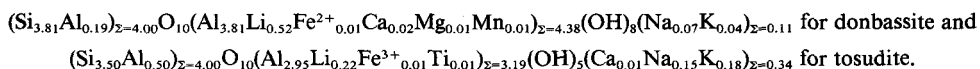


LITHIUM-BEARING DONBASSITE AND TOSUDITE FROM ECHASSIÈRES, MASSIF CENTRAL, FRANCE

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Abstract—Lithium-bearing donbassite and tosudite were found in veins in hydrothermally altered granite (Beauvoir granite) in the northern part of the Massif Central, France. The two minerals are characterized by their high Li contents and low Mg and Fe contents; their structural formulae are:



These chemical compositions indicate that the donbassite is an intermediate member of the donbassite-cookeite solid solution series and that the tosudite consists of interstratified Li-donbassite and beidellite. Both Li-bearing minerals show thermal behavior distinct from those previously reported for dioctahedral chlorite and tosudite.

Petrographic investigation of drill cuttings from the Echassières area indicates that the two minerals were formed in an intermediate stage of hydrothermal alteration following an early stage characterized by formation of muscovite ($2M_1$) at $>350^\circ\text{C}$ and before the latest stage characterized by deposition of kaolinite and randomly interstratified illite/smectite at $<100^\circ\text{C}$. Moreover, tosudite occurs in the upper part of the granite, whereas donbassite is restricted to the lower part, suggesting the formation of tosudite at lower temperatures.

Key Words—Donbassite, Granite, Hydrothermal alteration, Infrared spectroscopy, Lithium, Petrography, Tosudite, X-ray powder diffraction.

INTRODUCTION

Aluminous chlorite and related interstratified minerals occur in many geological environments, including sedimentary (Schultz, 1963; Müller, 1967), pegmatitic (Figueiredo Gomes, 1967; Foord *et al.*, 1986), and hydrothermal environments (Hayashi and Oinuma, 1964; Henmi and Yamamoto, 1965; Sudo and Sato, 1966; Fujii *et al.*, 1971; Fransolet and Bourguignon, 1978). According to the Nomenclature Committee of the Association Internationale Pour l'Etude des Argiles (Bailey, 1980), Al-chlorites having two dioctahedral sheets merit the name donbassite, whereas those having a dioctahedral 2:1 layer and a trioctahedral interlayer are called sudoite (Mg-dominant) and cookeite (Li-dominant). The three chlorites also exist as component layers in regularly interstratified chlorite/smectite, which is called tosudite (Bailey *et al.*, 1982).

Al-chlorite occurs as a vein-filling in intensively altered parts of the Echassières granitic cupola, Massif Central, France. Here, it is characterized by a significant content of Li_2O and occurs separately from tosudite. Li-bearing dioctahedral chlorite and tosudite have been reported by many workers (e.g., Sudo *et al.*, 1954; Shimoda, 1969; Nishiyama *et al.*, 1975; Ichikawa and Shimoda, 1976; Creach *et al.*, 1986; Foord

et al., 1986); however, unsolved problems remain, especially concerning their crystallochemical properties and the physicochemical conditions of their formation.

In this study, the modes of occurrence of donbassite and tosudite in the Echassières granitic cupola are described and the mineralogical properties of these minerals are reported. Detailed studies were made by X-ray powder diffraction (XRD), electron microprobe, and infrared spectroscopy (IR). An additional purpose of the present work was to clarify the difference in the physicochemical formation conditions between donbassite and tosudite.

GEOLOGICAL SETTING

The Echassières area is located in the northern part of the Massif Central, France (Figure 1) in which the Carboniferous Beauvoir Granite is widely distributed. In this area, drill hole G.P.F. ECHA No. 1 was sunk to a depth of 900 m, and the following lithologic units were encountered (Cuney *et al.*, 1986): mica-schist roof rocks containing a quartz-ferberite stockwork (to ~100 m depth); albite-lepidolite granite (Beauvoir Granite) containing disseminated Sn, W, Li, Nb, Ta, and Be minerals (from 100- to 789-m depth), and mica schist identical to the roof rocks (789- to 900-m depth).

The unaltered Beauvoir Granite is leucocratic and has the following average modal composition: quartz, 15–25%; albite (An_{0-3}), 40–70%; potassium feldspar, 5–15%; lepidolite, 10–25%; and topaz, 1–5%. Lepid-

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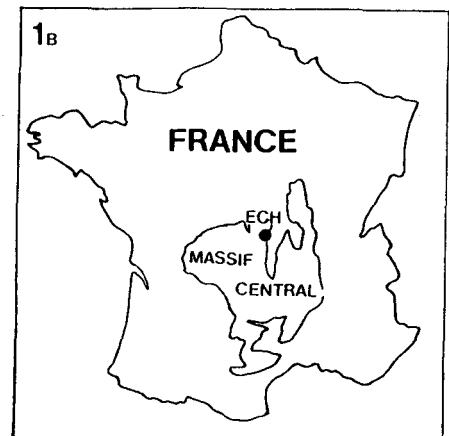
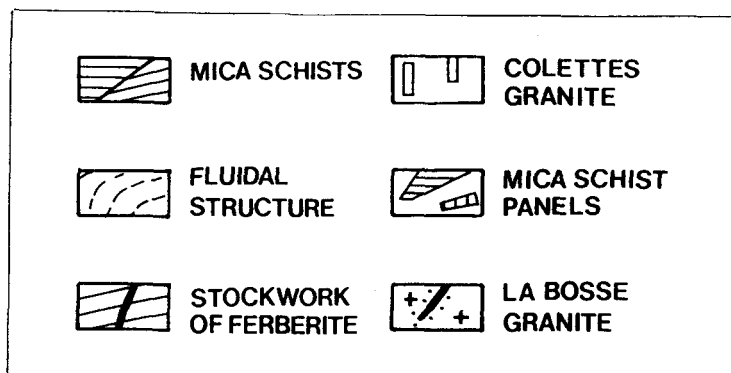
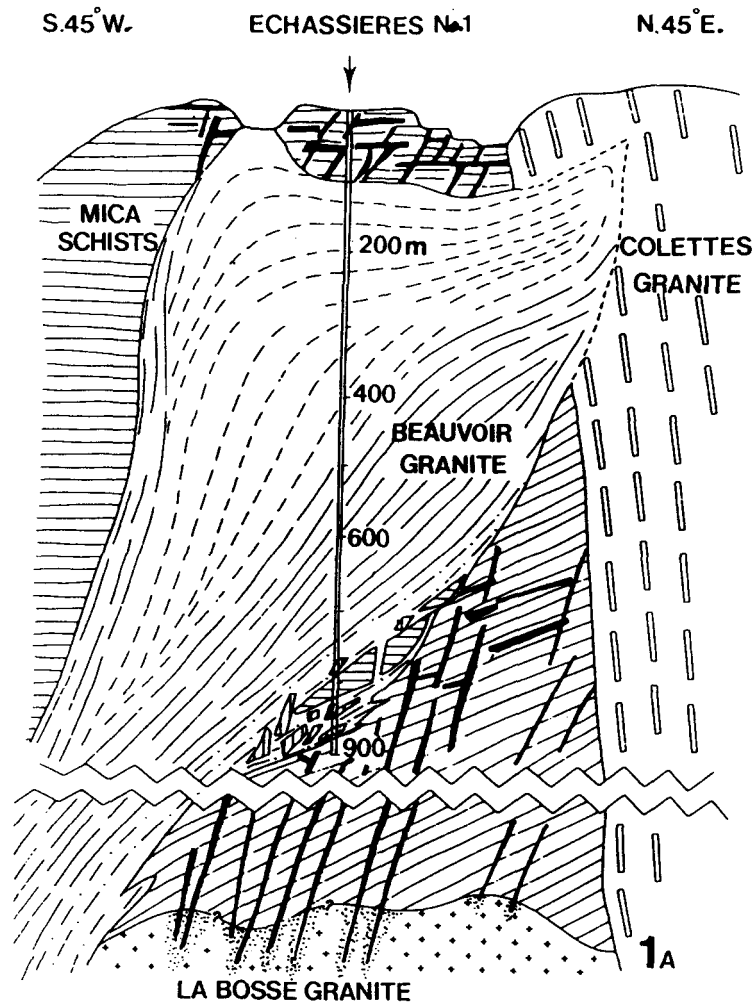


Figure 1. Schematic cross section (1A) of the Beauvoir albite-lepidolite granite based on data obtained in drilling hole GPF-ECH No. 1 (after Cuney *et al.*, 1986) and the situation map in France (1B).

olite decreases with depth in the granite (Cuney *et al.*, 1986).

The pelitic mica schist of the roof rocks, which was contact-metamorphosed by the Beauvoir Granite and other granitic intrusions, contains the mineral assemblage biotite + muscovite + garnet + staurolite.

EXPERIMENTAL

Fourteen samples were selected from the G.P.F. ECHA No. 1 drill cuttings. After clay minerals were located under an optical microscope, they were separated by using a microsampling method (Beaufort *et al.*, 1983). Powders and oriented samples were examined using X-ray powder diffraction techniques on a Philips PW 1730 diffractometer (40 kV, 40 mA, CoK α radiation) equipped with a linear location detector. Oriented samples were examined after four treatments: (1) air-drying at room temperature, (2) ethylene glycol (EG) saturation, and (3) heating at 350°C for 2 hr then (4) at 550°C for 2 hr. XRD patterns of donbassite and tosudite were compared with those calculated by the NEWMOD program developed by R. C. Reynolds, Dartmouth College, Hanover, New Hampshire in 1985. In addition, $d(060)$ values were determined on randomly oriented powders for donbassite and tosudite minerals.

Transmission infrared (IR) spectra were recorded using KBr disks (0.5 mg specimen in 300 mg KBr) on a Beckmann IR 4240 spectrophotometer over the 4000–300-cm⁻¹ frequency region.

Electron microprobe analyses were carried out with a Cameca MS 46 microprobe equipped with an ORTEC energy-dispersive X-ray analyzer. The accelerating voltage was 15 kV, the sample current was 1.5 nA, and the counting time was 120 s. According to Velde (1984), these conditions are necessary to prevent the loss of alkalis and the breakdown of clay minerals. Lithium was analyzed separately by atomic absorption spectrophotometry (AA) with a Perkin-Elmer 2380.

RESULTS

Occurrence

Three different stages of hydrothermal alteration have been identified in the Beauvoir Granite (Dudoignon and Meunier, 1984).

1. After the magmatic stage, the whole granite body was subjected to an intensive hydrothermal alteration, which is characterized by veins of muscovite ($2M_1$) crystals. Muscovite also replaces the primary minerals of the granite.
2. Veins of donbassite and tosudite crosscut the early muscovite veins and disseminated muscovite.
3. A final hydrothermal event again affected the whole granite body and is characterized by the paragenesis:

Table 1. Clay mineral assemblages found in selected intervals of the G.P.F. ECHA No. 1 drill cuttings as determined by X-ray powder diffraction.

Depth (m)	Donbassite	Tosudite	I/S	Mica ¹	Kaolinite
306.65 ²		**	**	tr	*
309.70	***			tr	
366.40		**	*	tr	tr
408.10	***		**	* ($1M$)	*
456.65		*		tr	
479.60	***		*	tr	tr
479.65	**			tr	
479.70	**		*	* ($1M$)	*
479.80	***		*	* ($1M$)	*
487.80	**		*	tr	
706.85	**			tr	
706.90	***			tr	
707.40	**			* ($2M_1$)	
708.10	***			* ($1M + 2M_1$)	

I/S = random illite/smectite mixed-layer (R=0).

***, **, * = decreasing abundance.

tr = trace.

¹ Mica represents illite (polytype $1M$) or muscovite (polytype $2M_1$).

² This sample also contains pyrophyllite.

illite ($1M$) \pm random illite/smectite (>60% smectite) \pm kaolinite.

Table 1 shows the distribution of the clay minerals in the bulk rocks in the ECHA No. 1 deep drill hole as a function of depth. Donbassite occurs in veins in strongly altered rocks from the lower part of the drill hole; the veins seem to concentrate in intensively argillized parts (479.60, 479.80 m depths) and crumbled ones (707.40 m depth). Based on optical microscopic observations, donbassite occurs not only in small veins (1 mm wide), but also as a replacement of primary minerals such as lepidolite and albite adjacent to the veins (479.60, 479.80 m depths). Donbassite is locally replaced by random illite/smectite, illite, or kaolinite, which were formed by the latest hydrothermal event, as shown in samples from 408.10, 479.70, and 479.80 m depths (Table 1).

Tosudite occurs in veins (Table 1). In the sample from 306.65 m depth, tosudite coexists with smectite, illite, pyrophyllite, and kaolinite (Figure 4A). Tosudite replaces the primary minerals of the host granite and is also locally altered to illite/smectite, illite, or kaolinite. Although the occurrences of tosudite and donbassite appear to overlap in the transitional zone (309.70 to 456.65 m), cross-cutting relationships between tosudite and donbassite veins were not recognized optically.

Mineralogy of donbassite

X-ray powder diffraction. Figure 2 shows XRD patterns of chlorite from the sample from 708.10 m depth. Al-

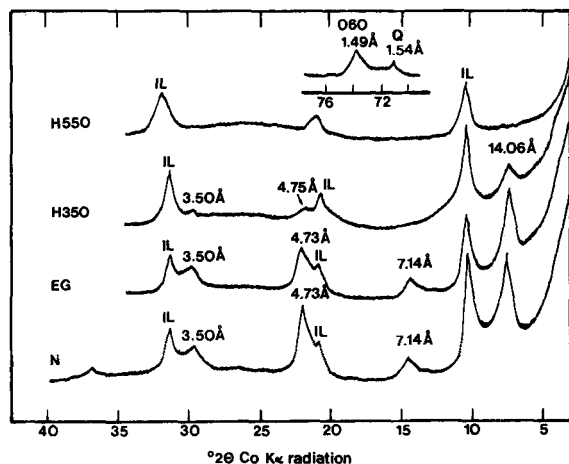


Figure 2. X-ray powder diffraction patterns of Echassières donbassite. N = natural; EG = ethylene glycol saturated; H350 = heated to 350°C; H550 = heated to 550°C; IL = illite; Q = quartz.

though this sample is contaminated by illite, a series of reflections indicative of a chlorite mineral are present at 14.06, 7.14, 4.73, 3.50, and 2.84 Å. The d-values of these reflections seem to be somewhat smaller and the peaks seem to be broader compared with those in typical di/dioctahedral chlorite. The d-values did not change after EG-saturation. Upon heating at 350°C, d(001) decreased slightly and the 002 reflection disappeared; after heating at 550°C, all chlorite reflections disappeared, but the illite reflections remained intact. This behavior is distinct from that of sudoite for which the intensity of the 001 reflection increases at higher temperatures to more than the original (Müller, 1967). The d(060) value for the mineral is 1.49 Å, which is consistent with the dioctahedral structure of this mineral.

Infrared absorption. An IR spectrum of the chlorite is given in Figure 3 and is compared with IR data for sudoite (Hayashi and Oinuma, 1965, 1967) and Li-donbassite (Van Oosterwyck-Gastuche and Deliens, 1968) in Table 2. Peaks in the 4000–3200-cm⁻¹ region (Figure 3) were assigned to OH-stretching vibrations and this chlorite shows different spectral features from those of trioctahedral chlorites. The spectrum is typical of dioctahedral chlorites according to Hayashi and Oinuma (1967). The peaks at 3645 and 3660 cm⁻¹, which may be attributed to Al–OH stretching vibrations, are similar to those of Li-donbassite (Table 2). Hayashi and Oinuma (1965) reported that the peak near 550 cm⁻¹ shifts according to the octahedral Al content in the chlorite structure. As indicated in Table 2, this chlorite shows a peak at 535 cm⁻¹, a wavenumber that is distinctly smaller than the value for sudoite (555 cm⁻¹) and similar to that of Li-donbassite (538

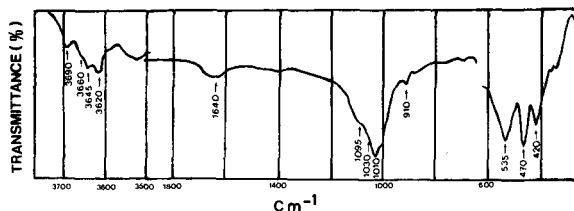
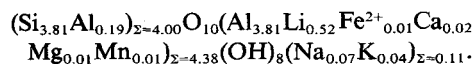


Figure 3. Infrared absorption spectrum of the Echassières donbassite.

cm⁻¹). The present IR data suggest that the chlorite is a donbassite-like species.

Chemical analysis. An average (of 10 determinations) chemical analysis is given in Table 3 and is representative of chlorite from two modes of occurrence: in veins and in rock adjacent to the veins (samples at 309.70, 479.60, and 707.40 m depths, Table 1). The structural formula calculated on the basis on O₁₀(OH)₈ is:



Comparison of the chemical composition of this Al-chlorite with those previously reported (Table 3) shows that Echassières Al-chlorite is donbassite. Moreover, the Echassières donbassite is notable because of its high Li-content in the octahedral layers, an assignment previously made by Aleksandrova *et al.* (1972). The Echassières donbassite shows less substitution of Al for Si in the tetrahedral layers compared with that from Novaya Zemlya, but is chemically very similar to the donbassite from Itaya.

Mineralogy of tosudite

X-ray powder diffraction. Figure 4A shows XRD patterns of clay minerals from the sample at 306.65 m depth. This material contains an interstratified mineral having a reflection of 28.65 Å, in addition to significant amounts of impurities such as smectite having a minor illite component, illite, kaolinite, and pyrophyllite.

The 28.65-Å reflection expanded to about 31 Å after EG-saturation and disappeared upon heating at 550°C. The d(060) value of the mineral is 1.50 Å, indicating a dioctahedral structure. Therefore, the mineral can be identified as tosudite composed of regularly interstratified dioctahedral chlorite and smectite. This identification was confirmed by comparing the experimental XRD patterns with theoretically calculated XRD patterns using Reynolds program (NEWMOD), as shown in Figure 4B.

Thermal behavior. In terms of the thermal behavior of tosudite, Ichikawa and Shimoda (1976) pointed out that a regular series of the basal reflections of tosudite remains unchanged even after heating at 550°C. The value of d(001) of the Echassières tosudite shrank, how-

Table 2. Comparison of infrared spectral data for Al-chlorites.

Echassières ¹ donbassite	Li-donbassite ²	Sudoite ³
420	422	
	454	
470	480	475
535	538	528
	560	555
		692
		825
910	915	
1010		1004
1030	1028	
1095	1164	
		3340
1640		
	3540	3520
3620	3620	3620
3645	3635	
3660	3652	
3690	3700	

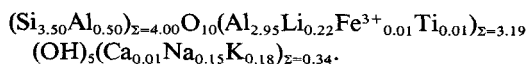
¹ Echassières donbassite.

² Li-donbassite from Atondo, Mozambique (Van Oosterwyck-Gastuche and Deliens, 1968).

³ Sudoite from Kamikita, Japan (Hayashi and Oinuma, 1965, 1967).

ever, to 9.60 Å at this temperature; this behavior is similar to that of the Montebras Li-bearing tosudite (Creach *et al.*, 1986).

Chemical analysis. An average of 10 microprobe analyses is shown in Table 4. A structural formula of tosudite calculated on the basis of O₁₀(OH)₅ is:



According to Nishiyama *et al.* (1975), the total iron determined as FeO by electron microprobe is expressed as Fe₂O₃. Compared with the literature data for tosudite (Table 4), the Echassières tosudite contains more Si, Na, and K and less Al, Fe, and Mg on the average than the others. The mineral is distinguished by its elevated Li-content like the others. Nishiyama *et al.* (1975) pointed out that Li is in the octahedral layers of the chlorite component. The chemical analysis suggests that the Echassières tosudite is a regularly interstratified mineral composed of Li-bearing dioctahedral chlorite and beidellite layers. The Li-bearing dioctahedral chlorite layer appears to be nearly identical to the Li-bearing donbassite mentioned above.

DISCUSSION

Chemically, the Echassières donbassite appears to be very close to the di/dioctahedral chlorite sub-group (Bailey, 1975); however, its Li₂O content (about 1.6%) suggests that the Echassières donbassite is intermediate in Li content between end-member donbassite and end-member cookeite. The Echassières tosudite also con-

Table 3. Chemical analyses of Echassières donbassite and two Li-donbassites.

Oxide	Wt. %			
	ECH ¹	SD ²	IT ³	NOV ⁴
SiO ₂	47.36	(1.85)	43.43	35.36
Al ₂ O ₃	42.09	(2.30)	39.21	47.35
FeO ⁵	0.21	(0.10)	0.15	0.54
MgO	0.06	(0.05)	0.13	0.67
TiO ₂	0.11	(0.06)	—	0.14
MnO	0.07	(0.07)	—	—
CaO	0.27	(0.16)	0.47	0.45
Na ₂ O	0.43	(0.16)	1.41	0.15
K ₂ O	0.39	(0.39)	0.49	0.25
Li ₂ O ⁶	1.60	—	1.43	0.76
Total ⁷	92.59	(1.15)	86.72	85.67

Cations per 14 O (anhydrous)			
Si	3.81	3.79	3.12
Al(IV)	0.19	0.21	0.88
Al(VI)	3.81	3.83	4.04
Fe ²⁺	0.01	0.01	0.03
Mg	0.01	0.02	0.08
Ti	0.01	—	0.01
Mn	0.01	—	—
Ca	0.02	0.04	0.04
Na	0.07	0.24	0.03
K	0.04	0.05	0.03
Li	0.52	0.50	0.27

¹ ECH = Echassières.

² SD = standard deviation.

³ IT = Itaya, Japan (Henmi and Yamamoto, 1965).

⁴ NOV = Novaya Zemlya, U.S.S.R. (Loskutov, 1959).

⁵ Fe total determined as FeO.

⁶ Li₂O determined by atomic absorption spectroscopy (3 determinations with a standard deviation of 0.05%).

⁷ Total does not include water.

tains a considerable amount of Li₂O (0.7%) and very small amounts of Fe₂O₃ and MgO.

Sudo (1978) pointed out the possible existence of continuous solid solution for tosudite minerals between donbassite and cookeite insofar as the Li contents were concerned. The Li contents of Al-chlorites and tosudite minerals from the literature in addition to the present data are plotted against the total octahedral cation content per half cell unit in Figure 5. Line A connects the ideal cookeite and ideal donbassite compositions. A few points (samples a to e, and Echassières donbassite) are in the intermediate area of line A. These samples show characteristically small Fe + Mg contents in the octahedral sheets; such Li-bearing chlorites can be regarded as intermediate species in the true donbassite-cookeite solid solution. Several Li-bearing chlorites other than those mentioned above are also known; however, they contain considerable amounts of Fe + Mg as well as Li and therefore may be identified as Li-bearing sudoite.

In terms of the Li content in tosudite, if it can be assumed that the smectite layer in the interstratifica-

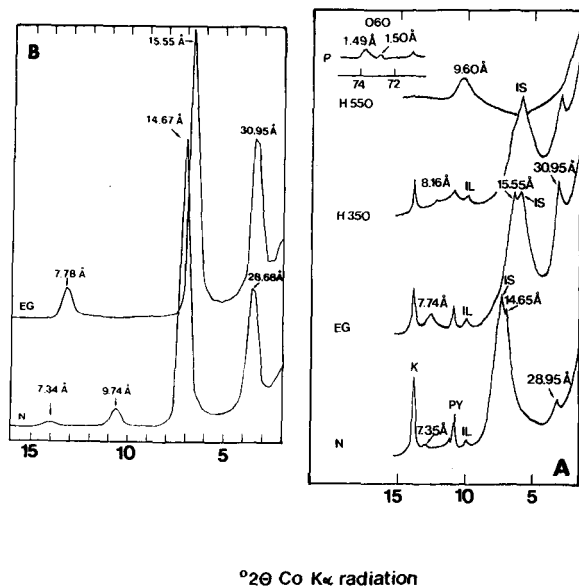


Figure 4. (A) X-ray powder diffraction patterns of Echassières tosudite. N = natural; EG = ethylene glycol saturated; H350 = heated to 350°C; H550 = heated to 550°C. IS = random illite/smectite mixed layer (R=0); IL = illite; PY = pyrophyllite; K = kaolinite; Q = quartz. (B) Calculated X-ray powder diffraction patterns of tosudite (regular interstratification of donbassite and dioctahedral smectite) using the program NEWMOD of Reynolds (1985). N = natural; EG = ethylene glycol saturation.

tion has ideally an octahedral cation content of 2.0 and does not contain Li in the structure, the composition of tosudite composed of end-member cookeite and smectite layers may be located at the point of $\text{Li}_2\text{O} = 1.425\%$ and octahedral cation content = 3.5; that composed of end-member donbassite and smectite layers may be located at the point of $\text{Li}_2\text{O} = 0\%$ and octahedral cation = 3.0 (g). Thus, if the chlorite layer in tosudite exhibits a donbassite-cookeite solid solution similar to true Al-chlorite, the Li contents in tosudite minerals can be expected to range along line B in Figure 5. Li-bearing tosudites (i to l, and Echassières tosudite) having smaller Fe + Mg contents are distributed along line B, suggesting the existence of donbassite-cookeite continuous solid solution even in the chlorite layer in tosudite. On the other hand, some data show a considerable deviation from line B. These samples (e.g., sample h) contain larger amounts of Fe + Mg, therefore the chlorite layer in such tosudite minerals may be regarded as Li-bearing sudoite as is the base for the Al-chlorite as mentioned above.

Our knowledge of the physicochemical conditions of formation of Li-bearing donbassite and tosudite is still incomplete. Recently, Foord *et al.* (1986) specified possible thermal stability fields for cookeite and Li-tosudite, based on the synthetic studies of Eberl (1978a, 1978b). The formation of cookeite, in the Himalaya

Table 4. Chemical analyses of Echassières tosudite and Li-bearing tosudites.

Oxide	Wt. %				
	ECH ¹	MONT ²	TOOH ³	HUY ⁴	SAN DIEGO ⁵
SiO ₂	45.59	43.43	41.60	39.74	44.70
Al ₂ O ₃	38.06	35.35	36.40	35.87	37.80
Fe ₂ O ₃	0.21	0.35	1.82	0.98	0.14
FeO	—	—	—	2.77	—
MnO	—	0.27	—	—	0.06
MgO	—	0.17	0.29	3.08	0.84
TiO ₂	0.12	0.07	—	0.01	0.01
Li ₂ O	0.72	1.60	1.04	0.51	1.70
CaO	0.16	0.27	0.38	0.06	1.00
K ₂ O	1.87	1.90	0.38	0.62	0.98
Na ₂ O	1.02	0.28	0.14	0.12	0.15
Total ⁶	87.75	83.69	82.05	83.76	87.38

Cations per 12.5 O (anhydrous)

Si	3.50	3.50	3.40	3.26	3.43
Al(IV)	0.50	0.50	0.60	0.74	0.57
Al(VI)	2.95	2.85	2.91	2.72	2.85
Fe ³⁺	0.01	0.02	0.11	0.06	0.01
Fe ²⁺	—	—	—	0.19	—
Mg	—	0.02	0.04	0.25	0.09
Li	0.22	0.52	0.34	0.17	0.52
Ti	0.01	—	—	—	—
Oct	3.19	3.41	3.40	3.39	3.47
Mg	—	—	—	0.12	—
Ca	0.01	0.02	0.03	—	0.08
Na	0.15	0.04	0.02	0.02	0.02
K	0.18	0.20	0.04	0.07	0.10
Charge	0.35	0.28	0.12	0.33	0.28

¹ ECH = Echassières.

² MONT = Montebas, France (Creach *et al.*, 1986).

³ TOOH = Tooho, Japan (Nishiyama *et al.*, 1975).

⁴ HUY = Huy, Belgium (Brown *et al.*, 1974).

⁵ SAN DIEGO = San Diego County, California (Foord *et al.*, 1986).

⁶ Total does not include water.

dike system, probably occurred at 350°–400°C; Li-tosudite was formed at somewhat lower temperature. From laboratory experiments, Fransolet and Schreyer (1984) concluded that the possible thermal stability field for sudoite ranges from 150° to 350°C at 1 kbar. Matsuda and Henmi (1973) observed the transformation of a randomly interstratified illite/smectite to tosudite in pure water at 360°C and 1 kbar. Fujii *et al.* (1971) described an Al-chlorite that was formed from pyrophyllite, through sericite as an intermediate alteration product, under acid conditions as temperature decreased. Maksimović and Brindley (1980) demonstrated that the concentration of Si and Al due to pH decrease is necessary for the crystallization of tosudite. These facts suggest that the formation of donbassite and tosudite occurs at relatively high temperatures and low pHs. In the Echassières area, such conditions probably existed during the intermediate hydrothermal event between the early stage hydrothermal alteration resulting in formation of muscovite (>350°C) and the

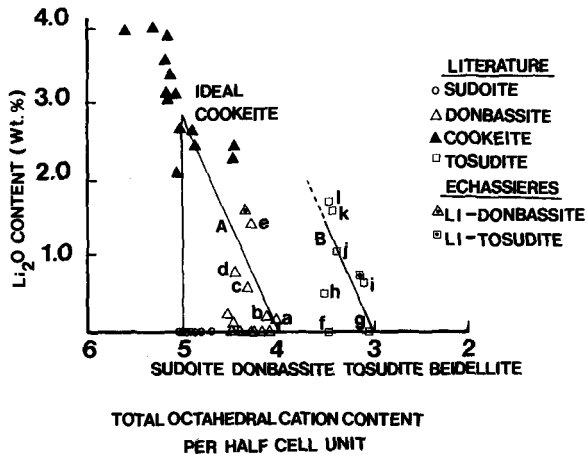


Figure 5. Diagram showing the relationship between Li_2O content and total octahedral cation content per half cell unit. Analytical data on cookeites are in Černý (1970); those on sudoites and donbassites are in Newman and Brown (1987). Other analytical data (a to l) are given with their Mg + Fe cations per half cell unit. Li-donbassite: (a) Zhuravaka, U.S.S.R., Lazarenko (1940), Mg + Fe = 0.23; (b, c) Namivu, Mozambique, Figueiredo Gomes (1967), Mg + Fe = 0.07, and 0.08; (d) Novaya Zemlya, U.S.S.R., Loskutov (1959), Mg + Fe = 0.11; (e) Itaya, Japan, Henmi and Yamamoto (1965), Mg + Fe = 0.03. Tosudite: (f) Niida, S. Odota, Japan, Sudo and Shimoda (1978), Mg + Fe = 1.13; (g) Takatama mine, Japan, Shimoda (1969), Mg + Fe = 0.03. Li-tosudite: (h) Huy, Belgium, Brown *et al.* (1974), Mg + Fe = 0.63; (i) Chassole, France, Turpault *et al.* (1986), Mg + Fe = 0.27; (j) Toho, Japan, Nishiyama *et al.* (1975); (k) Montebbras, France, Creach *et al.* (1986), Mg + Fe = 0.04; (l) Himalaya mine, San Diego, California, Foord *et al.* (1986), Mg + Fe = 0.11.

latest stage of deposition of randomly interstratified illite/smectite (<100°C). The vertically zoned distribution of donbassite and tosudite in the Echassières drill hole suggests that tosudite formed at slightly lower temperatures than donbassite. The chemical analyses from Echassières as well as from other localities indicate that the formation of Li-donbassite and tosudite requires the availability of Li in circulating hydrothermal solutions. At Echassières, this element was probably supplied from the wall rock because the host granite contains large amounts of lepidolite and possibly Li-phosphates (amblygonite-montebbrasite) as primary minerals.

ACKNOWLEDGMENTS

Financial support for this study was provided by the "Géologie Profonde de la France" program. The authors are indebted to E. E. Foord for his review of the manuscript and useful comments.

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(Received 1 April 1987; accepted 16 July 1987; Ms. 1668)