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Statistical microthermometry of synthetic fluid inclusions in quartz during decompression reequilibration

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Abstract Fluid inclusion microthermometric data are often reported as homogenization temperature frequency histograms. Interpretation of such histograms for a single fluid inclusion assemblage (FIA) of non-reequilibrated fluid inclusions is usually straightforward and provides an accurate determination of the original density (*T*h) of that FIA. However, interpretation of such histograms for reequilibrated inclusions is more problematic. Decompression experiments using synthetic inclusions in natural quartz and conducted at 2–5 kbar and 600–700 °C with a maximum internal overpressure of 2 kbar indicate that histogram shape reflects the sample's *P*-*T* history. Our results further indicate that the mean, mode, range, standard deviation, extreme values, etc., all have a significance with respect to the *P*-*T* history of the sample. Thus, a mound-shaped, unimodal histogram with low range is indicative of a nearly isochoric cooling *P*-*T* path. A unimodal histogram that is slightly skewed to the right, and with a low standard deviation but high range, results from inclusion deformation in the plastic regime (high temperature/low strain rates). Fluid inclusions deformed plastically show no correlation between size and density. Histogram outliers should not be ignored and may be used to determine an isochore that passes close to the conditions of entrapment (minimum *T*h) or close to the final reequilibration conditions (maximum *T*h). The histogram mean *T*h value corresponds to an isochore that represents the internal overpressure (about 1 kbar) that can be maintained over geologic time by a majority of reequilibrated fluid inclusions. A multimodal histogram with high range and high standard deviation indicates inclusion brittle deformation (low temperature/high strain environments). Fluid inclusions deformed in a brittle manner show strong positive correlation between size and density. Histograms produced in the laboratory show many similarities to histograms for natural samples, offering the hope that laboratory results may be used to interpret *P*-*T* histories of natural samples.

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Introduction

Roedder (1984) summarized the three assumptions that must be invoked in order to use fluid inclusions for geothermobarometry. These are: (1) the inclusion traps a single, homogeneous phase; (2) the inclusion volume remains constant (isochoric); (3) nothing is added to, or lost from, the inclusion after trapping. An additional assumption is that no reactions have taken place within the fluid inclusion between the time of trapping and the time when the inclusion is examined (cf., Dubessy 1984). Roedder also noted that assumptions (2) and (3) are not always valid, and several dozens of papers (and hundreds of abstracts) have now been published on experimental studies that support this conclusion. These many studies serve to illustrate a very important point, namely, that *some* fluid inclusions in *some* samples *may* reequilibrate if the inclusions are subjected to *certain* *P*-*T*-*t* histories. We know, for example, that fluid inclusions often reequilibrate if they follow a *P*-*T* path that strays significantly from the *P*-*T* path for the original fluid isochore, and this often results in a wide range of fluid inclusion homogenization temperatures (densities) and corresponding isochores. It should also be noted that the converse is also true. That is, fluid inclusions in natural samples do not reequilibrate (or do so to a lesser extent) if the *P*-*T* path of the rocks closely follows the isochore for the fluid inclusions, as demonstrated clearly by Srikantappa et al. (1992). However, our understanding of how to apply results from laboratory studies to the interpretation of natural inclusions is still incomplete.

Homogenization temperatures of fluid inclusions in natural samples are usually displayed in temperature frequency histograms. However, little is known concerning the relationship between the shape of the histogram and the *P-T-t* history of the sample. Thus, one may ask why microthermometric data for a single fluid inclusion assemblage (FIA; Goldstein and Reynolds 1994) from natural samples sometimes show a broad range of homogenization temperatures with a single, distinct peak, while other samples produce a multimodal histogram of *T*h. Other questions relate to the significance of the histogram shape, the mean or the mode of the data, the range and variability of the data, and the minimum and maximum values (see e.g., Touret 1977). Also little attention has been given to the application of laboratory reequilibration data to natural samples for which the rate and duration of the reequilibration process are considerably different from conditions in the laboratory.

In order better to understand the relationship between inclusion statistical microthermometric data and geologic *P-T-t* history, we have reequilibrated synthetic fluid inclusions in natural quartz along a decompression *P-T* path similar to that often proposed for natural samples. We then conducted microthermometric analysis of a large number of randomly selected fluid inclusions from these samples. These results were then compared to microthermometric data from natural samples. As described in more detail below, the experimental and natural samples show many similarities, offering the hope that laboratory results may be used to interpret *P-T-t* histories of natural samples. The goal of this research is to develop a predictive tool that may be used to infer *P-T-t* histories from inclusion microthermometric statistical characteristics. It is important to note, however, that the results and conclusions presented below are only valid when applied to microthermometric data for inclusions from a single FIA for which all inclusions have experienced the same *P-T* history.

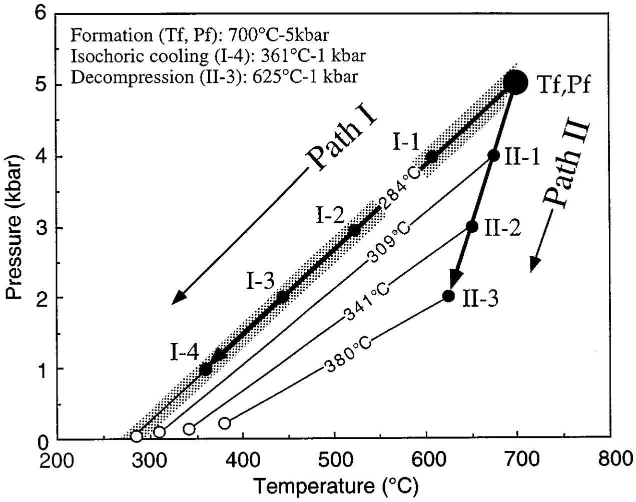


Fig. 1 *P-T* path simulating isochoric (PATH I) and nearly isothermal decompression (PATH II) during reequilibration of 10 wt% NaCl-H_2O synthetic inclusions in natural quartz. Iso-*T*h lines project from the conditions of re-equilibration (*filled circles*) to the liquid-vapor curve of 10 wt% NaCl-H_2O (*open circles*). *Filled circles* labeled 1–4 along the isochoric *P-T* path (PATH I) represent the conditions where the sample was held for 7 days each (total duration of the experiment was 35 days) during cooling along the isochore. The range in experimental *P-T* conditions corresponding to the observed range in homogenization temperatures after isochoric cooling is represented by the *shaded area* along PATH I. *Filled circles* II-1 and II-2 along Path II represent the *P-T* conditions where the sample subjected to incremental loading was held for 7 days before reaching the final reequilibration conditions represented by point II-3. The iso-*T*h lines for 10 wt% NaCl-H_2O solution were calculated using the equation of state of Bodnar and Vityk (1994)

Experimental procedures

In one set of experiments, an isochoric *P-T* path was simulated by trapping 10 wt% NaCl-H_2O inclusions in quartz at 700 °C and 5 kbar (*T*f, *P*f; Fig. 1) in two different cold-sealed vessels (for a description of the synthetic fluid inclusion technique see Bodnar and Sterner, 1987). After seven days, one quartz core (sample ITD-0) was removed from the pressure vessel and used as the "control" sample to determine the homogenization temperature for the original non-reequilibrated inclusions in subsequent experiments. (In the following discussion, "ITD" refers to isothermal decompression experiments, whereas "ISC" refers to isochoric cooling experiments). Another quartz sample, sample ISC-1, was gradually cooled to 361 °C and 1 kbar in approximately 80–90 °C increments along the isochore that passes through the formation conditions (PATH I, Fig. 1) . During isochoric cooling, sample ISC-1 was held at each new *P-T* condition (steps 1–4 along PATH I, Fig. 1) for 7 days, and the total duration of this experiment was 35 days.

A second experiment was conducted to examine the effect of *instantaneous loading* on inclusion reequilibration behavior. This experiment involved "rapid" application of effective pressure (sample ITD-3–30R, Table 1). With this approach, 10 wt% NaCl-H_2O synthetic fluid inclusions were formed at 700 °C and 5 kbar for 7 days (*T*f, *P*f; Fig. 1), and the inclusions were reequilibrated by rapidly changing confining pressure and temperature to the final reequilibration conditions of 2 kbar and 625 °C (PATH II, Fig. 1) over about a 20–30 min period. At the final reequilibration conditions, the original inclusions experienced an internal overpressure of about 2.1 kbar. The sample was held at these final reequilibration conditions for thirty days.

We also conducted experiments to examine the effect of slow *incremental loading* on inclusion reequilibration at elevated *P* and *T*. Inclusions were trapped at 700 °C and 5 kbar (*T*f, *P*f; Fig. 1) in five quartz samples (ITD-3-7, ITD-3-30, ITD-3-90, ITD-3-180, and ITD-3-270), each sample in a different pressure vessel. After 7 days, *P-T* conditions in all five vessels were reduced from the original 700 °C and 5 kbar to 675 °C and 4 kbar to generate about 0.7 kbar of internal overpressure in the original inclusions (PATH II, step 1, Fig. 1). This procedure was repeated in 25 degree Celsius and one kilobar steps to 650 °C and 3 kbar (step 2) and then to 625 °C and 2 kbar (step 3). Thus, trapping and subsequent reequilibration of inclusions was accomplished without quenching the samples to ambient conditions (see also, Vityk and Bodnar 1995b). The final conditions of reequilibration (625 °C and 2 kbar) resulted in a cumulative overpressure of about 2.1 kbar. At the final conditions (625 °C and 2 kbar), the samples were held for 7 (sample ITD-3-7), 30 (ITD-3-30), 90 (ITD-3-90), 180 (ITD-3-180) and 270 days (ITD-3-270), respectively. After quenching, the quartz cores were sliced in half. One half of each core was sectioned perpendicular to the c-axis and polished to produce disks for microthermometric study. The remaining half of each sample was thermally shocked (see, Bodnar and Sterner 1987) to produce a new set of microfractures (note

Table 1 Results from decompression reequilibration experiments. *T*h = homogenization temperature. Data in parentheses are internal overpressures (in kbar) calculated after each experiment. Mean density and strain were calculated from mean *T*h values for *T*h histogram

Sample	Time (days)	Count	*T*h min (°C)	*T*h max (°C)	Range (°C)	*T*h mean (°C)	*T*h median (°C)	*T*h SD (°C)	Density mean (g/cm^3)	Strain mean (%)
ITD-0	7	160	282	285	3	284	284	1	0.841	0
ISC-1	35	133	272	295	23	284	285	4	0.841	0
ITD-3-30R	30	154	307	380	73	345	350	18	0.790	6.1
			(1.6)	(0)		(0.7)	(0.6)	(0.4)		
ITD-3-7A	7	164	270	359	89	298	290	21	0.822	2.3
			(2.4)	(0.46)		(1.8)	(1.91)	(0.46)		
ITD-3-7B	7	164	282	359	77	306	294	23	0.811	3.6
			(2.1)	(0.46)		(1.6)	(1.9)	(0.5)		
ITD-3-30A	30	165	286	372	86	322	316	19	0.787	6.4
			(1.8)	(0.17)		(1.26)	(1.42)	(0.42)		
ITD-3-30B	30	160	293	365	72	319	315	17	0.792	5.8
			(1.9)	(0.33)		(1.35)	(1.44)	(0.3)		
ITD-3-90A	90	160	298	365	67	319	318	14	0.792	5.8
			(1.81)	(0.33)		(1.35)	(1.37)	(0.37)		
ITD-3-90B	90	135	294	369	75	317	316	12	0.795	5.5
			(1.9)	(0.24)		(1.38)	(1.42)	(0.26)		
ITD-3-180A	180	160	285	372	87	320	319	11	0.790	6.1
			(2.1)	(0.17)		(1.32)	(1.35)	(0.24)		
ITD-3-180B	180	169	292	360	68	321	320	10	0.788	6.3
			(1.95)	(0.53)		(1.30)	(1.33)	(0.22)		
ITD-3-270A	270	160	308	367	59	323	319	10	0.785	6.7
			(1.59)	(0.28)		(1.26)	(1.34)	(0.23)		
ITD-3-270B	270	175	310	360	50	323	319	11	0.785	6.7
			(1.55)	(0.44)		(1.26)	(1.34)	(0.24)		
ITD-3-90-90										
H_2O-NaCl	180	171	296	378	82	343	343	14		
			(1.8)	(0)		(0.8)	(0.8)			
H_2O	90	42	336	358	22	348	345	4		
ITD-3-180-180										
H_2O-NaCl	360	102	322	376	54	346	343	16		
			(1.3)	(0)		(0.7)	(0.8)			
H_2O	180	50	340	348	8	345	345	2		

that the samples already contained one set of microfractures with reequilibrated 10 wt% NaCl-H_2O inclusions from the just completed incremental loading experiment). Each of these twice-fractured quartz cores was loaded into a platinum capsule with pure water. Each capsule was sealed and placed into a separate pressure vessel for an additional 90 days (sample ITD-3-90-90) and 180 days (sample ITD-3-180-180) at 625 °C and 2 kbar to examine the effect of a second fracturing event and introduction of a new fluid on the reequilibration process. This experiment was designed to simulate the occurrence of two fracture sets, containing inclusions of distinctly different ages and compositions, commonly observed in high-grade metamorphic rocks (cf., Touret 1981).

Approximately 120–160 randomly selected fluid inclusions were measured from each sample to monitor the distribution of homogenization temperatures (density) for reequilibrated inclusions. Within each sample several groups of inclusions were selected from different healed fractures. Inclusion size was estimated by measuring the longest and shortest dimensions of each inclusion and calculating the area assuming an ellipsoidal and/or rectangular geometry.

Two different tactics were used during collection of microthermometric data. For one set of samples, homogenization temperatures were measured in the order from lowest to highest *T*h to assure that no inclusion was overheated before its *T*h was measured the first time. The second approach consisted of selecting inclusions randomly and measuring their homogenization temperatures without regard to *T*h of other inclusions in the sample. With this approach, some inclusions were necessarily overheated by some unknown amount *before* the *T*h was measured. It is important to note that whichever method of selecting inclusions is used, the worker must be certain that the group of inclusions from which the selection is being made represents a single FIA. Selection of inclusions from multiple FIAs invalidates any reasonable interpretation of the fluid inclusion data.

Initially, we did not know how many inclusions in each sample had to be measured to obtain a statistically valid number. This was determined by plotting the number of measured inclusions versus the mean *T*h calculated for that number of inclusions. For preselected inclusions, the mean increased continuously until about 120 inclusions were measured, but remained constant as additional inclusions were measured (Fig. 2). For randomly selected inclusions, the mean approached a constant value of about 321 °C (± 1 °C) after measurement of 60 inclusions (Fig. 2). Regardless of which selection technique was used, the final histograms showed similar statistical parameters as discussed below. An implication of this result is that the amount of pressure generated in the lower temperature inclusions during measurement of some of the higher temperature inclusions using the random selection technique was insufficient to cause further reequilibration of the low temperature inclusions. However, it should be noted that in some samples fluid inclusions measured using the random selection method may stretch or decrepitate during measurement of some higher temperature inclusions if the total range in *T*h is sufficiently large and/or if the internal pressures generated during heating are sufficiently high (Bodnar et al., 1989). *Thus, we recommend that in all fluid inclusion studies the preselection method described above be used to avoid overheating inclusions before Th is measured the first time.* Measured homogenization temperatures were plotted in frequency histograms. Each histogram was described by a set histogram pa-

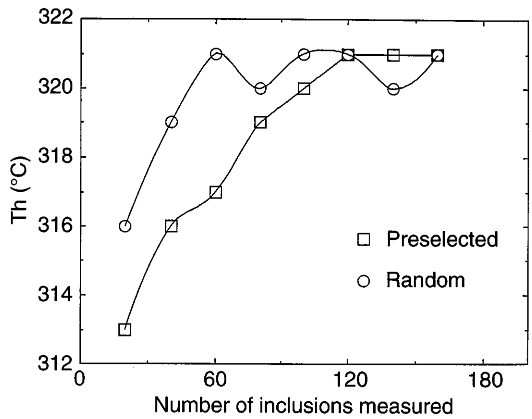


Fig. 2 Relationship between mean homogenization temperature for reequilibrated fluid inclusions in sample ITD-3-180 and number of inclusions measured. *Squares* represent the mean *Th* calculated for preselected fluid inclusions, whereas *circles* represent the mean *Th* calculated for randomly measured fluid inclusions.

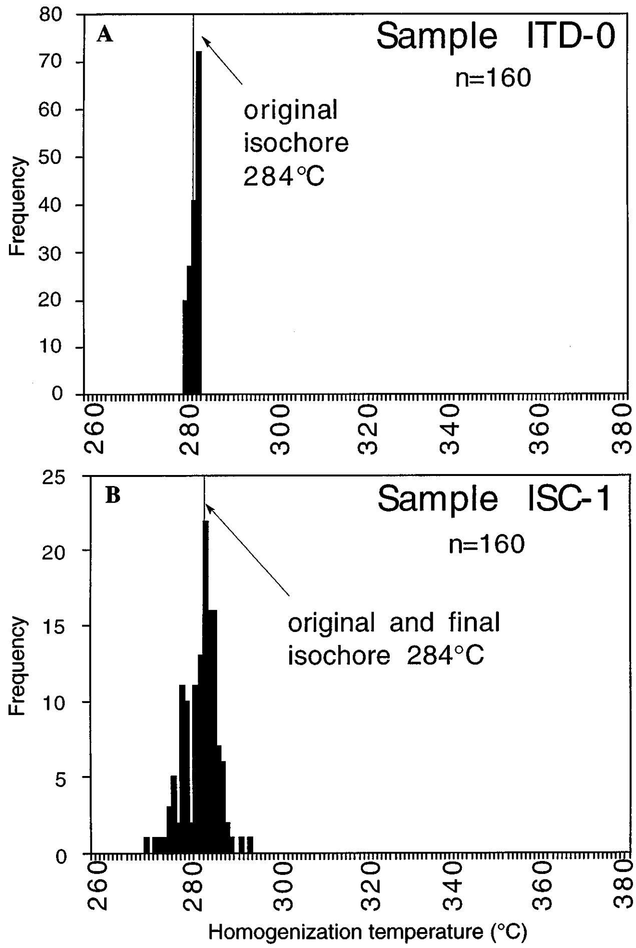


Fig. 3A,B Histograms of homogenization temperatures for 10 wt% NaCl-H_2O synthetic fluid inclusions in quartz after quenching (sample IT-0, histogram **A**) from formation conditions (700 °C and 5 kbar), and after isochoric cooling along PATH I shown in Fig. 1 (sample ISC-1; histogram **B**). *Th* corresponding to the original isochore for the synthetic fluid inclusions is 284 °C (±7 °C)

rameters (see, Ott 1992), including: extreme values (minimum and maximum *Th* values, i.e. outliers), range (the difference between the outliers), mean, median, and standard deviation (Table 1).

Results

Fluid inclusions originally formed at 700 °C and 5 kbar for 7 days homogenize at 282–285 °C with a mean *Th* value at 284 °C (sample ITD-0, Fig. 3A). After the incremental isochoric cooling experiment (PATH I, Fig. 1), the range in *Th* increased by 20 degrees Celsius compared to the range of *Th* values observed after quenching from the original formation conditions (sample ISC-1, Fig. 3B). The range in *Th* observed for the fluid inclusions after isochoric cooling can partly be explained by the combined effects of the experimental temperature error (about 7 °C, which is approximately 1% of the total range for maximum *T* of 700 °C) and the error involved in isochore location (about 10 °C, Bodnar and Vityk 1994). Note, however, that in both cases ITD-0 and ISC-1 the mean *Th* values (284 °C) are identical (Table 1).

A frequency plot of homogenization temperatures of inclusions from the sample that was subjected to reequilibration under instantaneous loading is shown in Fig. 4 A. Reequilibrated inclusions produce a multimodal histogram with a wide range in *Th* and relatively high mean *Th* of 345 °C. Inclusions reequilibrated in this manner show a positive correlation between size and *Th* (density) (Fig. 4B).

Histograms of homogenization temperatures of fluid inclusions from samples that were reequilibrated under conditions of internal overpressure for 7, 30, 90, 180 and 270 days during incremental loading are shown in Fig. 5. Note that the left column in Fig. 5 shows results for the sample in which *Th* was measured in the order from lowest to highest values (preselected inclusions), whereas the right column shows results for inclusions that were randomly selected for *Th* measurement (random sampling). After 7 days, (Fig. 5A, ITD-3-7A and ITD-3-7B), a large percentage of the inclusions have maintained their original density. The mean value for preselected inclusions (298 °C; ITD-3-7A) differs from the mean for randomly selected inclusions (306 °C; ITD-3-7B) (Table 1). The 7 day histograms are skewed to the right and are characterized by a large standard deviation (about 22 °C, Table 1). The 30 day reequilibration experiment (ITD-3-30 A and ITD-3-30B, Fig. 5B) also produced a unimodal skewed histogram for both selection criteria but with less variability (standard deviation 18 °C) and a mean *Th* value (320 °C) which is considerably higher than the mean (298 and 306 °C) calculated for the ITD-3-7 samples. The histograms for the 90 day experiments are more symmetrical but still skewed to the right (sample ITD-3-90 A and ITD-3-90B, Fig. 5C); the mean *Th*s (319 and 317 °C) changed little compared to the 30 day experiment (322 and 319 °C). The standard deviation decreased to about 13 °C after 90 days, com-

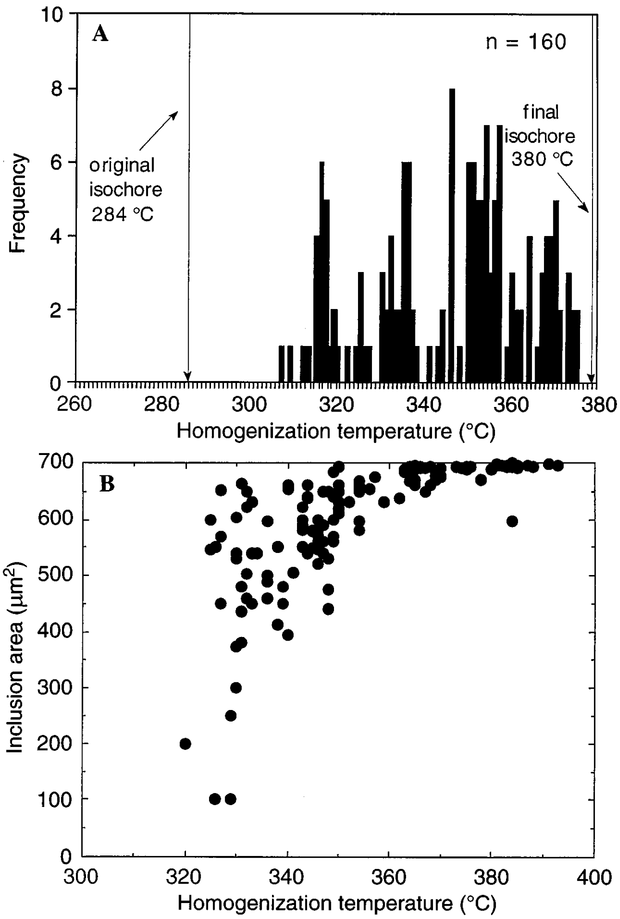


Fig. 4A,B Histogram of homogenization temperatures (**A**) and relationship between the homogenization temperature and inclusion size (**B**) for 10 wt% NaCl-H_2O synthetic fluid inclusions reequilibrated under conditions of instantaneous loading (sample ITD-3-30R; PATH II, Fig. 1)

pared to an average standard deviation of 18 °C after the 30 day experiment. Finally, inclusions reequilibrated for 180 and 270 days display unimodal histograms that are slightly skewed to the right and with relatively small standard deviations (about 10 °C) and a mean *T*h between 320 and 323 °C (Fig. 5D and E). For both selection criteria, the mean, median and standard deviation values differ by less than 1 °C. Fluid inclusions from the experiments described above have ice-melting temperatures of −6.7 ± 0.1 °C, which correspond to about 10.1 ± 0.1 wt% NaCl-H_2O, indicating that the inclusions may have lost small amounts of water during the reequilibration process. Although this loss of water is not directly relevant to the discussion here, it does have important implications concerning the mechanisms controlling the reequilibration of fluid inclusions (Vityk and Bodnar 1997).

Results of two experiments, ITD-3-90-90 and ITD-3-180-180, with 10 wt% NaCl-H_2O inclusions that first were reequilibrated at 625 °C and 2 kbar (2.1 kbar of internal overpressure) for 90 and 180 days, respectively, and then were subjected to thermal shock followed by continued reequilibration at 625 °C and 2 kbar in pure H_2O for an additional 90 and 180 days are given in Table 1 and plotted in Fig. 6. As expected, inclusions with two different compositions were observed after the experiments. One group of inclusions shows *T*m ice ranging from −6.6 to −7.4 °C, which corresponds to 10.0–11.0 wt% NaCl (Bodnar 1993), and the other set of inclusions shows *T*m ice ranging from 0.0 to −1.1 °C, which corresponds to 0.0–1.9 wt% NaCl (Fig. 7). In the following discussion, the inclusions containing 10.0–11.0 wt% NaCl-H_2O solution are referred to as the NaCl-H_2O inclusions, and the inclusions containing 0–1.9 wt% are referred to as the H_2O inclusions. After reequilibration, the H_2O inclusions in both ITD-3-90-90 and ITD-3-180-180 samples produce histograms with mean values at about 345 °C and 348 °C, respectively. These values are in good agreement with the expected *T*h for pure water synthetic inclusions trapped at 625 °C and 2 kbar (Bodnar and Vityk 1994). The fractures containing the H_2O inclusions did not contain NaCl-H_2O inclusions, i.e., these most likely represent new fractures opened during the second fracturing event. The NaCl-H_2O inclusions display a multimodal distribution of *T*h with very high data variability and mean at 343 °C (ITD-3-90-90) and 346 °C (ITD-3-180-180) (Table 1; Fig. 6A and B). Note that the NaCl-H_2O inclusions from both samples, ITD-3-90-90 and ITD-180-180, show a well defined frequency peak at 327 °C corresponding to about 1.2 kbar of internal pressure. Also note that a large percentage of NaCl-H_2O inclusion *T*h values cluster between 340 and 360 °C to produce a continuous unimodal distribution for ITD-3-90-90 and bimodal distribution for ITD-3-180-180. After both experiments, none of the H_2O-NaCl inclusions showed *T*h corresponding to the original H_2O-NaCl iso-*T*h line (Fig. 6A and B).

Discussion

Histograms for fluid inclusions that deform plastically

The first and perhaps the most important question that must be considered is whether laboratory-generated *T*h histograms for an FIA are indicative of the sample's *P*-*T* history. In order effectively to apply results from our experiments to geologic problems, potential laboratory effects of time and loading rates must also be considered. In our earlier reports (Vityk and Bodnar 1995a, b, 1996) we suggested that loading rates may affect the behavior of fluid inclusions during experimental reequilibration. In the present experimental study we attempted to minimize shock-induced modification of fluid inclusions arising from instantaneous loading. By applying differential pressure gradually over a period of several weeks, we found that fluid inclusions deformed in a plastic manner (Vityk and Bodnar 1997). These observations differ from instantaneous loading experiments (e.g., Bodnar et al. 1989; Sterner and Bodnar

1989), in which many inclusions showed evidence of brittle failure.

To examine the effect of time on inclusion reequilibration, inclusions formed at 700 °C and 5 kbar were reequilibrated along a decompressional *P*-*T* path to simulate retrograde *P*-*T* conditions typical for many high-grade (amphibolite- to granulite-grade) metamorphic terranes (Spear 1993). The experiments were designed so that the inclusions were deformed plastically at a constant loading rate beyond their elastic limit into the plastic regime (Vityk and Bodnar 1996). At the final conditions (625 °C and 2 kbar), the samples were held for 7, 30, 90, 180 and 270 days, respectively. The histograms for these samples show different spread, or variability, about the mean as a function of time: the shorter the duration of the experiment the higher the variability (standard deviation) of measurements (Fig. 8). Thus, the 7 day experiment produced a highly skewed histogram with a standard deviation of about 22 °C. On the other hand, the 270 day experiment produced a nearly symmetrical distribution with a standard deviation of 10 °C. Accordingly, our results suggest that a nearly symmetrical histogram that is slightly skewed to higher temperatures and with low data variability is characteristic of fluid inclusion reequilibration resulting from plastic deformation under conditions of internal overpressure. The mean, median and mode for this histogram are the same, and the isochore corresponding to the mean *T*h represents a *P*-*T* path along which the inclusions would have had an overpressure of about 1 kbar. The observed differences in the data variability arise mostly because inclusions can volumetrically and chemically adapt more fully to changes in differential pressure in 270 days at the final reequilibration conditions (625 °C and 2 kbar) than they can in 7 days (Vityk and Bodnar 1997).

In this respect, it is important to consider how additional time affects the relationship between inclusion size and density (*T*h). A majority of the inclusions reequilibrated for 7 days showed a weak correlation between size and homogenization temperature (Fig. 9A), and this correlation continued after the 30 day experiment (Fig. 9B). However, with additional time the correlation becomes less obvious (Fig. 9C and D). Overall, the results demonstrate that during the early stages of deformation, the response of fluid inclusions to an imposed internal overpressure is highly variable and to some extent depends on the inclusion size. However, additional time eliminates this dependence. This behavior can be explained by considering the differing mechanical properties of the quartz surrounding the large and small inclusions before loading: the larger the inclusion the higher the activity of preexisting dislocations in the inclusion walls (Vityk and Bodnar 1997). Because the larger inclusions already have a considerable number of dislocations around them, they begin to deform as soon as they are subjected to a differential pressure. However, smaller inclusions must first develop dislocations in the host before deformation can begin. This "incubation" period for the development and multiplication of dislocations is less than 90 days, because after 90 days the correlation between inclusion size and *T*h is absent.

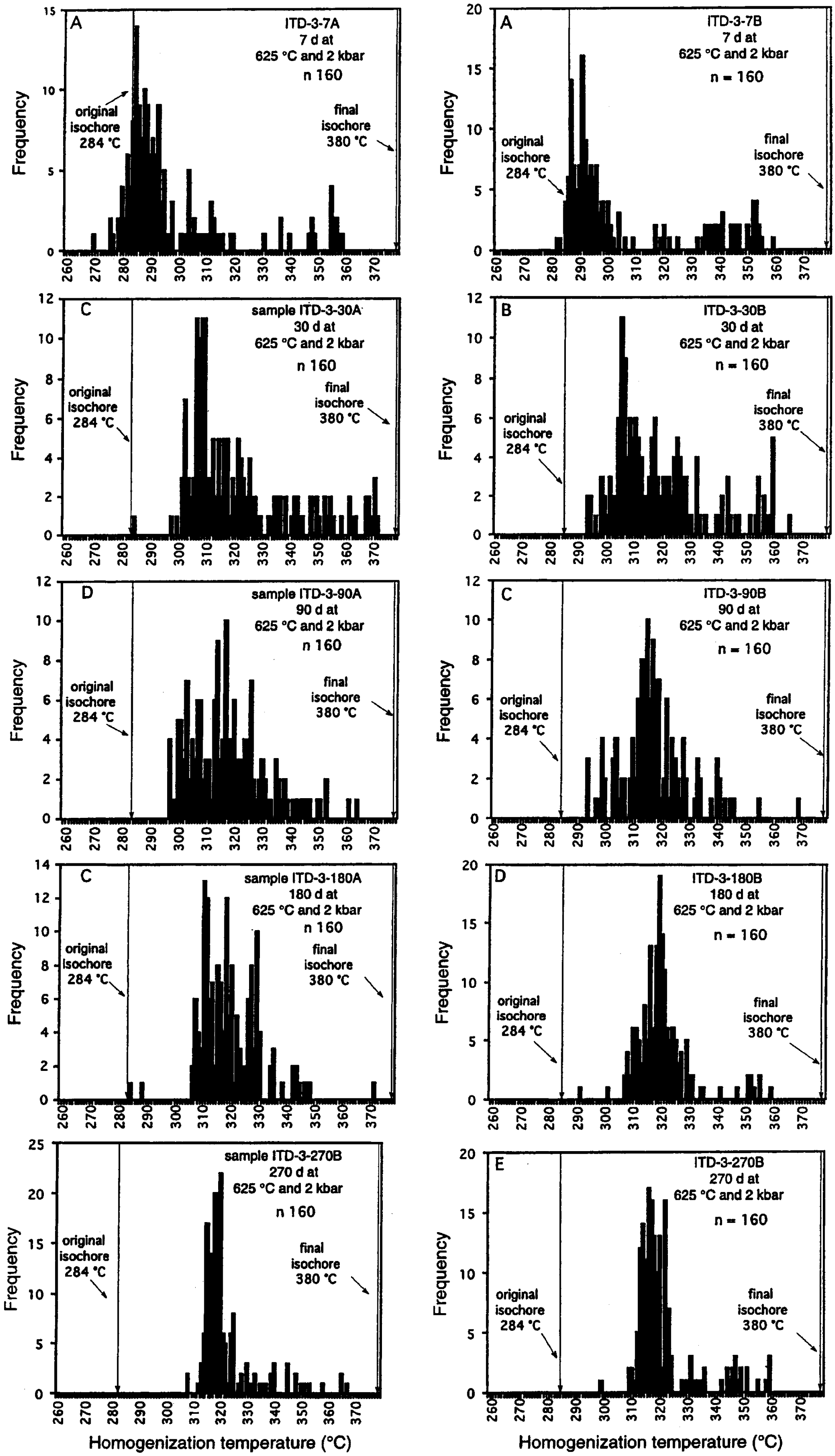
Inclusion densities can also be affected by decrepitation (followed by rehealing) of large inclusions and/or continued healing of incompletely healed fractures isolated from the bulk fluid. For example, we noted that after the 7 day experiment, samples contained networks of interconnected fractures filled by fluid and tubular voids. On the other hand, after the 180 and 270 day experiments these structures were mostly healed to produce new inclusions.

Based on results of our experiments, histogram mean values are a useful measure of the overall density shift for fluid inclusions that deform in the plastic regime. It should be emphasized, however, that the mean may be subject to distortion due to the presence of extreme values in a set of measurements. As can be seen from Fig. 5, histograms representing short duration experiments are strongly skewed to higher temperatures, i.e., each histogram has a long tail and a single peak at lower temperature. The histogram peak(s) or mode(s) (290–320 °C) are thought to represent inclusions reequilibrated as a result of plastic deformation. On the other hand, the histogram tails (340–380 °C) represent inclusions that experienced brittle deformation and reequilibrated by fracturing and rehealing (see discussion in the next section). In these histograms, *T*h values in the 340–380 °C range are relatively rare (less than 15%). However, these values shift the mean in the direction of higher homogenization temperatures, resulting in a mean that is an incorrect measure of inclusion density shift resulting from plastic deformation. By trimming the data, however, we can reduce the impact of the few high *T*h values on the mean. Thus, each histogram was trimmed by dropping 15% of the highest *T*h values and averaging the remainder (see Table 2).

Results of experimental reequilibration of synthetic fluid inclusions under conditions of gradual loading are presented in Fig. 10. Shown are the average *trimmed mean* values calculated for *T*h histograms (Fig. 10 A), the *total apparent strain* of the inclusions (Fig. 10B), and the inclusion overpressure (Fig. 10 C), all as a function

▶

Fig. 5 Histograms of homogenization temperatures for 10 wt% NaCl-H_2O synthetic fluid inclusions reequilibrated at 625 °C and 2 kbar for 7 days (**A**), 30 days (**B**), 90 days (**C**), 180 days (**D**) and 270 days (**E**). *T*h values of original inclusions formed at 700 °C and 5 kbar are shown in Fig. 3 A. The inclusions were reequilibrated under conditions of gradual loading (0.7 kbar $week^{-1}$). Histograms in the *left column* (samples ITD-3-7A, −30A, −90A, −180A, and −270A) show results for samples in which inclusions were measured starting from the lowest *T*h and proceeding to the highest *T*h (preselected inclusions), whereas the histograms shown in the *right column* (samples ITD-3-7B, −30B, −90B, −180B, and −270B) represent inclusions that were randomly selected for *T*h measurements. The *T*h labeled "original isochore 284 °C" represents the expected *T*h if no reequilibration occurred, whereas the *T*h labeled "final isochore 380 °C" represents the *T*h expected if the inclusions completely reequilibrate to final conditions.


A
ITD-3-7A
7 d at
625 °C and 2 kbar
n 160
original isochore 284 °C
final isochore 380 °C
A
ITD-3-7B
7 d at
625 °C and 2 kbar
n = 160
original isochore 284 °C
final isochore 380 °C
C
sample ITD-3-30A
30 d at
625 °C and 2 kbar
n 160
original isochore 284 °C
final isochore 380 °C
B
ITD-3-30B
30 d at
625 °C and 2 kbar
n = 160
original isochore 284 °C
final isochore 380 °C
D
sample ITD-3-90A
90 d at
625 °C and 2 kbar
n 160
original isochore 284 °C
final isochore 380 °C
C
ITD-3-90B
90 d at
625 °C and 2 kbar
n = 160
original isochore 284 °C
final isochore 380 °C
C
sample ITD-3-180A
180 d at
625 °C and 2 kbar
n 160
original isochore 284 °C
final isochore 380 °C
D
ITD-3-180B
180 d at
625 °C and 2 kbar
n = 160
original isochore 284 °C
final isochore 380 °C
sample ITD-3-270B
270 d at
625 °C and 2 kbar
n 160
original isochore 284 °C
final isochore 380 °C
E
ITD-3-270B
270 d at
625 °C and 2 kbar
n = 160
original isochore 284 °C
final isochore 380 °C
Frequency
Homogenization temperature (°C)
Homogenization temperature (°C)

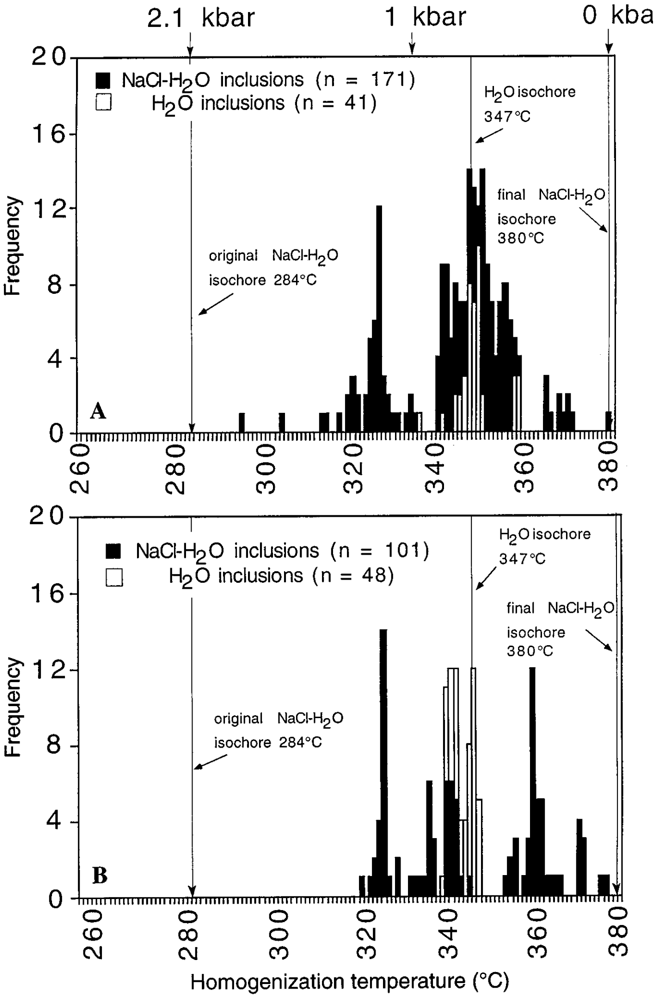


Fig. 6A,B Histograms of homogenization temperatures for two populations of synthetic fluid inclusions (NaCl-H_2O and H_2O inclusions) in samples reequilibrated for 90 days (histogram **A**) and for 180 days (histogram **B**). Calculated internal pressures of earlier generation NaCl-H_2O fluid inclusions, corresponding to the measured *T*h, are shown along the *top* of the histogram in **A**

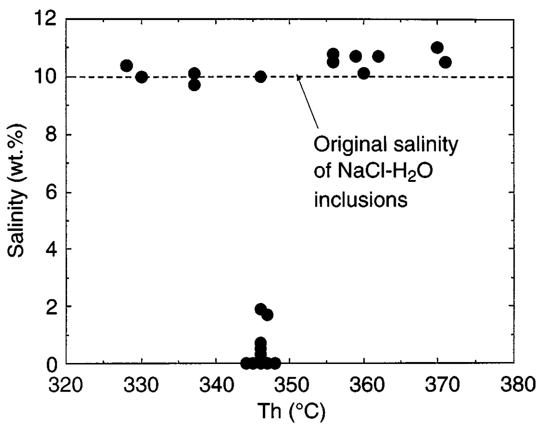


Fig. 7 Relationship between salinity and homogenization temperature for fluid inclusions in sample ITD-3-180-180

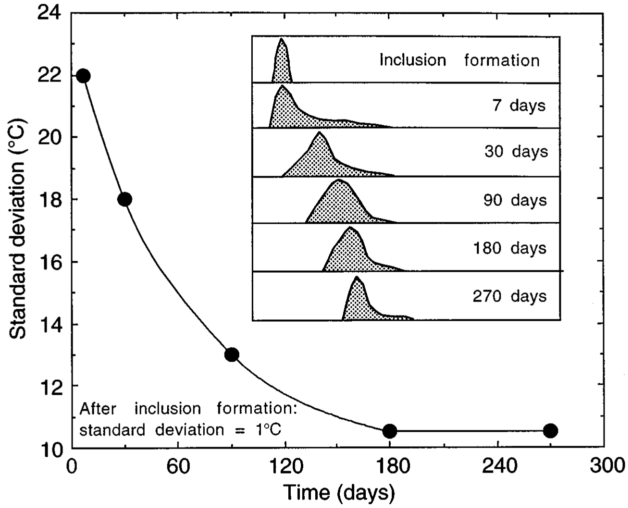


Fig. 8 Relationship between standard deviation of inclusion *T*h histograms and duration of reequilibration experiments involving incremental loading. Also shown is a schematic representation of the manner in which histogram shapes evolve during incremental loading (*see insert*)

of the duration of the reequilibration experiment. Inclusion total apparent strain represents the percent change in inclusion density during the experiment, and was calculated from homogenization temperatures of the inclusions measured before and after the experiment. Calculation of the total apparent strain assumes that composition of the inclusion remains constant (i.e., no water loss occurred during the experiment). Thus, for example, if a 10 wt% NaCl-H_2O inclusion with original density of 0.841 g/cc (*T*h = 284 °C) reequilibrates under conditions of internal overpressure to generate a new density of 0.799 g/cc (*T*h = 311 °C) the change in density is 0.042 g/cc and the total apparent strain is 0.05 or 5%. If this change in volume occurs over 90 days (7.776×10^6s), then the total strain rate is $0.05/((7.776 \times 10^6\text{s}) = 6.4 \times 10^{-9}\ \text{s}^{-1}$(see Table 2).

The calculated percent density change described above assumes that nothing has been added to or lost from the fluid inclusions. However, a salinity increase of as much as 0.2 wt% NaCl noted after the 180 day run may have been the result of preferential water loss (see also, Sterner and Bodnar 1989; Hall and Sterner, 1993). This loss of water corresponds to about a 2% change in inclusion density. Accordingly, true plastic strain ($\varepsilon_{plastic}$) for the inclusion can be calculated as: $\varepsilon_{plastic} = \varepsilon_{total} - \varepsilon_{leakage}$, where ε_{total} is the total apparent strain, and $\varepsilon_{leakage}$ is the proportion of inclusion density change resulting from water loss (Fig. 10B).

The increase in inclusion total strain reduces the internal overpressure of the fluid inclusion (Fig. 10 C), shifting (reducing) inclusion density toward the final reequilibration isochore. The most dramatic changes in fluid inclusion density occurred during the first 30 days of reequilibration (Fig. 10 A and B). After 30 days, the histogram mean value was shifted towards the final re-

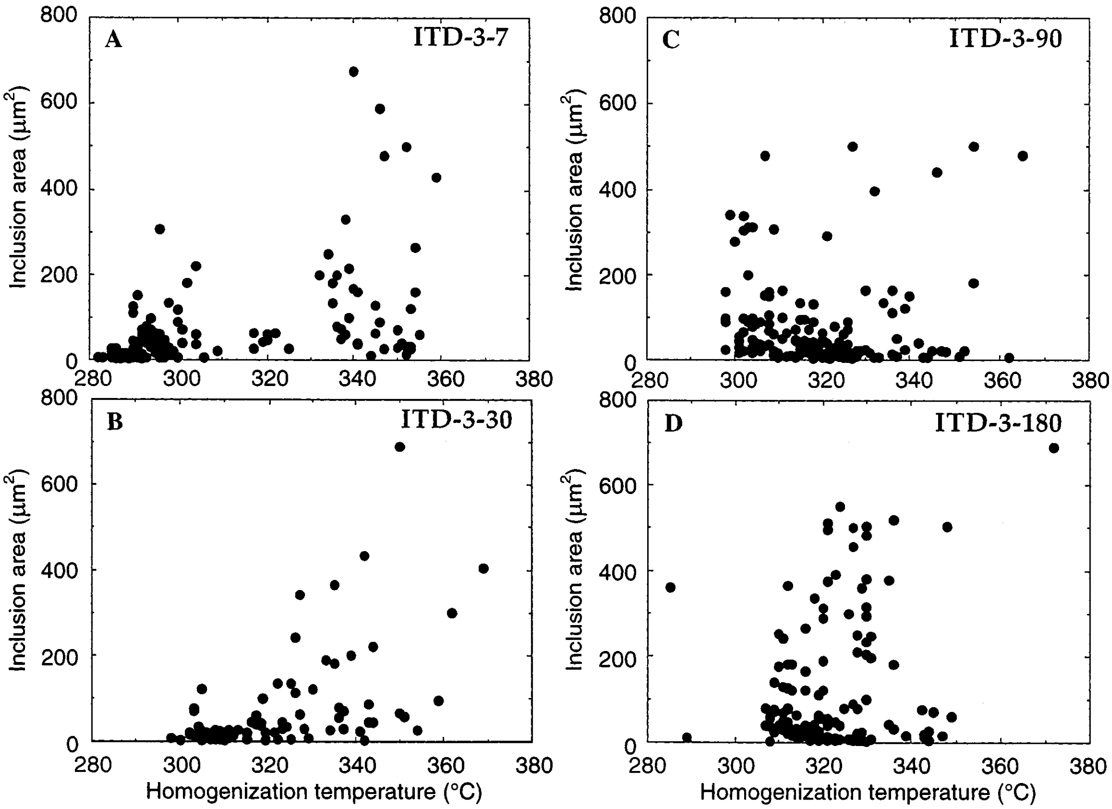


Fig. 9A–D Relationship between homogenization temperature and inclusion size (*area*) for 10 wt% NaCl-H_2O synthetic inclusions reequilibrated under conditions of incremental loading for 7 days (**A**), 30 days (**B**), 90 days (**C**), and 180 days (**D**)

Table 2 Mean and trimmed mean values for *T*h of fluid inclusions from reequilibration experiments and calculated inclusion density, internal overpressure, and deformation parameters. Data in parentheses are values corresponding to stretching strain, i.e., strain values corrected for loss of water

Sample	*T*h[a] (°C)	*T*h[b] (°C)	*T*h[c] (°C)	Density (g/cc)	*P*internel (kbar)	Total strain (%)	Total strain rate (sec^{-1})
ITD-0	284	284		0.841	–	–	–
ITD-3-7A	298	291					
			292	0.831	1.94	1.2	2×10^{-6}
ITD-3-7B	306	293					
ITD-3-30A	322	312					
			311.5	0.803 (0.819)	1.53	4.5 (2.5)	1.7×10^{-8} (9.6×10^{-9})
ITD-3-30B	319	311					
ITD-3-90A	319	314					
			314	0.799 (0.815)	1.46	5.0 (3.0)	6.4×10^{-9} (3.8×10^{-9})
ITD-3-90B	317	314					
ITD-3-180A	320	317					
			317.5	0.795 (0.814)	1.39	5.4 (3.4)	3.5×10^{-9} (2.2×10^{-9})
ITD-3-180B	321	318					
ITD-3-270A	323	319					
			319	0.792 (0.807)	1.35	5.8 (3.8)	2.5×10^{-9} (1.6×10^{-9})
ITD-3-270B	323	319					

[a] *T*h – mean value obtained for inclusions from each quartz disk measured
[b] *T*h – trimmed mean value
[c] *T*h – average sample trimmed value. Data in parentheses are values corresponding to stretching strain, i.e., strain values corrected for loss of water.

equilibration isochore by approximately 25 °C (about 4.5% of total strain) from the original iso-*T*h line. Longer duration experiments (more than 30 days) had relatively little effect on inclusion *T*h (less then 10 °C change, which corresponds to about 1.5% of total strain), suggesting that a state close to equilibrium (i.e., some steady flow state) had been achieved by many fluid inclusions after about 30 days.

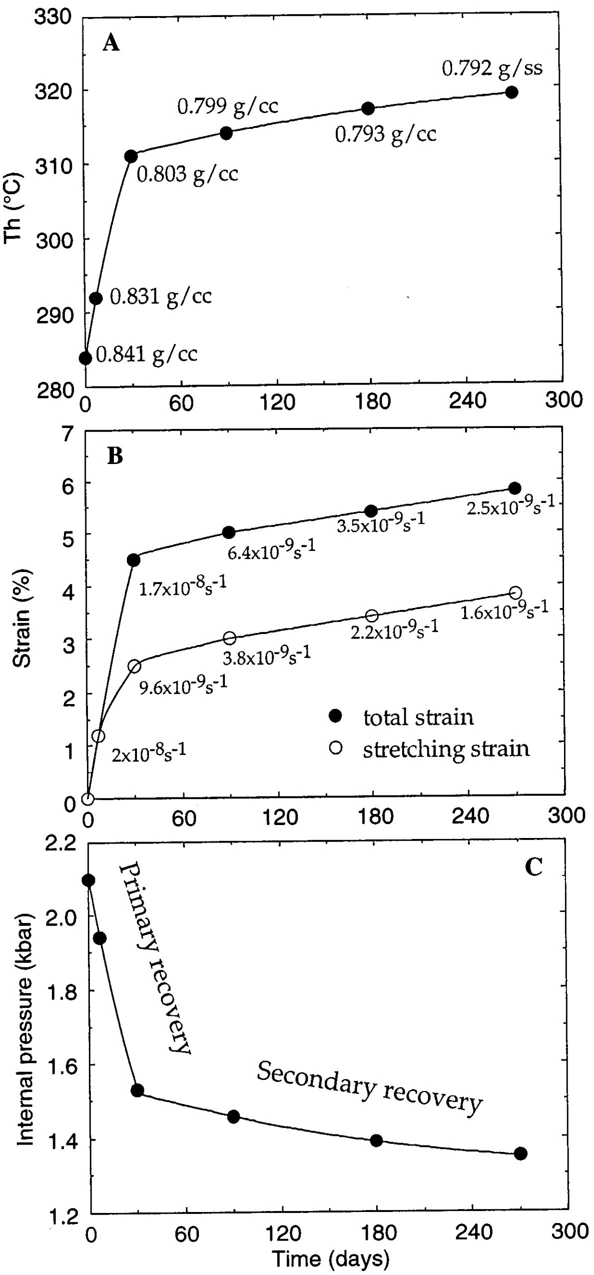


Fig. 10A–C Evolution of the trimmed mean homogenization temperatures (**A**), total strain and stretching strain (**B**) and internal overpressure (**C**) calculated from the trimmed mean values for *Th* histograms, plotted as a function of the duration of the experiment (samples ITD-0, ITD-3-7, ITD-3-30, ITD-3-90, ITD-3-180, and ITD-3-270; see Table 2). The *numbers adjacent to circles* in **A** are the density values calculated from the trimmed mean *Th* values. The *numbers adjacent to circles* in **B** are inclusion deformation strain rates (see text for additional explanations)

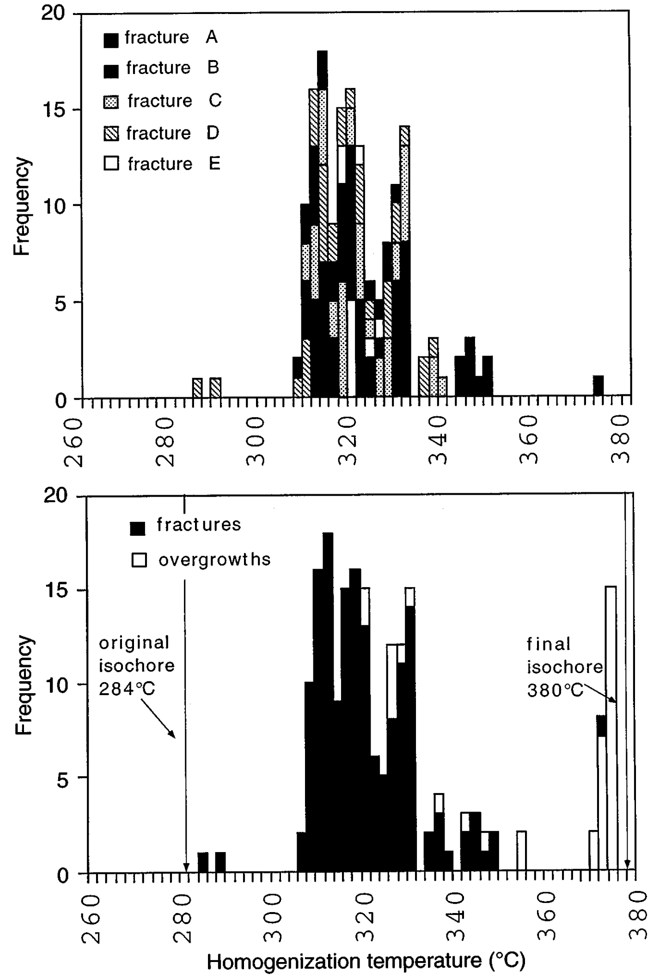


Fig. 11 Histogram of homogenization temperatures for fluid inclusions trapped in five different fractures in sample ITD-3-180 (*top histogram*). The *bottom histogram* shows *Th* from two different FIAs. One represents secondary fluid inclusions in healed fractures and the other FIA represents primary inclusions trapped during overgrowth formation

These results indicate that even fast experimental loading rates allow the inclusion volume to adjust (relax) such that the inclusion recovers from high internal stresses over a relatively short period of time (Fig. 10). The driving force for recovery of inclusions in quartz from the high internal stresses presumably arises from plastic deformation of the inclusion walls (dislocation glide) and/or inclusion leakage to produce a unimodal *Th* histogram with low variability and no correlation between inclusion size and density (see also, Boullier et al. 1989; Bakker and Jansen 1994; Vityk and Bodnar 1996). Recovery of the inclusions from the high internal stresses is a relatively rapid process, and can be achieved in about 30 days. After this "primary" recovery, the estimated internal overpressure for the inclusions at the final conditions of reequilibration was about 1.5 kbar. This internal overpressure was further reduced by about 200 bars, to 1.3 kbar, with 240 additional days of reequilibration (secondary recovery, Fig. 10 C), indicating that inclusion deformation over this time occurred at very low strain rates.

An important feature of experimentally reequilibrated inclusions is that a small percentage of the inclusions (1–2%) maintained their original (or close to the original) density. As was noted earlier (Vityk and Bodnar 1995a) and observed in this study, these inclusions do

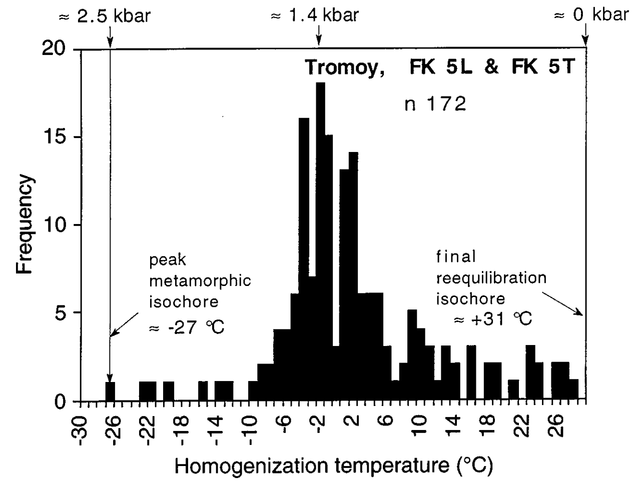


Fig. 12 Histogram of homogenization temperatures for coeval CO_2 inclusions from two samples (FK-5L and FK-5T) from Tromoy granulites, Proterozoic Bamble sector of southern Norway (unpublished data, A.M. van den Kerkhof). Calculated internal pressures of fluid inclusions, corresponding to the measured Th, are shown along the *top* of each histogram. The internal pressures for the inclusions from Tromoy were calculated using the estimated *P-T* path for the sample and isochores corresponding to measured *Th* of −27 °C, +2 °C, and +31 °C at 300 °C (see Fig. 6 in van den Kerkhof et al. 1994). Statistical parameters calculated for the internal pressures (in kilobar) are the following: min = 0.1, max = 2.5, mean = 1.23, median = 1.28, and st dev = 0.4

not appear to be different from other inclusions in the sample. They were observed in the same fractures with deformed inclusions that did change density (Fig. 11 A). This is an extremely important point which cannot be over-emphasized. The inclusions that maintained their original density cannot be characterized as having a special shape, size, distribution with respect to fractures or mineral surfaces, etc. The reasons why some inclusions maintain their original density while their presumably identical siblings reequilibrate represents fertile ground for future research on the fluid inclusion reequilibration phenomenon. Our long term experiments further indicate that a small percentage of the inclusions in fractures show *Th* that approximates the final reequilibration *Th* (380 °C). Inclusions with *Th* = 380 ± 7 °C were also found in overgrowths formed during the experiment (Fig. 11B). The inclusions in fractures with *Th* close to (but not equal to) the final *Th* could be original inclusions that decrepitated, or they could represent new inclusions trapped either during incremental decompression or at the final reequilibration *P* and *T* during continued fracture healing. If the latter origins apply, then it can be concluded that all fractures in the sample had been isolated from the bulk fluid in the capsule before the sample arrived at the final reequilibration conditions. Otherwise we would find fractures healed with inclusions having *Th* corresponding to the final iso-*Th* line. The density of secondary fluid inclusions formed as a result of continued healing of the isolated fractures may not correspond to the final isochore. However, the density of primary inclusions formed during overgrowth precipitation will represent the final conditions.

Histograms for fluid inclusions that reequilibrate by brittle deformation

The question remains whether inclusion histograms from rocks deformed in low temperature and/or high strain (fast loading) environments are considerably different from the inclusion histograms for rocks deformed under conditions of slow plastic flow at great depth. To provide some insight into this problem, we have conducted an experiment using synthetic inclusions in which a differential pressure of about 2 kbar was applied over a 20–30 min period. After loading, the sample was kept at the final conditions (625 °C and 2 kbar) for 30 days. Instantaneous loading results in a large spread of inclusion densities to produce a multimodal histogram with data considerably shifted toward the final reequilibration iso-*Th* line (Fig. 4). These results differ significantly from incremental loading experiments which produced a unimodal distribution with low data variability (Fig. 5).

Naumov et al. (1966), Leroy (1979), Swanenberg (1980), and Bodnar et al. (1989) showed that during heating of natural or synthetic fluid inclusions in quartz at one atmosphere confining pressure, 2 kbar of overpressure exceeds the rupture strength for inclusions larger than about 10 μm. This amount of internal overpressure generated over a short period of time (usually 10–20 min) causes failure of the inclusions in a brittle manner (decrepitation). The nucleation of subcritical microcracks in stressed walls of the fluid inclusions leads to an irreversible drop in density of the inclusion. Bodnar et al. (1989) also showed that the internal overpressure required to cause decrepitation during rapid heating is strongly dependent on inclusion size, with large inclusions decrepitating at lower internal stresses. Similar behavior was observed for synthetic fluid inclusions during our instantaneous loading experiment at elevated *P* and *T*. After the experiment, many large fluid inclusions showed associated planes of tiny fluid inclusions (healed micro-fractures), suggesting that inclusion failure occurred in a brittle manner. We also noted that after the experiment, large inclusions have relatively lower densities (higher *Th*) than smaller inclusions (Fig. 4B), suggesting either volume dependent decrepitation of the fluid inclusions or different deformation mechanisms for inclusions with different sizes, namely, brittle deformation for larger inclusions and slow plastic flow for smaller ones. Brittle deformation of the larger inclusions occurred immediately when the inclusions were rapidly subjected to 2 kbar of internal overpressure. These inclusions homogenize between 340 and 380 °C (Fig. 4). Plastic deformation of the smaller inclusions occurred during the additional thirty day period following loading. These inclusions homogenize at about 310–340 °C (Fig. 4)

To investigate further some possible effects of brittle deformation of rock hosting fluid inclusions, we have conducted two experiments involving superimposed fracturing of the host (experiments ITD-3-90-90 and ITD-3-180-180). Inclusions from these experiments produce a specific multimodal distribution (two or three modes) with high range and variability. This distribution is similar to that produced during instantaneous loading of synthetic fluid inclusions (compare Figs. 4 and 6), suggesting that the superimposed fracturing resulted in successive brittle modification of earlier inclusions. In both ITD-3-30-30 and ITD-3-180-180 samples, a large percentage of fluid inclusions maintained about 1.2 kbar of internal overpressure to produce a frequency peak at 327 °C (Fig. 6A and B), similar to that measured for fluid inclusions that were reequilibrated in the plastic regime. This indicates that many inclusions survived the fracturing event. One fluid inclusion maintained high internal overpressure of about 1.7 kbar (Fig. 6A), after being reequilibrated under conditions of thermally induced shock fracturing followed by overpressure conditions at 2 kbar and 625 °C for as long as for 180 days. This inclusion does not appear to be different from other inclusions in this same sample, an observation that was also noted for the 180 day experiments involving incremental loading.

Inclusions from the superimposed fracturing experiments showed a broad range of salinities with some that differ from the original 10 wt% NaCl and pure H_2O compositions (Fig. 7). Some inclusions have salinity slightly higher than 10 wt%, implying that some amount of water was preferentially lost from the fluid inclusions during the experiment to affect both inclusion density and composition. Inclusions with higher salinities also have higher homogenization temperatures (350–370 °C) that are thought to be related to micro-fracturing. This correlation between increased salinity and higher *T*h in the same inclusions might suggest that fracturing promotes preferential water loss. Fluid inclusions with salinities of about 2 wt% NaCl most likely represent NaCl-H_2O inclusions that were re-filled with H_2O fluid. Early NaCl-H_2O inclusions and late H_2O inclusions were observed along different fractures, suggesting that reactivation of old fractures and rehealing with new fluid did not take place.

Summary and geological implications

In laboratory experiments, when the stress exceeds some critical value, the response of fluid inclusions to high internal overpressure at elevated *P* and *T* involves two types of permanent deformation: deformation accompanied by fracturing of the host (inclusion *decrepitation* or *brittle deformation*), and deformation whereby strain is controlled by dislocations in their slip systems (*stretching* or *plastic deformation*). Each type of deformation produces a characteristic histogram of homogenization temperatures. Thus, a mound-shaped unimodal histogram with low range is indicative of a nearly isochoric cooling *P-T* path. During slow incremental loading, inclusions deform in a plastic manner to generate a unimodal, slightly skewed *T*h histogram with low data variability but high range and no correlation between inclusion size and density. Conversely, during instantaneous loading, many inclusions deform in a brittle manner to produce a multimodal *T*h histogram with high range and data variability and strong positive correlation between inclusion size and density.

During slow incremental loading at 625 °C and 2 kbar, synthetic aqueous fluid inclusions in natural quartz are able to recover from high internal overpressure (about 2 kbar) without generating fractures to release stress. Recovery is a relatively rapid process and can be achieved within about 30 days, and is accommodated by plastic flow (by stretching and diffusion) of the inclusion walls (Vityk and Bodnar 1997). Assuming that inclusion deformation proceeds in a similar manner in nature, we conclude that for most high *P* and *T* conditions decrepitation of natural inclusions with an average size (5–20 μm) and a regular negative crystal morphology is unlikely to occur. Decrepitation is only likely to take place in low temperature, high strain metamorphic environments. Similar conclusions were reached by Küster and Stöckert (1997) in a study of natural fluid inclusions in high pressure, low temperature rocks from Crete.

The internal overpressure that can be maintained by the majority of fluid inclusions over a 270–360 day period is about 1.2–1.3 kbar. This closely approximates the overpressure at which the inclusions start to *flow* by stretching and leakage, i.e., the inclusion yield stress (see, Vityk and Bodnar 1996, 1997). We suggest that the yield stress corresponds closely to the internal overpressure that can be maintained by inclusions in quartz over geological time. Our experiments also indicate that a small percentage of the fluid inclusions can maintain about 2 kbar internal overpressure for as long as 180 days (see also, Vityk and Bodnar 1995a). These inclusions represent histogram outliers and can be used to infer the original conditions (original isochore) of entrapment. However, we emphasize that in order to use outliers for this purpose it is necessary to document that all the inclusions being studied (including the outliers) belong to the same FIA. At this moment, we do not have a defensible explanation for original density preservation by some synthetic fluid inclusions in our long term experiments.

In order to test the relevance of our experimental data to samples which have been deformed in nature we compared our results with microthermometric data from the Tromoy granulites. Lamb et al. (1986) conducted a detailed petrologic study of the Tromoy granulites of southern Norway, and concluded that peak metamorphism occurred at around 800 °C and 7 kbar. Following peak metamorphism, the rocks followed a decompression *P-T* path in which the pressure decreased from about 7 to 1 kbar while temperature dropped from about 800 to 300 °C (see, van den Kerkhof et al., 1994). Van den Kerkhof et al. (1994) suggested that CO_2-rich inclusions in quartz segregations in enderbites from Tromoy were trapped at near peak *P-T* conditions, and then subsequently were gradually reequilibrated during unroofing to produce a *T*h histogram spanning a wide range (see Fig. 6 in van den Kerkhof et al., 1994). The final conditions of reequilibration would have generated about 2.5 kbar of overpressure in the inclusions. Note that in van den Kerkhof et al. (1994) the *T*h histogram shown in their Fig. 6 represents many samples. We have obtained data for one sample from van den Kerkhof (personal communication, 1995) and *T*h for this sample is plotted in Fig. 13 for comparison with synthetic inclusions (Fig. 5D and E).

The mean *T*h value for the experimental results corresponds to an internal overpressure of 1.3 kbar. This value is in good agreement with the internal overpressure for the Tromoy granulites (1.2 kbar) (Fig. 12). Note, however, that the standard deviation of the histogram for natural inclusions (0.4 kbar) is higher than that for synthetic inclusions (0.2 kbar). This can be explained by the presence of more then one FIA in the Tromoy sample, whereas the laboratory reequilibrated sample contains a single FIA. This can also be explained if the reequilibration process in natural samples is more complex than that for synthetic fluid inclusions described here. Our experimental data further indicate that after the long term experiments, a small percentage of aqueous synthetic inclusions preserve their original density. Similarly, in the Tromoy samples, a very few CO_2 inclusions have homogenization temperatures of −27 °C, and this isochore projects through the peak *P-T* conditions. Tromoy samples show no correlation between inclusion size and density, regardless of their decompressional retrograde history (van den Kerkhof et al. 1994). A similar relationship was documented in our experiments. However, many natural samples from metamorphic environments show evidence that large inclusions decrepitate at *lower* effective pressure than smaller inclusions (e.g., Swanenberg 1980). The same relationship was also documented in our high *P-T* experiments conducted under conditions of instantaneous loading, suggesting that the positive size-density correlation might be indicative of deformation in high strain environments.

Finally, it is important to note that we used aqueous inclusions in our experimental reequilibration study, whereas the inclusions reported by van den Kerkhof et al. (1994) were pure CO_2. We recognize that composition may affect reequilibration behavior (e.g., Hall and Wheeler 1992; Vityk et al. 1995), and that a rigorous comparison of synthetic aqueous inclusions with Tromoy granulite CO_2 inclusions may not be valid. Nevertheless, our experimental results with water-rich inclusions and the results from natural CO_2 inclusions show many similarities in their microthermometric behavior, and this might suggest that the CO_2 inclusions originally contained a small amount of water which was lost during plastic deformation (e.g., Lamb et al. 1987).

Our data indicate that by using experimentally reequilibrated synthetic inclusions it is feasible to identify parameters such as the mean, mode, extreme values, etc., which are characteristic of a given *P-T* history. Each of these parameters has a significance and may be used in tectonic reconstructions. This offers encouragement that fluid inclusion reequilibration behavior is not a random process but, rather, occurs systematically and reflects the changing physical and chemical conditions of the inclusions. As such, fluid inclusion microthermometric statistical characteristics may supplement other techniques used to infer *P-T* histories of samples that have experienced complex tectonic histories.

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