STABLE AND METASTABLE FLUID INCLUSION DATA, BROWNS CANYON FLUORSPAR DISTRICT, CHAFFEE COUNTRY, COLORADO, AND SIMILAR EPITHERMAL AND HOT-SPRING (?) DEPOSITS

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INTRODUCTION

Fluid inclusions in fluorite were examined from a series of apparently shallow Tertiary deposits in Mexico and the western U.S., particularly Browns Canyon, Chaffee County, Colorado. Many of these deposits are too fine-grained to provide usable inclusions, but some are suitable. Although the inclusions are difficult to work with, due to their small size and several types of metastability, they have provided useful data on the environment of fluorite deposition. The data obtained from Browns Canyon and apparently similar deposits indicate a hot-spring-type environment, with circulating heated ground waters, rather than the more saline fluids that formed many epithermal ore deposits, and the highly saline brines responsible for the deposition of fluorite in several other types of ore deposits (Roeder, 1972).

GEOLOGY OF THE BROWNS CANYON DISTRICT

Vann Alstine (1969) described the mineralogy and geology of the district and the fluorite deposits, and presented very few of the author's inclusion data. The fluorite occurs as veins along steep normal faults in Tertiary volcanic rocks and Precambrian igneous and metamorphic rocks. The veins, up to 12 m thick, consist mainly of fine-grained fluorite and microcrystalline to chalcedonic quartz, and constitute a large fluor spar resource. Most of the fluorite is a fine-grained varicoloured, thinly layered coating ("ribbon spar" or "baconspar"). Euhedral crystals are scarce and generally less than 1 mm in diameter. The crystal forms present are mainly the cube and octahedron, but include at least 5 other forms. Minor amounts of opal, calcite, barite, pyrite, marcasite, manganese oxides, iron oxides, and clay minerals also occur. The wall rock shows only minor silicification and fluoritization. Warm springs
(moderately alkaline fluoride waters, with 500 to 800 ppm total salinity and 12 to 15 ppm F) are found in and near the mine workings. The depth of cover at the time of deposition was estimated at \( \sim 76 \) m.

NATURE OF THE SAMPLES AND INCLUSIONS DISTRICT
FROM BROWNS CANYON DISTRICT

A variety of types of fluorite samples were examined from seven locations in the Colorado-American deposit, two in the Alderman deposit, and one each from the last chance open pit, stayover claim and Curly Lee shaft (samples ER 62-50 and ER 65-76 through – 86). These included green, grey, purple and yellow-coloured fluorite with various structures (coarsely crystalline, chain structure, fibrous, finely layered and botryoidal). As no differences were recognized in the inclusion data from the various types, they are not listed separately. Doubly-polished plates approximately 1 mm thick were cut from each. Almost all contained two-phase primary inclusions (liquid+vapor; Fig. 1.1), of sharply faceted negative tetrahedral shape. These were generally in isolated, random distribution through the crystals, but with the orientation of their flat sides obviously controlled by the host crystal (i.e., one facet parallel to an octahedral growth surface, but with the points facing both inward and outward). Some occurred in groups, outlining growth

Fig. 1. Photomicrographs of the fluid inclusions (the bar is 10 \( \mu m \))

1 — Typical two-phase (gas plus liquid) tetrahedral inclusion, ER 65-85; 2 — Typical one-phase (all liquid) tetrahedral inclusion, presumably containing stretched liquid under negative pressure. ER 85-85; 3 — One-phase (all liquid) tetrahedral inclusion with small modifying facet of the second tetrahe-
dron (?). ER 63-76; 4a, b — Two inclusions from adjacent growth bands of a fluorite sample, each re-
presentative of the shape of inclusions in its band. Sample ER 66-67, from the Rock Candy mine, Yale
district, British Columbia, Canada, provided by W. W. Atkinson, Jr. 4a-d — Appearance of superheated ice in tetrahedral inclusion in sample ER 62-50, after equilibration at the temperatures indicated. The time between the individual photographs was 5 to 10 min. 1 — metastable, superheated ice; L — li-
quid water solution; V — vapour bubble; F — fluorite host crystal. At some temperature (or time) between photos b and c, a vapour bubble nucleated, relieving the negative pressure that maintained the metastable superheated ice, permitting it to melt instantly, at \( +3.8 \pm 0.1 \) \( \circ \) (R·o·d·d·w·e·g, 1967)
zones. Inclusions in some samples were too small to run on heating or freezing stages but their phase ratios were optically comparable to the other, larger inclusions. All samples contained at least some inclusions that were full of liquid at room temperature (Figs. 1.2, 1.3). These are generally the smaller inclusions, and are not found near to other inclusions with bubbles, from which they might have necked down. Most samples also contained at least some all-gas primary inclusions that do not appear to have leaked, i.e., they appear to represent a primary gas phase.

**Laboratory Technique**

After appropriate inclusions were selected, they were checked for bubble movement in a thermal gradient (Roeder, 1965). All bubbles that were free to move were attracted toward a source of heat. This uniformity of direction of movement is a necessary but not sufficient proof of uniform fluid composition.

The homogenization temperatures ($T_H$) were determined with a Leitz model 350 stage calibrated with organic compounds of known melting point (136 and 149.5°C) sealed in vacuum in tiny glass capillaries, run on the stage with the unknowns. The runs lasted several hours, with stepwise heating, letting the temperature level off after each increment. As a result of the technique used and the thermal gradients known to exist in the stage, most of the individual results are probably precise to only ±2°C. The accuracy is probably within this same range of error.

The freezing studies, yielding information on the salinity of the fluids, were made using the technique and equipment described earlier (Roeder, 1962). Calibration was obtained mainly by the use of a tiny sealed capillary of distilled water run with the unknowns. For lower temperatures, calibration was made at the melting point of n-dodecane (−9.6°C). After freezing (usually at −78°C), melting of ice was watched during very slow warming. Heating schedules varied from run to run, but were always sufficiently slow that thermal equilibration was essentially complete, since the sample plate was completely immersed in thermostated acetone flowing at 7 litres per minute. The largest source of error in the measurement of the freezing temperature ($T_F$) comes from the fact that in these low-salinity inclusions, the bulk of the heat flow to cause melting of the ice must occur in the last tenth of a degree (Roeder, 1962, p. 1060). For this reason, heating rates as low as 3°C/h were used in some runs. The precision of the individual measurement probably averages ±0.1°C. The accuracy is believed to be in this same range, but is exceedingly difficult to evaluate exactly.

**RESULTS FROM BROWNS CANYON SAMPLES**

The 179 homogenization temperatures range from 119 to 161°C, and average 141°C (Fig. 2). Seven inclusions homogenizing at higher temperatures are not plotted on Fig. 2 (one at 183°C and six in the range 229 to 265°C). These are believed to be spurious results, most likely from “stretching” of the samples on previous overheating (Larsen et al., 1973), or on freezing.

Notable for their absence on Fig. 2 are the many inclusions that appear homogeneous at room temperature. Inclusions trapped at room temperature
will have no bubble, but these one-phase, liquid-filled inclusions are believed to represent metastable equilibrium (at room temperature), i.e., they were trapped at \( \sim 140^\circ \text{C} \) but now contain stretched liquid under negative pressure, from failure to nucleate a vapor bubble since the original cooling \( \sim 7 \) mil-

![Graph](image)

Fig. 2. Plot of measurements of temperature of homogenization \( (T_H) \) vs. frequency for 179 primary inclusions, from Browns Canyon samples

lion years ago. Three facts support this contention: First, no two-phase inclusions were found with intermediate \( T_H \) (i.e., \(< 119^\circ \text{C} \)). Second, the samples did not decrepitate even at \( 160^\circ \text{C} \), nor did the liquid-filled inclusions become stretched \((\text{Larson et al., 1973})\). Yet if these liquid-filled inclusions had actually formed at room temperature, they would have developed internal pressures of 1,500–2,000 bars at \( 160^\circ \text{C} \), and \( \text{Khotchikov et al. (1968)} \) have shown that inclusions in a much stronger mineral, quartz, decrepitate when the internal pressure reaches only 800±30 bars. Third, some previously liquid-filled inclusions developed a normal-sized vapour bubble at room temperature after a freezing cycle, yet showed no signs of stretching. Similar reasoning precludes formation of the liquid-filled inclusions by necking down at near-surface temperatures.

The freezing data are shown in Fig. 3. The most common problem with these inclusions, metastable supercooling, was eliminated easily by freezing at \(-78^\circ \text{C}\) for 30 minutes (or more if needed). On warming the frozen inclusions to near \( 0^\circ \text{C} \), 20 were found to hold the stable assemblage ice + water + vapour bubble. \( T_F \), the melting of the last ice crystal under stable equilibrium occurred in five of these at \( 0.00 \pm 0.05^\circ \text{C} \), in one at \(-0.07 \pm 0.03^\circ \text{C} \), in eleven at \(-0.10 \pm 0.05^\circ \text{C} \), in two at \(-0.13 \pm 0.03^\circ \text{C} \), and in one at \(-0.15 \pm 0.10^\circ \text{C} \). These yield an average stable freezing temperature of \(-0.08^\circ \text{C} \), corresponding to \( \sim 1,400 \) ppm NaCl equivalent salts in solution.

Expansion on freezing generally eliminates the small vapour bubbles in these inclusions. Failure to renucleate a vapour bubble on reheating resulted in the persistence of metastable "superheated" ice, at temperatures as high as \(+6.6^\circ \text{C}\) and for times as long as four hours. Once the bubble has been eliminated, four different sequences involving metastable phases may occur on warming \((\text{Roden, 1967})\); only three of these were recognized in the Browns Canyon samples (Fig. 3). In sequence A, the superheated ice in the metastable assemblage ice+liquid (at negative pressure) melts suddenly

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but incompletely when a bubble nucleates and relieves the negative pressure. This was observed in only one Browns Canyon inclusion, at $-0.9\pm0.3^\circ$C (not plotted on Fig. 3); further warming of the then stable assemblage ice+liquid+vapor resulted in a determination of $T_F$ at $-0.10\pm0.05^\circ$C. In sequence B, the metastable assemblage superheated ice+liquid persists to temperatures above $T_F$ when nucleation of vapour then occurs, the ice melts suddenly and completely, leaving stable liquid+vapor (Fig. 1.5). In sequence C, the superheated ice melts gradually with increasing temperature under reversible but metastable equilibrium, until only metastable stretched liquid remains (Roe der, 1967, Fig. 2).

Why do these particular samples display such extensive metastability, in contrast to the occasional metastability found in many other studies? In part, this may be because metastability is more obvious in low-salinity inclusions such as these, where one can see ice at $>0^\circ$C. The basic causes for metastability in these samples are probably a combination of small inclusion size and very clean, slow-moving fluids, free of suspended solids to act as nuclei for the formation of a vapour bubble (Roe der, 1962; 1971). In addition, it is possible that nucleation of vapour in the stretched liquid occurs at the liquid/mineral interface, rather than by “homogeneous” nucleation out in the liquid. The apparent predominance of fluorite as a host for inclusions exhibiting metastable superheated ice may thus be an indication that the interface liquid/fluorite is stronger (in tension) than is that with other minerals. However, similar metastability (but generally lesser in magnitude) has been found in inclusions in barite, quartz, calcite, and sphalerite (Roe der, 1967). Theoretically, one should be able to observe the actual nucleation site, but this is not very feasible. The process of nucleation of vapor and complete melting of ice via sequence B was seen to occur within an estimated 0.1-0.2 seconds in several inclusions. High-speed microcinematography
should suffice to pinpoint nucleation, but as it may occur at any moment (over a period of minutes or hours), and at any point within the inclusion (which is usually thicker than the depth of field of the microscope lenses at these high magnifications), such a method is presently impractical.

STUDIES OF OTHER PRESUMABLY SHALLOW FLUORITE DEPOSITS

Northgate Fluorite District, Jackson County, Colorado. The fluorite deposits of the Northgate district are among the largest in the Western United States. They occur along large late Tertiary (?) faults, and consist mainly of botryoidal layers of fluorite and fine granular to chalcedonic quartz encrusting open vuggy rubble-filled veins (S t e v e n, 1960). Small amounts of clay and manganese oxides are also present. The wall rocks (sedimentary, metamorphic and igneous) are in part fluoritized. The depth of cover is unknown, but fluorite has been deposited over a vertical range of at least 300 m (S t e v e n, 1960). \( T_H \) was determined on then primary inclusions in a sample of coarsely granular green fluorite from this deposit (sample ER 65-75; Penbrook Shaft). The results are of low precision due to the nature of the sample, but are probably accurate to \( \sim \pm 5 \)°C. These determinations were 114°C (3 inclusions), 121°C (3), and 128°C (4). Presumably pseudosecondary inclusions \( (\sim 20) \) homogenized in the same range. S t e v e n (1960) reported \( T_H \) of 100-150°C for 44 similar inclusions in fluorite from Northgate, and N a s h (1971) reported salinities of \( \sim 0.2 \) weight per cent NaCl for inclusions from a presumably similar sample.

Wagon Wheel Gap, Mineral County, Colorado. This fluorospar deposit consists largely of a fissure vein of fluorite and barite, with lesser quartz and pyrite, on the east margin of the Creede caldera. An active hot spring issues a few hundred meters on strike west-northwest from the deposit (S t e v e n, L i p m a n, 1973). The fluorite occurs mainly as pale lilac, 2-cm, cubic crystals lining vein walls, with later coarse white bladed barite in the core of the veins (samples ER 59-27 and 28). The fluorite contains small, sharply cubic primary inclusions in cubic zonal array. The inclusions in the barite were all full of liquid. \( T_H \) determined on five primary inclusions in fluorite, ranged from 133 to 140°C. \( T_F \) was determined on 39 primary inclusions, with only minor problems from metastability; all were 0.00±0.1°C (R o e d d e r, 1963).

Cougar Spar Mine, Indian Peak Range, Beaver County, Utah. This mine, one of a group of presumably related small epithermal fluorospar deposits, has been described by T h u r t o n e t al. (1954). The deposit is in a fault zone near the contact between an intrusive quartz diorite and a Tertiary latitic to rhyolitic pyroclastic volcanic series. The fault zone is marked by a belt of quartz veins and vuggy quartz-filled breccia, with lamellar calcite and fluorite, and the adjacent tuff has been silicified. \( T_H \) was determined on 18 primary inclusion; it ranged from 188 to 195°C and averaged 191°C. Approximately 50 primary inclusions from a pale green, 2-cm fluorite cleavage fragment (ER 61-50), mostly with flat triangular wedge shape, were run on the freezing stage. All showed \( T_F \) of \( -0.10 \pm 0.05 \)°C, with little metastability. (Preliminary studies on this sample by R o e d d e r (1963) listed \( T_F \) as 0.1°C.)
Rampart Range, Colorado. This sample (ER 62-49) came from an undescribed but possibly similar barite-fluorite vein in the Rampart Range. It consists of white, green, and purple 1-cm cleavage masses. Five primary irregular to cubic inclusions in this sample all had \( T_F = 0.3 \pm 0.1 \)°C. Eleven showed metastable ice via sequence B at +0.32 to +6.23°C, and two via sequence C at +4.52 and +5.91°C.

Cuatro Palmas, Pico Eléreo, Coahuila, Mexico. This deposit, one of many epithermal fluorite deposits in Mexico, has been described by Van Aistine et al. (1962). It consists of fluorite replacements and cavity filling in Tertiary limestone cut by a Tertiary rhyolite dike. Twenty-four irregular to cubic, possibly primary inclusions in a sample of fine-grained purple to colourless “ribbon spar” (ER 62-52) yielded \( T_F \) ranging from \(-1.31 \pm 0.01\) to \(-8.12 \pm 0.03\)°C, and averaging \(-4.2\)°C. These fluids are thus \( \sim 50 \) times more saline than those from Browns Canyon, and the variation suggests that the fine banding may reflect a changing fluid composition. Four of these inclusions also showed metastable equilibria in other runs, via sequence B, at \(-0.63\), \(-0.95\), \(-1.35\), and \(-2.0\)°C. One, with \( T_F \) of \(-1.31 \pm 0.01\)°C showed sudden nucleation of a vapour bubble via sequence A at \(-1.35\)°C one time and \(-1.60\)°C another time (after holding at the respective temperature for 30 min).

Ohl Vein, Creede, Colorado. This deposit is one of a series of steeply dipping epithermal Ag-Pb-Zn-Cu veins in Tertiary rhyolitic welded tuffs in the Creede district, Mineral County, Colorado (Barton, Bethke, and Roeder, ms. in preparation). Although it is not a fluorite deposit, it does have some fluorite as pale green to colorless octahedra and cleavage masses \( \sim 3 \) cm, fairly early in the paragenesis, before much of the sphalerite that has been studied extensively (Roeder, this volume). It is discussed here as a typical example of near-surface epithermal polymetallic vein deposits. Field studies suggest a maximum of 600 m of cover at the time of ore deposition. Much of the fluorite has been removed by Pater fluids, leaving casts.

A total of 449 inclusions were run on the heating and freezing stages from 22 fluorite samples taken throughout the mine. Unfortunately, the origin of many of these inclusions is uncertain. However, the data for all 449 fall into one or another of three rather narrow ranges of values for \( T_H \) and \( T_F \), respectively, as follows (in each case the minimum, average and maximum are listed in sequence, in °C): 255, 261, 268, and \(-6.5\), \(-6.93\), \(-8.5\) (79 inclusions); 210, 214, 229 and \(-6.5\), \(-7.04\), \(-7.5\) (330 inclusions); 150, 160, 168, and \(-4.5\), \(-4.5\), \(-4.5\) (40 inclusions). The first of these groups consists mainly of inclusions believed to be primary, the second of uncertain origin, and the third of two planes (\( \sim 20 \) inclusions each) of obviously secondary origin. It is interesting to note that of the three separate fluids indicated by these data, only the first overlaps with data found for inclusions in later sphalerite (Roeder, this volume); the second and third, which one might expect to have the greatest possibility of being contemporaneous with the fluids responsible for the later sphalerite deposition, differ greatly from them.
POSSIBLE SIGNIFICANCE OF TETRAHEDRAL INCLUSION SHAPE

Although inclusion shape can be transient and ambiguous (Roeder, 1971), the inclusions described here do not appear to have changed shape significantly since trapping. As such it is of importance to examine the problem of how these regular and distorted tetrahedra were formed. Similar tetrahedral inclusions are common in fluorite from some other localities, but most inclusions in fluorite, from most localities, are either composite cubes, or wedge-shaped but still subparallel to the cube. The origin of such cubic shapes is evident from inspection of the step-like growth on the outer surface of most fluorite cubes. Similarly, distorted tetrahedral inclusions might be trapped by growth on an octahedral face covering over trigonal pits outlined by faces of any of several forms, e.g., the cube or the trioctahedron. Regular tetrahedral inclusions could form by covering over pits outlined by three other octahedron faces. Etch pits formed on fluorite have been studied intensively since the early careful work of F. Becke. Etch pits of both regular and distorted tetrahedral shapes commonly form on octahedron faces, which, if covered over by later growth, would yield tetrahedral inclusions very similar to those described here. (Universal stage measurements should be made on the inclusion facets to determine their actual crystallographic orientation.)

Although there seems to be no X-ray evidence for tetrahedral symmetry in the structure of pure fluorite, it might be possible that substitution of rare earths for calcium could lower the symmetry to tetrahedral. The problem of recognizing lower symmetry in crystals that exhibit only simple crystal forms, that are possible with either high or low symmetry, is common in morphological crystallography. In this connection it is important to note that Kieffer (1932) reported systematic differences between the etch pits on adjacent octahedral faces, possibly suggesting the lower symmetry for a specific fluorite sample. Most fluorite samples show either tetrahedral or cubic inclusions, but not both. An exception is shown in Fig. 1.4, from adjacent growth bands in a fluorite crystal from the Rock Candy mine, Yale district, British Columbia, Canada, first brought to my attention by William Atkinson, Jr. (sample ER 66-67). He noted that this 4-cm banded fluorite crust shows early green growth bands parallel to the octahedron, outlined by slight colour differences and by planes of solid and tetrahedral liquid inclusions (Fig. 1.4a), and an outer zone of purple color, which grew as cubes ~1 cm in length, and has sharply cubic inclusions (Fig. 1.4b). Electron microprobe traces were made across these bands, by E. J. Dwornik, U. S. Geological Survey, but unfortunately, the rare earth content was too low to reveal any differences.

CONCLUSIONS

1. Metastability can cause serious errors in freezing measurements on inclusions, and must not be ignored.
2. Primary inclusions in fluorite from the deposit at Browns Canyon generally have a tetrahedral shape, of uncertain origin.
3. Data on the homogenization temperatures ($T_h$) and freezing temperatures ($T_f$) of the fluids in these inclusions are compatible with the field evidence of a hot-spring type of environment at the time of formation. The solu-

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tions in the inclusions have twice as much salts in solution (∼1400 ppm) as the present hot-spring fluids, but are still very dilute, and it is to be expected that this concentration would decline during the waning of any hot-spring system. The presence of all-gas inclusions, representing effervescence or boiling, would also be expected at the shallow maximum depths for this deposit (76 m) inferred from the geology. Even if the fluids were at the highest measured \( T_H \) (161°C), the pressure from a column of pure water, at the boiling point throughout and just adequate to prevent boiling, would be only 6 bars (57 m depth) (Haas, 1971), and fluids at the average \( T_H \) (141°C) would require only 28 m depth. The differences are easily explicable in terms of depth to the water table or small amounts of gas in solution.

4. The difference between the fine grain size of much of the ore at Browns Canyon (and similar deposits) and the coarse crystals of fluorite in some of these deposits may be a result of relatively minor variation in the degree of supersaturation of the fluids, making the difference between continuous nucleation and growth, or just continued growth on existing nuclei. As \( \text{CaF}_2 \) exhibits retrograde solubility in most aqueous solutions above \( \sim 100^\circ \text{C} \) (Holland, 1967), changes that are likely to cause a high degree of supersaturation would come most probably from varying the mixing ratio of waters of different compositions.

5. The solubility of \( \text{CaF}_2 \) is generally in the range of 10 ppm in these dilute solutions, requiring that large volumes of water must have passed through these deposits (Roedder, 1960, Fig. 6).

6. The low solubility and the relatively large crystal size in some deposits, and the common occurrences of metastable phenomena in the inclusions, all suggest exceedingly quiet and stable conditions, very low flow rates and long times for deposition.

7. The normal epithermal ore deposits (either fluorite or polymetallic), appear to have formed from hotter and necessarily deeper fluids that are 50 or more times as saline as the hot-spring type, although intermediate types have been described (Roedder, 1963), and there may well be a continuum between these two environments.

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