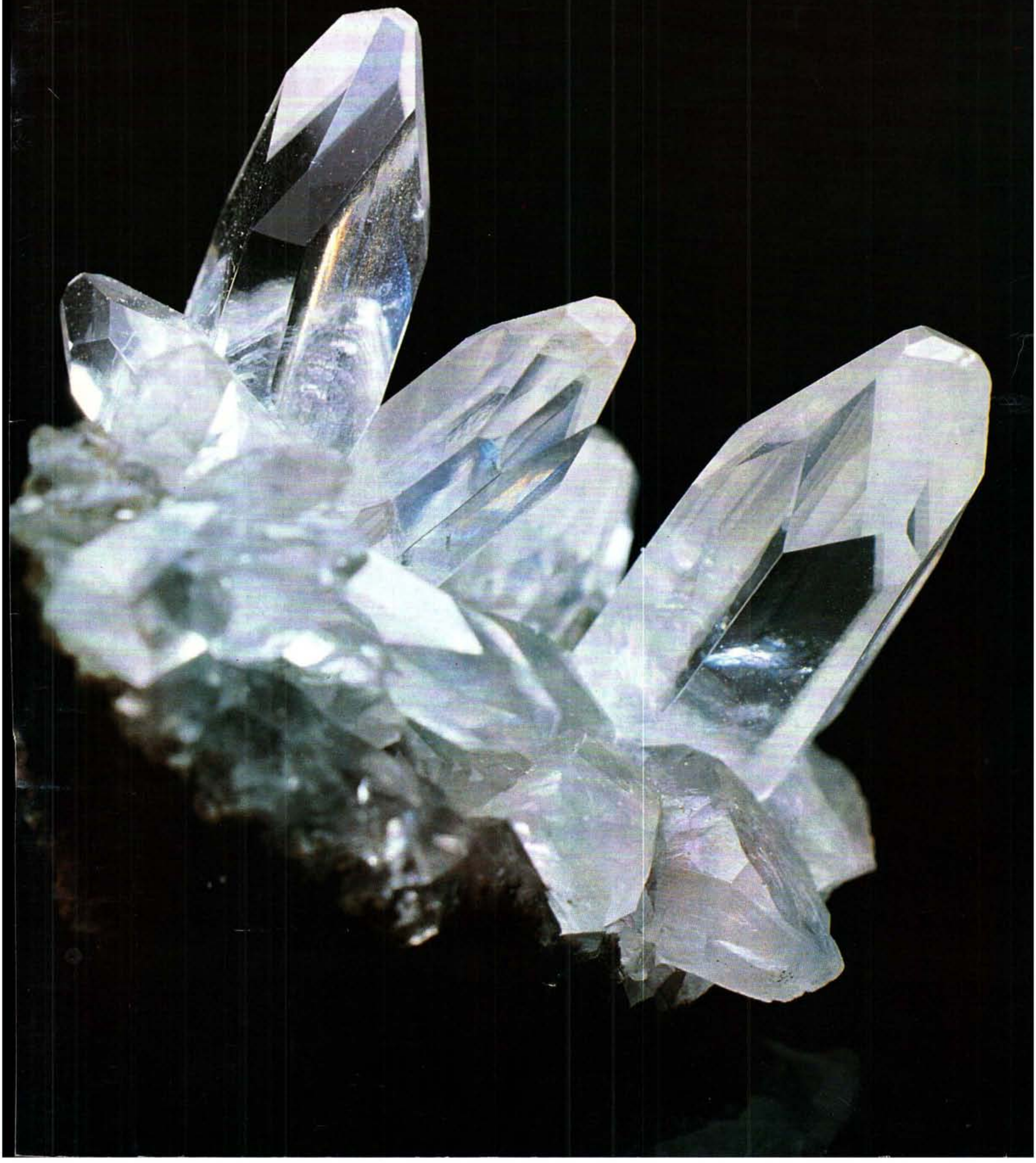
JOURNAL
OF
The Russell Society

Volume 3, Part 1 1990



JOURNAL OF THE RUSSELL SOCIETY

The Journal of British topographical mineralogy

Aims and Scope

The Journal publishes articles and reviews by both amateur and professional mineralogists dealing with all aspects of mineralogy. Contributions concerning topographical mineralogy are particularly welcome.

In addition to full articles, short notes (up to 1000 words) may be submitted. Book reviews and correspondence will also be accepted with a view to publication. Notes for contributors can be found at the back of the Journal.

Subscription rates: The Journal is free to members of the Russell Society. Subscription rates per volume (two issues per year) £10. Enquiries should be made to the Journal Manager at the above address. Back copies of the Journal may also be ordered through the Journal Manager.

Advertising: Details of advertising rates may be obtained from the Journal Manager.

Published by The Russell Society.

ISSN 0263 7839

FRONT COVER: Calcite crystals, Bigrigg Mine, Egremont, Cumbria, U.K. (National Museum of Wales Specimen, NMW 83.41G.M5199, *ex* King Collection). The largest crystal is 2.8 cm in length. Photo: M.P. Cooper.

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EDITORIAL

The establishment of a new journal is not an easy task, particularly in the present financial climate. Nevertheless, the Journal of the Russell Society has now reached Volume 3, and production has been placed on a more regular basis. It is the intention to produce one volume in two parts per annum, one appearing early in the year (February-March), the other later (September-October). Production costs of course are high, too high to be borne solely by funds received from membership subscriptions. We have been extremely fortunate to have been in receipt of considerable sponsorship from Bardon Hill Quarries Ltd., Redland Aggregates Ltd., and for the colour cover of this and the last issue from Ralph Sutcliffe. We are very grateful for these commitments, and would hope to attract further sponsorship for future issues. However, we must also look to additional sources of income to help offset production costs (hence the advertising in this and the last issue) and we are also grateful to those advertisers who have shown faith in the Journal and the Society by placing advertisements with us. We hope that they will continue to support us in the future.

The Journal itself has continued to evolve as comments and criticicms have been received and considered. Volume 2(2) had, we believe, more of a balance than the previous issue, presenting contributions from the amateur field as well as from professionals. Those amateurs who have contributed are to be congratulated in their works, and we hope that they will provide encouragement to others who have something to record but who feel uneasy about comitting themselves to print. We can only reiterate that all contributions will be welcomed and that those from amateurs will be dealt with sympathetically and with appropriate discretion. Members of the Editorial Board will do their utmost to offer assistance in transforming initial drafts into articles of an appropriate style and format for publication.

This issue of the Journal sees publication of a paper describing the use of X-ray diffraction studies in the identification of minerals. It aims to explain to the non-professional some of the theory behind the technique and to illustrate applications by way of a worked example. This is intended as the first of a series of such papers detailing the various techniques available for mineralogical investigations including, for example, electron microprobe analysis, infrared spectrophotometry and so on.

At the end of the day, however, the Journal can only expand its scope and content as far as contributions permit. Copy for the next issue is presently being received, but we would encourage even more contributions, particularly from the non-professional sector. If contributions are received only from professionals, then the Jounal will have failed in one of its principal aims. Members of the Editorial Board have provided stirling support for me and the Journal co-editor; we now need similar support from members of the Russell Society by way of submitted articles and notes. We see this as an important way of putting the Journal on a sound footing as far as contents are concerned.

R.E. Bevins

The Russell Society gratefully acknowledges the
generous financial support of
BARDON HILL QUARRIES LTD. and **RALPH SUTCLIFFE**
towards publication costs

J. Russell Soc., 3(1), 1–6 (1990).

ALSTONITE AND BARYTOCALCITE FROM LLANTRISANT, SOUTH WALES, AND BARYTOCALCITE FROM HOLWELL, MENDIP HILLS, ENGLAND

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Alstonite and barytocalcite are both recorded from new localities. Alstonite associated with witherite, calcite, dolomite, barite, and quartz accompanies low temperature hydrothermal Pb-Zn-Fe-Cu sulfide mineralization in veins and cavities in dolomitized Triassic marginal deposits immediately to the south of Llantrisant, Mid Glamorgan. Barytocalcite forms part of an extensive suite of late-stage minerals associated with an Upper Triassic age iron and manganese oxide vein exposed in a working Carboniferous Limestone quarry in the eastern Mendip Hills, Somerset. These occurrences are compared with similar examples elsewhere.

INTRODUCTION

Three naturally occurring polymorphs of the compound $BaCa(CO_3)_2$ are known, namely alstonite (triclinic), barytocalcite (monoclinic) and paralstonite (hexagonal). Alstonite and barytocalcite were first described from the northern Pennine Pb-Zn orefield where they occur as primary hydrothermal vein minerals, usually in association with witherite. To date alstonite has been found in very small amounts at five localities in the northern Pennines (Young, 1985), although in addition to the record from Llantrisant (descibed here) only from another 4 localities world-wide (U.S.S.R., Kapustin, 1971; U.S.A. (California and Illinois), Rogers, 1932, Lillie, 1988; and Poland, Dziedzic and Ryka, 1983). Barytocalcite, although locally a major vein mineral in the northern Pennines, is likewise of very limited occurrence outside of this area. Brief mention is made of its occurrence in the Llantrisant area by Prior (*in* North, 1916). Paralstonite has, to date, only been recorded from the Cave-in-Rock district, Illinois, U.S.A. (Roberts, 1979).

GEOLOGY AND MINERALIZATION OF THE LLANTRISANT AREA

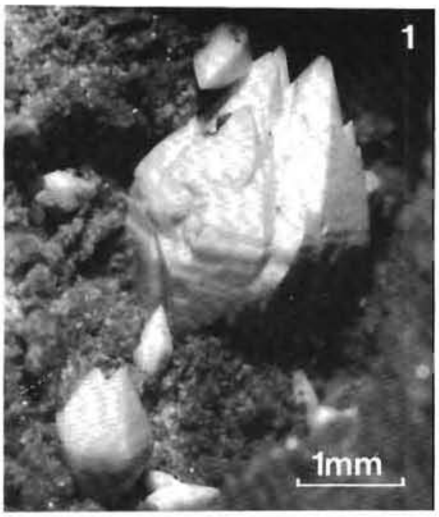
Llantrisant (Mid Glamorgan), situated approximately 10 km northwest of Cardiff, lies on the southern margin of the South Wales coal basin. The district falls within the former Taffs Well-Llanharry iron ore field and numerous derelict workings for iron ore within the Carboniferous Limestone occur throughout the area. Immediately to the south of the town, Triassic marginal deposits, known as the Dolomitic Conglomerate, comprise partially dolomitized conglomerates with limestone and sandstone clasts and interbedded calcareous marls. They form an E-W elongated (*ca* 0.3 × 1 km) outlier which rests unconformably upon Carboniferous sedimentary rocks. Small-scale Pb-Zn-Fe-(Cu) sulfide mineralization with a predominantly calcite-witherite-dolomite-barite gangue occurs throughout the Triassic rocks. A geochemical survey of the outlier (Bowler and Kingston, 1970) identified eight former mining sites (Pb-Zn) and showed that the mineralization consists of both simple narrow fissure fillings along a prominent vertical E-W joint set, and complex brecciated veins up to 1 m in width.

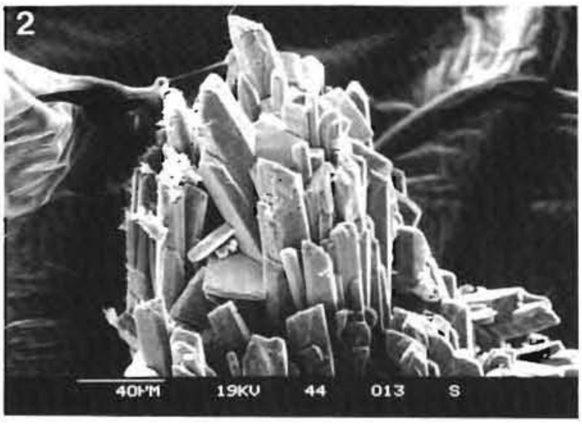
The Dolomitic Conglomerate is well exposed in the bypass road cutting [Nat. grid ref. ST 053 820]. At this locality, ovoid calcite-lined geodes up to 25 cm diameter and much smaller irregular cavities occur throughout the conglomerates, and are developed within both the limestone clasts and the matrix. The walls of the vughs and geodes consist of saccharoidal dolomite which is commonly intergrown with other filling minerals. Calcite-witherite (+ minor barite) geodes and cavity fillings are abundant and sometimes contain chalcopyrite grains and/or secondary copper minerals. Small quantities of quartz may also be present, but quartz-lined cavities are rare. Similar mineralized cavities and calcite-lined geodes can be found at several of the old mine sites. Celestine-barite-calcite geodes have previously been reported from the road cutting (Thomas, 1968), but none were found during this or an earlier (Bowler and Kingston, 1970) study. At several localities in the area the usually massive witherite fill is occasionally associated with and grades into a compact, coarsely-prismatic to acicular variety (the latter showing a silky lustre) composed of parallel groups of inseparable crystals. Similar material occurs in some of the witherite veinstone at mine site 'BK3' of Bowler and Kingston (1970) [ST 063 824]. X-ray powder photographs give no indication of any mineral other than witherite. Quantitative microprobe analysis of the witherite reveals trace levels of strontium.

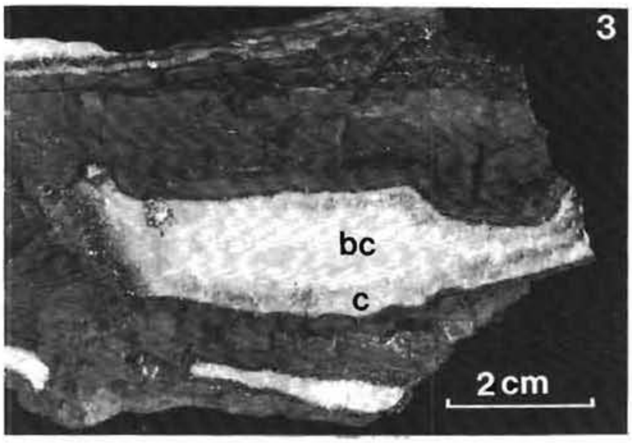
BARIUM MINERALS IN THE LLANTRISANT AREA

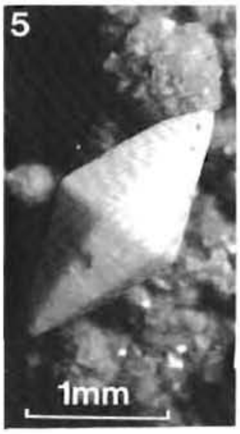
Alstonite occurs as a minor component of the carbonate gangue at one old mine site, [ST 063 824], locality 'BK3' of Bowler and Kingston (1970), and very sparingly in dolomite-lined microcavities in the conglomerate at the southern end of the bypass road cutting [ST 053 819].

Alstonite is most abundant at the mine site where it is found both within dolomite microcrystal-lined cavities in the conglomerate and as a very minor component of









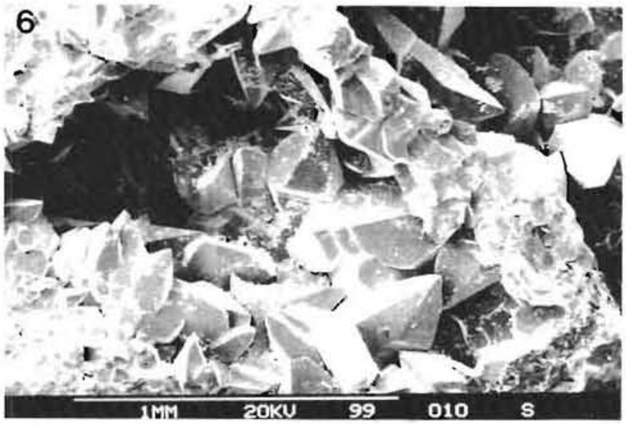


FIGURE 1.1. Barite pseudomorphing compound crystal of alstonite. Mine BK-3 (of Bowler and Kingston, 1970), Llantrisant; **1.2.** Bladed aragonite crystals totally encrusting relic pyramidal alstonite crystal in an area where alstonite has been partially replaced by barite. Mine BK-3, Llantrisant; **1.3.** Barytocalcite (bc) filling a calcite (c)-lined cavity in banded pyrolusite-manganite ore. Colemans Quarry; **1.4.** Barite pseudomorph after alstonite, broken open to show cavernous internal structure. Mine BK-3, Llantrisant; **1.5.** Barite pseudomorphing dipyramidal alstonite crystal. Mine BK-3, Llantrisant; **1.6.** Alstonite crystals lining a vugh in massive witherite veinstone. Mine BK-3, Llantrisant; **1.7.** Group of alstonite crystals. Mine BK-3, Llantrisant.

some of the brecciated veins. In the cavities alstonite commonly occurs as clusters of pseudohexagonal, colourless, transparent to greyish translucent, acute pyramidal or doubly terminated dipyramidal crystals, up to 6 mm in length (Fig. 1.6, 1.7); because of multiple twinning these commonly exhibit characteristic horizontal striations on pyramid faces, and occasionally shallow vertically-bisecting re-entrant angles. Some specimens have a distinctly pink tinge. This colour, unlike that of similar crystals from the northern Pennines (Palache *et al.*, 1951) appears to be stable under normal daylight conditions. Quantitative microprobe analyses show no difference between the pink and colourless crystals. Trace amounts of strontium are invariably present in both types. Lead, copper and iron were detected in some specimens. Manganese, reported as a common trace impurity (Palache *et al.*, 1951), was not found. The crystals occur either singly, encrusting the cavity walls, or are accompanied by calcite, witherite, barite, or sulfides, the chief of which is pyrite. Examination of about 100 alstonite-bearing cavities revealed pyrite euhedral against alstonite (common), alstonite encrusted by pyrite (very rare), alstonite containing very small (presently unidentified) sulfide inclusions (rare), alstonite encrusting or being encrusted by calcite (rare), alstonite encrusting euhedral dolomite microcrystals (common) and platy or granular barite (rare), replacements of both alstonite and witherite by barite (very common), and alstonite euhedral against witherite (universal). In the latter case pyramidal alstonite crystals are sometimes developed upon earlier euhedral calcite. Very rarely cavities up to 3 cm diameter are lined or completely filled with radiating groups of colourless prismatic alstonite which locally show hexagonal pyramidal crystal terminations.

'Breccia-ore' (Bowler and Kingston, 1970) comprises the majority of the sulfide-bearing vein material on the dumps. It typically consists of angular polymineralic clasts of galena, pyrite, sphalerite, rare chalcopyrite, and wall rock set in a matrix of vughy white to grey translucent witherite, with subordinate calcite and dolomite. Witherite appears to be the principal barium mineral in the veins at this locality. Barite occurs both as early-formed platy crystals pre-dating witherite, and as late-stage replacements of witherite and alstonite, as well as, and sometimes accompanied by, extensive euhedral encrustations on earlier vein components. However, except where the barite is clearly seen to encrust and replace witherite and/or alstonite, differentiation of the two generations of platy barite, in hand specimen, is virtually impossible. Quartz is widely distributed but only occurs in minor amounts. Alstonite is a very rare component of these veins although at least two distinct generations can be recognised. Early-formed alstonite consists of rare, grey to colourless pyramidal or dipyramidal crystals up to 2 mm long which are typically euhedral against witherite. It encrusts dolomite, sulfide and wall-rock clasts, platy barite and calcite and generally shows similar relationships to the principal vein minerals to those displayed by alstonite in the cavities. Later-generation alstonite occurs in the form of minute, coarsely crystalline colourless vughy veinlets, up to 0.5 mm wide, and hair-line fracture fillings which cut massive witherite. The vughs are either lined with pyramidal or dipyramidal crystals or traversed by crystal-encrusted septae, some of which are continuous with the veinlets in the witherite. Crystalline encrustations of alstonite on euhedral witherite crystals in small vughs (Fig. 1.6), and massive witherite encrustations on euhedral alstonite crystal cavity-linings, in turn developed upon massive witherite, indicate that some degree of overlap in the deposition of these two minerals has locally occurred. Euhedral colourless quartz crystals and, locally, sulfide (pyrite, chalcopyrite) grains occasionally accompany the alstonite. No alstonite encrustations on clearly identifiable late-stage barite have been seen. Networks of vughy calcite veinlets, sometimes containing alstonite and sub-millimetre sized sulfide grains, (galena, chalcopyrite, pyrite, sphalerite) are found in many blocks of shattered conglomerate wallrock. In this material alstonite occurs both as colourless granular mixtures with calcite and as euhedral crystals lining vughs where it both encrusts and is encrusted by coarsely crystalline calcite.

In both the cavities and veins alstonite occurs partly altering to aragonite. In the early stages acicular aragonite crystals form felted overgrowths, their long axes aligned parallel to the length of the alstonite crystal. This ordering is lost where radiating sprays of acicular crystals develop as alteration proceeds. The occurrence of sheaves or tufts of aragonite crystals, especially within the veins, is often a good indictation of the presence of alstonite. Partial or complete replacements of both alstonite and witherite by white powdery to granular barite are widespead. A thin replacing barite surface layer is often found on witherite crystals throughout the area. Acicular aragonite sprays or crusts may be developed where alstonite has been replaced (Fig 1.2). Hollow, white, opaque pseudomorphs of barite after alstonite and witherite also occur (Fig. 1.4) . In these very fine surface detail may be present but usually only the general shape of the crystal is preserved (Fig. 1.5). Barite pseudomorphs after alstonite have also been noted in the northern Pennines (B. Young, personal communication).

Unaltered alstonite dipyramids (up to 0.5 mm length) and hollow barite pseudomorphs occur sparingly within microcavities at the southern end of the bypass road cutting [ST 053 819]. No alstonite was identified in any of the witherite-calcite geodes collected during this study or in similar material collected during the construction of the bypass, and now incorporated into the collections of the National Museum of Wales. Alstonite was not found in any of the sufide-bearing, calcite-barite joint fillings exposed in this section.

Barytocalcite was provisionally reported from the Llantrisant area by Prior (*in* North, 1916). He described a specimen, possibly obtained from the Mwyndy iron mine, now in the collections of the Natural History

Museum, London as specimen BM 62133, which has been confirmed as barytocalcite (R. F. Symes, personal communication). This closely matches a specimen labelled as coming from the Llantrisant area, donated to the National Museum of Wales by the Cymmer Colliery Workman's Library (specimen NMW 25.554. GR7). This specimen consists of a crust of colourless to yellowish transparent bladed crystals and massive material accompanied by minor pyrite, lining a cavity in siliceous goethite. The crystal habit is identical to that of barytocalcite from the northern Pennines.

GEOLOGY AND MINERALIZATION OF THE HOLWELL AREA

Colemans Quarry [ST 724 454], at Holwell, near Frome, Somerset, situated on the northern flank of the Beacon Hill Pericline, works well-bedded, southerly dipping limestones of the Lower Carboniferous Black Rock Group which in the quarry area are overlain unconformably by up to 5 m of massive, horizontally-bedded Middle Jurassic bioclastic limestones (Doulting Stone).

Since 1985, when quarrying began, small pockets and veins of iron and manganese oxides, associated with a prominent N-S trending system of faults and joints, have periodically been encountered within the Carboniferous Limestone. Some of these fractures were subsequently developed into deep narrow fissures and are now seen to contain a sedimentary fill which ranges in age from Upper Triassic to Middle Jurassic (*c.f.* Moore, 1867). The iron-manganese mineralization is truncated by the plane of unconformity but galena-, pyrite-, and sphalerite-bearing calcite veins, some of which also cut the iron-manganese oxide bodies and/or the sediment-filled fissures, continue up into the Jurassic limestones. A few of these veins also carry chalcopyrite, but this mineral appears to be restricted in occurrence to the immediate vicinity of the vein intersections with the manganese oxide concentrations. Patchy silicification of the Doulting Stone, recorded elsewhere in the eastern Mendips by Stanton (1981), is present. Very similar iron and manganese oxide mineralization within the Carboniferous Limestone occurs at several localities in the eastern Mendips. An Upper Triassic age has been ascribed to these deposits by Symes and Embrey (1977). A small derelict iron mine, with associated manganese oxides, lies approximately 300 m NE of the quarry.

BARYTOCALCITE FROM THE HOLWELL AREA

Barytocalcite was found in one small vein, now quarried away, associated with a vertical N-S trending joint swarm at the southern end of the quarry. Continuous over a strike length of *ca* 30 m, the vein averaged 10 cm in width with local fluctuations from <5 cm to pockets 30 cm wide. It appeared to narrow with depth and pinched out along strike. The vein was seen only in the second quarry bench, the top bench having already been cut back, indicating a probable total vertical development over at least 15 metres. Pyrolusite, manganite, and smaller amounts of hausmannite were major constituents. Iron oxides (geothite and hematite) formed only a small component of the vein, occurring as discrete pockets of massive ore and thin discontinuous ferruginous selvages along the vein margins. Rhodochrosite (confirmed by XRD) was sparingly developed but the limestone adjacent to the vein margins often had a distinctly pink tinge, especially where fractured and veined by manganese oxides. Thin discontinuous vughy seams of white or banded black and white calcite and elongated calcite geodes, flattened in the plane of the vein, highlighted a coarse banding along strike.

Barytocalcite forms part of the late-stage mineral assemblage which, in common with similar manganese oxide deposits throughout the Bristol district, is mostly developed within calcite-lined cavities in the manganese oxide. To date, late-stage minerals found in the manganese veins in this quarry include manganite, hausmannite, crednerite, goethite, cuprite, quartz, kentrolite, melanotekite, chloroxiphite, mendipite, diaboleite, paralaurionite, mimetite, pyromorphite, cerussite, hydrocerussite, aragonite, malachite, rhodochrosite, barytocalcite, calcite, fluorite, barite, and two as yet unpublished new minerals (R.F. Symes, personal communication).

In this one vein, barytocalcite was fairly common. In every case it occurred within calcite-lined cavities in the manganese oxides, always in the form of closely packed groups of mutually interfering transparent to translucent colourless to yellowish-white or greenish-yellow platy crystals showing the prominent {210} cleavage, vitreous to resinous lustre and sub-conchoidal fracture. Well-formed crystals have not been found. In most cavities, barytocalcite is intergrown with cerussite, rarely cerussite and hydrocerussite. Barytocalcite is euhedral against the lead carbonates. Less commonly associated minerals include massive pale yellow pyromorphite, developed at the margin of the cavity, a partially altered orange as yet un-named new mineral, and submillimetre diameter clusters of euhedral manganite crystals as inclusions within and encrustations upon the barytocalcite, cerussite, and enclosing calcite cavity lining. Barite, a ubiquitous mineral in this deposit, occurs as colourless, minute tabular or lensoid crystals up to 0.1 mm long encrusting uncorroded barytocalcite, in microcavities between adjacent barytocalcite crystals, and also as larger (to 0.5 mm length) tabular crystals, projecting inwards from the cavity walls, which appear to predate the barytocalcite. No barite replacements or pseudomorphs after barytocalcite have been seen. Alteration of the barytocalcite appears to result in no more than a slaggy corroded mass of relic crystals. Examination of specimens from other iron and iron-manganese deposits in the Bristol district, in the collections of the Bristol City Museum, failed to reveal any further examples of baryocalcite. Witherite has not been found in this assemblage.

DISCUSSION

Parallels can readily be drawn between the mode of occurrence of alstonite in the northern Pennines (Young, 1985) and those at Llantrisant. However, in the northern Pennine veins most of the alstonite can clearly be shown to be a late-stage mineral which post dates associated witherite or barite, although some degree of overlap between witherite and alstonite has locally occurred (Young, 1985). At Llantrisant, the relative ages of the alstonite in the cavities, breccia-veins, and wall rock are less certain. The similarities in the relationships between associated minerals and alstonite in the cavities and first generation vein alstonite suggest that they may be more or less contemporaneous. It is probable that at least some of the calcite-alstonite veinlets in the wall rock are related to the alstonite veinlets which cut the massive witherite in the breccia veins. Dunham and Wilson (1985) noted the occurrence of coexisting calcite and witherite (±alstonite or barytocalcite) in some of the northern Pennine veins, whilst Young (personal communication) has observed the common association of alstonite and calcite, the latter apparently both pre- and post-dating alstonite, from at least two of the northern Pennine localities.

Descloizeaux (1881) briefly descibed a barytocalcite-like mineral associated with hedyphane and hausmannite from the skarn assemblage accompanying thermally metamorphosed and hydrothermally veined Precambrian, dolomite-hosted volcanogenic-sedimentary manganese oxide deposits at Långban, Sweden. These deposits are notable for the occurrence of an extensive suite of unusual accessory minerals (Moore, 1971) some of which have also been found in association with the Mn-Fe mineralization in the Bristol district and South Wales.

The barytocalcite from the Llantrisant area provides a closer parallel to the Mendip occurrence. The iron (and minor manganese) mineralization in South Wales and the Mendip Hills is thought to be contemporaneous and of low temperature, hydrothermal formation (Rankin and Criddle, 1985).

Young (1985), in a detailed review of the northern Pennine occurrences, noted that alstonite and barytocalcite appear to be mutually exclusive. Although they have been reported to occur in different veins in the same mine, they have never been found together. A close relationship was also demonstrated between the presence of calcium-bearing carbonate wall rocks and the occurrence of alstonite or barytocalcite in the veins. These minerals may have formed as a result of the influx of sulfate-deficient, carbonate-saturated fluids during the later stages of vein filling. Calcium was probably leached from the limestone country rock. A similar situation may have occurred at Llantrisant, except that the final phase of mineralization involved sulfate-rich fluids which precipitated barium as barite and effected widespread replacement of the earlier-formed barium carbonates. The apparent lack of a sulfate-deficient carbonate and barium-rich mineralizing phase in the geologically similar environment of the Mendip Hills probably accounts for the absence of witherite from the calcite-barite-sphalerite-galena veins in this area.

It is not known what factors influence the crystallization of $BaCa(CO_3)_2$. In the case of alstonite and barytocalcite, solution temperature and degree of ordering in the crystal lattices are thought to be important (Chang, 1965). Barytocalcite has been prepared under hydrothermal conditions over a range of temperatures >200°C. Attempts to crystallize alstonite from the same starting materials were unsuccessful, suggesting that it may only be stable at temperatures below 200°C. Alstonite (but not barytocalcite) was synthesized in aqueous solution at ambient temperatures by Kaushansky and Yariv (1986). How these findings relate to natural occurrences of alstonite and barytocalcite is not known.

At Llantrisant, the relative abundance of alstonite in cavities in the wallrock compared to its scarcity in the veins is striking. Kaushansky and Yariv (1986) synthesized small amounts of alstonite by equilibrating a 1 molar solution of $BaCl_2$ with powdered calcite for 20 days in a closed vessel. Reaction products included aragonite, recrystallized from the calcite, and major calcian witherite. No alstonite was formed when the $BaCl_2$ solution was percolated through calcite powder. In the Llantrisant deposit it seems probable that once suitable conditions for crystallization of alstonite had developed they were more easily maintained in the partially closed chemical environments of the cavities (thereby allowing significant quantities of alstonite to be precipitated) than in the more open environment of the veins. Deep, narrow embayments and crevices (having restricted fluid movement) in the developing breccia-veins would also have provided suitable microenvironments for the crystallization of alstonite. The work of Kaushansky and Yariv (1986) also suggests that the stability fields of alstonite and witherite share a common boundary. Within this critical zone minor fluctuations in solution chemistry would account for the localized overlap in deposition of these minerals. A similar relationship may exist between alstonite and calcite.

A highly complex chemical environment existed within the iron and especially the iron-manganese oxide orebodies during the development of the late stage mineral suites, with a wide variety of reactions occurring simultaneously in different parts of any one deposit (Abdul-Samad *et al.*, 1982). Although the chemistry of the interstitial fluids in each type of orebody (iron, and iron-manganese) appears to have followed the same general evolutionary trend, as evidenced by the similarity in their respective late-stage mineral suites, each deposit was still a unique chemical system and the inevitable minor differences are reflected in the presence of rarer species. The cavities in which the late-stage minerals are found were, by and large, the sites of these reactions. An overall abundance of carbonate in solution is evidenced by the common occurrence of carbonate-bearing minerals. That barytocalcite is present at Colemans Quarry almost certainly reflects the localised and transient development of essentially sulfate-free conditions which enabled evolving solutions with the correct chemistry to precipitate barium (probably desorbed from the crystallizing Mn oxide gels) as a carbonate instead of the more usual sulfate. The absence of witherite from this assemblage may well be more apparent than real. In the absence of precise locality information, the paragenesis of the Llantrisant barytocalcite remains an open question. Although it may well be a late-stage mineral formed under similar conditions to that at Colemans Quarry, the presence of pyrite in the cavity strongly suggests a genetic link with the alstonite occurrences.

ACKNOWLEDGEMENTS

The author wishes to thank the proprietors of Brofiskin Farm, Groes-Faen, Mid Glamorgan, and E.C.C. Quarries Ltd for permission to visit mines and quarries on their properties, Dr R. E. Bevins (National Museum of Wales) and Dr P. Crowther (Bristol City Museum) for granting accesss to material in their collections, Dr D. Thompson (Department of Chemistry, Bristol University) and Dr D. Dingley (Department of Physics, Bristol University) for microprobe facilities, Dr R. Bradshaw (Department of Geology, Bristol University) for reading the manuscript, and Mr B. Young (British Geological Survey, Newcastle) for prompting this study.

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IDENTIFICATION OF MINERALS USING X-RAY DIFFRACTION

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The use of simple X-ray diffraction techniques for identification of mineral species is described. Attention is focussed on the Debye-Scherrer photographic method of obtaining powder diffraction data and examples are given of the way in which unknown species can be identified using this method in conjunction with published powder diffraction data files.

INTRODUCTION

For any person interested in mineralogy, the variety and beauty of the shapes of crystals is amazing. It is important to realize that the shapes of the crystals (their *morphologies*) are not accidental. Of course, we make frequent use of this fact when attempting to identify unknown species.

The crystalline state is a special state of matter. It refers to solids, but solids with a very important characteristic. This is that the atoms, ions or molecules contained in a crystal are not arranged in a haphazard fashion (such an arrangement is said to be *amorphous*). Rather, the ions, atoms, *etc.* are packed in a regular fashion in a *repeating* three-dimensional array. This mode of packing is maintained throughout the crystal. For example, if a crystal of galena, PbS, is shattered, it will break into a number of cubic cleavage fragments. Each individual fragment has exactly the same structure, or packing arrangement of ions, as any other, and the same structure as that of the original crystal (here we refer to internal structure).

The same process may be repeated more or less indefinitely, far beyond the resolution of the human eye or the most powerful optical microscopes. However, a stage would be ultimately reached when the tiniest fragment cannot be subdivided further without destroying the arrangement of ions in space that is present in the crystalline solid. The smallest fragment of the crystal thus obtained in this imaginary way is called the unit cell of the crystalline compound, and a description of the structure inside it is a description of the structure of the whole substance. In effect, the unit cell is the smallest repeat unit of the whole crystal. A crystal of the compound is built up of many identical unit cells packed together in three dimensions. For galena, a picture of the unit cell is shown in Figure 1.

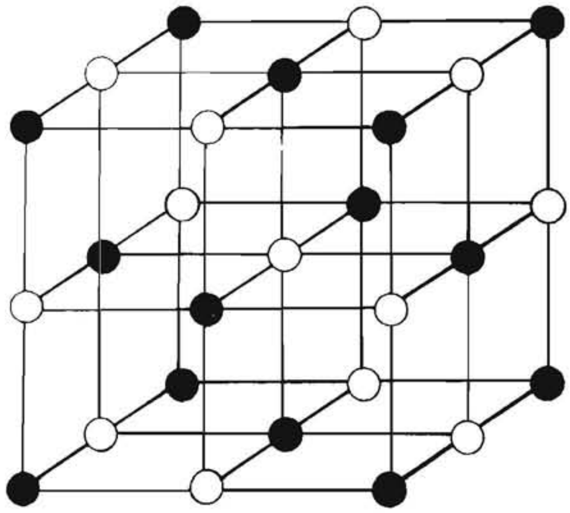


FIGURE 1. The unit cell of galena, PbS, and its structural analogues.

The cell is represented by the outside boundaries of the large cubic box. Here, the filled circles represent lead ions (Pb^{2+}) and the open circles represent sulfide ions (S^{2-}). If we imagine that a whole crystal is built up of packing cells face-to-face in all directions it is appreciated that atoms at the corners are shared by 8 other unit cells, atoms in the middles of the faces by 2 other unit cells and atoms (or ions) on the edges by four other unit cells. Simple counting leads us to conclude that in the unit cell of galena there are 4 whole lead ions and 4 whole sulfide ions. This explanation is entirely consistent with the *formula* of galena, PbS.

There are many ways of packing atoms and so on in regular arrays (230 in fact!) but that need not concern us here. What is important to realize is that every distinct chemical compound in the crystalline state has a *unique* packing arrangement. This is achieved in three ways. First, the basic structural arrangement may be different (one of the 230 possible, so-called *space groups*). Secondly, *groups of atoms or ions* may take the positions of the single points in the lattice shown in Figure 1 (in this case the spatial arrangement of the group is fixed as well). Finally, a third possibility exists. It is possible that two compounds (or indeed minerals) have the same structure. To continue with our example, we might consider that halite (sodium chloride) has the same structure as galena with Na^+ ions and Cl^- ions replacing Pb^{2+} and S^{2-} ions, respectively. However, it is true that Na^+ ions are of different size than Pb^{2+} ions; the same is true of the anions. Thus, in a crystal of NaCl, the distances between the ions are different than those between the ions in PbS. That is to say, the size of the unit cell is different.

Given these few simple ideas, it is possible to appreciate the fact that the unit cell, and thus the detailed structure of every single crystalline substance, is unique with respect to any other. If a method could be devised that measured these differences, one would have a very powerful tool for the unambiguous identification of crystalline solids. Some kind of measurement of the unit cell, or the spacings between atoms or planes of atoms, would give a 'fingerprint' of the particular substance. X-ray diffraction provides the most powerful and convenient method for carrying out just this kind of measurement. In certain special cases some difficulties arise, but for the vast majority of solids the measurement of an X-ray diffraction pattern does serve to identify the compound unambiguously if access to previously measured data is available. For mineralogists, the method is extremely useful, and measurements can be compared with those of all but a handful of known mineral species. One drawback, however, is the requirement for special, costly equipment.

THE DIFFRACTION OF X-RAYS

X-rays were discovered in 1895 by Wilhelm Conrad Röntgen (1845-1923). These 'rays' are simply part of the normal electromagnetic spectrum, which, in terms of decreasing wavelength and increasing energy, includes radio waves, infrared radiation, visible light, ultraviolet radiation, X-rays and gamma rays. Wavelengths of radio waves are typically measured in metres and X-rays have wavelengths very approximately equal to three times the diameter of an atom, say 1-2Å (1Å, or Ångström is equal to 10^{-8} or one hundredth of one millionth of one centimetre).

Uses for X-rays became apparent immediately after their discovery. Obvious medical applications were quickly developed; less radiation penetrates the dense mineralization of bone as compared with fleshy tissue and thus medical X-ray scans are, at their simplest level, employed to examine the skeleton. Effects which are important for mineral identification and determination of the structure of crystalline substances were first noted during the study of the interaction of X-rays with single crystals.

The experiments simply involved passing an X-ray beam through a tiny, stationary single crystal about which photographic plates had been positioned to record the sites of the emerging beams. When the photographic plates were developed, an initially surprising result was obtained. Instead of the X-rays emerging in a random, scattered, diffuse fashion, discrete spots were developed on the photographs. These corresponded to the shape of the tiny crystal used and furthermore the spots or reflexions were arranged in geometric patterns. It was quickly established that there was a relationship between the geometric arrays of spots and the morphologies of the crystals used in the experiments, or their symmetries. For example a tetragonal crystal would give rise to spots on the plate with four-fold symmetry, and hexagonal crystals, such as quartz, spots with six-fold symmetry. Obviously, the patterns of spots were related to the internal structures of the crystals, and it was a matter of only a short time before the relationship was properly established.

It is important for us to understand this idea, because it forms the basis of the use of X-rays to identify mineral species. The father of X-ray crystallography, W.H. Bragg, devised a simple mathematical relationship between the structure of crystalline substances and the patterns of spots (or reflexions) on the photographic plates. This is best viewed in terms of a two-dimensional representation of the lattice of galena as shown in Figure 2. Various sets of planes with regular spacings may be identified in the two-dimensional lattice, designated as AA, BB, CC. Many sets of planes could of course be envisaged. The distance between each plane is termed the *d-spacing*, and this varies from plane set to plane set. Since each unit cell has a different size, at least, then for any crystalline substance the set of d-spacings will be unique. An important fact is that X-rays are *reflected* (or *diffracted*) from these planes in a regular fashion; the angle of incidence of the X-ray beam is equal to the angle of reflection or diffraction, just as in the case when visible light is reflected by a mirror. This is illustrated in Figure 3, for two planes. Bragg simply pointed out that the single condition for an X-ray reflexion to occur is that the extra distance travelled in the crystal by ray b in Figure 3 must be exactly equal to an integral number of wavelengths of the radiation (in more formal terms, the emergent radiation must be *in phase*). The X-radiation used is *monochromatic*; that is,

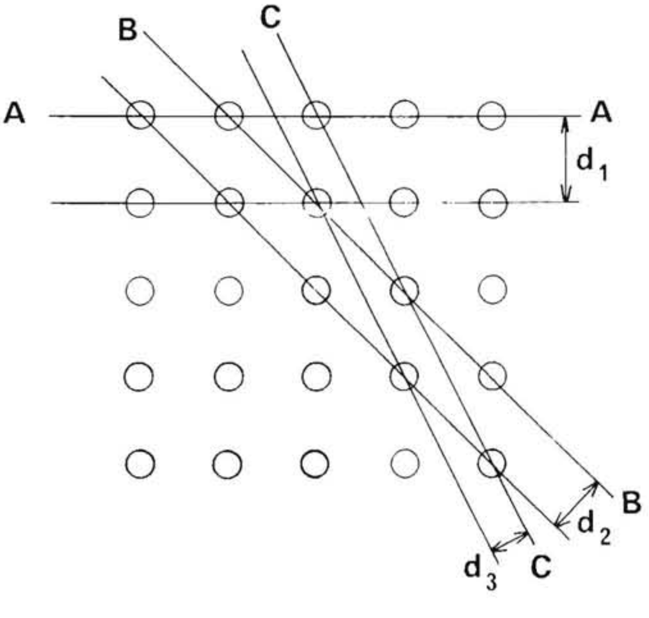


FIGURE 2. A two-dimensional representation of a lattice depicting various sets of planes and d-spacings.

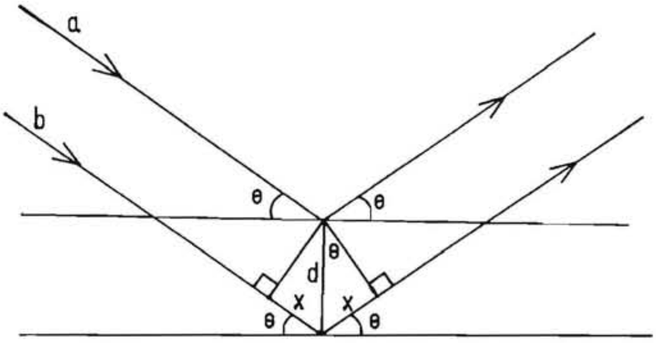


FIGURE 3. Geometrical derivation of Bragg's law.

it consists of X-radiation of one wavelength only. A little simple maths permits us to calculate the extra distance travelled by the ray b in Figure 3. That distance is equal to 2x.

Now, since $\sin\theta = x/d$, $x = d \sin\theta$ and the extra distance travelled, 2x, must be equal to an integral number, n, of wavelengths, λ, of the radiation used.

$$n\lambda = 2d \sin\theta \qquad (1)$$

This simple equation, (1), is known as the *Bragg equation* and it merely states that if we can measure the angle through which the spots are reflected and if we know the wavelength of the X-radiation used, then we can easily calculate the distance between the planes in the lattice that have given rise to the reflexion. D-spacings then will then form a family of distances for any one crystal, and these are calculated according to equation (2).

$$d = \lambda / 2 \sin\theta \qquad (2)$$

Measurements of many more reflexions allow us to calculate the size of the unit cell for any crystal.

There is another important piece of information that can be gained from the diffraction experiment. It had been observed that the *intensities* of the reflexions were not all equal, with some being very intense compared with others, and some being missing in places where one might have expected to observe them in terms of the geometric arrays developed on the plates. Subsequently it was pointed out that the intensities of the reflexions were related to the numbers of electrons which lay in the planes cutting the crystal or unit cell. Some atoms have more electrons than others and more atoms lie on certain planes than others. With this in mind it is easy to appreciate how these differences in intensity arise.

Finally, it should be pointed out that there is an easy way to *index* the reflexions. For our purposes we are interested in the relationship of the sets of planes to the unit cell. The unit cell is classically defined in terms of three dimensions, *a, b,* and *c* (the lengths of the edges of the unit cell) and the angles between the edges, α, β and γ (α is the angle between *b* and *c*, β the angle between *a* and *c*, and γ the angle between *a* and *b*; for a cubic cell $a = b = c$ and $\alpha = \beta = \gamma = 90°$, but for a triclinic unit cell, $a \neq b \neq c$ and $\alpha \neq \beta \neq \gamma \neq 90°$. Other possible arrangements form the *crystal systems*. The sets of planes are given the nomenclature *hkl* where *h, k* and *l* are whole numbers or zero. The set of planes *hkl* are those which cut the unit cell edge *a* into h^{ths}, *b* into k^{ths} and *c* into l^{ths}. If any set of planes does not cut an axis, the integer zero is employed. Thus the 200 reflexion arises from the set of planes which cut the unit cell in half (and at 0 and 1) along *a*, but do not cut the *b* or *c* edges. Once these coordinates have been assigned, the plane is said to have been 'indexed.' Interested readers may consult the texts given at the end of this article for further examples.

If we put these simple ideas together, a very powerful conclusion results. We could collect the d-spacings and their intensities for all available reflexions from a crystal. Suitable mathematical manipulations would give us the size of the unit cell, and the density of electrons on a series of planes which cut the cell at many different angles. We thus build up a three-dimensional electron density map inside the unit cell, and since we know that most electrons reside around the nucleii of atoms, we would in effect have located all of the atoms in their positions inside the unit cell. Thus we would have solved the crystal structure of the substance. This, in a nut-shell, is the basis of the 'single-crystal' method, and it has been used to determine structures as simple as that of sodium chloride and as complicated as that of haemoglobin, the molecule that transports oxygen in our blood.

Bragg (1914a,b) carried out the first structure determination on halite, NaCl. Shortly afterwards (Bragg, 1914c,d) the structure of sphalerite, ZnS, among other species, was reported. Since that time, roughly half of all known minerals have had their single-crystal X-ray structures determined. New structures appear at the rate of approximately 100 per year. An important source of this kind of information is found in *Mineralogical Abstracts*, which lists X-ray structural studies in a separate section.

IDENTIFICATION OF MINERAL SPECIES USING X-RAYS

Single crystals of any mineral *can* be uniquely characterized using the above methods, supplemented by a good chemical analysis. However, this would be a laborious process and one which turns out to be quite unnecessary for identification purposes. Only a few d-spacings are in fact required to distinguish one mineral from almost any other, and more convenient techniques in terms of effort, and certainly in terms of time, are thus employed. Instead of single-crystal measurements, a powder diffraction technique is employed. This has other advantages in that for a particular sample, single crystals may not be available, or they may be so small as to render impossible a single crystal determination.

Several kinds of measurements of X-ray powder

FIGURE 4. A Debye-Scherrer camera with collimator (left) and beam stop (right) mounted. The cover plate has been removed to aid viewing. Devices to centre the sample and retain the film are at the top of the camera.

diffraction have been devised. Perhaps the most common in use is the Debye-Scherrer method and this will be described here. A picture of a typical Debye-Scherrer camera is shown in Figure 4. It consists of a cylinder around the inside of which is fitted the film used for recording the diffraction pattern. The camera is mounted next to an X-ray source of fixed wavelength (commonly $CuK\alpha, \lambda = 1.5418$Å or $MoK\alpha, = 0.71069$Å). The X-ray beam is admitted to the camera (a close fitting plate fits over the exposed face) *via* a collimator (on the left of the camera) and radiation passing through or around the sample is trapped by the beam stop (right). Two circular holes are punched in the film strip to fit the beam stop and collimator.

The sample to be analysed is ground in a mortar and pestle to a fine powder (it should be dry) and either collected in a small bead of glue on the end of a glass fibre or packed into a thin 'Lindemann' glass capillary, typically of 0.2 mm inside diameter. About 1mg of sample or less is sufficient for the experiment. It can be sometimes difficult, but not impossible, to grind and pack smaller amounts, and perhaps 0.25mg will suffice. The sample is positioned in the beam and then rotated by an electric motor fixed to the rear of the camera to expose the randomly packed crystals to the beam in all possible orientations. Bragg's law for the observation of the diffraction is still obeyed, but the effect of having many tiny crystal fragments in the sample in a variety of orientations is to produce, instead of single spots on the film, cones of reflexions, which are recorded on the film as circles. If θ is large not all of the circles are recorded on the film, but a series of arcs, on either side of the exit (beam-stop) hole corresponding to sections of the circles of reflexion, are obtained (the circles are in fact a cross section of the cones of reflexion).

Films are removed from the camera after a suitable exposure time (1-12 hrs) and developed. Some examples are shown in Figure 5. Different sizes of Debye-Scherrer cameras are available, but the most convenient is made such that the internal circumference is equal to 360 mm. The *diameter* of the circles recorded on the film correspond to an angular measurement of 4θ. For a 360

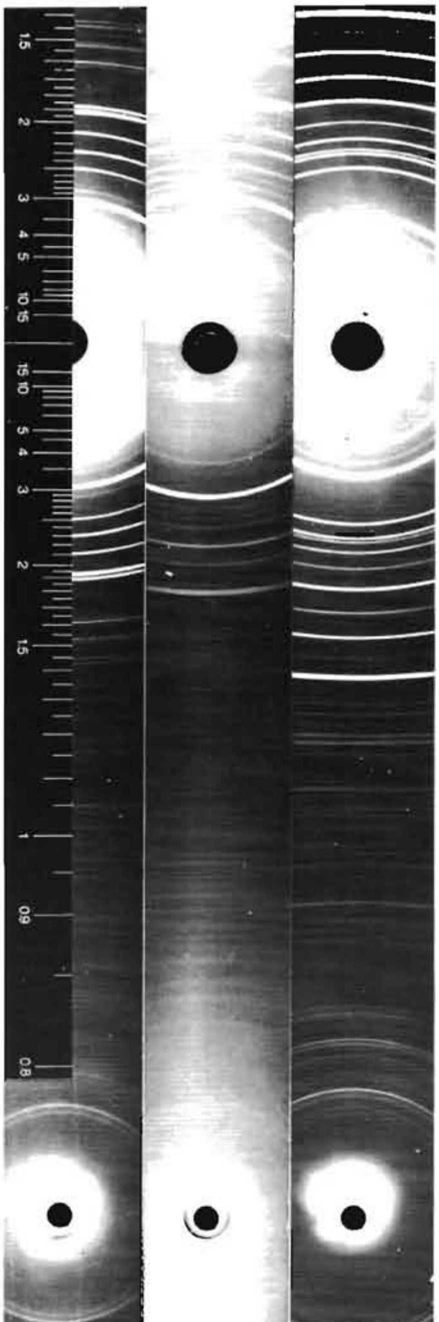


FIGURE 5. Debye-Scherrer powder photographs of a: calcite; b: dolomite; c: quartz; 114.6 mm diameter camera, CuKα radiation.

mm circumference camera, the angles are measured directly in millimetres. For accurate measurements, especially for low values of θ, some Vernier-type device is frequently employed. Values of θ in degrees, corresponding to individual d-spacings are calculated and, from a knowledge of the wavelength of the X-radiation used, the value of d are obtained in Ångström units.

We have already established that the list of d-spacings thus obtained provides a unique fingerprint of the particular mineral (or any crystalline) species involved. It is now a simple matter to compare the results obtained with the data file for d-spacings of known minerals. One should remember that the file contains most minerals, but not the most recent ones to be published. However, save for exceptional circumstances, and in the case of the occasional amorphous mineral, characteristic powder X-ray data must be supplied with any description of a new mineral. For recent publications, the primary literature ought to be consulted prior to drawing the conclusion that the sample is a new mineral, even if it is not found in the file.

The experimental data (d-spacings) should be tabulated and an estimate of intensity made. The latter can be in terms of 'strongest, strong, medium, weak, very weak' and so on, or intensity can be estimated on a relative scale with the strongest line being assigned a value of 100. An extremely useful data base for X-ray powder data is known as the IPDF (International Powder Diffraction File) for minerals. It is available in two parts. First a series of search manuals is provided. One of these lists minerals in alphabetical order and lists the eight strongest diffraction lines in terms of d-spacings together with their relative intensities on a scale of 1 to 10, with the strongest line being designated 'x' (see Figure 6 for an example of the numerical data in relation to the next comment). Another manual lists

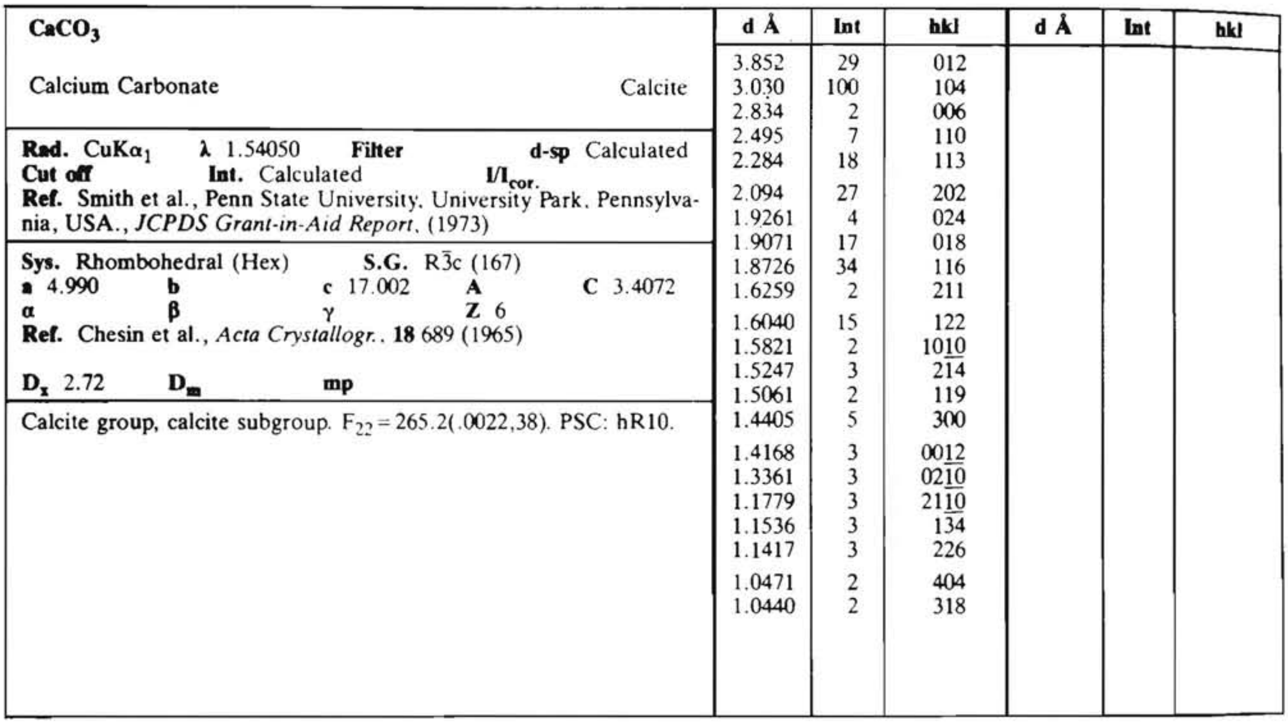
3.04 – 3.00 (± .01)

										File No.
3.02_x	1.94_7	2.18_5	3.25_3	3.11_3	2.70_3	2.64_3	3.75_2	**Miharaite**	$Cu_4FePbBiS_6$	33– 461
3.02_x	1.91_3	3.05_2	2.45_2	1.50_2	1.75_1	1.74_1	1.51_1	**Cooperite**	PtS	26–1302
3.02_x	1.91_4	1.51_4	0.78_3	1.75_2	2.45_2	1.73_2	1.23_2	**Cooperite, syn**	PtS	18– 972
3.01_9	1.90_x	1.75_9	2.29_7	3.51_3	2.26_3	4.99_2	2.48_2	**Alunite**	$KAl_3(SO_4)_2(OH)_6$	4– 865
3.05_x	1.87_8	1.59_8	2.64_6	1.52_4	1.21_4	1.18_4	1.02_4	**Stannomicrolite**	$Sn_2(Ta,Nb)_2O_7$	23–1441
3.05_x	1.87_9	1.59_7	1.93_6	2.50_5	2.08_4	1.71_4	2.83_3	**Giraudite**	$(Cu,Zn,Ag)_{12}(As,Sb)_4(Se,S)_{13}$	35– 525
3.04_x	1.87_x	1.59_5	1.08_3	2.77_1	1.82_1	1.66_1	1.32_1	**Arsenosulvanite**	Cu_3AsS_4	25– 265
3.03_x	1.87_3	3.85_3	2.09_3	2.28_2	1.91_2	1.60_2	2.50_1	**Calcite**	$CaCO_3$	24– 27
3.05_x	1.86_7	2.64_5	1.59_5	3.73_4	2.07_4	1.71_4	1.08_4	**Freibergite, syn**	$(Cu,Ag,Zn)_{12}Sb_{4.4}S_{12.6}$	27– 190
3.05_x	1.86_9	1.59_7	1.58_6	1.20_6	1.08_6	1.32_5	1.07_5	**Luzonite, syn**	Cu_3AsS_4	10– 450
3.04_x	1.86_4	1.59_3	1.87_2	1.58_1	2.64_1	1.21_1	1.08_1	**Chalcopyrite**	$CuFeS_2$	35– 752
3.04_x	1.86_7	1.59_5	1.32_2	2.63_1	4.30_1	5.25_1	1.52_1	**Arsenosulvanite, syn**	$Cu_3(As,V)S_4$	35–1017
3.04_x	1.86_x	1.59_x	1.01_x	3.17_9	1.37_9	1.02_9	2.02_8	**Cesstibtantite**	$(Cs,Na)SbTa_4O_{12}$	35– 672
3.03_x	1.86_9	1.58_9	2.63_8	6.07_6	3.17_5	1.21_5	1.17_5	**Natrobistantite**	$(Na,Cs)Bi(Ta,Nb,Sb)_4O_{12}$	35– 706
3.02_x	1.86_5	2.66_4	3.14_3	2.44_3	2.03_3	1.78_2	1.17_2	**Argyrodite**	$Ag_8(Ge,Sn)S_6$	14– 356
3.02_x	1.86_9	1.58_9	1.08_8	1.02_8	1.21_7	2.62_6	1.18_6	**Plumbopyrochlore**	$(Pb,Ln)_{2-x}(Nb,Ta)_2O_6(OH)$	25– 453
3.00_x	1.86_8	1.58_6	2.62_4	2.06_4	1.20_4	1.07_4	2.47_3	**Tetrahedrite, argentian**	$(Cu,Ag,Fe)_{12}Sb_4S_{13}$	11– 101
3.02_x	1.85_8	1.58_7	2.61_4	1.21_3	3.34_2	1.56_2	1.31_2	**Sinnerite, syn**	$Cu_6As_4S_9$	25– 264
3.01_x	1.85_3	2.61_2	1.58_1	5.97_1	2.40_1	1.51_1	3.15_1	**Bindheimite, syn**	$Pb_2Sb_2O_6(O,OH)$	18– 687
3.05_x	1.84_8	1.18_8	2.73_7	2.49_7	2.16_7	1.25_7	1.08_7	**Hauerite, syn**	MnS_2	25– 549
3.01_x	1.84_x	7.32_7	1.08_8	0.99_7	3.10_6	2.09_6	1.28_6	**Daomanite**	$(Cu,Pt)_2AsS_2$	29– 573
3.01_x	1.84_2	2.61_2	1.56_1	6.03_1	0.00_1	0.00_1	0.00_1	**Pyrochlore**	$(Ca,Na)_2(Nb,Ti)_2O_6F$	17– 746
3.01_x	1.84_8	1.58_6	7.30_4	2.61_3	1.20_2	1.17_2	1.72_1	**Uranpyrochlore**	$(U,Ca,Pb)(Nb,Ta)_2O_7$	29–1411
3.00_x	1.84_6	1.57_5	5.98_3	3.13_2	2.60_2	1.19_2	1.50_2	**Pyrochlore**	$(Na,Ca,U)_2(Nb,Ta)_2O_6(OH,F)$	13– 254
3.00_x	1.84_9	1.57_9	3.14_8	5.94_6	2.60_6	2.00_6	1.76_6	**Bismutomicrolite**	$(Bi,Ca)(Ta,Nb)_2O_6(OH)$	26–1042
3.00_x	1.83_4	1.56_3	2.59_2	0.78_1	1.19_1	0.88_1	0.79_1	**Pyrochlore, heated**	$(Ca,Na)_2(Nb,Ti)_2O_6F$	17– 747
2.99_x	1.83_6	1.56_5	2.58_4	5.94_3	1.50_3	1.19_3	1.16_3	**Stibiobetafite, heated**	$(Ca,Sb)_2(Ti,Nb,Ta)_2(O,OH)_7$	35– 695
3.01_8	1.82_x	4.17_8	3.12_8	3.58_6	1.61_3	1.57_3	2.79_2	**Tungusite**	$Ca_4Fe_2Si_6O_{15}(OH)_6$	19– 231
2.99_9	1.81_x	2.13_8	1.15_9	1.07_9	1.01_8	1.23_7	3.49_6	**Tolovkite**	$IrSbS$	35– 656
3.03_x	1.77_x	1.93_3	5.79_3	2.90_3	1.02_2	2.30_2	0.79_2	**Pentlandite**	$(Fe,Ni)_9S_8$	30– 657
3.02_8	1.77_7	5.99_6	1.21_x	1.03_8	1.02_8	1.51_6	1.34_6	**Xingzhongite**	$(Ir,Cu)S$	29– 551
3.01_x	1.77_x	3.53_8	2.50_8	1.92_8	1.30_8	1.25_8	2.04_6	**Daubreelite, syn**	$FeCr_2S_4$	4– 651
2.99_x	1.77_9	2.04_8	1.90_8	2.77_7	2.27_6	1.44_6	5.88_5	**Steigerite, chromian**	$(Al,Cr)VO_4\cdot3H_2O$	29– 20
3.01_x	1.76_x	1.92_3	5.76_3	2.88_3	1.02_2	2.29_7	0.79_2	**Cobalt pentlandite**	$(Co,Ni,Fe)_9S_8$	30– 444
3.01_x	1.76_x	1.92_8	5.75_6	2.88_6	1.02_6	2.29_5	1.30_5	**Cobalt pentlandite**	$(Co,Fe,Ni)_9S_8$	12– 723
3.01_x	1.50_8	1.74_7	1.84_6	1.78_6	1.91_5	2.80_4	1.57_4	**Euxenite, syn**	$YNbTiO_6$	14– 643
3.01_x	1.15_x	1.73_8	1.16_8	3.07_7	2.99_7	2.14_7	1.76_6	**Bowieite, syn**	Rh_2S_3	35– 736

2.99 – 2.95 (± .01)

2.98_8	28.0_x	14.1_8	2.70_8	4.85_6	4.44_5	5.27_4	5.60_3	**Tyrolite**	$CaCu_5(AsO_4)_2(CO_3)(OH)_4\cdot6H_2O$	11– 348
2.96_x	15.4_5	3.51_4	3.08_4	3.27_3	2.90_3	3.21_2	2.53_2	**Traskite**	$Ba_9Fe_2Ti_2Si_{12}O_{36}(OH)_6\cdot6H_2O$	18– 171
2.98_8	14.1_8	2.70_8	4.85_6	4.44_5	5.27_4	5.60_3	4.34_3	**Tyrolite**	$CaCu_5(AsO_4)_2(CO_3)(OH)_4\cdot6H_2O$	11– 348
2.98_8	12.1_x	6.96_6	3.43_6	2.78_6	6.16_4	4.06_4	4.55_2	**Fibroferrite**	$Fe(OH)SO_4\cdot5H_2O$	16– 935
2.99_x	12.0_x	3.54_6	5.95_5	2.89_5	4.73_4	3.73_4	3.28_4	**Tlapallite**	$H_6Ca_2Cu_3(SO_4)(TeO_3)_4TeO_6$	29– 319
2.94_8	11.7_x	2.45_8	4.43_7	3.54_7	2.69_6	2.56_6	3.25_5	**Agardite**	$Cu_6(Y,Ca)(AsO_4)_3(OH)_6\cdot3H_2O$	25– 183
2.94_x	11.4_x	2.65_x	4.50_8	3.80_6	2.48_6	1.64_6	1.58_6	**Raite**	$Na_4Mn_3Si_8(O,OH)_{24}\cdot9H_2O$	25–1318
2.98_9	10.2_x	3.44_8	3.27_8	2.73_7	3.86_5	3.75_5	2.83_5	**Chudobaite**	$(Mg,Zn)_5H_2(AsO_4)_4\cdot10H_2O$	12– 643
2.95_9	8.90_x	2.72_x	3.29_9	2.22_8	1.65_8	5.64_5	1.50_5	**Kolfanite**	$Ca_2Fe_3O_2(AsO_4)_3\cdot2H_2O$	35– 662
2.96_x	8.88_7	2.60_5	4.43_5	2.49_5	2.81_4	1.94_4	2.77_3	**Katoptrite**	$(Mn,Mg,Fe)_{13}(Al,Fe)_4Sb_2Si_2O_{28}$	19– 274
3.00_8	8.87_x	6.29_6	5.11_6	5.34_4	4.07_4	3.74_4	3.50_4	**Botryogen**	$MgFe(OH)(SO_4)_2\cdot7H_2O$	17– 157
2.94_9	8.81_x	3.33_7	3.16_7	2.86_7	10.9_6	4.74_6	4.47_6	**Ludlockite**	$(Fe,Pb)As_2O_6$	29– 774
2.94_x	8.68_x	3.09_x	6.09_8	5.82_8	2.50_8	2.31_8	3.02_6	**Barringtonite**	$MgCO_3\cdot2H_2O$	18– 768
3.00_x	8.40_x	5.20_6	4.26_6	2.13_6	4.20_4	3.30_4	3.21_4	**Francevillite, syn**	$Ba(UO_2)_2V_2O_8\cdot5H_2O$	21– 381
2.97_8	8.18_x	4.09_9	4.82_7	6.10_6	3.52_6	2.21_6	2.72_5	**Cuprosklodowskite**	$Cu(UO_2)_2(SiO_3OH)_2\cdot6H_2O$	8– 290

FIGURE 6. Hanawalt group for 3.04 – 3.00Å from the IPDF for minerals (this group contains 'key-in' data for calcite)



$CaCO_3$

Calcium Carbonate — Calcite

Rad. $CuK\alpha_1$ **λ** 1.54050 **Filter** **d-sp** Calculated
Cut off **Int.** Calculated **$I/I_{cor.}$**
Ref. Smith et al., Penn State University, University Park, Pennsylvania, USA., *JCPDS Grant-in-Aid Report*, (1973)

Sys. Rhombohedral (Hex) **S.G.** $R\bar{3}c$ (167)
a 4.990 **b** **c** 17.002 **A** **C** 3.4072
α **β** **γ** **Z** 6
Ref. Chesin et al., *Acta Crystallogr.*, **18** 689 (1965)

D_x 2.72 **D_m** **mp**

Calcite group, calcite subgroup. $F_{22} = 265.2(.0022,38)$. PSC: hR10.

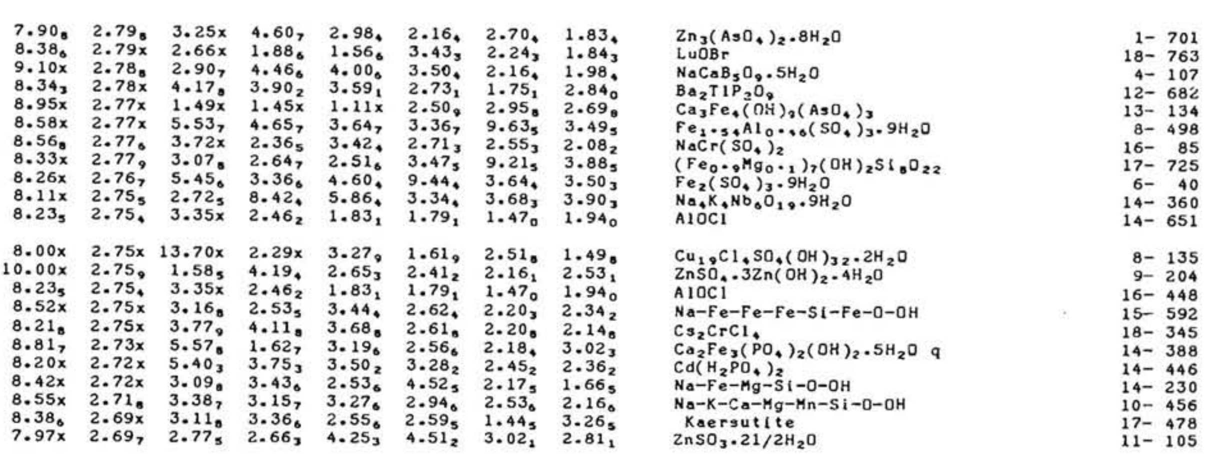
d Å	Int	hkl	d Å	Int	hkl
3.852	29	012			
3.030	100	104			
2.834	2	006			
2.495	7	110			
2.284	18	113			
2.094	27	202			
1.9261	4	024			
1.9071	17	018			
1.8726	34	116			
1.6259	2	211			
1.6040	15	122			
1.5821	2	$10\underline{10}$			
1.5247	3	214			
1.5061	2	119			
1.4405	5	300			
1.4168	3	$00\underline{12}$			
1.3361	3	$02\underline{10}$			
1.1779	3	$21\underline{10}$			
1.1536	3	134			
1.1417	3	226			
1.0471	2	404			
1.0440	2	318			

FIGURE 7. IPDF file card for calcite.

eight strongest lines in terms of groups based on the d-spacing of the most intense reflexion. There are 42 search groups (called Hanawalt groups) ranging from 50 to 1.0Å. In order to use the search index, the strongest line is taken and the appropriate search group consulted. In Figure 6, the group (Hanawalt group) for the strongest line corresponding to a d-spacing from 3.04 – 3.00Å is shown. The data is viewed in terms of attempting to match the next seven strongest lines with data in the table for the various minerals listed. There will always be errors associated with measurements, so *exact* correlations between experimental and tabulated data should not be expected. Very close correlation should, however, be observed when a likely candidate is found, and the file number of the particular mineral should be noted (for example, 24-27 for the mineral calcite). The second part of the IPDF consists of individual cards containing a more complete set of data for each mineral as shown for calcite in Figure 7. Experimental data should now be compared with all of the tabulated data for the mineral. If the two sets of data do correspond, then you have identified the unknown species with certainty.

A PRACTICAL EXAMPLE

Most of the foregoing discussion is a little dry, and it is suggested that a practical example might be useful in illustrating the procedure involved in identifying a particular unknown. In this case, a few small needles of a blue mineral from the Britannia Mine, North Wales, were hand-picked, freed of any adhering material,



7.90_8	2.79_8	3.25x	4.60_7	2.98_4	2.16_4	2.70_4	1.83_4	$Zn_3(AsO_4)_2 \cdot 8H_2O$	1- 701
8.38_6	2.79x	2.66x	1.88_6	1.56_6	3.43_3	2.24_3	1.84_3	LuOBr	18- 763
9.10x	2.78_8	2.90_7	4.46_6	4.00_6	3.50_4	2.16_4	1.98_4	$NaCaB_5O_9 \cdot 5H_2O$	4- 107
8.34_3	2.78x	4.17_8	3.90_2	3.59_1	2.73_1	1.75_1	2.84_0	$Ba_2TlP_2O_9$	12- 682
8.95x	2.77x	1.49x	1.45x	1.11x	2.50_9	2.95_8	2.69_8	$Ca_3Fe_4(OH)_9(AsO_4)_3$	13- 134
8.58x	2.77x	5.53_7	4.65_7	3.64_7	3.36_7	9.63_5	3.49_5	$Fe_{1.54}Al_{0.46}(SO_4)_3 \cdot 9H_2O$	8- 498
8.56_8	2.77_6	3.72x	2.36_5	3.42_4	2.71_3	2.55_3	2.08_2	$NaCr(SO_4)_2$	16- 85
8.33x	2.77_9	3.07_8	2.64_7	2.51_6	3.47_5	9.21_5	3.88_5	$(Fe_{0.9}Mg_{0.1})_7(OH)_2Si_8O_{22}$	17- 725
8.26x	2.76_7	5.45_6	3.36_6	4.60_4	9.44_4	3.64_4	3.50_3	$Fe_2(SO_4)_3 \cdot 9H_2O$	6- 40
8.11x	2.75_5	2.72_5	8.42_4	5.86_4	3.34_4	3.68_3	3.90_3	$Na_4K_4Nb_6O_{19} \cdot 9H_2O$	14- 360
8.23_5	2.75_4	3.35x	2.46_2	1.83_1	1.79_1	1.47_0	1.94_0	AlOCl	14- 651

8.00x	2.75x	13.70x	2.29x	3.27_9	1.61_9	2.51_8	1.49_8	$Cu_{19}Cl_4SO_4(OH)_{32} \cdot 2H_2O$	8- 135
10.00x	2.75_9	1.58_5	4.19_4	2.65_3	2.41_2	2.16_1	2.53_1	$ZnSO_4 \cdot 3Zn(OH)_2 \cdot 4H_2O$	9- 204
8.23_5	2.75_4	3.35x	2.46_2	1.83_1	1.79_1	1.47_0	1.94_0	AlOCl	16- 448
8.52x	2.75x	3.16_8	2.53_5	3.44_4	2.62_4	2.20_3	2.34_2	Na-Fe-Fe-Fe-Si-Fe-O-OH	15- 592
8.21_8	2.75x	3.77_9	4.11_8	3.68_8	2.61_8	2.20_8	2.14_8	Cs_2CrCl_4	18- 345
8.81_7	2.73x	5.57_8	1.62_7	3.19_6	2.56_6	2.18_4	3.02_3	$Ca_2Fe_3(PO_4)_2(OH)_2 \cdot 5H_2O$ q	14- 388
8.20x	2.72x	5.40_3	3.75_3	3.50_2	3.28_2	2.45_2	2.36_2	$Cd(H_2PO_4)_2$	14- 446
8.42x	2.72x	3.09_8	3.43_6	2.53_6	4.52_5	2.17_5	1.66_5	Na-Fe-Mg-Si-O-OH	14- 230
8.55x	2.71_8	3.38_7	3.15_7	3.27_6	2.94_6	2.53_6	2.16_6	Na-K-Ca-Mg-Mn-Si-O-OH Kaersutite	10- 456
8.38_6	2.69x	3.11_8	3.36_6	2.55_6	2.59_5	1.44_5	3.26_5		17- 478
7.97x	2.69_7	2.77_5	2.66_3	4.25_3	4.51_2	3.02_1	2.81_1	$ZnSO_3 \cdot 21/2H_2O$	11- 105

FIGURE 8. IPDF Hanawalt data used to identify connellite.

TABLE I

Powder X-ray diffraction data for connellite.

Observed data[a]		IPDF data[b]	
d (Ångströms)	Intensity	d (Ångströms)	Intensity
13.6	ss	13.70	100
8.00	ss	8.00	100+
		6.90	10
5.53	m	5.51	60
		5.20	70
		4.59	60
		4.35	60
		3.98	20
3.80	s	3.82	60
3.42	m	3.48	60
3.24	s	3.27	90
3.07	ww	2.96	20
		2.85	20
2.77	ss	2.75	100−
2.65	m	2.62	70
		2.59	60
2.50	ms	2.51	80
2.46	ww	2.46	10
2.38	w	2.38	40
2.24	ss	2.29	100
		2.25	50
2.173	w	2.19	50
2.092	w	2.09	40
2.051	ww	2.04	40
1.982	w	1.97	50
1.914	ww	1.91	20
1.860	ww	1.855	20
1.810	m	1.811	50
		1.799	50
		1.754	70
		1.725	10
		1.673	20
		1.644	10
1.613	s	1.613	90
		1.580	60
		1.546	40
1.521	w	1.520	20
		+ 9 lines to 1.313 Å	

[a] Data from Pollard *et al.* (1989); s = strong, m = medium, w = weak.

[b] Card 8-135, data taken from Bannister *et al.* (1950).

crushed and ground and packed in a Lindemann tube. A powder photograph was recorded using a Debye-Scherrer camera with a narrow-bore beam stop (to record reflexions with low θ). Measurements of 4θ were abstracted from the developed film using a Vernier measuring device. Using the relations given above, a list of d-spacings was calculated, and these are detailed in Table I. The radiation used was CuKα (λ = 1.5418Å). Estimates of intensity were also made in simple terms, and these, too, are listed in the Table. A d-spacing of 8.00Å was seen to correspond to the strongest reflexion.

Reference to the IPDF was made, the appropriate section of which is shown in Figure 8. Of the various possibilities, only that of connellite fitted the data in terms of the strongest lines (experimental: 13.5, 8.00, 2.77, 2.24 (all very strong), 3.80, 3.24, 1.61 (strong), 5.53, 3.42, 2.65, 2.50, 1.81 (medium or medium to strong)Å; tabulated data: 13.7, 8.00, 2.75, 2.29 (all very strong), 3.27(9), 1.61(9), 2.51(8) and 1.49(8)Å, with estimated intensities in parentheses). Next, the individual file card (8-135) for connellite was consulted. Tabulated data from the file are also listed in Table I. It is seen that there is a very good match for the reflexions listed, and those which were recorded experimentally. We could be certain that this sample was connellite. Subsequent investigations using other techniques bore this result out, and the material was shown to contain small amounts of carbonate and nitrate substituting for sulfate in the lattice (Pollard *et al.*, 1989).

SOME FINAL REMARKS

It is hoped that this article has been helpful in outlining the use of X-ray powder diffraction for the identification of minerals. As mentioned above, other camera types are available, but that fact need not bother us here. In fact, the measurement can be automated by the use of a *diffractometer*, which detects reflexions and measures their position and intensity using a Geiger-Müller tube or its equivalent.

Unless a sample of a new mineral is chosen, if it is crystalline, the above procedure will serve to identify it. There are, however, a few complications that should be mentioned. First, the *habit* of certain crystals, or their cleavage characteristics, may serve to give particles of preferred shapes (or *orientations*) upon grinding. This sometimes causes difficulty in measuring certain reflexions, or gives undue weight (intensity) to others. Secondly, a mineral species may be isomorphous with another (same structure and unit cell) but there may be only slight differences in the size of the unit cell. Alternatively, two related species might be isostructural (same strucure) but with very subtle differences in detail (different space group). It can be sometimes difficult to separate species in these cases on the basis of X-ray diffraction measurements alone. An example might be that of the pyromorphite-mimetite series (end members isostructural not isomorphous, but with very slight structural differences between the two). Minerals exhibiting complex solid-solution can also prove difficult to distinguish. In these cases, simple (sometimes otherwise!) chemical tests are essential adjuncts to the X-ray diffraction measurements, as can be examination using microscopical techniques of various kinds. These cases are outside the scope of this article, although other articles covering these aspects of mineralogy are planned for subsequent issues.

It is finally worth observing that measurements of X-ray powder diffraction data have proved to be the single most important technique used to discover new

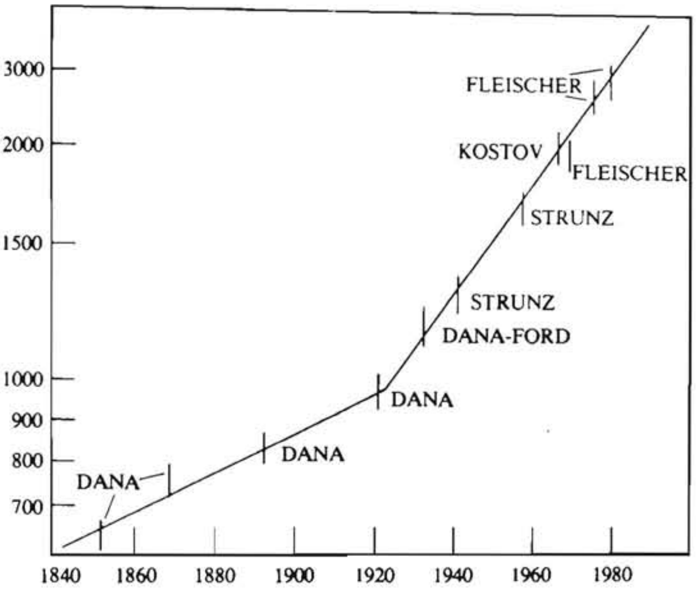


FIGURE 9. Estimated change in numbers of new mineral species from Skinner and Skinner (1980). Rate of increase rises sharply soon after the introduction of X-ray diffraction techniques (reproduced by courtesy of the Mineralogical Record).

minerals. Approximately 3500 species are now known, and this number is increasing at a rate of about 60-70 per annum, from all over the world. The small sample size necessary for X-ray powder diffraction lends itself ideally to the characterization of minerals when only small quantities of material are available. The effect of the technique in terms of the rate that new minerals have been discovered is readily appreciated from Figure 9 which estimates the number of new species described since 1840 (Skinner and Skinner, 1980).

ACKNOWLEDGEMENTS

We wish to thank Mr C. Chaplin for assistance in preparation of the figures. Grateful thanks are due to JCPDS (International Centre for Diffraction Data and publishers of the Powder Diffraction File, 1601 Park Lane, Swarthmore, Penn., U.S.A.) for permission to reproduce published X-ray data. We wish to thank the Mineralogical Record for permission to publish Figure 9.

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CHROMIAN MINERALS IN MICRODIORITE FROM THE ENGLISH LAKE DISTRICT

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Accessory minerals in an augite-phyric microdiorite from Scawgill Bridge include an unnamed Cr-Ti-rich oxide mineral and Fe-rich, Mg-poor chromite. The former underwent an early stage of Zn and V enrichment with probable Mg loss, and both minerals suffered Cr, Fe, Zn and V loss with Si and K enrichment during pervasive hydrothermal alteration of the intrusion. Despite alteration, the Cr-Ti-rich mineral retains areas interpreted as reflecting an original composition comparable with material previously described from kimberlite and lunar soils, and is interpreted as early-formed phenocrysts preserved by rapid emplacement and enclosure within the margins of augite phenocrysts.

INTRODUCTION

Chromite and an unnamed Cr-Ti-rich oxide mineral have been observed as accessories, together with titanite, rutile and Co-Ni-As sulfides, in a specimen of augite-rich, porphyritic, granophyric microdiorite from Scawgill Bridge [BGS specimen number LC226; NGR NY 1755 2577] near the Winlatter Pass in the Lake District (Fig.1). Chromite is rarely reported from the Lake District. It was recorded in segregations in leucogabbro of the Carrock Fell Complex (Eastwood *et al.*, 1968; Young, 1987), but recent re-examination of the specimen [BGS collection no E15248] has indicated only magnetite (a qualitative X-ray fluorescence analysis indicated no significant Cr: D.J. Bland, personal communication, 1989). The present occurrence may be the first confirmed reporting of chromite from Lake District igneous rocks.

The Cr-Ti-rich mineral at Scawgill Bridge is of additional interest because of its unusual composition, similar to minerals described from kimberlite and lunar rock (see below). Few polished sections of rocks from the Scawgill Bridge and related intrusions are available, and the true distribution of the minerals in the Lake District is not known. The present paper sets out the

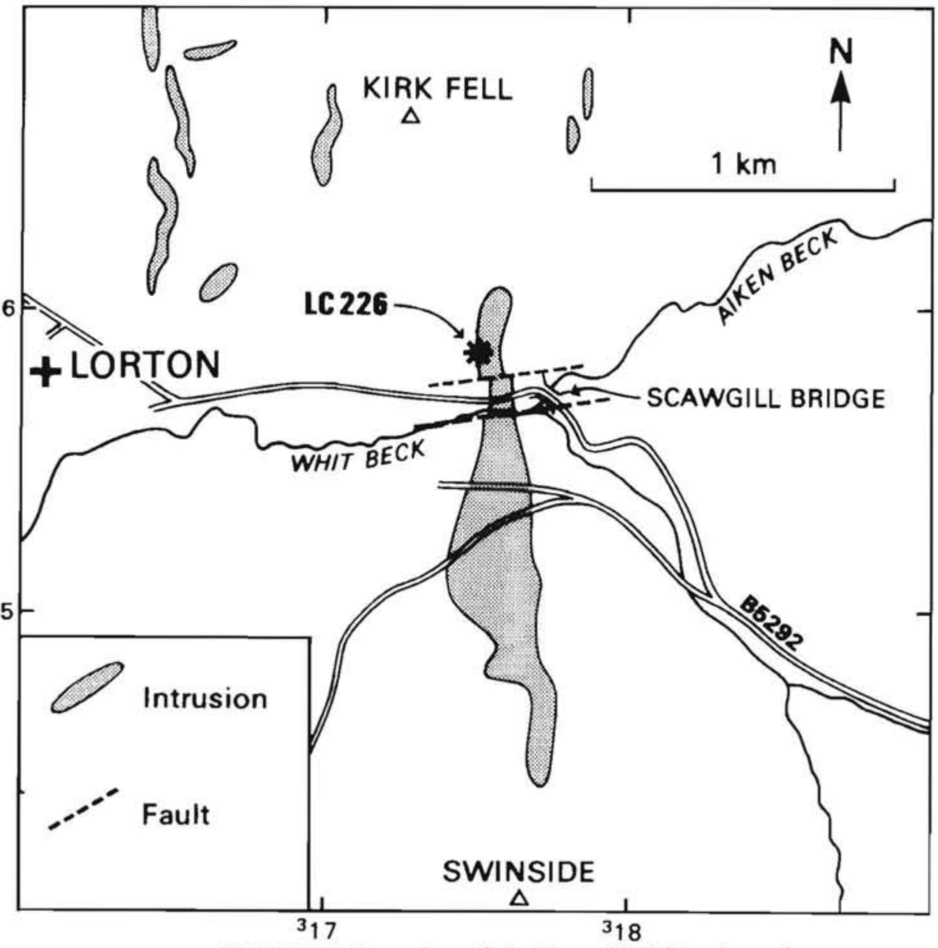

FIGURE 1. Location of the Scawgill Bridge intrusion.

currently available results with particular reference to the chemistry and genesis of the Cr-Ti-rich mineral.

GEOLOGICAL SETTING

The igneous province of the English Lake District comprises two major series of basic to acid extrusive rocks and related sediments, gabbroic to granitic plutons, and several sets of dykes and other minor intrusions (Firman, 1978; Millward *et al.*, in press). Most of the rocks are transitional tholeiitic to thoroughly calc-alkaline (Fitton and Hughes, 1970), and the province results from late-Ordovician arc volcanism followed by plutonism and intrusive activity related to continental collision and the final closure of the Iapetus oceanic basin (Moseley, 1978; Hutton, 1987). No major ultrabasic bodies are present at outcrop, though minor intrusions emplaced into the Skiddaw Group contain rocks whose silica content is below 48 % (T.C. Colman, personal communication, 1988) including the appinitic 'picrites' at Barkbeth and Dash in the Bassenthwaite district which are variably altered augite-hornblendites with olivine and/or orthopyroxene (described by Eastwood *et al.*, in 1978 as 'cortlandtite').

The Scawgill Bridge intrusion, a very poorly exposed, N-S oriented dyke-like body recently mapped by Dr. A.H. Cooper (unpublished BGS geological data), is one of an appinitic suite of porphyritic microdiorite-spessartite minor intrusions which crop out in the Skiddaw Group slates which underlie the thick Lake District sequences of extrusive volcanic rocks, and which includes the Barkbeth and Dash intrusions. Despite pervasive deuteric and hydrothermal alteration, considerable primary modal and textural variation is evident in the Scawgill Bridge intrusion and more generally among the members of this suite. The intrusions collectively display a calc-alkaline chemistry similar to the Borrowdale Volcanic Group but more primitive in that Zr and Ti are consistently lower while Mg and Cr attain high values (*e.g.* Cr > 1000 ppm in some samples). Their variability relates to patchy concentrations of augite phenocrysts which formed in deep level magma-chambers and were transported up to the final site of consolidation with, in some cases, little disruption.

PETROGRAPHY

The petrography of the intrusions can be considered in terms of three primary components: augite phenocrysts accompanied by minor olivine and accessory Cr minerals; ilmenite-bearing hornblende-microdioritic groundmass with minor biotite; an interstitial micro-granophyric component which uncommonly forms the groundmass hosting concentrations of augite phenocrysts. Olivine occurs as rounded, completely altered grains enclosed within augite crystals. The Scawgill Bridge intrusion shows considerable variation among the proportions of these components, although melanocratic augite-phyric microdiorite (spessartite) is the major lithology. Sericite, chlorite and carbonate, with titanite and epidote, form a widespread alteration mineral assemblage, but several specimens reveal an earlier deuteric alteration marked by saussuritised plagioclase, green hornblende and actinolite.

The single specimen currently known to contain chromite originally consisted of hornblende-rimmed augite phenocrysts, up to 1 cm in width, set in microdioritic groundmass in which small clusters of biotite flakes are associated with pockets of micro-granophyric material. The rock has undergone severe pervasive carbonate-sericite-chlorite alteration, although the darkened, iron-stained biotite crystals have remained conspicuous. A small number of grains of the Cr-Ti mineral up to 200 microns wide were located during electron-probe microanalysis (EPMA) of minerals in the rock, and were found subsequently to be accompanied by smaller grains of chromite and granules (10-20 microns in size) of sulfide minerals. The Cr-Ti mineral grains are situated in narrow trails within highly altered material apparently in the margin of an augite phenocryst. The chromite grains which have been observed are less frequent, but occur in the same general area.

Sulfide granules include pyrite, chalcopyrite and Co-Ni-As-S minerals. Definitive data on the latter are not yet available, but qualitative analysis during SEM investigation indicated the presence of a Ni-As-S phase (possibly gersdorffite) with minor Fe, Co and Ag, and a Co-As-S phase with subordinate Fe and Ni (possibly cobaltite, glaucodot or villamaninite). They form granules spatially associated with the Cr-minerals though probably formed during the alteration. Titanite and rutile, apparently formed by alteration of ilmenite, occur in close association with the relict biotite flakes.

MINERALOGY

The grains of the Cr-Ti-rich mineral are opaque although of very low reflectance. They display internal reflections from weak, dark brown to strong, pale yellow to white, corresponding with variation from high to relatively low density seen in backscattered electron images described below. Their rounded, anhedral, internally fissured forms suggest partial dissolution of grains originally formed early in the history of the rock. The minute chromite grains are also anhedral, indicating a degree of dissolution.

The EPMA data (Tables I and II) indicate wide compositional variation within individual grains of the Cr-Ti mineral, confirmed by scanning electron-microscope backscattered electron images (BSE) which show (Figures 2 and 3) micro-scale inhomogeneity throughout the grains and extending down to the effective limit of spatial resolution (*ca* 2 microns) of the technique (Fig. 3b). Changes in brightness of the BSE images result from changes in the mean atomic number (approximately the density). The pattern observed is interpreted as pervasive alteration imposed upon

TABLE I

EPMA analyses[a] of individual spots (*ca* 4 microns in diameter) on polished sections through grains of the Cr-Ti-rich mineral in specimen LC226.

	1	4	5	6	7	8	9
SiO_2	2.54	4.02	1.30	17.25	20.73	56.86	19.77
TiO_2	12.39	11.82	3.51	19.29	18.62	40.14	75.49
Al_2O_3	12.65	13.24	16.62	15.31	14.63	0.80	2.03
V_2O_3	1.26	1.33	0.59	0.78	0.89	nd[c]	nd
Cr_2O_3	33.34	34.30	35.44	17.84	14.10	nd	nd
FeO[b]	24.83	23.79	29.44	12.45	12.82	nd	1.76
MnO	nd	nd	nd	nd	nd	nd	nd
MgO	0.72	0.59	nd	2.75	4.77	nd	1.63
ZnO	5.85	5.50	6.01	2.24	1.62	nd	nd
K_2O	0.47	0.31	0.40	3.19	2.43	nd	nd
Total	94.05	94.90	93.31	91.10	90.61	97.80	100.68

[a] EPMA data in this paper were obtained using a Cambridge Microscan V instrument with LINK EDA attachments operating at 15 kV. Samples 1,4,5: dense areas detected by SEM-BSE imaging; 6 to 9: lower density areas of incipient alteration.

[b] Total iron as Fe(II).

[c] Not detected.

TABLE II

EPMA analyses of chromite (1-3) and altered chromite (4 and 5) in LC226, the former recalculated to give formulae to four oxygens, adjusting the iron oxidation-ratio to give three total cations.

	Weight-percentage results						Chromite formulae		
	1	2	3	4	5		1	2	3
SiO_2	0.50	0.34	0.48	42.73	26.04	Si	0.022	0.014	0.021
TiO_2	0.83	0.91	0.77	14.20	16.61	Ti	0.027	0.029	0.025
Al_2O_3	11.26	11.97	10.35	6.30	8.61	Al	0.572	0.595	0.530
V_2O_3	nd	nd	nd	0.87	0.96	Cr	0.718	0.775	0.810
Cr_2O_3	42.20	46.52	47.20	19.62	23.82	Fe^{3+}	0.613	0.543	0.568
FeO[a]	39.02	30.38	34.74	13.18	17.95	Fe^{2+}	0.792	0.528	0.694
MnO	0.87	0.67	nd	nd	nd	Mn	0.032	0.024	
MgO	3.50	7.81	5.44	0.28	0.46	Mg	0.225	0.491	0.352
ZnO	nd	nd	nd	2.99	3.35	Cr:Al[b]	55.7	56.6	60.4
K_2O	nd	nd	nd	0.22	0.34				
Total	98.18	98.60	98.98	100.39	98.14	Fe:CrAl[c]	32.2	28.4	29.8
						Mg:Fe[d]	22.1	48.1	33.7

[a]Total iron as Fe(II). [b]Cr/(Cr + Al). [c]Fe(III)/(Fe(III) + Cr + Al). [d]Mg/(Mg + Fe(II)).

already fractured and partially dissolved anhedral grains (Figs 2 and 3). The most dense areas indicated by the brightest parts of the BSE images (*e.g.* Fig. 2b) show consistency of composition with major Cr, Ti, Fe, Al and Zn accompanied by V, Si and K. The Cr-Ti mineral contains more Al and V than the true chromite, but less Fe and very little Mg. It is also rich in Zn, suggesting that this element rather than Mg occurs in divalent sites. Si and K occur at significant concentrations in the Cr-Ti mineral, but are near or below the detection limit in the true chromite.

Variation diagrams of the EPMA data such as Fig. 4 show the pattern of alteration. Fe and Zn decrease progressively as Cr decreases, maintaining approximately constant Fe/Cr and Zn/Cr ratios (Fig. 4a). Other elements indicate a more complex, two-stage process. Increasing Si with decreasing Cr is initially accompanied by increases in Ti (Fig. 4b) and Al, but beyond a certain stage no further Ti or Al increase is seen, and the diverse SiO_2- and TiO_2-rich compositions in the lowest density parts of the grains (*e.g.* Table I, analyses 8 and 9) suggests alteration to micron-scale intergrowths of quartz, TiO_2 polymorph and possibly other minerals.

The true chromite grains show a similar alteration towards Si- and Ti-rich compositions (Table II) but so

Fig. 2a

Fig. 2b

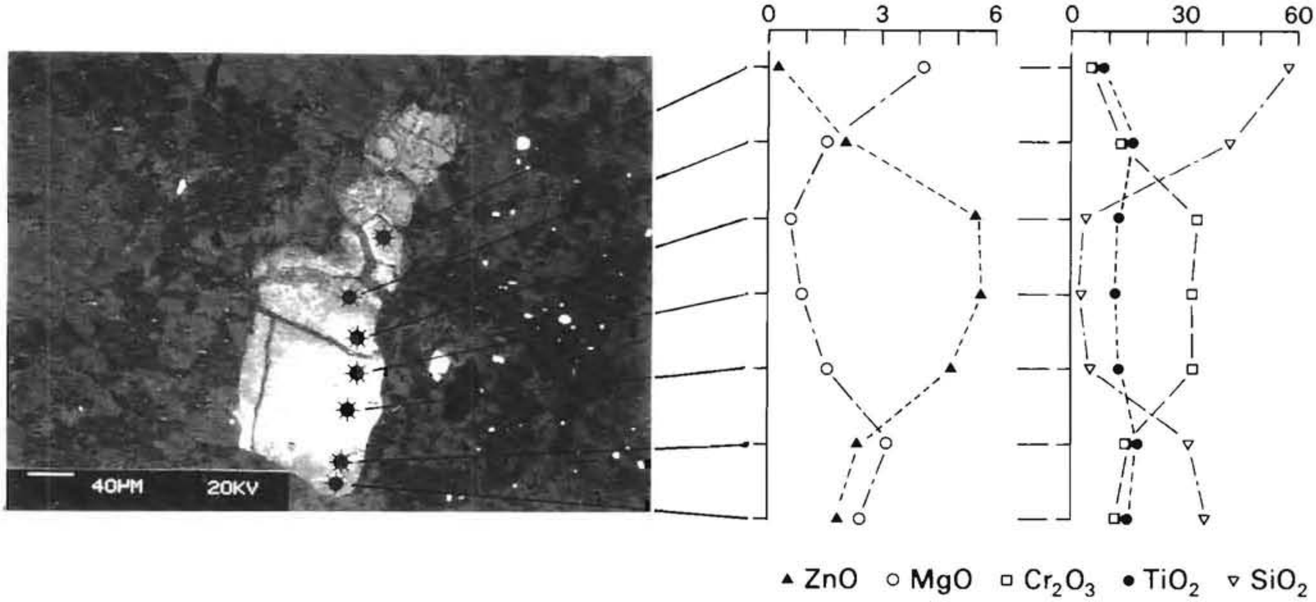

FIGURE 2 (a): SEM-BSE micrograph showing a Cr-Ti-rich grain with a relict area of high density Cr-Fe-Ti-Al-Zn-rich material; (b): variation in Zn, Mg, Cr, Ti and Si determined by EPMA spot analyses.

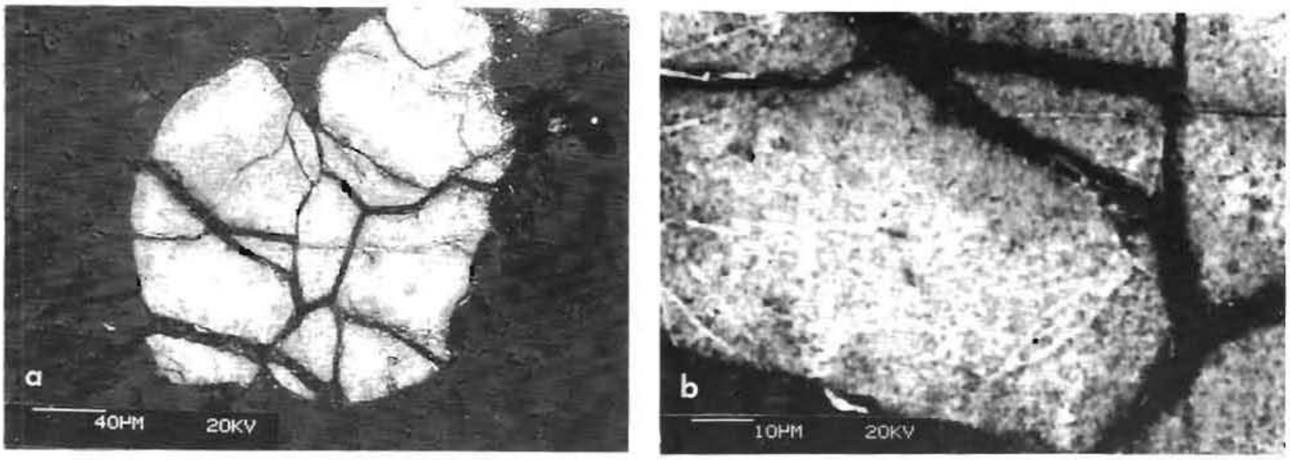

FIGURE 3 (a): SEM-BSE micrograph showing pervasive alteration of a Cr-Ti-rich grain; (b): shows part of this grain at higher magnification to demonstrate that compositional inhomogeneity extends to the limit of resolution.

far no intermediate zones of the Cr-Ti-rich composition have been observed.

DISCUSSION

The chromite analyses were recalculated to atomic proportions by constraining the total cations to exactly 3 or 4 oxygens (Table III). Although this gives only an approximation of the ferrous-ferric ratio, the results correspond closely with chromite stoichiometry. The chromite is Fe- and Ti-rich while poor in Al and Mg when compared with most chromites (see Dick and Bullen, 1984). This may reflect crystallization at a relatively high oxygen fugacity and temperature (Hill and Roedder, 1974). The composition is however comparable with chromite from Mount St Helens basalt (Dick and Bullen, 1984) and ultramafic intrusions in SE Alaska (Irvine, 1967) which Murray (1972) recognised as roots of andesitic volcanic centres. Chromites from the Muskox Intrusion and from the Merensky Reef of the Bushveld Complex also show similar low-Mg and high-Fe_2O_3 characteristics, although the iron-enrichment is less extreme (Irvine, 1967). The compositions of chromite grains in sample LC226 from Scawgill Bridge could thus be consistent with derivation of the intrusion by tapping a fractionating basaltic magma-chamber possibly related to the calc-alkaline Borrowdale extrusive volcanic

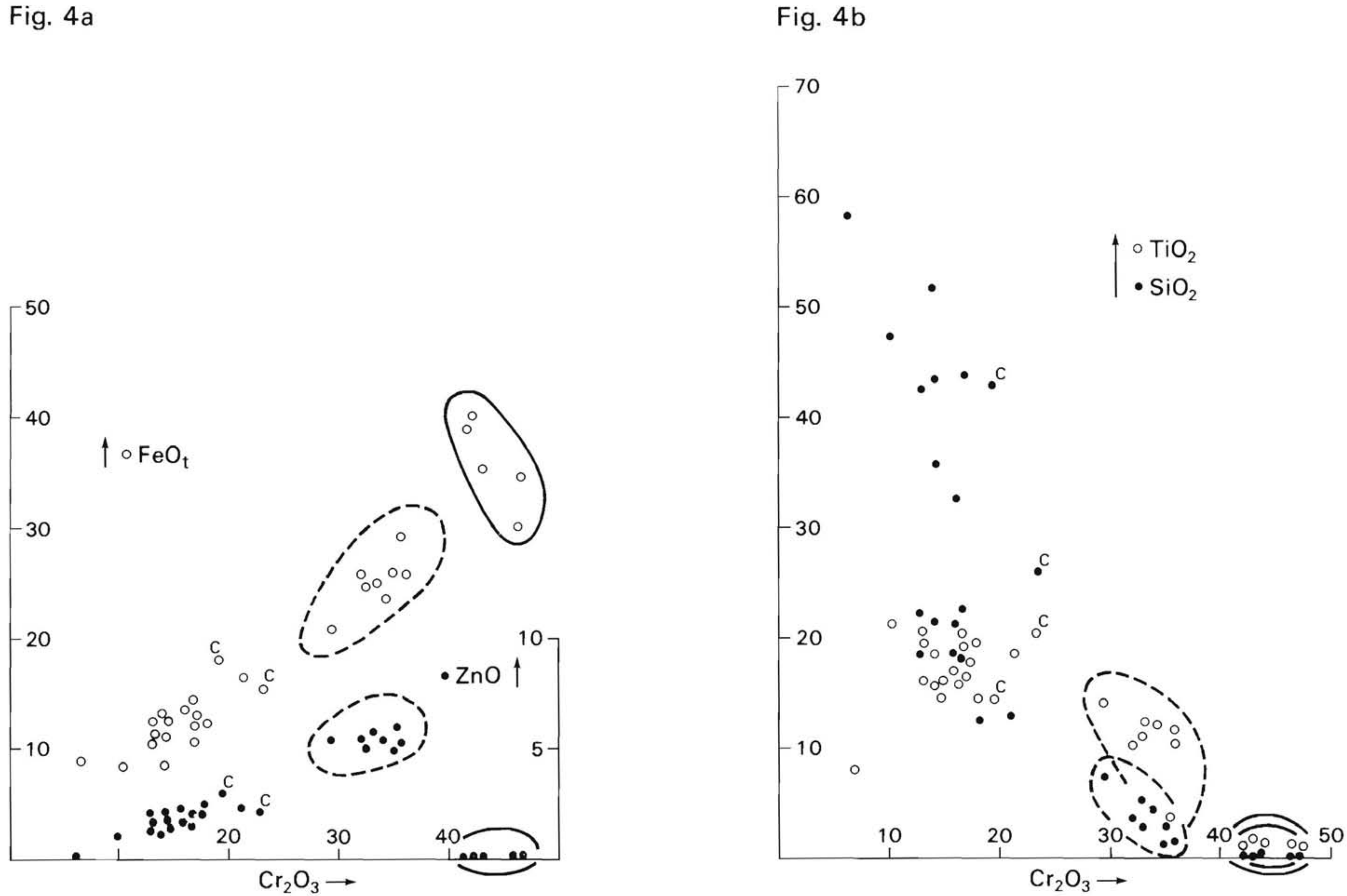

FIGURE 4 (a): Variation of Fe(total) (open circles, left hand scale) and ZnO (closed circles, right hand scale) with Cr_2O_3, and (b): of TiO_2 (open circles) and SiO_2 (closed circles) with Cr_2O_3 in Cr-Ti-rich and chromite grains (EPMA data). Ringed area: chromite; c: altered chromite; dashed ring: Cr-rich parts of Cr-Ti grains; other points: altered

rocks of the area. It is interesting to note that chromite has recently been reported from the petrographically comparable Port-na-Blagh appinite in northern Donegal (Elsdon and Todd, 1989) and is probably more widespread in such intrusions.

The Cr-Ti-rich grains are chemically distinct from the true chromite grains. Although their variable compositions can be attributed to post-consolidation alteration, the high Ti, V and Zn contents of the most Cr-rich parts suggests incorporation of these elements at an earlier stage. Danchin and d'Orey (1972) reported titanium-rich chromite intergrown with ilmenite in kimberlite from the Premier Diamond Mine, Pretoria, following an earlier report of titanian chromite from kimberlite in Lesotho (Nixon *et al.*, 1963). Chromite of similar composition was reported from Luna 20 soil (Haggerty, 1973; Brett *et al.*, 1973) as discrete grains and as lamellae in ilmenite. Chromian-ulvospinel was also reported from Luna 20 as well as from Apollo 11 samples (Haggerty, 1971; Haggerty and Meyer, 1971). However, none of these authors record measureable Zn, and only Brett *et al.* (1973) record V_2O_3 (up to 0.84 wt%). Weiser (1967) and Bevan and Mallinson (1980) report zincian chromite from Outokumpu, Finland and Mashaba Mine, Zimbabwe respectively, both concluding that Zn had diffused into originally non-enriched chromite during metasomatic or hydrothermal reactions.

Derivation of the high Cr content of the Cr-Ti-rich mineral by metasomatic alteration of an original Timineral seems unlikley in view of the extremely fine-scale intergrowth of Cr and Ti components required and the excess of Cr over Ti in the high density areas. Metasomatic Ti-enrichment is also unlikley in view of the very low mobility of this element in most metasomatic processes. We therefore conclude that the high Cr-Ti character is an original, magmatic feature of the mineral, which is interpreted as altered phenocrysts formed at deep, possibly sub-crustal levels. The original phase may have unmixed as the magma reached shallower levels to give intimate chromite-ilmenite or chromite-rutile intergrowths, and Zn may have been exchanged for Mg during metasomatism at this or some other stage in the ascent of the magma. The presence of Si and K even in the most Cr-rich parts of the grains suggests a third minor component, possibly feldspar or sericite formed during the post-emplacement alteration of feldspar.

On the premise that the dense Cr-Ti-rich areas reflect an original composition, the weight-percentage analyses were recalculated in an attempt to derive a realistic original mineral formula. Since neither the appropriate number of oxygen equivalents nor the Fe oxidation ratio is known, this is an arbitrary process which can give no unique result, although it is desirable that whole numbers of total cations and of individual cations or subgroups of cations should be achieved. The recalculations were made using a BBC-BASIC program for mineral formulae calculations modified to allow the iron oxidation ratio, (Fe(III)/Fe(total), to be adjusted to achieve a preset number of cations for the number of oxygen atom equivalents. If the calculated oxidation ratio falls outside the range 0 to 1 then the number of cations is unacceptable and the program is re-run with different numbers of cations (or of oxygen equivalents) until a plausible though probably not unique solution is reached.

Table III gives the recalculated results for five analyses of the Cr-, Ti- and Zn- rich material in three grains (see Table I). Four of the recalculations gave

TABLE III

Possible atomic formulae calulated for analyses of high Cr-Ti parts of mineral grains in specimen LC226. Note that the results represent only one of many possible recalculations of the weight percent results and are not necessarily true mineral formulae.

	1	2	3	4	5
Si	0.303	0.197	0.322	0.475	0.174
Ti	1.111	0.987	0.954	1.050	0.353
Al	1.777	1.835	1.901	1.843	2.620
V	0.050	0.053	0.057	0.052	0.026
Cr	1.571	1.734	1.644	1.601	1.873
Fe(III)	1.847	2.071	1.884	1.502	0.495
Fe(II)	0.627	0.584	0.653	0.847	2.797
Mg	0.128		0.119	0.104	
Zn	0.515	0.478	0.426	0.480	0.593
K	0.071	0.061	0.039	0.047	0.068
Total cations[a]	8.0	8.0	8.0	8.0	9.0
Fe(III)/ Fe(total)	0.75	0.78	0.74	0.64	0.15

[a] Calculated on the basis of 12 oxygen atoms.

solutions for 12 oxygen equivalents, with 8 total cations, 1 Ti atom and a value between 0.6 and 0.8 for Fe(III)/Fe(total). Summation of subgroups of cations in one of these (analysis 4) suggests a possible formula, $(Al,V,Cr,Fe^{3+})_{5.0}(Fe^{2+},Mg,Zn,Si,K)_{1.95}Ti_{1.1}O_{12}.(H_2O)_x$ but the other three are less satisfactory, having up to 5.6 trivalent atoms and as little as 1.3 divalent atoms plus Si and K. The remaining analysis, with less Ti and more Al than the others, gave 9 cations to 12 oxygen equivalents, and an oxidation ratio of 0.15. Summation of cation subgroups suggests a formula, $(Al,V,Cr,Fe^{3+})_{5.0}$ $(Ti,Fe^{2+},Zn,Si,K)_{4.0}O_{12}.(H_2O)_y$. In each case, the low weight percent total is tentatively ascribed to uncertain numbers of water molecules (x and y above). The last example resembles spinel in its total number of cations, but has excess divalent to trivalent ions. Conversion of Fe(II) to Fe(III) will reduce the total number of cations (to maintain the total charge), but this could be compensated for by the need to use some of the oxygen with the Si and K in a proposed sericite component. It is therefore possible that this analysis approaches an original chromite phenocryst composition. The other analyses, with about 1 Ti atom to 12 oxygens are more difficult to interpret in this way, possibly because Ti was present both as ilmenite and as a constituent of the chromite in original composite phenocrysts, and because of the higher Si-K component of the present material. Given the degree of alteration, we are unable to be more precise regarding the composition of the original material.

CONCLUSIONS

EPMA and SEM examination of opaque grains in sample LC226 from the Scawgill Bridge intrusion shows that chromite and an unnamed zincian Cr-Ti-rich mineral occur, both in a state of alteration involving Cr, Fe and Zn loss with development of variable Si-Ti- (and Mg-) -enriched compositions. The chromite is interpreted as having formed during fractionation of the parental basaltic magma in a deep-level chamber. The Cr-Ti- rich mineral may have originally been precipitated as titanian chromite in an even deeper magma-chamber within the deep crust or upper mantle and have unmixed into an intimate chromite-ilmenite intergrowth too fine to resolve by the backscattered detector. Zn and V enrichment took place during early alteration of the Cr-Ti- rich mineral, possibly concurrently with its partial dissolution, but has not been observed in the true chromite. The preservation of this material attests to the deep origin and rapid emplacement of the Scawgill Bridge intrusion, in keeping with its mineralogical character and textural inhomogeneity. It can be anticipated that such minerals will be more widespread in the appinitic augite-phyric hornblende microdiorite intrusions of the Skiddaw Group.

ACKNOWLEDGEMENTS

The writers are indebted to A.H. Cooper for field data and samples, and to T.C. Colman for access to whole rock analyses. Rob Ixer is thanked for his valuable criticisms of the manuscript. This paper is published by permission of the Director of the British Geological Survey (Natural Environment Research Council).

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SCHULENBERGITE AND NAMUWITE FROM SMALLCLEUGH MINE, NENTHEAD, CUMBRIA

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During the nineteenth and early twentieth centuries the now abandoned Smallcleugh Mine, at Nenthead in Cumbria, worked lead and zinc ore from veins and replacement flats in the Great Limestone of Namurian (Carboniferous) age. Post-mining groundwater has oxidised primary minerals, leading to the formation of gypsum, which in places encrusts the mine walls accompanied by various iron oxides, epsomite (Dunham, 1948) and rare serpierite (Bridges, 1987). Locally these are associated with the secondary copper-zinc basic sulfates schulenbergite, $(Cu,Zn)_7(SO_4,CO_3)_2(OH)_{10}.3H_2O$, and the recently described species namuwite, $(Zn,Cu)_4(SO_4)(OH)_6.4H_2O$. Both minerals have been identified by X-ray powder diffraction on specimens collected from the Middlecleugh First Sun Vein. Representative specimens have been deposited in the mineral collections of the Royal Museum of Scotland (reg. no. RMS 1985.42.2) and the National Museum of Wales (reg. no. NMW 88.43G.M1).

Schulenbergite was first described from dumps of the Glucksrad Mine, near Oberschulenberg, in the Harz Mountains, Germany (Hodenberg *et al.*, 1984). The mineral was also found at a further five localities by these authors, associated with serpierite, linarite, brochantite, cerussite, smithsonite, posnjakite and namuwite. In Britain, schulenbergite has been identified at the Dyfngwm and Eaglebrook Mines, in central Wales (S. Rust, personal communication), although details have not been published. At Smallcleugh schulenbergite is sparsely present as pearly, blue-green trigonal crystals which exhibit either sharp or rounded corners, together with a perfect basal cleavage.

Namuwite was first descibed as a new mineral by Bevins *et al.* (1982) from a specimen in the Mineral Collection of the National Museum of Wales from Aberllyn Mine, Llanrwst, Gwynedd. On this specimen, labelled as 'hydrozincite coloured by malachite', namuwite forms pale sea-green aggregates or stacks of perfect to sub-perfect hexagonal plates up to 60 microns across. It occurs with hydrozincite on a breccia composed of black, pyritic slate cemented by sphalerite, calcite, ankerite and quartz. Subsequently namuwite has been found more widely in Europe, including Min Trentini, Torrebelvicino, Vincenza, Italy (Bertoldi *et al.*, 1984), Bastenberg, Ramsbeck, Nordrhein-Westfalen, Federal Rebublic of Germany (on National Museum of Wales specimens NMW 84.3G.M2 and NMW 86.30G.M2 respectively) and Laurion, Greece (Schnorrer-Köhler *et al.*, 1988). In Britain it has been found as small hexagonal crystals on the dumps of Waterbank Mine, Ecton Hill, Staffordshire and in Wales from the Dylife and Dyfngwm mines (S. Rust, personal communication). This note records the first *in situ* British occurrence of the mineral. At Smallcleugh Mine pearly sea-green namuwite forms flat, triangular plates up to 200 microns across. Only rarely do the crystals show development of the full hexagonal plates seen on the type specimen. The plates are commonly curved, and are typically grouped together as sheaves (Figure 1).

The serpierite is present as turquoise to copper-blue I

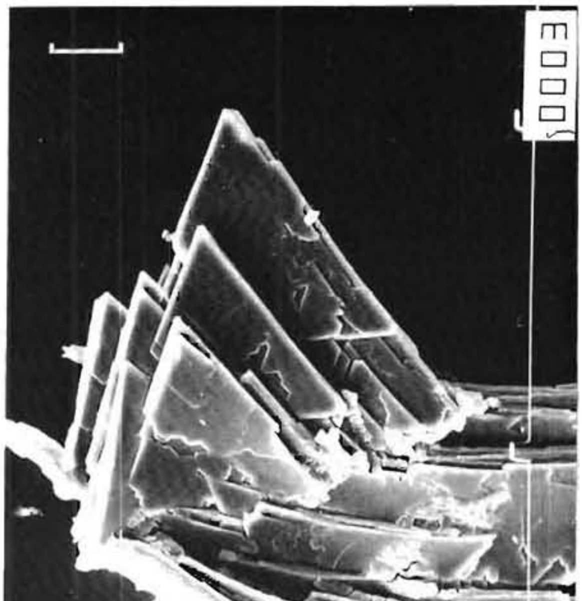

FIGURE 1. Scanning electron photomicrograph of namuwite sheaf from Smallcleugh Mine. Scale bar (20μ) indicates size. From specimen NMW 88.43G.M1.

TABLE I

Electron microprobe analyses of schulenbergite (1-4) and namuwite (5-7) from Smallcleugh Mine, Nenthead. Analyses from specimen RMS 1985.42.2.*

	1	2	3	4	Av.1-4	5	6	7	Av.5-7
CuO	34.34	38.23	34.80	35.74	35.77	24.23	24.03	22.18	23.48
ZnO	25.29	27.76	24.80	24.04	25.47	33.35	33.62	29.88	32.28
SO_3	16.44	17.07	16.05	17.03	16.65	13.89	14.27	13.02	13.73
Total	76.07	83.06	75.65	76.81	77.89	71.47	71.92	65.08	69.49
Cu:Zn	1.38	1.41	1.43	1.52	1.45	0.74	0.73	0.76	0.74
Cu + Zn:S	3.62	3.85	3.70	3.49	3.66	4.12	4.02	3.98	4.03

*Each analysis represents a single spot per grain

mm long bundles comprising wispy or stellate acicular groups. Bridges (1987) reported the presence of devilline in association with the Smallcleugh serpierite, although none has been found during this investigation.

Electron microprobe analyses of schulenbergite and namuwite are presented in Table I. Apart from S, Cu and Zn no other elements of any significance greater than atomic number 8 were detected. The average schulenbergite analysis yields the following empirical formula on the basis of 13 oxygens in the anhydrous component $(Cu_{4.12},Zn_{2.87})_{6.99}(SO_4)_{1.91}(OH)_{10.16}.3H_2O^*$ and namuwite (formula based on the presence of 7 oxygens) yields $(Zn_{2.31},Cu_{1.71})_{4.02}(SO_4)_{0.99}(OH)_{6.06}.4H_2O^*$, where * = H_2O idealized. Copper-zinc ratios in Smallcleugh serpierite and namuwite are fairly constant, averaging 1.45 and 0.74:1 respectively, whereas that reported from the type locality for schulenbergite is 1.33:1. Smallcleugh serpierite possesses a Cu:Zn ratio of 3.16:1.

Textural evidence tentatively suggests that the schulenbergite and namuwite at Smallcleugh Mine formed later than the serpierite and as the former have lower Cu-Zn ratios than serpierite it appears that within the supergene assemblage Cu became progressively depleted.

ACKNOWLEDGEMENTS

We thank Mr C. Chaplin and Mr R.J. Reekie at the Royal Museum of Scotland for preparation of the electron microprobe slide and Ms J.M. Horák and Mr M.P. Lambert at the National Museum of Wales for assistance with X-ray diffraction. B. Young kindly commented on an early version of the manuscript.

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J. Russell Soc., 3(1), 25–28 (1990).

SUPERGENE MINERALS FROM LOW PIKE, CALDBECK FELLS, CUMBRIA

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A suite of rare supergene minerals occurs in a hitherto undescribed trial working on the north slopes of Low Pike, near the Potts Gill Mine, Caldbeck Fells. Supergene minerals present include bayldonite, beudantite, cornwallite, duftite, malachite, mimetite, philipsburgite and pseudomalachite.

INTRODUCTION

The numerous abandoned mines and trial workings of the Caldbeck Fells have long been celebrated for a wide variety of supergene minerals. Many of these are very rare species. Most prominent are lead-, copper-, zinc-, and arsenic-bearing minerals but tungsten, molybdenum, antimony and vanadium species have been reported from some localities. The most recent and most comprehensive compilation of Caldbeck Fells minerals is that by Young (1987). Interesting new finds continue to be made and published and already several additional rare species can be added to Young's list (Cooper and Stanley, *in press*). These recent discoveries have mainly been made at already well known sites. Described here is a small suite of rare supergene minerals from a hitherto undescribed trial working on the north slopes of Low Pike [Nat. grid ref. NY 320 358], south of the former Potts Gill barites mine.

GEOLOGICAL SETTING

Much of the Caldbeck Fells consists of lavas and volcanic sediments of the Ordovician Eycott Volcanic Group. Mudstones and sandstones of the Lower Ordovican Skiddaw Group underlie the volcanics and outcrop in the area around Great Sca Fell [NY 292 339] and Knott [NY 296 330]. A smaller outlier of Upper Ordovician Dry Gill Shales is present at the head of Dry Gill [NY 321 345]. Intruded into these rocks are the gabbros and granophyres of the Carrock Fell Igneous Complex. Numerous faults cut the area. Many of these are mineralized and form the veins which carried the deposits of lead, copper, zinc and tungsten ores and barite which were formerly the basis of the local mining industry. Over much of the area the solid rocks are concealed beneath a thick mantle of glacial deposits, mainly boulder clay.

The detailed geology of Caldbeck Fells has been described by Eastwood *et al.* (1968). The area has had a long history as a mining field, extending back over at least four centuries. Much has been published on some of the area's minerals, particularly the rarer species, notably by A.W.G. Kingsbury and J. Hartley. However comparatively little has ever been published on the mineral deposits themselves or the mines that worked them. Brief outlines of some were given by Eastwood (1921), Dewey and Eastwood (1925), Dunham and Dines (1945), Eastwood (1959) and Eastwood *et al.* (1968). Useful but incomplete accounts of the mining industry have been published by Shaw (1970) and Adams (1988).

The two most prominent and longest mineralized fractures of the Caldbeck Fells are those generally referred to on maps of the British Geological Survey as the Roughton Gill North and South Veins. In detail these are likely to be complex features and not simple single fractures. During the eighteenth and nineteenth century these veins were known as the Silver Gill and Roughton Gill Veins, respectively, and the eastern extremity of the former was referred to as Low Pike Vein by Dunham and Dines (1945, p. 100). Both the Roughton Gill North and South Veins have been worked extensively for lead and copper in Roughton Gill and Silver Gill where rocks of the Carrock Fell Complex form one or both walls. The South Vein, in lavas of the Eycott Volcanic Group, has also been worked between Driggith and Sandbeds Mines [NY 328 352 and NY 332 362]. Little is known about the veins in the intervening ground in the High Pike and Low Pike areas, where Eycott Volcanic Group rocks form the wall rocks.

Dunham and Dines (1945) recorded a trial, made in 1927, on Low Pike Vein on the lower northeastern slopes of Low Pike [NY 325 360]. Here a narrow string of barite followed the footwall of the vein for up to 12 m. No other details of the mineralization at this site were recorded. Further west the course of Rougthon Gill North Vein across the north slopes of Low Pike is marked by a slight break in slope. Three small trial adits, all now collapsed, have been driven to the vein in this area. The dumps consist mainly of altered lava wall rock together with abundant vein quartz, in places with coatings of manganese oxides, and locally a little barite. The vein is exposed as a rib of massive white quartz up to 0.75 m wide adjacent to the collapsed adit entrance at the easternmost of the trials [NY 320 358]. Apart from iron and manganese oxide stains no other minerals have been observed in the vein outcrop there. However the adjacent dump has yielded an interesting suite of copper, lead and zinc supergene minerals, mostly as thin encrustations or stains on vein quartz and wall rock. Supergene minerals present include bayldonite, beudantite, cornwallite, duftite, malachite, mimetite, philipsburgite and pseudomalachite. Primary sulfides are rare though small amounts of chalcopyrite and galena have been seen.

SUPERGENE MINERALS OF THE LOW PIKE TRIAL

The supergene minerals found on the dump from the Low Pike trial are described below. Most were identified by X-ray diffraction. Numbers shown in brackets thus (Ph 7411, 6765F) are British Geological Survey and Natural History Museum X-ray numbers respectively. Infrared spectrophotometry analyses were performed on some specimens. They are indicated thus (MPC74).

BARITE, $BaSO_4$

Although much of the compact white to cream-coloured barite on the Low Pike dump is clearly part of the primary vein filling, a few specimens of pseudomalachite and cornwallite show later encrustations of small clear colourless complex crystals up to 3 mm long which are of supergene origin. A few of these barite crystals contain minute acicular green inclusions presumed to be malachite or brochantite.

BAYLDONITE, $PbCu_3(AsO_4)_2(OH)_2.H_2O$

This is a comparatively common mineral at Low Pike. It occurs as emerald-green to grass-green finely to coarsely crystalline patchy crusts up to 2 cm across on quartz and, in places, wall rock (Ph 7411, Ph 7414, Ph 7417, Ph 7422). In several specimens bayldonite encrusts or replaces a pyromorphite-mimetite group mineral.

BEUDANTITE, $PbFe_3(AsO_4)(SO_4)(OH)_6$

A few specimens have been identified as beudantite. The mineral occurs on quartz as very thin bright yellow crystalline crusts composed of rhombohedral crystals about 50 microns across (Ph 7421) and as greenish-yellow slightly iridescent rhombohedra up to 0.1 mm across (6765F). In one specimen examined (Ph 7429) X-ray lines suggestive of a crandallite group mineral were noted but it has not been possible to determine the precise identity of this.

BROCHANTITE, $Cu_4(SO_4)(OH)_6$

This mineral has been found on a very few specimens as sprays of small dark green acicular crystals up to 1 mm long.

CORNWALLITE, $Cu_5(AsO_4)_2(OH)_4.H_2O$

This occurs sparingly as microcrystalline turquoise-green crusts up to 10 mm across lining cavities in quartz (5939F) and as banded dark green crusts filling narrow cracks in quartz (MPC 92). Some of the latter closely resemble malachite in appearance. Specimens examined by infra-red spectroscopy show major substitution of phosphorous for arsenic and material from Low Pike may exhibit a more or less complete passage to the phosphate end-member. Cornwallite and pseudomalachite from Low Pike cannot be distinguished from one another visually.

DUFTITE, $PbCu(AsO_4)(OH)$

Duftite has been identified in two specimens. In one it occurs as a bright lemon-yellow crust intimately associated with bayldonite (Ph 7412). In the second specimen (Ph 7416) duftite is present as a coating of minute yellowish-green crystals which encrust bayldonite.

MALACHITE, $Cu_2(CO_3)(OH)_2$

At Low Pike malachite occurs as tufts of minute green acicular crystals. At first sight some of the cornwallite and pseudomalachite from this locality can easily be confused with malachite.

MIMETITE, $Pb_5(AsO_4)_3Cl$

Pyromorphite-mimetite group minerals occur in several generations at Low Pike. All specimens analysed by microprobe or infra-red techniques show considerable substitution of phosphorus for arsenic. Mimetite occurs as crusts of yellow to yellowish-green crystals up to several millimetres long on quartz and barite (Ph 7430, MPC74) or as small white hexagonal crystals (Ph 7413). The mineral is commonly coated with, and locally replaced by, bayldonite.

MOTTRAMITE, $PbCu(VO_4)(OH)$

A single specimen of a thin greenish-brown crust, associated with bayldonite, has been identified (7155F) as mottramite (N. Thomson, personal communication).

PHILIPSBURGITE, $(Cu,Zn)_6(AsO_4,PO_4)_2(OH)_6.H_2O$

Philipsburgite was first described as a new mineral from Montana by Peacor *et al.* (1985). Recently the mineral was reported, for the first time from Britain, by Braithwaite and Ryback (1988) who described its occurrence in a loose block on the dumps of Potts Gill Mine [NY 320 367] and also commented briefly on its presence at Low Pike.

Several specimens of philipsburgite have been collected from Low Pike (*e.g.* Ph 7418, Ph 7424, Ph 7425). The mineral exhibits several different habits at this locality. It commonly forms crusts of radiating acicular pale turquoise-blue crystals forming spherulitic masses up to 2 mm across on quartz (MPC72). These masses locally display a concentric shell-like structure. Globular aggregates, up to 1 mm across, of radiating pale turquoise-blue bladed crystals are also common (MPC76). These aggregates typically exhibit a comparatively deep bluish-green minutely crystalline drusy outer surface and are paler, almost white, within. Less commonly philipsburgite forms bluish-green to apple-green bladed crystals lining cavities up to 4 mm across in cellular iron-stained quartz.

X-ray diffraction data for one specimen of philipsburgite (Ph 7418) are given in Table I. Ni-filtered CuKα radiation was used in a 114.6 mm diameter Debye-Scherrer camera. The (*hkl*) indices given are by analogy with the published data for philipsburgite (Peacor *et al.*, 1985), with the exception of those annotated by an asterisk, which are by analogy with the data for kipushite (Piret *et al.*, 1985). The most significant difference between the present data and that of Peacor *et al.* (1985) is the occurrence of the reflection at 6.10Å, corresponding to the (200) reflection of kipushite at 6.06Å, and consistent with the slightly larger cell size of philipsburgite. The relative intensities (I_{vis}) are by visual estimation. Semi-quatitative microprobe data for three specimens of philipsburgite are given in Table II.

PSEUDOMALACHITE, $Cu_5(PO_4)_2(OH)_4.H_2O$

This mineral is relatively common at Low Pike. It is present as bright turquoise-blue botryoidal crystalline crusts up to 5 mm across in cavities in iron-stained cellular quartz (Ph 7410), as dark grass-green botryoidal crusts up to 7 mm across on quartz (Ph 7426), as thin blue crystalline crusts on quartz (Ph 7419) and as dark green crystalline crusts up to 5 mm across lining irregular cavities in iron-stained quartz (Ph 7427). In this latter form the mineral appears bright blue when removed from the ferruginous matrix. Specimens have also been obtained which show pale turquoise-blue pseudomalachite crusts with a smooth porcellaneous surface (MPC90). As noted above pseudomalachite and cornwallite from Low Pike can not easily be distinguished and specimens may exhibit compositions intermediate between the two species.

TABLE I

X-ray powder diffraction data for philipsburgite (Ph7418) from Low Pike.

d (Å)	I_{vis}	hkl	d (Å)	I^{a}_{vis}
12.35	vs	100	2.45	vvw
6.96	vvw	011	2.43	w
6.30	vw	111	2.40	vvw
6.10	s	200*	2.36	m
5.87	vvw	111	2.34	vw
5.09	mw	102 210	2.33	mw
4.65	vvw	102	2.29	w
4.53	vvw	112*	2.26	w
4.31	mw	120 202	2.18	vw
4.09	vvs	300	2.16	m
3.78	vw	202	2.15	m
3.74	vw	310	2.07	w
3.57	mw	221	2.03	vvw
3.50	vvw		1.99	vvw
3.46	vw		1.97	vvw
3.40	ms	122 311 221	1.945	ms(432)*
3.31	w	013	(+13 m to vvw lines)	
3.24	w	312	1.53	ms
3.15	w		1.505	ms
3.10	vvw		(+25 m to vvw lines)	
3.07	vw	302 400		
		213 320		
2.89	mw	131 411 321		
2.67	ms	412 123		
		104 032		
2.56	s	132 114		
2.55	vvw	014		
2.50	vw	232		

[a] Abbreviations: s = strong, m = medium, w = weak, v = very.

TABLE II

Semi-quantitative microprobe analyses (wt %) of philipsburgite in two specimens (Ph 7425 and Ph 7424) from Low Pike.

	Ph 7425		Ph 7424	
	a	b	a	b
CuO	49	45	44	45
ZnO	10	9	9	10
FeO	na	na	2	nd
Al_2O_3	1	<1	1	<1
P_2O_5	7	6	5	5
As_2O_5	27	22	23	24
SO_3	na	na	<1	nd
SiO_2	na	na	1	<1
Total	94	83	86	85

Energy-dispersive analyses, 50 seconds livetime, on crystal faces of unpolished grains. Computed values of over 1% oxide were rounded to the nearest 1%. Analytical errors are likely to be larger than this. The low totals are due in part to the undetected (OH) and H_2O present; na = not analysed, nd = not detected.

ACKNOWLEDGEMENTS

We wish to thank the staff of the British Museum (Natural History) for X-ray diffraction determinations, J. Faithfull, Department of Geology, University of Leicester and M. Rothwell, Unilever Research for electron microprobe analyses, and R.S.W. Braithwaite, Department of Chemistry, University of Manchester Institute of Science and Technology for infrared spectroscopy analyses. Mrs J. Dunkley is thanked for preparing the typescript. This paper is published with the permission of the Director, British Geological Survey (NERC).

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J. Russell Soc., 3(1), 29–30 (1990).

HAUSMANNITE FROM WEST CUMBRIA

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Hausmannite, manganite and pyrolusite have been identified as constituents of the manganese mineral assemblage at Wyndham Pit, Bigrigg, Cumbria. Hausmannite is thus confirmed as a British mineral.

Hausmannite, Mn_3O_4, was first recorded as a British mineral by Goodchild (1875) from the iron mines of the Cleator area, Cumbria. It was reported to occur well-crystallized associated with pyrolusite in veins and pockets near the margins of the hematite ore body. Perhaps surprisingly no precise locality details were quoted and no determinative data were given. More recently Smith (1924) referred to the presence of manganese ores adjacent to the margins of the hematite ore body in the Carboniferous Limestone at Wyndham Pit, near Egremont [NY 003 126]. Apart from manganite, MnO(OH), Smith did not specify the manganese minerals present. Passing reference was made to hausmannite, together with pyrolusite, MnO_2, braunite, Mn_7SiO_{12}, and rhodochrosite, $MnCO_3$, at Wyndham Pit by Davidson and Thomson (1948). The occurrence of hausmannite in the Whitehaven area was also noted by Palache *et al.* (1944). Specimens labelled hausmannite from Wyndham Pit are present in many collections of British minerals although no description of the mineral has been published. Criddle and Symes (1977) described hausmannite from the Tŷ Coch Mine, in Mid Glamorgan. In a recent review of British minerals Macpherson (1983) noted the lack of determinative data for this mineral, commenting that the occurrence of hausmannite as a British species requires confirmation.

Wyndham Pit has long been abandoned and none of the underground workings is now accessible. Several years ago landscaping operations at the disused Langhorn Quarry, adjacent to Wyndham Pit, uncovered a small amount of dump material from the Wyndham Pit workings. From this a very few fragments of manganese ore were collected.

Examination of these by X-ray diffraction confirmed the presence of hausmannite (BGS X-ray powder photograph number Ph 7244). The hausmannite occurs as compact dark grey crystalline masses up to 8 cm across in which bright cleavage surfaces up to 2 mm across are commonly conspicuous. Locally streaks and pockets of white crystalline calcite up to 1 cm across occur in the ore. Adjacent to these the hausmannite locally exhibits well-developed striated pseudo-octahedral faces up to 1.5 mm across. This suggests that the calcite filled early cavities in the manganese ore. A few small crystals of barite are also present.

A small chip of the ore with visible crystal faces was immersed in 2N HCl to dissolve the carbonate, thus revealing small cavities lined with well-formed hausmannite crystals (Figures 1 and 2), showing characteristic form and twinning. The latter is particularly well displayed where adjacent faces of a twin pair are striated parallel to [100], the striae exhibiting strongly re-entrant angles emphasising the position of the twin plane (Figures 3 and 4). Parallel groups aligned on the *c* axis are also common (Figure 5), suggesting that the striae on individual crystals might be due to alternations of (011) and (011). In many cases however, the striae themselves are re-entrant grooves of assymetric cross-section, rather than simple steps, implying that a higher order (0*kl*) form is also present.

Blocks of recrystallized and partially dolomitized limestone collected from the dump contain calcite-lined vughs up to 3 cm across. In these occur a few clusters of lustrous black bladed crystals up to 2 mm long which are commonly grouped into sub-parallel bundles. Examination of this material by X-ray diffraction identified both pyrolusite and manganite (Ph 7245, 7246). Lines suggestive of ramsdellite (MnO_2) were also noted but it was not possible to confirm the presence of this mineral with certainty.

ACKNOWLEDGEMENTS

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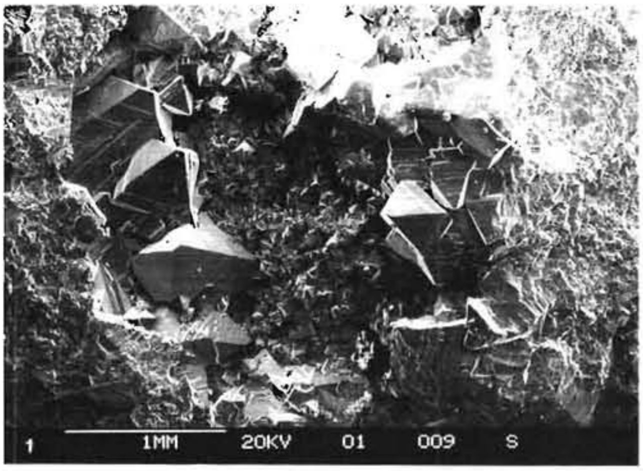

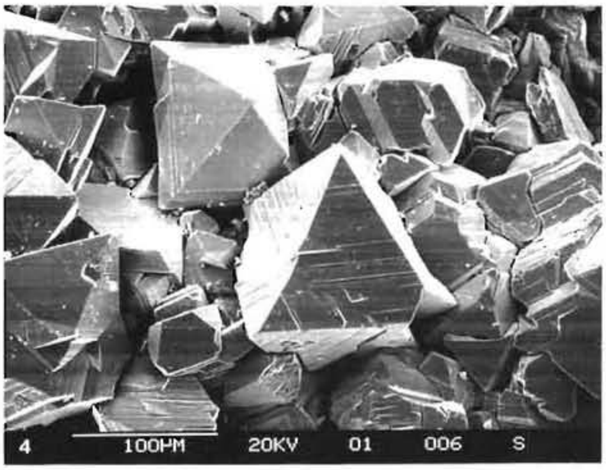

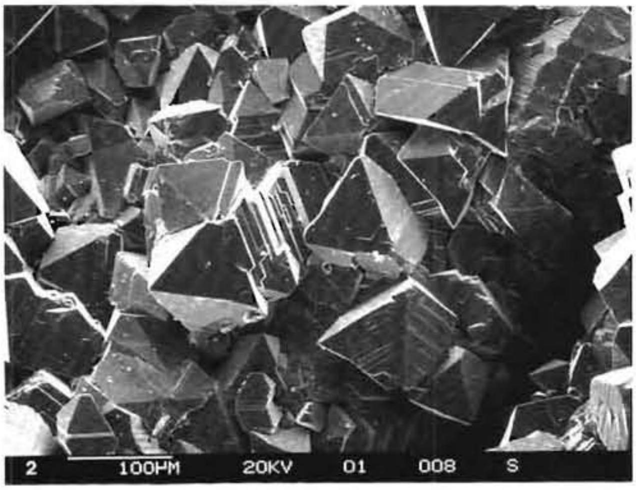

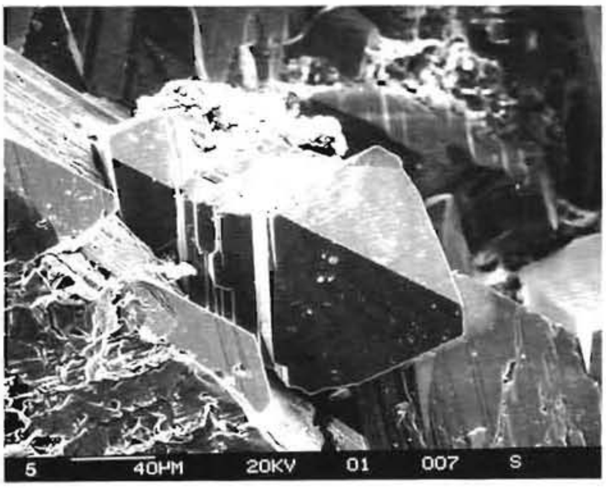

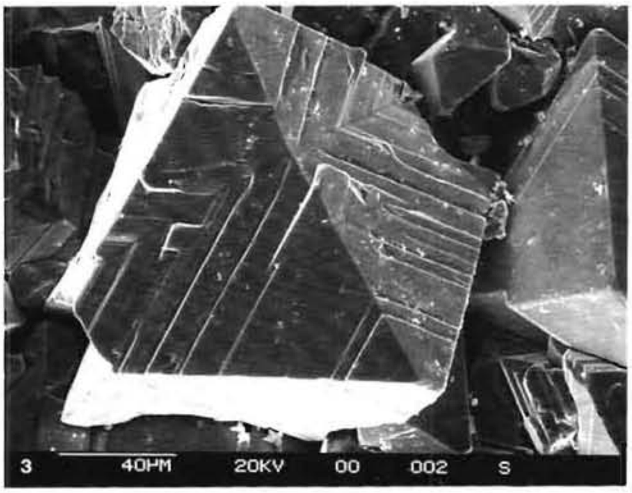

FIGURES 1 TO 5. Scanning electron microscope photomicrographs of hausmannite crystals from Wyndham Pit, Cumbria. Scale bars are indicated on individual plates.

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J. Russell Soc., **3**(*1*), *31* (*1990*).

NOTE

BERTRANDITE FROM THE PASS OF BALLATER, GRAMPIAN REGION, SCOTLAND

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Bertrandite, $Be_4Si_2O_7(OH)_2$, was first recognised as a new mineral species and described by E. Bertrand from the quarry of Petit Port near Nantes, France (Bertrand, 1880), although the mineral was formally described as a new species a little later by Damour (1883). Since then the mineral has been reported from a number of localities around the world, generally in pegmatite or granite, and often associated with beryl. Sometimes, bertrandite occurs in cavities formerly occupied by beryl crystals, and it has been noted as pseudomorphs after that mineral (Dana, 1932).

In the United Kingdom, the occurrence of bertrandite from Cheesewring Quarry, near Liskeard, Cornwall was reported by Bowman (1911). Sir Arthur Russell subsequently found the mineral at the nearby Gold-diggings Quarry, as well as at East Kit Hill Mine, near Callington (both in Cornwall), although at these localities it does not appear to be associated with beryl (Russell, 1913). Phemister (1940) described a further Cornish occurrence from South Crofty Mine, Camborne. Bertrandite has also been reported from Scotland (Morgan, 1967) as crystals up to 3 mm across, associated with genthelvite in miarolitic cavities in adamellite from Coire an Lochain in the Cairngorm Mountains.

Bertrandite has been identified by X-ray powder diffraction in specimens collected in 1986 from the screes below Creag an t-Seabhaig, on the north side of the Pass of Ballater [National Grid Reference NO 368 972]. This is believed to be only the second recorded occurrence of the mineral from Scotland. The occurrence of beryl from the Pass of Ballater area was recorded by Heddle (1901) and has been subsequently confirmed by the writer as masses up to 8 cm across in pinkish granite. Bertrandite has also been collected *in situ* from the Pass of Ballater area by later workers (J. Faithfull, personal communication).

Lustrous colourless crystals of bertrandite up to 1 mm across were found to thickly encrust the walls of a 10 mm cavity in coarsely crystallized pinkish granite. The crystals exhibit characteristically brilliant pinacoids and have striated prism faces. The bertrandite is associated with minor pale purple to colourless fluorite, and colourless, doubly-terminated quartz crystals.

No beryl is present on the specimen described, but given the relatively common occurrence of this mineral in the scree material and large boulders, it seems reasonable to suggest that the bertrandite has formed by hydrothermal alteration of earlier beryl crystals.

ACKNOWLEDGEMENTS

Thanks are due to Dr J. Faithfull, Department of Geology, University of Leicester, for the X-ray confirmation of bertrandite.

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Adams, J. *Mines of the Lake District Fells*. Clapham, Lancaster. Dalesman Publishing Co. Ltd., 1988. 160 pp. Paperback £8.95. ISBN: 085206 931 6.

Recent years have seen a proliferation of publications on mining, and particularly metalliferous mining, history. Almost every corner of the country in which minerals have been sought or worked seems to have attracted the attention of mainly amateur mine explorers and historians. Whereas much of the resulting literature is incomplete and often poorly researched a few texts constitute worthwhile additions to the knowledge of British mining history. John Adam's recent book on the *Mines of the Lake District Fells* falls decidedly into the latter category.

The book is, as described on the cover, a '. . . compact, comprehensive catalogue and history . . . ' of mines and trial workings in the Lake District. It begins with a very brief introduction to the nature of mineral deposits and a short review of methods of prospecting and working the type of metalliferous deposits found in the Lake District. The bulk of the book then consists of descriptions of as many of the area's mineral workings as the author has been able to discover and research, illustrated by numerous plans and sections. In addition to fairly lengthy descriptions of the larger and better known Lake District mines Adams presents a wealth of information on numerous lesser known and generally smaller workings. Indeed some of his descriptions are the only published accounts of many of these. For an area with such an important part in the long history of British metalliferous mining it is astonishing that so many of the Lake District's old mines and trials should never even have been mentioned in print until now. This book goes some way to correcting this neglect. For this alone the book is a useful reference to all wishing to study mineral deposits in Britain and those of the Lake District in particular. It will also serve as a fascinating companion for visitors to the Lake District who wish to discover more about this once important aspect of Cumbrian life and industry.

No book of this sort is ever completely free of flaws and this one is no exception. Happily these are comparatively few and generally not serious. A surprising omission from the introductory section is any discussion of the system of mine leases and royalty payments. The descriptions of individual mines would be easier to follow if they had been grouped by areas under clearly separate headings as in the index. In some instances there is no indication of what mineral was worked or sought at a particular site. Whereas there may be no documentary records of this a brief comment on what may be seen at the site today would be helpful. For some mines Adams provides rather less information than is available in other publications. Admittedly it is difficult to decide on how much of this should be included but perhaps rather more could have been included on for example the Potts Gill mines. Certainly the reader could have expected more on the Coniston mines without being invited to buy another author's book.

The absence of a bibliography is disappointing and is probably the book's most serious defect.

The descriptions of mines are accompanied by numerous plans and sections. Unfortunately these are not numbered and not referred to in this text. Whereas most are quite clear there is some inconsistancy of style, particularly in the almost universal free-hand lettering. A few plans contain insufficient surface topographic features to fix them on the ground. Some are reproduced with a strange orientation. For example, Hay Gill Mine (p. 80) appears at the bottom of the page while the section of Kinniside Mine (p. 105) has no scale!

These criticisms are generally minor and do not detract from an interesting and useful book published in an attractive format by Dalesman. John Adams is to be congratulated on a fine piece of amateur research. It is clear from reading the book that it is the product of genuine enthusiasm. He has produced a worthwhile addition to the literature of British mining and the Lake District. The book is strongly recommended.

B. Young

Farra, O. and Spencer, C. *Mineral Wealth of Saudi Arabia*. London (IMMEL Publishing Ltd.), 1986. 128 pp. Price £23.75.

This book is a photographic record of some of the more interesting of the minerals, rocks and fossils that the authors have come across during their travels in Saudi Arabia. It does not claim to be a scientific work, although a thread of geological background has been woven into the presentation. In addition to ores of Fe, Zn, Cu, Mo, W and Ti, Au occurs at King Solomon's mine at Mahd adh Dhahab. Also illustrated are olivine nodules, desert roses, fluorite, magnesite, barite, graphite, diatomite, etc.

R. A. Howie

Boscardini, M. and Sovilla, S. *Il giacimento mineralogico di Pietro in Montecchio Maggiore (Vicenza)*. Montecchio Maggiore (Museo Civico 'G. Zannato'), 1988. 93 pp., 37 figs., 45 colour photos.

A local development of amygdaloidal basalt, basaltic tuffs and explosion breccias which overlie the Tertiary sediments in this area of northern Italy contains relatively abundant zeolites. This nicely produced publication describes and illustrates each species, together with morphological drawings and some infrared spectra. The commoner species are analcime, natrolite, apophyllite, calcite, celadonite and smectite, while gmelinite, heulandite, thomsonite and celestine are less common, and chabazite, erionite, stilbite, phillipsite and levyne are rare.

R. A. Howie

Taucher, J., Postl, W., Moser, B., Jakely, D. and Golob, P. *Klöch: ein Sudösterreisches Basaltvorkommen und*

Seine Minerale. Graz, Austria (J. Taucher), 1989. 160 pp., 200 colour photos. Price: Austrian Schillings 1080.00.

This work presents an unusual combination of scientific mineral descriptions and aesthetically inspired mineral photography. More than 70 mineral species are described from the nepheline basanite and basalt of the large quarry at Klöch in SE Austria and illustrated in 200 colour photographs and in 200 black-and-white illustrations (including morphological drawings and SEM photographs). Calcite, aragonite and numerous zeolites feature strongly.

R. A. Howie

Ineson, P. R. *Introduction to practical ore microscopy.* Longman Earth Science Series, 1989. 181 pp. Paperback £11.95. ISBN : 0-582-30140-8.

Most students of geology spend hours, if not years, looking at minerals in transmitted light, but usually have little chance to develop skills in reflected light microscopy. It is therefore hardly surprising that the technique is often viewed as a difficult one and best left alone. In most branches of geology there is a shortage of low-price introductory texts, and one concerning the practical aspects of ore microscopy could form an important addition to the mineralogy student's library.

This text is essentially a very basic introduction to ore microscopy, and most aspects are covered, albeit rather briefly. The first half of the book consists of seven chapters concerning the methods used in reflected light microscopy. After a very short introduction (chapter 1), there are sections describing reflected-light microscopes (chapter 2), sample preparation (chapter 3), methods used for mineral identification (chapter 4), mineral textures (chapter 5), paragenesis (chapter 6), and structural etching (chapter 7). Most of the remainder of the book is taken up by tables listing the properties of the most common ore minerals. Appendices contain brief information on common ore mineral associations and reflected light optics. The coverage of minerals and ore deposit types is reasonably comprehensive, although rather surprisingly there is very little discussion of supergene effects. Other important omissions include at least some indication of the complexities of reflected light optics (and their differences to those of transmitted light), and any description of the appearance in reflected light of common gangue minerals, such as quartz, silicates, and calcite. Advertisements for the book state that it 'incorporates information on the milling and processing of materials'; this would certainly have been a useful subject to cover, but it is not dealt with at all!

However, as a *practical* guide it has certain deficiencies. For instance, there is a lengthy description of textures and how to determine paragenesis, but little advice on how to use this information. It would have been helpful to see some conclusions derived from the descriptions of these features. But my main criticism lies in the lack of any photographs of minerals as viewed under the microscope. In their place are numerous line drawings, but these often have no labels or scales, and are really of very limited value.

The elementary nature of this book means that it will be most suited to a complete beginner, who needs to obtain a rapid introduction to the principles and possibilities of ore microscopic techniques. However, more serious users will rapidly progress to a higher level and soon have need of more detailed information. It will naturally be compared to 'Ore Microscopy and Petrography', a similar text by Craig and Vaughan. This latter work appears to represent much better value, as for a similar price one gets a text which is more comprehensive, goes into more detail, and contains numerous photographs.

D. H. M. Alderton

Brodtkorb, M. de (Editor) *Nonmetalliferous Stratabound Ore Fields.* Van Nostrand Reinhold, 1989. 332 pp. Price £38.50.

This book is the fourth of a rather disparate number of volumes in the 'Evolution of Ore Fields Series', a series designed to help to 'know' more about the orefield . . . . .and not just study individual deposits'. To this end, Dr Brodtkorb has collected and edited fourteen chapters, with an average length of thirty pages, from seventeen authors including four chapters where she is the author or co-author. The book therefore reflects her interests with eight chapters devoted to barium deposits (mainly barite), two to strontium and celestite and one each to barite plus celestite, magnesite, fluorite and a final summary and synthesis chapter.

The first two short chapters outline the geochemistry of strontium and barium, respectively, and stress the importance of $^{87}Sr/^{86}Sr$ ratios in helping to determine the genesis of celestite and barite deposits – a useful introduction, as these data are used quite extensively in the following nine chapters. Here, celestite and barite orefields are described on a geographical basis, from world-wide or continental to small district. Inevitably these chapters show the regional experience and/or nationality of their authors and this has produced further inbalances. Barite deposits in Europe have two chapters, deposits in Western Europe and deposits in Sardinia similarly; North America has stratabound barite deposits of North America except for Arkansas, and Arkansas separately. All of the chapters are very descriptive and often highly detailed but the most useful and interesting ones are those that deal with broad areas ('Stratabound barite ore fields of North America excluding Arkansas') or broad classes of deposits ('The barite ore fields of Kuroko-type of Japan'). Chapters on magnesite and fluorite are followed by a final review of stratabound celestite, barite, magnesite and fluorite deposits. This neatly encapsulates the earlier chapters and concludes that the deposits are widely distributed in time and found within many different tectonic settings – conclusions that show that these deposits

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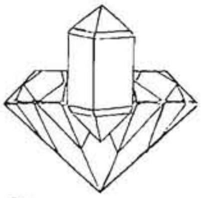

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have been neglected academically and suggest that further synthesis is possible.

The volume is excellently produced, the photographs, figures and tables are well reproduced and generally the text is in readable English with few typographical errors.

Nonmetalliferous Stratabound Ore Fields is a valuable contribution in highlighting the current uncertainties regarding barite genesis as well as a first step towards solving them.

R.A. Ixer

Macpherson, H.G. *Agates*. London (Natural History Museum) and Edinburgh (National Museum of Scotland), 1989. 72 pp, 165 colour photographs. Price £4.95. ISBN: 0 565 01100 6.

An immediate question when faced with a book such as one dedicated solely to agates springs to mind: 'Is it too specialized for me?'; maybe some may ask 'How can you produce a book describing only agates?' The answers are simple. No, it is not too specialized, and yes, you can write a book describing only agates. The author is to be congratulated on the publication of such an interesting and informative work.

This lavishly illustrated booklet, co-published by the National Museum of Scotland and the Natural History Museum, is produced in the format of the other popular Geological Museum publications, including the recently published *Gemstones*. At £4.95 this, and indeed the other booklets, represents excellent value.

Following a brief description of what agates are, the author provides an account of agate-bearing rocks in the British Isles. A very informative section then presents the various hypotheses concerning the formation of agates. Here the reader is introduced to some of the more subtle aspects of agate structure such as tubes of escape, dilation features, agate dykes and so on. It all shows that there is more to agates than immediately meets the eye! There then follows a section detailing British agate localities, which collectors will find of enormous interest and value. Of course, the majority of these are Scottish localities, and were taken from Heddle's *The Mineralogy of Scotland* (1901). For these localities, national grid references are provided. Agate localities in England, Wales and Northern Ireland are then briefly described.

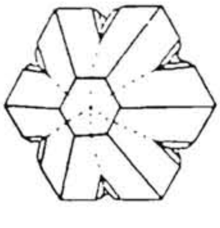
Arguably the most interesting section of the book then follows, entitled 'A Portfolio of British Agates.' Again, this largely relates to Scotland, but the detailed descriptions and illustrations of agates from such classic localities as the Blue Hole at Usan, Scurdie Ness, Ballindean, Middlefield Farm, and so on, are exceptional. The photographs are in their own right an impressive record for each site. In addition, the accounts of early collectors and discoveries at these sites are extremely interesting, and it is very pleasing to see that specific contemporary or recent collectors are acknowledged for their individual contributions, collections and donations of agates.

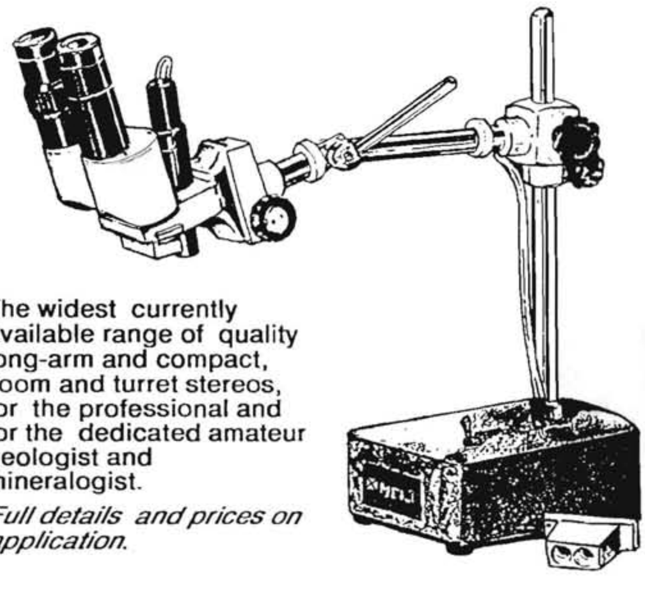
Brief mention is made of agate collecting, pointing out not only what to look for in the field, but also the measures which must be adhered to before actual collection takes place. Emphasis is given to the need to establish land ownership matters and permission for collecting.

Techniques of agate cutting and polishing are next described and illustrated, as are some of the various products from lapidary suppliers. A particularly interesting section describes pebble jewellery, which is infinitely more subtle than the name implies. Agates from around the world are briefly dealt with in the final section of the booklet, which is followed by a useful glossary and a bibliography.

On the back cover another superb photograph of an agate is provided, but this leads me to my only criticism of the work. I cannot understand the choice of front cover, which features a picture which means nothing to me and which does not even resemble an agate. The choice is puzzling, particularly when there are 165 or so other marvellous photographs of agates to choose from. Of course, I am sure that to the author it is significant, maybe something very personal, and good luck to him after providing us with such an enjoyable contribution to the mineralogical literature.

R.E. Bevins

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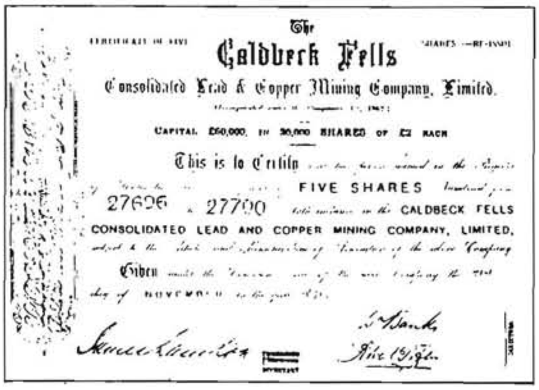

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Both full articles, covering all aspects of mineralogy including topographical mineralogy, and notes (up to 1000 words) are accepted for publication. Each full length paper should contain an abstract of up to 250 words summarizing the significant points of the paper. No abstract is required for notes.

The first sheet of the typescript should give, in order, the title of the paper, the names and addresses of the authors, and the name and address of the author for correspondence and to whom proofs are to be sent. Text pages should be numbered serially. Papers should be submitted in the style and format of the Journal.

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If varietal names are employed in the text, these should appear underlined (for italics) after the formal definition and should be referred to in the same way throughout the text. Chemical formulae for minerals, wherever appropriate, should be included.

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Nomenclature should be that adopted by the International Union of Pure and Applied Chemistry. If authors are in doubt, they should consult the Editor.

Crystal structure studies should be reported in the manner outlined by the International Union of Crystallography (*Acta Crystallographica*, **22**, 45 (1967)). Tables of anisotropic thermal parameters and observed and calculated structure factors should be deposited with the Editor.

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References and notes should be indicated in the text by the author and year of publication, thus: (Brown, 1967) or, 'as stated by Brown (1967)'. If two authors are involved, both should be named: (Green and Brown, 1985), and if more than two authors: (Green *et al.*, 1986). When more than one reference is quoted, each should be separated by a semicolon thus (Brown, 1969; Green *et al.*,1986).

A list of references should be provided in alphabetical order. References should be given in full. Some examples are given below.

Manning, D.A.C. and Henderson, P., 1984. The behaviour of tungsten in granitic melt-vapor systems. *Contributions to Mineralogy and Petrology*, **86**, 286–293.

Pankhurst, R.J., Sutherland, D.S., Brown, G.C. and Pitcher, W.S., 1982. Caledonian granites and diorites of Scotland and Ireland. *In*: Sutherland, D.S. (ed.) *Igneous Rocks of the British Isles*. Wiley, London, 149–190.

McGarvie, D.W., 1985. Volcanology and Petrology of mixed-magmas and rhyolites from the Torfajokull Volcano, Iceland. Unpublished Ph.D. thesis, University of Lancaster.

Ramsay, J.G., 1967. *Folding and Fracturing of Rocks*. McGraw-Hill, New York.

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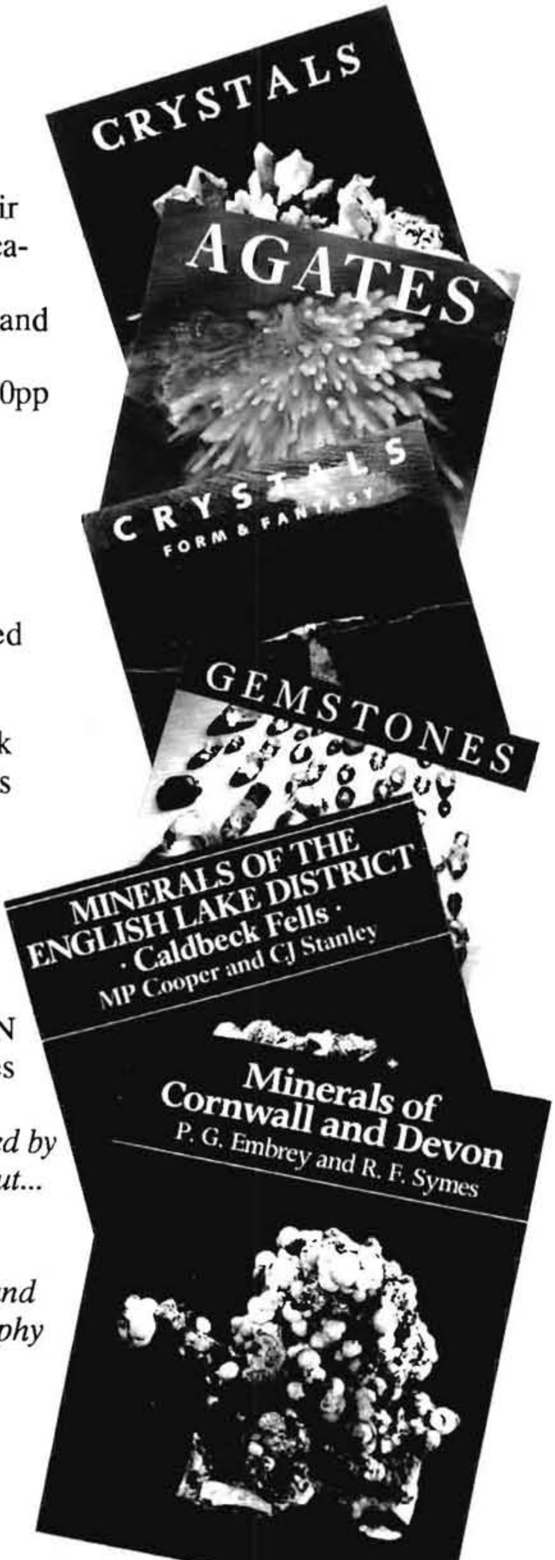

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