Halite and sylvite as solid inclusions in high-grade metamorphic rocks

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Abstract. Solid inclusions of halite and sylvite, formed during regional and contact metamorphism have been identified by microscopy and by electron microprobe analysis in rocks from Campolungo, Switzerland and Cornone di Blumone, Italy. The solid inclusions occur in several of the major minerals crystallized during metamorphism and have been observed as idiomorphic crystals and dendrites. The compositions measured in 100 analyses from Campolungo, Switzerland and 40 analyses from Cornone di Blumone, Italy extend across the two-phase region in the system, KCl − NaCl, indicating that the salt inclusions are high temperature precipitates. In both localities compositionally zoned and unzoned crystals have been found. Measured compositions on the temperature maximum of the two-phase region indicate at least 500° C which can be compared with 500° ± 20° C determined by Mercolli (1982) and Walther (1983) from the Mg content of calcites from Campolungo. The solid inclusions have been trapped apart from CO_2-rich and saline, H_2O-rich fluid inclusions which have been described by Mercolli (1982) as the earliest preserved fluid inclusions in the rocks. The early precipitation of salt minerals at Campolungo indicates that fluids were saturated with NaCl and KCl at 500° C and pressures of 2,000 bars or higher. Similar relationships exist between solid and fluid inclusions in the rocks of Cornone di Blumone which formed at temperatures as high as 800° C and pressures between 0.5 and 1 kilobar (Ulmer 1983). The entrapment of halite and sylvite as solid inclusions preserves the composition of the minerals which may therefore be useful as geothermometers.
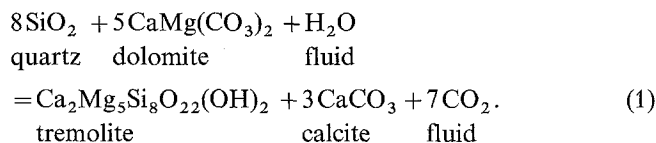
Introduction

The evolution of volatiles during the progressive metamorphism of carbonate rocks has frequently been considered as a process involving a single fluid phase in the binary system, $CO_2 − H_2O$ (see for example, Carmichael 1970; Trommsdorff 1972; Greenwood 1975; Flowers and Helgeson 1983; Walther 1983). The study of fluid inclusions in metamorphic rocks has, however, indicated the presence of salt-bearing fluids (for example, Poty et al. (1974); Crawford et al. (1979); Kreulen (1980); Touret (1981); Sisson et al. (1981). Bowers and Helgeson (1983a, b) have calculated the position of phase boundaries in the ternary system, $NaCl − H_2O − CO_2$, using experimental data obtained by

Gehrig (1980). Bowers and Helgeson have drawn attention to the location of the two-phase region H_2O-rich liquid + CO_2-rich vapour, and commented on the likelihood of ternary, two-phase fluids being present in metamorphic rocks to much higher temperatures than in the binary system, $CO_2 − H_2O$. Mercolli (1979) has provided evidence from fluid inclusions in support of coexisting liquid and vapour in the tremolite zone of the amphibolite facies in marble from Campolungo in the Lepontine Alps. Sisson et al. (1981) have found evidence of CO_2-brine immiscibility at 6.5 kilobars and 600° C. We have re-examined rocks from Campolungo and other localities and found petrographic evidence not only for the co-existence of two fluids at high metamorphic grade, but also for the saturation of fluids with respect to chlorides such as halite and sylvite prior to their entrapment.

The system, $NaCl − H_2O − CO_2$

A series of isothermal sections in the ternary system, $NaCl − H_2O − CO_2$, has been described by Gehring (1980) for temperatures from 100° C − 800° C and a pressure of 1,000 bars. Bowers and Helgeson (1983a) have calculated isothermal sections from 400° C to 550° C at 2,000 bars. The major features of these isothermal sections have been summarized in a schematic polythermal projection presented in Fig. 1. This figure illustrates the large three-phase region, halite + liquid + vapour, and its surrounding two-phase regions. The two-phase region, liquid + vapour, passes into a supercritical fluid in moving towards the binary, $CO_2 − H_2O$. Also shown in Fig. 1 is a series of isothermal equilibrium curves along which the following metamorphic reaction is at equilibrium for temperatures increasing from 1 to 6.

$$8\,SiO_2 + 5\,CaMg(CO_3)_2 + H_2O$$
quartz   dolomite   fluid

$$= Ca_2Mg_5Si_8O_{22}(OH)_2 + 3\,CaCO_3 + 7\,CO_2. \tag{1}$$
tremolite   calcite   fluid

The intersection of the equilibrium curves for reaction 1 with the binary system, $CO_2 − H_2O$, is known from experimental measurement by Eggert and Kerrick (1981) as well as from calculated $T − X_{CO_2}$ diagrams (e.g. Walther and Helgeson 1980). Equilibrium curves for temperatures 3 and 4 are joined across the two-phase region, liquid + vapour, by tielines which have been calculated by Bowers and Helgeson (1983b) for 2,000 bars. At temperature 5 on Fig. 1,
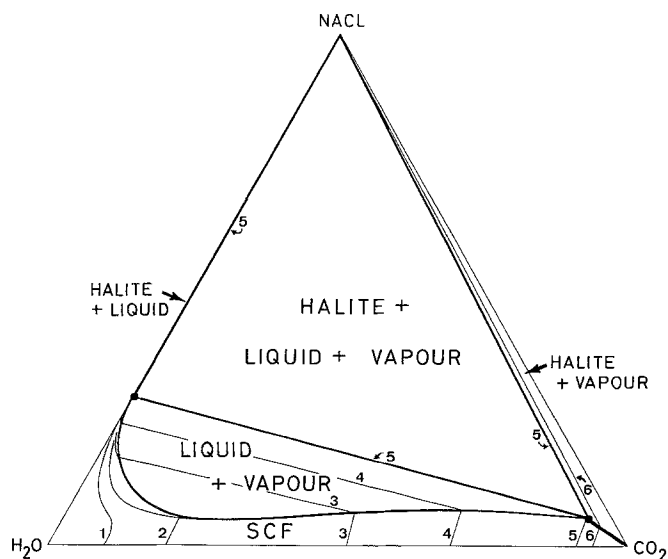
Fig. 1. Schematic, isobaric, polythermal projection of the system $NaCl-CO_2-H_2O$ with a series of isothermal equilibrium curves for a reaction such as 5 Dolomite + 8 Quartz + H_2O. Tremolite + 3 Calcite + 7CO_2. Temperatures represented by the equilibrium curves increase from 1 to 6. Isotherm 2 encounters the two-phase region, liquid + vapour, at the consulate composition. Isotherms 3 and 4 are joined across the two-phase region by tie-lines as calculated by Bowers and Helgeson (1983a). Isotherm 5 represents the temperature for equilibrium in the three-phase region, halite + liquid + vapour. SCF (supercritical fluid)

the equilibrium curve includes the three-phase triangle, halite + liquid + vapour.

The position of equilibrium curves for reaction 1 on Fig. 1 suggests that the composition of fluids buffered by the mineral phases during progressive metamorphism could be carried towards the three-phase region and might, under certain conditions, become saturated with respect to salt. It was therefore decided to look at natural parageneses to see if evidence for such saturated fluids could be found. The Campolungo area in the Lepontine Alps of Switzerland was considered a suitable study area because it has been previously subjected to careful study of fluid inclusions by Mercolli (1979, 1982) and of metasomatic processes by Walther (1983).

Sample Preparation

The most likely means of preserving salt crystals precipitated at a high grade of metamorphism is as solid inclusions within the more abundant phases formed during metamorphism. Salt crystals exposed to fluids migrating along grain boundaries during retrograde metamorphism or during sample preparation could be wholly or partially dissolved or recrystallized. In order to minimize dissolution of salt crystals during sample preparation in this work, petroleum distillate rather than water was used during the cutting of thin sections and polished sections. Sections for electron microprobe analysis were slowly polished with abrasive paste and coated with 200 Å of carbon. Care must be taken to clean any residues on the surface of polished or thin sections. Salt may precipitate from wet fingerprints!

Fluid Inclusions from Campolungo

Siliceous carbonate rocks at Campolungo have been metamorphosed to the lower amphibolite grade with the devel-

opment of talc and tremolite. Bianconi (1971) describes two textural types of tremolite-bearing marbles at Campolungo. A mosaic including calcite, dolomite, some quartz and prismatic tremolite oriented parallel to the foliation is typical of the main body of marble. A second textural type of marble is observed in unfoliated veins and nodules that contain radiating clusters of tremolite crystals up to 20 cm in length and megacrysts of calcite. Tremolite and calcite form an armouring texture that separates quartz veins and nodules from the mosaic-textured dolomite. These latter textures associated with veins and nodules have been referred to as "chaotic" by Mercolli (1982, Fig. 6). Walther (1983, Fig. 2) has discussed the development of this armouring texture as a consequence of metasomatism. The two types of tremolite are also distinct in their compositions (Mercolli 1982). The earlier prismatic tremolite is rich in edenite component; the later, radiating tremolite is Al-poor with a higher F content. Fluorite has been identified at one vein locality.

Mercolli (1979) has described the contemporaneous development of two types of consanguinous fluid inclusions in these veins. One type of inclusion has a very high CO_2 content (more than 85 volume %) while the other type has a high H_2O content with up to 50 weight % of dissolved salt. The water-rich inclusions now contain liquid, a gas bubble, an isotropic salt crystal and may also contain additional and unidentified solid phases. Our petrographic examination confirms the existence of these inclusions in calcite and tremolite as well as quartz. These inclusions have features consistent with an early origin, as suggested by Mercolli (1982, p. 293). Mercolli has also recognized later generations of both CO_2 and H_2O inclusions. Walther (1983) has described three types of fluid inclusions, two of which are aqueous and a third contains a mixture of CO_2, H_2O and CH_4. Mercolli (1982) and Walther (1983) have both estimated temperatures of $500° \pm 20°$ C from measurements of the Mg content of calcites in equilibrium with the other phases of reaction 1 at Campolungo. It is more difficult to define a specific pressure for these rocks. Pelitic schists associated with the marbles in the area contain staurolite, garnet and kyanite. Rosenfeld (1969) estimated a pressure of seven kilobars for these schists on the basis of piezobarometric observations. Bianconi (1971) has correlated the development of the mosaic-textured marbles with the pelites. For the mineral assemblages in the veins and nodules, Walther (1983) estimated a pressure of 3.25 ± 0.5 kilobars and Mercolli (1982) estimated 2 kilobars; both of these studies are based on measurements of fluid inclusions.

Solid inclusions from Campolungo

Duplicate samples of rocks studied by Mercolli (1982) have been prepared as described above and examined petrographically. Salt crystals up to 80 μm in size were visible under the petrographic microscope and their identification was confirmed with an ARL-SEMQ electron microprobe. Such crystals are represented in Fig. 2. The salt in this figure shows a dendritic habit and is intergrown with quartz. Sufficient crystals were exposed on or near the surface of the thin sections to obtain over 100 analyses with the microprobe. Because of the dendritic nature of certain of the crystals the microprobe analyses may include elements from the matrix mineral. Analytical results for typical halite-syl-
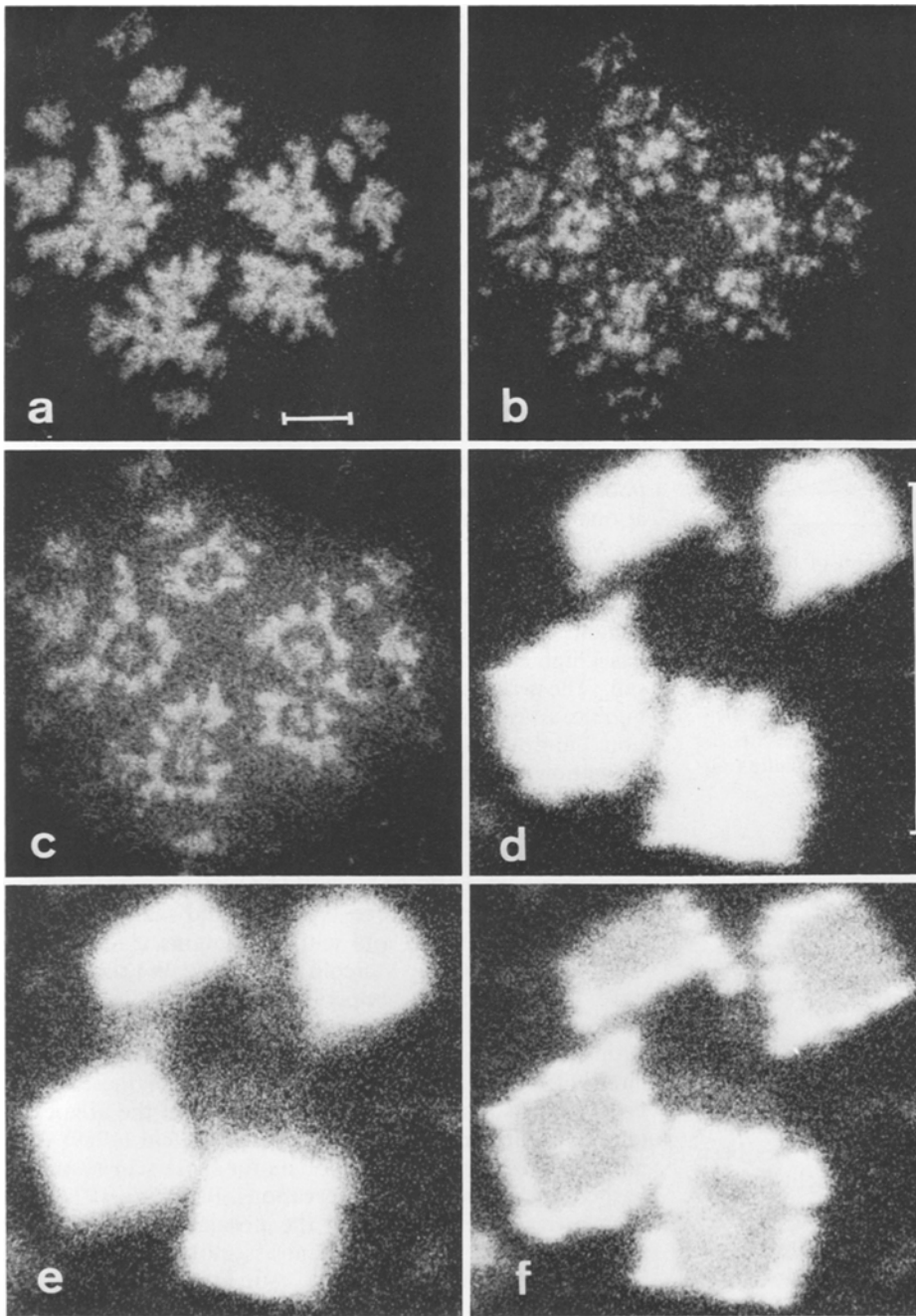
Fig. 2a–d. Crystal X-ray spectrometry of halite and sylvite solid inclusions in quartz crystals from Campolungo, Switzerland. A 25 μm scale is indicated by bars on **a** and **d. a–c** are of the same dendritic crystal. **d–f** are the same idiomorphic crystals as detected by the following radiation: Cl K_α (a, d), Na K_α (b, e) K K_α (c, f)

vite solid inclusions from Campolungo are given in Table 1. The entire spectrum of elements was scanned with the microprobe in the energy-dispersive mode and halite and sylvite as standards. Only Na, K, and Cl were detected in substantial amounts with minor Ca, apart from elements present in the enclosing crystal. The majority of analyses were made with quartz as the matrix; inclusions in tremolite and calcite can also be analyzed. The weight percent sums in Table 1 are substantially less than 100 because of detection of elements such as silica from within the matrix. The ratio of alkalies to chlorine exceeds unity for some analyses in Table 1, apparently because of analytical difficulties with chlorine. The values of X_{NaCl} were calculated from the measured Na and K contents.

The analyses from Table 1 indicate that salt crystals from Campolungo have compositions that are intermediate in the solid solution series between sylvite and halite. (Thompson and Waldbaum 1969; Skeaff et al. 1979). The two-phase region in the system NaCl − KCl at one atmosphere is reproduced in Fig. 3 after Waldbaum (1969). This diagram is taken as an approximation of the two-phase region at higher pressures. The range of compositions measured in 100 analyses from two samples is shown as a bar, labelled C, in the upper part of the diagram. The existence of compositions corresponding to the maximum of the immiscibility region indicates temperatures of at least 500° C. Analyses (3A) and (3B) from Table 1 are from different parts of the single solid inclusion shown in Fig. 2 and are indicated as circles on the two phase region in Fig. 3. These data are consistent with unmixing of the crystal.

Table 1. Representative microprobe analyses of halites and sylvites

Wt.%	B1B(1)	B1B(2)	B1B(3)	B1B(3B)	AU 45
Na	16.77	35.35	7.65	28.21	25.84
K	23.99	2.49	36.04	5.62	8.83
Cl	45.00	54.44	35.26	42.22	45.91
Atomic ratios					
$\Sigma(K+Na)/Cl$	1.05	1.04	1.19	1.15	1.04
X_{NaCl}	0.54	0.95	0.26	0.90	0.83

Sample B1B is from Campolungo, Switzerland. (1) and (2) are unzoned solid inclusions in quartz. (3A) and (3B) are from different zones in a single inclusion in quartz. Sample AU 45 is a solid inclusion within clintonite from Cornone di Blumone, Adamello, Italy. Accelerating voltage 15 KV; beam current 10 nA on brass for B1B and 20 nA for AU 45; beam diameter 2 microns. Ca max. 1 wt. %. Matrix minerals of samples B1B are quartz and tremolite; and of sample AU 45 clintonite and forsterite. Analyst P. Ulmer
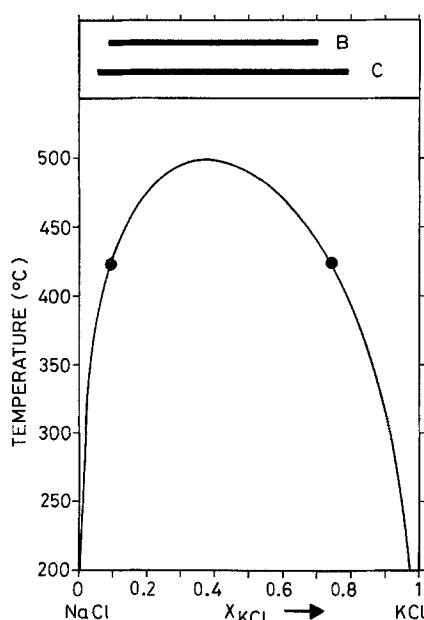

Fig. 3. The two-phase region in the system, NaCl−KCl, after Waldbaum (1969). Horizontal bars labeled B and C correspond to the range of compositions measured by electron microprobe. B is based on 40 analyses of inclusions in 2 specimens from Cornone di Blumone, Italy. C is based on 100 analyses of inclusions in 2 specimens from Campolungo Switzerland. Two points shown in full circles on the two-phase curve correspond to analyses 3a and 3b from Table 1 for two zones from the dendritic inclusion shown in Fig. 2a to 2c

Cornone di Blumone, Adamello

Solid inclusions of halite and sylvite have also been observed in metamorphic rocks from Cornone di Blumone in the Adamello Complex, northern Italy. Ulmer (1983) has described the petrography of these clintonite and for-sterite-monticellite marbles which occur as large pendants in gabbroic and dioritic rocks. He estimates pressures between 0.5 and 1 kilobar on the basis of stratigraphy. His temperature estimates are 800–850° C for the monticellite marbles, based on the solvus, forsterite-monticellite (Warner and Luth, 1973). Clintonite marbles appear to have formed at lower temperatures.

Clintonite megacrysts form large poikiloblastic crystals that enclose diopside, spinel and calcite. The clintonite con-

tains two types of fluid inclusions. One of these is typically dark with a liquid and a gas, i.e. characteristics of CO_2-rich inclusions. The second type of fluid inclusion contains a liquid, a gas and a cubic solid, i.e. features of a salt-satu-rated aqueous fluid. Halite and sylvite occur within the clintonite as dendritic and idiomorphic solid inclusions up to 50 µm in size. Similar features have been observed in the solid and fluid inclusions within large monticellite over-growths. A representative analysis corresponding to the top of the two-phase region in the system, NaCl−KCl, is given in Table 1 for a solid inclusion within clintonite. The range of compositions measured in 40 analyses is indicated by the bar, labeled B, at the top of Fig. 2.

Halite and sylvite have also been discovered at Blumone as intergranular minerals within the matrix of the rock. It appears, that intergranular salt minerals can survive re-trogression of metamorphic rocks and can be identified in specimens cut and polished without water. Salts tend to precipitate rather than dissolve during retrogression as indi-cated by fluid inclusions. Intergranular salt minerals as well as the monticellite may have been isolated from undersatu-rated ground waters by the relatively ductile calcite matrix. Intergranular salt minerals, however, are less likely than solid inclusions to retain early compositions. We have ob-served that intergranular salts are homogeneous and ap-proach end-member composition.

Origin of solid salt inclusions

The compositions of the chloride inclusions plotted on Fig. 3 demonstrate that these crystals are high temperature precipitates. The concordance of temperatures obtained from solid salt inclusions and from the magnesium content of calcite (Mercolli 1982; Walther 1983) containing these inclusions at Campolungo is consistent with their contem-poraneous formation. The high metamorphic temperatures estimated for Cornone di Blumone rocks are above the minimum in the liquidus curve of the binary system, NaCl−KCl (Waldbaum 1969; Chou 1982). It is possible, therefore, that molten salts existed in these rocks prior to the development of solid inclusions.

Alternative mechanisms can be considered for the for-mation of these solid inclusions. Exsolution can be elimi-nated because of salt immiscibility with entrapping minerals such as quartz. A late secondary origin is unlikely in view of the temperature concordance of salt minerals with mag-nesium calcites from Campolungo. Necking down or leak-ing of fluid inclusions containing daughter salt crystals at the time of vein formation cannot be entirely excluded as a possibility for the origin of the solid inclusions. However, fluid inclusions and their decrepitates contained significant amounts of S and other elements under the microprobe (see also Mercolli 1982, Table 2) that could not be detected in the solid inclusions.

Roedder (1984, p. 246) has suggested that the best proof for the existence of fluids saturated with respect to NaCl before trapping would be NaCl crystals enclosed in other minerals. We believe that the halite and sylvite inclusions at Campolungo and Cornone di Blumone indicate the pres-ence of saturated brines during the formation of the entrap-ping mineral asemblages.

Many geological processes could lead to the develop-ment of salt-saturated fluids, for example changes in pres-sure and temperature, or preferential escape of volatile com-
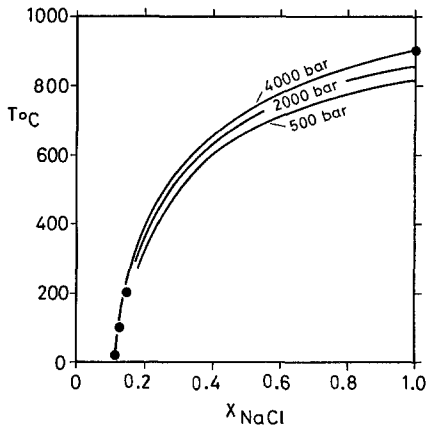
Fig. 4. Isobaric curves giving the composition of liquids saturated with NaCl in the system, $NaCl-H_2O$. Solid circles represent data points for 4,000 bars. The melting of pure NaCl is from Akella et al. (1969) and the remaining points from Hilbert (1979). Curves at 2,000 and 500 bars are from Bowers and Helgeson (1983a)

ponents from the fluid. Changes in fluid compositions which lead to concentration of salts may also result from the progress of mineral reactions during prograde or retrograde metamorphism. For example, metamorphic reactions that consume H_2O are likely to concentrate salts in the residual aqueous fluid.

The influence of pressure and temperature on the composition of the liquid in equilibrium with vapour and halite can be assessed with the information on Fig. 4. This figure gives the composition of liquids in equilibrium with solid NaCl. The curves for 500 and 2,000 bars are taken from Bowers and Helgeson (1983a) the curve for 4,000 bars is based on data from Akella et al. (1969) and Hilbert (1979). The amount of dissolved NaCl in the aqueous liquid varies considerably with respect to temperature and pressure as indicated by Fig. 4. The composition of the CO_2-rich vapour does not vary greatly between 400° and 550° C and between 0.5 and 2.0 kilobars (Bowers and Helgeson 1983a). The composition of the liquid is therefore the principal factor in the expansion or contraction of the three-phase region on Fig. 1 and Fig. 5. An increase in pressure or a decrease in temperature causes expansion of the three-phase triangle such that salt could precipitate from a fluid that originally had a bulk composition in the two-phase or one-phase region.

Shifting bulk composition of the fluid as a result of metamorphic reaction is illustrated on Fig. 5. The four dashed "fluid-evolution-curves" corresponding to the addition of CO_2 and the subtraction of H_2O from the fluid in the ratio, 7:1, have been drawn on this figure to correspond with the stoichiometry of reaction (1). The four curves have been arbitrarily chosen from the family of such curves to illustrate the evolution of the bulk composition of fluids with progress of reaction (1). One such curve, which is tangent to the three phase region, is shown in a heavy-dashed signature on Fig. 5. Bulk compositions above the heavy curve will encounter the three-phase region during reaction progress while those below this curve will remain in the two-phase or one-phase region if there is not preferential loss or gain of components. An increase in temperature from 5–6 on Fig. 1 may cause rapid progress of the reaction across the three-phase region. The dashed reaction curves on Fig. 5 also indicate that rapid reaction
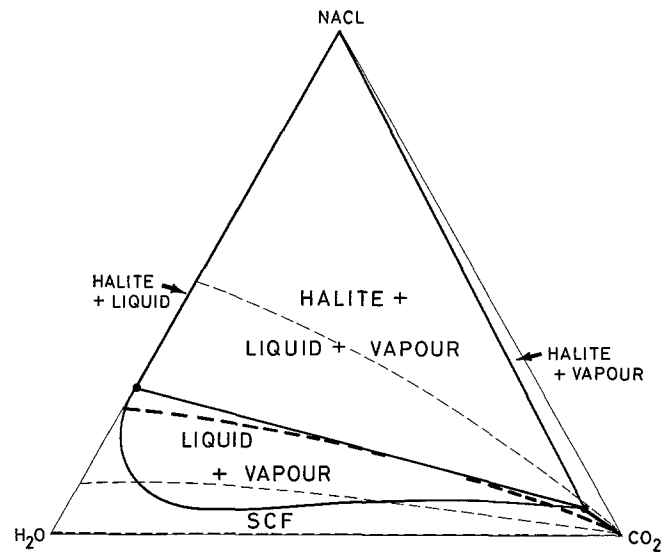

Fig. 5. Schematic, isobaric, polythermal projection of the system, $NaCl-CO_2-H_2O$. Dashed curves correspond to typical reaction paths for production of CO_2 and removal of H_2O from the fluid in the ratio, 7:1, as for the metamorphic reaction, 5 Dolomite + 8 Quartz + H_2O = Tremolite + 3 Calcite + 7 CO_2. The heavier dashed curve corresponds to the reaction path that is tangent to the three-phase region, halite + liquid + vapour. Bulk compositions of the fluid phase falling above the heavy dashed curve will progress through the three-phase region with continuing reaction. Bulk compositions beneath the heavy dashed curve will remain in the two-phase region, liquid + vapour, or in the region of supercritical fluid (SCF)

could occur in recrossing lower temperature isotherms along certain "fluid-evolution-curves".

Concluding statement

The observation and data presented in this paper demonstrate the existence in metamorphic rocks of salt-saturated liquids in equilibrium with vapour over a wide range of metamorphic temperature and pressures. It appears from our studies to date that saturation of fluids with respect to salt components such as KCl and NaCl is associated with development of characteristic textures in metamorphosed carbonates. These poikiloblastic and/or armouring textures that characterise product minerals crystallized from salt saturated fluids may be related to a different reaction mechanism than applies to rocks with a mosaic texture.

Equilibrium models for the metamorphism of carbonates must be extended to take account of this new range of saline metamorphic fluids as suggested by Bowers and Helgeson (1983b). It will then be possible to re-evaluate the $P-T-X$ paths of carbonate rocks during progressive and retrograde metamorphism to take account of isograds that result from variation in salt content of the fluid as well as factors that have been previously considered.

The recognition of co-trapped halite and sylvite crystals as solid inclusions provides an independent geothermometer for the interpretation of fluid inclusions. The bulk composition of an entrapped solid inclusion is preserved by the enclosing mineral even though exsolution may occur. Intergranular salt crystals, on the other hand, may be subject to changing composition or dissolution through contact with fluids.

We have observed textures similar to those described

in this paper in samples collected at many localities documented by other workers including Ben Suardal, Scotland (Tilley 1948), Cima Uzza, Adamello, Italy, (Callegari 1963), Val Schiesone, Bergell, Switzerland (Oterdoom and Gunter 1983), Monzoni, Italy (Masch 1977). In addition to the characteristic textural features referred to above, we have observed petrographically the existence of two sets of fluid inclusions with separate solid inclusions similar to those described in this paper at Campolungo and Blumone.

The common occurrence of evaporites in Triassic rocks from the Alps may have contributed to the evolution of saline solutions during Alpine regional metamorphism. Release of saline solutions from igneous rocks and concentration of salt in the formation waters of sediments are well known processes. In the case of the shallow intrusions, such as Adamello and Monzoni, sub-oceanic convection cells may have circulated saline solutions originating from sea water. We anticipate that evidence for salt saturated fluids will be found in a variety of rock types in addition to the carbonate localities described in this paper[1].

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[1] After revision of this manuscript it came to our attention that A. Irouschek (unpublished Dissertation, University of Basel) has detected halite-sylvite as inclusions in cordierite from the Central Alps. The nature of these inclusions, whether solid inclusions or formed from decrepitates or necking down of fluid inclusions remains to be investigated