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7th May, 1976

# FLUID INCLUSION STUDIES OF S.A. ORES, ROCKS AND MINERALS

Progress Report No. 1

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#### SUMMARY

Recently-purchased microthermometry apparatus for studying fluid inclusions in rock samples has been set up, calibrated and used on a trial basis on a number of selected rock-types in South Australia. The apparatus is suitable for determining homogenisation and freezing temperatures of fluid inclusions and for estimating the temperature of their decrepitation. The results have enabled the minimum temperature of entrapment (or crystal growth) to be determined, together with estimates of fluid salinities and pressures, thus contributing to knowledge on the petrogenesis of barites, pegmatites, quartz veins etc. and on certain problems in the fading of colour in granite. The apparatus is now available on a routine basis for fluid-inclusion investigations and a number of petrogenetic applications are suggested.

#### 1. INTRODUCTION

#### 1.1. Aims of the Project

A proposal (No. 5/0/2613) to purchase and set up microthermometry apparatus, in order to commence fluid inclusion studies of selected rock-types and minerals in South Australia, was accepted by the Mines Department on 4 September, 1975.

The aims of the project for the current financial year (1975-6) were to purchase the most suitable type of equipment, to set it up, calibrate it and use it on preliminary tests to measure homogenisation temperatures on a range of suitable substances. It was anticipated that subsequently the technique would be applied in detail to a number of petrogenetic problems in South Australia.

## 1.2. Summary of the Applications of Fluid Inclusions Studies

Many minerals, on crystallising, incorporate small inclusions of the surrounding fluid, which, if preserved, represent one of the few types of natural evidence of ore- and mineral-forming conditions. From the study of such inclusions it may be possible to determine the temperature and pressure of formation of the host mineral and possibly also the density and chemistry of the fluid itself.

In conjunction with other evidence, such as from field relations, petrology etc., it may be possible to establish the following:

- (1) The genetic relationship of igneous intrusion to related hydrothermal veins (e.g. by establishing progressive changes in temperature and salinity towards the intrusion).
- (ii) Progressive changes of temperature and composition during mineralisation.
- (iii) Thermal gradients in ore veins.
- (iv) The likely genesis and classification of individual ore deposits (e.g. porphyry coppers, Mississippi Valley-type Pb-Zn deposits).
- (v) The effects of metamorphism and the cause of 'facies' changes.
- (vi) The nature of mineralisation as being of hydrothermal or of supergene origin.
- (vii) The origin or source of minerals in sedimentary rocks.

(viii) The physical conditions necessary for the formation of fracture porosity (e.g. in petroleum reservoir rocks).

Inclusions may be of primary, secondary or pseudoseconary origin. Primary inclusions represent fluids trapped at the time of growth of the enclosing mineral. Secondary inclusions represent fluids trapped during recrystallisation of the host, perhaps by annealing of fractures, while pseudosecondary inclusions are formed when a crystal fractures during growth. Consequently it is important to determine the nature of the inclusions present, though it may not always be possible to achieve this.

Primary inclusions tend to be sparsely distributed and large. If inclusions show a relationship to a formal crystal shape or to growth phenomena, they are presumed to be primary. In contrast, secondary inclusions tend to be smaller than primary inclusions in the same sample and to be concentrated in transverse or cleavage planes, which may cut across crystal boundaries. Pseudosecondary inclusions often lie in curved planes, which do not cut across crystal boundaries. Cofirmation that more than one set of inclusions is present may come from the different results obtained, though a range of homogenisation temperatures does not necessarily imply a number of It is also considered that inclusions in euhedral crystals which generations. project into vugs can be taken as primary, but whereas negative crystal shape is also taken by some as a primary feature, it is known that secondary inclusions can assume Clearly the history of the host mineral, if established, could assist in discriminating primary from secondary inclusions.

Large fluid inclusions are very rare, with the frequency of occurrence increasing with decreasing size (Roedder, 1967). Consequently such phenomena have to be studied by microscopic means, usually in conjunction with thick sections, polished on both sides. For these, clear crystals provide better viewing conditions than do milky or cloudy crystals. The presence of bubbles or of two immiscible liquids greatly aids the recognition of fluid inclusions, distinguishing them from solid inclusions or inclusions which have lost their contents through leakage or decrepitation.

When a crystal grows it may entrap an inclusion which is solid, liquid, gas or fluid. Many crystals grow from aqueous solution and in general one phase (referred to as a 'fluid' but usually a liquid) is trapped. Unless this occurs within the boiling range (when liquid and gas are in equilibrium) the liquid has to cool by a certain amount to relieve the excess pressure which prevented it from boiling, before it shrinks and forms a vapour phase. By heating up such inclusions once again until the vapour phase disappears, one can thus establish a minimum temperature for the entrapment, and usually a correction can be applied to account for the extra pressure on the liquid. This can be done if the depth of formation of the rock sample is known or if the salinity etc. of the liquid can be determined from the depression of the freezing point (see below).

The apparatus required must therefore be capable of heating the average fluid inclusion to its homogenisation (single phase) point. This usually takes place by the contraction and disappearance of the gas bubble (homogenisation in the liquid phase), but sometimes the homogenisation point is reached instead by expansion of the gas bubble to fill the whole area of the inclusion (homogenisation in the gas phase), or by the curved meniscus between gas and liquid suddenly fading and disappearing, giving a supercritical fluid (homogenisation at the critical point). An additional feature may be the decrepitation ('explosion') of inclusions. If all the inclusions decrepitate rapidly at the same temperature this may indicate the actual temperature of entrapment of the fluid by the host. Temperatures of entrapment may be as high 500° C or more, (e.g. some porphyry copper deposits and pegmatites) but for hydrothermal veins are

generally less than this (commonly  $150^{\circ}$  to  $400^{\circ}$ C).

The apparatus must also be capable of cooling the average fluid inclusion sufficiently low to enable the freezing point to be measured for aqueous solutions. These are usually NaCl brines, but solutions with KCl and  $\rm CO_2$  are also found. From the depression of the freezing point of water the approximate salinity etc. and density can be estimated and this can be used to calculate the corresponding pressure, and hence a temperature correction can be arrived at. Most phenomena to be observed with cooling techniques occur from  $\rm O^{O}C$  to  $\rm -30^{O}C$ , but since liquids tend to resist freezing (becoming supercooled) it may be necessary to cool down to  $\rm -170^{O}C$  or less, to promote freezing before raising the temperature to find the melting point.

#### 2. SETTING UP THE APPARATUS

## 2.1. Purchase and Date of Arrival

Following acceptance of Amdel's proposal the apparatus was ordered on 10 October, 1975. The delivery date had been indicated to be about two months from receipt of order, and the apparatus duly arrived on the 9th February, 1976.

#### 2.2 The Apparatus

The Chaixmeca microthermometry apparatus consists of a heating/freezing stage designed to fit onto a Leitz petrological microscope, a console with temperature read-out and controller, and a gas cooler. A crushing stage is also available to release gases from fluid inclusions for collection under oil.

By filling the gas cooler with liquid nitrogen it is possible to achieve a sample temperature as low as  $-180^{\circ}$ C. The built-in heating element permits a temperature of  $+600^{\circ}$ C to be achieved, while the console monitors temperature, with a digital read-out to  $\pm 0.1^{\circ}$ C.

## 2.2 Calibration

Calibration of the apparatus using standard substances must be carried out before routine measurements can be made. The method recommended involves determining the melting point of several substances (supplied by the manufacturer) for high temperature calibration. For low temperature calibration a section of quartz containing 3-phase inclusions with pure  $\rm CO_2$  was supplied. In these inclusions solid  $\rm CO_2$  was claimed to melt at the triple point of  $\rm CO_2(-56.6^{\circ}C)$ .

The melting point of the 8 solid substances supplied was determined, in some cases a number of times, and a correction curve was drawn up. Irregularities in this curve were related to samples which gave variable melting point readings, depending upon the procedural details. As a check, a similar set of substances, originally supplied to Adelaide University for the same purpose, were determined with the same results. This established that, provided these substances were subjected to the same procedures, the results were reproducible. It is of interest to note that the reproducibility of these standard products was indicated by the suppliers to decrease rapidly with age.

With regard to low temperature calibration, melting of solid  ${\rm CO_2}$  at  $-56.6^{\circ}{\rm C}$  was not observed in the  ${\rm CO_2}$  inclusions in the quartz plate supplied, though indications of a phase change at  $-60.8^{\circ}{\rm C}$  were noted. Another section from the same source, measured on Adelaide University equipment, gave similar indications

at  $-61^{\circ}$ C (Ypma, 1976: private communication). An indication of the sublimation point (theoretically  $-78.5^{\circ}$ C (Roedder, 1962)) was obtained at between  $-78^{\circ}$ C and  $-79.6^{\circ}$ C. Upon heating, measurement of several of these inclusions gave homogenisation temperatures around the critical temperature of  $CO_2$  (31°C). Usually the temperature of homogenisation could be repeated on any given inclusion to within  $\pm 0.2^{\circ}$ C, provided the same approach was made (i.e. heating up or cooling down). These results are taken to indicate that while individual inclusions may vary slightly in a rock section, and independent of the absolute accuracy of the measurements, the equipment is capable of excellent reproducibility.

Since heating fluid inclusions to establish homogenisation temperatures gives only minimum entrapment temperatures, the absolute accuracy of instrumental readings is not critical, particularly as corrections for pressure may be highly approximate. With this calibration completed, it was considered, therefore, that the apparatus was suitable to commence investigations.

#### 3. RESULTS OF FLUID INCLUSION STUDIES

As well as carrying out test measurements on fluid inclusions in miscellaneous rock samples, a number of rocks from specific areas in South Australia were selected so that experience would be gained in examination of a wide range of samples and the applicability of fluid inclusion studies assessed. These samples included pegmatites from Olary, auriferous quartz veins from Wadnaminga, barites from Mount Frome, and one from Oladdie, granites from the Eyre Peninsula and sandstones from the Cooper Basin.

In all cases thick sections, polished on both sides, were prepared for study. In most instances repeat determinations of the homogenisation temperature on the same inclusion gave closely similar results, usually to within a few degrees, but different inclusions in the same section, and even in the same crystal, did not always agree. Consequently a range of temperatures, representing the lowest to the highest has been recorded.

## 3.1. Olary Pegmatites

The Olary region is abundant in a wide variety of pegmatitic rocks (Campana and King, 1958), from thin sheets and stringers to huge dykes and sills. Some of these may be of metasomatic origin, others merge with granite, while others have definite cross-cutting relationships with the country rocks.

The two samples examined had originally been submitted as part of the Olary Feldspar Project (Project 1/1/170); one sample had a graphic granite texture, the other being perthitic.

Sample: P207/75; TS35348. Graphic granite/pegmatite; 1.3 km West of Ameroo Hill.

Inclusions in the slightly strained quartz are not abundant and are generally small, up to about 10 to 20 µm. Much smaller inclusions tend to lie in planes, perpendicular to elongation of the quartz, possibly related to mild shearing. The former types tend to be less regular in shape and are possibly primary, particularly those with some indication of negative crystal shape. Inclusions found include 2-phase and 3-phase types, comprising liquid with gas bubble; and liquid, gas bubble and cube-like or rectangular solid (probably salt(NaCl)), respectively. The relative size of bubbles and solid phases to inclusions appeared constant. One larger, isolated inclusion was found to contain a

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gas bubble and two birefringent crystals (anhydrite?).

Heating of this section gave a range of homogenisation and decrepitation values that is, different inclusions gave different values, as explained earlier. The inclusion with two solids decrepitated at around 345°C before the salts dissolved: in other inclusions no change was noted in the solid phases at up to 442°C. The indications are that these solids could be NaCl or anhydrite, but not KCl. However, in one probably secondary inclusion, the solid phase disappeared at 237°C, suggesting KCl.

In general homogenisation (in the liquid phase) ranged from  $228^{\circ}\text{C}$  to  $423^{\circ}\text{C}$ , with decrepitation ranging up to  $427^{\circ}\text{C}$ . The minimum temperature of formation of the quartz is thus in excess of  $420^{\circ}\text{C}$ .

Sample: P292/75; TS35350. Perthitic pegmatite; 4 km West of Dome Rock Copper Mine.

There are a number of irregularly-shaped small inclusions ( $\sim 10 \mu m$ ) with gas bubbles, displaying homogenisation temperatures ranging from 233°C to 383°C.

## 3.2. Wadnaminga Quartz Veins

The quartz veins at Wadnaminga (Olary area) are of interest because of their association with gold. Sections were prepared from four samples (submitted as part of Project 1/1/176), two from unmineralised veins and two from mineralised veins.

In general, numerous, fairly small inclusions are present. Gas bubbles vary from occupying a small portion of the liquid inclusions to almost filling them, with both primary and secondary inclusions being represented. Homogenisation occurred in the liquid phase, by shrinkage of the gas bubbles. The highest minimum quartz-crystallisation temperatures have been determined as from  $435^{\circ}$ C up to  $504^{\circ}$ C. The highest homogenisation temperatures on secondary inclusions ranged from  $190^{\circ}$ C to about  $357^{\circ}$ C, possibly indicating a post-crystallisation metamorphic event.

Three of the four samples were found to have a solid phase also present in some of their inclusions (i.e. 3-phase inclusions) this being identified as salt (NaCl) and it is thus likely that the inclusions are essentially of H<sub>2</sub>O (liquid and gas). Freezing experiments indicated the two-phase inclusions to have salinities of 15-20% (by wt.), the three-phase varieties being at least 26% NaCl. The minimum indicated pressure is around 340 bars, which in turn suggests that the correction required on the homogenisation temperature would be relatively insignificant.

Individual details are as follows:

Sample: A; TS35364. Unmineralised quartz vein.

Numerous small two-phase inclusions with variable degree of fill; possibly secondary. Less abundant, larger and usually three-phase inclusions; (i.e. with salt cubes) possibly primary. (homogenisation temperature of both types ranges from  $220^{\circ}$ C to  $>400^{\circ}$ C. Highest homogenisation temperature recorded:  $480^{\circ}$ C.

Sample: B; TS35365. Mineralised quartz vein.

All inclusions, as well as those containing salt cubes, lie in planes, probably of secondary origin.

Homogenisation range 200°C-357°C.

Salinity; ∿15% NaC1 to >26% NaC1.

Sample: C; TS35366. Mineralised quartz vein.

Numerous two-phase and a few three-phase (NaCl) inclusions. Some inclusions have negative crystal shape, and may thus have a primary origin.

Homogenisation range:  $200^{\circ}\text{C}-350^{\circ}\text{C}$  for secondary inclusions; up to  $434^{\circ}\text{C}$  for primary ones.

Salinity: ∿10% NaCl to >26% NaCl

Sample: D; TS35367. Unmineralised quartz vein.

Numerous, small inclusions, two-phase only (liquid and gas) with variable degree of fill. Some display negative crystal shape (primary).

Homogenisation range:  $\sim 195^{\circ}$ C for secondary inclusions;  $400^{\circ}$ C- $504^{\circ}$ C for primary inclusions.

Salinity: ~20% NaCl.

#### 3.3 Mount Frome Barites

The Mount Frome area provides one of the more important barite deposits in the State. Several samples from this area, submitted for study under report MP 149/76, were further examined using the microthermometry apparatus.

Originally the clearest, coarsest-grained barites were selected, but subsequently sections were made from finer, more milky varieties. In general the clearer the host crystal, the better the conditions for viewing fluid inclusions, but the fewer the inclusions there are likely to be. Although thick sections are normally preferred for fluid inclusion studies (to increase the numbers of inclusions available for microthermometry and to permit a standardisation of thermal gradients) it was found that thinner sections of the milky barites greatly enhanced viewing conditions.

In general, fluid inclusions are relatively abundant but difficulty was found with some samples in distinguishing primary from secondary types. Usually the larger less regular inclusions yielded slightly higher homogenisation temperatures - up to about  $340^{\circ}\text{C}$  - while the smaller, more equant inclusions, some with negative crystal shape gave temperatures about  $256^{\circ}\text{C}$  at the most. Decrepitation was detected as low as  $300^{\circ}\text{C}$ . rising to a hiatus at up to  $400^{\circ}\text{C}$ .

Salinities (assuming NaCl) were found to be from 2 to 21% by wt. with indicated pressures of up to, and possibly over 100 bars, though mostly much less. On this basis temperature corrections are minimal.

Individual details are as follows:

Sample: P1812/75; TS(unnumbered) Grey and white mottled barite.

Two-phase inclusions, homogenising in the liquid phase.

Homogenisation range:  $95^{\circ}\text{C-}137^{\circ}\text{C}$  for possibly primary inclusions;  $228^{\circ}\text{C-}266^{\circ}\text{C}$  for secondary inclusions.

Strong decrepitation of the sample (possibly indicating filling temperature) took place at 286°C. Cooling tests failed to cause nucleation of ice.

## Sample: P1825/75; TS35480 White to semitranslucent barite.

Few good inclusions; generally irregular, two-phase secondary inclusions, with vapour bubble 2-5% of total volume, homogenising in liquid phase. Possibly primary inclusions with larger gas phase  $(20\% \text{ vol.} - \text{CO}_2?)$ 

Homogenisation range:  $116^{\circ}\text{C}-266^{\circ}\text{C}$  for secondary inclusions. Primary inclusions uncertain:  $100^{\circ}\text{C}-244^{\circ}\text{C}$ ?

Salinity = 7% NaCl.

## <u>Sample: P1830/75; TS35481</u> White barite.

Generally fairly large, irregular, two-phase inclusions, parallel to cleavage faces and thus probably secondary.

Homogenisation range:  $145^{\circ}\text{C}-220^{\circ}\text{C}$  for secondary inclusions. Primary inclusions less certain:  $110^{\circ}\text{C} - 158^{\circ}\text{C}$ ?

Salinity ≡ 2% NaCl.

## Sample: P1832/75; TS 35482 White barite.

Numerous two-phase inclusions, a few possibly with a small, solid third phase present. Some inclusions lie parallel to cleavage planes (i.e. secondary); others with negative crystal form appear to be primary.

Homogenisation range: 195°C - 240°C (large, irregular inclusions) 192°C - 277°C (small, equant inclusions)

Freezing produced ice, plus a second solid phase ( $CO_2$  hydrate?). Salinity uncertain: in one inclusion indicated to be 23% NaCl, in others much less, but the probable presence of  $CO_2$  makes salinity uncertain.

#### Sample: P1837/75; TS35483 Coarse, white barite.

Fairly numerous two-phase inclusions, many being largish and irregular (probably secondary), some being small (primary?). One or two inclusions contain a large 'dark' vapour bubble, probably of CO<sub>2</sub>.

Homogenisation range: 181°C - 278°C (small (primary?) inclusions)
156°C - 302°C (large, irregular (secondary?) inclusions)

Salinity ≡ 3% NaCl.

## Sample: P1853/75; TS35484. White to semitranslucent barite.

Numerous two-phase and some three-phase inclusions with small gas bubbles. In some three-phase inclusions the solid appears to be more cubic and isotropic (NaCl), but in others the solid appears more rhombic in outline and shows apparent variation in relief with rotation of the stage (calcite?). Rarely, the solid phase is less voluminous than the gas phase. The distinction between primary and secondary inclusions is uncertain though the majority are probably secondary, with some of the larger, irregular ones being possibly primary.

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three-phase inclusions: Homogenisation range:

205°C? (Decrepitation √300°C)

two-phase inclusions:

248°C - 256°C (small, regular inclusions) 261°C - 320°C (large, irregular inclusions) 307°C - 341°C (equant, negative crystal shape)

In some equant inclusions, vapour was still present when the sample 'exploded' at 379°C. No freezing tests were carried out.

Sample: P1856/75; TS35485 White to pale pink barite.

Two-phase inclusions ranging in size and shape as in P1853/75, with uncertainty as to primary/secondary, but all homogenising in the liquid phase.

Homogenisation range:

155°C - 237°C (large irregular inclusions) 261°C - 325°C (small, equant, negative crystal shape)

Decrepitation: 300°C increasing to 400°C.

Salinity = √21% NaCl.

## 3.4. Barite Sample from Oladdie

A thick section was prepared from sample P289/75, a pink barite, intergrown with quartz and iron oxide-stained dolomite. The barite itself was not suitable for study but some inclusions were tested in the intergrown quartz and in a small area of late clear quartz. The former gave temperatures for homogenisation in the liquid phase of up to 280°C, the latter being around 242°C.

#### 3.5. Eyre Peninsula Granite

Sections of 4 samples of Eyre Peninsula rose-red granite were subjected to microthermometric examination, principally in an attempt to determine the cause of fading in this granite. A summary of the results from 3 samples has already been presented (see Report MP 1491/76 (Part 1)). This established that low-temperature filling had recurred in some inclusions, with a general homogenisation temperature (minimum trapping temperature) of "up to 321°C" (316°C when corrected for instrumental error) for two-phase fluid inclusions in the quartz. Decrepitation was becoming pronounced when heating reached about 300°C.

Subsequent examination of a fourth sample showed a similar reaction, with some inclusions homogenising below 180°C and decrepitation occurring at up to 274°C.

#### 3.6. Cooper Basin Sandstones

A number of samples were originally submitted (supplied as part of Project 1/1/159). These consisted of core-plugs taken from the Tirrawarra Sandstone, a thick fluvial sand which occurs near the base of the Permian succession in the Cooper Basin. In Progress Report No. 3 (MP 1/1/159) it was postulated that secondary quartz overgrowths had developed relatively early in the history of these sandstones and fluid inclusion studies were aimed at assessing the conditions of formation of these overgrowths.

Thick sections, prepared from four core samples, were initially studied. In two samples (Gidgealpa #7 (7350') and Tirrawarra #8 (9570')) no suitable inclusions were found in the overgrowth zones, the inclusions in the original quartz grains being probably of primary origin. The results from the other two samples, Mudrangie (10135') and Coonatie #1 (10204'), though clearly insufficient to support firm conclusions, suggest that the mantling of quartz grains commenced at relatively low temperatures but continued as moderately high temperatures were reached in aqueous solutions with possibly very low salinity, but containing  $CO_2$ .

The sections show detrital grains of quartz with secondary overgrowths marked by lines of fluid inclusions. Primary inclusions within the original grains are numerous. The grains also contain cross-cutting planes of secondary inclusions which terminate at the original grain boundary. The overgrowth zones also contain some inclusions. The details are as follows:

## Sample Mudrangie (10135'); TS35509

Primary inclusions displayed shrinkage of the gas bubble to homogenise in the liquid phase from  $140^{\circ}$ C to  $166^{\circ}$ C, while secondary inclusions in the original quartz grains homogenised at  $230^{\circ}$ C.

Inclusions in the overgrowth quartz homogenised at from  $224^{\circ}\text{C}$  to  $310^{\circ}\text{C}$ , but inclusions at the overgrowth boundary homogenised by vaporisation of the liquid phase (homogenisation at the critical point) at  $288^{\circ}\text{C} - 320^{\circ}\text{C}$ . The critical temperature for pure water is  $374^{\circ}\text{C}$ , this being generally raised by the presence of salts in solution and lowered by the presence of  $\text{CO}_2$ . Thus the presence of some  $\text{CO}_2$  is indicated.

The results of freezing tests on primary inclusions indicate a very low salinity.

## Sample Coonatie #1 (10204'); TS35510.

Some primary? inclusions were found to contain solid phases (not identified) as well as a gas phase, homogenisation occurring in the range  $269^{\circ}$ C to  $308^{\circ}$ C. Homogenisation of inclusions in the overgrowth zone was not determined, decrepitation occurring between about  $240^{\circ}$ C and  $334^{\circ}$ C. One inclusion at the overgrowth boundary homogenised at about  $125^{\circ}$ C.

## 4. SUMMARY AND CONCLUSIONS

It has been shown that the acquired microthermometry apparatus is capable, as expected, of covering the range of temperatures necessary to carry out heating/freezing tests on fluid inclusions in various rock types. Furthermore, the recorded temperatures appear to show good reproducibility.

The results of a preliminary series of tests indicate that valuable information, of a type otherwise unavailable, is to be had with regard to the petrogenesis and possibly the classification of barites, pegmatites etc. It may also be possible to study the history of diagenesis etc. in sedimentary rocks, and to successfully apply microthermometry to more specific problems, such as the cause of fading in granite.

There is in fact no reason to suppose that other applications might not be productive, for instance regional studies of ore and mineral genesis in complex areas such as Mount Painter, or studies of the genesis of particular mineral deposits. Indeed, 'new' applications might even be considered for investigation, such as the characterisation of quartz grains in soils and sands, or of gemstones.

In selecting topics or individual rock types for study, consideration must be given to certain limitations. The method is time-consuming and for various reasons should be used on several different sections. The field relations of samples should be known to allow for possible temperature corrections, account for 'overprinting' effects, or to establish the history and relative age of the host minerals. Finally, it must be remembered that ultimately the results on the temperatures of formation of minerals rest upon the presence and recognition of primary fluid inclusions - which is not always possible. There is, however, virtually no other way of studying the original mother fluids from which minerals of interest have crystallised.

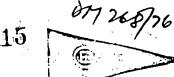
#### 5. RECOMMENDATIONS

It is recommended that during 1976-77 the methods outlined be applied on a project basis to one or more selected petrogenetic problems. These are suggested as follows:

- 1) Barites from the Flinders Ranges.
- 2) Gold-bearing quartz veins from the Adelaide geosyncline.
- 3) Olary pegmatites.
- 4) Copper-mineralisation in diapiric structures (e.g. Mountain of Light deposit).
- 5) Rocks from Mt. Painter.

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## FLUID INCLUSION STUDIES OF S.A. ORES

ROCKS AND MINERALS

Progress Report No.2

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#### FLUID INCLUSION STUDIES OF S.A. ORES, ROCKS AND MINERALS

#### 1. INTRODUCTION

Following on the fluid inclusions studies carried out in the first half of 1976 (see Progress Report No.1), it was agreed to continue studies in the period 1976/77 under the same project, but in two sections. Originally these sections (1/1/182A "Industrial Minerals" and 1/1/182B "Metallic Minerals") had an equal allocation of funds.

As it was anticipated from preliminary discussions that work on barite would be extended, (under "Industrial Minerals"), some initial investigation of further samples already to hand was carried out and a limited number of measurements were made. However, no official request for work was submitted and ultimately it was requested instead that the study of barite be deferred until other samples were available and more recent field information had been co-ordinated.

During this period the opportunity was taken to recalibrate the microthermometry equipment (see below). Subsequently a number of rock samples from Mt. Painter were received for examination under section 1/1/182B ("Metallic Minerals"). After partial completion of this part of the project, it was agreed to carry out fluid inclusion studies under section 1/1/182A on samples of smoky quartz (currently being analysed under a separate request for service). It was also agreed that the funds remaining in this section should be used in continued studies of Mt. Painter specimens. At a later date a small batch of samples, more specifically related to Mt. Gee, was received in an attempt to resolve certain problems associated with the Mt. Painter samples (also discussed below). As a result, the remaining time was used to carry out heating tests on some of these Mt. Gee samples, and no freezing runs were made. Likewise no low-temperature investigations have been made of the smoky quartz.

#### 2. RECALIBRATION OF THE EQUIPMENT

According to the manufacturers of the Chaixmeca microthermometry apparatus, recalibration should be carried out each year. As the apparatus, had been calibrated only once (on arrival), no indication of possible variation with use was known, and for this reason alone it was considered necessary to repeat the calibration as recommended, at least for heating tests. Generally, there is little need for great accuracy during heating operations, reproducibility being much more desirable. None-the-less, when inclusions homogenise at or close to critical points, the deviation of the measured value from the theoretical one for a pure substance (e.g. water) can be used to indicate the nature and amount of dissolved impurities (salts, CO<sub>2</sub> etc.). Thus errors in measurement cannot be completely ignored.

The original calibration runs had shown irregularities in the correction curve (see Progress Report No.1, section 2.2). Repeat determinations, using the same standards, gave essentially the same results, indicating that the apparatus is relatively free from drift, and that the standards, although claimed to lose quality with age, have not materially changed since receipt.

The accuracy of the correction curve was, however, still considered to be unsatisfactory for low (room) temperatures and hence for the measurement of salinites via the depression of freezing points etc.. Consequently several additional standards of organic compounds were obtained, enabling a satisfactory curve for the range 20-180°C to be produced and allowing a better extrapolation into the freezing range.

#### 3. METALLIC MINERALS - MT. PAINTER HEMATITIC BRECCIA

#### 3.1 General Setting

The rock samples and cores from the Mt. Painter area generally represent the results of the brecciation of a largely granitic basement, with the introduction (from an unknown source) of much iron and lesser amounts of other minerals, including uranium in some. Most of the samples received for study are thus hematitic breccias, though one or two fine-grained clastic sediments, also rich in hematite, are represented. Some previous reports (e.g. MP2714/76) have indicated that hydrothermal conditions were the most likely environment for the formation of the original breccia(s), but reworking to produce sedimentary breccias has also occurred, together with late-stage fracturing tending to obliterate earlier sturctures. The main group of samples relate to the older Radium Ridge Beds, which rest unconformably on the crystalline basement of the Mount Painter Complex, and which are tentatively correlated in part with Sturtian tillites.

The Mount Gee Beds more specifically represent a younger and largely sedimentary series of units with material derived from the older breccia(s). Mineral paragenesis is believed to be similar in both groups, with monazite as an early development, ultimately followed by magnetite and hematite, then quartz (often euhedral) and uraninite, sulphides, etc., ending with fluorite and cementing quartz. Theoretically, therefore, fluid inclusion studies of monazite, quartz and fluorite (the most suitable minerals) should give some indication of the nature of formation of the breccias and of any subsequent changes.

The problematical aspects of these breccias have not, however, been confined to field relations and petrology, as partly foreshadowed in report MP1140/76. As a guide to future considerations the various problems actually encountered are summarised below.

#### 3.2. Problems Encountered

## 3.2.1. Friable and Opaque Nature of Breccias

At the outset, the nature of the breccias themselves has made section-preparation of many a difficult, if not impossible, task. It is not normally advisable to use impregnating agents, since when heated in the section they seriously impair the optics of the equipment. Secondly, the ferruginous nature of the breccias results in up to 90% of some sections being effectively opaque, especially as thicker sections are required for fluid inclusion studies. Of the transparent minerals expected to provide evidence of entrapped fluids (monazite, fluorite and quartz), only quartz was found in

relative quantity, and although duplicates were prepared of a few sections with coarser and more numerous crystals of monazite, very little information was obtained from this mineral (see below). To some extent this must reflect the difficulty in recognising individual crystals of monazite in thick section, and, once identified, in being able to study inclusions in a mineral whose contrasting hardness does not readily permit a suitable polish to be obtained.

## 3.2.2. Abundance and nature of inclusions

With regard to the fluid inclusions themselves, they were found overall to be relatively small and varied from below lum to about 30µm. Most of the homogenisation temperatures were obtained from inclusions in the 5-15 µm range. Gas bubbles and solid phases (when present) tended to be correspondingly small, the latter usually too small for identification, and rarely as more than one phase. The abundance of inclusions also varied, from sections with few, sparsely distributed examples (more common) to a few sections where, in quartz, the inclusions were sufficiently numerous as to be more like a "network" of pores. The inclusions were most commonly rounded to irregular in shape, but many in the former condition could be seen in detail to be partly bounded by straight outlines or to resemble negative crystal shapes partly rounded off, possibly representing modified primary inclusions. In general, therefore, it was seldom possible to find unequivocal examples representing the original mother fluid, though many inclusions are believed to have developed initially as primary phenomena (see also section 3.3).

#### 3.2.3. Decrepitation

Even where, as in rare monazite crystals, few of these problems were encountered, the majority of fluid inclusions failed to show any change on heating to  $400^{\circ}$ C or even  $500^{\circ}$ C, and instead appeared to have already decrepitated.

Prior decrepitation appeared to have occurred in not a few quartz crystals also, thus highlighting the problem of whether the decrepitation (usually resulting from heating at too fast a rate and/or to excessively high temperatures above homogenisation) was a natural or an induced phenomenon. As a natural occurrence this could in theory yield useful information as to how high post-crystallisation temperatures had reached above initial temperatures, and might also in some circumstances distinguish early-formed from late-formed inclusions.

As an induced phenomenon, decrepitation would indicate that the sample had been overheated during preparation, most probably during initial heating of the cut slab, prior to its being bonded to the glass slide. The glue in question was recommended for this work, largely because of its solubility, that is to enable the section, once finished, to be removed by solvent from the glass slide and cleaned. The glue is not sufficiently soluble for cold bonding, however, and must be heated, with the rock slab, to melt at about 150°C. No suitable alternative is known at this stage, so that future section preparation will necessitate an investigation of the problem, possibly by repreparing control sections by cold-mounting and comparing the inclusions in them with those in the present sections. Other possible areas to be considered include the heat generated by cutting the rock slice and by subsequent polishing. The former is not thought to be a likely problem, since the operation water-cooled, and the latter would have local effects, confined virtually to the surface of the polished section.

Decrepitation of, and partial loss of fluid from, inclusions may also occur by the mechanical processes of section-preparation. Normally this would be observed as a series of fractures intersecting inclusions, but subsequent heating can permanently influence results by causing inelastic stretching along virtually invisible micro-fractures(Larson et al,\* 1973) The effect of this is normally to raise the apparent homogenisation temperature. Results similar to those quoted have been observed in some of the present samples and presumably are more likely to be manifest in samples of breccia than in most other rock-types.

Notwithstanding these factors, the presence of fluid inclusions in the current samples, with homogenisation temperatures well below 150°C, is clear proof that even if prior decrepitation of many inclusions has occurred as a result of sample preparation, at least some inclusions have been unaffected. Thus, by examining a sufficient number of samples as in the present case some evidence of the true homogenisation temperature(s) should emerge. Also, in cases where a number of adjacent or related inclusions show similar homogenisation temperatures, these can be assumed to be unaffected by partial decrepitation, which would have caused divergence in homogenisation temperatures.

#### 3.3 Summary of Results

Of the 41 samples of breccia etc. submitted from the Radium Ridge Beds and Mt. Gee Beds, 31 were selected for polished thin section preparation, and in a few cases duplicate sections were made. Two of these polished sections were not examined in detail and 9 were found to be unsuitable for study. The remaining 20 samples were used to determine homogenisation temperatures by heating, but although several may have had inclusions suitable for freezing point studies, no time was spent on this. The results are summarised in Table 1 & 2; individual section descriptions are given later.

During the examination of the various samples it was seldom possible to distinguish between "primary" and "secondary" (or "pseudo-secondary") fluid inclusions. This to a large extent reflects the fact that most of the inclusions were found in quartz, which, being readily capable of recrystallising, does not provide the best environment for preservation. The criteria for recognising primary inclusions are reportedly when they occur in euhedral crystals projecting into vugs, when they relate to original crystal shape (parallel to crystallographic directions, growth or colour bands), and in some circumstances when they form negative crystal shapes. Numerous, small inclusions with negative crystal shape, lying in a single plane are, for instance, usually regarded as secondary, (having formed by "necking down" from a fracture plane), but where larger and more randomly situated they are more probably primary in origin.

The apparently most reliable primary fluid inclusions were found not surprisingly in the Mt. Gee beds, where late quartz had crystallised in cavities etc., as in P306/76. Although other features suggestive of primary origin were noted, for instance in quartz in P308/76, where inclusions were associated with growth zones, no inclusions could be related to all the criteria listed as indicating primary formation. Some crystals of vuggy quartz in fact seemed to display 3 types of fluid inclusion (eg P1304/76). Since the quartz, too, may occur within one sample as separate crystals of more than one generation, the results of homogenisation determinations have been listed in the table under "earlier" and "later" inclusions rather than "primary" or "secondary" etc.. However, it

<sup>\*</sup> Larson, L.T., Miller, J.D., Nadeau, J.E., and Roedder, E., 1973. Econ. Geol., v.68, p.113-116.

must be stressed that there may be more than two generations of fluid inclusions, and that almost inevitably some results will reflect intermediate or transitional stages. This division has been applied also to samples from the Radium Ridge Beds, the inclusions in some of which must be earlier as a whole than those from the Mt. Gee Beds. In general there is less evidence of the primary nature of inclusions in samples from the Radium Ridge Beds, and in some cases it was not even possible to distinguish between early euhedral quartz and late cementing quartz.

In contrast to quartz, such monazites as could be measured appeared to have primary inclusions almost exclusively, and therefore most represent the earliest events in the thermal history of the breccias.

From the tabulated results it is possible to recognise an ill-defined trend or grouping of homogenisation temperatures. Firstly the earlier (and usually primary) inclusions in the Radium Ridge Beds tend to range from 300°C to slightly in excess of 400°C. This is more marked in the East Painter samples, less in the Mt. Gee Prospect, with little evidence of such a range in the Armchair Prospect. Secondly, it appears that in all areas there are both earlier and later inclusions homogenising from about 150°C up to just below 300°C. The very limited evidence from monazite (an early-formed mineral) does not conflict with this.

By contrast, the Mt. Gee Beds show very few instances of temperatures in the  $300^{\circ}\text{C+}$  range. Most inclusions are probably primary and of these the later primary inclusions in the vuggy quartz seem to agree with the earlier setting a general range of  $110\text{--}200^{\circ}\text{C}$  with a few readings reaching up to  $300^{\circ}\text{C}$ . Only two points of conflict are evident: in one area of vuggy quartz in sample Pl304/76, temperatures up to  $412^{\circ}\text{C}$  were recorded; and in late zoned fluorite from P308/76 one well-formed primary inclusion gave a result (reproducible to less than  $0.5^{\circ}\text{C}$ ) of  $412^{\circ}\text{C}$ , with irregular, apparently secondary inclusions in another zone ranging up to  $315^{\circ}\text{C}$ . (See also NOTE p.14.)

The picture which emerges, therefore, is that of brecciation and contemporaneous growth of monazite at moderate to high temperatures, probably reaching to in excess of 400°C. Those conditions were probably maintained until and during the formation of early quartz. Limited evidence from subsequent quartz overgrowths suggests that by this stage the temperature had fallen to the lower range recorded, this fitting in with the evidence from the "later" and secondary fluid inclusions in the quartz. These may be in part contemporaneous with the development of late cementing quartz in other sections but for which no reliable temperatures have been obtained.

Similar low temperatures continued during the formation of the Mt. Gee Beds, but there is some evidence of a very late rise in temperature during the formation of fluorite and some of the vuggy quartz, the reasons for which remain unknown.

With regard to the composition of inclusions, virtually all appear to represent aqueous solutions, the exceptions being P1067/76 and P1112/76 (from the Radium Ridge Beds) which, although also having "normal" inclusions in quartz, locally also contain groups of small inclusions with CO<sub>2</sub> liquid and gas. The quartz in both these instances appears to be late or recrystallised and the inclusions in question in P1112/76 at least appear to be related to fracture planes, inferring that the development of a CO<sub>2</sub>-rich environment was probably a late and local one.

Although no salinity estimates were made, it is reasonable to suppose from the paucity of solid phases that salinities at the time of crystallisation were generally low, but with some variation. The identity of the solid phases also remains uncertain. Some apparently isotropic solids with square sections may represent salt (NaCl), those with noticable birefringence being possibly potassium chloride.

#### 3.4. Detailed Descriptions

#### 3.4.1. Mt. Painter Hematitic Breccia

Sample: P1062/76

Hematitic breccia, East Painter. No thin section, nor polished thick section prepared.

Sample: P1065/76; TS37242.

Hematitic breccia; East Painter.

Traces of monazite have been recorded in thin section but none was found in polished thick section. However, the thermal history recorded by inclusions in quartz appears to be complex. The quartz tends to form large, partly recrystallised areas, with some euhedral crystals reaching up to 2mm across. The enclosed fluid inclusions range from irregular to regular, elongate (up to 100 µm long) to small and equant The last-mentioned, with very small gas bubbles, give in outline. an indication of homogenisation starting as low as 158°C; but some equant inclusions, ranging to hexagonal in outline(negative crystal shape) and probably of primary origin, have homogenisation temperatures in the range  $373-412^{\circ}\text{C}$ , with decrepitation from  $412^{\circ}\text{C}$  to about  $470^{\circ}\text{C}$ . The larger, less regular inclusions tend to show higher relief, and to be multi-phase, with relatively larger gas bubbles, and birefringent solid phases (as well as inclusions of ?hematite). The solids tend to dissolve around 336°C, resolidifying on cooling to 234°C; but some solids dissolve below 300°C (KCl?). The gas bubbles indicate high temperatures of homogenisation, mostly from 337°C upwards, some of them appearing to almost reach the homoginisation point then expanding at varying rates. In some cases this would appear to result from decrepitation (range 371-460°C) but in others the slow expansion of the bubble with failure to form a single phase, even when heated to 524°C, leaves doubt as to their temperature of origin. these inclusions may be secondary, but a few which are smaller and possibly primary, also yield high temperatures in the range 373-412°C. In one part-euhedral crystal of quartz a group of small, well-spaced inclusions with negative crystal shape lying in a plane parallel to a pyramid edge, appeared to have already decrepitated. These seemingly primary inclusions must presumably have had lower homogenisation temperatures than some of the secondary inclusions. The highly elongate inclusions appear to have formed by "necking down" in shear or fracture planes in strained areas of quartz. Few of them yield definite homogenisation temperatures and many have no evidence of more than one phase. One was found to homogenise at 128°C, while one or two others appeared to have a phase expanding at temperatures around 365-375°C(decrepitation?). It is possible, therefore, that there are several generations of inclusions represented and some of the quartz would appear to have formed or recrystallised at fairly high temperatures and salinities.

#### Sample: P1067/76; TS37243

Granitic breccia; Underground East Painter.

Again no obvious monazite was found in polished thick section. Most of the quartz forms small subhedral to anhedral interstitial crystals, the inclusions in this sample also suggesting a complex thermal history. Local areas have small primary inclusions with two phases homogenising in the liquid phase at low temperature (from 22.5-24.5°C), and presumably representing CO<sub>2</sub>-rich liquid.

Generally, however, small primary inclusions show homogenisation temperature ranges of 193-245°C and 324-387°C with decrepitation from 381° to 479°C. As in the previous sample some of the gas bubbles display a fairly quick, but not rapid, expansion to virtually fill the whole inclusion at temperatures in excess of 340°C. Rapid expansion at 479°C is presumed to be a result of decrepitation.

#### Sample: P1071/77; TS37244.

Hematitic breccia; Underground East Painter.

As before, quartz is post-monazite, but more abundant in this sample, with both large and small areas, ranging from euhedral to anhedral. One relatively large crystal of monazite was found. Some of the quartz contains numerous small inclusions, often in bands or zones apparently parallel to crystallographic outlines (growth lines), yet irregular in shape and commonly without gas bubbles. These homogenise from 149°C to 200°C, with some up to 241°C. Slightly larger inclusions, some with negative crystal shape and occasionally also with a solid phase (NaCl?) homogenise from 343-402°C. The solid phase in one inclusion seemed to have dissolved by about 270°C, and in a second inclusion by about 320°C, but in others it was unaffected by temperatures up to 510°C. Larger secondary and possibly also modified primary(or pseudosecondary) inclusions display a wide range of thermal history as previously described. Temperatures as low as 105°C were noted, with decrepitation or sudden gas bubble expansion starting at 195°C. Homogenisation occurs at various temperatures up to 506°C. When present the solid phase has moderate briefringence and generally shows no change of state with heating (KC1?).

The monazite crystal contained several generally elongate inclusions (up to 30  $\mu m$ ) but these were optically unsuitable for study. Structures closely resembling small (3-4  $\mu m$ ) dark bubbles could be discerned, but heating to about 520  $^{\circ}$ C produced no visible change in them. The evidence again suggests a wide range of temperatures and salinities.

#### Sample: P1073/76; TS37245.

Finely laminated, hematitic siltstone; Underground East Painter.

The sample is largely composed of detrital quartz, cemented by secondary quartz. In some instances it is uncertain whether isolated areas of coarser quartz represent relict clasts or recrystallised quartz. Coarser detrital grains occasionally display small primary inclusions homogenising in the range  $299-308^{\circ}$ C, with a solid phase being unaffected by heating to  $\sim 345^{\circ}$ C. However, one large area of quartz, possibly a fragment of a euhedral crystal now apparently transected by the

matrix, was found to give a homogenisation temperature (?primary) of about 390°C, but the small solid phase had dissolved before this, possibly by about 300°C. Other (later?) inclusions indicated lower homogenisation temperatures, circa 188°C. No monazite was detected, and the late cementing quartz was much too fine for fluid inclusion studies.

#### Sample: P1082/76

Hematite-bearing siltstone; Underground East Painter.

Quartz and rare grains of monazite not generally suitable - no polished thick section prepared.

#### Sample: P1085/76; TS37247

Arkose; Underground East Painter.

This rock contains some large clastic quartz grains, plus late interstitial quartz, but no suitable grains of monazite. The late cementing quartz was not found to be suitable for study in thick polished section. The coarse clasts themselves are probably of two generations, the more iron-stained from granitic basement and the clearer, sometimes part-euhedral, probably from related breccias. Inclusions in the latter range from well-formed primary? types to slightly more rounded (but probably also primary) ones. Homogenisation of gas bubbles varies from 423°C for well-formed inclusions down to 234°C for others. Solid phases generally dissolve on heating, but results tend to be inconsistent. Repeated heating on one well-formed primary inclusion caused solution of the solid phase between 137°C and 145°C, with crystallisation at 114°C.

#### Sample: P1087/76; TS37248

Hematitic breccia; Underground East Painter.

The polished thick section was found to be unsuitable for study.

## Sample: P1088; TS37249.

Finely laminated siltstone; Mt. Gee Prospect.

Quartz is highly variable, from fine-grained detrital to coarse clasts to secondary or partly recrystallised. Monazite is more abundant than usual but small and fragmentary. Inclusions in the coarser (?recrystallised) quartz have strong relief, with gas bubbles homogenising from 160°C to approximately 312°C, and the more obviously secondary inclusions ranging from 334°C to 370°C. However, some narrow elongate inclusions decrepitate at about 241°C. In one monazite fragment a lenticular inclusion displays homogenisation at the critical point of 376°C. Moderate to high temperatures of formation are thus indicated, and if the evidence from the monazite is typical, very low salinities are also indicated.

#### Sample: P1089/76B; TS37250

Hematitic breccia; Mt. Gee Prospect.

Locally there are moderate amounts of monazite, mostly as fractured crystals (or possibly aggregates). These tend to enclose regularly distributed fluid inclusions (and some of ?hematite) ranging up to 20 µm in length and generally angular to prismatic in outline. The The relatively high relief and dark interior of most of them prevent the enclosed bubble-like shapes from being positively recognised as such in most cases, and other inclusions appear to have already decrepitated. However, homogenisation temperatures of 305-334°C are indicated, with some decrepitation around 370°C. Heating above this level tends to inhibit bubbles from reverting to normal on subsequent cooling.

## Sample: P1091/76; TS37251

Hematitic breccia; Mt. Gee Prospect.

This sample is largely of opaque material and no suitable crystals of quartz or monazite were encountered.

#### Sample: P1092/76; TS37723

Hematitic breccia; Mt. Gee Prospect.

Much of the quartz is granulated, but there are some coarser unstrained masses (resembling late quartz but not obviously replacive) with areas or zones of inclusions. In some places their distribution is suggestive of growth lines, in the host, though most inclusions are irregular and locally even become linked to form a network effect. Small individual fluid inclusions with negative crystal shape appear to be randomly distributed in these areas. Few were found suitable for study but those investigated (probably primary) gave homogenisation values around 340-350°C. Although local concentrations of monazite are to be found in this rock, no suitable material was encountered in polished thick section.

## Sample: P1094/76; TS37724

Hematitic siltstone; Mr. Gee Prospect.

No suitable quartz or monazite could be found.

## Sample: P1095/76; TS37725

Hematitic breccia; Mr. Gee Prospect.

Some sections show sporadic, but at times relatively large anhedral to subhedral fragments of monazite. Although few have noteworthy fluid inclusions some display numerous small to medium-sized inclusions, mostly dark (decrepitated?) or clear but without evidence of gas bubbles etc.. The occurrence in some monazites of numerous fine cracks, some apparently intersecting inclusions, lends weight to the view that decrepitation may be widespread, possibly a mechanical effect of brecciation. However in one large, entirely euhedral crystal full of inclusions, only one inclusion appeared to have a gas bubble, and heating to 407°C produced no change. From the few indications available, homogenisation occurs at temperatures in excess of 200°C, with possible

decrepitation in one instance at 245°C. Homogenisation may occur at higher temperatures (up to 300°C recorded) but reheating to this level gave consistently higher results, indicating that some form of inelastic stretching or leakage caused by the opening up of tiny cracks leading from inclusions was giving false readings.

## Sample: P1097/76; TS37726.

Breccia; Mt. Gee Prospect.

The section was too friable to be of any use.

## Sample: P1098/76; TS37727.

Hematitic breccia; Mt. Gee Prospect.

No suitable material present.

## Sample: P1099/76; TS37728

Porous detrital sediment; Mt. Gee Prospect.

Section too friable.

#### Sample: P1100/76; TS37729

Hematitic breccia; Mt. Gee Prospect.

Monazite is generally very limited and with no suitable inclusions. Quartz, too, is almost inclusion-free. Most of it is probably of late replacive origin, though it varies from coarse anhedral crystals to a network of partly euhedral crystals. Some of the former crystals have what resembles an overgrowth zone with possibly primary (rel. to overgrowth quartz) inclusions homogenising between 97°C and 130° and secondary inclusions related to fractures also low (87°C). Other inclusions (pseudosecondary?) have higher temperatures (237-267°C). There may also be solid phases in a few inclusions but only one example was recognised with certainty, this dissolving between 241°C and 248°C.

## Sample: P1101/76; TS37730

Hematitic breccia; Mt. Gee Prospect.

The section was found to be friable and was not studied in detail.

## Sample: P1103/76; TS37731

Laminated hematitic siltstone; Mt. Gee Prospect.

There are some "horizons" of secondary or recrystallised quartz, while even in some of the laminated areas the coarser quartz crystals appear to be mantled with secondary growths. Very few fluid inclusions suitable for measurement were found, however, and none of the limited monazite was suitable. The inclusions in quartz all appear to be related to the secondary overgrowths and give homogenisation temperatures starting possibly as low as 80°C, but mostly over 115°C and ranging up to about 156°C.

#### Sample: P1105/76; TS37732.

Hematitic breccia; Mt. Gee Prospect

The section of this sample was also friable and not studied in detail.

#### Sample: 1107/76; TS37733

Hematitic breccia; Mt. Gee Prospect.

No quartz suitable for fluid inclusion studies was found and although fluorite was present it was too fine grained to be of value either. Careful searching revealed a fragment of monazite with small fluid inclusions indicating a homogenisation temperature of between  $200^{\circ}\text{C}$  and  $250^{\circ}\text{C}$ , with some decrepitation at not much higher temperatures.

#### Sample: 1108/76; TS37734

Silicified breccia; Mt. Gee Prospect.

Although brecciated, some of the monazite crystals in this sample still retain virtually euhedral outlines. Moderate-sized inclusions, lying parallel to margins and presumably of primary origin, are by no means sparse, but many display no gas bubble and some may indeed have already decrepitated. The evidence from a few inclusions indicates homogenisation occurring in the range 280°C to about 300°C.

Two inclusions resembling gas bubbles showed no changes when heated to about  $515^{\circ}\mathrm{C}$ , possibly a result of partial decrepitation. The monazite in this sample may thus have been subjected to higher post-crystallisation temperatures.

The evidence from the quartz, most of which post-dates the monazite, tends to support this view, with inclusions showing homogenisation phenomena between  $364^{\circ}\text{C}$  and  $379^{\circ}\text{C}$ . One secondary inclusion gave a value of  $165^{\circ}\text{C}$ .

#### Sample: P1110/76

Hematitic breccia; Armchair Prospect. No thin section prepared; thick polished section unsuitable.

#### Sample: P1112/76; TS37736

Hematitic breccia; Armchair Prospect.

The quartz in this sample is anhedral, slightly strained and in most instances appears to be secondary. Fluid inclusions occur mostly in concentrations defining bands and planes, some cutting across crystals (annealed fractures?), some radiating and others following old curved grain boundaries. The fluid inclusions themselves vary in type from smaller regular forms to more elongate and irregular types. Locally small inclusions with negative crystal shape lie in the same plane as larger, irregular ones. Consequently no clear sequence can be established for the thermal history of this sample. Possibly the earlier inclusions are those homogenising in the range 200-267°C, with the later and less regular ones at 68°C ranging to 186°C, with decrepitation in some at 268-270°C. Inclusions with CO<sub>2</sub> liquid and

gas components homogenise in the liquid phase at between 23.7°C and 24.6°C.

#### Sample: P1113/76; TS37737

Granitic rock/Hematitic breccia; Armchair Prospect.

In this sample neither monazite nor quartz was suitable for fluid inclusion studies.

#### Sample: P1114/76; TS37738

Granitic breccia; Armchair Prospect.

Most of the polished thick section was too friable for study.

#### Sample: P1118/76; TS37739

Hematitic breccia; Streitberg Ridge Prospect.

In view of the greater amounts of monazite present in this sample two polished thick sections were prepared, but only two crystals afforded information on homogenisation temperatures. A number of relatively very coarse subhedral to anhedral fragments appear to have small( $\sim$ 5µm) oval to prismatic inclusions, but most are dark (decrepitated?) or difficult to examine internally. Nearly all the remainder are single-phase inclusions. One of these apparently primary inclusions was found to have a tiny gas bubble, homogenising at about 96 °C. In the second monazite some irregular (secondary?) inclusions, apparently with very small solid phases, failed to display proper homogenistation, whereas an inclusion with a gas phase was found to homogenise at about 210 °C.

Crystals of fluorite, though readily recognisable in this sample are small and with few inclusions, none suitable for study. In contrast, some crystals of quartz, usually angular and possibly of clastic origin, are unstrained and sometimes display growth lines marked by horizons of inclusions. These vary even in adjacent zones from irregular types with higher relief to more equant or euhedral examples with lower relief. The former, which are generally coarser, may already be decrepitated; certainly no gas bubbles are present, and heating produces no effects. By contrast, the latter types generally have a small gas bubble, homogenisation occurring mostly below 100°C, but also at temperatures up to 138°C. Further heating appears to have no effect on such inclusions until approximately 460°C, when decrepitation begins, reaching a peak about 480-495°C. In some this takes the form of a quickly expanding bubble filling the inclusion and causing the rim to appear heavy (i.e. increasing the relief). This may also be accompanied by the formation of tiny fractures running out from the inclusion, providing conclusive proof of decrepitation. Thus, although only low homogenisation temperatures have been recorded in this sample some of the quartz at least may have been formed at much higher temperatures.

#### Sample: P1124/76:

Chloritised granitic breccia; Streitberg Ridge Prospect.

No section prepared

Sample: P1129/76;

Granitic breccia; Hodgkinson Prospect.

Not examined

3.4.2. Mt. Gee Beds

Sample: P306/77; TS38151

Vuggy quartz; East flank Mt. Gee Ridge.

Quartz crystals are mostly very thin and form sub-parallel to slightly radiating groups, fluid inclusions (small) mostly occurring in irregular patches and being presumably of primary origin. Homogenisation ranges from  $136^{\circ}\text{C}$  to  $206^{\circ}\text{C}$ .

#### Sample: P308/77; TS38153

Zoned fluorite and interbanded quartz; East flank Mt. Gee Ridge.

The fluorite varies from pale green or almost colourless to purple. In the section examined the former areas display only rare inclusions, apparently in the form of small tetahedra. Most of these also appear dark internally, but whether a result of high relief or decrepitation could not be determined. However, one partly dark multiple inclusion was found to have a heavy-rimmed bubble representing a relatively high degree of fill, with a high homogenisation temperature (in the liquid phase), at 352°C. The consistency of this value on a number of repeat determinations tends to support this as being a genuine indication of the minimum temperature of formation. The purple areas in contrast contain occasional planes of irregular inclusions with lower apparent relief. Most of these look to be typical of medium-sized secondary inclusions and many have low homogenisation temperatures (~107°-117°C), though some groups (possibly modified) have higher ones (257-293°C). Smaller secondary inclusions tend not to have gas bubbles, but those that do give even higher figures (max. recorded = 315°C).

The associated quartz varies in crystal size and has certain horizons marked by concentrations of very small inclusions, presumably representing growth levels. Local horizons have rare, coarser inclusions of more suitable size for study, but all are irregular in shape and very few contain more than one phase. Two inclusions with gas bubbles gave values of 182°C and 207°C respectively.

#### Sample: P261/76; TS38148

Laminated chert and interbanded quartz; Top ridge North of Mt. Gee.

Although some vuggy-type quartz is present, few of the inclusions, present in local zones, are large enough for study. When coarser, most are either dark or have a heavy margin, possibly it is believed, resulting from decrepitation, particularly as heating to 440°C produces no changes whatever. Only one suitable inclusion with a gas bubble was noted, this homogenising at 163°C. There was found to be some evidence of decrepitation in other inclusions between 300°C and 350°C.

Sample: P263/76; TS38146

The quartz in this sample varies from anhedral to euhedral. In the latter condition growth tends to resemble vuggy quartz. Such crystals are commonly clouded with inclusions and may radiate out from clear, perfectly euhedral crystals, which probably acted as nucleation centres. Inclusions in the cloudy quartz are generally unsuitable for study, but the clear areas, although abruptly delineated, may have well-spaced inclusions of variable shape, some with gas bubbles, and rarely also a solid phase with moderate birefringence. The general homogenisation temperature range is 123°C to slightly in excess of 163°C. In one area some inclusions had bubbles surviving up to 276°C, but not before others had decrepitated. Information on the solid phases was difficult to obtain because of their small size and the effects of decrepitation.

Sample: P1304/76; TS38145

Hematitic breccia; North of "The Waterfall".

No suitable monazite appears to be available for study. Quartz is present as medium/coarse grains, possibly as partly or slightly recrystallised remnants with overgrowths (uncertain), and also as areas of late quartz, including veins and a thin top horizon of vuggy quartz. In the "original" areas, inclusions of possibly primary generation display small bubbles which are unaffected in heating tests until 130°C. Between this and 158°C some bubbles apparently shrink and disappear but other remain, 200°C being the highest recorded temperature for the presence of a gas phase. The vuggy quartz contains numerous and varied inclusions, the smaller types in vaguely-defined bands/areas roughly perpendicular to growth horizons, or rhombic outlines, the larger and less regular varieties (some with gas bubbles) being more isolated, and tending to be aligned parallel to bands and crystal boundaries. There are also areas of "network" type of inclusions, many of which appear dark (decrepitated?) Few two-phase inclusions of any type still retain the gas phase at temperatures much above 150°C, but individual inclusions were measured at 280°C, 334°C and 380°C; while two inclusions appeared to have tiny gas bubbles still moving when heating stopped at about 410°C. Therefore, although there may be some evidence for high temperatures of formation, most. inclusions tend to support a low to medium-temperature origin.

NOTE: An un-numbered sample of banded fluorite from the slopes of Mt. Gee, received separately (S. Whitehead, 1976-personal communication), was also used for microthermometric studies. Fluid inclusions were found to be of two main types: (i) individual occurrences up to 40µm in size with perfect negative crystal outlines(tetrahedra), and (ii) small to large inclusions varying in shape from oval to highly irregular, and lying in curved zones or groups, as if derived from annealed fractures. Both types displayed gas bubbles. The former (primary) gave homogenisation temperatures of 183-185°C but some may homogenise as low as 140°C. The second type (pseudsecondary?) range from 146-221°C.

TABLE 1: SUMMARY OF HOMOGENISATION TEMPERATURES ETC. (°C)

## RADIUM RIDGE BEDS

Sample	Mineral	Earlier Inclusions	Later Inclusions
P1062/76	(no section prepared)		
P1065/76	Euhedral Q ('Early idiomorphic')	373-412; 412-470	128,150,228;337-460;>524?
P1067/76	Anhedral Q (+CO <sub>2</sub> )	193-245; 324-387 479	129–167
P1071/76	Subhedral Q(+Early idiomorphic'?)	149-200; 343-402?	105-266-506
P1073/76	Detrital Q	299-308; 370-390	188; 344?
P1082/76	(no section prepared)	,	
P1085/76	Part-euhedral Q('Early idiomorphic	') 234-290; 342	423
P1087/76	(unsuitable)	,	
P1088/76	∫ Subhedral Q	160-290-312 295?	334-370 241
•	Mz	376	
T1089/76	Subhedral Mz	<b>305–334</b> <i>370</i>	
P1091/76	(unsuitable)		
P1092/76	Variable Q	340-350	
P1094/76	(unsuitable)		
P1095/76	Anhedral/subhedral Mz	>200;298? 245	
P1097/76	(unsuitable)		
P1098/76	(unsuitable)		
P1099/76	(unsuitable)		
P1100/76	Part-euhedral Q (Late?)	97-130	87: 237-267
P1101/76	(unsuitable)	•	•
P1103/76	Detrital Q (W. overgrowths)		80? 118-125; 135-155
P1105/76	(not examined)		
P1107/76	Anhedral Mz	>200-245	
P1108/76	Anhedral Q	364-379	165
	Euhedral Mz	280-300	
P1110	(no section prepared)		·
P1112/76	Anhedral $Q(+CO_2)$	200-267	68-186 <u>270</u>
P1113/76	(unsuitable		
P1114/76	(unsuitable)		
P1118/76	Anhedral Q	×100−138 4 <u>80−495</u>	
	Anhedral/subhedral Mz	96	215
P1124/76	(no section prepared)		
P1129/76	(not examined)	·	•
		· .	

Q = Quartz Mz = Monazite

Figures in italics refer to decrepitation temperatures

TABLE 2: SUMMARY OF HOMOGENISATION TEMPERATURES ETC. (°C)

## MT. GEE BEDS

Sample	Mineral	Earlier Inclusions	Later Inclusions	
P306/76	Vuggy Q	<136 - 206		
P308/76	Zoned Ft	352	∿107-117;257-293; 315	
	Interbanded Q	182-207		
P261/76	Interbanded vuggy Q	163 <u>300-350</u> *		
P263/76	Euhedral/anhedral 'vuggy' Q	123-163; 223-276		
P1304/76	'Vuggy' Q	129-149; 280- 410?	<b></b> ?	
	Early Q	130-198		
	Zoned Ft	183-185	146-221	
· .		·		

Q = Quartz Ft = Fluorite

<sup>\*</sup> Figures in italics refer to decrepitation temperatures

## 4. INDUSTRIAL MINERALS - ZONING IN SMOKY QUARTZ (MORION)

#### 4.1. Introduction

Samples of zoned smoky quartz, from the Miltalie Morion Deposit (Eyre Peninsula), were received initially under the File No. 1/16/5/0, as job 1514/77, with the request to determine the cause of colour zoning. The specimens comprised a number of related smaller single crystals and groups of both clear and smoky varieties, and also a larger crystal of smoky quartz, some 12cm long. The smoky types were generally zoned and passed from clearer quartz at the "base" of the clusters to smoky quartz towards the outer termination. It was understood that the completely colourless crystals were surface samples, while the smoky crystals occurred 0.3 metres below the surface of the deposit.

After discussion it was agreed that microthermometric measurements on fluid inclusions in selected samples might assist in determining the cause of the colour variation, particularly if a relationship between the homogenisation temperatures of the inclusions and the intensity of the colour could be established. It was further considered that chemical analysis of the zones might assist in future interpretation of fluid inclusion studies of quartz, particularly as the present samples were obviously primary in origin, with good zoning and crystal habit.

## 4.2. The Nature of Smoky Quartz (after Dana)

The cause of the smoky colour in quartz has been the subject of much discussion and investigation. Smoky quartz (morion) grades into colourless quartz and may also be interbanded with amethyst. The colour is stable up to about 225°C, but is bleached above this. A smoky colour can be induced in colourless quartz by irradiation, but the temperature stabilities of the natural and the irradiated smoky colours are different. Most properties of smoky quartz lie within the range of ordinary quartz, though it is generally agreed that the presence of aluminium is a prerequisite to the presence of a smoky colour. Not all quartz with aluminium, however, is smoky, possibly indicating that the location of the aluminium is the critical factor, and indeed in some occurrences of the two types of quartz, the colourless one has the higher aluminium content. Smoky quartz had a characteristic absorption at 460nm related to aluminium substituting for silicon.

Although smoky quartz is said to contain fluid inclusions, sometimes abundantly, this was not found to distinguish the smoky quartz in the present samples.

#### 4.3. Procedures of Investigation .

The largest sample was sectioned length-wise (perpendicular to the zoning) and marked off into 10 convenient zones. Portions of these zones, together with three portions of other crystals, were analysed spectrographically for selected elements. Polished thick sections were then prepared

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from the remaining parts of the main section and used for fluid inclusion studies.

Heating tests were also carried out on some samples in an attempt to find out whether or not the occurrence of the clear quartz at the surface of the deposit was a climatic effect.

#### 4.4. General Description

The largest quartz crystal was found to be clear to milky towards the base, grading outwards to smoky. The milky effect was generally somewhat irregular, an effect caused by the presence of fracture planes etc. and inclusions (many solid), yet in detail the junction between milky and clear bands was locally sharp. The smoky zones, though not so well-defined in detail, were more persistent overall. The various marked zones were individually described as follows:

1)	Clear to slightly milky
2)	Clear to slightly milky
3)	Clear
4)	Milky to clear
5)	Milky
6)	Slightly smoky to smoky
7)	Clear to v.slightly milky
8)	Clear to smoky
9)	Smoky (loc. v. smoky)
10)	Smoky

Slight traces of a more yellow colour were found in zones 1-3.

In thin section the large quartz crystal was seen to have certain areas characterised by undulose extinction, locally reaching such a degree as to resemble the texture of a quartzite, with fine sub-zones usually but not always roughly aligned perpendicular to growth zones. Fluid inclusions were generally rather limited in quantity. No very definite relationship could be found between the presence of fluid inclusions and strain effects, though the outer zones (7-10) displaying least strain tended to have fewer inclusions. Also, where more abundant, the inclusions tended to be aligned or elongated nearly perpendicular to growth zones and thus appeared to be roughly parallel to some strain shadows, but planes of inclusions lying oblique to these directions, and more rarely, inclusions showing the influence of two directions of elongation, could also be observed. Local concentrations of fluid inclusions (some reaching almost to the stage of being a "network") appeared to be related to areas of milky quartz and associated with solid inclusions.

The fluid inclusions themselves seldom reached above 15µm in size, the bulk being closer to 5µm. With few exceptions they were elongate to irregular, but with a slight tendency to rounding. Many displayed a small gas phase (i.e. 2-phase inclusions) but inclusions with a solid phase were very rare. In one instance inclusions, possibly with two liquids and a gas phase (CO 2), were noted, but they were too small for

positive identification.

In the absence of any positive primary features, such as negative crystal shape and relationship to growth bands or to significant crystall ographic direction, the majority of the inclusions might be considered as secondary or pseudosecondary. Indeed the elongation and alignment of many inclusions roughly perpendicular to growth bands and roughly parallel to strain shadows is most typical of secondary inclusions, and there is no doubt that secondary inclusions are represented. However, the quartz itself is clearly of primary origin. It may be, therefore, that many of the fluid inclusions are modified primary or pseudosecondary types.

#### 4.5. Summary of Results

#### 4.5.1. Emission Spectrographic Analysis

The results of emission spectrographic analysis of a small clear quartz crystal, a small smoky quartz crystal, the 10 described zones of the large quartz crystal, plus a piece of related clear surface quartz, are shown in Tables 3 and 4.

The small clear and smoky crystals were analysed for a range of trace elements in addition to those sought in the other samples. (See Table 3). The results show firstly that with respect to these two crystals only aluminium differs significantly, being an order of magnitude higher in the clear quartz, though in both cases there being no more than a trace amount present. The additional presence of traces of Ni is considered to be unusual.

In the remaining samples only the elements Al, Ca, Fe, Mg, Li and Na were sought, these being among the most common elements found in quartz (except perhaps Ti and Na). The results show clearly that there are some zonal variations in composition and that the smoky zones comprise purer quartz than the colourless zones. This is most typified by Al levels. In apparent conflict with this, however, is the close agreement in trace element composition between the smoky zones in the large crystal and the sample of clear surface quartz.

## 4.5.2. Microthermometry of Fluid Inclusions

The homogenisation temperatures of the inclusions in the various zones in the large quartz crystal are summarised in Table 5. Individual descriptions of zones are given in Section 4.6. No obvious pattern is discernible from the results, though it could be argued that there is a trend of increasing temperature from zone 1 to zone 3, with a return to low temperatures at the milky zone 4, followed by another temperature increase. However, the results are based in some cases on too few well-formed inclusions to be reliable on such details, and unfortunately no usable inclusions were found in zone 10, which might have confirmed the apparent rise in temperature towards this outer zone.

#### 4.5.3. Heating/Fading Tests

One half of a smoky quartz crystal was kept in an oven at about 105°C for a day. No colour change was recorded and so the temperature was increased to 160°C for a further 7½ hours, but again with no change in the colour of the quartz being observed. The same half of the sample was then transferred to a furnace at 240°C (above the supposed colour stability temperature) and slowly brought to 300°C. Within 24 hours a slight fading was noted when the sample was compared with the unheated reference half, and within a further 3 days at the same temperature the sample had become completely clear.

Having confirmed that fading of the present samples can be achieved by elevating temperatures, another piece of smoky quartz was held in the oven at 150°C for 4 months as a test of time-dependency. At the end of the period it was considered that the sample had faded slightly. This thus raises the possibility that fading does not have an absolute temperature threshold but more probably has an inverse exponential time relationship with respect to temperature.

## 4.6. Discussion and Conclusion

No obvious relationship has been found to exist between the temperature of formation (as judged by the homogenisation temperature of fluid inclusions) of the quartz and either its smoky colour, or its trace element levels. However, within a zoned clear to smoky crystal there appears to be an overall progressive decrease in aluminium content with outward crystal growth, which generally (though with local exceptions) corresponds to an increase in smokiness. Although these results are not necessarily in conflict with the general belief that aluminium is an essential constituent of smoky quartz, they tend to suggest rather that in a given environment, higher levels of aluminium are required for the formation of clear quartz than for smoky quartz.

The situation is complicated by indications that relatively low temperatures may induce fading in smoky quartz, provided they are maintained for a long enough period. Thus fading might explain why the surface quartz samples are clear (as against the less-exposed smoky quartz) and also why their Al-content is not as high as the clear zones in smoky crystals: in other words the surface quartz may originally have been smoky. This, of course, cannot explain the presence of clear zones in single crystals of smoky quartz.

It is concluded that the present investigation has not in fact established the complete cause of colour variation. The effects of fading on large-scale colour variation might be better confirmed by a statistical comparison of surface and deeper quartz samples from a number of deposits. Likewise, analysis of zones in crystals from a range of deposits would have to be considered to confirm that aluminium plays a key role in colour zoning of individual quartz crystals.

TABLE 3: COMPARISON OF TRACE ELEMENTS IN CLEAR AND SMOKY

QUARTZ - EMISSION SPECTROGRAPHIC ANALYSIS

Results in ppm  Element		Detection limits in brackets		
		Clear	Smoky	
Al.	(100)	200	20	
Ca	(<10)	<10	<10	
Fe	(<10)	<10	<10 (equal)	
Mg	(<10)	<10	<10 (equal)	
Li	(1)	100	100	
Na	(50)	30	30	
Cr	(20)	30	50	
Ni	(5)	10	10	
Ti	(100)	<10	<10	
Zr	(10)	<3	<3	

Elements sought but not found include Ba(200), Be(1), Ce(300), Co(5), La(100), Mn(10), Mo(3), Nb(20), Sc(3), Sr(50), Ta(100), Th(100), V(10), W(50), Y(10), Yb(1), Ag(0.1), As(50), Bi(1), Cd(3), Cu(1), Ga(1), Ge(3), In(10), Pb(1), Sb(30), Sn(1), Zn(20).

TABLE 4: COMPARISON OF SELECTED TRACE ELEMENTS IN ZONED

QUARTZ - EMISSION SPECTROGRAPHIC ANALYSIS

Results in ppm.		Detection limits in brackets				
Sample	A1(10)	Ca(1)	Fe(10)	Mg(10)	Li(1)	Na(10)
Zone 1	1,000	40	30	20	200	100
Zone 2	2,000	20	40	- 30	50	30
Zone 3	1,500	70	150	15	200	100
Zone 4	1,000	2	250	<b>1</b> 5	100	100
Zone 5	900	. 3	60	15	100	100
Zone 6	800	5	30	15	80	30
Zone 7	750	20	15	15	100	200
Zone 8	250	. 2	20	10	30	30
Zone 9	200	4	20	15	70	30
Zone 10	<b>7</b> 5	3	15	15	30	80
Clear "sur	· <b>-</b>		•			
face quart	z* 50	2	50	15	30	80

TABLE 5: SUMMARY OF HOMOGENISATION - TEMPERATURES ETC. (°C)

Zone 1	102?; 123-169; 199-306
Zone 2	130 - 199
Zone 3	154-166; 275-376
Zone 4	∿125 -∿180; 138-151
Zone 5	135-189; ∿150-257
Zone 6	125-162; 123-202
Zone 7	140-235; 280?-307? 290 - 335 - 350 *
Zone 8	>270->350
Zone 9	>290-∿325
Zone 10	<del></del>

<sup>\*</sup> Figures in italics refer to decrepitation temperatures

#### 4.7. Detailed Descriptions of Fluid Inclusions

- Zone 1: Inclusions in this zone are relatively sparse, though in places concentrated and aligned approximately parallel within planes running almost perpendicular to growth bands. Such inclusions range from medium-small (5-10µm ) down to sub-microscopic in size. Although some suggestion of negative crystal shape may be visible, they are generally elongate to slightly angular. Some of the larger individual examples (up to 20µm) are less "regular" in shape, but these, too, tend to display some common directional growth control. Inclusions with gas bubbles appear to be in the minority, and with few, if any, exceptions they display a low degree of fill(i.e. small bubbles). Fortunately these 2-phase inclusions are sufficiently numerous to give a fair indication of the range of homogenisation temperatures, but no solid phases can be recognised with certainty. In a few cases the gas bubble only became indiscernible after heating to slightly above 100°C, but 123°C represents the lowest confirmed homogenisation temperature, and 306°C the highest figure obtained. Isolated instances of decrepitation occur below 200°C, but these are probably not an indication of the true temperature of crystallisation, particularly as decrepitation is seldom to be observed even up to 310°C. One plane was found to contain medium-sized (20µm) flat inclusions resembling secondary types, their range of homogenisation being 232-281°C, while another plane was found to contain similar inclusions which had already decrepitated.
- Zone 2: Again inclusions are not abundant generally, though locally sufficiently numerous to show a rough alignment broadly perpendicular to zone boundaries. Most are small (<10µm), and although as in zone 1, some evidence of negative crystal outline is visible, an elongate outline is dominant. Gas bubbles, when present, indicate that the homogenisation range starts below 130°C and continues up to 199°C.
- Zone 3: Even fewer inclusions were encountered in this admittedly narrower zone, some planes containing inclusions without gas bubbles. The local cluster of inclusions was found which displayed two mutually perpendicular directions of growth control (one parallel to zone bands), with sufficient gas bubbles present to give homogenisation temperatures in the range 275°C to about 376°C. Other narrow planes in this zone intersect and sometimes curve across one another. Two slightly larger inclusions, associated with these planes have "bubbles" which behave more like a second liquid phase, shrinking and disappearing with a range of 154-166°C. These features are suggestive of later (pseudosecondary?) inclusions.
- Zone 4: The "milkiness" in this zone is clearly related to patches of quartz with myriads of inclusions. The majority resemble fluid inclusions, but a number are opaque, sometimes with a reddish

tinge (haematite?). Their general alignment, though not always of the planes in which they lie, is approximately perpendicular to the zone boundaries, but many show other sometimes curving alignments, or are unorientated. Very few gas bubbles are represented, though many of the larger (irregular) inclusions look decrepitated. Homogenisation occurs in the range from about 125°C up to 180°C. One transverse plane with larger, elongate inclusions confirms this range, but several of them also have solids which do not dissolve up to 400°C or more, by which stage numbers of inclusions have decrepitated.

- Zone 5: Another milky zone, this contains similar patches of inclusions, mostly aligned perpendicular to the zone and locally resembling "network"-type inclusions. Here and there, larger individual inclusions, also aligned, appear to be related, though irregular examples can also be found. The lowest homogenisation temperature recorded is ~150°C, with irregular types reaching to 257°C, but too few observations were made for reliability. In another non-milky part of the zone inclusions tend to be individually elongated as before, but also to be distributed along the sometimes curved boundaries separating crystal domains. Such inclusions are typical of pseudosecondary types, but whether in fact they post-date the others just described remains uncertain Their range of homogenisation is more definite, however, from 135°C to 189°C.
- Zone 6: By contrast with the two previous, much of this zone has few inclusions. It is similar to zones 1-3, however, in having many inclusions grouped in ill-defined planes. Most are small, elongate and commonly curved, their tiny gas bubbles homogenising between 125°C and 162°C. One inclusion in this group was found to have a length of nearly  $50\mu\text{m}$ , tapering off at one end and merging into an apparently decrepitated zone at the other. The relatively large gas phase contracted upon heating until about . 305°C, then expanded irreversibly to fill the inclusion by about These unusually higher temperatures are taken as an indication that partial decrepitation had already occurred in this particular inclusion, while its highly elongated shape points to a post-crystallisation influence (e.g. an annealed fracture). It is thus possible that many inclusions in this zone reflect later temperatures. In other parts of the zone where patches of irregular to "network" inclusions occur, one or two seemingly remnant regular primary fluid inclusions indicate minimum temperatures of formation of up to 202°C.
- Zone 7: In the strained areas of quartz "network"-type inclusions grade into more regular varieties following various curved planes (possibly recrystallised domain boundaries). Homogenisation probably ranges up to about 280°C, but no good readings were obtained. In the unstrained areas of quartz, inclusions range up to 307°C, but groups of small primary-looking types vary from 140°C to 235°C. Some decrepitation was recorded after 290°C,

but more generally in the range 330-345°C.

- Zone 8: This zone comprises unstrained quartz, with very few inclusions, mostly confined to relatively isolated and randomly-orientated planes. The inclusions range from those with negative crystal shape to elongated and angular forms, in the homogenisation span from at or above 270 C up to at least 350 C.
- Zone 9: There are rare, short planes, often terminating abruptly, which contain small irregular, yet slightly elongated, fluid inclusions, giving a temperature range of over 290°C to about 325°C. Few suitable inclusions were found.
- Zone 10:No inclusions suitable for microthermometric measurements were found in this zone.

FLUID INCLUSION STUDIES, OF SOUTH AUSTRALIAN ORES, ROCKS, AND MINERALS

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> FLUID INCLUSION STUDIES OF SOUTH AUSTRALIAN ORES, ROCKS, AND MINERALS

PROGRESS REPORT NO. 3

GOLD-BEARING VEINS AND QUARTZ FROM SOUTH AUSTRALIAN GOLDFIELDS

by Dr B. Collins

Investigated by: Geological Services Division

Manager: Dr Keith J. Henley

for R.E. Wilmshurst Acting Managing Director

#### 1. INTRODUCTION

During 1975/76 some initial studies were made of gold-bearing quartz veins from the Wadnaminga Goldfields (Project 1/1/182; Progress Report No.1). In the following year similar studies were commenced on barren quartz veins occurring between Anabama and Wadnaminga (Project 1/1/182; Progress Report No. 2; Project 1/1/176; Progress Report No. 2). Preliminary indications suggested that the Wadnaminga gold veins were unrelated either to the barren veins or to the Anabama Granite intrusion. Consequently, further work was proposed for gold-bearing vein quartz samples to be collected from various goldfields in the Olary region in an attempt to throw more light on their origin.

Although only a few samples were used in the initial study of Wadnaminga auriferous quartz, the information obtained indicated that primary fluid inclusions had a minimum temperature of formation in the range  $380^{\circ}-510^{\circ}\text{C}$ , whereas secondary inclusions displayed the range  $180^{\circ}-360^{\circ}\text{C}$ . Inclusions from the barren veins appeared to range from  $140^{\circ}\text{C}$  up to about  $400^{\circ}\text{C}$  for primary types, whereas secondary inclusions generally fell into the range  $115^{\circ}-300^{\circ}\text{C}$ . The current investigation has not been completed, but it appears at this stage that the suspected primary fluid inclusions have a wide range of minimum temperatures (see Table 1). However, it has been found difficult to make accurate determinations on some inclusions, and to assign many inclusions measured to a primary or secondary origin, partly because of their small size, lack of diagnostic properties and sometimes because of observational problems.

#### 2. GENERAL DESCRIPTION

Nearly all the samples examined comprised medium to coarse or even very coarse quartz, usually displaying strain-shadows and apparently partly or locally recrystallised to form granular zones. In a few cases a lithological banding was evident, this probably relating to the "incorporation" of lenses of quartzose country rock (eg P1837/76 and P1839/76 - see Project 1/1/176, Progress Report No. 2, page 8 and page 10). Thus, although coarse areas of quartz were selected for study wherever practicable, the possibility exists that some of the material measured represents "incorporated" quartz, with homogenisation temperatures which may be unrelated to the formation of the (later) veins. In most vein samples the quartz was generally cloudy, due to the presence of numerous inclusions, apparently fluid-bearing, some with gas bubbles (2-phase) and rarely also with a solid phase (3-phase). However, most samples also had patches of clearer quartz where the inclusions were more widely dispersed. With the exception of those which looked to have previously decrepitated, the inclusions were small, ranging from the limits of observation up to about  $12\mu\mathrm{m}$  in a few cases, with rare individuals reaching  $20~\mu\mathrm{m}$  in size.

These inclusions probably represent more than one generation, but as already mentioned, no specific features were found to reliably distinguish between primary and secondary types. Very few with even a partial indication of negative crystal shape were encountered. Instead, the smaller ones tended to be rounded to slightly elongate (sometimes with a common alignment) while the larger were often less regular. A significant percentage of fluid inclusions occurred as apparently randomly dispersed individuals, unrelated to any planes (nor yet to obvious crystallographic directions); these, therefore, have been considered as primary in origin. Similar, but smaller, inclusions tended to lie in narrow planes resembling otherwise completely annealed fractures; these, it has been assumed, represent secondary inclusions. However, in some cases the alignment of apparently similar inclusions defined

close-spaced, parallel, short planes, the abundance and regularity of which, it is considered, tend to support a primary, rather than a secondary origin. In some sections vague bands rather than planes of aligned inclusions followed two directions, usually one roughly parallel to strain shadows. Inclusions in this direction were assumed to be of secondary origin, but only in one section (P1846/76) were the bands more obviously distinguishable as primary and secondary.

A possible third and later(?) set of inclusions may be represented by mostly larger, irregular structures, resembling decrepitated inclusions, which may have smaller counterparts in local areas with a fine "network" of inclusions. No reliable measurements could be made on these inclusions, however.

#### 3. INDIVIDUAL DESCRIPTIONS

#### P1824/76 TS 38741: Mt. Grainger Gold Mine

Although numerous tiny inclusions accounted for the cloudy appearance of the quartz, virtually none with gas bubbles suitable for measurement was found in the material so far examined. Larger inclusions appeared to have previously decrepitated.

#### P1826/76 TS 38742: Mt. Grainger Gold Mine

This sample, too, was generally cloudy, though with limited clearer areas. The many small inclusions had a tendency to common alignment with the slight elongation of crystal domains, which was roughly perpendicular to a vestigial banding (from the country rock). Some bands of inclusions appeared, in contrast, to be parallel to this lithological banding, probably indicating more than one generation of fluid inclusions. The well-dispersed and possibly primary inclusions gave low readings, from 152°C up to at least 178°C.

#### P1829/76 TS 38743: Mt. Grainger Gold Mine

This sample was very cloudy, and again the smaller inclusions tended to be commonly orientated, with well-dispersed, larger varieties resembling those in the previous sample. Some of the latter had outlines consistent in part with negative crystal shape but apparently grading to the smaller, rounded varieties. In a number of cases decrepitation occurred before the disappearance of the gas phase, possibly owing to the presence of a small solid phase. The lowest temperature recorded was 145°C, though whether primary or secondary remained uncertain. Other possibly primary types started to decrepitate about 230°C, with high homogenisation temperatures found, up to 502°C. Very small inclusions, elongated and aligned in secondary(?) planes homogenised around 220°C, the range probably being 145°-278°C. The largest and usually least regular types appeared to be decrepitated.

#### P1831/76 TS 38744: Mt. Grainger Gold Mine

This sample was generally less clouded with inclusions. Where abundant, the inclusions tended to be small and in places two rough alignments were detectable, one agreeing with the elongation of strain shadows in the quartz; but the more-individual inclusions also tended to show a local alignment. Subsequent re-examination indicated that some inclusions resulted from the annealing of fractures (i.e. secondary), but although others may be more consistent with growth bands (i.e. primary), the majority remain uncertain. The lowest reading recorded on

a possibly primary fluid inclusion was about 110°C. All others were higher, starting at 285°C. The smaller inclusions ranged up to at least 465°C, probably reaching in excess of 500°C. One such group of nevertheless individual occurrences displayed negative crystal outline and ranged from about or above 400°C to in excess of 530°C, when heating stopped.

#### P1833/76 TS 38745: Mt. Grainger Gold Mine

This sample showed some concentrations of inclusions, with indications of possible growth bands, but size limitations precluded homogenisation tests. Some areas had a network of inclusions, also unsuitable for use, which tended to "follow" strain-shadows and may therefore represent zones of recrystallisation rather than fracture- or shear-planes. Inclusions of a coarser and more isolated nature, some with negative crystal outline and possibly also containing a solid phase were presumed to be of primary origin. Most of the temperatures obtained were fairly high to very high with decrepitation tending to confirm the range. The lowest recorded was 205°C, two inclusions failing to homogenise by 500°C.

#### <u>P1834/76 TS 38746: Mt. Grainger Gold Mine</u>

Similar to P1833/76 and generally sieved with inclusions, this sample provided little evidence of the nature of these inclusions. In the absence of distinct planar features (such as fractures) or strong alignments, most of the inclusions large enough to be measured were assumed to be primary. Two ranges may be present  $(168^{\circ}-228^{\circ}\text{C} \text{ and } 392^{\circ}-509^{\circ}\text{C} \text{ or possibly higher})$ , but insufficient readings were obtained to be certain of this.

#### P1837/76 TS 38747: Ajax Gold Mine

This sample had relatively larger areas of "clear" quartz and again evidence of possibly two groups was found. Re-examination confirmed that at least two generations of inclusions were present, with apparently primary regular to slightly irregular types varying from relatively isolated individuals to those associated in short planes, and smaller more rounded to elongate secondary inclusions lying in more distinctive planes. The more-obviously secondary inclusions gave low homogenisation temperatures around 150°C to 170°C, whereas the possibly primary ones were more variable and sometimes displayed partial decrepitation, rendering some of the readings less reliable. The latter ranged from 220°C or 240°C up to 420°C. The section also contained larger and distinctly irregular structures, resembling decrepitated inclusions. One genuine fluid inclusion of similar shape, containing a gas bubble, homogenised/decrepitated at somewhat less than 140°C and may be an indication that these structures represent another generation of lowtemperature secondary inclusions.

#### P1839/76 TS 38748: Ajax Gold Mine

This sample displayed a ?remnant lithological banding comprising granular quartz with coarser crystals of vein quartz, the latter having inclusions showing a tendency to oblique alignment. Elsewhere in the vein quartz only vaguely defined (?primary) bands could be discerned and no overall pattern was noted, but most of the associated inclusions could be primary. Only a few temperature observations were made and both low (circa 133°C) and high (349°-460°C+) values were recorded. One inclusion appeared to homogenise at a critical point, the temperature of  $\sim 349$ °C indicating a fairly saline solution.

No definite growth bands or alignments were found in this sample. Many nebulous, very fine "networks" were noted, but so, too, were larger, individually-occurring inclusions, some with gas bubbles. Several measurements, from about 158°C to 314°C were obtained.

#### P1846/76 TS 38750: Waukaringa Gold Mine

This sample displayed the best evidence of inclusions defining primary growth bands which seem to pre-date recrystallised areas. Some of the inclusions were relatively well formed with part-euhedral outlines, reached up to 20  $\mu$ m in size and not uncommonly contained both gas and solid phases. Homogenisation temperatures ranged from 170°C up to possibly 330°C, but the presence of a solid phase which tended to dissolve after homogenisation of the gas and liquid phases may have influenced these values. Further work may be required on this sample.

#### P1860/76 TS 38751: Taltabooka Goldfield

The sample had a very cloudy appearance due to the presence of numerous tiny inclusions and "network"-type zones. The presence was also evident of distinct though seldom regular bands, probably representing primary inclusions. There were also moderate numbers of slightly coarser "individual" inclusions, most slightly irregular in outline, but some with negative crystal shape. The temperatures obtained ranged from about 200°C to probably in excess of 340°C. Inclusions of possibly secondary origin homogenised between 138°C and 168°C.

#### P1861/76 TS 38752: Taltabooka Goldfield

This is another sample with a cloudy appearance arising from the presence of numerous small inclusions. Generally no banding was observed, though one crystal displayed a fine alignment of inclusions with a vaguely defined banding perpendicular to it. Few, if any, decisive secondary planes or fractures with typically small inclusions were encountered, the larger "individual" types of inclusions present being too numerous to have formed from fracture- or shear-planes, and thus considered to be of primary origin. In contrast to most other samples, the homogenisation temperatures of a large number of fluid inclusions were almost exclusively fairly low (117°-197°C), with two higher values, at 247°C and 271°C. It is possible, however, that some inclusions with gas bubbles, as yet unobserved, have higher homogenisation temperatures, since heating tests on this sample reached only to about 271°C. Further work may thus be required on this sample also.

#### 4. SUMMARY AND CONCLUSIONS

Microthermometric work has not yet been completed; a number of samples have not been examined at all and several warrant further heating tests. A summary of data obtained so far is given in Table 1. No freezing tests (to estimate salinities) have yet been carried out. The work so far carried out is believed to indicate that the vein quartz in the goldfields has formed under widely varying temperature conditions, even within the same field, though where measured, secondary inclusions, possibly related to recrystallisation, have been formed under low-temperature conditions. No obvious relationship with the previously-studied samples has so far been established.

Future work will involve obtaining homogenisation temperature measurements on the remaining samples not yet studied (from various goldfields). Selected samples will be investigated with regard to freezing temperatures to allow estimates of fluid salinity. The results of these tests will be given in the next Progress Report.

## TABLE 1: SUMMARY OF FLUID INCLUSION HOMOGENIZATION DATA

Goldfield	Sample	Nature of Quartz.	Homogenization Temperature, °C Primary ?Secondary		
	No.	•	Inclusions	Inclusions	
Mt. Grainger	P1824/76	Coarse, slightly strained. Granular.	Unsuita	ble —	
Mt. Grainger	P1826/76	Medium to coarse, slightly strained	152-178 (-192?)	(not measured)	
Mt. Grainger	P1829/76	Coarse, strained. Granular.	230?257; 406-528+	145–278	
Mt. Grainger	P1831/76	Coarse, unstrained. Locally granular.	<b>~110; 285–535+</b>		
Mt. Grainger	P1833/76	Coarse, strained	205-508+	•	
Mt. Grainger	P1834/76	Coarse, strained	168-228; 392-509+		
Ajax	P1837/76	Coarse, slightly strained. Granular.	~180-380	∿150–170	
Ajax	P1839/76	Coarse. Granular bands.	?133; 349-460+	÷ .	
Waukaringa	P1842/76	Coarse, strained.	?158; 241-314	,	
Waukaringa	P1846/76	Coarse.	170-308; ?330		
Taltabooka	P1860/76	Coarse, slightly strained	>198-\233; ?>339	138–168	
Taltabooka	P1861/76	Very coarse.	117-197; 247; 271		

FLUID INCLUSION STUDIES OF GOLD-BEARING VEINS AND QUARTZ FROM SOUTH AUSTRALIAN GOLDFIELDS.

Department of Mines & Energy

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October 1978



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4 October 1978

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FLUID INCLUSION STUDIES OF GOLD-BEARING VEINS AND QUARTZ FROM SOUTH AUSTRALIAN GOLDFIELDS

PROGRESS REPORT NO. 4

Investigation and Report by: Dr B. Collins

Manager, Geological Services Division: Dr Keith J. Henley

for Norton Jackson Managing Director

051

#### 1. INTRODUCTION

During 1975/76 some initial studies were made of gold-bearing quartz veins from the Wadnaminga Goldfields (Project 1/1/182; Progress Report No. 1). In the following year similar studies were commenced on barren quartz veins occurring between Anabama and Wadnaminga (Project 1/1/182 - reported in Project 1/1/176; Progress Report No. 2). Preliminary indications suggested that the Wadnaminga gold veins were unrelated either to the barren veins or to the Anabama Granite intrusion. Consequently, further work was proposed for gold-bearing vein quartz samples to be collected from various goldfields in the Olary region in an attempt to throw more light on their origin.

Although only a few samples were used in the initial study of Wadnaminga auriferous quartz, the information obtained indicated that primary fluid inclusions had a minimum temperature of formation in the range  $380^{\circ}-510^{\circ}\text{C}$ , whereas secondary inclusions displayed the range  $180^{\circ}\text{C}-360^{\circ}\text{C}$ . Inclusions from the barren veins around Anabama appeared to range from  $140^{\circ}\text{C}$  up to about  $400^{\circ}\text{C}$  for primary types, whereas secondary inclusions generally fell into the range  $115^{\circ}-300^{\circ}\text{C}$ . The current investigation has now been completed, and it has confirmed that the suspected primary inclusions in quartz veins from the Olary goldfields do indeed have a wide range of minimum formation temperatures (see Table 1). Continuing difficulties in determining and differentiating some inclusions have been found, as previously reported, but this has not prevented further information from being obtained (in most cases) on salinities by freezing experiments, the freezing data being incorporated in Table 1 and in the individual descriptions.

#### 2. GENERAL DESCRIPTION

Nearly all the samples examined from the Olary goldfields comprised medium to coarse or even very coarse quartz, usually displaying strain-shadows and apparently partly or locally recrystallised to form granular zones. In a few cases a lithological banding was evident, this probably relating to the "incorporation" of lenses of quartzose country rock (eg P1837/76 and P1839/76 see Project 1/1/176, Progress Report No. 2, page 8 and page 10). Thus, although coarse areas of quartz were selected for study wherever practicable, the possibility exists that some of the material measured represents "incorporated" quartz, with homogenisation temperatures which may be unrelated to the formation of the (later) veins. In most vein samples the quartz tended to be cloudy, due to the presence of numerous inclusions, apparently fluid-bearing, some with gas bubbles (i.e. 2-phase) or with a second liquid with gas (i.e. 3-phase) and sometimes also with a solid present (i.e. 3- or 4-phase). However, a number of samples had, in addition, patches or individual crystals of clearer quartz where the inclusions were more widely dispersed. With the exception of rare individuals, including some which appeared to have previously decrepitated, the inclusions were found to be small to medium in size. They ranged from the limits of observation up to about 12 μm, in some cases reaching 20 μm in size. The maximum size recorded was 70 µm.

These inclusions may represent more than one generation, but as already mentioned, no specific features were found to distinguish reliably between primary and secondary types, with the exception of some samples in which grain and subgrain boundaries were marked by partly annealed planes and inclusions mostly too small for study. These apart, few inclusions with distinct negative crystal shape were encountered. Instead, the smaller ones tended to be rounded to slightly elongate (some with a common alignment), while the larger were often less regular.

A significant percentage of fluid inclusions occurred as apparently randomly dispersed individuals, seemingly unrelated to any planes (nor yet to obvious crystallographic directions); these, therefore, have been considered as primary (or at least early) in origin. Similar, but usually smaller, inclusions tended to lie in narrow planes resembling otherwise completely annealed fractures; these, it has been assumed, represent secondary inclusions, and less attention has been devoted to them. However, in some cases the alignment of apparently similar inclusions defined close-spaced, parallel planes of short length, the abundance and regularity of which, it is considered, tend to support a primary rather than a secondary origin (i.e. growth zones). In some sections vague bands rather than planes of aligned inclusions followed two directions, usually one of these roughly parallel to strain shadows. Inclusions obviously related to this direction were assumed to be of secondary origin, but only in one section (P1846/76) were the bands more obviously distinguishable as primary and secondary. In general, therefore, the well-dispersed inclusions have been preferentially selected for study as being the most likely to represent the entrapment of primary fluids.

A possible third or fourth (and later?) set of inclusions may be represented by generally larger, irregular structures, resembling decrepitated inclusions, which may have smaller counterparts in local areas with a fine "network" of inclusions. In some instances the partly annealed planes/inclusions along subgrain boundaries resembled "network" areas.

#### 3. INDIVIDUAL DESCRIPTIONS

#### P1824/76: Mt. Grainger Gold Mine

Although numerous tiny inclusions accounted for the cloudy appearance of the quartz, virtually none with gas bubbles suitable for homogenisation studies was found. Attempts to freeze selected areas with inclusions were unsuccessful.

#### P1826/76: Mt. Grainger Gold Mine

This sample, too, was generally cloudy, though with limited clearer areas. The many small inclusions had a tendency to common alignment with the slight elongation of crystal domains, which was roughly perpendicular to a vestigial banding (from the country rock). Some bands of inclusions appeared, in contrast, to be parallel to this lithological banding, probably indicating more than one generation of fluid inclusions. The well-dispersed and possibly primary inclusions gave low readings, from  $152^{\circ}\text{C}$  up to at least  $178^{\circ}\text{C}$ . As with the previous sample, however, attempts to freeze inclusions were not successful.

#### P1829/76: Mt. Grainger Gold Mine

This sample was very cloudy, and again the smaller inclusions tended to show common orientation, with well-dispersed, larger varieties (mostly 5-10 µm) resembling those of the previous sample. Some of the latter type had outlines consistent in part with negative crystal shape, but apparently grading to the smaller, rounder varieties. Also, in some of the larger ones a solid phase could be recognised, its roughly square outline being suggestive of halite (salt). In rare cases a second, birefringent solid accompanied the other. The lowest homogenisation temperature recorded upon heating was 145°C, but whether primary or secondary remained uncertain. Some inclusions, especially the largest and usually least regular, looked as if they had previously decrepitated. Other possibly primary types started to decrepitate at about 230°C, though more widespread decrepitation did not become apparent until above 350°C by which stage some of the salt? cubes

had dissolved. The birefringent solids did not dissolve below  $430^{\circ}\text{C}$ , while some inclusions had very high homogenisation temperatures, up to and possibly in excess of  $500^{\circ}\text{C}$ . Very small inclusions, elongated and aligned in secondary(?) planes, gave values around  $220^{\circ}\text{C}$ , the range probably extending from  $145^{\circ}$  to  $278^{\circ}\text{C}$ . Freezing experiments caused the outlines of some bubbles to appear "heavier" than normal, but in only one case was a "double bubble", probably representing  $C0_2$  liquid + gas, recognised, this becoming a single phase once more on being warmed to  $-26^{\circ}\text{C}$ . In one or two cases the solid phases, which had disappeared on cooling, reformed with rise of temperature, the birefringent mineral at  $-18^{\circ}\text{C}$  and the halite(?) at around  $+10^{\circ}\text{C}$ . What appeared to be ice in two inclusions was still present during heating up to at least  $-29^{\circ}\text{C}$ , but the inclusions were too small for precise melting-point data to be obtained.

The evidence as a whole indicates variable but generally very high temperatures of quartz crystallisation from solutions rich in carbon dioxide and sodium chloride, at least.

#### P1831/76: Mt. Grainger Gold Mine

This sample was generally less clouded with inclusions. Where abundant, the inclusions tended to be small and in places two rough alignments were detectable, one agreeing with the elongation of strain shadows in the quartz; but the more-individual inclusions also tended to show a local alignment. Subsequent re-examination indicated that some inclusions resulted from the annealing of fractures (i.e. secondary), but although others may be more consistent with growth bands (i.e. primary), the majority remain uncertain. The lowest reading recorded on a possibly primary fluid inclusion was about 110°C, but as this is so out of character, it may in fact be a secondary inclusion. All other inclusions were much higher, starting at 247°C, the smaller inclusions ranging up to at least 465°C and probably reaching to in excess of 530°C. One such group of nevertheless individual occurrences displayed negative crystal outline and ranged in homogenisation temperature from around  $400^{\circ}\text{C}$  to in excess of  $530^{\circ}\text{C}$ , when heating stopped. However, the inclusions in this section were regarded as being not large enough to warrant freezing experiments to be carried out.

#### P1833/76: Mt. Grainger Gold Mine

This sample showed some concentrations of inclusions, with indications of possible growth bands, but size limitations precluded homogenisation tests on such inclusions. Some areas had a network of inclusions, also unsuitable for use, which tended to "follow" strain-shadows and may therefore represent zones of recrystallisation rather than fracture- or shear-planes. Inclusions of a coarser and more isolated nature, some with negative crystal outline and possibly also containing a solid phase were presumed to be of primary origin. Most of the temperatures obtained were fairly high to very high with decrepitation tending to confirm the range. The lowest recorded was 205°C, two inclusions failing to homogenise by 500°C. In an area selected for freezing tests, several larger and less regular inclusions contained bubbles and two solids - one resembling halite, the other with moderate birefringence. On heating up after freezing by cooling to -170°C, the salt-like solid dissolved at about -36°C. The melting point of "ice" appeared to occur at about -29°C, which, though theoretically slightly low for a pure NaCl-H $_2$ O saturated solution,\* nevertheless tends to confirm the evidence of the

\*A genuine depression of the melting point of ice to  $-29^{\circ}$ C implies the presence of another component, since a saturated solution should melt at  $-21^{\circ}$ C. The presence of CO<sub>2</sub> would have little effect, but the presence of, for instance, CaCl<sub>2</sub> could account for such a discrepancy.

salt-like solid in being consistent with a solution saturated with respect to NaCl. On further warming the solid phase (presumably halite) reappeared at about +5°C. With further heating the birefringent mineral in one inclusion seemed to have dissolved by about 235°C, another inclusion had partially decrepitated, while several others were close to homogenisation by about 250°C. Although limited, the evidence again is suggestive of moderate to very high temperatures of formation from highly saline solutions.

#### P1834/76: Mt. Grainger Gold Mine

Similar to P1833/76 and generally sieved with inclusions, this sample provided little evidence of the nature of these inclusions. In the absence of distinct planar features (such as fractures) or strong alignments, most of the inclusions large enough to be measured were assumed to be primary. Two ranges may be present  $(168^{\circ}-228^{\circ}\text{C} \text{ and } 392^{\circ}-509^{\circ}\text{C} \text{ or possibly higher})$ ; insufficient readings were obtained to be certain of this, but no doubt exists that, as with the previous sample, some of the temperatures obtained were very high. Again, too, a moderate percentage of fluid inclusions contained cube-like solids, probably of halite (salt). Rarely, the presence of a birefringent solid was noted. Freezing tests gave possible indication of the presence of CO<sub>2</sub> in a solid phase, melting at about  $-60^{\circ}\text{C}$ .

Cooling and reheating appeared to show the disappearance of the salt(?) phase very approximately at  $-40^{\circ}\text{C}$  and its reformation between  $-18^{\circ}\text{C}$  and  $+5^{\circ}\text{C}$ , as has been found before. Further heating of such inclusions indicated that the salt(?) cubes in some started to dissolve at about  $+170^{\circ}\text{C}$ , possibly also with homogenisation of gas and liquid phases, but higher temperatures for the homogenisation of the gas and cubic solid phases were also obtained.

#### P1837/76: Ajax Gold Mine

This sample had relatively larger areas of "clear" quartz, plus areas of granular quartz. Evidence of possibly two groups of inclusions was found. Detailed examination confirmed that at least two generations of inclusions were present, with apparently primary regular to slightly irregular types varying from relatively isolated individuals to those associated in short planes, and smaller more rounded to elongate secondary inclusions lying in more distinctive planes. The more-obviously secondary inclusions gave low homogenisation temperatures around  $150^{\circ}\text{C}$  to  $170^{\circ}\text{C}$ , whereas the possible primary ones were more variable and sometimes displayed partial decrepitation, rendering some of the readings less reliable. The latter ranged from  $220^{\circ}\text{C}$ or  $240^{o}\text{C}$  up to  $420^{o}\text{C}$ . The section also contained larger and distinctly irregular structures, resembling decrepitated inclusions. One genuine fluid inclusion of similar shape, containing a gas bubble, homogenised/ decrepitated at somewhat less than 140°C and may be an indication that these structures represent another generation of low-temperature secondary inclusions. There was little indication of the presence of solid phases, and the material was found to be generally unsuitable for freezing tests.

#### P1839/76: Ajax Gold Mine

This sample displayed a ?remnant lithological banding, comprising granular quartz with coarser crystals of vein quartz, the latter having inclusions showing a tendency to oblique alignment. Elsewhere in the vein quartz, only vaguely defined, possibly primary bands could be discerned, there being no overall pattern, though it was considered that most of the related inclusions were of primary origin. Few heating observations were recorded though both low (circa  $133^{\circ}$ C) and high ( $349-460+\ ^{\circ}$ C) values were obtained. One 3-phase inclusion was found to contain  $\rm H_2O$  liquid +  $\rm CO_2$  liquid +  $\rm CO_2$  gas. Another inclusion apparently homogenised at a critical point, the temperature of  $\rm ^{\sim}349^{\circ}C$  being possibly indicative of a  $\rm CO_2$ -rich solution (as well as being a saline one). The saline aspect was confirmed by the presence in some

inclusions of salt-like solids (i.e. halite). A few also had birefringent solids; one such presented a rhombic outline, and is thus possibly a carbonate. Although no carbonate was directly recognisable in thin section, the textures found in the central parts of Ajax veins are reportedly after carbonate in part (Progress Report No.2, p.11). Freezing experiments also indicated fairly saline solutions, with ice melting at about  $-24^{\circ}\mathrm{C}$ , suggesting an equivalent salinity of at least 23% by wt. NaCl. The salt-like solids displayed a wide range of solution temperature, this commencing well below  $0^{\circ}\mathrm{C}$  and continuing to  $+17^{\circ}\mathrm{C}$  in one case and to over  $140^{\circ}\mathrm{C}$  in others.

#### P1842/76: Waukaringa Gold Mine

No definite growth bands or structural alignments were recognised in this sample. Many nebulous, very fine "networks" were noted, but so, too, were larger, individually-occurring inclusions, some with "bubbles". Several homogenisation measurements, from about 158°C to 314°C, were obtained. evidence from freezing experiments was complex, incorporating the separation on cooling of a second phase in the "bubble" (probably CO2 liquid + gas), followed on further cooling by ice-formation and by solid CO2(presumably). Several phase changes also occurred during warming up, notably one at  $-67^{\circ}\text{C}$ (possibly CO2 + another gas), followed by the melting of much of the ice, etc. at  $-24^{\circ}$ C. Although some inclusions appeared to be all liquid by this temperature, others had a solid phase continuing to melt/dissolve up to -13°C. Rarely, salt-like solids were observed at temperatures up to about 180°C. Some birefringent solids were also noted. Homogenisation of the  $\rm CO_2$  (liquid + gas) phases took place between  $\rm 0^{\circ}C$  and  $\rm 25^{\circ}C$ . Further heating caused general homogenisation from 160°C up to at least 295°C. In summary, the indications point to quartz formation from fairly saline solutions (>23% wt. NaCl) enriched in  $CO_2$  ( $\equiv 0.75g/ml$ ) at moderate to high temperatures.

#### P1846/76: Waukaringa Gold Mine

This relatively coarse sample displayed the best evidence of inclusions defining primary growth bands which seem to pre-date the recrystallised areas. Some of the inclusions were relatively well formed, displaying part-euhedral outlines and reaching up to 20 µm in size. Many inclusions had a gas phase, from which homogenisation values of from 170°C to at least 330°C were obtained. Some inclusions had a solid phase (i.e. 3-phase) with the usual square outline, and in one or two such instances anomalous homogenisation temperatures up to about 480°C were recorded, representing the re-appearance of a gas? phase after initial homogenisation at a lower temperature in the presence of a now-dissolved solid (the solids tending to dissolve around 240°C). Freezing of the 2-phase inclusions, usually between  $-80^{\circ}$ C and  $-126^{\circ}$ C, tended to induce the formation of a salt-like solid. The ice, etc. started to melt at about  $-29^{\circ}$ C and by  $-26^{\circ}$ C had almost disappeared. Some solid material was judged to be present in certain inclusions at up to  $-15^{\circ}$ C or even  $-13^{\circ}$ C, and solids became visible in certain inclusions at even higher temperatures before disappearing again (gas hydrates?). The presence of CO2 was inferred from the formation of a second phase within the main bubble at very approximately  $+15^{\circ}$ C ( $\equiv \sim 0.85 \, \text{g/ml}$ ). The evidence indicates low to at least moderate homogenisation temperatures, with salinitica in the 20%+ range by weight of NaCl.

#### P1860/76: \_Taltabooka Goldfield

The coarse quartz in this sample had a very cloudy appearance due to the presence of numerous tiny inclusions and "network"-type zones. The presence was also evident of distinct though seldom regular bands, probably representing primary inclusions. There were also moderate numbers of slightly coarser "individual" inclusions, most slightly irregular in outline, but some with negative crystal shape. The temperatures obtained ranged from

about  $155^{\circ}\text{C}$  to probably in excess of  $340^{\circ}\text{C}$ . Inclusions of possibly secondary origin homogenised between  $138^{\circ}\text{C}$  and  $168^{\circ}\text{C}$ . No solid phases were visible in these inclusions, though cooling to below  $-50^{\circ}\text{C}$  caused ice and/or solid  $CO_2$  to form. The entrapped bubbles appeared to continue contracting (phase change?) even when warmed up again slightly, with some seeming to disappear. Further warming eventually caused melting of the ice, which was accurately fixed in one specimen at between  $-11^{\circ}$  to  $-12^{\circ}\text{C}$  ( $\equiv 15\%$  wt NaCl). There was evidence in some inclusions of the presence of  $CO_2$ , in one case with a solid phase ( $CO_2$ -hydrate?) melting at about  $O^{\circ}\text{C}$ . The inclusions used for salinity measurements had homogenisation temperatures at the low end of the range.

#### P1861/76: Taltabooka Goldfield

This is a similar, but coarser, sample with a cloudy appearance arising from the presence of numerous small inclusions. Generally no banding was observed, though one crystal displayed a fine alignment of inclusions with a vaguely defined banding perpendicular to it. Few, if any, decisive secondary planes or fractures with typically small inclusions were encountered, the larger "individual" types of inclusions present being too numerous to have formed from fracture- or shear-planes, and thus considered to be of primary origin. In contrast to most other samples, the homogenisation temperatures of a large number of fluid inclusions were almost exclusively fairly low (117° to 197°C), with two higher values, at 247°C and 271°C. Very few inclusions gave any indication of the presence of a solid phase. Several fairly accurate determinations showed that the melting point of ice ranged in different inclusions from  $-14^{\circ}$  to  $-25^{\circ}$ C, two inclusions giving values of between  $-5^{\circ}$  and  $-10^{\circ}$ C, but with another solid phase (CO<sub>2</sub>-hydrate) melting just before  $0^{\circ}$ C.

#### P1865/76: Westward Ho Gold Mine

A variable sample, but mainly fine to medium in grain size with local areas of granulation, now appearing as fine equigranular clear quartz. Some inclusions in these areas were small to medium, and lay in zones probably representing growth bands of the originally coarser quartz. Homogenisation temperatures were generally low to medium possibly starting as low as 115°C, but some reached 377°C. Inclusions with square shaped solid were found in the coarser quartz. These solids were observed to be dissolving by about  $-60^{\circ}$ C, on warming up from lower temperatures, and had disappeared by about  $0^{\circ}\mathrm{C}$ , only to reform once more when allowed to stabilise at or slightly above room temperature. When heated, the same solids began to dissolve once more, having disappeared by  $+180^{\circ}$ C. The incidence of birefringent solids was much rarer. The melting point of ice was estimated to be about  $-27^{\circ}$ C, which is lower than the theoretical value for any aqueous solution containing NaCl and/or KCl, pointing (as in P1833/78) to the presence of another component. such as CaCl<sub>2</sub>. In salinity terms it represents >23% by wt. NaCl. Two inclusions displayed another phase, probably CO2.

#### P1867/76: Westward Ho Gold Mine

This specimen comprised slightly coarser quartz but with all gradations in grain size down to strained and recrystallised quartz. Numerous tiny inclusions were clearly related to later planes and sub-grain boundaries, etc. The coarser areas displayed varying though usually low concentrations of small, irregular inclusions also with variable degree of fill, but few with solid phases. A limited number had "double" bubbles, presumably due to the presence of  $\mathrm{CO}_2$ . On being heated, most of the 2-phase inclusions appeared to homogenise irreversibly in the gas phase, a not uncommon phenomenon in the present study. This could reflect a high incidence of  $\mathrm{CO}_2$ -filled inclusions,  $\mathrm{CO}_2$  having a much greater coefficient of expansion than water. Such homogenisation fell into the  $140^{\circ}\mathrm{C}$  to  $339^{\circ}\mathrm{C}$  range. One

inclusion was noted to homogenise at a critical point, the temperature of  $430^{\circ}\text{C}$  being equivalent to 9 wt.% NaCl. Other evidence of CO<sub>2</sub> was found in the freezing rims, which generated in some inclusions the formation of a third phase (H<sub>2</sub>O liquid + CO<sub>2</sub> liquid + CO<sub>2</sub> gas), the CO<sub>2</sub> homogenising in the liquid phase at from +8°C to +14°C ( $\equiv \sim 0.9 \text{g/ml}$ ). The melting point of ice was not measured accurately, though appeared to occur at about -22°C. Although the evidence from this sample appears to be somewhat ambiguous, it does indicate the original liquid to be saline and CO<sub>2</sub>-enriched and at low to high temperatures.

#### P1868/76: Westward Ho Gold Mine

This sample was represented by coarse strained quartz with finer (probably recrystallised) areas. Some areas displayed 2-phase inclusions in abundance, from larger irregular varieties to smaller kinds with negative crystal shape, all generally well distributed and only rarely giving an indication of primary? banding. The apparent degree of fill was relatively low but not constant, and many of the smaller inclusions had an appearance and behaviour suggestive of aqueous solution with liquid  $\rm CO_2$  phase. One inclusion decrepitated at  $168^{\rm O}{\rm C}$ , but otherwise homogenisation on heating took place from  $\rm 208^{\rm O}$  to  $\rm 424^{\rm O}{\rm C}$ , with ice-melting being variable, from  $\rm -20^{\rm O}{\rm C}$  to  $\rm -16^{\rm O}{\rm C}$  or higher (roughly 20 wt.% NaCl). The  $\rm CO_2$  liquid and gas phases homogenised between  $\rm +6^{\rm O}$  and  $\rm +16^{\rm O}{\rm C}$ . Variation in the nature of homogenisation was suggestive of entrapment on the boiling curve for some groups of inclusions.

#### P1869/76: Teetalpa Goldfield

The quartz in this case was very coarsely crystalline and relatively strainfree. Fluid inclusions were abundant, and though mostly not large, reached lengths of 40 and 70 µm in two cases. The inclusions showed a tendency to lie along 3 directions, which in places corresponded with fracture planes, and which may have been inherited from carbonate (rhombohedral boxwork textures). Many inclusions were 2-phase, some also with cubic solids (halite?). Homogenisation temperatures possibly fell into two ranges:  $189^{\circ}$  to  $299^{\circ}$ C and  $353^{\circ}$  to  $479^{\circ}$ C. However, apparent decrepitation (possibly of a  $C0_2$ -rich inclusion) occurred as low as  $175^{\circ}$ C, and in another as high as  $510^{\circ}$ C, with bubble-like structures still preserved in several inclusions at about 520°C. The solid phases had generally started to dissolve by about 180°C, this being completed around 218°C. For a system in equilibrium, this would represent 20-30 wt.% NaCl. During the freezing cycle evidence of the presence of CO2 became apparent, and although the phenomena observed were not completely understood, there appeared to be a phase change involving CO<sub>2</sub> at -46°. to  $-50^{\circ}$ C. It is suspected that two types of inclusion were represented: (i) smaller, more regular, commonly with negative crystal shape and containing saturated aqueous solution; (ii) larger, less regular, with aqueous solution enriched or even saturated in  $CO_2$ , in which ice melted at  $-15^{\circ}C$ .

#### P1870/76: Kirkeek's Treasure Gold Mine

Mostly coarse-grained quartz was to be found, with minor occurrences of fine granulated crystals, though much of the quartz showed strain-extinction and the development of sub-grain boundaries with related short fracture-planes. The abundant and often elongate fluid inclusions lay parallel to these structures, though sometimes appearing more as a "network". Salt-like solids were fairly common in the inclusions, but birefringent solids much less so. Heating caused homogenisation mostly in the range 253° to 395°C, the lowest recorded being 128°C. Although only a few direct measurements were made, nearly all observable fluid inclusions had homogenised or decrepitated by 430°C. The salt-like solids dissolved in the range 185°-230°C, though in one with a high homogenisation temperature, the solid survived to 350°C.

Some of the freezing phenomena were complex, with the last ice, or possibly  $NaC1.2H_2O$ , melting at  $-25^{O}C$  to  $-26^{O}C$ . All the evidence points to saline solutions of at least 25 wt.% NaCl, possibly with another component, such as  $CaCl_2$ , giving rise to quartz formation at moderate to high temperatures.

#### P1872/76: Eukaby Hill

This sample was characterised by coarse quartz crystals often outlined by fine, granular quartz. Fluid inclusions were abundant, well distributed and apparently earlier than the numerous short fractures observed. Most had a distinctive single-phase bubble, but without a solid phase. Heating this sample to about  $270^{\circ}\text{C}$  caused most fluids to homogenise or decrepitate. A few individual readings gave a range of  $255^{\circ}$  to  $289^{\circ}\text{C}$ . Cooling produced evidence of  $\text{CO}_2$  in the system, with a phase change occurring at about  $-45^{\circ}\text{C}$  and homogenisation of  $\text{CO}_2$  liquid and gas at  $17^{\circ}$  to  $18^{\circ}\text{C}$ . Ice-melting was not fixed with certainty, but probably occurred at about  $-19^{\circ}\text{C}$  (22-24 wt.% NaCl).

#### P1873/76: Ulooloo Goldfield

This section consisted of moderately coarse quartz displaying various stages of straining - from uneven extinction to the development of sub-grain boundaries and the formation of granular zones. Some very small structures resembling (secondary) inclusions were associated with partly annealed planes lying along sub-grain boundaries, but these were too small for study. The main inclusions were less common than in previous sections, and generally small, equant with negative crystal shape, and irregularly distributed. Apparently locally interspersed were larger, irregular and flatter inclusions up to 35 µm in size. The former displayed bubbles with heavier outlines (CO<sub>2</sub>?) than the latter, but this could reflect merely a difference in "depth" of the inclusions. Phases almost certainly representing CO<sub>2</sub> (liquid + gas) were noted during cooling, and most melting on reheating had been completed by  $-20^{\circ}$ C. In one instance a solid phase (CO<sub>2</sub>-hydrate?) finally melted at about  $+1^{\circ}$ C. Salt-like solids, observed in some inclusions, dissolved over the range  $-4^{\circ}$  to  $+18^{\circ}$ C, though in two instances salt-like solids, observed at room temperature, had not dissolved until heated to about 165°C. Homogenisation of liquid and gas phases occurred in the range 118°C to 183°C, with one exception at about 200°C.

#### 4. DISCUSSION

From the results obtained on the quartz veins from various Olary goldfields, a number of generalisations can be made. Although several generations of fluid inclusions may be present, there appears to be a better physical contrast in these Olary samples between what have been described as individual types (even where abundant and/or variable from small rounded or "euhedral" types to larger, irregular varieties) and those much smaller to "network" types, lying in planes related to subgrain boundaries. There is little doubt that the latter group are genetically linked to the straining and local granulation/recrystallisation of the quartz. Most of the former group have thus been identified as "primary", in the sense at least of being pre-straining of the quartz, and most of the results have been obtained from them.

These "primary" homogenisation temperatures display a considerable range - from low ( $\sim 120^{\circ}$ C) up to relatively very high ( $>500^{\circ}$ C), in contrast with the low values obtained from the secondary or later inclusions. The Taltabooka samples seem to be the only exceptions in having low values of homogenisation overall. A moderate percentage of samples provide evidence of the presence of CO<sub>2</sub> in solution. In a few of these the indicated density of the CO<sub>2</sub> component points to fairly high filling pressures, probably in excess of 500 atmospheres, and possibly

up to 1000 atmospheres. Many inclusions would appear to be saline, sometimes quite highly so, with a solid phase (almost certainly of NaCl) sometimes maintaining a saturated solution to moderately high temperatures. Salinities in the Olary samples would thus appear to range from 20 wt.% NaCl to higher values, with the possible exception again of Taltabooka. Other components, such as CaCl<sub>2</sub>, may also be present in some samples. Although accurate pressure corrections cannot be made, the salinity results suggest that the highest homogenisation temperatures measured could be at least 50°C lower than actual entrapment temperatures. Evidence from CO<sub>2</sub> inclusions tends to confirm this. Other solid phases are less commonly represented and too small for identification. The birefringent mineral reported is considered most likely to be carbonate though anhydrite is also a possibility. The occurrences of hematite within fluid inclusions, though rare, are not unexpected, but could represent solid phases trapped during quartz growth rather than daughter minerals of the inclusions.

In regard to the comparison of these Olary goldfield's samples with those from Wadnaminga and with the barren Anabama group, the Olary samples do not appear to have a preferential affinity for either, and probably represent a third group. Their highest homogenisation temperatures seem to equate for instance with those from the Wadnaminga auriferous quartz, but the range is much wider and the salinity values are higher, with possibly greater enrichment in  $CO_2$ . Contrasts between them and the Wadnaminga samples are hardly surprising, in view of the difference in gold/silver ratio between the two groups. Although freezing/salinity determinations have not been made on the barren Anabama group, the evidence via incidence of solid NaCl in the inclusions from this group points to lower salinity values than in many of the Olary samples.

One of the immediate problems, of course, lies in the relationship of the gold mineralisation to the formation of the quartz veins. In the Olary samples the general sequence of events appears to have been:

- (i) Formation of vein quartz
- (ii) Deposition of sulphides (with some gold?), locally in quartz fractures
- (iii) Slight deformation
- (iv) Introduction of carbonate with gold

With regard specifically to Wadnaminga, the native gold is contemporaneous with the vein quartz, though with a slightly later mineralisation also. Whether the time-span between quartz-formation and gold-precipitation in the main Olary area is sufficient to invalidate any assumptions about the temperature of gold-formation (and therefore comparisons based on it) is uncertain, but could be important. For instance, the temperature of solutions related to gold-formation in Kyzylkum, U.S.S.R., is reported to range from 60° to 600°C, but the actual precipitation of gold was restricted to the range 200-300°C (Adryanova et al, 1973).

In regard to wider comparisons, particularly with a view to making inferences on the origin(s) of the Wadnaminga and other Olary mineralising fluids, a brief review of the literature indicates firstly, that fluid inclusion data as obtained are seldom sufficient alone to invoke a particular process (e.g. magmatic, metamorphic). Usually information on the geochemistry of leachates from inclusions, on the chemistry of daughter minerals and sometimes isotope ratio studies, are required, together with detailed field relationships and mineral/ore paragenesis. Secondly, there is a variety of possible origins for auriferous ores. Thirdly (though partly as a consequence of the second indication), there is a large range of temperatures, if not of salinity and CO<sub>2</sub> content, over which the gold-mineralising fluids can be effective.

In Progress Report No. 1 of Project 1/1/176, (p.89 and Fig. 8) a contrast was invoked between the Wadnaminga auriferous deposits and a number of gold-deposits of the western United States, investigated by Nash (1972). The latter would appear to relate to the Carlin-type, shallow depth, hydrothermal to epithermal deposits, with low salinities and temperatures in the 200°-300°C range. Visible gold and gold-bearing quartz veins are reportedly sparse (Radtke etc al., 1974). Clearly, neither the Wadnaminga nor the other Olary goldfields fall into this type. This does not, however, indicate that they cannot be of hydrothermal or related origin, since gold-ore deposits of hydrothermal origin can display a wide range of temperatures, such as 180°-420°C, (Karobeinikov, 1974) and those of pneumatolytic-hydrothermal origin may result from both higher- and lower-temperature ore-forming fluids (Laz'ko et al., 1976). As was shown in Progress Report No. 1 (Fig. 8) the Wadnaminga data plotted much closer to those of porphyry copper deposits investigated by Nash but again it cannot be assumed on those grounds alone that an acid magmatic source for the fluids was ultimately responsible, for auriferous quartz veins have also been reported in intermediate/basic igneous complexes (Valyashko et al., 1973).

There appears as yet to be no published information on fluid inclusion studies in Australian goldfields, but the gold deposits of the USSR have probably been the subject of more fluid inclusion research than They can be divided into 7 ore formations on those of any other area. dominant mineral groups (Timofeevskii and Adryanova, 1973). Gold deposited during silicate-sulphide formation formed at very high temperatures (550 $^{\circ}$ -750°C), with gold-carbonate-sulphide at 340°-480°C, and gold-quartz as low as 210°C for the beginning of the ore stage in some deposits. Generally, early gold was produced in the 210°-410°C range and late gold in the range 100°-320°C, the main productive mineralisation being 150°-250°C. In the Chukotka fold system a combination of structure, mineral composition, nature of inclusions, together with textural and structural peculiarities of the ores, has provided the main criteria for classification of individual deposits as plutonic, volcanic, sub-volcanic contact-metasomatic etc. Consequently, gold-bearing deposits appear to be (Davidenko, 1973). capable of ranging from very shallow to deep-seated, from plutonic igneous and volcanic to metamorphic in genesis, from acid to basic in related composition and from high to low temperatures of crystallisation. According to Grebenchikov (1973) the temperature of ore-formation does not even have a real influence on the mineral composition of productive ores.

Fortunately, the literature does suggest that some generalisations can be made. With regard to temperature, it can be said that overall the highest temperature values for fluid inclusions do tend to relate to plutonic or contact metamorphic origins (often >500°C). Volcanic and sub-volcanic contact-metasomatic deposits are usually lower, perhaps ranging down to 350°C, hydrothermal normally below 400°C and pithermal below 300°C. Although a very wide range of formation-temperatures is known, many deposits, particularly involving vein mineralisation, tend to commence with high temperatures and end with low ones (Roedder, 1967). Although the gold itself can be formed at variable temperatures, such as 150°-450°C (Naiborodin & Sidorov, 1973), sometimes with pulsations and inversions (ibid.), nevertheless in many cases there is a tendency for a high-temperature pre-ore mineralisation or vein-formation followed by one or

two stages of auriferous ore formation, completed by a low-temperature set of non-auriferous veins. This may explain in part the occurrence of the low-temperature barren veins between Anabama and the more productive high-temperature area at Wadnaminga, but it does not explain the co-existence at Wadnaminga itself of both gold-bearing and barren veins formed at the same temperature. In cases like this, where fluid inclusion data cannot distinguish between productive and barren quartz, it may be necessary to study some other feature. For instance, Adryanova et al. (1973) have reported that IR absorption at specific wavelengths in quartz increases regularly from pre-ore to post-ore varieties.

In considering composition, gold-bearing veins(in contrast to barren ones) not uncommonly comprise multiphase inclusions (Davidenko & Valpeter, The presence of the latter has been correlated 1969), and contain  $CO_2$ . with gold content in South American, French and Canadian quartz veins, for instance (Machairas, 1970). Some reportedly contain more inclusions per volume of sample than the barren varieties (Ryabova, 1973). Although there are many chemical analyses and other data on ores and fluid inclusions available in the literature, much of the information seems to point in diverse directions, hence the lack of complete agreement as to the composition and origin of the fluids responsible for gold mineralisation. One aspect which commonly emerges is that the mineralising fluids appear The high salt, or at least chloride, content to be rich in CO2 and NaCl. appears to be necessary for the solubility of ore metals, (1,000-2,000 ppm metallic gold or platinum reportedly dissolved in chloride brines (Anderson & Burnham (1964) in Roedder, 1967). Dense Na-K-Ca-Cl brines are claimed to transport metals as chloride complexes particularly in hydrothermal situations (Cunningham, 1975), the solutions being highly acidic (Korobeinikov, 1974), or when transported in sulphide complexes, being alkaline (ibid). Some authorities regard the gold as being transported instead in the form of ions of thioaurates (e.g. AuHS and Rytuba and Dickson (1974) have  $Au(HS)_{2}^{-}$  (Letnikov et al., 1975). suggested that the gold goes into solution as Au(HS) 2 ions, but that the amount in solution is strongly affected by Cl ions, which thus have Timofeevskiy and Adryanova (1975) an indirect effect on gold solubility. considered that CO2 plus carbonate ions represent the main composition of parent solutions in auriferous quartz veins.

Whatever the precise answer, most authorities agree that the boiling of  $\rm H_2O-CO_2$  solutions and subsequent degassing of  $\rm CO_2$  stimulates the formation of commercial gold ores (e.g. Andrusenko et al., 1975). The latter authors further specify temperatures of  $240^{\circ}-320^{\circ}\rm C$  for nearsurface deposits, and  $260^{\circ}-380^{\circ}\rm C$  for intermediate depths. Popivnyak (1975) specifies that the presence of heterogeneous fluid inclusions containing  $\rm CO_2$  with a formation range  $200-260^{\circ}\rm C$  constitute a perspecting tool.

Auriferous metamorphogenetic quartz veins probably show a similar transfer of gold (Letnikov et al., 1975), though the temperature of formation of the ore-deposits may be a function of metamorphic facies (Konavlov, 1975) with ranges, for instance of 260°C up to 460°C.

Information via fluid inclusion studies on the pressure of gold-forming ores is more limited. The evidence from shallow deposits naturally points to relative low pressures, usually well below 500 atmospheres (e.g. Nash & Cunningham, 1973). In others the pressure may be variable (600 to 1100 atmospheres, for instance, in different stages of oreformation for 70 deposits studied by Popivnyak (1975) in the Muyskiy gold ore region). In deposits related to regional metamorphism the pressure can be much higher (e.g. 1000-2000 atm. (Konovalov, 1975)). In the

auriferous quartz veins from Olary (excluding Wadnaminga) precise indications of pressure cannot be given, but in some cases the evidence suggests a minimum of 400 atmospheres, while the evidence from  $\rm CO_2$  in some inclusions indicates that it may be at least 1000 atmospheres. There is no reason, however, to invoke such high pressures for those inclusions of low salinity and high degree of fill, which may, of course, represent a different generation from the highly saline,  $\rm CO_2$ -rich inclusions.

If reliable estimates of pressure can be made in conjunction with temperature measurements, then there is hope that in a given goldfield the different genetic types of quartz may be recognised. Veres et al. (1973) have reported that thermobarograms are typomorphic features of minerals and have typical curve configurations, enabling gold-bearing quartz to be distinguished from barren quartz.

Taking all the factors discussed together, it could be argued that the auriferous deposits of Olary generally fit into the main trend of the goldfields of the USSR - moderate to high temperatures and pressures of fluids similar to dense, CO2-rich brines, and possibly of magmatic/hydrothermal origin. This leaves the problem of explaining the range of features noted (grading to low temperature/pressure and lower salinity conditions) - does this represent a range in conditions under which gold was precipitated, or does it represent a time-span of increasingly later and lower-temperature post-ore conditions? might be resolved by a closer study of the problem of primary v. secondary inclusions in quartz veins though it must be expected that, since the quartz veins in the Olary region are probably much older than most of those investigated on a world-wide basis, the original fluid inclusions may have been destroyed or modified sufficiently to provide unreliable information. Attempts to relate the gold more specifically to quartz deposition might also be enlightening, possibly by checking for thermal gradients across and along specific gold-bearing veins, as reported for instance by Kolkovski et al. (1974).

Although the auriferous veins around Wadnaminga have given no indication of a zonation related to the Anabama Granite intrusion, it may be that the fluids responsible relate to a more deep-seated origin. If the Wadnaminga and other auriferous quartz veins in the general Olary area are instead ultimately of metamorphic origin, further work might be expected to disclose a temperature zonation related to metamorphic facies across the region.

#### 5. SUMMARY AND CONCLUSIONS

Fluid inclusion studies of barren and auriferous vein quartz from the general Olary region have indicated that the formation of the quartz (and possibly therefore of the gold) has apparently occurred over a wide range of conditions, even within a single goldfield. The fluids responsible have at some time been high-temperature, fairly to strongly saline, and in some cases enriched in  $\rm CO_2$  at high pressures. These conditions appear to be the most conducive to gold formation, and may be magmatic/hydrothermal in origin.

More specifically, there appears to be no immediate difference in the fluid inclusion data from the auriferous veins as compared to the barren veins at Wadnaminga, both having formed at high temperatures. The data are, however, limited. The barren veins between Wadnaminga and the Anabama Granite intrusion were formed at lower temperatures, from less saline conditions, and appear to be unrelated to those at Wadnaminga. The main goldfields in the Olary region may not be related to either of these groups, but further work would be required to indicate whether all of them fit into a regional, and possibly therefore a metamorphic, pattern or are instead related to more deep-seated intrusions. Even if this were not possible there is still reason to believe that the information from fluid inclusion studies in the Olary region could be used as a prospecting tool for gold. To firmly establish this potential would also require the study of considerably more samples in conjunction with their techniques, such as the use of electron microprobe analysis and/or scanning electron microscopy as an aid to identifying solid phases in inclusions.

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<sup>\*</sup>Abstract in Proceedings of COFFI

TABLE 1: SUMMARY OF FLUID INCLUSION DATA

		•	Homogenisation Temperature, °C ?Primary ?Secondary				
Goldfield, etc	c. Sample No.	Nature of Quartz	Inclusions	?Secondary Inclusions	Decrepitation Temp., °C	Freezing Data	Comments
Mt. Grainger	P1824/76	Coarse, slightly strained; finer,granular. Numerous ting inclusions	-	Unsuitable	-	-	Inclusions mostly single-phase; large ones decrepitated.
Mt. Grainger	P1826/76	Medium to coarse, slightly strained. Preserved sedimen- tary structures in places.	152 to 178 (to 192?)	?		Unsuitable	Inclusions did not appear to freeze.
Mt. Grainger	P1829/76	Coarse, strained; finer, granular areas. Numerous small inclusions.	232 to <300; >364 to 528+	145 to 278	364 to 440		l Limited CO₂. Solid NaCl? up to C.∿340°. Rare birefringent solid.
Mt. Grainger	P1831/76	Very coarse, rel.unstrained; loc. granular	110? 247 to 530+	110?	>400	Unsuitable	<del>-</del>
Mt. Grainger	P1833/76	Coarse, strained. Concentrations of inclusions.	205 to 350; >500		241 to ∿345; 425	Ice, etc. melts	Solid NaCl + birefringent solid
Mt. Grainger	P1834/76	Coarse, strained. Sieved with inclusions.	168 to 228; 392 to 509+		228-258	Uncertain	Solid NaCl dissolves 170°C to >330°C. Birefringent solid.
Ajax	P1837/76	Coarse, clear quartz, slightly strained; finer, granular quartz.	∿180 to 420	∿150 to 170	270–363	Unsuitable	
Ajax	P1839/76	Coarse with remnant(?) granular bands. "Network" inclusions + larger individua	182; 349 to 460+	133?	184; <300	Ice, etc. melts	Solid NaCl dissolves 143°C to 160°C. Birefringent solid.
Wankaringa	P1842/76	Coarse, strained. Fine "network" inclusions + larger individuals.	∿162 to 314	158	168 - 7	Ice, etc. melts -24°C	CO <sub>2</sub> + 2nd gas? - L + G homogenise up to 22°C. Solid NaCl up to ~180°C.
<b>V</b> a≱karinga	P184 /76	Coarse, slightly strained. Primary growth bands with inclusions.	170 to >330 Up to 480??	٠ .	~276-460	Ice finally melting - 3°C	CO <sub>2</sub> liquid+gas homogenises at or above +15°C. Solid NaCl? tarely above O°C.
Taltabooka	P1860/76	Coarse, slightly strained. Cloudy due to inclusions, including "networks".	155 to ~233; >339?	138 to 168	∿199-340	Ice melts -11°/ -12°C	Limited CO2.
Taltabooka	P1861/76	Very coarse; cloudy	117 to 197; 247; 271		197 <b>- t</b>	Ice melting variable; -25° to -5°C	Solid NaCl rare.
Westward Ho	P1865/76	Fine to medium; loc. granular	115; 200 to 377		1957 237 to 377	Ice, etc. melts -27°C	Some solid NaCl? dissolves at up to $180^{\circ}\text{C}$ .
Westward Ho	P1867/76	Coarse to fine, granulated quartz with numerous tiny inclusions (prob.secondary)	140 to 339; 430		175 to 313		CO <sub>2</sub> liquid+gas homogenises +8° to +14°C. Homogenisation at critical point = 430°C.
Vestward Ho	P1868/76	Coarse, strained to granular, loc. abundant inclusions.	208 to 424	· .	216 to 326	variable: -20°	CO <sub>2</sub> liquid+gas homogenises +6° to +16°C. Some evidence of boiling.
Ceetalpa	P1869/76	free. Abundant inclusions.	189 to 299; 353 to 479; up to 520?	٠	175? up to ∿510		NaCl solids in some case's dissolving 180°C to 218°C; Others CO <sub>2</sub> -rich.
irkeek's 'reasure	P1870/76	Most coarse, strained, with loc. fine granular areas. Inclusions fairly abundant.	128; 253 to 395				Evidence of CO <sub>2</sub> . Solid NaCl dissolves 185° to 230°C.
ukaby Hill	P1872/76	Coarse quartz, intergrown or coutlined by fine granular quartz.	255 to 289	,			CO₂ liquid+gas homogenise ∿+17°C.
100100	P1873/76		118 to 183; ∿200		216; ~363		CO <sub>2</sub> ? Solid NaCl dissolves -4° to +165°C.

066

FLUID INCLUSION STUDIES OF S.A. ORES, ROCKS AND MINERALS

Department of Mines & Energy, South Australia

P.R. Report No.4

1/1/182.

July 1979



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24 July 1979

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067

FLUID INCLUSION STUDIES OF S.A. ORES,

ROCKS AND MINERALS

PROGRESS REPORT NO. 4

Investigation and Report by: Dr Barry Collins

Manager, Geological Services Division: Dr Keith J. Henley

for Norton Jackson, Managing Director.

#### FLUID INCLUSION STUDIES OF MT. PAINTER MINERAL CONCENTRATES

#### 1. INTRODUCTION

In the period 1976/77 microthermometric studies of fluid inclusions in quartz and, to a limited extent, in monazite, were carried out on hematite breccias, etc., from Mt. Painter, as a possible means of shedding light on the origin of these rocks. The results of such studies were presented in Progress Report No. 2 under Project 1/1/182B ("Fluid Inclusion Studies of S.A. Ores, Rocks and Minerals - B. Metallic Minerals").

Subsequent to this a service request was received in July 1978, for fluid inclusion studies of specular hematite with monazite from Radium Ridge (Mt. Painter). This was commenced as job GS 30/79, file no. 12/05/182, but owing to technical problems the investigation was extended to include monazite concentrates and residues, the work being carried out under Project 1/1/182. The results are now reported below.

#### 2. THE NATURE OF THE MATERIAL

#### 2.1 Introduction

The specular hematite sample submitted (RBM 68/76, P919/78) was located in No. 2 workings on Radium Ridge (NFM R17/092  $(35)_{\rm SP}$ ) and had previously been examined by thin and polished sections as sample P1300/76 (Report MP 305/77). Here it had been described as having up to 80% of opaques, mainly as masses of intergrown hematite (martite), probably after magnetite. According to the report, 5-10% monazite was present, occurring as aggregates and crystals intergrown with the hematite, and in a few cases reaching 3 mm in length. Textural evidence was claimed to indicate that the monazite was pre-hematite, though possibly synchronous with the earlier magnetite, and that the remaining interstices were occupied by a mass of intergrown quartz crystals (secondary).

In a general preface to the petrographic descriptions in this same report, the comment had been made that the quartz-hematite rocks described therein (of which P1300/76 was one) differed from previously-described hematitic breccias in the absence of clasts of granitic or granitoid rocks, and that because of this and the presence of much secondary quartz, the origin of the quartz-hematite rocks was not evident. However, the author did point out the many aspects of similarity between the two groups of rocks as supporting the suggestion of genetic relationship.

Clearly, mineral paragenesis is critical to the use or interpretation of fluid inclusion results to determine the conditions of formation and subsequent thermal history. However, even without knowing the precise order of mineral formation, at least some information on the temperature and pressure to which the rock was at some stage subjected could be usefully obtained. Consequently, it was decided again to attempt microthermometric studies on the monazite, as being the more stable mineral (in contrast to quartz).

#### 2.3 Monazite in Section

The problems encountered in preparing from hematite breccias the special double-polished sections required for microthermometry have already been outlined in the Progress Report referred to. The specific examination of monazite in these breccias presents compounding difficulties, as also pointed out previously. Nevertheless, sample P1300/76 (now re-submitted as P919/78)

presented an instance in which two of the problems - sparsity and small crystal size of monazite - appeared to be at a minimum.

Four double-polished thin sections were prepared, two initially at AMDEL and two subsequently at LaTrobe University (Melbourne), in order to augment the monazite available for study and to compare methods of preparation, particularly with respect to preventing induced decrepitation of inclusions (see previous report, section 3.2.3, p3). This material was used where possible for both heating and freezing studies, with limited success. Owing to the rarity of suitable inclusions, however (many being apparently decrepitated), attention then turned to the possibility of using monazite concentrates, already available from other studies of Mt. Painter breccias. The main project thus commenced with a view to: (1) overcoming problems in sample preparation likely to induce decrepitation; (2) establishing a method of preparing and studying mineral concentrates for microthermometry; and (3) carrying out such a study on some nine monazite mineral concentrates from Mt. Painter.

#### 2.3 Monazite Concentrates, etc.

Six samples of monazite concentrates were received initially, the bulk of the material having been used elsewhere for age-determination. Of these, two samples (00925/1 and 00932/1) provided insufficient material for use, leaving no's. 00928/2, 00929/3, 00930/4 and 00931/3, in which the majority of the grains were in the 100-200 µm size range, and only about half of which appeared to be of crushed monazite. Although attempts were later made to purify these samples, virtually no material worthy of study was obtained, and it was then agreed to re-examine the monazite residues with a view to extracting the clear quartz grains (="late idiomorphic quartz") for microthermometric studies, this quartz being the nearest suitable mineral to monazite in the paragenetic sequence. The presence of other generations of quartz made separation difficult and the problem was approached by firstly concentrating the quartz and then hand-picking the clearer and (where obvious) more euhedral crystals.

Material thus selected was used to prepare double-polished thick sections of grain mounts, the finished grains then being liberated from the section and individually arranged on the microscope heating stage. In addition, material from the monazite concentrates and hand-picked grains of quartz were inspected in refractive index oil for the presence of suitable inclusions. Preliminary low-temperature heating runs were carried out on some grains temporarily mounted in oil, as described later.

#### 3. SAMPLE PREPARATION

#### 3.1 Double-Polished Thick Sections of Grain Mounts

Previous methods of sample preparation have relied upon the normally-accepted technique of mounting a polished slab of rock on a glass slide using a glue melting at temperatures of up to 150°C. The possibility of such temperatures, either alone or in conjunction with frictional temperatures, being responsible for the overheating of inclusions to induce decrepitation, was discussed with other workers at Adelaide and LaTrobe Universities. It was generally felt that frictional problems would be minimal, but that mounting temperatures might affect the very low-temperature fluid inclusions. Communication on the subject was also made with the American authority, Dr Edwin Roedder, who recommended a method of grain mount preparation and a suitable low-temperature bonding material. Samples of the latter (Aremco 'Crystalbond') were obtained from America and were used for most of the sections of grain mounts prepared for the project.

The method involved mounting the selected grains on a glass slide by slow heating from cold to the minimum temperature required to effect bonding. After grinding and polishing, the glass slide was trimmed and the polished surface was then bonded onto another glass slide as before. The original portion of the glass slide was then ground away to expose the back surface of the grains, which, after polishing, were removed by soaking in acetone. After microscopic examination groups or individually slected grains were placed on the heating stage in the usual manner, making sure that each grain rested on a polished surface.

In terms of operational efficiency, the method was found to be laborious, quite apart from the time spent on further purification of the monazite by heavy liquid separation. In addition, a not insignificant percentage of grains was lost in polishing and in cleaning and transfer to the heating stage, necessitating duplication of sections in several instances. The quality of polish was inferior to that obtainable with normal rock sections, especially with the smaller grains. In fact, since the grains supplied in concentrate no. 00931/3 were below average size, no material worthy of study emerged from preparation.

However, the method has been shown to work, and where studies of rare minerals necessitate the use of mineral concentrates, it could provide a means of doing so if attention is paid to liberation and separation designed for this purpose. The bonding material used is also superior to any other known in minimising working temperatures and in its ease of solvency for releasing polished material. As it happened, the monazites prepared by this method showed roughly the same degree of inclusion decrepitation as observed in the previous project. This tends to indicate that the monazite in Mt. Painter has decrepitated by natural means.

#### 3.2 Other Methods of Preparation

As briefly mentioned, normal methods of double-polished thick section-making were employed on the rock sample P919/78. Examination of the monazite inclusions showed no significant differences as between the material prepared at AMDEL and that prepared at LaTrobe, with regard to proportion of apparently decrepitated inclusions. Comparison with the polished grain mounts specially prepared from the monazite concentrates showed little difference in this regard either.

As an extra check on the possible effects of section preparation on fluid inclusions, unpolished grains of monazite concentrate were mounted in high refractive index oil to facilitate optical study of their inclusions. Again, no significant differences in the state of the fluid inclusions were observed.

A similar method of temporarily mounting grains of quartz in a matching refractive index oil was used, and attempts were subsequently made in both cases to heat the oil-mounted grains as an additional check on the few suspected two-phase inclusions present. No further information was obtained from the monazite, but confirmation of inclusions with very low temperatures of homogenisation was obtained for the quartz in question. Heating of the oil in the case of monazite was limited to about 150°C, with difficulties arising not only from oil evaporation but also from the liberation of sulphurous fumes, since the transparent thermal insulation cap for the heating stage could not be used in position over the oil-mounted samples. In the case of quartz, temperatures of up to about 200°C were achieved before oil evaporation and/or breakdown occurred.

Finally, all the available material was re-examined and, where practical, heated without the insulating cap in place on the heating stage, in order to improve optical resolution. By so doing, some inclusions, previously precluded from observation, were able to be studied during heating and some valuable information was obtained. Although such results are less reliable since the sample is thus heated effectively on an open stage without the glass insulating cap, the errors were reduced to a minimum by carrying cut calibration runs with and without thermal insulation, and then applying a correction factor. The 'corrected' results will clearly lie closest to true values at lowest temperatures (room temperature) and diverge upwards. It is estimated that by this method the total error on quoted results at 350°C would be ±15°, at most, which, for most purposes, is quite acceptable.

#### 4. DESCRIPTION OF SAMPLES & RESULTS

#### 4.1 Monazite

Re-examination of earlier sections of samples used in Progress Report No. 2 failed to reveal any hitherto unmeasured inclusions. Sample P919/78 thus provided the only new material available in rock section for study.

Sample P919/78: Here, the monazite was relatively abundant and coarse, occasionally as isolated hypidiomorphic crystals, but usually as aggregates of what strongly resembled crystal fragments, as found in previous samples (Progress Report No. 2). Many of these appeared shattered and, rarely, bent.

Virtually all monazites contained inclusions, and although the decisive presence of gas bubbles was very seldom evident in these inclusions, most of them appeared otherwise typical of fluid inclusions. Of these the majority further appeared to be primary. They ranged in size from about 1 µm up to around 50 µm, usually as well-formed, evenly-distributed individuals, varying from slightly rounded to those with negative crystal shape and often aligned roughly parallel to a single direction (possibly in the 001 plane). Gradations to large, irregular varieties were sometimes encountered, however. At first sight there appeared to be two types of fluid present: a relatively low relief 'clear' variety and a generally more common type giving rise to inclusions with heavy outlines and/or dark interiors, but the latter may be no more than decrepitated instances of the former (see below). Although fluid inclusion alignment possibly coincided with a cleavage direction (which might imply a secondary origin), no cleavage itself was obvious. Instead, a less regular fracture pattern, probably related to the brecciation of the monazite, was observed to cut across some of these inclusions.

In addition to the above, some monazite crystals displayed other generally low relief irregular inclusions, also without gas bubbles, but usually highly elongated and lying parallel to the main inclusions. In one instance this direction coincided with a fracture cleavage, along and across which some inclusions were interconnected, indicating that they were formed from fractures as later, secondary inclusions, now preserved in the process of 'necking down'. Rarely the distal ends of such inclusions displayed negative crystal outlines, thus raising some doubt as to the truly primary nature of the earlier inclusions, many of which had negative crystal shape.

Although literally thousands of inclusions were examined, less than 1% gave any indication of the presence of a gas phase. Most of these rare two-phase inclusions were relatively small in size and none was of the 'secondary' kind. A number of larger ones with what seemed certain to be a gas bubble were raised to high temperatures without any effect or change at all in the supposed bubble. Nearly all inclusions which did have a gas phase homogenised at low temperatures, the lowest being just above 50°C, with several below 100°C, increasing in

frequency up to around  $160^{\circ}\text{C}$ , the highest readings in this range being about  $180^{\circ}\text{C}$ . Decrepitation temperatures were correspondingly low, some individuals 'decrepitating' by slow leakage around  $100^{\circ}\text{C}$ , others more rapidly from  $125^{\circ}\text{C}$  up to about  $210^{\circ}\text{C}$ . Two inclusions had reproducible homogenisation temperatures of  $335^{\circ}\text{C}$  and  $342^{\circ}\text{C}$  with some evidence elsewhere of decrepitation just above  $400^{\circ}\text{C}$ .

There were virtually no inclusions with recognisable solid inclusions other than hematite. One or two suspected of having daughter crystals showed no change in appearance even when heated to high temperatures. One single instance was noted in which five separate, moderately birefringent, solid phases were present, none of them altering in any way, even after heating to  $500^{\circ}$ C.

Freezing experiments were more severely limited by the quantity of suitable inclusions, since small inclusions are more difficult to freeze and the ice, etc., in them more difficult to recognise and to observe when melting occurs. Several suitable, relatively large inclusions were able to be frozen, this occurring at varying temperatures in different inclusions from  $-50^{\circ}$ C downwards. Some inclusions could not be induced to do so, even as low as  $-170^{\circ}$ C, possibly an indication of high purity (in  $\rm H_2O$ ). The gas bubbles generally contrated on cooling and effectively disappeared on freezing in some instances. In one, however, no changes to the bubble were noted with cooling.

During initial warming no distinct phase changes were observed, except in the possible re-appearance of the bubble in small inclusions. Some re-organization of the ice was observed from  $-50^{\circ}$ C upwards, but commencement of melting of ice or what may have been NaCl.2H<sub>2</sub>O could not be pin-pointed. Indications of melting varied widely, as did the temperature of reappearance of the gas bubble (from the instance where it survived freezing, to temperatures right up to  $-2.4^{\circ}$ C, after last melting). The last melting of ice itself varied from about  $-13^{\circ}$ C to about  $-3^{\circ}$ C (equivalent to up to 17% wt. NaCl) and time was thus not spent in its accurate measurement. The formation of other solid phases was not observed.

The information to be derived from freezing experiments is that, though variable, the fluids present are equivalent only to weakly or very weakly saline ones. This is only to be expected from the low homogenisation temperatures and absence of solid phases.

Sample 00925/1: Insufficient monazite material remained for section-making.

Sample 00928/2: The potentially useful monazite material was considered to be too limited to warrant section-making.

Sample 00929/3: Again, examination in oil of mineral concentrates failed to reveal any two-phase inclusions of value.

Sample 00930/4: Some 80% of the monazite grains appeared to contain fluid inclusions, but gas bubbles were not evident. A sample of the concentrate was heated in oil to about  $150^{\circ}\text{C}$  without movement of any gas bubble being detected. Oil evaporation limited further temperature increases. However, a double polished grain mount was prepared. Two polished grains were found in which two-phase inclusions (liquid + gas) could be recognised by the movement of the gas phase at room temperature. In one, homogenisation occurred at  $37^{\circ}\text{C}$ , in the other at  $40^{\circ}\text{C}$ . A possible explanation is that the fluid is a mixture of  $\text{CO}_2$  and  $\text{H}_2\text{S}$ . In a third grain containing the usual apparently decrepitated inclusions, two still retaining gas bubbles gave homogenisation values of about  $150^{\circ}\text{C}$  and  $162^{\circ}\text{C}$ .

Sample 00931/3: The polished grains available were found to be too small for detailed optical work and no obvious two-phase inclusions were observable.

#### 4.2 Quartz

Owing to prior crushing of the samples for liberation and separation of the monazite, the 'late idiomorphic quartz' in the residues could seldom be recognized by its idiomorphic nature. It was generally selected as being represented by the clearest quartz grains, though the clearest grains of all, having thus virtually no fluid inclusions, were of little value for the current exercise. To some extent, therefore, the material selected for study may be regarded as a compromise.

As expected, the volume of fluid inclusions actually able to be observed in detail was not great, and in most cases the number of quartz grains available could not compensate for this. The size and shape variation of individual inclusions was not atypical of quartz, but more restricted than that described in Progress Report No. 2. Generally absent were the 'network types' found in most other Mt. Painter breccias, etc.; also missing were concentrations of inclusions in obvious growth bands, indicating a primary origin. Instead, most of those measurable inclusions were more individual in occurrence, or in small groups, ranging from partly rounded to more 'euhdral' shapes and to irregular (the biggest group). The size range was probably less than for those of previous studies, the maximum noted being 70 µm, with most in the 5-15  $\mu m$  range. These are assumed to be primary, or at least to be the earlier inclusions, in contrast to those lying either in regular planes (annealed fractures) or to those following possible sub-grain boundaries. The last-mentioned (and least common) may, of course, be related to an older quartz fabric still preserved in the now-recrystallized quartz.

The quartz in the current study also contrasted slightly with that of the previous Mt. Painter report in having a higher percentage of single-phase inclusions, (i.e. liquid but no bubble) to the extent that in some samples the information obtained was very limited. Also restricted was the incidence of daughter minerals, except in two samples (00929 and 00931) where some of the fluid inclusions appeared to contain several different types of solid.

Sample 00924: The quartz in this sample was examined only in oil. Most quartz grains had inclusions preserved, some in relative abundance, and some approaching the spongy, 'network' situation previously referred to. Nearly all were irregular in form. The proportion having a gas phase was also variable, from grain to grain, and it is suspected that more than one type of quartz was present, even after hand-picking. Individual grains were heated in oil to  $130^{\circ}$ C, with variable results. Some gas bubbles were still evident, but homogenisation could not have been greatly in excess of this temperature, the lowest recorded being 95°, but one shrinking out of view at 45°C. One grain displayed inclusions still with relatively large gas bubbles at  $130^{\circ}$ C. The indicated temperature of formation is thus very low to moderate at most.

Very few inclusions contained solid phases other than small specks of hematite, identified from its orange-red colour.

Sample 00925: Most inclusions here, too, were irregular in outline, but in some grains there were smaller inclusions with a tendency to a more regular or partly 'euhedral' shape, with heavier outlines and moving gas bubbles, resembling two-phase  $\rm CO_2$  inclusions. Heating, however, failed to cause homogenisation below  $\rm 32^{\circ}C$ , which would have virtually confirmed the presence of  $\rm CO_2$ . The minimum homogenisation temperature could not be measured accurately owing to limits on optical resolution, but most could not have homogenised below  $\rm 80^{\circ}C$ . In one grain with numerous such examples only a few survived  $\rm 90^{\circ}C$  as two phases, thus providing a reasonable estimate of the minimum value. In other grains the upper limit appeared to be  $\rm 140^{\circ}C$  for these types.

The more normal inclusions (with no rigid distinction from the previous) also had generally low homogenisation temperatures, from just over  $100^{\circ}\text{C}$  to about  $145^{\circ}\text{C}$ . Solid phases, not uncommon, appeared to be cubic (?halite) and gave indication of contraction at over  $110^{\circ}\text{C}$ , but not completely dissolving in some cases until about  $240^{\circ}\text{C}$ . This would indicate an equivalent wt.% NaCl of up to 30, or even 35.

Small, often elongate inclusions were found in some grains to lie parallel to slightly curved planes, sometimes in two directions, and possibly representing old grain boundaries or fractures during growth (?pseudosecondary). Whatever their origin, the fluid inclusions in them were of low-temperature origin, at or below  $120^{\circ}$ C, except in rare instances of, again, up to about  $140^{\circ}$ C, with one value of around  $235^{\circ}$ C being noted.

In this sample some generally large irregular inclusions appeared to have suffered prior decrepitation. During tests a number of 'normal' inclusions, when heated above their homogenisation points, suffered a form of partial decrepitation or inelastic stretching, manifest as the relatively sudden appearance or re-appearance of a small gas phase. With further heating these bubbles underwent a further and usually reproducible homogenisation event at temperatures up to 355°C. Complete decrepitation also occurred. This may be an explanation for the isolated higher than normal values obtained, and may also give an indication of the temperatures responsible for the occurrence of previously-decrepitated inclusions, the implication being that post-crystallisational temperatures may have been temporarily much higher than the generally very low ones assumed for initial quartz crystallisation.

Sample 00926: Few grains with usable fluid inclusions were to be found in this sample. Nevertheless, a range of shapes and sizes of usually well-dispersed inclusions was noted. Homogenisation ranged from about  $105\,^{\circ}\text{C}$  to  $215\,^{\circ}\text{C}$ . One group of secondary inclusions, clearly produced by 'necking down' from fracture planes gave a spuriously high value of over  $420\,^{\circ}\text{C}$ .

Sample 00927: Initial inspection of selected quartz grains showed that very few had two-phase fluid inclusions and section-preparation was not considered to be feasible. Many of the visible inclusions resembled previously-decrepitated fluid inclusions. Some grains were heated in oil which revealed that no two-phase fluid inclusions were visible above 128°C. This sample is thus presumed to have a generally very low temperature of homogenisation. No solids were observed.

Sample 00928: This sample had a range of inclusions, from smaller, more regular kinds to bigger more irregular ones, with several of intermediate size and partly with negative crystal outline. The largest could be recognised in some cases as three-phase, and although the homogenisation temperature of the two bubbles (inner gas and outer liquid, presumably) could seldom be fixed, their appearance was typical of  $\rm CO_2$ -filled inclusions. In two instances a value of 24°C was obtained, this being normal room temperature, and indicating the possibility of other  $\rm CO_2$  inclusions being not recognised because their homogenisation point was just below room temperature.

The homogenisation of the aqueous phases ranged from very low  $(75^{\circ}\text{C})$  up to moderate temperatures  $(198^{\circ}\text{C})$ , though two apparently primary isolated inclusions in one grain reached values of approximately  $285^{\circ}\text{C}$  and  $300^{\circ}\text{C}$ . The apparent presence of a solid phase in some was investigated, but no changes were observed up to the limit of heating  $(200^{\circ}\text{C})$ . Very rare hematite was noted, however.

Grains of apparently secondary origin also gave mostly very low homogenisation values (about  $110^{\circ}$ C to  $140^{\circ}$ C), but in a few instances these, too, had higher values, up to  $320^{\circ}$ C.

Sample 00929: Very few suitable grains survived the polishing stage of preparation in this sample. From grains mounted in oil some could be recognised as having small regular CO<sub>2</sub> fluid inclusions, homogenising around 25°C, similar to the previous sample, and equivalent to a CO<sub>2</sub> density of about 0.7g/ml. Inclusions were generally limited in this sample, however, and even when present no evidence of a gas phase could be found in all but a few. Of these few, some were probably secondary, produced by 'necking down' after the annealing of fractures, and now occurring in groups of irregular inclusions in which only one or two had a gas bubble. Not unexpectedly, homogenisation temperatures derived from these were variable, ranging from low up to about 230/240°C, and possibly thus unreliable.

The inclusions more likely to be of primary origin were insufficient in numbers to give an indication of the true range of homogenisation temperatures. These may start as low as 105°C, but certainly by 128°C confirmatory evidence from dissolving solids was obtained. A number of ?primary inclusions contained solid phases of hematite and/or a moderate/high birefringent mineral. latter could be calcite, but positive identification of such small entities (seldom more than 2 μm) is hazardous. One instance was found in which almost the entire 10 µm inclusion was occupied by a medium-sized gas bubble, two larger solids (one isotropic, one weakly to moderately birefringent), two different and smaller solids (one isotropic, the other moderately to highly birefringent) and two small, flake-like masses of hematite. The larger birefringent solid dissolved at about 125°C (?gypsum), the gas phase disappearing at about 226°C accompanied by that in an adjacent inclusion. The smaller isotropic solid had disappeared by 300°C. Melting or dissolving of the other isotropic solid was apparent by about 380°C, by which time most other similar inclusions had decrepitated, but complete melting or dissolving had not occurred when, just below 480°C, the inclusion itself decrepitated. Consequently, the range of homogenisation temperatures may be considerable in this sample and would certainly seem to reach well above 200°C. Equivalent salinities could be up to or even above 40 wt.% NaCl.

Sample 00930: A number of large, irregular inclusions in the few polished grains examined appeared to be decrepitated. Other fairly large individuals, and possibly of secondary origin, contained  $\rm CO_2$  and homogenised once again in the 23-27°C range. Other more probably primary inclusions displayed a somewhat variable range of temperatures from about  $\rm 125^{\circ}C$  to  $\rm 250^{\circ}C$  or so. Although the available information is too limited for definite conclusions on formation temperatures, the higher values were supported by the temperatures at which small isotropic solid phases (?halite) in some inclusions dissolved. Larger birefringent solids were also present but showed no inclination to dissolve within the temperature range specified.

Sample 00931: This sample appeared to have a better representation of probable primary inclusions, many of which displayed a gas bubble and one or more solid phases. Some, in fact, could be likened to that described in sample 00929, in having a gas phase plus up to four solid phases, one or more of which was birefringent. This situation is thus relatively complex, and presumably represents a more complex mother fluid from which the quartz crystallised.

Generally, the simpler fluid inclusions (few phases) gave lower indications of homogenisation temperatures, starting at about 83°C. Unfortunately, slow apparent leakage of some of these caused later reappearance of the gas phase, which underwent two and sometimes three cycles of homogenisation, thus tending to undermine the confidence which can be placed on the other data. It is considered, however, that too many other closely-related inclusions give moderately high temperatures of homogenisation for this to be entirely spurious. One contributing factor, of course, is the presence of solids, some of which were slowly dissolving over the observed range of gas/liquid homogenisation and which must have affected the apparent homogenisation. Taking the results

at face value, the gas phase disappeared on heating in the range of  $\sim 100^{\circ}\text{C}-282^{\circ}\text{C}$ , with some isotropic solids also dissolving in this range (equivalent to a salinity range of 26-36 wt.% NaCl). Usually, towards the end of this stage, the remaining isotropic solid was apparently larger and more obvious in hexagonal outline, most probably as a thin hexagonal plate, then starting to dissolve just before the birefringent solid(s). In some inclusions the isotropic solid was readily visible as a cube. The highest recorded temperature for dissolving of solids was roughly 395°C ( $\equiv 45$  wt.% NaCl), but in two instances solid phases (?birefringent) had not completely dissolved (nor seemed further reduced in the last  $100^{\circ}\text{C}$  rise) when heating stopped at about  $475^{\circ}\text{C}$ .

The identification of the solid phases again remains uncertain, but the presence of two soluble isotropic phases (one sometimes visible as a cube) tends to suggest halite (NaCl) and sylvite(KCl).

Sample 00932: This sample displayed a range of fluid inclusion size from small, slightly elongate or rounded, to relatively very large (70 µm), irregular and sometimes elongate ones. There was no obvious indication of their being primary or secondary. Most had a gas phase, some also with one or even two solid phases present. With regard to the gas phase, all had low homogenisation temperatures, ranging from about 82°C in several instances, up to 200°C, the majority being below  $110^{\circ}$ C. The limited evidence from the solid phases (one isotropic (?halite) and the other birefringent) provided a contrast, the isotropic phase apparently dissolving just below 300°C ( $^{\circ}$ 37 wt.% NaCl), but the birefringent solid remaining even slightly above 500°C (?calcite). Precise measurements were precluded by the tendency of inclusions to decrepitate at lower temperatures.

# 5. SUMMARY OF RESULTS AND CONCLUSIONS

# 5.1 Methodology

The method of preparing double-polished sections of grains from mineral concentrates as described has been used to help obtain microthermometric data from fluid inclusions in monazite and quartz from Mt. Painter breccias. Although thus established, the method has suffered in the present study from certain limitations as to amount and grain-size of the material available. It is concluded that, overall, more information could have been more conveniently obtained by making and using repeat double-polished sections in the normal manner until sufficient data had accrued. The critical factor would appear to be the size fraction required to liberate the mineral(s) of interest. Once below a certain size it is unpractical, or even impracticable, to produce double-polished grain mounts of suitable quality. However, the use of a different bonding material has enabled the problem of pre-decrepitation to be decided.

## 5.2 Microthermometry

With regard to monazite, microthermometric results obtained from rock sections have shown firstly that many of the fluid inclusions present have suffered a prior (natural) decrepitation. Of the rest, most look to still contain fluid, but only a few of them actually have a gas phase also visible. No direct evidence was found as to the relative ages of these various inclusions. The surviving two-phase fluid inclusions generally exhibit very low homogenisation temperatures and, with the exception of a few containing  $\rm CO_2$  and/or  $\rm H_2S$ , would appear to be of aqueous origin with low salinities. Temperature correction factors on these would be expected to be minimal, indicating their actual entrapment temperatures to be low also.

If these are primary fluid inclusions, then the decrepitated inclusions can hardly belong to a later generation, and must therefore also represent

primary inclusions having the same or even lower formation temperatures. A slight post-crystallisation temperature rise is thus indicated to explain the decrepitation of some of them. The very few with high temperatures apparent could then represent partly-decrepitated or 'stretched' equivalents.

If, however, the low-temperature inclusions are of later (secondary) origin, and the decrepitated inclusions represent primary phenomena, then it is probable that the relatively rare high-temperature two-phase inclusions are, in fact, the survivors of this primary generation. This would put the temperature of monazite formation in the region of  $300-400^{\circ}$ C, followed by periods of even more-elevated temperatures (to explain their subsequent decrepitation). As stated above, the evidence from monazite alone is insufficient to solve this problem.

With regard to the 'late idiomorphic quartz', the bulk of the evidence points to its formation at very low temperatures (possibly rarely reaching to the vicinity of  $200^{\circ}\text{C}$ ) from mostly weakly saline solutions. The more  $\text{CO}_2\text{-rich}$  fluids found in some instances are probably secondary. In addition, a small but significant percentage of multi-phase inclusions, especially in samples 00929 and 00931, has preserved evidence of a more complex fluid at higher temperatures (possibly approaching  $400^{\circ}\text{C}$ ) and containing relatively high levels of salts (chlorides), with 'sulphate and/or 'carbonate'). From the occurrence in the same crystals of inclusions apparently derived from one or other of these fluids, it is tempting to conclude that the two fluids are genetically related. If the 'late idiomorphic' quartz is post-monazite in origin (and if the grains of quartz selected from the monazite concentrate residues do, in fact, all represent 'late idiomorphic quartz'), then it is further tempting to relate the high-temperature stage of multi-phase fluid inclusions with the period when the decrepitation of the inclusions in the monazite took place.

This indicated temperature of up to almost  $400^{\circ}$ C is in reasonable agreement with the few temperatures previously recorded on monazites from the Radium Ridge Beds (Progress Report No. 2, Table 1), though almost no low-temperature inclusions could be conclusively identified in these monazites. The present evidence, though limited, is not necessarily in conflict with the general picture which emerged from previous work. Although more than one interpretation is possible in detail, it appears to be generally established that during the crystallisation (including recrystallisation) history of both monazite and quartz, fluid inclusions were formed at very low to low and also at moderate to high temperatures (under more saline conditions). A relatively pronounced postcrystallisation rise in temperature at some stage is indicated for all monazite, with a late change to low-temperature conditions with lower salinities but higher levels of CO2. What appears to be lacking is good evidence as to the relative time of the various events. Further clues might still be found in comparing data from the earliest known occurrences of monazite with data from the most recent examples, but there can be no doubt that the imprint of later events has destroyed much of what might have been valuable evidence, in the monazite at least.

# FLUID INCLUSION STUDIES OF MT PAINTER ROCK SAMPLES

PR No. 6

Department of Mines & Energy
South Australia
1/1/241-GS April 1980



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3 April 1980

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GS 1/1/241

Director-General,
Department of Mines & Energy,
EASTWOOD.

FLUID INCLUSION STUDIES OF MT PAINTER ROCK SAMPLES

PROGRESS REPORT NO. 6

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## INTRODUCTION

Four rock samples were submitted for fluid inclusion studies. These samples comprised: a hematitic breccia (RS 1088-RBM 41A/79) from No. 2 workings, Radium Ridge, for which the identification of the pink mineral was also required (see also AMDEL Report 1902/80); a barite-hematite rock from the British Empire Mine; and two barite samples from the hematitic breccia area on the southern flank of Radium Ridge.

These samples in effect represented further work of similar type to that already carried out under Project GS 1/1/182 (Progress Reports Nos. 2 and 5). Being whole rock samples (as against mineral concentrates), they were used for the preparation of double-polished thick sections in the standard manner. Owing to the limited scope of the Project, heating studies were carried out firstly on all four samples and subsequent freezing studies were restricted to the single most suitable sample, RS 1126-RBM 2A/79.

#### 2. DESCRIPTION OF SAMPLES AND RESULTS

## 2.1 Hematitic Breccia

Sample RS 1088-RBM 41A/79: Microscopic examination showed this sample to comprise mainly coarse and apparently anhedral hematite, though occasionally displaying slender, lath-like crystals. The sample also contained local areas of clear to cloudy-brown, fine, recrystallised quartz and small amounts of monazite. These darker areas of quartz were found to correspond to the "pink mineral" of the hand specimen, and as such resembled weathered feldspar in both hand specimen and thin section. The cloudy aspect arose from the presence of minute inclusions, some arranged in bands, but too small to be identified either as solid or liquid varieties.

Parts of the clearer quartz appeared as vein-like zones probably representing recrystallised areas of the cloudy-brown type, with rare single crystals resembling vuggy quartz. Such clear areas of quartz are thus considered to be of late development, and could possibly be the equivalents of the "late idiomorphic quartz" of previous reports. Though clear by contrast with the cloudy quartz, some of these crystals nevertheless were found to contain small but recognisable fluid inclusions, sometimes with two phases (liquid and gas) and probably of more than one generation. One clear, subhedral crystal displayed inclusions disposed in zones parallel to the crystal outlines, a condition implying primary inclusions formed at successive growth stages. The temperature of homogenisation (tH) here ranged upwards from about 125°C, falling off after 175°C with nearly all inclusions converted to single-phase liquids by around 250°C. However, in another similar crystal of quartz the primary(?) inclusions tended to have no gas-bubbles visible, while the secondary or pseudosecondary inclusions (those that appeared to lie in slightly curved transverse planes, or in planes marking earlier sub-grain boundaries) were usually 2-phase inclusions. In some of these possibly pseudosecondary fluid inclusions the gas-bubbles disappeared at or just below 100°C, the upper limit being 150°C for most, the highest recording being nearly 230°C. One or two of these inclusions, however, resembled decrepitated or partly decrepitated types, which they probably were, since partial decrepitation/leakage was observed to occur with heating up to 300°C.

The larger inclusions in monazite also appeared to have suffered prior decrepitation, though tiny daughter solids of reddish hematite were recognized in some. Generally only the few very small (5  $\mu$ m) equant inclusions appeared to still contain fluid, giving homogenisation temperatures (in the liquid phase) in the range 60 or 70°C up to about 182°C. One monazite crystal contained, in addition to the usual elongated decrepitated inclusions, two thin, perfectly regular, tubular inclusions lying parallel to the others (see also RS 1127-RBM 76/79). The longer of the two contained a small crystal of hematite and a small bubble, which homogenised at 117°C.

Thus, although limited in scope, the evidence from both the quartz and the monazite in this hematitic breccia points to very low to low temperatures existing at some stage of the crystallisation/recrystallisation history, presumably followed by an increase in temperature (to account for the observed decrepitation). Similar evidence has already been recorded in the previous reports mentioned.

## 2.2 Barite-Hematite Rock

Sample RS 1126-RBM 2A/79: The barite appeared in section as originally coarse anhedral crystals, but now largely fragmented. All bore small inclusions in the size range 2-20 µm, some crystals being virtually packed with such inclusions, which, nevertheless, remained well-distributed and maintained slightly irregular to subhedral negative crystal outlines. Their large numbers and even distribution establish them as primary inclusions. Virtually all appeared initially to be empty, in that they had the very heavy outlines and absence of a second phase typical of gas-filled or decrepitated inclusions. Only extremely rarely (once in hundreds or even thousands of examples) could an inclusion with a "tell-tale" bubble be found, and three different determinations gave homogenisation values of just below 70°C, ~195°C and <280°C. In another crystal, with limited occurrence of 2-phase inclusions, homogenisation commenced at about 92°C and no normal homogenisation behaviour was found at temperatures above 100°C.

However, on cooling the sample from around 280°C to room temperature, numerous previously "empty" inclusions (indeed the majority of them) were observed to have moving gas bubbles. Most of them began to homogenise by disappearance of the gas phase towards and close to 200°C, the upper limit being 230°C. The explanation for this behaviour remains unknown. It seems improbable that so many could have been inelastically stretched (thus increasing the volume and decreasing the pressure to promote the formation of a vapour or gas phase) or partially decrepitated to the same extent by heating. If the sample, when first observed, was in a highly supercooled state (i.e. cooled slowly well below the homogenisation temperature but with failure of the gas phase to form), it must have previously been heated by at least the amount required to induce the homogenisation as subsequently observed, i.e. 200-230°C. Heating during sample preparation is usually much less than this (on this occasion  $65^{\circ}$ C - for mounting - though friction during polishing is an unknown factor). A more feasible explanation rests on the increased solubility of barite (especially in the presence of chlorides) with increased temperature, which would also tend to increase the volume of the inclusion by solution around the walls. Also, of course, barite is known to be prone to "decrepitation" even with gentle heat to give off  $H_2S$ , which could further account for the expansion of the gas phase. Whatever the explanation, however, it is clear that the upper limit of homogenisation for the sample is 270°C. The general absence of gas bubbles suggests that the formation temperature may, in fact, be somewhat lower than this.

Several freezing runs were carried out on this sample. Crystals were chosen which had fewer but larger fluid inclusions, with indications of arrangement in broad bands likely to represent growth stages. As before, few of these inclusions displayed gas bubbles. In the majority of cases

where freezing was induced this occurred between -60° and -70°C. No changes were observed on warming up, until -32°C when the "ice" in a number of inclusions was observed to undergo a change of appearance in its granularity. This "reorganisation" must reflect a recrystallisation due to a phase change or the onset of melting. By -28°C in one, and slightly higher in others, true melting could be observed, the "last ice" value lying between -7.5°C and -18°C. This is equivalent to 12 to 22 wt% of NaCl. The evidence of actual melting at temperatures as low as -28°C suggests that there are other salts in solution also, such as CaCl<sub>2</sub> or possibly  $\rm H_2S$  (which is common in barite).

There is other evidence that the entrapped fluid itself may not, in fact, be a simple saline solution. During the warming up period, and usually at or above the "last melting" stage, a number of single-phase inclusions became double-phase by the appearance of a gas bubble. Some of these bubbles continued to expand towards room temperature before ultimately contracting again, but others increased in size until ultimately decrepitation was reached, even at relatively low temperatures.

## 2.3 Barite in Hematite Breccia

Sample RS 1127-RBM 76/79: The barite here occurred generally as mediumgrained, sometimes anhedral individual crystals, but more usually in groups of radiating, near-euhedral types. Many of these were tranversed by numerous cleavage and fracture planes and contained opaque inclusions (presumably decrepitated fluid inclusions) and were thus unsuitable for study. Fortunately, some of the coarser crystals were packed with welldispersed small inclusions, some "following" crystallographic directions and almost certainly being of primary origin. The majority fell into the size range of 5-10 µm, there being a few crystals with larger inclusions up to 20  $\mu m$ , and rarely 40  $\mu m$  for elongate shapes. Nearly all inclusions were partly irregular in outline yet partly bound by straight edges. Gas bubbles in the inclusions were seldom observed. The homogenisation temperatures obtained indicated a range from 104°C up to 280°C. of the larger single-phase inclusions decrepitated just below 200°C, while other such large inclusions formed gas-phases once cooled down again to around 100°C (cf RS1126).

In one group of partly-radiating barite crystals, displaying as before numerous small inclusions, there were in addition many coarse and often highly irregular individuals, a few exceeding 150 µm in length. Their irregular outlines coupled with indications of recrystallisation by "necking-down" are suggestive of secondary origin. Gas bubbles, seldom present in this group, did not behave normally, in that some expanded on heating but did not contract again on cooling, nor expand further on reheating; while one expanded on cooling to a much larger size than before. Although uncertain, the evidence is again taken to indicate that some of the inclusions had previously decrepitated, and that the heating of others has induced partial decrepitation or dissociation.

Sample RS 1128-RBM 78D/79: The barite in this sample also tended to form coarse, sub-radiating groups of crystals, but appeared to be partly replaced by hematite. In one instance where the crystals had grown in vuggy cavities the terminating faces were marked by a thin capping of hematite which itself was overgrown by clearer barite with fewer inclusions than the earlier-formed barite. Quartz, probably of late development, was found associated with the barite mostly as idiomorphic single crystals but occasionally also in groups with triple-point junctions.

The evidence of fluid inclusions in the barite tended to be overshadowed

by the presence of hematite, which ranged down to fine specks resembling other inclusions in the barite, while some of the quartz was riddled with dark-looking ?fluid inclusions of highly irregular outlines, ranging from a network-like distribution to fan-shaped arrangements. Indeed, the abundance of such inclusions in these two minerals at times made it difficult to identify one from the other.

Some of these inclusions in the quartz were recognized as decrepitated types, and these may thus be counterparts in the barite. Some barite crystals were inclusion-free, others had regular, well-spaced fairly large and slightly elongate single-phase fluid inclusions, so that information was severely limited, and a second polished section was required to gain any useful information from this sample.

The barite in this second section generally contained numerous fluid inclusions. In summary, the bulk of these appeared to relate to growth bands, leaving little doubt that they were primary, though some areas had "flatter", partly interconnected inclusions, more probably derived from the annealing of fracture planes in the barite (i.e. secondary inclusions). Locally, the "interconnected" inclusions took on a more regular aspect and in extreme cases became distinctly tube-like (cf sample RS 1008-RBM 41A/79). This would imply that regular tube-shaped inclusions are of secondary rather than primary origin.

The information on homogenisation from both primary and secondary inclusions nevertheless appeared to be the same, as was that from the inclusions in quartz, though more limited.

Gas bubbles were relatively rare, on initial inspection, and the behaviour of many inclusions on heating and cooling down again was not "normal", in that the majority of single-phase inclusions formed a gas bubble once the temperature fell back below about 100°C. Homogenisation of those initially having gas bubbles commenced as low as 60°C and generally was low, but effectively covered the range up to about 280°C. Some of these in the upper part of this range (>175°C) were cooled and reheated a number of times always with the same results, indicating from their stability that this reflects the true range (subject to pressure corrections) of crystallisation. Those in the lower part of the range did not become two-phase again on cooling, possibly because they were not supercooled enough.

Homogenisation of those inclusions *eventually* forming a gas bubble on cooling after the first heating run tended to be complete by about 205°C. Again, however, the range extended to nearly 280°C, and some individuals which were heated and cooled several times showed a tendency for tH to rise slightly each time.

The information from the secondary inclusions and from those in the quartz was, as already indicated, similar. It would thus appear that many, if not most, of the fluid inclusions in this sample are characterised by slightly anomalous homogenisation behaviour, but this an upper limit for homogenisation of about 280°C.

## 3. SUMMARY AND CONCLUSIONS

The four samples studied are all from the same general area of hematite rocks and hematitic breccias, three of them containing barite, usually with quartz, and one also with monazite (RS 1088-RBM 41A/79).

Initially, direct evidence on the temperature of crystallisation of these minerals was highly limited. For instance, in one sample (RS 1126-RBM2A/79), although the barite was riddled with fluid inclusions, only a fraction of one percent of them had an observable gas bubble. After heating and recooling, however, single-phase inclusions in the samples generally displayed a second (gas) phase while some two-phase inclusions had enlarged gas bubbles. Although this phenomenon appeared to push up many individual readings of homogenisation temperature, the overall range is still at the low end of the spectrum. The precise reason for the unusual behaviour of so many inclusions with heating/cooling in uncertain but seems likely to be due either to solution of the inclusion walls or dissociation of the liquid phase, both of which are quite possible with barite, though with quartz solution is much less likely.

The lowermost values of tH lie around 60°C and the upper limit seems to be, or to be very close to, 280°C in all cases. Undoubtedly the great majority of inclusions have homogenisation temperatures between these values. In view of the tendency of re-measured values to rise and in view of the fact that inclusions with very low values of tH may not form a gas phase at all (and thus tend to be overlooked), it is considered that the "average" homogenisation temperature will lie towards the lower end of the range quoted.

The actual temperature of formation, of course, depends upon the pressure correction to be applied to the measured value of homogenisation (to account for the effects on the caused by the pressure of the system operating at the time of crystallisation). The pressure can be calculated from the depth of burial during crystallisation, if known, or from the density of the fluid. In the latter case the composition and volume of gas to liquid phases must be known fairly accurately. In the present circumstances none of these factors can be specified, though the evidence of usually relatively small gas bubbles, low to moderate equivalent salt-content of the liquid phase and the tendency to decrepitate at low temperatures, points to small temperature corrections.

Although the evidence from monazite has been extremely limited it has supported the findings of the immediately prior study (GS 1/1/182, Progress Report 5, dated 24 July 1979) that some of the (presumably fluid) inclusions have suffered an earlier decrepitation and that those few remaining tend to have low homogenisation temperatures (in that case below 200°C)\*. The evidence in the present study from the barite and quartz (though probably of relatively late development) would appear to support formation at very low to low temperatures (see also Progress Report No. 5 on "late idiomorphic quartz"). Finally, there is some evidence that hydrogen sulphide may be present in the host fluid, though this would require further investigation to substantiate.

<sup>\*</sup> It should be noted, however, that in an even earlier study (GS 1/1/182, Progress Report 2, dated 21 September 1977) much higher temperatures were noted for some monazites - possibly indicating an earlier generation of monazite or at least of fluid inclusions.

# **DEPARTMENT OF MINES AND ENERGY - SOUTH AUSTRALIA**

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# APPLICATION FOR EXAMINATION OF SPECIMENS OR SAMPLES

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# FLUID INCLUSION PROCEDURES FOR EXPLORATION

A LITERATURE SURVEY

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# FLUID INCLUSION PROCEDURES FOR EXPLORATION

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JUNE 1980

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	Page		
INTRODUCTION	1		
WHAT ARE FLUID INCLUSIONS	2		
MEASUREMENT OF FLUID INCLUSION PARAMATERS	4		
BRIEF HISTORY	6		
FLUID INCLUSIONS IN EXPLORATION	7		
EXPLORATION EXAMPLES	9		
Porphyry Copper Deposits	9		
Massive Sulphide Deposits	10		
Sulphide Veins and Pipes	10		
Gold Deposits	12		
Tin-Tungsten Deposits	13		
Replacement/Skarn Deposits	15		
Hydrothermal/Pneumatolitic Deposits	17		
ADVANTAGES OF FLUID INCLUSION METHODS	18		
LIMITATIONS OF THE METHODS	20		
PRACTICAL EXPLORATION PROCEDURES	21		
CONCLUSIONS	23		
GLOSSARY	25		
BIBLIOGRAPHY			

#### INTRODUCTION

The purpose of this study is to review the extensive literature on Fluid Inclusions from the viewpoint of their use as an aid to established techniques in the exploration for ore deposits. As such it is not intended to be a complete review of the literature, much of which deals strictly with research or ore genesis and no apology is made for any resulting bias towards methods which are considered or known to be directly relevant to exploration.

In view of the vast amount of information reported on fluid inclusions, and the importance of this data in studies of ore genesis, the lack of application of fluid inclusions to exploration is enigmatic. This is possibly due to the expense of collecting fluid inclusion data by the slow techniques commonly used in research institutions. Consequently this study focuses on the possibility of using rapid, automated techniques with a view to their use in a manner analogous to geochemical rock and soil surveys. Some loss of accuracy is inherent in such procedures but there is reasonable justification from the existing literature to expect the resulting information to be of substantial value to exploration if not to ore genesis investigations.

In conjunction with this literature study an automated, computer controlled apparatus has been constructed to obtain decrepigrams. Orientation surveys are now being carried out to assess the use of these decrepigrams in exploration for various types of ore deposits.

This project is being undertaken by Burlinson Geochemical Services, Darwin.

## WHAT ARE FLUID INCLUSIONS

Fluid inclusions are small volumes of fluid which are trapped within crystals by any of several mechanisms either during their growth or by later processes. They are generally representative of the fluids present during crystal growth or in which it was later bathed. They occur commonly in most minerals, are rarely greater than 1 mm and generally less than 10 microns in size. Small ones can be observed in ordinary petrological thin sections but in general doubly polished thick sections are preferred. The ordinary petrological microscope used at magnifications of X200 to X1000 is quite suitable for observing them.

Their abundance varies, but may be as many as a billion per cubic centimetre. Those trapped during host crystal growth are referred to as primary inclusions and contain a sample of the formation fluid. Later shearing and rehealing of fractures will cause trapping of large numbers of secondary inclusions containing samples of the fluids present at such time.

When trapped the ore forming fluids are most commonly (but not always) a single phase. During cooling to present temperatures these fluids may separate into several phases depending on their compositions. They may now contain any combination of solid phase (referred to as daughter crystals), liquid phase and gas (often as a bubble).

Materials which commonly occur in inclusions are water, water vapour, halite and carbon dioxide (either as a liquid or gas phase). An enormous variety of less common materials have been recorded, and their identification is frequently difficult due to the small amounts involved.

From the investigation of these fluid inclusions it is possible to deduce much about the environment in which the crystal grew. Such information includes:-

- 1) The temperature of formation;
- 2) The salinity of the formation fluids;
- 3) Presence of such constituents as carbondioxide, hydrocarbons, hydrogen sulphide;

4) Occurrence of boiling during formation; .... and much more.

This data is very commonly used in developing models for ore genesis and to a lesser extent has been used to define targets for exploration programmes.

# MEASUREMENT OF FLUID INCLUSION PARAMATERS

The most commonly made measurements on fluid inclusions are the temperatures at which various phase changes occur. These measurements are made by observing a single inclusion (or a few inclusions within the one field of view) under a microscope while either heating or cooling the sample. The temperatures of various phase changes within the inclusion can be accurately measured, generally within a degree or two. Temperatures of interest are:-

- 1) Homogenisation temperature  $(T_H)$  the temperature at which the several phases in the inclusion become a single homogenous phase when the sample is heated;
- 2) The temperature of freezing of the aqueous phase can provide information on the salinity and occasionally an indication of the salt composition (K, Ca, Na). The presence of CO<sub>2</sub> can be determined in some cases as it can form clathrates, which are recognizable during cooling;
- 3) Filling temperature. In some cases when an inclusion containing a daughter crystal (S) is heated, the liquid may expand to fill the inclusion without totally dissolving the daughter crystal. As 2 phases (solid and liquid) remain, this is not the same as the homogenisation temperature, and is referred to as the temperature of filling.

Less commonly made measurements include:-

- l) Heat the sample until expansion of the fluids in the inclusion causes the mineral to physically burst. This is referred to as the temperature of decrepitation  $(T_D)$ . A microscope is not necessary for this technique;
- Opening the inclusion within a pool of oil by using a sharp point while observing it under the microscope. This gives an estimate of internal pressure by estimating the expansion of the gas phase;
- 3) Various chemical, microprobe or mass spectrometer analyses. These usually require opened inclusions and are without exception long, involved and demanding processes which are beyond the scope of this study.

Data from these measurements is used to deduce the chemical composition, nature and temperature of the fluid during crystal formation. The temperature of formation  $(T_F)$  is greater than or equal to  $T_H$  depending on corrections necessary to allow for the fluid pressure and salinity. Note that the pressure is not usually discernable from fluid inclusion measurements alone and is estimated from geological considerations.

Most of these measurements require a skilled operator and take a considerable amount of time as the sample must be heated or cooled slowly (usually less than 5°C per minute). As only a very few inclusions can be measured simultaneously many days of work may be involved in obtaining a statistically meaningful result for a single sample.

In contrast, measurement of the decrepitation temperature  $(T_{\rm D})$  can be made quite quickly and is amenable to automation. It is for this reason that this study concentrates on the measurement of  $T_{\rm D}$ , despite some limitations which are outlined later.

#### BRIEF HISTORY

Sorby first proposed the use of fluid inclusions to estimate crystal formation temperatures in 1858. There was little interest in inclusions until the late 1940's when Smith, Little and Peach became involved with measurements of  $T_D$ . Their apparatus is best described by Peach (1949). Some controversy arose between the schools of thought favouring  $T_H$  and  $T_D$  and by about 1955 the homogenisation method had superseded the decrepitation method in the western world because of the inherent limitations of the decrepitation method.

The Russians however, took to using  $T_{\rm D}$  measurements and used this technique extensively in mineral exploration programmes. Their use of the  $T_{\rm D}$  method has continued to the present day, although, perhaps declining slightly in favour of  $T_{\rm H}$  measurements in recent years.

In the western world there has been no significant use of  $T_{\rm D}$  measurements by the exploration community, and the academic community has preferred the more precise  $T_{\rm H}$  method.

While several decrepitation instruments are manufactured in Russia there are no commercial instruments available in the western world and only a few individually constructed instruments seem to be in existence.

Although the major use of fluid inclusion measurements in the western world is in research on ore genesis, several studies have looked at fluid inclusions from an exploration viewpoint. (Nash 1972, 1975, 1976; Bradshaw, 1968; Roedder 1977). These studies have used the  $T_{\rm H}$  method and have shown some potential for the further use of fluid inclusion data in mineral exploration programmes.

## FLUID INCLUSIONS IN EXPLORATION

Many models of ore formation involve circulating fluids driven by a heat source. Such models range from the generation of quartz vein systems around intrusives to large scale convection cells either responsible for or associated with porphyry copper deposits. Samples of such fluids will be trapped in both the ore and gangue minerals during formation and the study of these trapped fluids can assist in determining the conditions of ore formation. However, in contrast to the various ore minerals which may be concentrated at only a few points in the fluid system, fluid inclusions will be more widely dispersed throughout the fluid system. The resulting pattern of fluid inclusions can be likened to a primary geochemical dispersion halo of trace elements. However, this fluid inclusion halo has several important advantages over conventional primary geochemical haloes, such as:-

- It is generally much more extensive as inclusions can still form after the fluid composition or temperature is no longer favourable for the transport of the ore elements or their associated indicator elements;
- 2) The fluid inclusion halo is little (if at all) affected by subsequent weathering processes;
- 3) In addition to the frequency of occurrence of fluid inclusions other paramaters such as salinity,  $T_{\rm H}$  or  $T_{\rm D}$  can indicate the direction towards the fluid system centre;
- 4) Fluid inclusion formation is relatively independent of pH, EH, pO<sub>2</sub> etc and therefore less subject to complications which can adversely affect trace metal transport.

The use of fluid inclusion haloes in exploration would therefore form a useful adjunct to conventional geochemical, geological and geophysical procedures.

Because the formation of a fluid inclusion halo presupposes little more than the existence of heated fluids a great variety of ore types should be amenable to exploration by fluid inclusion techniques. Such ore types include:-

- Late stage magmatic deposits;
- 2) Hydrothermal/Pneumatolitic deposits;
- 3) High level acid intrusive associations:
- 4) Deposits formed by country rock scavenging in convection cell systems;
- 5) Possibly even deposits formed due to dewatering during diagenesis or metamorphic dewatering;
- 6) Replacements and skarns (?)

This variety of ore types includes ores of a great many elements including Au, Ag, Bi, Sn, W, Mo, Cu, Pb, Zn, U etc as well as such commodities as fluorite.

In view of the wide variety of ore types and ore metals to which exploration by fluid inclusion techniques could be applied, the almost total lack of use of fluid inclusions in exploration (in the western world) is enigmatic!

## EXPLORATION EXAMPLES

Although there is a great deal of fluid inclusion data in the literature, most of this is concerned primarily with ore genesis rather than exploration. Most of the exploration examples are in the Russian literature and the data is sometimes less than complete. Nash, at the U.S. Geological Survey, has however carried out much work which is directly relevant to the use of fluid inclusion data in exploration.

# Porphyry Copper Deposits

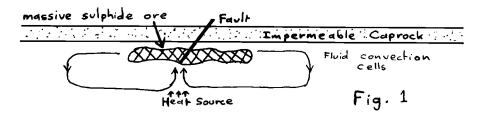
Extensive zoned haloes of fluid inclusions have been observed around many porphyry copper deposits by Nash. (Nash, 1976.)

He found the most useful measurement to be the salinities of the inclusions, and in most cases the salinity decreased away from the ore zone. The size of the anomalous salinity area was generally somewhat smaller than the pyritic alteration zone. He concludes that the fluid inclusion halo is a more satisfactory target than either minor element or alteration haloes as these latter are poorly developed in siliceous rocks. Nash has found evidence of vertical zonation of fluid inclusions in some but not all of the orebodies he studied and a change from boiling to non-boiling fluids seems to occur at the base of the system. Base and precious metal ores tend to occur at these lower levels. Similar effects were noted at Bingham by Roedder, 1971.

Although the porphyry copper deposits typically form at temperatures of  $400^{\pm}$   $100^{\circ}$ C, zonation of fluid inclusion filling temperatures around the orebodies is often subject to complications such as high temperature pulses and late stage activity and temperature zonation alone is a less reliable target than salinity zonation. Some studies have drawn attention to the zonation of the abundance of fluid inclusions (Nash, 1976; Chivas and Wilkins 1977). Simple counting techniques could be used to determine such zonation rather than the slow and involved heating/cooling stage procedures. Decrepitation measurements can also provide this information even more rapidly.

# Massive Sulphides

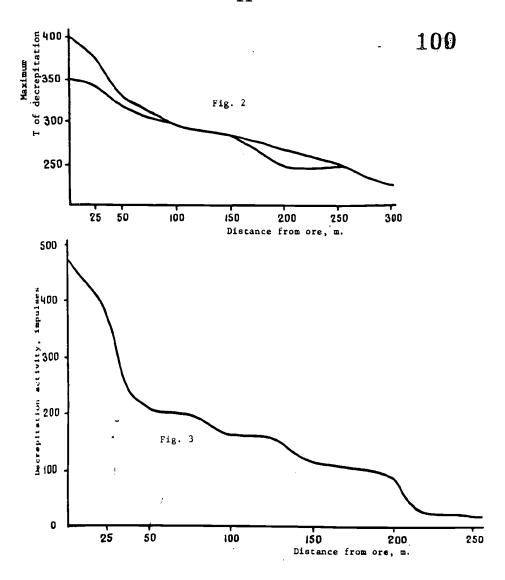
Franklin, at the 1980 Archaean Symposium, outlined a model for massive sulphide deposits of the Timmins type in which large convection cells driven by a deep heat source are involved. He considers this model to be relevant to a number of Canadian massive sulphides.

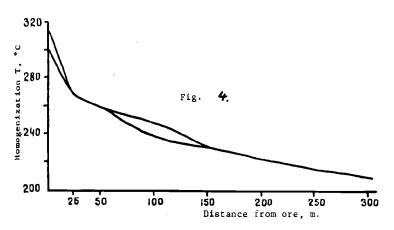


This model is similar to the convection cells set up around porphyry copper stocks and by analogy fluid inclusions should be a useful exploration tool, although no such studies seem to have been done to date.

# Sulphide Veins and Pipes

Vein and pipe-type base metal deposits have been well studied from an exploration viewpoint, particularly by the Russians. (Ermakov 1966; Ermakov and Gromov 1971; Ermakov and Kuznetsov 1972; Nash 1975). The Russians frequently report decrepigrams showing good contrast anomalies near such mineralisations. (Figs. 2,3,4,5). The extent of these anomalies seems to be quite variable and would seem to be very dependent on the sampling medium (rarely specified in the Russian papers). Nash's work at the Argentine vein, Colorado, found no systematic variation in filling temperatures which could be related to mineral zoning in an extensive vertical and lateral region of mineralised veins around the Silverton caldera. Unfortunately his sampling pattern is very irregular due to mine inaccessibility and the study stopped short of comparing barren and mineralised areas. Post sulphide stage veins were shown to have distinctly different filling temperatures  $(153^{\circ} - 188^{\circ}C)$  to the ore veins  $(220^{\circ} - 315^{\circ}C)$ and so there may be some value in using fluid inclusions to discriminate between the various vein systems. Bradshaw, 1968, studied the filling temperatures of vein materials in south-west England and suggested that filling temperatures can be used to determine the relationship between a particular





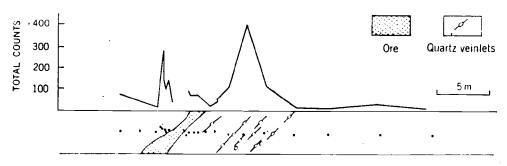


Fig. 5. Graph of decrepitometric activity of limestone from a drift in the Brichmull deposit, Uzbekistan (Polykovskii, 1971, p. 143).

vein and the economic zonal sequence. Nash's own work on the lower zones of porphyry copper deposits would confirm such zoning patterns although some reservations would be in order in south-west England where telescoping of the deposits is common.

# Gold Deposits

Exploration for gold is subject to several problems not encountered in base metal work and fluid inclusions may assist in dealing with these problems. The problem of sampling error is particularly acute for gold which is subject to the oft quoted "nugget effect" resulting in a random spotty distribution. Combined with very low economic cutoff grades for gold, expensive and involved sampling procedures are necessary to ensure reasonable accuracy. The dearth of useful geochemical pathfinder elements further complicates exploration procedures. If fluid inclusions could be used, these problems would be considerably eased.

Homogenisation temperatures for a number of gold deposits in Nevada (Nash 1972) do not show any regionally constant value. However, on a local scale the temperatures in any one deposit tend to be restricted to a range of about  $30^{\circ}\mathrm{C}$ . Salinity measurements showed that all the deposits formed at similar low salinities, and Nash concludes that fluid density measurements may have application in exploration on such a regional scale. Although he does not recommend exploration based on  $T_{\mathrm{H}}$  measurements he is referring to regional exploration and his data are consistent with the possibility of using  $T_{\mathrm{H}}$  on a local scale.

A limited amount of work has been carried out by the S.A. Department of Mines and Energy on gold areas in South Australia. Again this has shown that  $T_H$  measurements are not useful on a regional scale but the study has not tested their use on the local scale or as a discriminating paramater.

Some very rudimentary work I have already done in the Pine Creek area, Northern Territory indicates the potential of  $T_D$  measurements in discriminating between various stages of quartz veining on a local scale and more detailed studies are warrented.

Because fluid inclusions are quite common in quartz veins, using them should overcome the problems of sampling error for gold. Once familiarity is gained in a particular locality it may also be possible to use fluid inclusion temperature and salinity data to deduce a direction towards any auriferous zones within the veins.

## Tin - Tungsten

Several investigations on tin-bearing granitoids and vein systems have been carried out by the Russians. Unfortunately the reported results tend to be less than complete. Bradshaw, 1968 has looked at some of the quartz vein systems in southwest England and found a zoning of  $T_H$  measurements related to the mineralisation zoning of Sn, Pb, Zn, Cu. He also shows a temperature zoning around granitoids (Fig. 6), albeit from a fairly limited number of sample points.

Existing studies of base metal vein deposits should also be relevant for vein-type tin deposits. As cassiterite forms over a wide temperature range (200° - 350°C) fluid inclusion results are not likely to be of great use regionally, but could be used as an empirical method locally to pinpoint ore concentrations. Most vein-type tin systems have a confusing array of quartz veins, both barren and mineralised, and fluid inclusions may be of use as a discriminant for the various vein types.

It has also been suggested (Bradshaw, 1968) that by observing the zoning of quartz vein temperatures the source granitoid for the mineralization could be pinpointed. In many tin areas there are a great number of granitoid outcrops and often a great

number of different granitoid phases occur (eg some 20 odd in the Blue Tier Batholith, N.E. Tasmania). Pinpointing the source granitoid is usually not a trivial matter. This same technique may be of use as a guide in exploration for non-outcropping granitoids or cusps. Such unexposed areas are of interest in the search for high volume low grade greisen deposits.

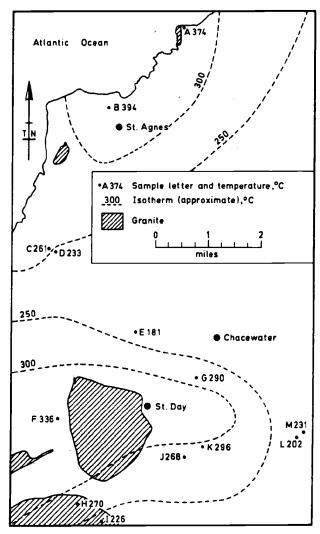


Fig. 6 Upper limit of filling temperatures recorded from individual deposits in St. Agnes—St. Day area

Little has been reported on exploration for quartz vein-type tungsten deposits, but they are so closely associated with tin that the same procedures should apply.

Although little data is available in the literature about tin/tungsten replacement or skarn deposits by analogy with other replacement and skarn deposits fluid inclusion data should be of use in exploration.

The problems of sampling for tin and tungsten are similar to those of sampling for gold and similarly there are few reliable geochemical pathfinder elements. Fluid inclusion methods may be of use in overcoming these limitations.

# Replacement/Skarn Deposits

Much of the work which has been done on these deposits is concerned with ore genesis rather than exploration and it is difficult to deduce how useful fluid inclusion measurements might be for exploration.

Nash, 1975, presents much data on the replacement Cu-Pb-Zn mineralisation at Idarado, Colorado, and shows the relationship between the replacement ore and the nearby crosscutting veins. However, no data is available from which to deduce any possible zoning.

Tan Teong Hing and Kwak 1979, have shown a  $T_F$  zoning in garnets at the King Island tungsten skarn (Fig. 7). This shows a consistent temperature drop along a section extending 500 m from the igneous contact. This diagram also shows the complications due to zoning of the individual crystals. Other skarn deposits have been studied by the Russians and Fig. 8 shows a decrepitometric traverse across a small skarn (Ermakov and Gromov, 1971; diagram from Roedder, 1977). This type of data presentation is common in the Russian literature.

Although previous studies of these deposit types from an exploration viewpoint are rare, there is justification to think that fluid inclusion data could aid in their exploration. More investigations seem to be warranted.

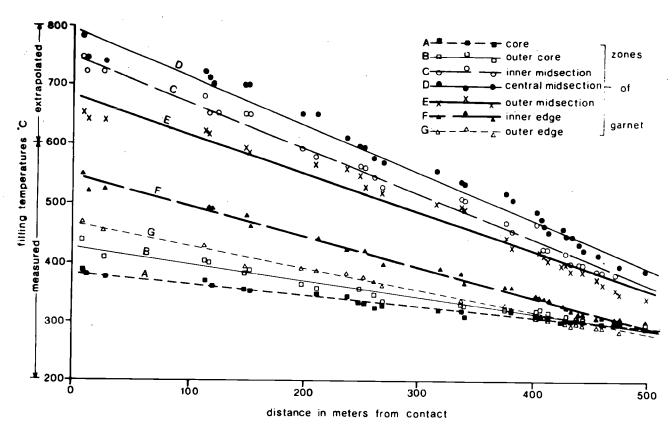


Fig. 7.—Measured temperature vs. distance curves from different parts of the garnets displayed in figure 2

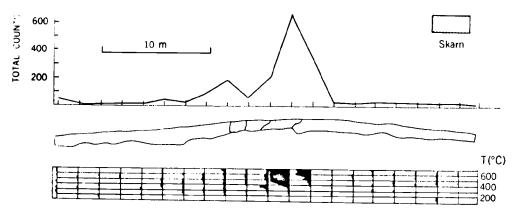


Fig. 8. Decrepitometric activity adjacent to garnet-pyroxene skarn in granite and tuffs in the Slushoky area, Central Kazakhstan (Ermakov and Gromov, 1971, p. 136). The individual decrepigraphs for samples every 2 m are given below the section, and the integrated (total) decrepitometric activity is shown by the graph above the section.

# Hydrothermal/Pneumatolitic Deposits

The close relationship between these deposits and fluid processes should make them good candidates for exploration by fluid inclusion techniques. The Russian literature contains numerous examples of studies of these deposits, but all too often the data is incomplete.

Fig. 9 shows the change in decrepitation activity (directly related to abundance of inclusions) away from an unspecified ore vein. This type of measurement can be quite simply made in comparison to  $T_{\rm H}$  or salinity measurements and is frequently used in exploration programmes in Russia.

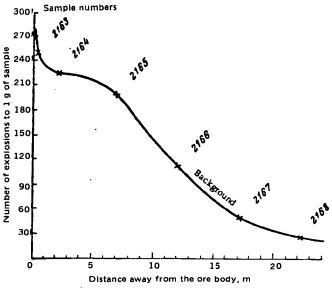


FIGURE 9. Decrepitation activity curve for sands tones, going away from the vertical hydrothermal vein

## ADVANTAGES OF FLUID INCLUSION METHODS

The use of fluid inclusion techniques in exploration would lead to a number of advantages over conventional geochemical techniques.

Foremost among these advantages is the persistence of inclusions in the resistent gangue minerals despite intense weathering. In fact many studies have been done on detrital grains and even on laterites, using fluid inclusions in quartz grains to determine the source of the host grains. (Roedder 1977, Ermakov 1966.) Few other exploration techniques can claim such immunity to weathering processes. Given the intense and deep weathering in many areas of Australia this advantage is of major significance.

The ability to overcome some of the sampling problems in exploration for Sn and Au have already been mentioned. While assay samples for these metals commonly need to be very carefully collected and as large as 10 - 30 kg to assure reasonable accuracy, samples of a few tens of grams are sufficient for fluid inclusion studies. The reason is the great and relatively homogenous abundance of inclusions in the quartz sampling medium. As sample collection and preparation for gold (and often tin) are much more expensive than the actual analysis, the savings in sampling costs could be substantial.

Geochemical techniques in exploration for gold and tin in particular are hampered by the lack of reliable pathfinder elements. Fluid inclusion measurements could help fill this void. Measurements of  $T_H$  or  $T_D$  are potentially of use on a local scale as an empirical exploration procedure in the same sense that geochemical and many geophysical techniques are now used.

Further potential for the use of fluid inclusion procedures is as a method of discriminating between potentially ore-bearing and ore barren veins or areas. Because the fluid inclusion characteristics are more intimately related to the actual ore forming processes than is the presence of associated elements (pathfinder elements) these paramaters should be

more reliable discriminators than the concentrations of associated elements.

The problem of determining the direction towards the ore centre frequently occurs in exploration. Measurements of  $T_H$ ,  $T_D$  or salinities provide a means of determining the palaeothermal gradients and hence of providing a direction towards the source of the original ore fluids. While this can be done using geochemical pathfinder techniques the fluid inclusion measurements are a much more direct method as they do not rely on assumptions about the relative mobilities of the various elements under the conditions prevailing during ore formation.

The target size is of major importance in any exploration procedures. Fluid inclusion haloes have the potential to be as large as the fluid system from which the ore formed. is most likely larger than either the primary geochemical dispersion halo of trace elements or the electrical/magnetic target of an orebody. Supergene processes may then serve to enlarge the size of the geochemical halo, however, this is at the expense of anomaly contrast. The fluid inclusion halo is therefore likely to be a relatively large, high contrast anomaly in comparison to other targets. While studies of porphyry copper deposits confirm this conclusion, many of the fluid inclusion anomalies reported in the Russian literature seem to be rather small (as are the "orebodies" on many occasions) and further work is necessary to determine whether a fluid inclusion target would be significantly larger than other targets.

# LIMITATIONS OF THE METHODS

Despite the great number of proven and potential applications outlined in the previous pages fluid inclusions are not the universal panacea to exploration problems.

The most important problem is that of the cost of many fluid inclusion procedures. Measurements of  $T_{\rm H}$ , salinity or fluid compositions require relatively uncommon apparatus, a skilled petrologist, a dcubly polished rock slice and considerable time and patience. Some measurements also require a supply of liquid nitrogen. Considerable operator skill is required in order to decide between the primary and secondary inclusions in a sample. The sample is then slowly heated and/or cooled while observing the inclusions. Because only a very few inclusions can be measured simultaneously the acquisition of a statistically meaningful amount of data can easily take weeks. These factors probably account for the lack of orientation surveys relevant to exploration.

Subsequent metamorphic events generally cause decrepitation of the inclusions and in effect erase the pre-existing inclusion patterns. While this has been used to advantage to determine the age relations of cross-cutting dykes, it is more likely to be a nuisance during exploration usage.

The applicability of fluid inclusion techniques to exploration will also depend on the type of deposit concerned. They are unlikely to be of much use for magmatic ores, stratiform sedimentary ores or other ores which are not associated with hot fluid systems. Such ores as the Mississippi valley type and submarine exhalative deposits are unlikely candidates but orientation surveys need to be done on these deposit types. The limitation of their use to only certain deposit types is a relatively minor limitation as all other exploration techniques are similarly restricted in the types of deposit to which they are relevant.

#### PRACTICAL EXPLORATION PROCEDURES

While many fluid inclusion measurements are difficult and costly to make, the measurement of decrepitation temperature can be more readily made. In this procedure the sample can be more rapidly heated, the measurements can be made by unskilled personnel, simultaneous measurements can be made on a statistically meaningful number of inclusions and the procedure is amenable to automation.

In Russia several decrepitometers are manufactured some of which are field operable. Using these instruments the Russians are able to produce decrepigrams on up to 12 samples simultaneously in the field and up to 90 samples per day. (Ermakov, 1966.)

A further advantage of this technique is that opaque minerals are just as easily studied as the transparent minerals. No other satisfactory method of investigating opaques is available. Conventional microscopic methods cannot deal with very small inclusions because it is too difficult to observe the phase changes within them during heating/cooling. Such small inclusions are less of a problem to the decrepitation method. Friable samples or detrital grains require more involved sample preparation for the microscopic methods while these materials present no problem at all to the decrepitation method.

These significant advantages are not, however, obtained without cost and considerable information is sacrificed to attain them.

While the homogenisation temperature is subject only to corrections due to the pressure at the time of formation, the decrepitation temperature is affected by many additional factors:-

- Degree of fill of the inclusions varying degrees of fill will result in different expansion behaviour of the fluids leading to variations in decrepitation measurements;
- 2) Mineral strength Different host minerals will require different overpressures before the inclusions will actually burst;

- 3) Salinity Varying salinities can result in different expansion behaviour of the fluids, hence influencing  $T_D$ ;
- 4) Partial pressure of CO<sub>2</sub> Gas rich inclusions may build up pressure only slowly and decrepitation will be delayed and may not even occur at all;
- 5) Homogenisation in the vapour phase This effect is similar to (4);
- 6) Secondary inclusions In microscopic techniques these are identified and avoided. They cannot be avoided in the TD method and the effect they cause on the resulting decrepigram can vary from beneficial to disastrous. Where the secondary inclusions decrepitate at a significantly different temperature to the primary inclusions they can actually provide useful additional data. In the absence of such a differential however, they may completely mask the primary inclusions. As secondary inclusions are most often lower temperature, and liquid filled there is some hope that the temperature differential between them and the primary inclusions will generally be sufficient to avoid swamping the primary inclusions.

A further consequence of using the decrepitation method is that no salinity data can be obtained.

Despite these limitations there is a considerable body of decrepitation data (almost all in the Russian literature) which attests to the usefulness of this technique in exploration. The resulting data must be interpreted empirically, in contrast to  $T_H$  data, but such practices are not new to exploration personnel, particularly geochemists and geophysicists.

By combining surveys using these rapid methods with a limited number of control measurements made by microscopic methods an effective and economic exploration technique could result.

## CONCLUSIONS

Fluid inclusions have long provided valuable information on the genesis of ore deposits. This same information has been shown by a great number of studies to be of practical importance to exploration, not only by improving our geological understanding and modelling, but also as a means of pinpointing ore environments and deposits.

While the commonly measured paramaters of fluid inclusions require considerable skill and time for their determination and interpretation, some of the more readily measured paramaters such as decrepitation temperature, decrepitation activity and abundance of inclusions can be used as a refinement of existing exploration techniques. Exploration procedures based on these methods have been shown to be both useful and practical and are in use in Russia today.

The simplifications involved in these exploration procedures can be compared to those made in the adoption of geochemical surveys by Atomic Absorption analyses of patterns of samples. To obtain rapid and cheap analyses the approximations of incomplete sample digestions, restricted measurement accuracy, tolerance of some inter-element interference and empirical interpretation procedures have been accepted. The resulting techniques, despite the approximations, have often played a significant role in exploration.

The absence of commercial instrumentation to make these rapid fluid inclusion measurements has mitigated against the adoption of these techniques in exploration. It has also resulted in a lack of well-controlled orientation surveys, many of the Russian surveys being only poorly described in the literature.

It is considered that fluid inclusion techniques do have an important role to play in exploration. They are already playing an important role in developing more accurate geological models. Simplifications of these high precision techniques could also directly aid in exploration procedures. The

decrepitation temperature/activity method has potential as a rapid and cheap technique which can provide an exploration tool which is uniquely immune to disturbance by weathering processes.

A microprocessor controlled instrument to obtain this decrepitometric data has already been constructed. A programme of orientation surveys is now underway to further test the applicability of this data to exploration and to develop useful exploration procedures.

#### GLOSSARY

Decrepigram:

A histogram of the number of decrepitations versus temperature. Most often used by the Russians.

Decrepitation:

The bursting of a fluid inclusion due to fluid expansion generating a pressure sufficient to fracture the host grain.

Decrepitation Activity:

A count of the total number of inclusions which decrepitate when the sample is heated through a certain temperature interval. Equivalent to cumulatively summing the decrepigram.

Decrepitation Temperature: Variably defined as either the temperature at which the greatest number of inclusions burst or the temperature of onset of massive bursting. The latter definition is preferred for comparions with homogenisation temperature, and is taken as the point of inflection preceding the main decrepitation peak of the decrepigram.

Decrepitometer:

An instrument to produce decrepigrams by counting the frequency of bursting while the sample is heated.

Formation Temperature :

The temperature at which the mineral is considered to have crystallized. Generally deduced from the Homogenisation temperature by making the necessary pressure corrections.

<u>Freezing</u> Stage :

A microscope attachment which allows the sample to be cooled while being observed. The cooling rate needs to be well-controlled to allow accurate determination of the temperatures at which the phases of the fluid inclusions freeze. Generally operate using liquid nitrogen and require an ancillary regulator and temperature measurement system. Usually have a heating system incorporated for both heating and cooling measurements.

<u>Heating Stage</u>:

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A microscope attachment which allows the sample to be heated while being observed. Require an external power supply and a temperature measurement and control system. May or may not be combined with a freezing stage. Generally limited to maximum temperatures around 500°C.

Homogenisation
 Temperature :

The temperature at which the phases in the inclusion become a single homogenous fluid. Generally considered to be an accurate estimator upon which to base determination of the Formation Temperature.

Primary inclusion:

A fluid inclusion trapped during the original crystallisation of the host grain. These are generally recognised by their close relationships to lines showing crystal growth.

Secondary inclusion:

Any inclusion trapped subsequent to original crystallisation. These may arise during metamorphic recrystallization or shearing after formation. They are generally recognised by their relationships to fractures, grain boundaries etc. Frequently they are liquid filled, low temperature inclusions. Although they may supply useful information about post mineral crystallisation processes they are usually only of nuisance value.

 $\frac{\mathrm{T}_{\mathrm{D}}}{}$ 

Decrepitation Temperature - see above.

 $T_{\mathbf{F}}$  :

Formation Temperature - see above.

T<sub>H</sub>:

Homognisation Temperature - see above.

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