Lithium-bearing sodium amphibole (Li$_2$O = 0.01–1.02 wt%) was found in a specimen of schistose manganese ore from the limori region in the Sanbagawa metamorphic belt, central Japan. The ore is composed mainly of quartz, albite, amphibole, Na to Na–Ca pyroxene, and braunite. The amphibole occurs as prismatic crystals with lengths of up to 400 µm and consists of a pale–green core and an orange–red rim observed in hand specimen. The chemical formulae of averaged compositions of the core and rim, based on $24$(O, OH, Cl) with $(OH, Cl) = (2 - 2Ti)$ atoms per formula unit, are $\Delta$(Na$_{0.468}$K$_{0.448}$)$_{2Ti}$O$_{22}$(Si$_{8.000}$Al$_{0.064}$)O$_{22}$W$_{[(OH)_{1.737}F_{0.157}]\Sigma 0.916}$B(Na$_{1.586}$Ca$_{0.393}$Mn$_{2+0.021}$)C(Mg$_{3.896}$Mn$_{2+0.124}$Fe$_{3+0.657}$)A$_{2Ti}$O$_{22}$Fe$_{5.000}$TSi$_{8.000}$O$_{22}$W$_{[(OH)_{1.771}F_{0.167}O_{0.062}]\Sigma 2.000C(Mg_{3.143}Mn_{2+0.332}Fe_{3+0.782}Al_{0.247}Mn_{3+0.081}Ti_{0.053}Li_{0.353}Cu_{0.009})\Sigma 5.000}$Si$_{8.000}$O$_{22}$W$_{[(OH)_{1.737}F_{0.157}]\Sigma 0.916}$B(Na$_{1.759}$Ca$_{0.241}$)$\Delta$(Na$_{0.468}$K$_{0.448}$)$_{2Ti}$O$_{22}$(Si$_{7.936}$Al$_{0.064}$)O$_{22}$W$_{[(OH)_{1.737}F_{0.157}]\Sigma 0.916}$B(Na$_{1.586}$Ca$_{0.393}$Mn$_{2+0.021}$)C(Mg$_{3.143}Mn_{2+0.332}Fe_{3+0.782}Al_{0.247}Mn_{3+0.081}Ti_{0.053}Li_{0.353}Cu_{0.009})\Sigma 5.000$Si$_{7.936}$Al$_{0.064}$O$_{22}$W$_{[(OH)_{1.737}F_{0.157}]\Sigma 0.916}$B(Na$_{1.759}$Ca$_{0.241}$)

**Keywords:** Amphibole, Lithium, LIBS, Manganese ore, Iimori region, Sanbagawa belt

**INTRODUCTION**

Lithium is an essential component of lithium amphiboles and of sodium amphiboles (Hawthorne et al., 2012). Hawthorne et al. (1992, 1994) studied sodium amphiboles from the Kajlidongri manganese mine, India and showed that (1) the sodium amphiboles have significant amounts of octahedral Li (C-group Li) and (2) they belong to the series magnesio-arfvedsonite–ferri-leakeite [NaNa$_2$(Mg$_4$Fe$^{3+}_2$)Si$_8$O$_{22}$(OH)$_2$–NaNa$_2$(Mg$_2$Fe$^{3+}_2$Li$_2$)Si$_8$O$_{22}$(OH)$_2$], demonstrating that octahedral Li is an important constituent of sodium amphiboles in Li-bearing environments.

In Japan, the occurrence of Li-rich sodium amphibole with Li$_2$O > 1.0 wt% is very rare, and there has been a report only of potassic–ferri–leakeite [KNa$_2$(Mg$_2$Fe$^{3+}_2$Li)$_2$Si$_8$O$_{22}$(OH)$_2$] from the contact-metamorphosed manganese ore of the Tanohtake mine, Iwate Prefecture (Matsubara et al., 2002). We found Li-bearing sodium amphibole (Li$_2$O = 0.01–1.02 wt%) in a schistose manganese ore from the Sanbagawa metamorphic belt, which is a high-P/T metamorphic belt in the limori region, Wakayama Prefecture, Japan. This is the first report of Li-bearing amphibole from the Sanbagawa belt. We present the mode of occurrence and chemistry of this amphibole and discuss its chemical formula. The amphibole nomenclature used in this paper follows Hawthorne et al. (2012).

**GEOLOGIC SETTING AND PETROGRAPHY**

The Sanbagawa metamorphic belt belongs to the high-pressure intermediate group of the metamorphic facies series (Miyashiro, 1961). It represents the deeply subducted portion of a Mesozoic accretionary complex at the eastern margin of the Eurasian continent. The Sanbagawa metamorphic belt in the limori region is divided into three mineral zones, namely, chlorite, garnet, and biotite (in order of increasing metamorphic grade) on the basis of mineral paragenesis in the pelitic schists (e.g., Wang and Maekawa, 1997; Makimoto et al., 2004). The manganese ore that contains the Li-bearing sodium amphibole was collected from a waste–rock pile at the limori mine (34°15′N, 135°26′E). This mine is located in the garnet zone, according to the metamorphic zonation maps of Wang and Maekawa (1997) and Makimoto et al. (2004). The manganese ore consists of quartz–rich and braunite–rich layers (Fig. 1a). The quartz–rich layer is composed mainly of quartz, albite, amphibole, Na to Na–Ca pyroxene (hereinafter termed sodic pyroxene), and braunite with minor hematite. The amphibole occurs as prismatic crystals with lengths of up to 400 µm and exhibits optical zoning observed in thin section, with...
a colorless core and a pale-yellowish–green to pale-reddish-brown rim (Fig. 1b). In hand specimen, the core and rim of the amphibole are pale green and orange-red, respectively (Fig. 1c). Sodic pyroxene forms subhedral to euhedral prismatic crystals measuring <300 µm long and shows a zonal structure with a pale-yellowish–green to pale-violet core and a violet to reddish-purple rim in thin section (Fig. 1d). Therefore, sodic pyroxene can be classified into an earlier greenish type and a later purplish type. In many cases, the greenish type is replaced completely by the purplish type (Fig. 1e). The braunite-rich layer consists mainly of braunite, with minor quartz, albite, carbonate minerals, and apatite.

**MINERAL CHEMISTRY**

Chemical analyses of the amphibole, sodic pyroxene, and braunite were performed on an electron microprobe analyzer (EMPA, JEOL JXA-8900R) installed at the GSJ-Lab, AIST. The Li₂O concentration of the amphibole was analyzed using laser-induced breakdown spectroscopy (LIBS, Applied spectra J200–EC) at Gem Research Japan, Osaka, Japan. LIBS is a valuable technique for performing qualitative and quantitative chemical analyses of all elements, including light elements (e.g., Li and Be), which have proved difficult to measure with conventional techniques (Tempesta and Agrosì, 2016). In recent studies LIBS has been used to perform quantitative chemical analyses of Li in silicate minerals (Imaoka et al., 2017; Nagashima et al., 2020). Operating conditions for EMPA were as follows: an accelerating voltage of 15 kV, a beam current of 12 nA, and a beam diameter of 5 µm for analyses of F and Cl or 2 µm for analyses of other elements. Synthetic quartz (for Si), rutile (Ti), corundum (Al), eskolaite (Cr), hematite (Fe), manganosite (Mn), periclase (Mg), wollastonite (Ca), F-phlogopite (F), copper (Cu for braunite), natural jadeite (Na), adularia (K), cuprite (Cu for amphibole), and Cl-rich hastingsite (Cl) were used as standard materials. Matrix corrections for braunite and other minerals followed ZAF and Bence-Albee methods, respectively. Detection limits for Cl and F were ~0.02 and ~0.08 wt% (3σ level), respectively. Cl content of the amphibole is below the detection limit. The Li concentration for the amphibole grains in a fractured surface of the ore specimen was determined using LIBS. The LIBS used in this study employed a 266 nm Nd: YAG laser coupled with an optical emission spectrometer. The Li peak used was at a wavelength of 670.776 nm. All analyses were carried out under a He gas flow of 1.0 L min⁻¹ with a laser pulse energy of 6 mJ at 10 Hz using a laser beam diameter of 35 µm. The gate delay and pre-ablation times were 0.3 µs and 0.3 s, respectively. An average of 10 shots for each standard and an average of 2 shots for the amphibole were used for each quantitative calculation of Li₂O contents. Emission spectra were collected in the 185.191–1050.512 nm spectral range covering the main emission lines of major elements. The NIST SRM 610 and 612 glasses and synthetic Li glasses (Imaoka et al., 2017) were used as calibration standards covering the Li₂O concentration range between 86.5 ppm (NIST 612) and 2 wt% (Li glass). A calibration curve using these standards was created by calculating relation between known Li₂O contents and values of observed Li count/(observed Si count/known SiO₂ content). Averaged SiO₂ contents of the core and rim of the amphibole, which were
obtained by EMPA, were used as internal standards to estimate Li2O contents. Internal standardization is used to improve the precision of the measurements and to minimize the effect of laser intensity shot-to-shot variation (Bhatt et al., 2020). The chemical compositions determined for the amphibole, sodic pyroxene, and braunite are given in Tables 1, 2, and 3, respectively (Tables 2 and 3 are available online from https://doi.org/10.2465/jmps.200728).

**Table 1.** Average chemical compositions of amphibole

| (wt%)    | Core n = 20 | Range | Rim n = 23 | Range |
|----------|------------|-------|------------|-------|
| SiO2     | 56.94      | 56.14-57.34 | 56.54 | 55.90-57.02 |
| TiO2     | 0.30       | 0.09-0.58   | 0.50  | 0.32-0.66   |
| Al2O3    | 1.50       | 1.16-2.06   | 1.48  | 1.09-1.87   |
| Cr2O3    | n.d.       | <0.06       | n.d.  | <0.07       |
| Fe2O3    | 6.26       | 4.81-7.93   | 7.34  | 6.60-8.16   |
| MnO      | 1.23       | 0.80-1.65   | 3.45  | 2.63-4.51   |
| MgO      | 18.75      | 17.14-20.26 | 14.90 | 13.83-15.96 |
| CuO      | 0.04       | <0.09-0.10  | 0.08  | <0.09-0.15  |
| CaO      | 2.63       | 1.53-3.61   | 1.59  | 1.32-2.03   |
| Na2O     | 7.60       | 7.01-8.18   | 7.97  | 7.06-8.40   |
| K2O      | 2.52       | 2.17-2.75   | 3.19  | 2.89-3.40   |
| Li2O**   | 0.19       | 0.01-0.44   | 0.62  | 0.31-1.02   |
| F        | 0.38       | 0.26-0.52   | 0.35  | 0.26-0.41   |
| -O=F+Cl  | 0.16       | <0.02       | 0.15  | <0.02       |
| Total    | 98.18      |          | 97.86 |          |

*Total Fe as Fe2O3 and total Mn as MnO. **Li2O content was analyzed using LIBS. Numbers of measurements for the core and rim are 63 (on 13 points) and 97 (on 21 points), respectively, n.d., not detected.

**Table 4.** Chemical formulae with analytical totals of amphibole

| (apfu)   | Core | Rim | 1   | 2   | 3   | 4   | 5   |
|----------|------|-----|-----|-----|-----|-----|-----|
| Si       | 7.925| 7.936| 7.996| 8.014| 8.000|
| Al      | 0.075| 0.064| 0.004| 0.000| 0.000|
| Sum T   | 8.000| 8.000| 8.000| 8.014| 8.000|
| Al      | 0.171| 0.182| 0.243| 0.247| 0.247|
| Ti      | 0.031| 0.031| 0.053| 0.053| 0.053|
| Cr      | n.d. | n.d. | n.d. | n.d. | n.d. |
| Fe**    | 0.656| 0.657| 0.781| 0.783| 0.782|
| Mn**    | 0.000| 0.000| 0.000| 0.000| 0.000|
| Mn**    | 0.141| 0.124 | 0.413| 0.407| 0.332|
| Mg      | 3.891| 3.896| 3.141| 3.148| 3.143|
| Cu      | 0.004| 0.004| 0.009| 0.009| 0.009|
| Li      | 0.106| 0.106| 0.353| 0.353| 0.353|
| Sum C   | 5.000| 5.000| 4.993| 5.000| 5.000|
| Mn**    | 0.004| 0.021 | 0.000| 0.007| 0.000|
| Ca      | 0.392| 0.393| 0.241| 0.241| 0.241|
| Na      | 1.604| 1.586| 1.759| 1.752| 1.759|
| Sum B   | 2.000| 2.000| 2.000| 2.000| 2.000|
| Na      | 0.447| 0.468| 0.426| 0.438| 0.428|
| K       | 0.447| 0.448| 0.576| 0.577| 0.576|
| Sum A   | 0.894| 0.916| 1.002| 1.015| 1.004|
| Total   | 15.894| 15.916| 15.995| 16.029| 16.004|
| F       | 0.167| 0.167| 0.157| 0.157| 0.157|
| Cl      | n.d. | n.d. | n.d. | n.d. | n.d. |
| OH      | 1.833| 1.771| 1.843| 1.737| 1.737|
| O       | 0.000| 0.062| 0.000| 0.106| 0.106|
| Sum W   | 2.000| 2.000| 2.000| 2.000| 2.000|

*Total Fe as Fe2O3. **Total Mn as MnO. ***Mn**/Mn** was calculated based on Si = 8 apfu. †OH was calculated assuming OH + F + Cl = 2.0 apfu (columns 1 and 3) and OH + F + Cl = 2 - 2Ti apfu (columns 2, 4, and 5). ‡Total weight percentage for EMPA and LIBS analyses. Columns 1 and 3: Formulae on the basis of 24(O, OH, F, Cl) with (OH, F, Cl) = 2 apfu. Columns 2, 4, and 5: Formulae on the basis of 24(O, OH, F, Cl) with (OH, F, Cl) = (2 − 2Ti) apfu. n.d., not detected.

**Amphibole**

Figure 1f shows a backscattered electron image of zoned amphiboles with darker and brighter areas that correspond to the core and rim observed in thin section (Fig. 1b), respectively. The Li2O contents of the core and rim are 0.01-0.44 and 0.31-1.02 wt%, respectively. The core is richer in MgO (17.14-20.26 wt%) compared with the rim (13.83-15.96 wt%). The core is poorer than the rim in total Fe2O3 (4.81-7.93 wt%) versus 6.60-8.16 wt%) and total MnO (0.80-1.65 wt%) versus 2.63-4.51 wt%). TiO2 contents of the core and rim are 0.09-0.58 and 0.32-0.66 wt%, respectively. Fluorine contents are less than 0.52 wt% for both the core and rim. Possible chemical formulae (Table 4) are discussed below.

**Sodic pyroxene**

The Fe3+/Fe2+ ratio of sodic pyroxene was estimated with the total cations = 4.00 atoms per formula unit (apfu) for O = 6. However, the calculated total cations are, in most cases, larger than 4.00 apfu, even when all of the Fe has been calculated as Fe3+. This suggests that a part of the Mn ion should be Mn3+, as in the namansilite molecule (NaMn3+Si2O6: Kalinin et al., 1992), and as reported for some Mn-bearing omphacites and diopsides (so-called ‘violan’: Griffin and Mottana, 1982) and for some Mn-rich aegirines (Ashley, 1986). Therefore, in the case of the total cations being greater than 4.00 apfu, the Mn3+/Mn2+ ratio was calculated on the basis of total cations = 4.00 apfu (O = 6), assuming all iron to be Fe3+. If a calculated value of Mn2+ was negative using the abovementioned Mn3+/Mn2+ estimation, then the formulae were calculated based on all Fe as Fe3+ and all Mn as Mn3+. In this case, the sum of all cations is slightly higher than...
Li-bearing sodium amphibole from the Sanbagawa belt

4.00 apfu (cf. the formula for the purplish type with total MnO = 7.21 wt% in Table 2). Sodic pyroxene belongs to the aegirine-aegirine-augite series. The end-member proportions of jadeite, aegirine, namansilite, and augite were calculated using the method proposed by Morimoto (1988). The compositional range of the greenish type is X$_{Jd}$ 0.19–0.32, X$_{Ca}$ 0.48–0.62, X$_{Nam}$ 0.00–0.04, and X$_{Aug}$ 0.13–0.25, whereas that of the purplish type is X$_{Jd}$ 0.05–0.19, X$_{Ca}$ 0.36–0.64, X$_{Nam}$ 0.04–0.24, and X$_{Aug}$ 0.13–0.27. The purplish type is more enriched in Mn$^{3+}$ (as the namansilite molecule) compared with the greenish type. Ashley (1986) reported that aegirine with X$_{Nam}$ = 0.27. The purplish type is more enriched in Mn$^{3+}$ (as Mn$_2^+$) compared with the greenish type.

Braunite

Mn$^{3+}$/Mn$^{2+}$ values of braunite were estimated on the basis of total cations = 8.00 apfu and all Fe being in trivalent state (O = 12). Braunite can be classified into an early-formed Ca-poorer type (Ca = 0.03–0.08 apfu) and a later-formed Ca-richer type (Ca = 0.19–0.26 apfu). Most of the braunite belongs to the Ca-poorer type. The Ca-richer type locally occurs in the braunite-rich layer and grows on the Ca-poorer type. The Ca-poorer braunite is zoned with increasing Mn$^{2+}$ and decreasing Mg from core to rim and has high Mg contents of up to 0.34 apfu, implying the substitution Mg = Mn$^{2+}$. Contents of Cu and Mn$^{2+}$ of the Ca-poorer type are 0.01–0.05 and 0.58–0.96 apfu, respectively. The Ca-richer braunite has up to 26% of the neltnerite molecule (CaMn$_{13}^+$SiO$_{12}$; Baudracco-Gritti et al., 1982). Compositional ranges of Cu, Mg, and Mn$^{2+}$ of the Ca-richer type are 0.00–0.01, 0.03–0.05, and 0.85–0.95 apfu, respectively.

DISCUSSION

Hawthorne et al. (2012) discussed the calculation of amphibole formulae and (OH) content, taking into account present knowledge of the crystal chemistry of amphiboles. It is usually recommended that amphibole formulae be normalized on the basis of 24(O, OH, F, Cl) with (OH, F, Cl) = 2 apfu, where H$_2$O is not known. This method is equivalent to the 230 calculation. However, Hawthorne et al. (2012) recommended that in the absence of direct measurement of H$_2$O, amphibole formulae be calculated on the basis of 24(O, OH, F, Cl) with (OH, F, Cl) = (2 − 2Ti) apfu. This normalization method is led by the substitution $^{M1}$(Ti$^{4+}$ + 2WO$_2^-$) = $^{M1}$(Mg, Fe$^{3+}$) + 2$^{M2}$(OH) using the approximation $^{M1}$(Ti$^{4+}$) = Ti (Hawthorne et al., 2012), although in some cases, a significant fraction of Ti may occur at the M(2) site (Hawthorne et al., 1998). Therefore, chemical formulae of averaged compositions of the core and rim of the amphibole from limori (Table 1) were calculated using the abovementioned two normalization methods. First, we calculated them based on 24(O, OH, F, Cl) with (OH, F, Cl) = 2 apfu assuming all Fe as Fe$^{3+}$ and all Mn as Mn$^{2+}$, and the calculated formulae of the core and rim are listed as columns 1 (core) and 3 (rim) in Table 4, respectively. The formula of the core satisfies the six criteria (stoichiometric limits) [(1) Si ≤ 8, (2) Si + Al ≥ 8, (3) sum of all cations exclusive of Ca, Na, and K ≥ 13, (4) sum of all cations exclusive of Na and K ≤ 15, (5) sum of all cations exclusive of K ≥ 15, and (6) sum of all cations ≤ 16] outlined by Schumacher (1997). The formula for the rim reveals that the sum of all cations exclusive of Ca, Na, and K is 12.993 apfu. This value is slightly under the stoichiometric limit (3), but the value is very close to the ideal number of 13 apfu. Thus, the formula is considered to be acceptable.

Next, the formulae of the core and rim were calculated based on 24(O, OH, F, Cl) with (OH, F, Cl) = (2 − 2Ti) apfu and an assumption of all Fe as Fe$^{3+}$ and all Mn as Mn$^{2+}$. The results are presented as columns 2 (core) and 4 (rim) in Table 4. The formula for the core also satisfies the abovementioned criteria, whereas that for the rim violates criteria (1) and (6), implying that a part of Mn should be Mn$^{3+}$ to reduce the amount of Si and the sum of all cations. Thus, we attempted to estimate the Mn$^{3+}/$(Mn$^{3+}$ + Mn$^{2+}$) ratio on the basis of Si = 8 apfu, and the recalculated formula with Mn$^{3+}$ = 0.081 apfu is given in column 5 in Table 4. This formula shows that the sum of all cations is 16.004 apfu. This value is slightly over the stoichiometric limit (6), but the value is very close to the ideal number of 16 apfu. Therefore, the formula is considered to be acceptable.

The core and rim are pale green and orange-red, respectively, in hand specimen (Fig. 1c). Despite their relatively similar chemical compositions (cf. Table 1), they have obviously different colors. Mn$^{3+}$–rich sodic amphiboles have characteristically reddish color: for example, cherry-red to dark red for mangano–mangani–ungarettite [NaNa$_2$(Mn$^{3+}$2Mn$^{3+}$)Si$_8$O$_{22}$O$_2$; Hawthorne et al., 1995], brownish lilac to dark red for potassic–mangani–leakeite [KNa$_2$(Mg$_2$Mn$^{3+}$2Li)Si$_8$O$_{22}$(OH)$_2$; Armbruster et al., 1993], pink to red for mangani–dellaventurate [NaNa$_2$(MgMn$^{3+}$2 Ti$_2$)Si$_8$O$_{22}$O$_2$; Tait et al., 2005], and deep red for holotype ferri–leakeite with Mn$^{3+}$ = 0.42 apfu (Hawthorne et al., 1992). This suggests the presence of an amount of Mn$^{3+}$ in the orange-red rim of the amphibole from limori. Therefore, we selected the formulae of columns 2 and 5 in Table 4 as the final formulae and which were calculated by the same normalization method, 24(O, OH, F, Cl) with (OH, F, Cl) = (2 − 2Ti) apfu. The chemical formulae of the core and
rim are \(\Delta(Na_0.468K_0.448)_{20.916}^{20.916}(Na_1.580Ca_0.392Mn_0.021)_{22.000}^{22.000}C(Mg_{3.896}Mn_{0.124}Fe_{0.365}Al_{0.635}A_0_{0.182}Li_{0.031}Li_{0.106}Cu_{0.004})_{25.000}^{25.000}T(Si_{7.936}Al_{0.064}O_{22.000})\) and \(\Delta(K_{0.676}Na_{0.257}A_{1.000})_{21.000}^{21.000}(Na_{1.759}Ca_{0.241})_{22.000}^{22.000}(Mg_{13.144}Mn_{1.332}Fe_{0.785}Al_{0.247}Mn_{0.081}Ti_{0.053}Li_{0.051}Li_{0.353}Cu_{0.009})_{25.000}^{25.000}Si_{8.000}O_{22}\). Consequently, the core amphibole has an intermediate composition between magnesio-arfvedsonite and potassic-magnesio-arfvedsonite [ideally KNa2(MgFe3+)Si8O22(OH)2: Dyulgerov et al., 2019], whereas the rim amphibole, which has a higher K/(K + Na) ratio than that of the core, is potassic-magnesio-arfvedsonite. The final formulae of the core and rim indicate that Li content increases from core to rim, but \((Mg + Mn)\) content decreases, with increasing \((Fe^{3+} + Mn^{3+} + Al)\) content. This implies that the Li contents are controlled mainly by the substitution \(2C(Mg, Mn^{2+}) \leftrightarrow (Fe^{3+}, Mn^{3+}, Al) + Li\).

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SUPPLEMENTARY MATERIAL

Color version of Figure 1 and Tables 2 and 3 are available online from https://doi.org/10.2465/jmps.200728.

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