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COMMONWEALTH OF AUSTRALIA.

DEPARTMENT OF NATIONAL DEVELOPMENT.

BUREAU OF MINERAL RESOURCES GEOLOGY AND GEOPHYSICS.

RECORDS:

1963/109

ECAFE SYMPOSIUM ON GEOCHEMICAL PROSPECTING, BANGKOK AUGUST 1963

Papers presented by the Bureau of Mineral Resources
A.D. Haldane, B.P. Ruxton, E.J. Howard, S. Baker, K.R. Walker

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GEOCHEMICAL PROSPECTING OVER LEAD AND ZINC MINERALIZATION AT McARTHUR RIVER, N.T.

by

A.D. Haldane

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GEOCHEMICAL PROSPECTING OVER LEAD AND ZINC MINERALIZATION AT McARTHUR RIVER, N.T.

INTRODUCTION

The McArthur River flows in a north-easterly direction from the Barkly Tableland into the Gulf of Carpentaria opposite the Sir Edward Pellew Islands. It flows through the survey area which is located approximately 180 miles east of the township of Daly Waters, and 60 miles inland from the coast.

The existence of lead mineralization in the McArthur River area has been known since 1887 but no systematic examination was made until 1955 when Mount Isa Mines Ltd began geological mapping and geochemical prospecting surveys which led to the discovery of a number of new prospects including one (the H.Y.C.) of substantial dimensions. The area contains both lead and zinc mineralization and it would be anticipated that, under normal circumstances, zince would mobilise and disperse giving well defined surface geochemical anomalies. However this was found not to be the case. The early reconnaissance geochemical prospecting by Crabb (1956) was disappointing. The anomalies were weak and ill defined even adjacent to rock outcrop carrying considerable zinc mineralization.

In 1961 the Bureau of Mineral Resources suggested that deep auger drilling might yield better results and A.G. Fricker of the Bureau undertook a geochemical survey over selected areas with deep alluvial cover, to test this method of sampling (Fricker, 1962).

PHYSICAL FEATURES

The general survey area is shown in Plate 1. A large proportion of this area is covered by the flood plain of the McArthur River which consists of alluvium up to 80 feet deep. The McArthur River itself is deeply entrenched in its flood plain and is the only permanent watercourse. A dense undergrowth with tall timber of Eucalypt sp. and Sheoak grow in the immediate vicinity of the river. This gives way within less than 100 yards of the river bank to a savannah woodland of Bauhinia and Eucalypt sp. with open areas covered by Mitchell grass.

The climate is monsoonal with an average annual rainfall of 25" - 30" and a short wet summer season followed by a long dry winter.

GEOLOGY AND MINERALIZATION

The geology of the McArthur River district is described in reports by Smith (1962), Dunn, Smith and Roberts (in prep.), Kreiwaldt (1957), Beresford (1957) and Cotton (1963).

The area of interest lies within the McArthur Basin which extends from the Queensland border in the south to Arnhem Land, in the north, paralleling the south and west coasts of the Gulf of Carpentaria. The rocks of the Basin are of Proterozoic age and are divided into three Groups viz., Roper Group - 15,000 feet dominantly sandstone and siltstone.

McArthur Group - 10,000 to 15,000 feet, dominantly carbonate rocks.

Tawallah Group - 13,000 feet, dominantly sandstone with volcanics.

The strata are mainly shallow-water marine shelf deposits and have been subjected to moderate faulting and folding; dips above 30° are rare.

All the known mineralization occurs within the Amelia Dolomite, a unit near the base of the McArthur Group. The unit is about 6000 feet thick and comprises a complex of biohermal and biostromal dolomite, ferruginous clastics, bioclastics and chemical deposits. Disseminated galena occurs over wide areas and throughout the unit but it is only of economic importance in the Barney's Creek Member, which contains the H.Y.C. lead/zinc deposit and the W-fold and Teena prospects (Plate 1). A substantial lead/zinc orebody has been proved at the H.Y.C. prospect.

In detail the Barney's Creek Member comprises from the base upwards; laminated dolomite, green vitric tuff, lower barren shales, sphalerite-and-galena bearing pyritic shale, barren shale and dolomite-chert breccia. The sequence is dolomitic throughout with many minor beds of breccia. The mineralization shows a marked sedimentary control and is considered to be syngenetic in origin.

SOILS AND WEATHERING

The soils developed on the deep alluvial cover deposited by the McArthur River are grey clays showing pronounced shrinking and cracking at the surface, and are underlain by brown clay commonly carrying travertine, gypsum and salt. River gravels and sands occur at depth. Residual soils are confined to the topographically high areas around Barney's Hill, W-Fold, Teena, and Reward Prospects. Patches of cemented ferricrete occur in places but there is no evidence of lateritisation in the area.

It is difficult to assess the extent of weathering as a large proportion of the area has apparently been stripped to bedrock by the McArthur River and subsequently buried beneath alluvial deposits. These deposits carry salt, travertine, and gypsum indicating little or no leaching of the alluvial soils. Dolomite outcrops are common and in the survey area the Amelia Dolomite carries fresh galena at the surface. In contrast to this are the pyritic shales of the Barney's Creek Member which are oxidized at the surface and carry only secondary lead/zinc minerals.

It is considered that the pyrite, which oxidizes readily, has accelerated the breakdown of the shales both chemically and mechanically resulting in the formation of secondary lead and zinc minerals. No extremes of weathering would be required for this. Diamond drilling has shown fresh shale immediately below the river sediments. It can be concluded therefore that weathering at the surface is mild and there has been no pronounced leaching of either the river sediments or the underlying bed rock.

METHODS AND EQUIPMENT

For sampling in the deep alluvial areas a truck-mounted Proline power auger was made available by Carpentaria Exploration Company. This brings a continuous flow of cuttings to the surface and samples can be taken at various depths while drilling continues. At depths of 20 feet there is a lag of approximately 2 feet taken for the sample to reach the surface from the bottom of the hole. In difficult ground this lag distance may be greater depending on the drilling techniques.

In places inaccessible to the Proline unit a Jarret hand auger was used to obtain samples at depths up to 30 feet. The general procedure adopted was to take a sample of the auger cuttings representing each two feet of penetration. These were laid out in sequence and at the conclusion of drilling samples for analysis taken from the individual piles at various depths, or combined to give any type of bulk sample such as soil, weathered rock, etc.

In using the Jarret hand auger the cuttings accumulate in the auger head and must be withdrawn and emptied at intervals of approximately three inches. Location of the sampling depth is more accurate with the Jarret auger but for holes much in excess of ten feet progress is slow and the auger becomes progressively more awkward to handle.

Analysis of the samples was carried out in a mobile field laboratory. After drying the samples were crushed and the less than 40 mesh fraction taken for analysis. Several methods of sample digestion were examined, and potassium bisulphate fusion chosen as the most suitable. Although slower than some of the other methods it gives the highest extraction of lead and zinc. The analyses for lead and zinc were made according to the standard procedures of the U.S. Geological Survey (1953).

H.Y.C. PROSPECT

The H.Y.C. prospect was chosen for the initial studies as a lead/zinc sulphide orebody was known to exist at depth, mineralization was evident at the surface in an outcrop approximately 300' x 100' and previous geochemical work by Mount Isa Mines Ltd. had given surprisingly low results. The outcrop at the H.Y.C. is a supergene enrichment of hemimorphite containing only traces of lead in the dolomite/chert breccia. The only other observable mineralization is anglesite in weathered shale exposed in a nearby gully; only traces of zinc are present in the shale. Plate 2 illustrates the vertical distribution of lead and zinc along several traverses across the mineralized shale and breccia beds which have a strike length of at least 3600 feet. Auger holes were drilled at 100 feet intervals along traverses 400 feet apart and approximately at right angles to the strike of the mineralized beds. The layout of the grid is shown on Plate 1.

The values obtained for zinc are uniformly low at the surface except in the immediate vicinity of the mineralized breccia outcrop. Obviously any geochemical survey using surface or shallow depth samples would give only poorly defined anomalies of very low contrast such as the poor results obtained in the earlier geochemical survey.

The vertical and lateral distribution of zinc is in accord with the suggestion made earlier of denudation and concomitant burial by barren sediments. There is no indication of dispersal by circulating ground water after burial of the mineralised beds. Exceptions to this generalisation will be seen in auger holes K13 and K15 where zinc values greater than 1% are recorded (K13) and anomalous zinc values occur throughout the profile from 6 to 32 feet (K15).

It is suggested that these two auger holes penetrated eluvial or residual material derived from the mineralised breccia, The traverse sections and the section through G13 to M13 normal to these (Plate 2) show that K13 and K15 are directly above a small depression in the bedrock surface. If this depression had been filled by eluvial material from the adjacent rocks high values for zinc would be expected. The depression is in the downstream direction from the mineralised H.Y.C. outcrop and would be in a "shadow area" during the period of denudation thus preserving at least in part the original eluvial and residual cover.

In the H.Y.C. area residual remnants are rare so that auger drilling to within a few feet of the bedrock is necessary to detect the mineralized zone representing the underlying ore body. It is anticipated that this will be the case throughout the areas covered by river alluvium at the McArthur River.

MITCHELL YARD PROSPECT

At Mitchell Yard the laminated dolomite and green vitric tuffs of the Barney's Creek Member crop out in a south-west plunging synclinal nose on the eastern side of the river. Four exploratory holes were drilled in the alluvium close to the river. two of these proved to be over the green vitric tuff and showed no sign of lead/zinc mineralization. The other two were over shale and gave values of 380 and 5000 ppm Zn, the latter with 560 ppm Pb, in samples taken just above bedrock. Samples shallower than this showed only background values for lead and zinc. On the western side of the river the syncline is buried under deep alluvium and three traverse lines normal to the projected synclinal axis were set out (see Plate 1) and auger holes drilled along these at intervals of 200 feet. The results obtained did not indicate the presence of the mineralized shale.

Diamond drilling by Carpentaria Exploration Co., based on geological mapping and the geochemical results proved the structure to be a closed syncline with a core of pyritic shale carrying sub-economic galena and sphalerite.

TEENA PROSPECT

In contrast to the H.Y.C. and Mitchell Yard prospects where the thick alluvial cover obscures the evidence of mineralization and necessitates the use of deep auger drilling, the Teena prospect is in an area of shallow residual soil cover. Like the H.Y.C. and Mitchell Yard prospects it also occurs in the shales of the Barney's Creek Member. The outcrop is silicified and visible evidence of mineralization is rare, but chemical tests indicate the presence of lead in the shales. Samples were taken at a depth of one foot on a grid pattern 100 feet x 150 feet. The results are shown in summarised form in Plate 3.

The zinc anomaly is associated mainly with the soil and rubble overlying shales. Lead is represented by a single anomaly at the vitric tuff/shale contact. A noticeable feature of the dispersion patterns is the greater spread of the zinc anomaly which must be taken as the result of chemical dispersion by groundwater activity. A similar behaviour is found at Rum Jungle N.T., with copper and lead in soils.

Costeaning revealed 4 percent of lead over a width of 200 feet, however diamond drilling did not show any high grade mineralization.

The results obtained at Teena prospect are included here to illustrate the differences that may be encountered in the same general area depending on the nature of the country. These differences are considerable and economically important. Thus Teena shows a well defined, substantial surface anomaly over uneconomic mineralization while the weak, ill-defined surface anomaly at H.Y.C. represents an orebody of considerable size.

With the normal surface sampling methods, the geochemical results obtained over Teena and other similar prospects in the area, would command more attention than prospects such as the H.Y.C.

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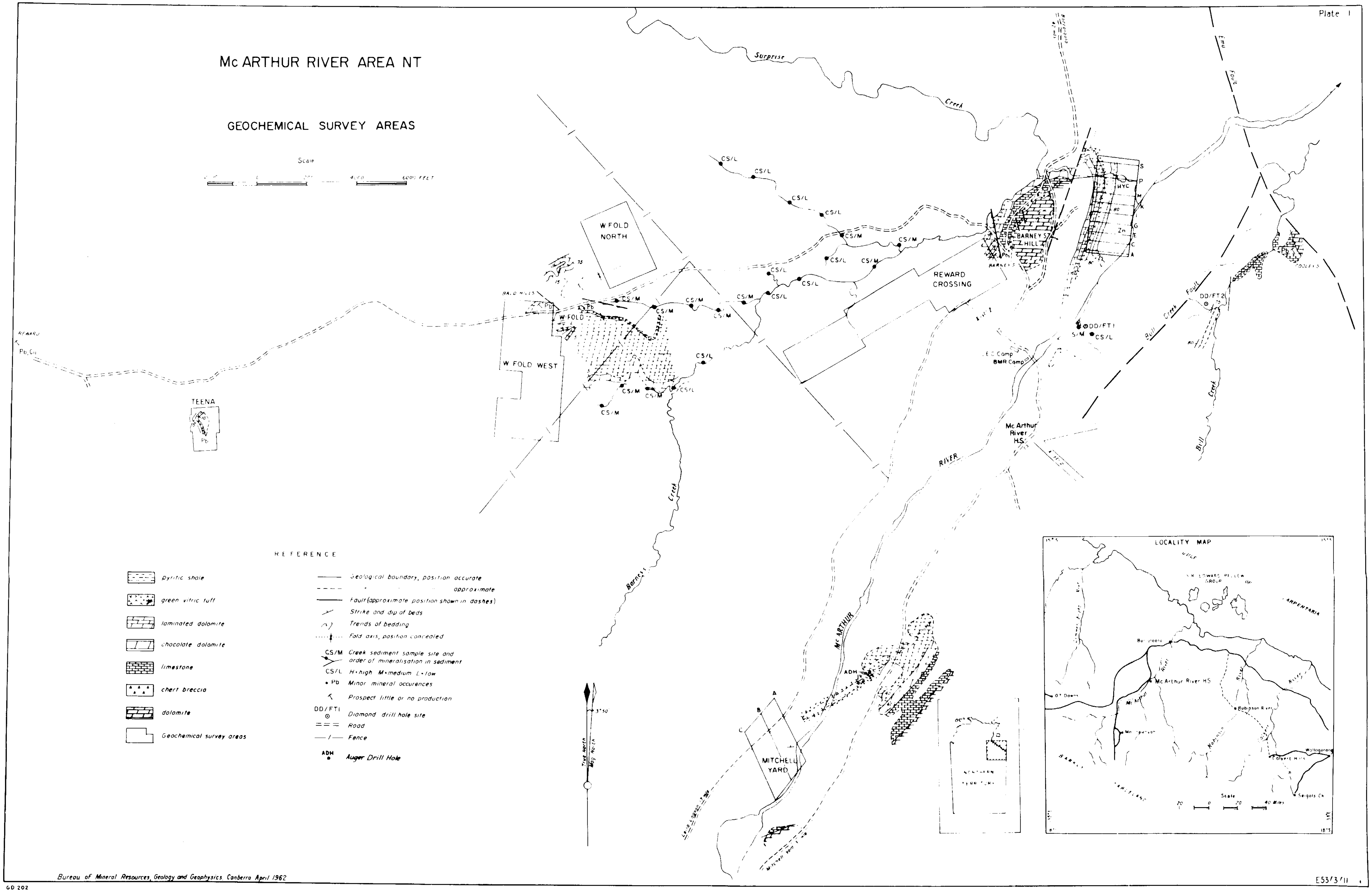

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To accompany Record No 1963/109

Plate 2

H.Y.C PROSPECT

McARTHUR RIVER

Zinc and Lead distribution with depth

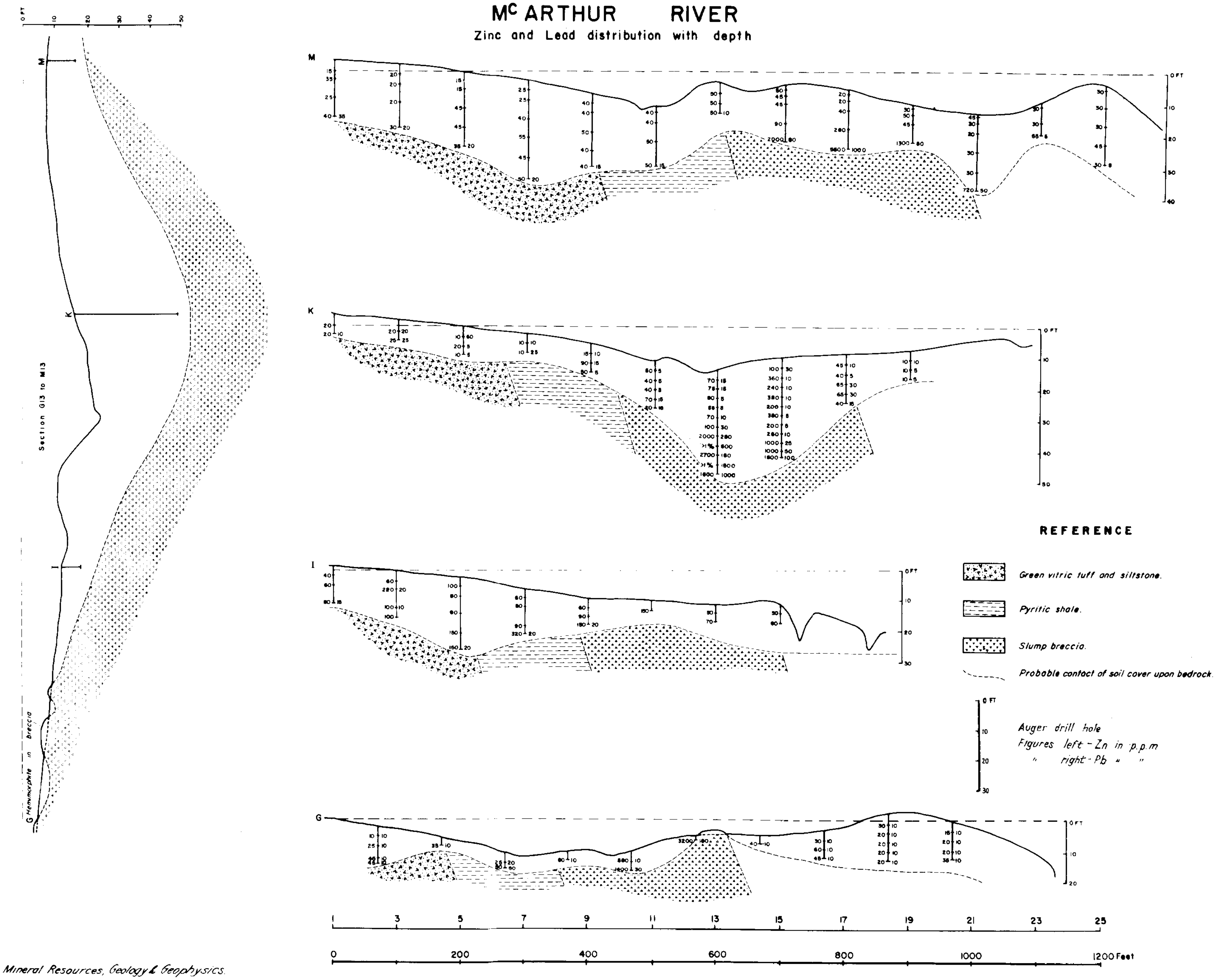

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PLATE 3.

SKETCH MAP OF TEENA PROSPECT

Mc ARTHUR RIVER AREA, N.T.

Geochemical Anomaly Centres and Geology

Geology and D.D.H. Information after
Carpentaria Exploration Co.Pty.Ltd.

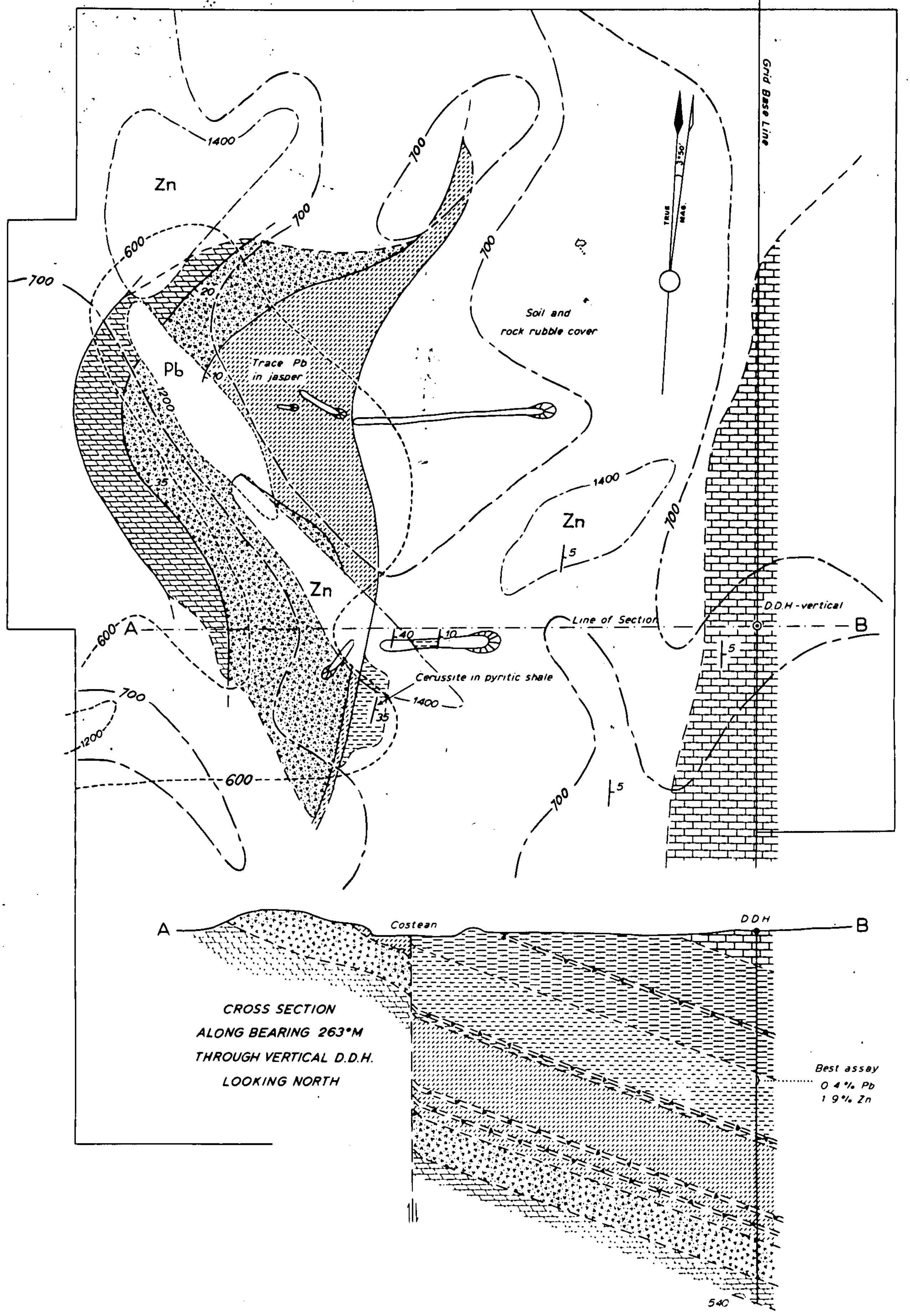
Geochemical Survey - 1961

Scale

100 0 100 200 300 Feet

REFERENCE

- ▦ *Limestone*
- ▤ *Barren shale*
- ▧ *Sedimentary breccia*
- ▤ *Pyritic shale*
- ▨ *Lower barren shale*
- ▩ *Green vitric tuffs and siltstone*
- ▦ *Laminated dolomite*
- *Costean*
- 1400 *Geochemical Anomaly Centres p.p.m. Zinc*
- 1200 *Geochemical Anomaly Centres p.p.m. Lead*
- *Geochemical Grid Boundary*



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SOME ASPECTS OF DETAILED GEOCHEMICAL TESTING AT RUM JUNGLE, NORTHERN TERRITORY, AUSTRALIA.

by

Bryan P. Ruxton

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PLATES

SOME ASPECTS OF DETAILED GEOCHEMICAL TESTING AT RUM JUNGLE, NORTHERN TERRITORY, AUSTRALIA.

INTRODUCTION

The discovery in 1949 of uranium minerals at an old copper prospect near Rum Jungle sixty miles south of Darwin, was followed by airborne radiometric surveys by the Bureau of Mineral Resources (B.M.R.) in 1950. The anomalies discovered were later examined on the ground by the B.M.R. and Territory Enterprises Pty Ltd (T.E.P.). Subsequently surface radiometric surveys, costeaning, churn drilling, surface geochemical surveys, and electromagnetic surveys were carried out over several of these anomalies. Economic uranium deposits were proved and exploited at White's, Dyson's and Mount Burton (Sullivan and Matheson, 1952; Fisher and Sullivan, 1954; Roberts, 1960). The regional geology was mapped by the B.M.R. in 1956 and this work confirmed the idea put forward in 1954 by W. Thomas (1956) that most of the uranium mineralization occurred in the "black slates" of the Golden Dyke Formation at or near the contact with the underlying Coomalie Dolomite.

The geology of the Rum Jungle area is shown in Plate 1.

Uranium was found to be associated with either copper or lead mineralization or both. Accordingly, in 1958, a surface geochemical survey was carried out by the B.M.R. over an area of about six square miles of the "black shale" dolomite contact north-west of the Giant's Reef Fault (Haldane and Debnam, 1959). Samples were taken at 2 foot depths on a 400 x 200 foot grid and were analysed by chemical methods, using biquinolyl, dithizone and rubeanic acid after fusion with potassium bisulphate, for Cu, Pb, Ni and Co. Several large copper and lead anomalies were delineated.

In 1957 T.E.P. began diamond drilling on the south-western side of Castlemaine Hill, south-west of Giant's Reef Fault, following a line of radiometric anomalies first outlined by the 1950 airborne survey (Wood and McCarthy, 1952). Between 1958 and 1960 pattern diamond drilling by T.E.P. of a weak surface radiometric anomaly at Rum Jungle Creek South disclosed a large uranium ore-body several hundred feet south-east of the surface anomaly. Although minor pyrite occurs with the ore there is no other base metal mineralization. As a result of this discovery it was decided to test other radiometric anomalies in the Rum Jungle District in detail.

In July, 1961 a programme of deep auger sampling was commenced by the B.M.R. on selected surface base metal/radiometric anomalies using Gemco auger drills. The drilling was undertaken to test whether there was any major dispersion of trace elements in the soil cover and to delineate targets for diamond drilling. The profiles cut by the augers were studied and three dimensional patterns built up for uranium, copper, lead and other elements. Auger cuttings from bedrock and Ca:Mg:Na ratios were used to interpret subsurface geology.

Details of the survey method have been described by Ruxton (1963). Briefly auger holes, 20 to 60 feet deep, were drilled on a 200' x 200' grid. Each auger hole was probed to test for radioactivity and samples of cuttings taken for chemical analysis. Initially samples were analysed in the field by chemical methods (using biquinolyl and dithizone after sulphuric acid extraction) for Cu, Pb, and Zn, but subsequently all samples, including thoses from the early drilling, were analysed spectrographically at the B.M.R. Laboratory in Canberra for Cu, Pb, Ni, Co, Mo, V, Sn, Be and P. By November, 1962, 2000 auger holes had been drilled to an aggregate depth of 60,000 feet and covering an area of four square miles (Ruxton and Shields, 1963a and 1963b). The survey is still in progress.

GEOLOGY

Geological mapping (B.M.R., 1960) shows two domes of granitic rocks surrounded by annular rings of Lower Proterozoic sedimentary rocks (Plate 1). A general synclinal structure occurs between the two domes. In the centre of this a topographic ridge (Castlemaine Hill) is capped by hematite quartz breccia which is interpreted as the basal bed of the Upper Proterozoic. A large transcurrent fault (Giant's Reef Fault) cuts obliquely across both domes and the synclinal structure, displacing the outcrops 3¼ miles north-east on the north-west side. Numerous sub-parallel shears and much dragfolding is associated with this fault. The fault affects the Upper Proterozoic rocks several miles south-west of Rum Jungle.

A basal arkose resting unconformably on the igneous complex, is followed by a thick dolomitic sequence which contains a bed of quartz pebble conglomerate and then by a pyritic carbonaceous or chloritic shale (Golden Dyke Formation). Most of the known mineralization in the Rum Jungle District occurs in the Golden Dyke Formation or at its contact with the underlying Coomalie Dolomite. Lenses of greywacke occur within the shale.

Bands of "amphibolite" and chlorite-actinolite schists most common at the dolomite/shale boundary, are partly of igneous and partly of sedimentary origin.

The rocks are weathered to depths of 60 to 80 feet, but the base of the weathering is very irregular and pyrite has been found in some places. The texture of the parent rocks is commonly preserved up to near the base of the soil which is normally 3 to 6 feet in thickness. Deep (10 to 30 feet) red soils, however, occur on the dolomitic shales which in many places are extensively silicified. Ferruginous concentrations, locally termed "laterite" are common on the pyritic shales and in the rubbly soils derived from the quartz breccia of Castlemaine Hill which has a hematitic silty matrix.

The landforms around Rum Jungle are of the typical "hill and dale" type of humid-temperate regions and there is no development of the pediment landform common in other savannah areas of the tropics (Cotton, 1961). Fifty inches of rain a year falls at Rum Jungle in a humid wet season lasting from November to April.

GEOCHEMICAL RESULTS

Base Metals

Plate 2 shows the relationship between the lead and copper contours on the surface (at 2 foot depth) and in weathered bedrock at Area 55 which may be taken as typical of the Rum Jungle Area. The auger holes in most cases were drilled to a maximum depth of 40 feet and the metal values in weathered rock are those measured for bulked samples taken from below the base of the soil to the base of the auger hole.

Lead contours in soil above 960 p.p.m. are smaller in area than the equivalent contours in weathered bedrock showing slight dilution of the lead. The 140 p.p.m. lead contour in soil, however, covers a larger area than the 240 p.p.m. contour in weathered bedrock and shows some displacement of the lead in the soil downslope towards and into a north-westerly trending depression. In both soil and weathered bedrock the high lead contours are aligned west, parallel and adjacent to a major fault (b, Plate 2).

The copper contours in the soil are consistently lower than the contour beneath in weathered bedrock showing greater dilution of copper than of lead. Some less intense copper anomalies in weathered rock south of the major fault are not well represented in the soil and this may be partly due to wider spacing of the 1958 surface sampling points (400 x 200 foot grid as against the 200 x 200 foot grid in the weathered bedrock).

Scout diamond drilling of part of the intense copper and lead anomalies has shown low grade copper and lead mineralization in the rock beneath.

Radioactivity

Background radioactivity in the Rum Jungle district is generally taken as 0.015 milli-roentgens per hour (mR/Hr). In the examples discussed below the radioactivity is due to uranium and so readings given in mR/Hr are a rough approximation of the uranium content in parts per million.

In contrast to copper and lead, several examples of extreme dilution of uranium from weathered rock to soil are known in the area. Thus on the south-western side of Castlemaine Hill three small surface radiometric anomalies of 0.030, 0.030 and 0.025 mR/Hr are underlain in weathered bedrock by values of 0.40, 0.15 and 0.20 mR/Hr respectively. In each case values rose very rapidly with depth in the soil. An example of the less extreme dilution is shown in Plate 3, b at the southern end of Dolerite Ridge. Here the centre of the anomaly in weathered rock is represented by a small area outlined by the 0.025 mR/Hr on the surface some 200 feet downslope.

Downslope displacement of the surface anomalies from the corresponding anomalies in weathered bedrock are particularly common on the flanking slopes of Castlemaine Hill.

During the initial stages of open cut operations at Rum Jungle Creek South on the western side of Castlemaine Hill (Plate 1) stripping uncovered a small pod of high grade uranium ore covered by eight feet of soil. A detailed radiometric survey of the area had failed to show any surface anomaly over this pod; the only surface anomaly in the area was about 400 feet downslope from the ore. A line of auger holes were drilled at 75 ft. intervals between the surface anomaly and the ore. Beginning at the anomaly and proceeding towards the ore the radioactivity was at first confined solely to the lower part of the soil layer. Approaching the ore the radioactivity increased and was found to extend progressively further into the weathered rock until at the last auger hole, 65 feet from the ore, anomalous radioactivity was recorded over the entire depth of 40 feet. A section cut through the mineralization by the edge of the open cut, exposed secondary uranium minerals which could be traced visually for a distance of eight feet along the boundary between the soil and in situ altered rock.

The contact between the altered rock and the overlaying soil is sharp and slickensided as a result of solefluction. Uranium has obviously migrated along this contact, which has a slope of about 5^o, for a distance of at least 400 feet downslope without any vertical dispersion into the soil. The soil cover varies from 8 feet over the uranium ore to 6 feet at the edge of the surface anomaly and completely blankets all radioactivity. The surface anomaly itself occurs on the lower level of a small bench out in the soil by a nearby creek. Here the soil cover is reduced to 3 feet bringing the uranium at the base of the soil profile within the range of detecting instruments at the surface.

Examples of the concentration of uranium in ferruginous soils are very common in the Rum Jungle Area. In two places around Castlemaine Hill highly ferruginised weathered carbonate rocks have surface values of 0.30 and 0.10 mR/Hr and are underlain by normal weathered carbonate rocks with background radioactivity. A less extreme example is shown in Plate 3, a from the West Finniss area. Here the 0.025 mR/Hr surface radiometric contour covers a much wider area than the equivalent 0.024 mR/Hr in weathered bedrock. There is no dilution and considerable surface displacement has occurred. The soil here contains numerous pisolitic ironstone concretions, particularly above the pyritic carbonaceous shale, and the uranium is slightly concentrated in these.

At Dolerite Ridge (Plate 3, b) the surface and weathered bedrock radiometric contours of 0.025 mR/Hr are similar in shape and size but the surface anomaly is displaced upslope. The surface anomaly here coincides closely with an outcrop of ferruginised gravel which has presumably fixed the uranium, whereas on the downslope edge of the subsurface anomaly area uranium has been leached from the normal clayey soil.

Several surface radiometric anomalies found by Bureau officers on the north-eastern side of Castlemaine Hill in 1961 were found to be due to uranium-bearing phosphate rock in the carbonate sequence. Outcrops of the phosphate rock give values up to 0.24 mR/Hr but considerable dilution occurs in the soil where values rarely exceed 0.025 mR/Hr. Base metal values are extremely low in these areas. The radioactive background of the enclosing rocks is also low, about 0.01 mR/Hr.

DISCUSSION

Results show that despite the wet savannah climate in this tropical lowland area only slight to moderate dispersion and dilution of the base metals has occurred from fresh bedrock through weathered bedrock to overlying sedentary soil. The surface anomalies are rarely displaced more than 100 to 200 feet from their parent anomalies in weathered bedrock. It is difficult to calculate meaningful threshold values for base metals in the Rum Jungle Area. Soil values above 960 p.p.m. lead and 800 p.p.m. copper seem to indicate mineralized bedrock below. Thus on the north-eastern portion of Area 55, 400-800 p.p.m. copper in the soil overlies 1600 - 3200 p.p.m. in weathered bedrock which in turn is underlain by fresh bedrock with about ½ - 1% copper.

These results contrast markedly with the geochemical behaviour of base metals in the mature tropical weathering profiles of Northern Rhodesia (Tooms and Webb, 1961) where surface anomalies of 100 to 400 p.p.m. copper overlie bedrock with 2 - 5% copper, and the threshold value for copper is only 75 p.p.m. Similarly at Tennant Creek (McMillan and Debnam, 1961) where deep, severely leached weathering profiles occur, peaks of 50 - 100 p.p.m. copper in soil are known to overlie ore grade mineralization in the fresh bedrock beneath.

Along the western flank of Castlemaine Hill where solefluxion is an important feature there is little surface expression of the underlying uranium mineralization. The soil near to the land surface appears to be leached free of uranium which is distributed lower in the profile, frequently concentrating at the soil/weathered rock boundary. The technique of auger drilling coupled with radiometric logging of the auger holes would readily locate uranium mineralization that had been obscured in this way.

The strong surface anomalies are explained by their association with concentrations of iron oxides and phosphate rock, both well known for their ability to collect and retain uranium from sources such as circulating uraniferous ground water.

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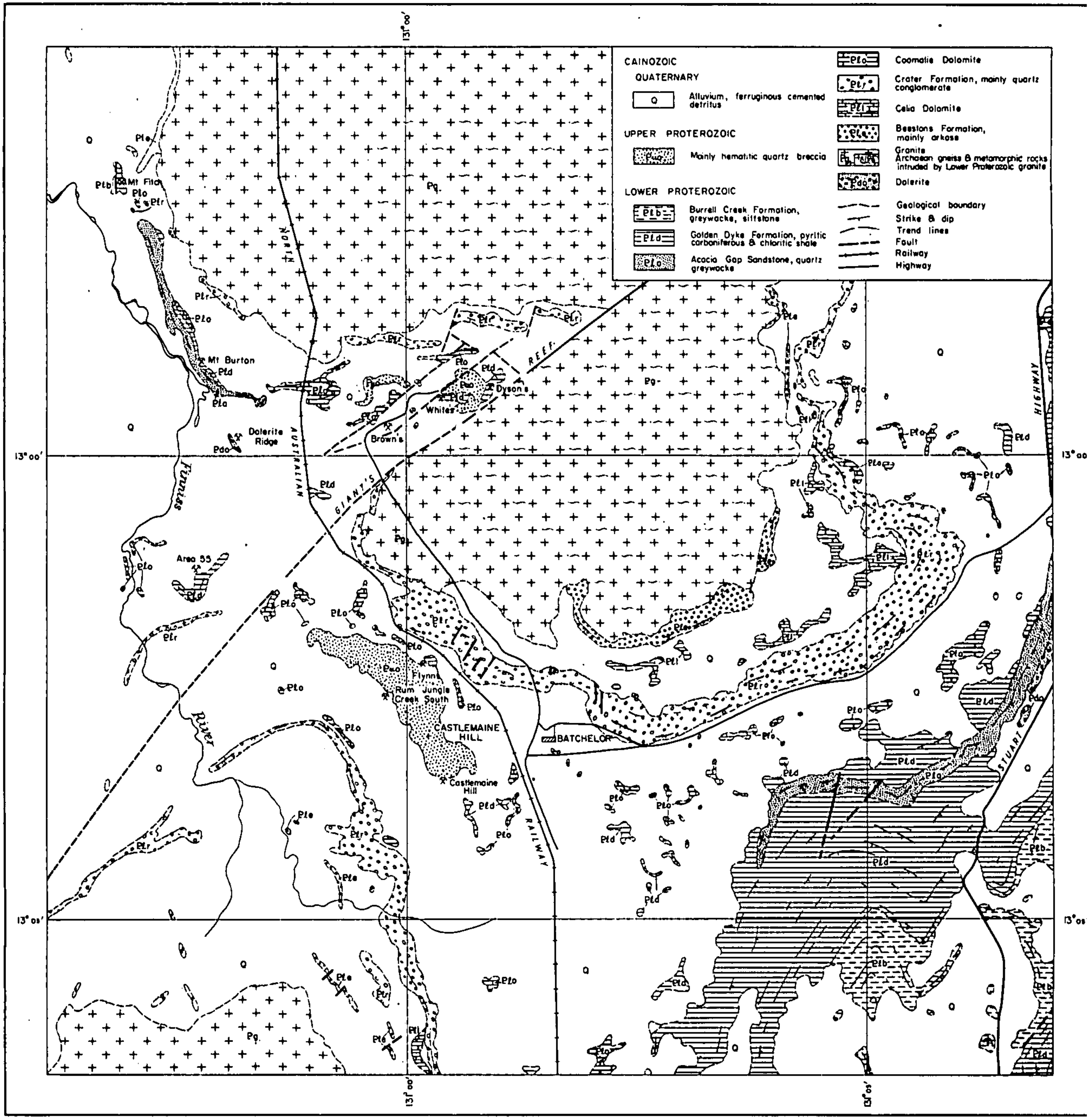
PLATE



RUM JUNGLE AREA, N. T.

GEOLOGY

AFTER BMR 1-MILE SPECIAL SHEET, 1960

SCALE IN MILES

1 0 1 2 3 4

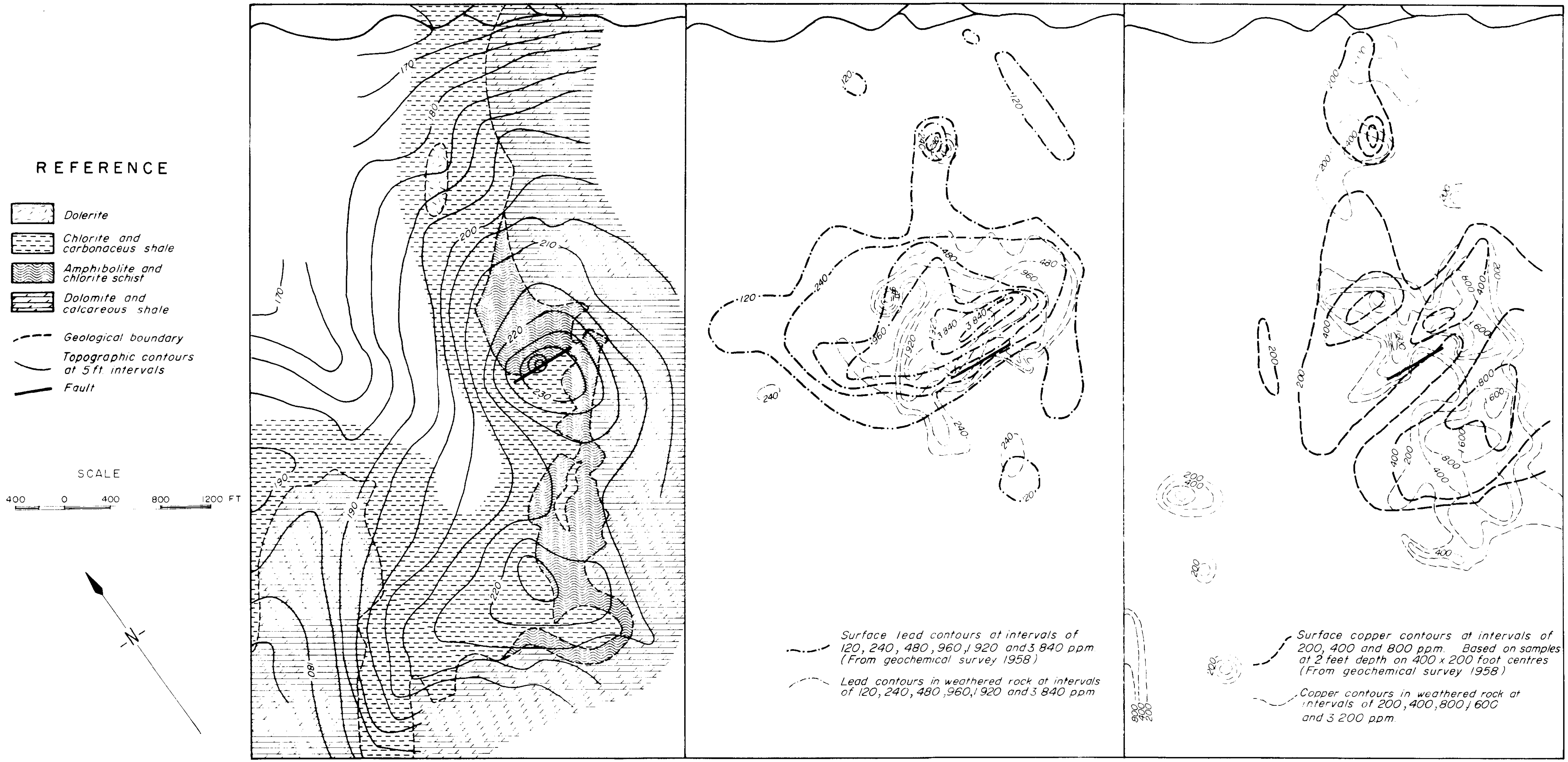
BUREAU OF MINERAL RESOURCES, GEOLOGY AND GEOPHYSICS

To accompany Record No 1963/109

D 52 /8/62

JULY 1963

AREA 55 RUM JUNGLE



a- Geology and Topography

b- Lead Distribution

Surface lead contours at intervals of 120, 240, 480, 960, 1 920 and 3 840 ppm (From geochemical survey 1958)

Lead contours in weathered rock at intervals of 120, 240, 480, 960, 1 920 and 3 840 ppm

c- Copper Distribution

Surface copper contours at intervals of 200, 400 and 800 ppm. Based on samples at 2 feet depth on 400 x 200 foot centres (From geochemical survey 1958)

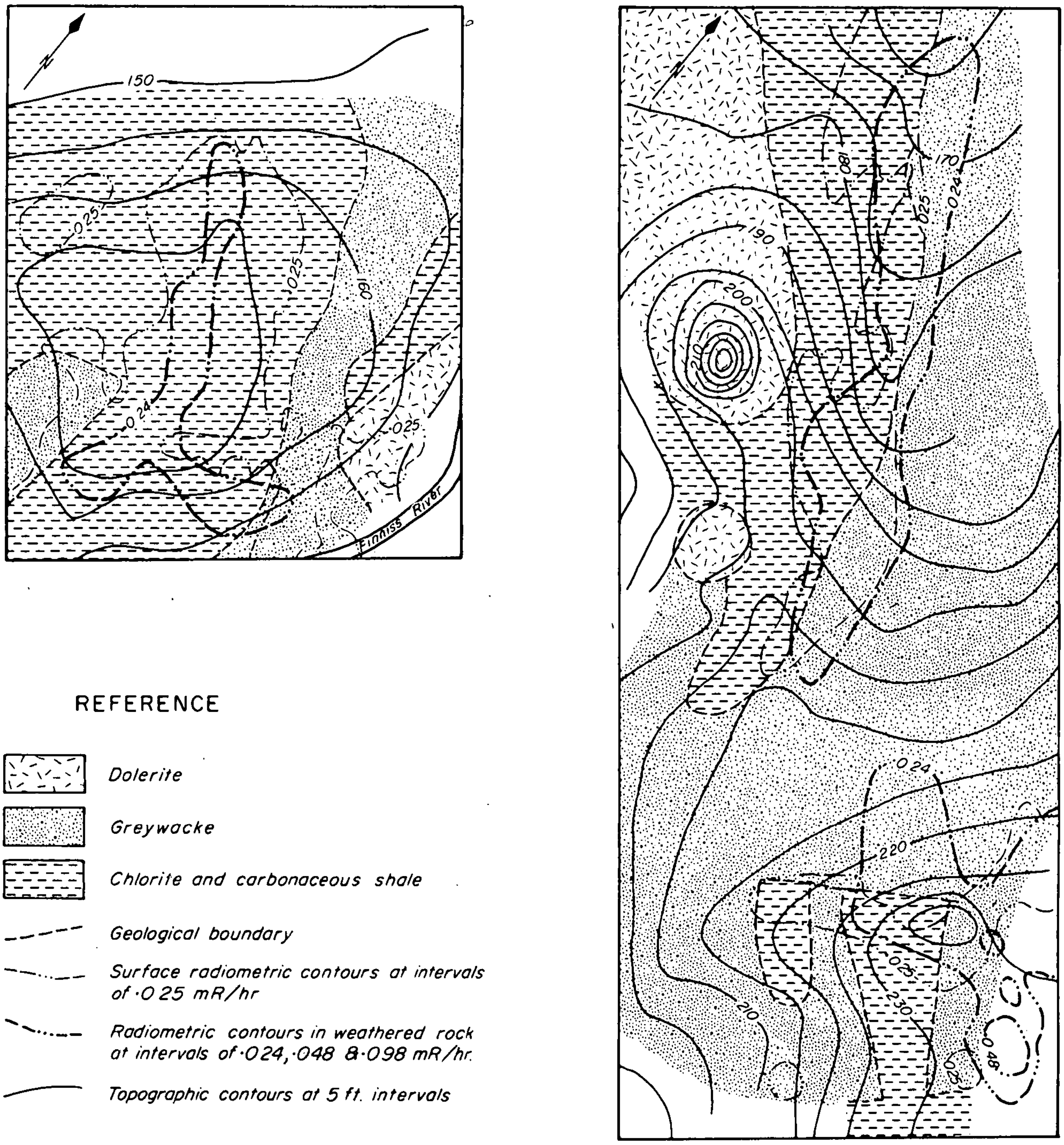
Copper contours in weathered rock at intervals of 200, 400, 800, 1 600 and 3 200 ppm

Bureau of Mineral Resources, Geology and Geophysics June 1963 To accompany Record No 1963/109

D52/A8/77 JK

PLATE 3

GEOLOGY, TOPOGRAPHY AND RADIOACTIVITY
at



a- WEST FINNISS

b- DOLERITE RIDGE

REFERENCE

- ▨ Dolerite
- ░ Greywacke
- ▤ Chlorite and carbonaceous shale
- _._--- Geological boundary
- —·..__ Surface radiometric contours at intervals of ·0 25 mR/hr
- \~·..__ Radiometric contours in weathered rock at intervals of ·0 24, ·048 & ·098 mR/hr.
- —— Topographic contours at 5 ft. intervals

800 0 800 1600 2400 FEET

Bureau of Mineral Resources, Geology and Geophysics. June 1963 To accompany Records No 1963/109 **D 52/A8/76**

A COMPARISON OF CHEMICAL AND SPECTROCHEMICAL METHODS USED BY THE COMMONWEALTH BUREAU OF MINERAL RESOURCES IN GEOCHEMICAL PROSPECTING IN AUSTRALIA.

by

E.J. Howard and S. Baker

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A COMPARISON OF CHEMICAL AND SPECTROCHEMICAL METHODS USED BY THE COMMONWEALTH BUREAU OF MINERAL RESOURCES IN GEOCHEMICAL PROSPECTING IN AUSTRALIA.

INTRODUCTION

An essential part of geochemical prospecting techniques is rapid analysis of a large number of soil and rock samples for trace elements with sufficient sensitivity and precision to clearly differentiate the anomalous samples from those considered to represent normal or "background" samples. Chemical methods using selective, sensitive reagents, many of them organic, are widely used in the analytical work; but spectrochemical procedures are becoming more common as an alternative to chemical methods. Spectrochemical analysis has contributed substantially to the development of geochemistry: but it is only in recent years that the spectrograph has been applied to rapid routine analysis in the geochemical prospecting field.

The Bureau used mainly colorimetric methods supplemented in special cases by polarography until 1961 when a general spectrochemical procedure was introduced. Since then all the analytical work for geochemical surveys has been carried out by this method.

THE GENERAL SPECTROCHEMICAL PROCEDURE

The method is designed to cover a selection of elements which are either commonly associated with economic mineralization or themselves form the ore minerals. The general procedure is classified as semiquantitative and includes the following elements in the wavelength range 3600A° - 2500A°.

Element	Detection Limit in p.p.m.	Analytical Range in p.p.m.
Nickel	1	5 - 2000
Cobalt	1	5 - 2000
Zinc	50	100 -10000
Copper	1	5 - 5000
Tin	10	10 - 5000
Vanadium	1	5 - 2000
Beryllium	5	5 - 1000
Molybdenum	1	1 - 1000
Lead	10	10 - 5000
Phosphorus	0.5%	1% - 30%

The apparatus used is a Hilger Large Quartz Littrow Spectrograph and Arc Stand. The power unit for the D.C. arc is an S.T.C. constant current rectifier, continuously variable from 3 to 15 amp at 240 volt. A Judd-Lewis Comparator is used for comparison of standard and analysis plates.

The optical set-up is arranged to give spectral lines of uniform density. This allows a two step filter of 100% and 10% transmission to be used to divide the spectrum and increase the range of concentration that can be recorded photographically in a single exposure. The prepared samples are loaded into preformed electrodes, preheated to 400°C and arced to completion at 10A, anode excitation. Comparison of the density of the analysis line is made against standard plates prepared under the same conditions using synthetic standards in a suitable base.

Preliminary preparation of the samples is kept as simple as possible. Experience has shown that, for soils and soft rock, hand grinding is the most efficient form of sample reduction in terms of output and lack of contamination. For hard rock machine grinding must be used and considerable attention paid to cleaning the equipment between samples.

A typical figure for the amount of work that can be carried out by a staff of one chemist and an assistant is 420 elemental analyses per day. This figure is based on a period of 90 working days and a coverage of 10 elements per sample.

Providing that the working conditions are standardised the main sources of error lie in large variations in the composition of the sample such as that between clay and limestone and the behaviour of the sample in the arc.

The results are given in terms of classes derived as follows: with a concentration range of 1 - 1000 p.p.m. covered by 9 standards it is possible to assign a value to a sample of equal to a standard, or between two standards. This gives 20 classes with the following class values in p.p.m. <1, 1, 2, 3, 5, 7, 10, 15, 20, 30, 50, 70, 100. 150, 200, 300, 500, 700, 1000, >1000. Other concentration ranges are simple multiples of this. This arrangement is similar to the colorimetric visual comparison technique used in the chemical methods.

CHEMICAL METHODS

During the early development of geochemical prospecting within the Bureau of Mineral Resources the analytical methods mainly followed those developed in England and the United States of America. Characteristic features of these chemical methods are speed, simplicity and sensitivity, a combination that is, however, attainable for only a small number of elements. One of the main restrictions is the number of sensitive, selective reagents available to form the basis of suitable field methods. Outstanding in this respect is dithizone which has been used extensively by the Bureau and others in geochemical surveys for copper, lead and zinc. Its main disadvantage is the instability of its solutions in carbon tetrachloride, chloroform and other organic solvents. This is a handicap in the tropical areas of Australia where most of the Bureau's geochemical prospecting has been carried out.

As the basis of a field method for the estimation of traces of zinc, dithizone has given satisfactory performance. The same cannot be said for the dithizone method for lead which requires careful attention to the working details for satisfactory results.

In recent years 2.2' diquinolyl (cupreine) has replaced dithizone as a reagent for copper. It is a stable, specific reagent for copper, and has been found to be of outstanding value both in the field and the laboratory.

Rubeanic acid has been used as a colorimetric reagent for nickel and cobalt after separation by paper chromatography. Because of the large dilution factor involved, the chromatographic procedure seriously reduces the sensitivity of the method.

Various methods of sample digestion have been employed for the extraction of the trace metals, and include heating with dilute acid, ammonium citrate extraction, fusion with potassium bisulphate, and concentrated sulphuric acid digestion.

COMPARISON OF THE METHODS

In comparing chemical against spectrochemical methods both economic and purely analytical factors are involved. In the spectrochemical procedure the ground sample is taken directly for analysis eliminating the sample digestion stage. Apart from the saving in time, handling of the sample is reduced and with it the risk of contamination and misidentification.

One of the unknown variables of chemical methods is the incomplete extraction of the element sought by a particular method of digestion. The various secondary minerals of the same element are not equally attacked by a given digestant e.g. copper as malachite versus chrysocolla or lead as cerussite versus anglesite to quote simple examples. Experience at Tennant Creek in Central Australia, where surface copper anomalies are small in magnitude, has shown that with some digestants apparent anomalies can be the result of extraction of a variable percentage (5 -90%) of the total copper present. For this reason potassium bisulphate fusion, which gives the highest percentage extraction, was used almost exclusively in the Bureau's geochemical prospecting work. Spectrochemical methods eliminate this factor and the values obtained represent the total content irrespective of the form in which the element concerned may occur.

A further drawback to chemical methods where analysis for more than one element is required, is the lack of a universal digestant applicable to rapid field methods. Potassium bisulphate fusion is not satisfactory for the extraction for example of tin or beryllium. In a case requiring the estimation of copper, lead, molybdenum, tin and beryllium as in the stream sediment geochemical survey in the Herbert River area in northern Queensland, several separate digestion methods would have to be used. A simple spectrochemical procedure eliminated this problem entirely.

Chemical methods generally require the preparation of concentrated buffer solutions free from trace metal impurities. In some cases, for example lead, this can be quite troublesome. Again certain reagents such as potassium bisulphate cannot be purified by the normal extraction methods. All this is avoided when using spectrochemical methods.

Identification of an element spectrographically is positive and unique if operator error and inexperience are discounted. This is rarely the case with chemical methods where a considerable amount of the development work is concerned with determining the interfering elements and setting limits to these.

In terms of concentration the detection limits generally achieved in field chemical analysis and spectrochemical analysis are much the same. However chemical methods can be made far more sensitive by virtue of the relatively greater weight of sample that can be taken for analysis. Any general spectrochemical method is at a disadvantage in this respect as increasing the weight of the sample arced does not necessarily increase line to background ratio, a critical factor in detecting faint spectral lines. There is an added disadvantage in that certain elements such as zinc, tungsten, arsenic, etc. have low arc sensitivities in the wave length range normally available.

As with chemical methods, the presence of large amounts of iron in the sample causes some trouble in the general spectrochemical procedure. Above about 30% iron oxide the sample is consistently ejected from the electrode cup at some stage of the arcing. This can be avoided but only at the cost of either reduced sensitivity for all elements or restriction of the number of elements that can be determined by a single arcing.

The biggest advantage with any spectrochemical method is the number of elements that can be determined simultaneously. In the general procedure at present used by the Bureau, ten elements are determined on a semiquantitative basis from each spectrum. These spectra also include sensitive lines of a number of other elements of interest in geochemical prospecting for example silver, gold, bismuth. It is a simple matter to scan each spectrum for the lines of these elements and in most cases give a rough indication of the amount likely to be present. Thus the analytical scope of the spectrographic technique is very wide. There are 50 elements, including the seven common silicate elements Si, Fe, Al, Mg, Ca, Na, and K, that can be detected spectrographically using ordinary D.C. arc technique. Those not included are all gaseous elements, the halogens, rare earths, radioactive elements and the non-metals carbon, sulphur etc. For geochemical prospecting the omission of these elements is not serious and for some of no consequence at all.

The initial cost of equipment for chemical analysis is small as only common laboratory apparatus and chemicals are required. Spectrographic equipment on the other hand represents an investment of at least several thousands of pounds.

Experience within the Bureau has demonstrated that productivity in terms of the number of analytical results produced is considerably higher for the spectrochemical method. With a staff unit of one chemist and one assistant carrying out chemical analyses for copper and zinc an output of 100 analyses per day (7½ hours) has been maintained. For the spectrochemical method covering the ten elements listed earlier, the output is 480 analyses per day with the same staff unit. These figures take into account all operations from sorting the samples to reporting the results.

It is not possible to make an estimate of the running costs other than a purely local one as labour in relation to equipment and reagent costs in one country do not necessarily apply in a different country. As employed by the Bureau of Mineral Resources the cost per element determination for the spectrochemical procedure is less than that by chemical methods. It is of course understood that the spectrograph is not used for the determination of only one or two elements simultaneously. In such a case chemical methods would cost less unless these elements present special problems.

One disadvantage inherent in the spectrochemical procedure is that samples must be sent to the spectrograph. Mobile spectrographic units have been used for field geochemical analysis in some countries, but a large Quartz Spectrograph is a difficult and sensitive instrument to move and the more complex direct-reading spectrographs cannot be moved. No serious inconvenience has been experienced in the Bureau's geochemical work by having to forward samples to a central laboratory; and in some respects it is beneficial to have the analytical work done under controlled conditions.

The introduction of spectrochemical analysis a tool for geochemical prospecting has proved of considerable benefit to the Bureau of Mineral Resources in coping with an increased programme of geochemical prospecting with no expansion in staff. It played a part in the discovery of high grade phosphate at Rum Jungle in Northern Australia and data on the major elements Mg, Ca and Na obtained as a bonus from the spectrograph plates have been used in the lithological interpretation of areas being surveyed. There is little likelihood that the Bureau will return to standard wet methods except, perhaps, in detailed surveys where only one or two elements need to be analysed.

PROPOSED MULTI ELEMENT ANALYTICAL EQUIPMENT AND SAMPLE CONTROL PROCEDURES FOR GEOCHEMICAL SAMPLES FOR THE BUREAU OF MINERAL RESOURCES, AUSTRALIA.

by

K.R. Walker

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PROPOSED MULTI ELEMENT ANALYTICAL EQUIPMENT AND SAMPLE CONTROL PROCEDURES FOR GEOCHEMICAL SAMPLES FOR THE BUREAU OF MINERAL RESOURCES, AUSTRALIA.

INTRODUCTION

Emission spectrography has been used in the analysis of geological materials for many years, but it is only in recent years that the development of the direct reading spectrograph has made the rapid accurate analysis of silicates possible. Similar developments have also taken place in the instrumentation of X-ray emission spectrography. The demand in industry for increased analytical speed and decreased possibility for human error (especially as a result of operator fatique in routine work) to meet the stringent conditions of production control, have been important factors stimulating the development of reliable direct reading spectrographs. Aware of these developments, geological organizations concerned about the limitations of present analytical procedures they employ, which are, comparatively, slow and determine only a few elements at a time, have been attempting very recently to adapt the direct reading instruments to the analysis of geological materials. The Bureau of Mineral Resources is installing a modified version of the standard direct reading optical spectrograph used in the metallurgical industry, and hope in the near future to obtain an automatic X-ray emission unit. Along with similar organizations in other countries, it hopes to contribute to further the development of these instruments for the analysis of geological materials.

The conversion to automatic analytical procedures is still only in the formative stages in the Bureau. Our direct reading optical spectrograph is at present undergoing trial runs in London; an automatic X-ray emission spectrograph has yet to be ordered, as do facilities for sample control, automatic plotting and data indexing and storage. The Bureau geochemical laboratory at present is equipped with a large Hilger quartz spectrograph and a Philips all-vacuum X-ray emission spectrograph. Routine geochemical analyses are currently done by the quartz spectrograph and the plates are scanned for 12 elements. Wet methods are still employed in some surveys but the trend has been increasingly towards multi element instrumental methods.

The main reason for this trend is an increasing awareness from experience, that important economic mineralization can be overlooked in surveys based only on wet analytical techniques. Particularly in orientation surveys, it is important to check for all elements, before deciding which are "indicator" elements and the best method for analysing them. To meet this requirement instrumentation which is capable of both rapid and precise multi element analyses is necessary. The automatic instruments will also have an application in fundamental geochemical research.

We plan to increase the scope of our mineral search programme by systematizing and streamlining sample collecting, analytical procedures, computation and plotting of data. This planning requires specialists, capable of developing equipment and of rationalizing analytical procedures and problems that continually arise in the course of routine work when sophisticated instrumentation is employed. Such instruments only give the right answer when all operational and experimental parameters are controlled. Providing they are in good working order therefore, successful operation of the instruments is determined by the specialist's knowledge of emission spectrography, and by his knowledge of geological and geochemical phenomena. For example, with the photographic optical spectrograph, the spectrographer is continually confronted with analytical problems which show up during analysis and computation. With the direct reading optical instrument, which eliminates the photographic procedure, similar problems can easily go unnoticed when analysing materials with complex spectra, such as silicates. In particular, background, line interference, self adsorption could occur unrecognized, and radically distort the results when analysing samples of unknown chemical composition.

As we do not as yet have our automatic instruments installed, the following comments are necessarily of a general nature only.

OPTICAL EMISSION DIRECT READING SPECTROGRAPH

With a direct reader accurate analysis for up to 33 elements in a rock or soil can be made in about 20 minutes. To achieve the same accuracy and element coverage by a photographic instrument would take about 2 days. The direct reading spectrograph is, therefore, a good instrument for the rapid analysis of samples in the accuracy range ± 5 to ± 20% of the element present for a wide element coverage. Provided a sample is analysed for 10 or more elements, the cost per element is probably less for the accuracy attained than by any other method, though it may be rivalled on this point by the automatic X-ray emission spectrograph. The direct reader finds its main applications for the analysis of samples where high element coverage and high sensitivity for each sample site are required, such as in regional geochemical surveys and orientation runs in detailed grid geochemical surveys. A direct reader would be wasted on problems requiring small element coverage and low accuracy, for much of the instrumental capacity would be idle and the recording equipment would be orders of accuracy greater than the analytical method employed.

By comparison the photographic spectrograph is more economical and rapid to operate where analyses are required for up to 10 elements in a sample in the accuracy range of ± 50% or less of the element present. It finds its main application in analysing samples for detailed grid geochemical surveys, other than those for orientation runs.

The cost of equipping an optical direct reader laboratory is about £A.40,000, and the annual running costs are estimated at about £A.17,000 for staff, consumables and amortization. After the equipment is installed we anticipate that a fairly lengthy period will be necessary for calibrating and establishing the equipment on routine analysis of geochemical samples. This to some extent is due to the fact that calibrating a direct reader and developing suitable analytical methods and working curves for the analysis of geological materials is so far being attempted by very few other scientific laboratories in the western world, none of which have yet advanced to a stage where we can have the benefit of their experience. No manufacturer makes a direct reader as a standard instrument for geochemical work. We hope the direct reader will have an annual capacity for up to 10,000 sample analyses, or between 200,000 and 330,000 elemental analyses, depending on how many elements are required, after a 12 month establishment period. The staff employed will be 2 geochemist-spectrographers, 1 technical officer and 2 laboratory assistants.

The direct reader will be programmed for: Bi, Ag, Li, Ga, Na, Pb, Zn, Tl, P, Ge, K, Rb, Cs, Cu, Sn, Fe, Al, Be, La, Sr, Mg, Cr, Co, Ni, V, Mn, Mo, Ca, Ba, Ti, B, Sc and Zr. Initially we propose to use a DC arc method, anode excitation of rock powder mixes with In as the internal standard for analysis of volatile elements, and Pd the internal standard for involatile elements.

SAMPLE PREPARATION

The sample preparation will require rapid reduction of a rock from about 4 in. diameter size, without contamination or differential loss of sample, to a homogeneous sample that will pass a 200 mesh seive. This can be achieved by having a well laid out sample preparation laboratory, with widely spaced crushing and grinding equipment. Enclosed demountable grinding vessels are essential. The equipment can be set in wall benches and arranged in order of reducing particle size, followed by facilities for cleaning grinding vessels; and by a muffle and drying oven. The entire sample preparation room should be air evacuated through holes in walls and ceiling, and this, with separate wall mounted vacuum hoses for cleaning each piece of equipment, means the room always remains dustless. We propose using ultrasonic tanks for cleaning grinding vessels.

The line up of sample preparation equipment will be something like the following -

Demountable jaw crusher - demountable roller mill (required only if the crushed product from the crusher exceed ¼" diameter) - swing mill (Siebtechnik type) - blender mill (Pica type, for homogenizing powdered rock sample or soil sample) - Spex 5000 or Glen Creston M270 mill (for mixing and homogenizing samples, graphite or flux and internal standard).

AUTOMATIC X-RAY EMISSION SPECTROGRAPH

The automatic X-ray emission spectrograph will complement the direct reading spectrograph. The acquisition of an automatic X-ray unit is still being negotiated, but we are at present developing analytical procedures for it which do not require internal standards, on the Philips vacuum unit. The method being tested is a fusion technique using a low absorbing matrix to overcome interference effects of sample composition.

Initially it is proposed to use the automatic unit for normal silicate rock analyses. Assuming a counting time of 2 minutes per element and including sample preparation time, it should be possible to determine all major constituents, except ferrous iron, water and alkalis, in about 40 minutes. The alkalis will be done by flame photometer, and FeO probably by spectrophotometer. The automatic X-ray unit will also be used for trace element analyses in geochemical samples, such as the study of heavy mineral concentrates from stream sediment samples; and for analyses of elements such as Re, Ta, Te, Nb, As, Cd, Sb, Sn, Hf, Se, W, P, Th, U etc. which can be done more satisfactorily by X-ray than by optical emission spectroscopy.

STANDARDS

The problem of standards is one faced by spectrographical laboratories everywhere. A number of rock and ore standards are in circulation, but these do not give a complete coverage of concentrations for each element in various matrices. Standards are a basic requirement for precise spectrographic work, and we plan to develop more of them for use in our analytical work, and, if arrangements can be made, we will develop them in co-operation with other interested laboratories.

SAMPLE CONTROL

Sample control is an important aspect of an efficient laboratory. We anticipate that the annual output of sample analyses in the Bureau laboratory from all sources will be about 25,000 sample analyses, and up to 400,000 elemental analyses, plus 2000 petrological examinations. Some form of mechanization will be necessary, therefore, to record, store, extract and process data relevant to a particular problem, and initially we plan to use punched cards for this.

The chemical and petrological classification systems will be integrated and all sample localities will be coded using a reference system based on the international grid. The more comprehensive and detailed the classification system, the more specific will be the information it is able to provide. The coding is therefore being designed to strike the balance between what information may be necessary to extract, and what the sampler may be able and willing to provide. In this context it should be noted that we handle material from organizations other than our own.

A simple indexing form has been devised to accompany and control the sample from point of collection, recording relevant field information, work requested and undertaken in the laboratory. The form is designed to simplify transfer of information to cards or tape for data processing or automatic plotting and ultimately storage. This procedure we hope, will encourage purposeful collection of samples, minimize sample handling, processing and interpretation of data, and avoid repetition of work.

Referring again to the direct reading optical spectrograph, this instrument has a digital read-out by I.B.M. typewriter, which tabulates element values from the sample analysis. The output in digits is uncorrected for ambient working conditions at the time of analysis and for analytical conditions such as matrix or inter element effects etc. Thus the read-out values have to be corrected for the analytical working conditions and to do this a working curve is constructed using standards, by which means the digits are converted to percent concentration of the element present in the sample. This manual conversion is unavoidable in the initial stages of calibrating the instrument and setting it up for routine analysis of geological materials. We plan to use punched cards while we are calibrating and setting up the optical direct reader for routine work, and probably for the first couple of years of operation. During this period our system will involve a card puncher - document writer unit, sorter and storage facilities. Plotting will be a manual operation following card sorting into element groups of various multiples of background. When experimental conditions and operating parameters of the instrument are understood for various combinations of elements in various matrix types, it should be possible to add a computer stage to the read-out, so that elemental values in percent concentration for each sample are recorded directly on punched cards or magnetic tape, in a form ready for data processing and automatic plotting.

Data sorting and processing facilities can be employed to determine background values for a geochemical survey. Samples with anomalous values can be sorted into multiples of background for each element (i.e. into concentration classes). Element ratios can be determined. Instructions for a plotting schedule can be worked out and applied to an automatic plotter which will plot detail of grid geochemical surveys automatically onto maps. Lines through points representing equal values for an element would be drawn manually to complete the map. This requires a grid reference of the metric type and co-ordinates for each sample to control the plotter. These are relatively simple processes and for our requirements about 10 hours computer time per year should be adequate.