Oxygen and hydrogen isotopic evolution of Variscan crustal fluids, south Cornwall, U.K.

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Abstract

Structural analysis of quartz vein systems and fluid inclusion criteria were used to distinguish five different fluid types which flowed through a segment of Palaeozoic crust in southwest England during the Variscan orogeny. Mineralogical constraints in combination with fluid inclusion thermobarometry enabled the temperature of vein formation to be estimated, and isotopic compositions of fluids were determined by analysis of vein material and direct measurement on fluid extracted from inclusions. Peak, low-grade (pumpellyite-actinolite facies) metamorphic fluids had a high δD and δ18O signature (δD = -18 to -10‰, δ18O = +10.6 to +11.9‰) which evolved to compositions in the range δD = -28 to -13‰, δ18O = +7.9 to +11.4‰ during later retrogression and uplift. Fluids in the contact aureole of the Cornubian batholith had δD-values intermediate between typical magmatic compositions and the regional metamorphic fluids (-23 to -43‰), and a similar range of δ18O-values to both magmatic and the regional metamorphic fluids (between +5.6 and +14.0‰). These compositions are comparable with those of fluids responsible for Sn-W mineralisation in the province. Post-erogenic fluid chemical and isotopic compositions were exotic and indicate significant infiltration of externally-derived fluids during late- to post-erogenic brittle faulting. Low-temperature, low-salinity fluids which circulated in ENE-WSW-trending brittle normal faults had low δ18O-values (-0.3 to +7.4‰) suggestive of a significant meteoric component. Low-temperature, high-salinity fluids, which flowed through N-S- to NNW-SSE-trending strike-slip faults and fractures and were responsible for Pb-Zn mineralisation, had significantly D- and δ18O-depleted compositions (δD = -80 to -49‰, δ18O = -0.1 to +4.7‰), typical of basinal brines. These data document the isotopic evolution of fluids in an external (Rhenodurcynian) part of the Variscan orogen, through the complete cycle of foreland thrust-belt development and low-grade regional metamorphism, S-type granite emplacement and associated hydrothermal systems, post-erogenic collapse and low-temperature fluid flow in regional fractures. There is limited overlap in isotopic composition between the different fluid types, indicating that fluids flowing through the same host rocks at each stage of orogenesis may be distinguished on the basis of their oxygen and hydrogen isotopic compositions. These data provide a framework for future studies involving fluids of unknown origin in the Variscan and are a reference for comparison with the isotopic evolution of fluids in other orogenic belts.

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1. Introduction

The relative chronology of complex quartz vein systems developed in a segment of Palaeozoic crust in southwest England has been documented by Wilkinson (1990a, b). The fluid inclusions present in these veins provide a direct record of fluid flow within a segment of Palaeozoic crust over a time period of >100 Ma during multiple phases of Variscan deformation, granitoid emplacement and associated hydrothermal circulation, and late- to post-orogenic faulting. The stable isotopic compositions of these fluids were determined in order to: (1) document the isotopic evolution of fluids in an external part of the Variscan orogen; (2) provide an indication of fluid source and the relative importance of locally-derived and exotic fluids; (3) provide constraints on fluid–rock interaction; and (4) evaluate the role of different fluids in mineralising processes in the province.

Fluid $\delta^{18}$O and $\delta^2$D compositional estimates are commonly based on analysis of mineral phases, either separated from whole rocks or from veins. Such determinations are subject to uncertainties: the parageneses of the minerals analysed, the recognition of re-equilibration during superimposed hydrothermal events, the choice of fractionation factors, the estimation of temperature required to apply these factors, as well as the practical difficulties of mineral separation. These problems are particularly acute in areas where rocks have been affected by multiple deformational, metamorphic and hydrothermal events such as in the Variscan province of southwest England. It is possible to eliminate some of these uncertainties by analysing the fluids directly for $\delta^2$D via fluid inclusions and, since quartz is extremely resistant to oxygen isotopic exchange after precipitation (Cole and Ohmoto, 1986), by analysing quartz vein material for $\delta^{18}$O. In addition, quartz is relatively resistant to post-entrapment leakage or decrepitation of inclusions (Roedder, 1984). This approach can be successful provided that detailed studies have demonstrated that samples are dominated by inclusions of one generation which are representative of the vein-forming fluid and that reasonable constraints on vein formation temperature are available.

Few direct analyses of the stable isotopic composition of palaeofluids in the European Variscides have been reported in the literature, and those are generally restricted to late- to post-orogenic mineralisation-related samples (Kelly and Rye, 1979; Turpin et al., 1990; Blamart et al., 1992; Munoz et al., 1994). A limited number of studies have been carried out in the Variscan (Cornubian) mineralised province of southwest England, both by direct analysis of fluid inclusions (Bray, 1980; Shepherd et al., 1983a; Lin, 1989; Alderton and Harmon, 1991) and via analyses of minerals of granite-hydrothermal origin (Sheppard, 1977; Jackson et al., 1982). The only constraints on the isotopic composition of Variscan metamorphic fluids comes from analyses of regional metamorphic mineral phases (Primmer, 1985).

Here we report stable isotopic data obtained on a subset of 18 samples from a thorough fluid inclusion study of 60 quartz veins. In all, over 1400 fluid inclusions were studied and in excess of 4500 microthermometric data collected (Wilkinson, 1990a, b). These data provided excellent constraints on samples containing fluid inclusion populations suitable for bulk isotopic analysis. The samples analysed were carefully selected to have minimal deformational or fluid overprinting and are thus thought to be truly representative of Variscan fluid isotopic evolution in the area. Emphasis was placed on regional metamorphic and contact metamorphic/metamorphic fluids for which no direct stable isotope data currently exist, but the results also contribute to our understanding of the hydrologic systems operating during mineralisation in the Cornubian metallogenic province.

2. Summary of geology

The Palaeozoic terrain of southwest England forms part of the Rhenohercynian zone of the Variscan orogen. It comprises a series of Devonian basins formed during localized late Palaeozoic extension (Ziegler, 1982) subsequently subjected to compressional tectonism and low-grade metamorphism during the Carboniferous. Multiple intrusion events over a period of ~25 Ma in the Late Carboniferous to Early Permian (Chesley et al., 1993) resulted in the formation of the Cornubian granite batholith which marked the end of Variscan tectonism in the province.

The study area is situated on the west coast of the Lizard Peninsula in south Cornwall (Fig. 1). The coastline from Porthleven to Polurrian Cove provides a cross-section through marine, fan and slope
“flysch”-type sediments deposited in the Gramscatho basin during the Middle to Late Devonian and subsequently stacked into a series of thrust-nappes (Holder and Leveridge, 1986). The Lizard Complex ophiolite crops out in the south of the area (Fig. 1) and is thrust over sedimentary breccias of the Carne Formation. These are successively structurally underlain by the Portscatho Formation, which comprises turbiditic sandstones and shales of presumed Middle–Late Devonian age (Holder and Leveridge, 1986), and a transitional sequence characterised by intercalated sediments of both Portscatho and Mylor lithofacies including a significant proportion of olistostromes. At Cacastull Zawn, these rocks are faulted against the parautochthonous Mylor Slate Formation of late Famennian age (Wilkinson and Knight, 1989) which consists of dark grey-green slates with thin rhythmic silty or sandy beds. Intruded into the Mylor Slates are a series of dolerite sills.

The entire sequence was deformed during low-angle brittle–ductile shear and nappe emplacement \( (D_1) \) in the Lower Carboniferous with synchronous pumpellyite–actinolite-grade regional metamorphism at \( 320 \pm 10^\circ C \) and \( 3.2 \pm 0.3 \) kbar (Barnes and Andrews, 1981; Harvey et al., 1994). Progressive coaxial refolding (Rattey, 1980) and imbrication \( (D_2) \) occurred during continued Variscan shortening under more brittle conditions. This phase was accompanied by widespread quartz veining and minor retrogression in fault zones at temperatures down to \( \sim 270^\circ C \) and pressures of \( \sim 1200 \) bar (Harvey et al., 1994). Emplacement of components of the Cornubian batholith over the period \( \sim 300–275 \) Ma (Chesley et al., 1993), and notably the Tregonning Granite in the north of the study area dated at \( 280 \pm 4 \) Ma (Darbyshire and Shepherd, 1987), postdated the compressive phase in south Cornwall and resulted in the development of contact metamorphic facies. This was apparently synchronous with low-angle extensional shear with reactivation of compositional structures and refolding \( (D_3) \), e.g. Shail and Wilkinson (1994). Associated with the granites was the extensive Sn–W–Cu mineralisation for which the area is famous (e.g., Jackson et al., 1989). Subsequent higher-angle brittle extensional and strike-slip faulting were accompanied by low temperature fluid flow and Pb–Zn mineralisation (e.g., Sawkins, 1966; Alderton, 1978).

### 3. Fluid characterisation

Vein systems developed during the geological events described above were classified by Wilkinson (1990a). Relative ages were constrained by the relationship between veins and regionally-correlated structures and deformation fabrics (see Rattey, 1980). Veins formed during each phase of deformation \( (D_1, D_2, D_3, \text{post}-D_3) \) occur in a range of structural and lithologic settings (Table 1). Detailed textural and microthermometric studies were carried out on 60 quartz vein samples to determine the origin and \( P–V–T–X \) characteristics of the fluid inclusions present (Wilkinson, 1990a, b).

Fluid inclusions in 100–200-\( \mu \)m-thick, doubly-polished wafers were analysed using a Linkam™ heating–freezing stage according to established procedures (e.g., Shepherd et al., 1985b). Salinity estimates for dilute aqueous inclusions were derived from final ice
Table 1
Vein classification in study area

<table>
<thead>
<tr>
<th>Vein type</th>
<th>Structural setting</th>
<th>Sulphide mineralisation</th>
<th>Relation to regional tectonics</th>
<th>Fluid type</th>
</tr>
</thead>
<tbody>
<tr>
<td>V1B</td>
<td>quartz in neck of boudin contained within S1, slaty cleavage</td>
<td>none observed</td>
<td>syn-D1</td>
<td>F1</td>
</tr>
<tr>
<td>V1F</td>
<td>quartz vein in outer arc of F1 fold and geometrically related to fold axial plane</td>
<td>none observed</td>
<td>syn-D1</td>
<td>F1</td>
</tr>
<tr>
<td>V1T</td>
<td>quartz vein on D1 thrust or in tectonic or shear veins associated with D1 thrust</td>
<td>minor pyrite</td>
<td>syn-D1</td>
<td>F1</td>
</tr>
<tr>
<td>V2T</td>
<td>quartz vein in outer arc of F2 fold and geometrically related to fold axial plane</td>
<td>minor pyrite</td>
<td>syn-D2</td>
<td>F2</td>
</tr>
<tr>
<td>V3S</td>
<td>quartz vein on D2 thrust or in tectonic or shear veins associated with D2 thrust</td>
<td>pyrite + late chalcopyrite</td>
<td>syn-D2</td>
<td>F2</td>
</tr>
<tr>
<td>V3o</td>
<td>extensional quartz vein related to D2 shear</td>
<td>none observed</td>
<td>syn-D3</td>
<td>F3</td>
</tr>
<tr>
<td>V3s</td>
<td>quartz vein in geometric array hosted by slate, contained within or crosscutting S3 crenulation cleavage in slate</td>
<td>none observed</td>
<td>syn-/post-D3</td>
<td>F3</td>
</tr>
<tr>
<td>V3o</td>
<td>quartz vein in geometric array hosted by greenstone sill, crosscutting S3, crenulation cleavage in slate (where observed)</td>
<td>arsenopyrite + galena + pyrite + chalcopyrite + sphalerite</td>
<td>syn-/post-D3</td>
<td>F3</td>
</tr>
<tr>
<td>V4N</td>
<td>quartz veining in high-angle brittle extensional faults</td>
<td>pyrite</td>
<td>post-D3</td>
<td>F4</td>
</tr>
<tr>
<td>V4w</td>
<td>quartz veining in subvertical strike faults and associated extensional veins</td>
<td>galena + sphalerite + chalcopyrite + pyrite + arsenopyrite</td>
<td>post-D3</td>
<td>F5</td>
</tr>
</tbody>
</table>

After Wilkinson (1990a).

melting temperatures based on the system NaCl–H2O using the regression of Bodnar (1993), and those for hypersaline aqueous inclusions in some V3 samples from halite dissolution temperatures (Sterner et al., 1988). The latter are minimum salinity estimates since halite dissolution generally occurred in the vapour-absent field (Bodnar, 1992). For CO2-bearing inclusions in V3 samples, clathrate melting temperatures were used to estimate salinity (Diamond, 1992). Homogenisation of the liquid and vapour phases generally occurred by disappearance of the vapour phase except for some CO2-rich inclusions in V3 samples (Wilkinson, 1990b).

The salinity and homogenisation temperature data obtained show that there were generally no significant differences between fluids trapped in different structural settings within one orogenic stage, but that characteristic microthermometric differences did exist between fluids at different stages of the orogeny (Fig. 2). This indicates relative homogeneity of fluids on a local scale at any one time, but that the fluid systems evolved during the course of the orogeny. The exceptions to this are the V4 veins which contain two distinct fluids (Fig. 2), indicating local heterogeneity during the post-orogenic stage.

Based on the fluid inclusion data, five fluid types were defined (Table 2), each characteristic of a particular stage of orogenic evolution. Determination of the major-element composition of the five fluid types by bulk decrpetation–inductively coupled plasma–atomic emission spectrometry (D–ICP–AES) analysis of fluid inclusions [see Rankin et al. (1993) for description of method] confirmed this subdivision (Fig. 3), with F1 and F2 fluids having overlapping chemical composi-
Regional peak (F1) and later retrogressive (F2) metamorphic fluids were H₂O-rich, with minor amounts of other volatiles, predominantly CO₂. The principal solute was NaCl, with lesser amounts of KCl and CaCl₂ (Fig. 3) and there was a slight increase in Ca content and total salinity from F1 to F2 (Figs. 2 and 3). Microthermometric properties are comparable with those reported for Variscan metamorphic fluids in Germany (Behr et al., 1987; Hein and Behr, 1991). Alkali metal compositions are consistent with the fluids being close to equilibrium with the dominant regional metamorphic mineral assemblage (quartz + albite + K-mica ± paragonite). Although classified separately, F1 and F2 fluids show similar microthermometric and chemical properties and probably represent a continuum in fluid evolution, consistent with the progressive nature of the compressional deformation. Based on mineral assemblage constraints, chlorite geothermometry and fluid inclusion isochores, fluid temperatures at this stage were estimated to decrease from 320°C to 270°C and fluid pressures from 3.2 to 1.2 kbar (see Table 3 and Harvey et al., 1994).

3.2. F3 fluids

Fluids active in the contact aureole of the Cornubian batholith during granite emplacement (F3) were complex H₂O-CO₂-N₂-CH₄ mixtures which were undergoing phase separation at the time of vein formation (Wilkinson, 1990b). Phase separation resulted in the development of extremely heterogeneous fluids with highly variable salt content and density. The optical identification of NaCl cubes in hypersaline inclusions indicates the dominance of NaCl but the F3 fluids also contain significant K and Ca and are generally enriched in these elements relative to the F1–F2 fluids (Fig. 3).

3.3. F4 fluids

Subsequent to granite emplacement, low-temperature, very low-salinity H₂O-rich fluids (Fig. 2) circulated in steep, ENE–WSW-trending brittle extensional faults in the section. These contain lower relative K concentrations than the syn-orogenic fluids (Fig. 3), reflecting in part their low-temperature origin. The exact age of the extensional faults is uncertain; they clearly postdate D₃ and focused fluids similar in composition to late-stage mineralising fluids in the granites.
Table 2
Summary of fluid types and associated geological events

<table>
<thead>
<tr>
<th>Fluid Relation to regional tectonics</th>
<th>Approximate age (Ma)</th>
<th>Geological events</th>
<th>Major components of fluid</th>
<th>Temperature(^a) ((^\circ)C)</th>
<th>Salinity (eq wt% NaCl)</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1 syn-D(_1)</td>
<td>360</td>
<td>peak low-grade regional metamorphism, nappe emplacement</td>
<td>H(_2)O–NaCl(–CO(_3))</td>
<td>290–330</td>
<td>1–4</td>
</tr>
<tr>
<td>F2 syn-D(_2)</td>
<td>?320</td>
<td>brittle thrusting and retrogression</td>
<td>H(_2)O–NaCl(–CO(_3))</td>
<td>265–315</td>
<td>1–7</td>
</tr>
<tr>
<td>F3 syn-D(_3)</td>
<td>?300–290</td>
<td>granite emplacement, contact metamorphism, early W–Sn-oxide mineralisation</td>
<td>H(_2)O–CO(_2)–N(_2)(–CH(_4))–NaCl,KCl, CaCl(_2), FeCl(_2)</td>
<td>200–440</td>
<td>1–46</td>
</tr>
<tr>
<td>F4 post-D(_1)</td>
<td>280–260</td>
<td>brittle extensional faulting, basin development, Sn–Cu–Zn–Pb-sulphide mineralisation</td>
<td>H(_2)O(–NaCl)</td>
<td>150–250</td>
<td>0–6</td>
</tr>
<tr>
<td>F5 post-D(_3)</td>
<td>270–230</td>
<td>brittle strike–slip faulting related to basin development, Pb–Zn mineralisation</td>
<td>H(_2)O–NaCl–CaCl(_2)</td>
<td>110–150</td>
<td>23–28</td>
</tr>
</tbody>
</table>

After Wilkinson (1990a, b).

\(^a\)See Table 3 for fluid temperature constraints.

...and wallrock brecciation, and structural relationships (Shail and Wilkinson, 1994) are consistent with a cogenetic origin. This interpretation implies that F5 brine migration occurred during late- to post-Variscan basin development in agreement with existing fluid inclusion data (Shepherd and Scrivener, 1987) and recent geochronology. Sm–Nd dating of fluorite and Rb–Sr dating of fluid inclusions in quartz from the “cross-course” vein paragenesis elsewhere in the province gave ages of 259–267 Ma (Chesley et al., 1993) and 236 ± 3 Ma (Scrivener et al., 1994), respectively. Based on homogenisation temperature data, and assuming hydrostatic pressure and depths of < 5 km, fluid temperatures are estimated at 110–150\(^\circ\)C (see Table 3) and fluid pressures < 500 bar.

3.4. F5 fluids

The Pb–Zn mineralised NNW–SSE-trending strike-slip faults and associated N–S extensional veins (“cross-courses”; see Jackson et al., 1989) in the study area were associated with the flow of distinctive low-temperature, high-salinity, NaCl–CaCl\(_2\)-dominated aqueous fluids (Figs. 2 and 3). Such fluids have also been described from other Variscan terrains, such as the Oberpfalz, Schwarzwald, Harz and Rheinisches Schiefergebirge regions of Germany (Behr and Gerler, 1987). The distribution of homogenisation temperature–salinity data (Fig. 2) are interpreted as a mixing trend, implying that exotic F4 and F5 fluids migrated into the area synchronously and were able to interact (cf. Tornos et al., 1991). The similar textures of both types of V4 veins, consisting of zoned, drusy quartz

4. Fluid inclusion populations and sample selection

For the stable isotopic analysis of inclusion fluids, a bulk sample of vein material is used. The validity of any bulk analysis of fluid inclusions is critically dependent on the nature of the fluid inclusions present in the sample. As a result, thorough investigation of the fluid inclusion populations present must be carried out...
to eliminate or minimise bias introduced by analysis of multiple generations of inclusions, in addition to providing the temperature and salinity information necessary for the interpretation of isotopic data. Such a study forms an essential prerequisite to chemical and isotopic analysis of inclusion fluids, and is often neglected in studies involving large numbers of samples where a statistically significant scatter of data is the object. Here, we have concentrated on the careful selection of samples for bulk analysis based on extensive petrographic and microthermometric studies. Thus, the selection of a limited number of what we believe to be truly representative samples forms a sound basis for the stable isotopic analyses of inclusion fluids and vein material.

Peak metamorphic vein quartz containing F1 primary and pseudosecondary fluid inclusions often also contains F2-type fluid inclusions due to significant fluid flow during the D2 deformation event (see Fig. 2). Fortunately, extreme F2 overprinting was found to be restricted to the leading edge of the allochthon (Blue Rock–Caca-stull Zawn; see Fig. 1) so that samples containing essentially only F1 fluid inclusions could be selected from other parts of the sequence. This effectively limited the suitability of F1 samples to those from the south of the area (Fig. 1). F1 fluid inclusion data are consistent with their origin during peak metamorphic conditions as deduced from independent thermo-barometric constraints (see Harvey et al., 1994, their fig. 3). Textural and cathodoluminescence studies of these samples could not resolve significant recrystallisation nor evidence for multiple generations of quartz, providing additional support for the hypothesis that the original isotopic signature of the F1 fluid is likely to have been preserved.

Due to the significant fluid flow during D2 and the extensive development of quartz veins at this stage, it was easier to find samples dominated by F2-type fluid inclusions. However, F3 fluids were identified in some samples (see Fig. 2), particularly in the vicinity of structures showing evidence of extensional reactivation during D2 (e.g., Blue Rock), and in samples from within the contact aureole of the Tregonning granite. The F2 samples selected for analysis were those showing no evidence of an F3 overprint (samples J48BR, D2BR2, D2bPC) or only a volumetrically insignificant overprint with <5% F3 inclusions (J99BR, J125LB). Cathodoluminescence studies showed that overprinted quartz could be identified; some dull purple-brown luminescing F2 samples were crosscut by brighter, bluish luminescing micro-veinlets containing F3 fluid inclusions.

D3 veins are dominated by F3-type fluid inclusions but overprinting by later fluids was recognised in some samples. Samples with significant overprinting were avoided. Another problem arose due to the phenomenon of fluid immiscibility demonstrated in these veins (Wilkinson, 1990b) which resulted in the synchronous trapping of two different types of fluid: a CO2-rich phase and an H2O–salt-rich phase. However, if representative trapping of the two fluids occurred, the isotopic analyses will reflect the isotopic composition of the bulk (unmixed) fluid.

The late (F4, F5) veins in the section are subject to minimal overprinting due to the lack of later significant tectonic or thermal perturbations. Overprinting that is observed is highly localised to the vicinity of brittle fault structures, so that F4 type fluid inclusions are
occasionally observed in microfractures in F5 veins (see Fig. 2) and vice versa. However, these were not volumetrically significant.

5. Sample preparation and analysis

Approximately 30-g samples of vein quartz were crushed and sieved to obtain the 0.5–2.0-mm fraction. Grains were then hand-picked under a binocular microscope to remove microcrystalline and contaminant particles. The clean quartz separates were boiled for 24 hr under reflux in 10% nitric acid, which was changed twice. Samples were then boiled in distilled, deionised water for 1 hr to remove acid contamination, thoroughly rinsed and air dried at 98°C.

Stable isotopic determinations were carried out using the methods described by Jenkin et al. (1994). The hydrogen isotopic composition of inclusion water was determined on water cryogenically separated from the gases released by decrepitation of 1–2 g of pure quartz separate. A mercury-piston inlet system to the mass spectrometer enabled routine measurement of the isotopic composition of the 20–140 μmol of hydrogen produced by reduction of this water. Oxygen isotopic compositions of the quartz vein samples were determined using conventional procedures and the isotopic composition of the fluid estimated by application of the quartz–water fractionation equation of Matsuhisa et al. (1979). Since the trapping temperatures were, in some cases, not well known, the most pessimistic (widest ranges) were used to indicate the degree of uncertainty in the δ18O(H2O) values. This leads to uncertainties in δ18O(H2O) of up to ±3.2‰, such that the analytical error of ±0.1‰ is negligible. Some fluids, notably F3 fluids, contain appreciable oxygen in CO2 as well as in H2O. All our estimated fluid δ18O-values are for the aqueous component; the CO2 component and bulk fluid will have higher δ18O (see Jenkin et al., 1994, p.437). Small corrections (up to +1.8‰) were applied to some of the δ18O data (see Table 3) because of the effects of dissolved salt on the quartz–water fractionation (Horita et al., 1995). These corrections and the choice of the fractionation equation of Matsuhisa et al. (1979) make little difference to the interpretation of the data, particularly with regard to the general trends observed. The δD-values are determined directly on the fluid so that the analytical error of ±2‰ is regarded as the level of uncertainty.

The estimated fluid compositional ranges for each of the samples analysed are presented in a δ18O–δD plot (Fig. 4). It is apparent that each of the five fluid classes defined show limited isotopic compositional overlap. This means that it may be possible to distinguish fluids flowing through the same segment of crust (i.e. the same host rocks) at different stages of orogenesis by their oxygen and hydrogen isotopic compositions.

6. Results and discussion

Results of the determinations of δD(H2O) and δ18O(quartz) are reported in Table 3. Based on estimated fluid trapping temperatures from a range of geothermometers (see Table 3), δ18O(quartz) was converted to δ18O(H2O) using the quartz–water fractionation equation of Matsuhisa et al. (1979). Since the trapping temperatures were, in some cases, not well known, the most pessimistic (widest ranges) were used to indicate the degree of uncertainty in the δ18O(H2O) values. This leads to uncertainties in δ18O(H2O) of up to ±3.2‰, such that the analytical error of ±0.1‰ is negligible. Some fluids, notably F3 fluids, contain appreciable oxygen in CO2 as well as in H2O. All our estimated fluid δ18O-values are for the aqueous component; the CO2 component and bulk fluid will have higher δ18O (see Jenkin et al., 1994, p.437). Small corrections (up to +1.8‰) were applied to some of the δ18O data (see Table 3) because of the effects of dissolved salt on the quartz–water fractionation (Horita et al., 1995). These corrections and the choice of the fractionation equation of Matsuhisa et al. (1979) make little difference to the interpretation of the data, particularly with regard to the general trends observed. The δD-values are determined directly on the fluid so that the analytical error of ±2‰ is regarded as the level of uncertainty.

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6.1. F1–F2 fluids

Peak and post-peak regional metamorphic fluids have the highest δ18O- and δD-values determined in this study, similar to those estimated for low-temperature metamorphic fluids in equilibrium with typical marine sediments (Sheppard, 1986) and to those for inferred metamorphic/formation waters from other Variscan terrains (e.g., Negga et al., 1986).

Primmer (1985) estimated the oxygen and hydrogen isotopic composition of southwest England (north Cornwall) metamorphic water based on illite and chlorite analyses and the application of appropriate fractionation factors for the temperature range 150–450°C. The calculated isotopic compositions for F1 and F2 fluids reported here are consistent with the higher-temperature sector of Primmer’s “metamorphic water” field (see Fig. 4). Since our data include δD determined directly from fluid inclusions and incorporate more precise fluid temperature estimates and the effect of dissolved salts on mineral–fluid fractionation, we believe that the F1/F2 data provide the best constraints to date on the oxygen and hydrogen isotopic composition of
Table 3
Measured and estimated stable isotopic compositions of fluids

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Fluid type</th>
<th>Modal salinity (wt% NaCl)</th>
<th>Temperature (°C)</th>
<th>δD (H₂O) (‰ vs. SMOW)</th>
<th>δ¹³C (CO₂) (‰ vs. PDB)</th>
<th>δ¹⁸O (H₂O) (‰ vs. SMOW)</th>
<th>δ¹⁸O (CO₂) (‰ vs. SMOW)</th>
</tr>
</thead>
<tbody>
<tr>
<td>J53JR</td>
<td>F1</td>
<td>2.3</td>
<td>315</td>
<td>-18</td>
<td>+18.2</td>
<td>+11.8</td>
<td>+11.9</td>
</tr>
<tr>
<td>J1PB</td>
<td>F1</td>
<td>2.8</td>
<td>316</td>
<td>-10</td>
<td>+17.8</td>
<td>+11.5</td>
<td>+11.6</td>
</tr>
<tr>
<td>J138GW</td>
<td>F1</td>
<td>2.5</td>
<td>330</td>
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Temperatures given in °C; n.d. = not determined.

*Temperature range is mean ±1σ for F1 fluids as estimated using the chlorite geothermometer of Walshe (1986) on vein chlorites; temperatures are consistent with D₁ pumpellyite–actinolite assemblages, vitrinite reflectance data and isochores for F1 fluid inclusions [see Wilkinson (1990a) for details].

*Temperature ranges as determined by the chlorite geothermometer of Walshe (1986) for vein chlorites coexisting with quartz. F1 temperatures are consistent with D₁ pumpellyite–actinolite assemblages, vitrinite reflectance data and isochores for F1 fluid inclusions; F2 temperatures are consistent with trapping of F2 fluid (~4 mole% CO₂–H₂O–3.1 eq wt% NaCl) in the one-phase region [data from Gehrig et al. (1979)], and isochores for F2 fluid inclusions; F3 temperatures consistent with homogenisation temperatures of inclusions trapped on the solvus [see Wilkinson (1990a) for details].

*Modal salinity for fluid group.

*Temperature range mean ±1σ for F2 fluids as estimated by chlorite geothermometry.

*Temperature range estimated from F3 fluid inclusion homogenisation temperatures for coexisting H₂O- and CO₂-rich inclusions trapped on the solvus where homogenisation temperature is equivalent to trapping temperature.

*Replicate analyses.

*Temperature range estimated from homogenisation temperature range with maximum +20°C correction to take into account trapping at a depth of up to 5 km, equivalent to hydrostatic pressures of ≤500 bar.

*Modal salinity based on H₂O–NaCl–CaCl₂ system.
Fig. 4. $\delta^{18}$O–$\delta$D plot of the isotopic compositions of fluids from south Cornwall. Crosses represent limits of uncertainty in isotopic determinations for each sample; $\delta$D uncertainty is analytical precision, $\delta^{18}$O uncertainty is dominated by vein formation temperature constraints.

1 Cornubian magmatic water as defined by Sheppard (1977) and Jackson et al. (1982).
2 North Cornwall Variscan "metamorphic water" as defined by Primmer (1985).
3 Pyrenees, France (Hercynian) "metamorphic" water, interpreted to be of seawater origin (Wickham and Taylor, 1985).
5 Composition of Cornubian (300–100 Ma) meteoric waters as estimated by Jackson et al. (1982).

Dashed lines indicate trends for modern basin waters from North America: M = Michigan, U.S.A.; A = Alberta, Canada (Clayton et al., 1966); C = California, U.S.A. (Sheppard, 1984).

low-grade regional metamorphic fluids in the province.

Relatively high $\delta$D-values ($>20\%$o) are indicated for syn-metamorphic water in a number of higher-grade, internal portions of the European Variscides based on analyses of micas (Frey et al., 1976; Hoernes and Friedrichsen, 1980; Wickham and Taylor, 1985; see Fig. 4). High $\delta$D-values and extreme isotopic homogeneity of mineral compositions were interpreted by Wickham and Taylor (1985) as evidence of deep ($\sim10$ km), pre-/syn-metamorphic penetration of seawater into the Trois Seigneurs Massif, Pyrenees, France.

In the present study, thermobarometric constraints from peak metamorphic mineral assemblages and fluid inclusions indicate depths of $\sim13$ km during $D_1$ (Harvey et al., 1994), assuming lithostatic pressure conditions. This would imply average geothermal gradients of $20$–$25^\circ$C km$^{-1}$, as are inferred for other thrust belts (e.g., Jamieson and Beaumont, 1989). The internal consistency of these observations, combined with the vein structural relationships seem to rule out sub-lithostatic fluid pressures during $D_1$. In addition, $F_1$ fluids appear to be close to chemical equilibrium with their host rocks making infiltration of large volumes of externally-derived fluids implausible. Syn-metamorphic deep penetration of surface water is thus considered to be unlikely in southwest England and we interpret our $\delta$D data as being representative of a peak, low-grade regional metamorphic fluid, locally-derived by prograde devolatilisation reactions. This may mean that it is unnecessary to invoke infiltration of exotic waters to account for high $\delta$D-values in other regional metamorphic terrains.
The slight shift in isotopic composition from F1 to F2 appears to be closely correlated with changes in fluid chemistry (compare Figs. 3 and 4) and is in accord with an evolutionary process. The trend of increasing salinity in F2 fluids and the minor propylitic alteration associated with them can both be explained by increasing (but slight) fluid-rock disequilibrium and consequent retrogressive hydration reactions. On this basis, the F2 fluids are interpreted as being residual metamorphic fluids released from more internal parts of the nappe complex during the waning stages of compressional tectonism. Although the possibility exists of late orogenic infiltration of O-shifted meteoric water (e.g., Jenkin et al., 1994), which could account for the isotopic signature of the F2 fluids, the structural relationships of the V2 veins indicate formation during compression with fluid pressures close to lithostatic. Hydrostatic pressures at the late stages of D2 would imply that the area was still at depths in excess of 10 km and that little or no erosional unroofing of the nappe pile had occurred during compression (see Harvey et al., 1994). This is considered to be geologically unrealistic.

The 6%2C-values for CO2 present in F1/F2 fluids are in the range -14.1 to -6.4‰ (Table 3). These data are consistent with an internal derivation of CO2 from a combination of organic carbon and carbonate present in the sedimentary sequence; no external source is required.

In summary, we believe that the isotopic compositions determined for F1–F2 fluids are representative of low-grade metamorphic fluids in the province and are consistent with the limited data from other Variscan terrains. The fluids were internally derived and generally close to chemical and isotopic equilibrium with their host rocks.

6.2. F3 fluids

The stable isotopic composition of the F3 aqueous fluids cannot be interpreted unambiguously in terms of a single fluid source. δ18O-values span the range estimated for regional metamorphic fluids in this study and Cornubian magmatic water as defined by Sheppard (1977) and Jackson et al. (1982). δD-values are intermediate between regional metamorphic fluids and magmatic fluids, with a small overlap of both fields (Fig. 4).

Chemical analyses of F3 fluids (Wilkinson, 1990b) showed that F3 fluids are also chemically intermediate between regional metamorphic fluids and the expected composition of magmatic fluids (see Fig. 3). On the basis of this, it was suggested that F3 fluids might be the result of either: (1) modification of a magmatic fluid by reaction with contact aureole rocks, or (2) mixing of a magmatic fluid with fluid locally derived in the contact aureole by devolatilisation reactions, or a combination of both processes. The first process would lead to a range of chemical compositions approaching that of equilibrium with the local rocks, over a range of temperatures governed by position within the contact aureole. Since the regional metamorphic (F1/F2) fluids are inferred to have been close to equilibrium with the same rocks at the lower end of the temperature range of the F3 fluids, a range of compositions between magmatic and the F1/F2 composition might be expected. Alternatively, mixing of a magmatic fluid with a contact metamorphic fluid at equilibrium with the aureole rocks (which, for the reason given above, would be similar in composition to the F1/F2 fluids), would also result in a range of compositions intermediate between magmatic and 'equilibrium metamorphic'.

The increasing δD with distance from exposed granite (compare Table 3 and Fig. 1) can be accounted for by a purely magmatic aqueous component exchanging with country rocks in the contact aureole. The trend can be produced by an infiltrating fluid because as it travels further and further from its source, the effective water/rock ratio continually decreases, which results in a positive δD shift (as long as water/rock ratios are generally low). Equilibration of a magmatic fluid with the Mylor Slates (bulk δ18O = +13.4‰, δD ≈ -58‰; Sheppard, 1977) could only result in a small shift in δ18O of <3‰, either in a positive sense at temperatures of 400–500°C or in a negative direction at lower temperatures. A significant positive shift in δD of up to 30‰ could occur if water/rock ratios were significantly less than unity.

However, due to the uncertainty in the F3 isotopic compositions, particularly in δ18O (Fig. 4), and the potential range in composition of magmatic and metamorphic fluids, the F3 data can be equally well explained by mixing of magmatic and contact metamorphic fluid. If a simple mixing model is considered (using average magmatic and F1 δ18O-and δD-values)
the mixed fluid would have had to contain 20–75% magmatic fluid. A similar calculation using the chemical data (Fig. 3) and the magmatic fluid composition of Bottrell and Yardley (1988) suggests mixtures containing 10–70% magmatic fluid. The uncertainty on these estimates is obviously large; nevertheless, the two sources of data give similar results and are consistent with a mixing hypothesis.

There is independent evidence that most of the volatile (CO$_2$–N$_2$–CH$_4$) components, and possibly also some of the aqueous component, of the F3 fluids were derived by devolatilisation of the Mylor Slates during granite emplacement (Wilkinson, 1991). This hypothesis is in accord with the $^{18}$O$_{(CO_2)}$ values of $-11.9$ to $-9.0\%$ obtained from two F3 samples (Table 3) which indicate that the CO$_2$ may be of organic origin. Unfortunately, the carbon isotopic signature of the Cornubian magmas is not known and it is possible that contamination of the melts by significant amounts of organic carbon-rich pelite, as has been suggested by a number of workers (see Jackson et al., 1989 and references therein), resulted in magmatic CO$_2$ of unusually low $^{13}$C. This is perhaps suggested by the data of Lin (1989) who obtained $^{13}$C$_{(CO_2)}$ values of $-11.7$ to $-8.8\%$ from fluid inclusions in pegmatites and sheeted veins. However, these data can also be explained by contamination of magmatic CO$_2$ by the involvement of non-magmatic volatiles in fracture systems close to the granite margin, as described for the Tanco pegmatite (Manitoba, Canada) by Thomas and Spooner (1988). Either way, it appears extremely likely that components ultimately derived from pelitic “aureole-type” rocks were involved in the F3 fluids, either directly via devolatilisation of pelite at a relatively high level in the crust or by assimilation of pelite into the melt and subsequent partitioning of pellet-derived components into a magmatic aqueous phase. The relative importance of these two processes remains unanswered.

Comparison of F3 isotopic compositions with previous work is difficult, primarily because most studies have not taken into account the effect of high concentrations of dissolved salts which may be present in aureole/metasomatic-type fluids. Our calculations indicate that this may have resulted in underestimates of $^{18}$O by up to 2$\%$ for high-salinity fluids (25 wt% salts) at temperatures above 400$^\circ$C (see Table 3). The work of van Marcke de Lummen (1985) suggested that skarn-forming fluids in the Land’s End district had $\delta^D$-values in the range $-38$ to $-6\%$ and $\delta^{18}$O-values in the range $+9$ to $+11\%$, assuming temperatures of 600–500$^\circ$C. These data show significant overlap with our F3 field (Fig. 4). Alderton and Harmon (1991) used the data of van Marcke de Lummen to infer magmatic $\delta^D$ compositions as high as $-15\%$; however, the solely magmatic nature of the skarn-forming fluids cannot be assumed and the close comparison in $\delta^D$-values with our F3 fluids, which probably contain an aureole-derived component, leads us to suspect that such a component is also present in the skarn fluids. We prefer to use an upper limit for $\delta^D$ of $\sim-40\%$ for purely magmatic fluids in Cornubia (e.g., Fig. 4) following the work of Sheppard (1977) and Jackson et al. (1982).

In conclusion, the origin of the chemical and isotopic composition of the F3 fluids remains unclear. It is probable that they contain a magmatic aqueous component and it is also probable that they contain CO$_2$, N$_2$ and CH$_4$ derived from the contact aureole. It is possible that they also contain some H$_2$O derived from the aureole rocks. We think it likely that both infiltration and equilibration of a magmatic aqueous fluid and mixing with locally-derived fluid occurred. The origin of the skarn-forming fluids described by van Marcke de Lummen (1985) is subject to the same uncertainty as our F3 fluids.

The significant overlap of the isotopic compositional field of the F3 fluids with the field for Sn–W mineralising fluids defined by Alderton and Harmon (1991) is significant (see Fig. 4). Rather than wholly magmatic fluids or magmatic–meteoric mixtures, the early (primarily Sn–W) mineralising solutions may contain an aureole-derived component, with the pelitic sediments and volcanics of the Mylor Slate Formation forming a potential reservoir of ore-forming elements. In particular, the extremely high-salinity aqueous fluids produced by phase separation in the aureole could have transported high concentrations of Sn, Cu, Pb and Zn as chloro-complexes (Wilkinson, 1990b). The clear association between zones of Cu and As mineralisation and Cu–As-rich basic intrusives (Jackson et al., 1989) can be explained by a local mobilisation process of this nature.

The similarity between F3 fluids and fluids associated with mineralisation in other Variscan Sn–W deposits (e.g., Kelly and Rye, 1979; Mangas and Arri-
bas, 1987) supports the hypothesis that fluids of this type are an integral part of such hydrothermal systems. In this context, the advantage of analysing samples from outside the main influence of granite-hydrothermal activity is made clear. Although only slightly mineralised, V3 veins do contain a typical suite of sulphide minerals (Table 1), and give a clear indication of the nature (chemical and isotopic) of mineralising fluids present on the flanks of the granite batholith, at an early post-emplacement stage. In contrast, the picture becomes blurred where fluid inclusions are analysed from mineralised veins within the granites (e.g., Alderton and Harmon, 1991) which almost invariably contain multiple fluid inclusion populations (Jackson et al., 1982).

### 6.3. F4 fluids

The low-temperature fluids circulating in E-W-trending faults in the area have distinct, low $\delta^{18}O$-values and are of low salinity. These features are consistent with an origin as meteoric waters which have been heated and have undergone chemical and isotopic exchange with country rocks. Although this hydrothermal activity postdates consolidation of the Cornubian batholith, it can be explained by the significant radiogenic heat produced by the high heat producing granites driving convection of surface waters in fracture systems (e.g., Sams and Thomas-Betts, 1988) continuously reactivated in an extensional tectonic regime. This is widely accepted as a process which played a role in mineralisation in the province (e.g., Jackson et al., 1989), although the relative importance of meteoric circulation as a mineralising process is still a subject of debate. The F4 fluids analysed in this study are chemically and isotopically comparable with the dilute "end-member" of the fluids responsible for the majority of the cassiterite-polymetallic sulphide-chlorite mineral paragenesis in the orefield (Jackson et al., 1982; Shepherd et al., 1985a; Alderton and Harmon, 1991; Farmer and Halls, 1993; Shail and Wilkinson, 1994), and are probably comparable with the Cu-Pb-Zn fluids of Alderton and Harmon (1991) (see Fig. 4). This supports the assertion that meteoric fluids did not become important until the Sn-polymetallic sulphide stage of mineralisation (e.g., Farmer and Halls, 1993). Our F4 data overlap with the data for Cu-Pb-Zn mineralised veins of Alderton and Harmon (1991); the extension to lower $\delta^{18}O$ is probably largely due to the fact that our samples are distal from the granite and formed at lower temperatures and also, perhaps, a lack of any magmatic input. The very dilute nature of the fluid in sample J78PS, and the fact that it has one of the lowest recorded $\delta^{18}O$ signatures in the region, suggest that the fluid inclusions in this sample represent the closest approximation to Cornubian palaeometeoric water yet measured.

### 6.4. F5 fluids

The low-temperature, high-salinity fluids responsible for Pb-Zn mineralisation in the study area are isotopically distinct, having low $\delta^D$- and $\delta^{18}O$-values, comparable with modern basinal fluids from North America (Fig. 4). A basinal origin for F5 (cross-course mineralising) fluids was first suggested because of the low temperatures, high salinities and high Ca contents indicated by microthermometry (Alderton, 1978). More recent detailed chemical (Gleeson and Wilkinson, 1993), isotopic (Alderton and Harmon, 1991) and geochronological data (Scrivener et al., 1994) support this interpretation. The reason for the higher $\delta^D$-values obtained by Alderton and Harmon (1991) is not clear; they may have analysed mixed F4/F5 populations from samples proximal to the granites or the data may just indicate that significant local heterogeneity exists (our data clearly indicate a spread of at least 30%0 within a single vein system).

Basinal fluid isotopic compositions are often related to local meteoric water compositions (e.g., Clayton et al., 1966) and have low $\delta^D$-values in high-latitude basins. A problem with the low $\delta^D$-values for F5 fluids is that there was no known local meteoric water of corresponding $\delta^D$ available during the Permo-Triassic. Due to the low latitude at that time meteoric $\delta^D$-values are thought to have been relatively high; Jackson et al. (1982) inferred values in the range $-14$ to $-3\%0$, although our F4 data suggest slightly lower values of $-25$ to $-21\%0$ (see Fig. 4). Similar mismatches in fluid $\delta^D$ between basinal fluids and local meteoric fluids have been observed in a number of other studies of ancient basins (Ghazban et al., 1991; Fallick et al., 1993; Munoz et al., 1994). Explanations of the low $\delta^D$-values in these studies include the incorporation of organic fluids, or exchange with organic material or hydrous minerals at low temperature. In southwest
England, Permo-Triassic basinal brines could have had their δD lowered by interaction with organic matter in the Palaeozoic basement or within the Permo-Triassic sediments themselves. An alternative hypothesis is that the ultimate source of low-δD fluids was an area of high altitude (cf. Turpin et al., 1990) located to the southeast of the Cornubian peninsula, perhaps an elevated Armorican massif (e.g., Matte, 1991) maintained as a positive feature during the Permo-Triassic. The actual δD-value of meteoric precipitation at any one place will depend on a number of factors including altitude (Yurtsever and Gat, 1981). However, an estimate of the altitude of the terrain required for precipitation with a given δD may be obtained by assuming an average sea level temperature of 15–20°C and a decrease of temperature with altitude of ~6.5°C km⁻¹, coupled with a relationship between temperature and δ¹⁸O (Yurtsever and Gat, 1981, their fig. 24) and the equation for the meteoric water line. For meteoric water with δD of −55 to −80‰, altitudes of 1.0–2.4 km are obtained by this method, consistent with an upland massif source region. More detailed evaluation of this problem is required and further work is in progress.

7. Summary and conclusions

A combination of careful fluid inclusion investigation, sample selection and isotopic analysis has provided a powerful methodology for constraining models of fluid flow and mineralisation in an external part of the Variscan orogen. Five fluid types, distinguished on the basis of vein chronology and fluid inclusion microthermometry, flowed through upper Palaeozoic metasediments during and subsequent to the Variscan orogeny in south Cornwall. Each fluid type may be largely distinguished on the basis of major-element chemistry and stable isotopic signatures. We report the first direct δD analyses of Variscan low-grade metamorphic water and also metamorphic/metasomatic fluids from the contact aureole of the Cornubian batholith. Such data are critical for evaluating the role of fluids generated by mineral dehydration reactions in the geological evolution of southwest England. The remaining data emphasise the role of two exotic low-temperature fluids involved in late-stage mineralisation in the area, namely, convecting evolved meteoric water and basin-derived brines.

Close parallels exist between the chemical evolution of the fluids studied and their isotopic evolution, indicating that the two processes are closely linked in a range of orogenic fluid systems. This is believed to be due to rock-dominance of the chemical and isotopic make-up of the fluids in such environments. Post-orogenic fluids diverge from this norm, with exotic, disequilibrium fluids infiltrating a fractured crust.

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