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**Speaker**

## GEOCHEMICAL EXPLORATION FOR ORES

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**O**RES — natural concentrations of metals which can be economically mined — are rare formations in the earth's crust. As they become more scarce, finding new ones grows increasingly difficult. Yet, the current and projected rate of metal consumption is so rapid and the pace of exhaustion of ore bodies so inexorable that locating new sources of metals is of utmost importance for the survival of modern civilization.

The earliest methods for locating ores relied solely on man's senses. An outcropping differing from the local terrain in luster, color, texture, or crystal structure would catch his eye and spark his interest. Copper and gold were first to be found and used, dating back to the Neolithic Age some 9000 years ago. Widespread as native metals, they were also easily melted and wrought into ornaments, utensils, tools and weapons. By 300 BC copper deposits on Cyprus had become so valuable that the island was honored by five successive invading nations and wound up occupied by the ancient Romans who returned the honor by naming the metal **cuprum**.

Native copper was quickly depleted everywhere. Then like most other metals, it could only be found locked within compounds, thus not detectable by human eyes. By the time of the Renaissance, man had to apply technology to locate these ores. A compass held vertically — a so-called dip needle — helped him spot iron-bearing magnetite. Rubbing pyrite with acid, he was able to differentiate iron ores from gold ores.

With the industrial revolution in high gear have come more sophisticated analytical tools. Airborne magnetometers now routinely map large areas seeking field discontinuities in rock structure below. Seismic techniques — relying on echoes from depth charges — electrical resistance and gravimetric measurements help locate disruptions in the rock structure. Radioactivity measurements are specific for uranium and thorium. As they decay, they release radon gas which trickles to the surface emitting gamma rays. Overall, geophysical surveying has probably been responsible for the discovery of the bulk of ore bodies in recent decades.

But as the demand-to-consumption ratio edges relentlessly higher, geologists are pressed for supplementary searching methods. Of these the most encouraging is geochemical pros-

pecting. Instead of physical differences, chemical differences between ores and surrounding rocks are mapped; the data are then combined with known behavior of ore metals during geological events. Isoconcentrational lines of metals occurring at trace levels in soil, water, plants and peats help to determine the presence of an ore body. Such prospecting was first seriously considered in the mid-1930's in the USSR and Scandinavia. Since then geochemical techniques have been sharpened, by refinement of analytical methods and computer treatment of raw data, and have become particularly valuable where geophysical methods have proved notably unsuccessful: with Mo, W and certain types of Cu, Zn, and Au ores.

A key distinction between geophysical and geochemical methods is specificity. Through the former some local discontinuity is sought. This abrupt change in magnetic, electrical or seismic field must first be sufficiently large to affect the needles on the gauges. It is, however, quantitative not qualitative; the cause of the discontinuity remains to be investigated by other techniques. By contrast, geochemical prospecting can reveal specific metals at a distance. This is made possible because ore metals are dispersed in the surrounds of the ore bodies. Geochemical anomalies, where the metallic contents of minerals are higher than normal, may thus lead to a recoverable ore body. Dispersion, incidentally, can occur in two different historical contexts. When studies reveal that the dispersion of the metals has taken place in solid rocks contemporary with the ore formation, it is referred to as primary dispersion. It is secondary when resulting from subsequent breakdown and redistribution of the components of rocks and ores under the weathering conditions prevailing at the earth's surface. Secondary dispersion can be observed by sampling and analyzing surface or near-surface materials such as soil, water, stream sediments, living or dead organic materials. The geochemical method includes regional and detailed prospecting.

Assuming that the dispersion from an ore body is horizontal, regional prospecting becomes a method of general reconnaissance for many long-range, large-scale projects. It is mostly a screening technique, separating barren areas from those where ore bodies can be expected. Sparse sampling — only a few samples per sq km — makes this technique attractive for it can be economically feasible over even thousands of sq km.

As distinguished from regional, detailed prospecting is applicable where the dispersion of ore metals is limited to a more or less vertical plane. Confined in this manner, concentrated dispersion of metals encourages the prospecting team to take as many as hundreds of samples per sq km as they close in on a deposit.

### Rock Geochemistry

It has long been recognized that certain rock types are genetically related to certain ores. Tin ores are associated with potassium-rich granites, chromium with ultrabasic rocks and nickel ores with both basic and ultrabasic rocks. This is not to say that the converse is true, however: potassium-rich granites do not always denote a tin deposit. Geological mapping of rock types therefore yields only preliminary information, revealing where the geological environment is hospitable for ore formation.

To sort out the real ore-bearing rocks from decoys, geochemical as distinct from geologic maps are prepared. Higher-than-normal traces of Sn, Li, B, and F in granitic rocks indicate potential tin mineralization. High concentrations of copper in granitic intrusives (and in biotite from these) have been correlated with copper ores of the porphyry type. In basic rocks, high Cu and Ni are favorable indicators for mineralization of Cu-Ni sulfides if at the same time their S-content is high.

Regional prospecting of this kind can thus help pinpoint local areas for further exploration. Isochemical boundaries for trace-metal constituents are drawn and analyzed together with data from other techniques. Dispersion "aureoles" of ore metals are formed both vertically and horizontally contemporary with the ores and diminish logarithmically with distance from the ore contacts. In rocks which do not react with the ore-forming agent, aureoles can be observed hundreds of meters from their mother ores. In reactive rocks such as limestone and dolomite the extension is reduced considerably and sometimes completely unobservable. A few elements which themselves are not ore-forming often form aureoles of much larger dimensions than ore-forming elements. Mercury is one such "pathfinding" element. Highly volatile, it is frequently found at large distances from its origin in certain sulfide ores. The U. S. Geological Survey reported in 1969 (1) that even the air above a deposit was laden with volatile mercury. At Cortez, Nev. the distribution of mercury in samples taken by a plane flying at 200 feet altitude correlated well with a deposit of gold-bearing rocks covered by as much as 100 feet of gravel. A copper deposit was outlined in like manner.

### Secondary dispersion methods

Secondary dispersion of ore elements is restricted to the earth's surface. Minerals containing these elements have weathered over the ages. Igneous rocks and ores derived from them were generally formed at high temperatures and pressures inside of the earth's mantle. When brought to the surface by

geological processes such as upheaval and erosion, they became unstable in their new environment. Lower temperatures and pressures, oxidizing conditions and the action of water and ice caused the rocks to change slowly but irreversibly. This weathering has been defined as "the change in rocks from the massive to the clastic state."

These massive rocks have changed as a result of physical as well as chemical weathering. Physically, the release of pressure on the rocks and ores when they are brought to the surface causes cracks along mineral grain boundaries because of differential rates of expansion. Widened by climatic temperature variations, freezing rain water, roots of plants, etc., the cracks weaken the rocks still further. So weakened, the rock is then attacked by other physical forces — wind, water and ice in motion — which may totally crumble the originally solid structure.

Chemical attacks on minerals compound the physical attacks. Over the eons, the two work together symbiotically. By increasing the surface area, physical weathering fosters chemical weathering. The chemical changes then promote further physical attack. Chemical weathering itself consists of a complicated interplay of a number of processes including hydration, hydrolysis, adsorption, oxidation, reduction, dissolution, precipitation, formation of solids and gels. These reactions take place mostly in an aqueous medium which makes water dominant in the chemical weathering process.

In its relentless chemical attack on minerals, water is assisted by two other agents: dissolved atmospheric gases and biologic matter. Carbon dioxide acidifies the water, enhancing its ability to dissolve many minerals. Oxygen oxidizes metals such as Fe and Mn, taking them to higher valency oxides and hydroxides. At the root tips of growing plants, very low pH conditions prevail, again aiding water in the chemical weathering process.

Once the minerals at or near the earth's surface have weathered, they seldom remain in place. As layers of loose materials on top of solid rock or solutes in water, they are transported and dispersed in the surroundings by gravity along slopes, wind, running water and moving ice including glaciers. To explore a mineralized area geochemically, it is thus necessary to sample water, soil, stream sediments, lake sediments, living and dead organic materials. And, as we have seen, even the atmosphere above.

### Water

Water being most important, it seems most natural to emphasize analysis of streams and pools both above and below ground. Water which participates in ore weathering will contain higher-than-normal concentrations of the ore metal ions, especially of those metals which form salts of high solubility. Although some of these may be lost by adsorption in clay minerals and hydroxides, often enough remains to be detected by modern chemistry.

Different bodies of water vary in value as exploration media. In principle, ground water should be very valuable in detailed prospecting but the difficulties entailed in obtaining samples often precludes analysis of this source. Where ground water comes to the surface in springs sampling is easier. But easiest to sample is a stream since sooner or later, ground water enters a stream. In spite of dilution from upstream barren areas, stream waters often are found to contain anomalous ore metals by sensitive chemical techniques. Certain mobile elements such as U, Mo, and Zn lend themselves to this type of prospecting. In northern Sweden, the first indication of a promising uranium deposit was obtained from water and organic samples. Stream water is being sampled every 200m in northern Sweden, a sampling density of about 3-6 per sq km.

Lake water is another source of geochemical samples. However, it does not appear to be advantageous over stream water except in inaccessible areas such as northern Canada where the lakes can be conveniently sampled with the aid of a helicopter.

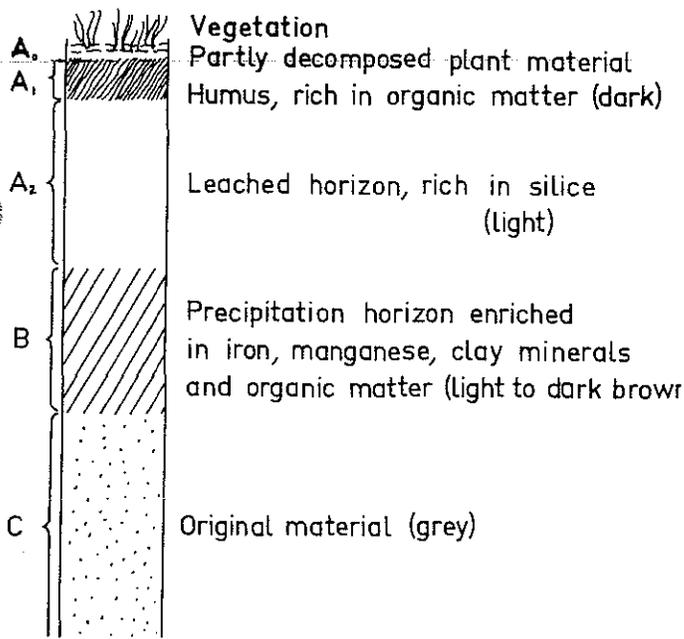


Fig. 1 Soil profiles vary widely depending on local and climatic conditions. Generally, three main horizons can be observed, A, B, and C, and often they can be further subdivided. A<sub>0</sub> consists of dead organic material in varying degrees of humification. A<sub>1</sub>, a mixture of dead organic and inorganic material, is generally darker colored. A<sub>2</sub> is light colored because organic material has been leached away by percolating rain water. Also leached out are clay particles and hydroxides so this layer consists mainly of weathering-resistant rock minerals. B, colored yellow to brown, contains much material leached from the A horizon above; its color results from the accumulation of iron and manganese hydroxides. C<sub>1</sub>, often gray in color, is the more or less weathered loose material from which A and B have developed.

### Soil

The upper part of the loose material produced by weathering, blanketing solid rock below, is usually differentiated in

layers, "horizons," forming what is called soil. All three of the layers depicted in Fig. 1 can be sampled for detailed geochemical prospecting; as many as 100 or more samples per sq km are taken. Anomalies in soil can be caused by the presence of weathering-resistant primary ore minerals, secondary ore minerals or by ore metals adsorbed by clay particles and hydroxides in the soil.

The position of a soil anomaly in relation to the ore from which it originates is different in residual and transported soil. In residual (unshifted) soil the anomaly is situated more or less above the ore (Fig. 2) but covering a somewhat larger area because of dispersion influences already mentioned. In transported soil it is dislocated in the transported direction (Fig. 3).

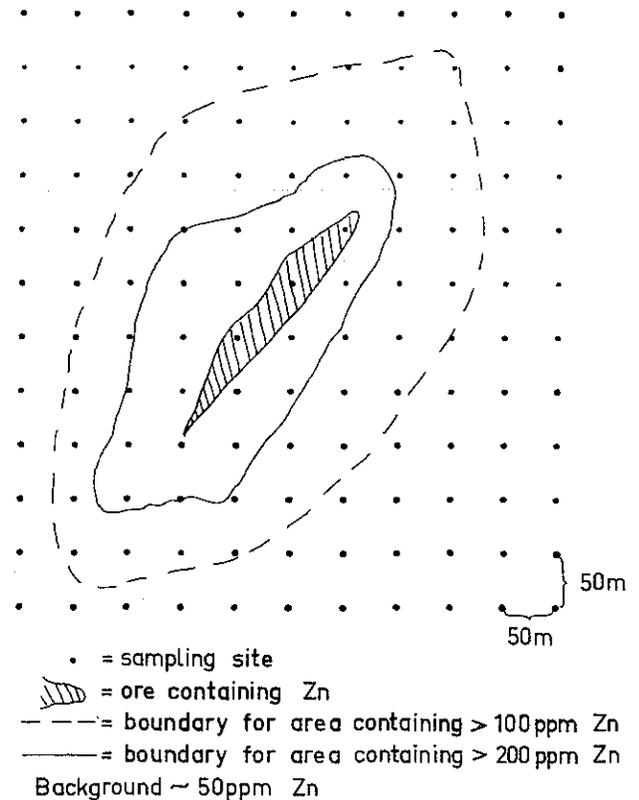
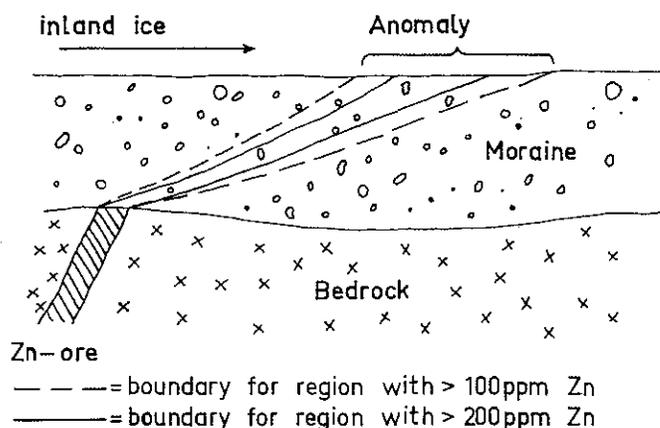


Fig. 2 Mapping a residual soil above an ore body shows traces of anomalous elements the peak concentrations of which are found directly above the ore.

### Stream and Lake Sediments

When a stream cuts through loose material covering solid rock, much of the finer constituents (clay minerals, hydroxides) are often swept away and only coarser material remains in the stream bed gradually moving downstream.

When passing an area, where the original loose material was anomalous because of the presence of ores, the stream sediment can be anomalous for several reasons. Ore grains resist-



**Fig. 3** Mapping a transported soil shows a metallic anomaly physically removed from the ore body below the surface. In this hypothetical illustration a glacier moraine has transported zinc ions. Such dislocations can amount to hundreds of meters in regions once covered by inland ice. To pinpoint the mother ore body, it might be necessary to take samples deep within the glacial till, "upstream" from the observed surface anomaly. Such samples can be taken in trenches or with the aid of special drills.

ant to weathering and secondary ore minerals can be constituents of the stream sediment. Clay minerals with adsorbed ore metals can remain in the stream sediment, especially if the current is not strong. Finally, ground water with anomalous contents of ore minerals can enter the stream where the ore metals are coprecipitated with the limonite or fixed by organic material.

In this way a stream sediment anomaly is formed and, gradually moving downstream, it can have a considerable extension (often several km) making stream sediments very useful in regional geochemical prospecting.

Lake sediments have also proved worthwhile in geochemical prospecting. In the permafrost regions of Canada, the bottoms of lakes are "windows" in the frozen ground. Here ground water can reach the bottom to deliver metal ions to clay particles and other adsorbing agents.

### Organic Material

Through their roots, plants take up water and dissolved salts from the ground. In the neighborhood of ores, unneeded ore metals can be taken up as well as those vital to the growing plant. The physico-chemical laws regulating the uptake of different elements are, however, complex and not well understood. The reliability of plant analysis for geochemical prospecting is thus questionable. Plants can, however, guide the observant ore prospector in another way. Certain plants prefer growing in soils with high contents of specific metals. When subjected to unusual metallic environments, others develop characteristic variations in the growth or color of their leaves or flowers. Geobotanical observations have frequently disclosed the presence of underlying ores. Copper mosses, for example, grow only over copper deposits (2). Increasingly,

airplane and satellite surveys are being conducted with color sensitive film to spot subtle reflectivity differences in the green, red and infrared regions.

When a plant dies, the speed at which it decomposes depends on local climatic conditions. In unusual circumstances, the organic material accumulates faster than it decomposes, forming swamps and bogs. Dead organic material in different states of humification (peat) has a great affinity for metallic ions binding them as metallo-organic compounds (3). Often the ash of such organic matter will reach upward of one percent, clearly indicating the presence of an ore. Peat bogs, which occur over large areas of Canada and Scandinavia where the climate is cool and humid, have been subjected to both detailed and regional prospecting. Because of the circulation of water seeping up from underlying bedrock and moraine, the presence of an ore can be detected by sampling and analyzing the peat. In contrast with stream sediments, organic samples from stream banks are always available in glaciated regions. Further, the high contents of ore metals facilitates analysis and anomalies are more continuous and elongated, permitting longer distances between sampling sites. For regional exploration of U, Mo, Cu, Pb, Ni, Zn, etc., in Sweden, organic stream material has almost totally replaced stream sediments. As reported later, sampling of swampy banks of streams in northern Sweden has led geologists to what may prove to be important deposits of U and Cu.

### Analytical Methods

Requirements of analytical methods in geochemical prospecting are stringent. They must be sensitive, ore metals occurring in the ppm range in solid samples and ppb range in water. They must be accurate to about  $\pm 20\%$  and they must be inexpensive, rapid and amenable to large-scale runs. Preferably, they should be multielement techniques because in at least regional prospecting unexpected ore metals may be as significant as specific ones sought. By virtue of this last stipulation, the emission spectrograph has become a powerful tool. One example of its applicability is in studying stream sediments. Here the limonite content (Fe + Mn) affects the content of Zn, Mo and to some extent Pb.

Of all known solution methods, atomic absorption has emerged as the most widespread. Earlier methods such as colorimetry, spot tests and paper chromatography, have been largely replaced by AA because of its inherent high accuracy and speed.

On the other hand, optical emission is the most important method for the analysis of solids which, in Sweden, are routinely studied for 30-40 elements. Speed and accuracy are high particularly with the so-called tape machine developed by Danielsson at the Geological Survey of Sweden (4). With this device, powdered samples are first mechanically picked up by adhesive tape. They are then transported between two electrodes and sparked in place. Intensities of selected spectral lines are fed to a computer which types out

concentration figures within seconds. Limit of detection of this technique is sufficient for most elements in prospecting with a few important exceptions: Au, U, W and Y. Trading sensitivity for higher analytical accuracy, x-ray spectroscopy often supplements optical emission in geochemical prospecting.

Treatment of the large mass of analytical data that pours out of geochemical prospecting laboratories is virtually impossible by normal hand statistical calculations. The computer has become indispensable. Through factor analysis, inter-correlation among different elements can be studied. To take into consideration the influence of matrices such as limonite and organic material, stepwise regression analysis can be applied. When fed raw data on geochemical samples, computer programs have been perfected to such an extent that they can relate these to the probable content and location of ore bodies.

By about 1960 the number of geochemical samples collected annually was already running in the hundreds of thousands. Having developed steadily during the past few decades, geochemistry is today recognized as an invaluable tool for ore exploration. Millions of samples will probably be analyzed this year.

**A**LTHOUGH examples of the successes of geochemical prospecting are numerous, only a few will be cited.

In 1966 the Swedish Geological Survey undertook a conventional regional survey in the Bittangi area, north of the Arctic Circle. Stream sediments were routinely collected and analyzed at intervals of 200m until success appeared. Along one stream near Kiskamavaara Mtn. highly anomalous contents of Cu were found. Yet no ore boulders or outcrops were known in the area. Further detailed prospecting was conducted through 1968. In an area of 4 sq km around the stream sediment anomaly, soil samples were taken along lines at right angles to the direction of the former inland ice. Separation between the sampling lines was 100m; between samples, 50m. An extensive soil anomaly was discovered: 60 to 2000 ppm against 25 ppm background. An electromagnetic survey was next carried out and combining information from the two, geologists drilled 10 holes. Nine of these passed through a mineralized zone consisting of 0.7% Cu. The deposit may prove economically interesting.

A current survey involves uranium. After uranium anomalies were found in several places, a comprehensive Swedish project was begun in which the entire country is being surveyed. A sparse net of organic samples was collected at road-stream intersections. During the summer of 1971 several anomalies of U and other heavy metals were obtained from an area in northern Sweden where little is known geologically. In 1972 streams feeding Lake West Kikkjaure were found to be highly anomalous, containing 100 ppm of U compared to <30 in the background. Geochemical and radiometric work is continuing this summer in the area which now is considered most promising as a potential source of uranium.

In its continuing program, the U. S. Geological Survey has at its disposal 26 mobile units. Six are outfitted with emission spectrographs, 9 are chemical units, 7 are reserved for sample preparation, 4 are generator-grinding units. By taking the laboratory to the samples rather than the other way around, efficiency has been impressively upgraded. Mineralization trends can be appraised on the spot thus helping prospectors decide on where and how frequently to take samples. Heavy bulk samples need not be transported over long distances.

In 1966, the USGS discovered a gold geochemical anomaly in central Nevada, the Cortez district (5). Previously established as an area containing anomalously high concentrations of As, Sb, and W, the area was found to contain high gold contents as well. Intensive sampling, 238 altogether, ultimately revealed an outcropping only 100 feet long with a Au content of up to 3.4 oz/ton. Even at the then-current price of around \$40/oz, the ore was valuable enough to be mined. At today's prices, nearly \$100/oz. as little as a few tenths of an ounce per ton represents a commercially recoverable ore. To those familiar with more traditional units, 0.09 oz/ton is equivalent to 0.3 ppm. Put differently, even with the emission spectrograph's unrivalled sensitivity it cannot, without prior concentration of the ore, detect gold at levels where it is economic to be mined.

A report issued last year (6) by the USGS has stimulated considerable exploration in the Coeur d'Alene district in Idaho. The most extensive silver-producing district in the United States, it was recently found to contain a number of other valuable metals including Sb, Te, Pb, Zn, Cu, Mn, Hg, As and Cd. Approximately 8000 soil samples and 4000 rock samples were collected along roads and ridges with a sample spacing of about 500 ft. Total traverse covered about 200 sq mi. Computer-generated geochemical maps reveal where the metallic content of soils is higher than that of rocks. Many of the metals are zoned horizontally as well as vertically. Patterns of trace silver clearly delineate the silver-bearing deposits; anomalous silver and lead outline gold and iron bearing galenas. Tellurium seems to have been driven well beyond the ore bodies, forming haloes far out from the centers of mineralization. According to the authors of the study, Te would seem to be an excellent pathfinder for deeply buried deposits in the Cortez region. Cadmium, almost always associated with zinc, is more temperature sensitive than the latter. The two metals tend to fractionate from each other in hot intrusions. In monzonite intrusions found in the Cortez district, Cd is found to be depleted with respect to Zn in veins. Because of the high temperature of the intrusion, Cd, like Te, has migrated in distant haloes. It, too, is a helpful pathfinder element.

Cripple Creek, Colorado, its hills pocked by numerous gold-mine shafts dating back to 1891, is being explored again by the Golden Cycle Corporation. Interest was largely stimulated by a report issued by the USGS in 1969 (7). In 1962,

the last active mines were closed threatening the economy of the district. As a result, USGS undertook a geochemical study in 1964, seeking extensions of known deposits or low-grade gold deposits near the surface. Several extensive Au-Ag-Te anomalies have been discovered. Au and Te were determined by an optical emission semiquantitative technique developed by Myers (8).

The United Nations has been active in exploring mineral resources of developing countries. Claude Lepeltier, who operates from his office in New York, is the technical adviser of its Geology and Mining Section. In this capacity, he travels to almost inaccessible hinterlands throughout the world. In cooperation with the Geological Surveys of the respective countries, the UN is currently conducting geochemical explorations in Africa (Morocco, Egypt, Mauritania, Ethiopia, Somalia, Senegal, Upper Volta, Togo, Dahomey, Rwanda, Burundi, Lesotho); in Latin America (Honduras, Colombia, Argentina, and Haiti); Yemen in the Middle East; India and Burma in the Far East. Last June, 21 UN mineral surveys were in progress. From 1970 until June, 1973, the number of samples collected amounted to 600,000 and the number of determinations, around 5,000,000. About 70% of these were from stream sediments, 25% from soils and 5% miscellaneous (rock, drill-core, water, vegetation). Atomic absorption has been the major analytical tool but a few emission spectrographs are in operation, one in Burma.

Results of the UN operation have proved impressive. In Malaysia, a 1965 UN survey of stream sediments indicated important copper anomalies in the headwaters of the Mamut River. Through later work by the Borneo Geological Survey, the actual ore body, estimated at 80-90 million tons and averaging 0.7% copper was discovered. Permission to develop was recently granted to a joint Japanese-Malaysian venture, the cost of which is estimated to be around \$70-million.

Geochemical prospecting by the UN has also unearthed a Cu-Mo deposit in Ecuador estimated at 80 million tons containing 0.7% Cu. In Panama UN teams have located ores containing valuable Cu, Au, Zn, Mo and Ag. One immense find in Chiriqui Province is estimated to consist of 2000 million tons grading 0.8% Cu. Copper has been found in Argentina by another UN team, Falconbridge having contracted with the government to follow up. Still other important copper deposits have been found by the UN in Mexico, Chile, and the Philippines.

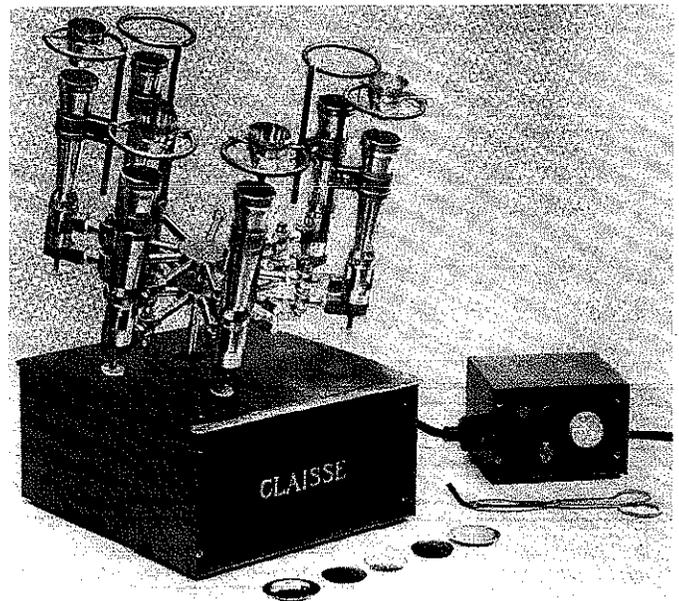
Lepeltier concludes his current report (9) with a paragraph relevant to this article, too:

"The relative importance of geochemical prospecting in the United Nations mineral surveys has increased steadily and is now established as a key technique in nearly all exploration undertaken. It has achieved its best results in reconnaissance

programs in the tropical or equatorial environment . . . in very rugged topographical conditions, helicopter-borne sampling programs have proven very effective . . . , the high transport cost being largely offset by the speed of coverage . . . ."

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## TETRABORATE FUSION FOR X-RAY SAMPLE PREPARATION

Fernand Claisse, University of Laval, Quebec, Canada

Developed 17 years ago by the author, the tetraborate disc technique has become well recognized for sample preparation prior to x-ray spectrochemical analysis. Fused with borax or lithium tetraborate, the sample is cast as a flat disc. Most variables are thereby eliminated, including particle size, microheterogeneities, surface roughness and mineralogical differences. The major variables remaining, interelement effects, are easily overcome by internal standardization, dilution, spiking or numerical corrections.

The original "Borax Bead" method consisted of fusing the sample in sodium tetraborate (borax) then casting the molten mixture into a ring. The operation, however, was delicate: internal strains often caused the disc to shatter eerily; material adhered to the platinum crucible; samples emitting gases developed tenacious bubbles which played havoc with accuracy. Some frustrated experimentalists compromised, grinding and pressing the powdered fusion into a pellet, settling for only the homogenizing advantage of the fusion. This also defeated one of its main purposes: to save time in sample preparation.

Two innovations are rapidly reviving the technique. One is the development of a non-wetting platinum-gold alloy. The other is the Claisse Fluxer, a semiautomatic device for fusing and casting (without sample transfer) up to six discs simultaneously. The discs so obtained are stable, homogeneous, bubble-free, transparent and have a smooth surface ready for insertion in an x-ray sample holder.

In the Claisse Fluxer (pat. pend.) six individual gas burners are on a special gyroscopic joint providing movement similar to hand agitation for each of the stations. Speed is adjustable through a motor controller separated from the apparatus for safety and comfort. After about five minutes of heating and agitation, the samples are usually ready for casting, accomplished by inverting the crucibles so the melts drop into the lids which double as molds. In one motion, one knob inverts all the crucibles and in three minutes samples may be slipped from the mold, cool and ready to be analyzed.

The special crucibles and lid-molds required are supplied by Englehard Industries and Matthey-Bishop, Inc. Since, among other things, successful casting depends on a flat-bottomed mold, these platinum manufacturers provide a reforming tool to keep it in shape through repeated usage.

If occasionally a droplet of fused material adheres to the wall of the "non-wetting" crucible, it can be removed by swishing molten flux inside the crucible on one of the burner stations. The cooled melt will solidify and separate clean.

Because of its relatively low cost, borax is preferred for the determination of easily detectable elements: those occurring in major concentrations and those of high atomic number. Lithium tetraborate is needed when the fluorescence intensities are low: for light elements and trace concentrations.

Additives are recommended for specific purposes. Heavy elements such as barium peroxide or lanthanum oxide increase the absorption of the glass thus decrease interelement effects. A greater dilution of borate: sample, however, provides the same result and saves a weighing operation.

Sulfide ores do not dissolve readily in borax and may react with the platinum crucibles. This can be avoided by oxidizing the ore before fusing it. An often better way is to combine the oxidation and fusion operations. The ore is mixed with a few times its weight of barium peroxide ( $BaO_2$ ) and placed in the bottom of the platinum crucible. The flux is poured above the mix. At the beginning of the heating the ore is oxidized, then as the flux melts the oxidized sample dissolves. Internal standards, if called-for, may also be added to the bulk flux, another time-saving step.

Metal samples can be fluxed after prior oxidation by heating in air or oxygen. They can also be dissolved in an acid and evaporated to dryness before fluxing. Another method found successful by the author is to heat the metal together with a small amount of potassium pyrosulfate until dissolution and then add borax. Organic materials should be ashed before fluxing.

With easily detectable elements such as Fe, Cu, or Pb, at concentrations above 1%, a flux: sample ratio as high as 100:1 is suggested. At such high dilutions, interelement effects are negligible and x-ray intensities are conveniently proportional to concentrations up to nearly 100%.

Lower dilution ratios of the order of 5:1 are recommended for determining elements below 1%. The only limit to the lower dilution ratio is the solubility of the material in the flux; ratios as low as 1:1 to lithium tetraborate have proved satisfactory for the preparation of Portland cements. Naturally, interelement effects increase with increasing sample: flux ratios and must be compensated for.

Calculations of concentrations from x-ray intensities are straightforward, less troublesome and more accurate than with other techniques because secondary interferences are normally absent.

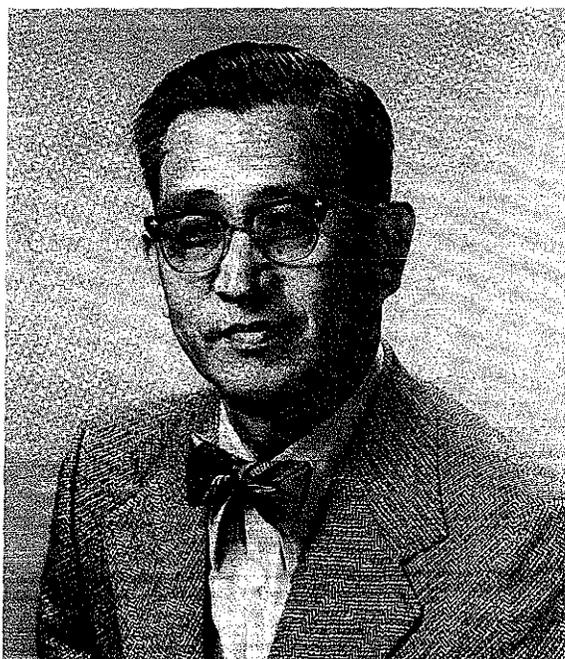
To summarize, the borate disc technique is applicable to almost any kind of material. With the Claisse Fluxer, fusion and casting of glass discs are simple and rapid operations, producing at least 36 discs per hour. Little, if any, personnel training is required.

## MEGGERS AWARD TO M. S. WANG

Continuing the objectives of the original Appl. Spec. Journal award initiated in 1961, the SAS Meggers Award is sponsored by Spex Industries. We are especially proud of this year's recipient because his subject, trace element analysis by emission spectroscopy, is close to the domain in which our business was launched.

The impact of M. S. Wang's paper, "Impurity Determination in Group III-V Compounds," is centered in two tables. Here, presented in typical scientific understatement, are presented detection limits of some 20 elements ranging from 0.05 to 40 parts per billion. As any recent text on the subject will authoritatively reveal, the limits of detection of these elements are in the ppm, not ppb, range. Yet, by squeezing double service out of his arc (preconcentrating impurities by boiling off the matrix and then exposing his photographic plate), by diligent optimization of parameters, by scrupulous avoidance of contamination, Wang has beefed up the detectivity of the emission spectrograph to a concentration range once reserved for nuclear activation spectrometry.

In so doing, Wang has greatly broadened the applicability of the emission spectrograph. As technology taxes the purity of materials still further, his paper will increasingly serve as a model and inspiration to other analysts faced with related problems. We salute Dr. M. S. Wang.



## SHATTERBOX UPGRADED

Thanks to its imposing number of installations, the Spex SHATTERBOX, first introduced in 1965, has become a labhold word. In under five minutes it pulverizes most samples. Even the toughest rocks, slags, ceramics, fluxes, glasses, and what-have-you succumb, swished to a dust fine enough for subsequent x-ray or emission spectrochemical analysis.

Although the SHATTERBOX has benefitted from numerous, unnoted minor surgeries over the years, we have now made a significant design improvement. Through several mechanical changes, the eccentric momentum has been substantially increased to intensify the grinding action and meaningfully cut grinding times. Not only did tests in an independent mining laboratory favorably rate the revamped SHATTERBOX with "the other brand" swing mill — noisier and more subject to breakdown, according to the investigators — compared with its own vanquished predecessor, the improved SHATTERBOX ground ASTM Standard Sand in a Hardened Steel and WC container for 5 minutes each, then dished out these effective results:

Mesh	43g in 8501 Steel		28g in 8504 WC	
	New	Old	New	Old
-325	100%	93%	100%	97%
-400	90	50	95	60

Also new with the SHATTERBOX is something dozens of you out there have asked for: a smaller capacity, and incidentally less expensive, tungsten carbide grinding dish. Here it is, the 8508. Identical in outside dimensions to the 8507 Hardened Steel Dishes, up to three 8508 WC Dishes may be loaded into the 8507R Rack. Its capacity of 95 ml is just under half that of the 8504 but is ample nonetheless for most x-ray spectrochemical sampling.

What happens to 20g of ASTM Standard Sand when three samples are ground simultaneously?

Mesh	8508	8508	8508
	Pos. 1	Pos. 2	Pos. 3
-200	100%	100%	100%
-325	77	77	77
-400	70	66	66

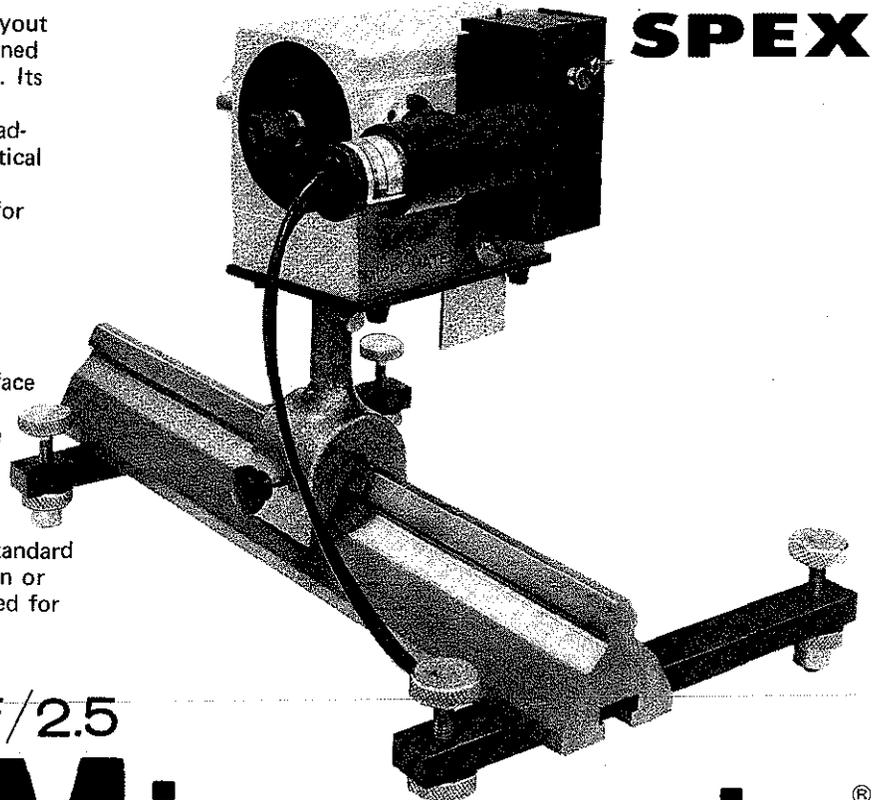
**8508 Tungsten Carbide Grinding Dish**, 95 ml total capacity, up to 30 ml grinding capacity (requires 8507R) . . . . . ea \$ 700.00  
3/ \$1960.00

**8507R Rack** for holding up to three 8507 or 8508 . . . . . ea \$ 110.00

An ingenious coupling of a Cassegrain optical layout and unique slit mechanism (Pat. Pend) has spawned the Spex Micromate, a versatile monochromator. Its low pricetag belies its extraordinary features. Compact, with an in-line optical path, digital read-out of wavelength and remarkably free from optical aberrations, the Micromate is ideal for many biological, chemical, and physical experiments, for student demonstrations of diffraction, as a component in commercial instruments or as a tunable filter for a microscope.

Unlike ordinary slits which consist of pairs of razor-sharp edges, the slits of the Micromate are defined by the space between the reflecting surface of a prism and the edge of a fixed aperture. As the prism is translated toward or away from the aperture, slit width is changed.

Spex Micromate is provided with a post to fit standard optical riders. Motor may be positioned as shown or inverted 180°. Either way, it is readily disengaged for rapid hand scanning.



**HIGH OPTICAL SPEED**

**NEGLIGIBLE** { COMA  
REDIFFRACTION  
SCATTERED LIGHT

**HIGH WAVELENGTH ACCURACY**

**IN-LINE OPTICAL PATH**

**3-DIGIT COUNTER, nm READOUT**

**CONVENIENCES:**

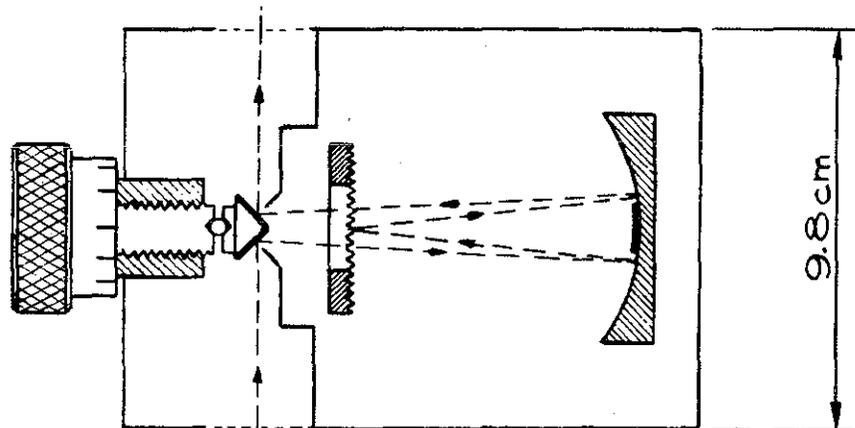
- coupled slits calibrated in bandpass units
- accessory mount available
- variable positioning
- small (17.6 x 10.4 x 12.5 cm)
- rugged, never needs adjustment

**Micromate your**

- Nephelometer
- Fluorometer
- Microscope Illuminator

f/2.5

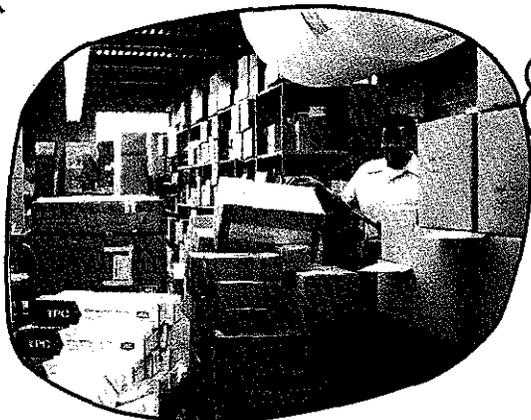
**Micromate<sup>®</sup>**  
100-mm MONOCHROMATOR



The optical diagram shows that after a 90° reflection from the first prism face, light passes through a slot in the grating, then to one side of a concave mirror, with a central stop. The light is collimated by the mirror to the grating, where it is diffracted, and reflected back to the mirror. After the second reflection from the mirror, the light is focused on the exit slit half of the slit-prism assembly. This design eliminates rediffracted light.

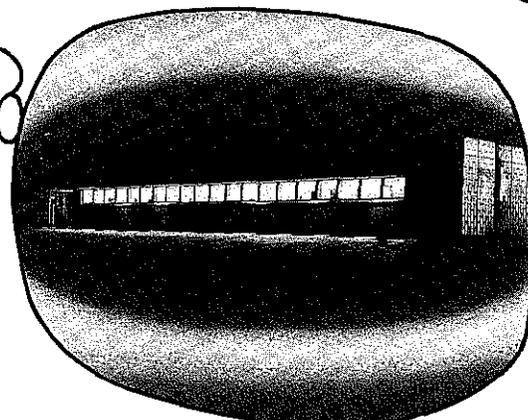
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Our precision automatic balance is faster, cheaper, more accurate and probably more enthusiastic about weighing diluents than your lab technician!

Individual chemicals or any mixture you specify are loaded to a tolerance of  $\pm 2$  mg into plastic or glass containers ready for the addition of your sample and popping into a Spex Mixer/Mill or Wig-L-Bug.

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May we send you a sample of high-purity preweighed KBr, in a little glass vial in which you can mix your sample and even store the 13 mm pellet when its work is done? Delivered, protected with dessicant in a double-sealed can, 200 mg portions, each with a 1/8"  $\phi$  stainless steel ball, are \$236.00 per 1000.



May we send you a price list of materials typically supplied or quote your special mixture or unit weight for your requirement? We'll work with your material or ours.

While we think of it, perhaps you'd like our Sample Preparation, Pure Materials, Standards and Supplies Catalogs. The catalogs, as well as most items in them, are in stock for immediate delivery.

## 7155 DECOMPOSITION CHAMBER

Whether called decomposition vessels, decomposition bombs, acid digestion bombs, or decomposition chambers, these devices are all similar and serve the same function: to hasten dissolution of difficult-to-dissolve substances. The sample is confined together with a strong acid or base in a sealed Teflon crucible and heated. Metallic contamination is avoided, volatile components are retained and the entire sample is recoverable.

The new Spex Decomposition Chamber incorporates all of the best features of each of the existing commercial units. Its capacity is 35 ml, large enough for preparing samples for x-ray fluorescence, emission, AA, and most wet chemical procedures. It is constructed entirely of 18-8 stainless steel and Teflon to deter corrosion and assure long, trouble-free life. The Teflon crucible has a flat bottom, and a Teflon-coated stirring rod is included to expedite reactions on a magnetic hot plate.

Safety was a prime goal in the design of the Chamber. In the unlikely event that excessive internal pressure does build up, the Teflon lid on the crucible will rupture. Pressure will be relieved through a hole in the rear of the outer stainless steel jacket, directed away from any likely observer.

Methods have been devised for dissolving organic as well as inorganic materials. A recently published article illustrates the dissolution of coal dust in two steps, first with  $\text{HNO}_3$  then with  $\text{HF}$  at 150C. Subsequent AA analysis was carried out for 10 elements without evidence of contamination or volatilization loss.

A further design goal was interchangeability of components. Although chemically inert to strong acids and alkalis, Teflon does exhibit porosity which will eventually require its replacement.

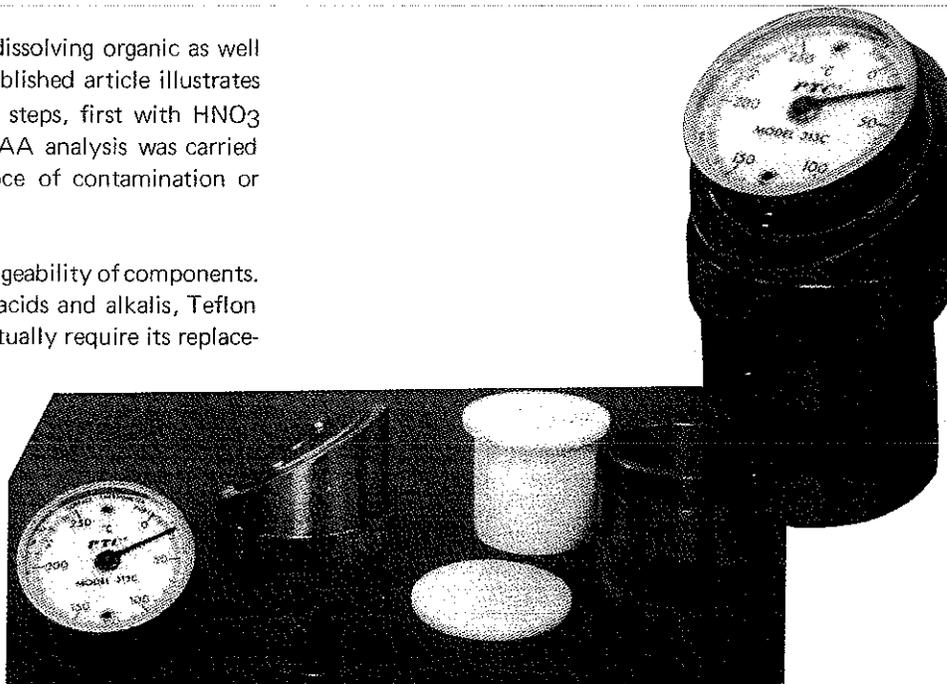
A unique feature of the 7155 is inclusion of a contact thermometer. Mounted obliquely on top of the Chamber, it is conveniently read even when inside a glass-windowed oven and permits accurate monitoring of temperature especially when exothermic reactions are involved. Under such conditions it also adds an element of safety to the procedure by indicating when the temperature approaches the safe limit of 180C.

### FEATURES:

- Long life —Teflon and stainless steel construction
- Safety —Teflon lid is also a rupture disc; blow hole in rear of outer case
- Extra capacity—35 ml
- Convenience —Thermometer for monitoring reactions
- Versatility —Place in oven or on magnetically stirred hot plate

**7155 Decomposition Chamber** . . . . . ea **\$175.00**

**7156 Teflon Crucible**, spare or replacement . . . . ea **30.00**  
6/ **160.00**



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