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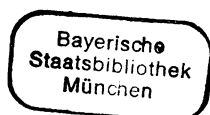
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THE SOLUBILITY OF H₂O IN MELTS IN THE SYSTEM SiO₂-Al₂O₃-Na₂O-K₂O AT 1 TO 2 KBARS¹

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ABSTRACT

The solubilities of water in six melts in the system K₂O-Na₂O-Al₂O₃-SiO₂ were determined at 970–1630 bars and 800°C. Melts were synthesized hydrothermally in Pt capsules in cold-seal vessels and then quenched isobarically. The solubilities were determined by micromanometric measurement of H₂O evolved during vacuum fusion of vesicle-free glass wafers and are reproducible to ±2.8% (2 s.d.) of the concentration. The solubilities at 970 bars for the granitic and phonolitic minimum melts are 2.88 ± 0.10 wt % and 5.01 ± 0.14 wt %, respectively. Both peralkaline and peraluminous granitic melts have higher H₂O solubilities than the 1 kbar P(H₂O) minimum melt and indicate the existence of minima in solubilities at molar (Na + K)/Al = 1 along joins of constant SiO₂ and (Na + K)/Al ratio, but varying in Na/(Na + K) (0.57, 0.69 molar), have the same solubility (2.88 ± 0.10, 2.74 ± 0.10 wt %). The compositional dependence of solubilities for peralkaline and peraluminous melts is not predicted by the solubility mechanism and calculation method of Burnham (1975, 1979, 1981) that was developed for metaluminous melts. The pressure dependence of solubility was investigated for a peralkaline melt with six determinations at four pressures from 970 to 1620 bars. The mole fraction of dissolved water (based on 8 moles of O) for this melt is proportional to the square root of the fugacity of water; the root mean square deviation from linearity is 1.4% and is equal to the analytical precision (1 s.d.). Our data for the haplogranite minimum are lower than some other determinations; however, the limitations of the various methods for determining solubilities in melts make detailed comparisons with our data difficult. The solubility of water in a Bishop Tuff melt was estimated from our measurements and used with direct measurements of H₂O in rhyolitic melt inclusions (4.9 ± 0.5 wt %, Druitt et al. 1982) to obtain the minimum pressure of water (2100 ± 300 bars) and minimum depth (8 ± 1 km) of crystallization.

INTRODUCTION

The solubility of water in granitic liquids of various compositions is a crucial property influencing the petrogenesis of intrusive and extrusive silicic rocks (e.g., Wyllie 1979; Burnham 1979). The dependence of solubility on melt composition is especially important during crystallization of granitic intrusions. Saturation of residual melts results in boiling phenomena closely associated with Cu-Mo porphyry systems and various pegmatites. The explosive eruption of magmas may be triggered by energy released during the expansion of a vapor phase evolved from melts (e.g., Sparks et al. 1977; Harris 1981*b*). Numerical models for the evolution of vapor (e.g., Wilson et al. 1980) from granitic liquids at low pressures require detailed knowledge

concerning the effects of composition and pressure on the solubility of water in such melts.

The solubility of water in granitic melts has been the topic of several experimental studies; however, there is no consensus on the results or their interpretation (e.g., Oxtoby and Hamilton 1978*b*; Day and Fenn 1982). These previous solubility determinations may be divided broadly into two categories: those using chemographic or phase equilibrium techniques (Burnham and Jahns 1962; Fenn 1973; Whitney 1975; Voigt et al. 1981) and those using a weight loss method (e.g., Goranson 1931, 1938; Bowen and Tuttle 1950; Yoder et al. 1957; Orlova 1963; Oxtoby and Hamilton 1978*a*). Solubility data obtained by such techniques were reviewed by Day and Fenn (1982), who discussed the assumptions and limitations associated with each method. A new capacitance manometric technique for micro-determination of H₂O evolved from vacuum fusion of glass (Harris 1981*a*) affords a method for measuring H₂O released from experimentally quenched vapor-saturated melts prepared as glass wafers free of fluid-filled vesicles. By using this technique we

¹ Manuscript received January 5, 1984; revised April 2, 1984.

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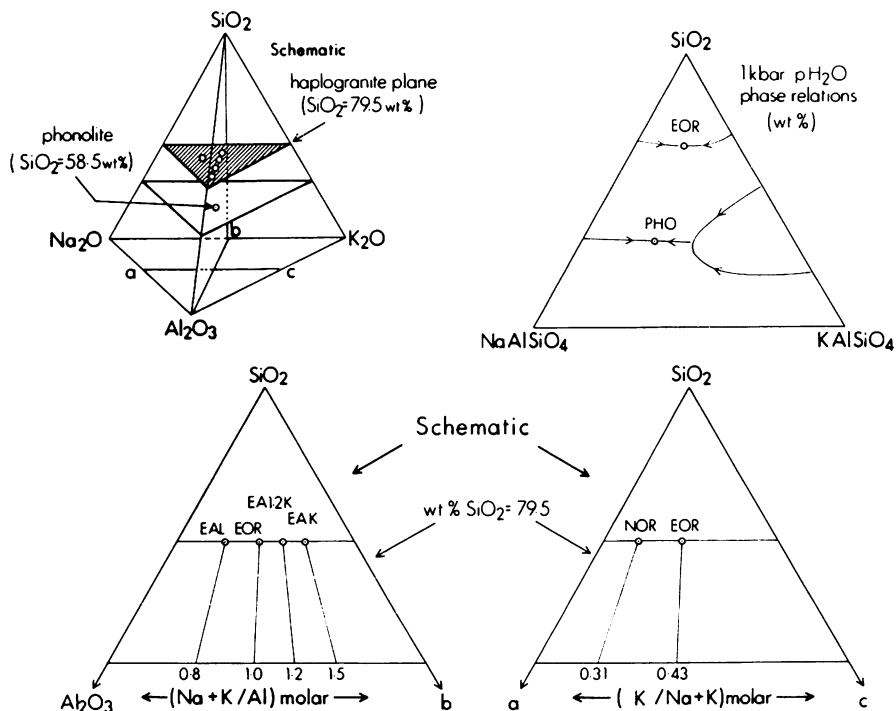


FIG. 1.—Melt compositions in the system K_2O - Na_2O - Al_2O_3 - SiO_2 (1 kbar phase equilibria data from Tuttle and Bowen (1958) and Hamilton and MacKenzie (1965)).

have determined the solubility of water in haplogranitic melts in the system K_2O - Na_2O - Al_2O_3 - SiO_2 .

The other unique aspects of our work are: (1) the study of haplogranitic melts to bridge the gap between melts of feldspar composition and natural granitic liquids; (2) the precise measurement of water solubility at up to four pressures in the range 1–2 kbars and determination of the pressure dependence for two compositions; and (3) investigation of the effects of increasing peralkalinity on water solubility at 1 kbar. The effects of composition and pressure on the solubility of H_2O are essential for understanding the petrogenesis of silicic plutonic and volcanic rocks.

EXPERIMENTAL AND ANALYTICAL PROCEDURES

The glasses used in the hydrothermal runs were synthesized from reagent grade Na_2CO_3 , K_2CO_3 , Al_2O_3 , and SiO_2 by using standard techniques described by Schairer and Bowen (1956). The compositions of the anhydrous glasses used as starting materials (fig. 1) were determined by electron microprobe.

Synthetic vapor-saturated melts were produced hydrothermally. Twenty-five to 50 mg of powdered glass were added to 10 to 20 mg of triple-distilled water in 3 mm Pt capsules and welded. The capsules were checked for possible leakage by testing for weight loss after drying in an oven at $110^\circ C$ for 15 minutes. Sealed capsules were loaded into cold-seal pressure vessels and raised to the desired run pressure by using an air-driven hydraulic pump. Pressurized capsules were heated to the run temperature ($800^\circ C$) by using Kanthal-wound resistance furnaces mounted concentrically about the vessels along a vertical axis. Temperatures were monitored with Chromel-Alumel thermocouples and the actual temperatures are believed accurate to $\pm 5^\circ C$. Pressures were measured with Bourdon-tube gauges previously calibrated with a Heise gauge. All run durations were 10^6 seconds. The charges were quenched isobarically by using compressed air. Pressure was maintained during the quench by opening the vessel to the pressure line and by pumping for the duration of the quench. Owing to the response time of the pump, there were slight

fluctuations in pressure during quenching. These fluctuations were monitored and are included in the pressure uncertainties listed in table 1. Except for water, the compositions of the hydrated glasses were determined by electron microprobe (table 1). All of the glass compositions, when recalculated H₂O-free and normalized to 100%, are within analytical error of their respective anhydrous starting compositions.

The quenched run products consisted of vesicular glass free of microlites. The vesicles contained liquid water and an enclosed vapor bubble and indicate that the charges were fluid-saturated during the experiments. It is extremely difficult to estimate accurately the volume fraction of vesicles in the run products. We avoided this difficulty by preparation of vesicle-free glass wafers.

Polished glass wafers free of occluded fluids were prepared manually from the experimental charges. The resulting wafers were typically 40–100 μm thick, depending upon the sizes and locations of vesicles within the glass wafer. Absence of occluded fluid was established by transmitted light microscopy at 200 \times magnification. The glass wafers were liberated from the Canada balsam mounting media by using acetone. The wafers were washed ultrasonically in acetone and checked again optically for purity at 200 \times magnification using transmitted and reflected light microscopy.

The amount of H₂O dissolved in the glass was determined by the vacuum fusion micro-manometric method described in Harris (1981a). The determinations of H₂O were made on duplicate glass wafers of different mass for each run, and the amounts reported are the duplicate means. The samples of glass ranged in mass from 0.020 to 0.600 mg. Sample masses were determined with a Cahn-G electronic microbalance and are considered precise to ± 0.003 mg. The analytical precision for water can be estimated by considering the variance of the relative deviations of the individual measurements about their duplicate mean. This method provides an estimate of $\pm 2.8\%$ of the concentration for the standard deviation of the duplicate means about their ensemble mean, if multiple pairs of determinations were made for each experimental charge. The overall reproducibility of our solubility measurements includes uncer-

tainties due to variation of run pressures, possible variable diminution of dissolved water during the quench, and analytical errors in the determination of H₂O (e.g., incomplete extraction of H₂O during vacuum fusion of samples, or loss of H₂O due to adsorption by the walls of the vacuum system). The overall reproducibility was estimated by repetition of experiments. Two solubility determinations have been repeated (table 1; EA1.2K at 970 bars, runs 80 and 38; EA1.2K at 1620 bars, runs 85 and 44). Both solubility determinations were within 0.06 wt % H₂O of their ensemble means, or about 1.4% of the concentration. The precision attained in these measurements exceeds that reported by Harris (1981a) owing to the greater precision of sample masses that was made possible in this study by using an electronic microbalance.

RESULTS

The glass compositions and solubility determinations at various pressures are given in table 1. The effects of pressure and composition are described next.

Pressure Dependence.—The effects of pressure on the solubility of water were determined for two melts with identical Na/(Na + K) ratios (0.57) and similar Si/Al ratios but with differing (Na + K)/Al ratios. Both liquid compositions were peralkaline. The experimentally determined solubilities, expressed as wt % H₂O in the hydrous liquid, are shown in table 1. The solubilities were recast as mole fractions by using the method of Burnham (1979) and fit (table 2) to linear equations as functions of the square root of the water fugacity (Burnham et al. 1969). The root mean square deviations from linearity are less than 1.4%. The equimolar solubility of water is slightly larger for the more peralkaline liquid at both 970 and 1620 bars $P(\text{H}_2\text{O})$. Hence, the derivatives with respect to square root of fugacity differ by 5%.

Effects of Na-K Exchange.—The effects of exchanging Na and K in haplogranitic melts may reveal whether there is a notable effect of alkali cation size on the solubility of water. Two experiments (runs 37 and 61) conducted at 970 bars show that the increase of the Na/K ratio from 1.34 at the eutectic to 2.23 caused no measurable change in solubility (maximum 0.14 wt %). Further research should determine whether this is also true for

TABLE 1.
EXPERIMENTAL DETERMINATIONS OF H₂O SOLUBILITY FROM MEASUREMENTS OF H₂O IN QUENCHED GLASS

Run No. Series	80 EA1.2K	38 EA1.2K	48 ^a EA1.2K	43 EA1.2K	85 EA1.2K	44 EA1.2K	36 EAK	83 EAK	37 EOR	61 NOR	60 PHO	58 ^a EAL
Pressure and fugacity of H ₂ O during experiments at 800°C												
Pressure ^b	970	970	1160	1450	1620	1630	970	1620	970	970	970	970
f _{H₂O} ^b	825	825	965	1175	1297	1305	825	1297	825	825	825	825
Microprobe ^c analyses and vacuum fusion determinations ^d of H ₂ O in glass												
SiO ₂	75.00	74.24		74.84	73.64	73.99	74.63	72.27	75.27	75.67	54.67	
Al ₂ O ₃	9.80	9.73		9.74	9.66	9.57	9.19	8.93	11.20	11.15	22.82	
Na ₂ O	4.02	4.08		4.08	3.96	4.10	4.70	4.41	3.79	4.53	10.56	
K ₂ O	4.69	4.55		4.68	4.59	4.60	5.33	5.25	4.32	3.09	5.08	
H ₂ O	3.38	3.29	3.72	4.36	4.67	4.55	3.44	4.98	2.88	2.74	5.01	3.46
Total	96.89	95.89		97.70	96.52	96.81	97.29	95.84	97.46	97.18	98.14	
Glass compositions recalculated to 100% totals												
SiO ₂	77.49	77.53	77.32	76.68	76.43	76.54	76.79	75.58	77.29	77.92	55.76	75.62
Al ₂ O ₃	10.13	10.16	10.06	9.98	10.03	9.90	9.46	9.34	11.50	11.48	23.28	13.24
Na ₂ O	4.15	4.26	4.15	4.18	4.11	4.24	4.84	4.61	3.89	4.67	10.77	3.52
K ₂ O	4.85	4.75	4.75	4.80	4.76	4.76	5.48	5.49	4.44	3.18	5.18	4.16
H ₂ O	3.38	3.29	3.72	4.36	4.67	4.55	3.44	4.98	2.88	2.74	5.01	3.46
Moles of cations per 100 g of melt												
Si	1.290	1.290	1.287	1.276	1.272	1.274	1.278	1.258	1.286	1.297	.928	1.259
Al	.199	.199	.197	.196	.197	.194	.186	.183	.226	.225	.457	.260
Na	.134	.138	.134	.135	.133	.137	.156	.149	.126	.151	.348	.114
K	.103	.101	.101	.102	.101	.101	.116	.117	.0942	.0676	.110	.088
H	.375	.365	.413	.484	.519	.505	.382	.553	.320	.304	.556	.384
Total	2.101	2.093	2.132	2.193	2.222	2.111	2.118	2.260	2.0522	2.0446	2.399	2.105
Na/Na + K	.565	.577	.570	.570	.568	.576	.574	.560	.572	.691	.760	.564
Na + K/Al	1.19	1.20	1.19	1.21	1.19	1.23	1.46	1.45	.97	.97	1.00	.777
Si/Al	6.48	6.48	6.53	6.51	6.46	6.57	6.87	6.87	5.69	5.76	2.03	4.84

X_w^m	X_w^m based upon solubility measurements and the model of Burnham (1979)												
	.363	.356	.386	.426	.443	.437	.372	.465	.322	.308	.379	.371	
				X_w^m calculated from Burnham's (1979) paper									
				.454	.465	.465	.409	.465	.409	.409	.409	.409	
X_w^m	.409	.409	.423	.293	.293	.293	.299	.299	.289	.285	.208	.296	
MW	293	293	293	1.58	1.53	1.53	1.79	1.53	1.79	1.79	1.79	1.79	
In K	1.79	1.79	1.72										

^a Microprobe analyses of hydrous products are not available for runs 48 and 58.

^b Pressure and fugacity are given in bars; pressure uncertainty is ± 10 , -30 bars. Fugacity was calculated from Burnham et al. 1969.

^c Glasses were analyzed by D. B. Dingwell with the University of Alberta ARL SEMO microprobe using 15 kV accelerating voltage and a 4 nanoamp probe current in the energy dispersive mode; a beam raster of 20×20 micrometers and a continuously moving sample were used to prevent alkali loss. Standards used were quartz, albite, and sanidine and the data were reduced using EDATA2 (Smith and Gold, 1979). Analyses have the following 3 standard deviation uncertainties quoted as percentages of the amount present: SiO₂, 0.69%; Al₂O₃, 1.78%; K₂O, 2.58%; Na₂O, 4.76%.

^d Determinations of H₂O (D. M. Harris) are duplicate means for individual experiments; reproducibility of solubility determinations is about ± 0.06 wt % (see runs 80, 38, 85, and 44).

other liquid compositions in the vicinity of the ternary eutectic.

Dependence of Solubility on the Alkali/Aluminum Ratio.—The equimolar solubilities of water in four liquids that differ in alkali/aluminum ratio were determined at 970 bars and are summarized in table 3. For the range of silica-saturated compositions investigated, the equimolar solubility at 970 bars water pressure increases linearly with increasing alkali to aluminum ratio, and the root mean square deviation from linearity is about 1.4%. However, the solubility of water in the peraluminous liquid (table 1, run 58) does not lie on the extension of this line to the peraluminous field. This suggests the existence of minima in the solubility of H₂O at (Na + K)/Al = 1 for melts along joins of constant molar SiO₂ in anhydrous melts.

Solubility in a Phonolite Liquid.—The 970 bar solubility of H₂O in the phonolite liquid exceeds that of all the granitic melts investigated. The phonolite composition, which has alkali/aluminum equal to 1 and very much less silica than those runs listed in table 3, does not satisfy the linear relation obtained in table 3.

DISCUSSION

Comparison with Earlier Determinations.—The determinations of water solubility in haplogranitic liquids from this study are shown (fig. 2) in relation to other determinations at low pressures. Although the results are generally similar to those of earlier workers, the effects of pressure and composition on the water solubilities are evident in our results and may help to explain some of the differences shown in figure 2.

All previous measurements of water solubility in granitic melts (except those of Khitarov et al. 1959, 1967) have employed one or both of the chemographic and weight-loss techniques. The chemographic technique involves the construction of isobaric T-X or rock-water pseudobinary phase diagrams (see Burnham and Jahns 1962 for discussion). In these diagrams the intersection of the water-saturated liquidus with the divariant L - L + V or L - L + C curve is determined. The intersection gives the concentration of water equal to the solubility limit. However, according to Day and Fenn (1982), water contents may be overestimated by as much as 1.0

TABLE 2.
 EFFECT OF PRESSURE ON THE SOLUBILITY OF H₂O IN TWO LIQUIDS

Run No.	Pressure bars	f _{H₂O} bars	f _{H₂O} ^{1/2} bars ^{1/2}	X _w ^m meas.	X _w ^m calc.	Residual
EA1.2K 970–1630 bars						
80	970	825	28.72	.363	.356	–.007
38	970	825	28.72	.356	.356	.000
48	1160	965	31.06	.386	.385	–.001
43	1450	1175	34.28	.426	.425	–.001
85	1620	1297	36.01	.443	.446	.003
44	1630	1305	36.12	.437	.447	.010
$X_w^m = 0.003 + 0.0123 (f_{H_2O}, \text{bars})^{0.5}$ r.m.s. error of fit = 0.005						
EAK						
36	970	825	28.72	.372	.371	–.001
83	1620	1297	36.01	.465	.465	.000
$X_w^m = 0.0006 + 0.0129 (f_{H_2O}, \text{bars})^{0.5}$ r.m.s. error of fit = 0.0006						

NOTES.—Solubility data and the origin were fit to linear equations by the method of least squares.

wt % by using the L – L + V or “dimple” technique. At pressures below two kbar the L – L + C technique requires the careful addition of small amounts of water to experimental charges and the execution of water-undersaturated experiments on melts for which equilibrium is difficult to achieve.

In the weight-loss technique, water concentrations are obtained by comparison of the weight of H₂O added to the charge before the experiment with the weight of the post-run hydrous glass after the charge has been pierced and heated to 110°C (Hamilton et al. 1964). Because the charges are generally oversaturated with H₂O, the run products, especially below 2 kbar, are vesicular, and the amount of H₂O in the vesicles must be

subtracted from the total H₂O determined by weight loss (Burnham and Jahns 1962). Alternatively, if the vesicles can be ruptured by stepwise heating without loss of any H₂O from the glass (e.g., Oxtoby and Hamilton 1978a), then it may be possible to determine the solubility from weight loss measurements during heating of vesicular glass. Uncertainties for weight loss techniques are predictably rather large.

In contrast, the experimental, sample preparation, and microanalytical techniques used in the present work permit the solubility to be determined with a variation of about ±0.10 wt % (2 s.d.) in replicate experiments at pressures from 1.0–2.0 kbars. The principal advantage over the previously mentioned tech-

TABLE 3

DEPENDENCE OF THE EQUIMOLAR SOLUBILITY OF WATER IN MELTS ON THE ATOMIC RATIO OF TOTAL ALKALIS TO ALUMINUM AT 970 BARS H₂O PRESSURE

Run	Series	SiO ₂ wt %	(Na + K)/Al molar	X _w ^m
61	NOR	77.92	0.97	.308
37	EOR	77.29	0.97	.322
80	EA1.2K	77.49	1.19	.363
38	EA1.2K	77.53	1.20	.356
36	EAK	77.53	1.46	.372
$X_w^m = 0.200 + 0.124 (\text{Na} + \text{K})/\text{Al}$ r.m.s. error of fit = 0.0110				

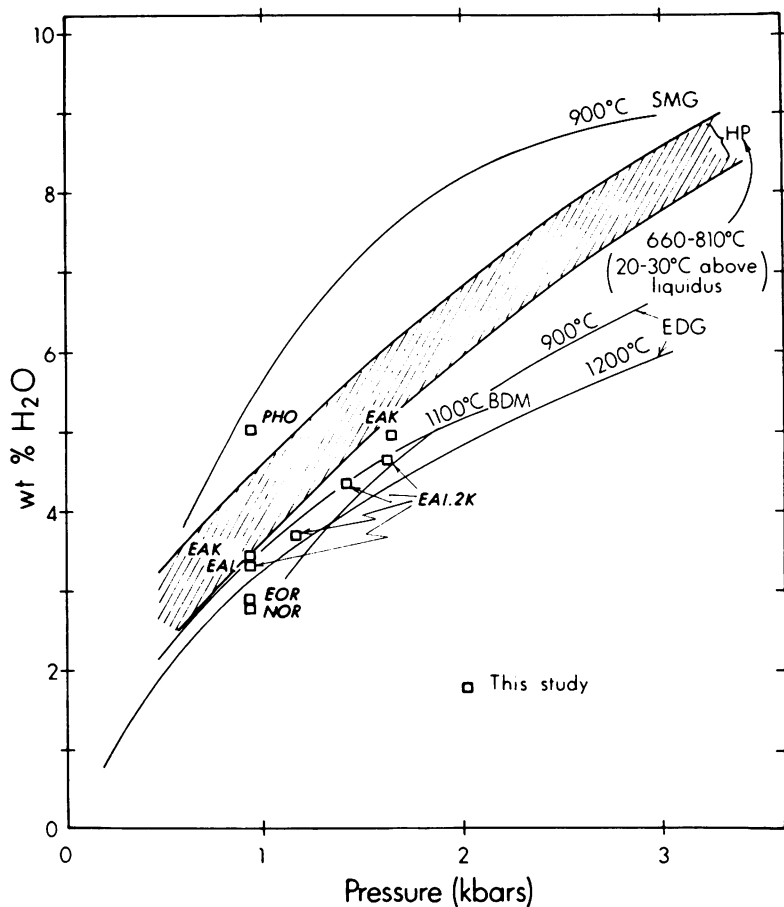


FIG. 2.—Solubility determinations in natural and synthetic granites. (BDM, Beinn an Dubhaich granite, Oxtoby and Hamilton, 1978*b*; SMG, Stone Mountain granite, Goranson, 1931; EDG, El'Dzurtinskii granite, Khitarov et al. 1959; HP, Harding pegmatite, Burnham and Jahns, 1962). Data from this study are for $T = 800^{\circ}\text{C}$.

niques are the high precision and the fact that the measured water is wholly derived from the glass. Other significant advantages are that the technique does not require multiple runs, precise determination of a phase boundary, or precise measurement of the amount of water added to an experimental charge.

From the comparison of the present data and previous work for granitic melts depicted in figure 2, two major conclusions can be drawn. The first conclusion is that our results at 800°C and those of Khitarov et al. (1959) for the El'Dzurtinskii granite at 900°C are approximately similar. On the other hand, the data of Oxtoby and Hamilton (1978*b*) on the Beinn an Dubhaich granite, although similar in magnitude, were obtained at 1100°C . The second conclusion is that our solubilities are

lower than those of Burnham and Jahns (1962) on the Harding pegmatite at temperatures between 660 – 810°C and those of Goranson (1931) on the Stone Mountain granite at 900°C . The Stone Mountain, El'Dzurtinskii, and Beinn an Dubhaich granites are approximately similar in alkali/aluminum ratios, but they vary considerably in their K/Na ratios. The Harding pegmatite is distinct in having high contents of Rb, Li, and F (see Burnham 1979). Oxtoby and Hamilton (1978*b*; see also Voigt et al. 1981) showed that the equimolar solubility of water is significantly greater in $\text{NaAlSi}_3\text{O}_8$ melt than in KAlSi_3O_8 melt. We conclude that compositional variations and measurement techniques are causes for these differences. These problems have been reviewed recently by Day and Fenn (1982). We

wish only to emphasize that the equimolar solubility of water is enhanced in increasingly peralkaline melts and this variation is not predicted by the model of Burnham (1979). The model of Burnham (1981) does reproduce our solubility determinations for PHO at 970 bars (wt % H₂O calc. = 5.02); and EA1.2K at 1620 bars (wt % H₂O calc. = 4.68) but overestimates all the other melt solubilities in table 1. Further discussion on solubilities in feldspathic melts is outside the scope of this paper which pertains to haplogranitic melts and natural granitic melts.

As far as we are aware, data on solubilities in phonolitic melts are not available in the literature. The fact that phonolitic melts show strongly enhanced solubility of H₂O over SiO₂-saturated compositions indicates the potential for a far more dramatic role for H₂O in the crystallization and emplacement of phonolitic magmas. Generally, it is clear that a range of water solubilities are to be expected in granitic melts of variable composition. These differences must be taken into account in the geochemical modeling of processes such as pegmatite formation, hydrothermal activity, crystallization, volatile exsolution, and eruptive mechanisms for granitic magmas.

GEOLOGICAL APPLICATION

The experimentally determined solubility of water in haplogranitic melts and determinations of water in rhyolitic melt inclusions in phenocrysts may be used to estimate the minimum pressure of crystallization for the Bishop Tuff. Druitt et al. (1982) determined the concentration of water in rhyolitic glass in quartz from the basal Plinian airfall unit of the Bishop Tuff to be $4.9 \pm 0.5\%$. A vacuum fusion gas analyzer method was used. The glass contained 77.6% SiO₂, 13.3% Al₂O₃, 0.6% FeO, 0.5% MgO, 0.3% CaO, 3.9% Na₂O, and 4.6% K₂O (A. T. Anderson, personal comm.). The gram molecular weight of the anhydrous melt (286 g) was calculated by the method of Burnham (1979). The Na/(Na + K) and Si/Al ratios are 0.563 and 4.95, respectively, and near to or within the range investigated. The alkalis Na and K account for about 90 mole % of the exchangeable cations, and the range of exchangeable cations to aluminum is 0.96. Therefore, we estimate the equimolar solubility for the stated compo-

sition to be $X = 0.0111 \times (f(\text{H}_2\text{O}), \text{bars})$. Accordingly, the pre-eruption fugacity of water estimated from the $4.9 \pm 0.5\%$ in the melt inclusions at 800°C is 1630 ± 200 bars. The pressure of water is estimated to have been 2100 ± 200 bars. The implied minimum depth of origin of these phenocrysts is 7.8 ± 1.1 km. The known presence in these inclusions of other components such as CO₂ (A. T. Anderson, personal comm.) would increase the minimum pressures and depths of crystallization. Druitt et al. (1982) reported a minimum concentration of water (2 wt%) in rhyolitic inclusions in quartz phenocrysts from the last erupted Mono Lobe. Our solubility measurements suggest minimum fugacities and pressures of H₂O of 480 and 530 bars, respectively. The direct measurements of pre-eruption water contents (Druitt et al. 1982) and solubility of water in related haplogranitic melts at 1–2 kbar and 800°C (this study) yield more direct estimates of pressures than are available from mineral equilibria (Hildreth 1979).

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