of these, only the third and last groups qualify as standard process control equipment in the sense that they can be obtained commercially and may be part of an overall program of plant control and process supervision. Among these applications the use of radioactive sources in thickness and density gauges for slurries, solutions, metal foil or paper by transmission or backscattering methods have received the greatest attention10, 11, 12, but many other applications are constantly being introduced. Similarly, a wide range of level gauges are in use to control liquid levels in leach tanks and oil refineries 13 , scrap metal feed in blast furnaces 14 , and coal or coke levels in fuel hoppers.

THE USE OF RADIOACTIVE TRACERS IN METALLURGY

18. The use of radioactive tracers, i.e., essentially unsealed, dispersed radioactive materials, calls for a different philosophy and more careful handling precautions. Tracer methods are most useful in development work and for occasional checks of proper operating conditions in routine process work. Their use in continuous process operations is rarely feasible, both for reasons of cost and the limitation in supply, and because of difficulties in supervision and waste disposal under such conditions.

19. There are many potential uses of tracers in the development of chemical processes, the development of mill circuits in extractive processes, and in pyrometallurgy. Typical uses include measurements of flow-patterns in leach plants¹⁵, of contact and residence times in kilns and furnaces¹⁶, of leak detection in pipe lines¹⁷, and of flow problems in hydrology and mining¹⁸. Although the technique of applying radioisotopes in such tracer tests is not difficult, they should be conducted by a properly qualified scientist or engineer to guard against unexpected exposures and serious contamination. However, the usefulness of tracer methods in the metallurgical field is so great, and the light they can shed on actual plant operations so useful, that one can only hope that a steadily increasing number of scientists from less-developed countries will be able to receive training in this field.

20. A few examples only can be given here of tracer applications in extractive metallurgy and related fields. By adding a suitable tracer it has been found possible to check flow conditions in leach tanks and flotation cells, and to remedy undesirable short-circuit conditions. Similarly, in kiln operations adsorption of a tracer solution on a feed sample can indicate residence times of different feed components, segregation effects, furnace brick wear, pile up and possible short-circuiting of finer fractions. In mines, radioactive tracers provide a better means than fluorescent tracers to tag mine waters and to investigate leakage or contamination problems.

21. In all these cases, careful analysis of the problem can lead to safe use and ultimate disposal of radioactive materials. However, it is not recommended that unsealed radioactive materials be used in any continuous application where the total quantity used may be excessive and ultimate recovery of the active material impractical or impossible. The "post-activation" of non-active tracers may be a possible answer to these problems.

ELECTRONIC EQUIPMENT AND PLANT CONTROL

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22. All these radioactive uses as well as other control applications depend on the development of reliable and relatively simple electronic circuits. By using transistors to the fullest possible extent, maintenance problems and down-time can be greatly reduced and the problem of replacement parts can be minimized.

23. There have been many developments of control equipment for mills and extraction plants in recent years and it may be useful to mention a few here that have been developed in Canada. Equipment is now available to control automatically the load of grinding mills, the conductivity of leach solutions of metalliferous ores, the oxygen content of cyanide solutions in gold extraction, or the iron content of iron ore concentrates. Considerable effort has been devoted to improve process control in the extraction of uranium. Automatic picker units have been devised to remove non-radioactive gangue from mine ore; methods for the rapid assay of ore and automatic control of effluent solutions have also been introduced. Many of the lessons learned in the uranium industry are now being applied to other branches of the mining and metallurgical industries. This is particularly relevant to some of the older industries like coal mining, gold mines, iron mines and steel plants. In all of them there is a gradual awareness of the benefits that can accrue from a detailed knowledge of the plant process and the direct control of plant conditions. Most of the instrumentation involved has been used in the laboratory for many years, but it is only in the more modern plants that routine records are kept continuously of such variables as pH, moisture, density, temperature, porosity, gas content, conductivity or pressure.

24. As the more mechanically-minded among the metallurgists are converted to accepting such electronic instruments as commonplace in the beneficiation and refining of metalliferous ores and other minerals, the level and value of production of such materials is bound to improve.

25. It may well be that those countries where a mineral technology is started without too many entrenched preconceptions may find it easier to proceed to optimum conditions of semi-automatic process control. The lessdeveloped countries are fortunate that much of this control technology is available for their use and can be readily adapted to their needs. By grasping these opportunities they will help to develop their own mineral resources and to utilize them as effectively and economically as possible.

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