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NEPHELINE-“IRON NEPHELINE” SOLID SOLUTIONS

by

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(with 3 Tables and 5 Text-figures)

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Abstract

The phase diagram of the join NaAlSiO_4 (nepheline) – “ $\text{NaFe}^{3+} \text{SiO}_4$ ” (iron nepheline) was determined, and the unit-cell dimensions of the nepheline solid solutions belonging to this join and of the nepheline solid solutions crystallized in the join $\text{NaAlSiO}_4 - \text{NaFe}^{3+} \text{Si}_2 \text{O}_6$ (acmite) were measured. “ $\text{NaFe}^{3+} \text{SiO}_4$ ” is incorporated into nepheline, by $\text{Fe}^{3+} - \text{Al}$ replacement, as much as 25 mol % $\text{NaFe}^{3+} \text{SiO}_4$ at 700°C at one atmosphere. The data on unit-cell dimensions also show that iron-bearing nephelines in the join $\text{NaAlSiO}_4 - \text{NaFe}^{3+} \text{Si}_2 \text{O}_6$ may be enriched in silica. Therefore, the nepheline in this join must be described with reference to the three components, NaAlSiO_4 , $\text{NaFe}^{3+} \text{SiO}_4$, and SiO_2 and the formula $\text{Na}_{x+y} \square_{8-(x+y)} \text{Fe}^{3+}_x \text{Al}_y \text{Si}_{16-(x+y)} \text{O}_{32}$ is given to this nepheline solid solution.

Introduction

In the study of the system nepheline-acmite, one of the authors (K.Y.) found that nepheline crystallizes below the solidus temperature, and that it is not associated with other phases between NaAlSiO_4 70 – 100 wt. %. He suggested that this nepheline might be an iron-bearing solid solution (YAGI, 1962). BAILEY and SCHAIRER (1966) have shown that nephelines which crystallize from compositions in the system $\text{Na}_2 \text{O} - \text{Fe}_2 \text{O}_3 - \text{Al}_2 \text{O}_3 - \text{SiO}_2$ have higher mean indices of refraction compared with those of pure nepheline, clearly indicating the effect of solid solution of the iron-bearing molecule. They called this molecule “iron nepheline” ($\text{NaFe}^{3+} \text{SiO}_4$) (henceforth abbreviated FeNe). They also prepared two compositions along the join $\text{NaAlSiO}_4 - \text{“NaFe}^{3+} \text{SiO}_4 \text{”}$ to investigate the replacement of Fe^{3+} for Al in nepheline.

In the present study pure nepheline was synthesized and mixtures of six compositions along this join were used to determine the phase diagram of the join. Unit-cell dimensions of the nepheline solid solution along this join and along the join $\text{NaAlSiO}_4 - \text{“NaFe}^{3+} \text{SiO}_4 \text{”}$ were also determined.

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Experimental Method

Ordinary quenching method was employed for investigation of the phase diagram. Homogeneous glasses were prepared by melting pure chemicals in a platinum crucible. In order to minimize the loss of iron from mixture into crucible, the platinum crucible used in this experiment saturated with iron by heating with Fe_2O_3 powder. Quartz (purified by the same method as described by SCHAIRER and BOWEN, 1955), certified reagents of Al_2O_3 and Fe_2O_3 , and pure sodium disilicate (prepared by the same method as described SCHAIRER and BOWEN, 1956) were used as a source. Sodium disilicate was used instead of sodium carbonate in order to prevent the loss of soda by volatilization at high temperature. The mixture was ground in an agate mortar with pure ethyl alcohol before melting. The glasses were heated at 700°C or 750°C for 30 days. The crystallization process of mixtures of $\text{Ne}_{95}\text{FeNe}_5$, $\text{Ne}_{90}\text{FeNe}_{10}$, and $\text{Ne}_{75}\text{FeNe}_{25}$ was checked by diffraction patterns. After annealing for 17 hours at 700°C the mixtures of $\text{Ne}_{95}\text{FeNe}_5$ and $\text{Ne}_{90}\text{FeNe}_{10}$ showed the diffraction pattern of low carnegieite, whereas $\text{Ne}_{75}\text{FeNe}_{25}$ showed that of nepheline. The diffraction pattern of nepheline was not obtained from the mixture $\text{Ne}_{95}\text{FeNe}_5$ even after annealing for 632 hours at 700°C . In the mixture $\text{Ne}_{90}\text{FeNe}_{10}$ the peaks of low-carnegieite disappeared and nepheline peaks appeared after annealing for 116 hours. The mixture $\text{Ne}_{95}\text{FeNe}_5$ showed the peaks of both the low-carnegieite and nepheline after heating for 72 hours at 750°C , however the peaks of low-carnegieite completely disappeared after heating for 158 hours at the same temperature. Thus the crystalline mixture consisting of minute granular crystals of nepheline were obtained.

These crystalline mixtures were used as starting materials for the quenching experiments. Pt-Pt₈₇Rh₁₃ thermocouples used to measure the temperature were calibrated by using the standard points of NaCl (800.4°C), Au (1062.6°C), and diopside (1391.5°C). Crystalline phases obtained by quenching were identified by microscope and X-Ray diffractometer. At high temperatures, over 1000°C , the duration of runs was 1/2–2 hours or 2 days to minimize volatilization of soda and reduction of Fe_2O_3 .

X-ray powder diffraction patterns of the nephelines along the join $\text{NaAlSiO}_4 - \text{NaFe}^{3+}\text{SiO}_4$ and along the join $\text{NaAlSiO}_4 - \text{NaFe}^{3+}\text{Si}_2\text{O}_6$ were obtained by the diffractometer with a crystal monochromator, using $\text{CuK}\alpha$ radiation at 35 KV and 20 mA at room temperature. An indexed powder pattern of pure nepheline was compared with the patterns of the nephelines along the two joins. Each peak of these nephelines corresponds to that of pure nepheline but its position shifts regularly as a function of the composition. Complete patterns were taken with a scanning speed of 1° per minute

from 70° to 10° in 2θ . Reflections of $(20\bar{2}2)$ and $(21\bar{3}0)$, from which the unit-cell dimensions were determined, were obtained at a scanning speed of $1/8^\circ$ per minute. Measurement of 2θ was made to $\pm 0.005^\circ$ and reproducibility for the 2θ measurements in triplicate was found to be $\pm 0.01^\circ$. The silicon line at 28.465° in 2θ was used as an internal standard.

Experimental Results

Phase diagram. The join NaAlSiO_4 – “ $\text{NaFe}^{3+}\text{SiO}_4$ ” is not stable because the composition $\text{NaFe}^{3+}\text{SiO}_4$ decomposes into hematite, acmite, and 5.1.8 compound ($\text{Na}_5\text{Fe}^{3+}\text{Si}_4\text{O}_{12} = 5\text{Na}_2\text{O} \cdot \text{Fe}_2\text{O}_3 \cdot 8\text{SiO}_2$) at atmospheric pressure (BOWEN et al., 1930). Therefore, the join should have the phase assemblage nepheline, acmite, hematite, and 5.1.8 at the solidus temperature. The assemblage nepheline, acmite, hematite, and 5.1.8 also appears at the solidus temperature in the system $\text{Na}_2\text{O}-\text{Fe}_2\text{O}_3-\text{Al}_2\text{O}_3-\text{SiO}_2$. BAILEY and SCHAIRER (1966) have reported the temperature of an invariant point, at which this phase assemblage appears is 763°C .

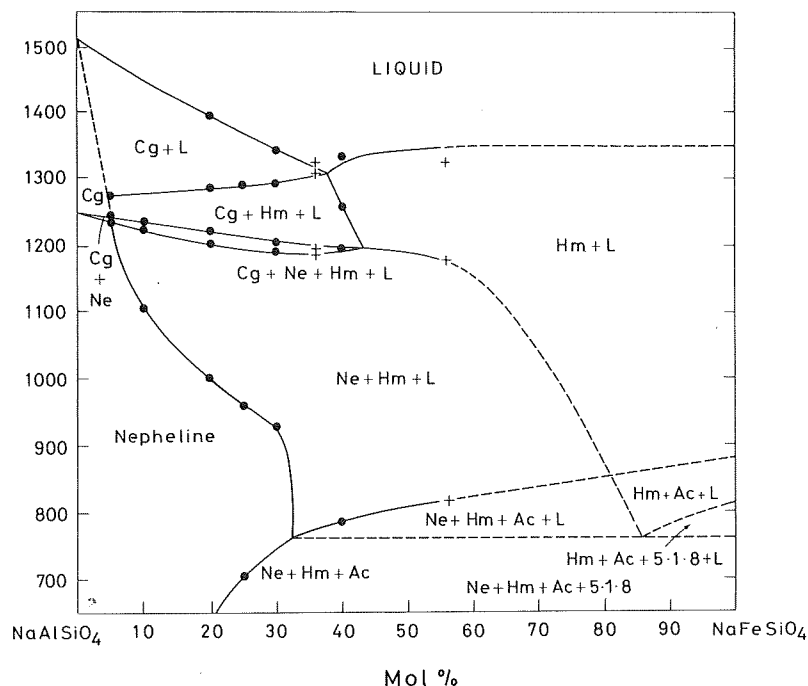


Figure 1 Phase diagram of the join NaAlSiO_4 – “ $\text{NaFe}^{3+}\text{SiO}_4$ ”. Broken lines is tentatively drawn from the data by Bowen et al. (1930) and Bailey and Schairer (1966). Cross, data by Bailey and Schairer (1966); Cg, carnegieite (solid solution); Ne, nepheline (solid solution); Hm, hematite; Ac, acmite; 5.1.8, $5\text{Na}_2\text{O} \cdot \text{Fe}_2\text{O}_3 \cdot 8\text{SiO}_2$; L, liquid.

Table 1. Results of quenching experiments for the
join NaAlSiO_4 - " $\text{NaFe}^{+3}\text{SiO}_4$ "

Composition (Mol %)		Temp. (°C)	Time	Phases
NaAlSiO_4	$\text{NaFe}^{+3}\text{SiO}_4$			
95	5	1275	6 hr	Cg + gl
		1270	24 hr	Cg + Hm + gl
		1250	48 hr	Cg + Hm + gl
		1245	72 hr	Cg + Ne + Hm + gl
		1230	72 hr	Cg + trace Hm + gl
90	10	1260	24 hr	Cg + gl
		1255	48 hr	Cg + trace Hm + gl
		1230	72 hr	Cg + Hm + gl
		1225	5 days	Cg + Ne + Hm + gl
		1220	5 days	Ne + Hm + gl
		1105	7 days	Ne + trace Hm + gl
		1100	7 days	Ne
80	20	1400	2 hr	gl
		1395	2 hr	Cg + gl
		1285	2 hr	Cg + gl
		1280	24 hr	Cg + Hm + gl
		1225	72 hr	Cg + Hm + gl
		1220	5 days	Cg + Ne + Hm + gl
		1210	7 days	Cg + Ne + Hm + gl
		1205	7 days	Ne + Hm + gl
		995	10 days	Ne + Hm + gl
		990	10 days	Ne
75	25	1290	2 hr	Cg + gl
		1285	2 hr	Cg + trace Hm + gl
		960	7 days	Ne + trace Hm + gl
		955	13 days	Ne
70	30	1350	1/2 hr	gl
		1345	1/2 hr	trace Cg + gl
		1295	2 hr	Cg + gl
		1290	2 hr	Cg + Hm + gl
		1205	72 hr	Cg + Hm + gl
		1200	72 hr	Cg + Hm + Ne + gl
		1195	72 hr	Ne + Hm + gl
		930	7 days	Ne + Hm ? + gl
925	14 days	Ne		
60	40	1335	1/2 hr	gl
		1330	1 hr	Hm + gl
		1260	48 hr	Hm + gl
		1255	48 hr	Cg + Hm + gl
		1200	72 hr	Cg + Hm + gl
		1195	72 hr	Ne + Hm + gl
		790	21 days	Ne + Hm + gl
		780	14 days	Ne + Ac + Hm + gl

The phase diagram of the join NaAlSiO_4 - " $\text{NaFe}^{3+}\text{SiO}_4$ " is shown in Fig. 1 and the data used to construct the diagram are given in Table 1. The data of BAILEY and SCHAIRER (1966) are also shown in Fig. 1. Their data are in good agreement with those of the present study within experimental error. We have no data for the " $\text{NaFe}^{3+}\text{SiO}_4$ "-rich region of the diagram beyond $\text{Ne}_{60}\text{FeNe}_{40}$. Consequently the phase boundary curves shown by broken lines of this region were tentatively drawn on the basis of data from the literature (BOWEN et al., 1930 and BAILEY and SCHAIRER, 1966). At the solidus temperature nepheline, acmite, and hematite were encountered, but 5.1.8 was not confirmed either by microscope or X-ray. The diagram has liquidus lines for both carnegieite and hematite. These cross each other at the temperature of $1305^\circ \pm 5^\circ\text{C}$ and a composition of about $\text{Ne}_{63}\text{FeNe}_{37}$. Carnegieite-nepheline inversion* takes place within a temperature range of about 15°C (at the composition of $\text{Ne}_{80}\text{FeNe}_{20}$). It is associated with hematite, as shown by the narrow field in which iron-rich carnegieite coexists with iron-poor nepheline, hematite, and liquid. The temperature of the upper boundary curve of this field decreases, with increasing " $\text{NaFe}^{3+}\text{SiO}_4$ ", to $1200^\circ \pm 5^\circ\text{C}$. A similar condition was found in the join NaAlSiO_4 - $\text{NaFe}^{3+}\text{Si}_2\text{O}_6$ (YAGI, 1962 and BAILEY and SCHAIRER, 1966). These facts indicate that the existence of Fe^{3+} in nepheline solid solution effectively lowers the inversion* temperature within a limited temperature range. The field of nepheline solid solution extends only to about $\text{Ne}_{70}\text{FeNe}_{30}$ at 765°C . The content of " $\text{NaFe}^{3+}\text{SiO}_4$ " is a maximum at this temperature, and decreases both with rising and falling temperatures.

The nephelines and carnegieites encountered in this join are somewhat different from the iron-free end-member. Both crystals are slightly pale brown in color and the nephelines have higher indices of refraction than those of pure nepheline. The mean refractive indices of nephelines crystallized at 700°C in the join are shown in Fig. 2 and Table 2. The indices of refraction increase with increasing " $\text{NaFe}^{3+}\text{SiO}_4$ ".

Unit-cell dimensions The values of a , c , and V of the nepheline solid solutions that crystallize from six compositions along the join NaAlSiO_4 - " $\text{NaFe}^{3+}\text{SiO}_4$ " as well as three compositions along the join NaAlSiO_4 - $\text{NaFe}^{3+}\text{Si}_2\text{O}_6$ are given in Table 3 and are plotted against the compositions in Figs. 3 and 4. Accuracies are evaluated to be $\pm 0.002 \text{ \AA}$ for a , ± 0.003 for c , and $\pm 0.4 \text{ \AA}^3$ for V . The values of a , c , and V in the join NaAlSiO_4 - " $\text{NaFe}^{3+}\text{SiO}_4$ " increase with increasing " $\text{NaFe}^{3+}\text{SiO}_4$ ". The extensions of curves a and c (broken lines in Fig. 3) do not coincide with those of pure nepheline, a 9.972 ± 0.002 and c $8.338 \pm 0.003 \text{ \AA}$. The pure nepheline was synthesized by

* This term is not used here in the strictest sense but rather as a general term for phase change involving minor variation in composition.

sintering the mixture of sodium disilicate and alumina at 1400°C. This may suggest the existence of a discontinuity between Ne_{100} and $Ne_{95}FeNe_5$,

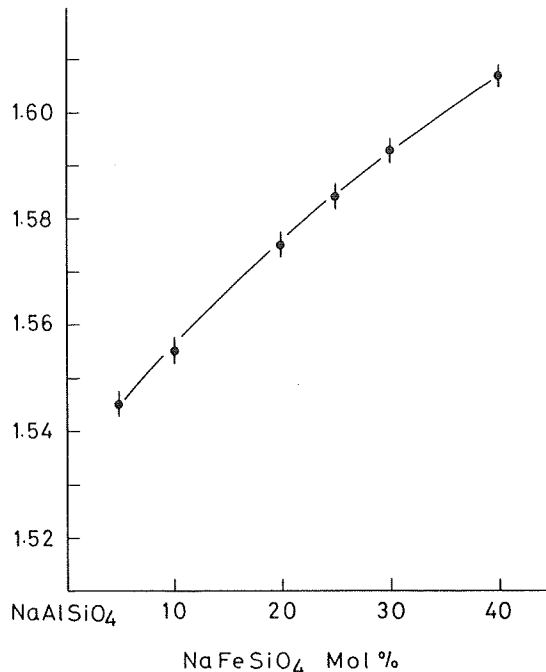


Figure 2. Mean refractive indices of the nepheline solid solutions crystallized from the join $NaAlSiO_4$ - $NaFe^{3+}SiO_4$, showing measurement accuracy ± 0.003 . $Ne_{95}FeNe_5$ crystallized at 750°C and the remainders at 700°C.

NaFe ³⁺ SiO ₄ (mol %)	5	10	20	25	30	40
refractive indices (± 0.003)	1.545	1.555	1.575	1.584	1.593	1.607

Table 2. The mean refractive indices of nepheline crystallized at 700°C in the join $NaAlSiO_4$ - $NaFeSiO_4$

though the condition of synthesis of pure nepheline is different from that of iron-bearing nepheline. It is noted that similar phenomenon was observed in the nepheline-kalsilite series (DONNAY et al., 1959).

Both a and c of nephelines that crystallize from three compositions at 800°C along the join $NaAlSiO_4$ - $NaFe^{3+}Si_2O_6$ increase linearly with increasing $NaFe^{3+}Si_2O_6$ as shown in Fig. 4. Since YAGI (1962) has shown that these nephelines are not associated with any other phases at 800°C in this join, it is expected that the nepheline should have a higher content of SiO_2 as compared with nephelines that crystallize from the join $NaAlSiO_4$ - $NaFe^{3+}SiO_4$

NaFe ³⁺ SiO ₄ (mol %)	a(A°) ±0.002	c(A°) ±0.003	V(A° ³) ±0.4
5	10.001	8.288	718.0
10	10.012	8.285	719.2
20	10.030	8.303	723.4
25	10.041	8.315	726.0
30	10.045	8.318	726.9
40	10.063	8.326	730.1

NaFe ³⁺ Si ₂ O ₆ (mol %)	a(A°) ±0.002	c(A°) ±0.003	V(A° ³) ±0.4
4.3	9.995	8.348	722.2
8.2	10.002	8.366	724.8
13.5	10.012	8.367	726.3

Table 3. The unit-cell dimensions of nepheline crystallized at 700°C in the joins NaAlSiO₄–"NaFe³⁺SiO₄" and NaAlSiO₄–NaFe³⁺Si₂O₆

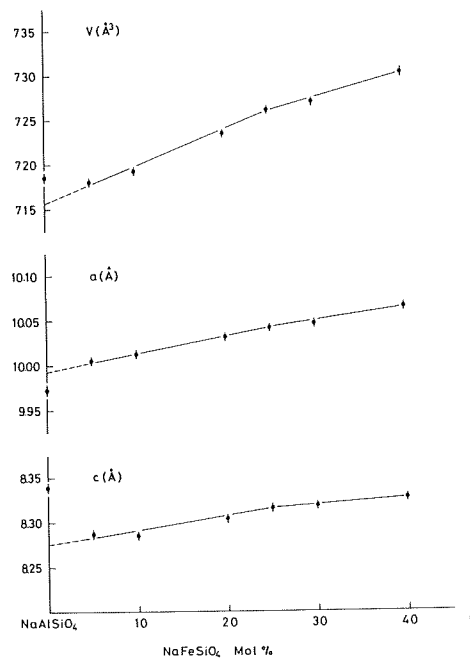


Figure 3. Unit-cell dimensions of the nepheline solid solutions crystallized from the join NaAlSiO₄–"NaFe³⁺SiO₄". Ne₉₅FeNe₅ crystallized at 750°C and the remainders at 700°C. Accuracies are evaluated to be ±0.002 Å° for *a*, ±0.003 for *c*, and ±0.4 Å³ for *V*.

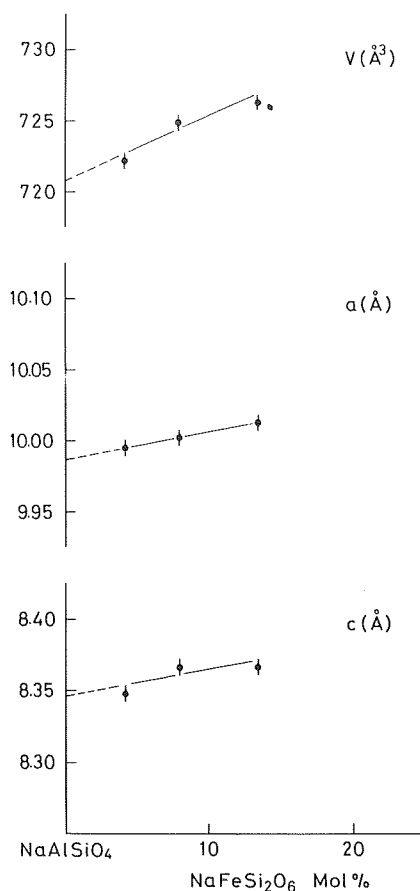


Figure 4. Unit-cell dimensions of the nepheline solid solutions from the join $\text{NaAlSi}_3\text{O}_8$ – $\text{NaFe}^{3+}\text{Si}_2\text{O}_6$ at 800°C .
 Accuracies are evaluated to be $\pm 0.002 \text{ \AA}$ for a , ± 0.003 for c and $\pm 0.4 \text{ \AA}^3$ for V .

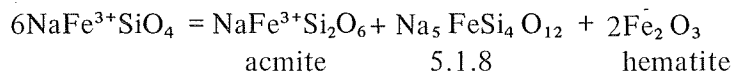
Discussion

The linear change of indices of refraction and unit-cell dimensions of the nephelines both in the joins $\text{NaAlSi}_3\text{O}_8$ –“ $\text{NaFe}^{3+}\text{Si}_2\text{O}_6$ ” and $\text{NaAlSi}_3\text{O}_8$ – $\text{NaFe}^{3+}\text{Si}_2\text{O}_6$ (Figs. 2, 3, and 4) strongly suggests that solid solutions are present between nepheline and “iron nepheline” and also indicates that the nephelines that crystallize in the join $\text{NaAlSi}_3\text{O}_8$ – $\text{NaFe}^{3+}\text{Si}_2\text{O}_6$ are solid solutions as suggested by YAGI (1962) and BAILEY and SCHAIRER (1966).

When only nepheline solid solution exists, probably replacement of Fe^{3+} for Al takes place and whole $\text{NaFe}^{3+}\text{Si}_2\text{O}_6$ molecules are incorporated into nepheline. Taking 32 oxygen atoms in a unit-cell, $\text{Na}_8\text{Fe}_x\text{Al}_{8-x}\text{Si}_8\text{O}_{32}$ must be given to this nepheline solid solution and the limit of the solid solution at 700°C is $x=2$, because acmite and hematite appear beyond $\text{Ne}_{75}\text{FeNe}_{25}$ at

700°C.

In the part with more than 25 mol. % FeNe the condition is rather complicated. BAILEY and SCHAIRER (1966) have given the equation,

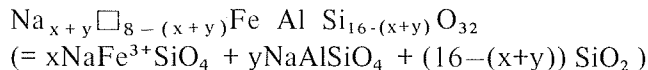


for the decomposition of $\text{NaFe}^{3+}\text{SiO}_4$. If four phases (nepheline, acmite, 5.1.8 and hematite) coexist in the solidus region, this phase assemblage is isobaric univariant because of the quarternary nature of the join. Therefore, the composition of nepheline solid solution should be fixed at constant temperature, 700°C. However, we did not confirm 5.1.8 in the mixture of $\text{Ne}_{70}\text{FeNe}_{30}$ and $\text{Ne}_{60}\text{FeNe}_{40}$. BAILEY and SCHAIRER (1963) have reported that they could not confirm 5.1.8 in the mixture of $\text{Ne}_{60}\text{FeNe}_{40}$ (wt. %). Even when the nepheline solid solutions coexist with other phases (acmite and hematite), the composition of the nepheline changes as indicated by increase of unit-cell dimensions. These facts, therefore, suggest that only three phases (acmite, nepheline, and hematite) coexist around the composition of $\text{Ne}_{60}\text{FeNe}_{40}$ and $\text{Ne}_{70}\text{FeNe}_{30}$ in the join $\text{NaAlSiO}_4 - \text{NaFe}^{3+}\text{SiO}_4$, indicating isobaric divariance. Since the phase assemblage acmite + nepheline + hematite has two degrees of freedom the composition of nepheline solid solution can change at constant temperature. Assuming the presence of a discontinuity in the curve of V, we may simply postulate that the appearance of iron-bearing phases (acmite and hematite) beyond $\text{Ne}_{75}\text{FeNe}_{25}$ at 700°C may lead to a flattening of the curve. It is reasonable to consider that the $\text{Na}_5\text{FeSi}_4\text{O}_{12}$ consists of acmite and sodium metasilicate ($\text{Na}_5\text{Fe}^{3+}\text{Si}_4\text{O}_{12} = \text{NaFe}^{3+}\text{Si}_2\text{O}_6 + 2\text{Na}_2\text{SiO}_3$), that Na_2SiO_3 enters into nepheline solid solution and that acmite is also incorporated into nepheline solid solution enriched in silica.

Assuming that Fe^{3+} enters into nepheline solid solution in the form of " $\text{NaFe}^{3+}\text{SiO}_4$ ", there always exists an excess of SiO_2 in the nepheline solid solutions that crystallize in the join $\text{NaAlSiO}_4 - \text{NaFe}^{3+}\text{Si}_2\text{O}_6$, so that nepheline solid solutions in this join are described with reference to the three components, NaAlSiO_4 , " $\text{NaFe}^{3+}\text{SiO}_4$ ", and SiO_2 . In their study, DONNAY et al. (1959) have suggested that the possibility of vacancy for some cation sites must be taken into account in order to explain the excess silica in natural nephelines. HAMILTON and MACKENZIE (1960) have shown experimentally the existence of such vacant sites in nepheline in the system $\text{NaAlSiO}_4 - \text{KAlSiO}_4 - \text{SiO}_2$. If we assume such vacant sites to explain excess SiO_2 in the nepheline

*However Tilley(1933) showed that there is little or no solid solution between Na_2SiO_3 and pure nepheline at low temperature.

solid solutions described in terms of the three components NaAlSiO_4 , " $\text{NaFe}^{3+}\text{SiO}_4$ ", and SiO_2 , the following formula is given to this nepheline solid solution.



where \square is a vacant site and $x + y < 8$.

This formula indicates the possibility of nepheline solid solution between NaAlSiO_4 and $\text{NaFe}^{3+}\text{Si}_2\text{O}_6$ and provides for nepheline solid solution enriched in SiO_2 . Therefore, we can assume two types of replacement for the iron-bearing nephelines:

(1) Fe^{3+} replacing Al and (2) vacant site replacing Na, and Fe^{3+} and Al replacing Si.

The iron content of natural nepheline is not negligible. DEER et al. (1963) have shown chemical analyses of twenty natural nephelines from various localities. Among them nine nephelines contain more Fe^{3+} than Ca, six of the nine have more than twice as much Fe^{3+} as Ca. Fig. 5 shows the relationship between the number of Si, Al, and Fe^{3+} atoms of these nephelines on the basis of 32 oxygens. When Si is plotted against Al a linear relation exists between these atoms for the smaller values of Al. However, the plot becomes more scattered with increasing Al (Fig. 5A). On the other hand, the plots of Si against the sum of Al and Fe^{3+} show completely linear change, indicating the complete replacement of Al and Fe^{3+} for Si (Fig. 5B).

BROWN (1970) has shown that the nephelines in nepheline tephrite from Ol Doinyo Lengai, Tanzania contain as much as 4.6 wt. % Fe_2O_3 and demonstrated a regular variation of iron with aluminum, aluminum increasing when

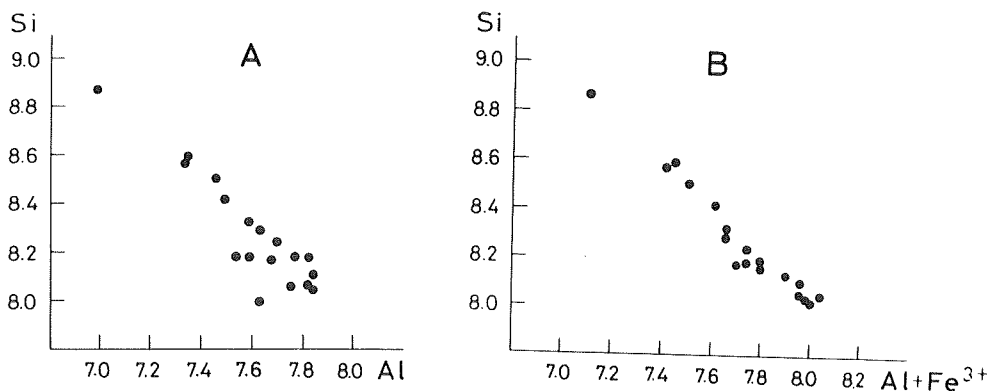


Figure 5. (A) Relationship between numbers of Si and of Al.
(B) Relationship between numbers of Si and sum of Al and Fe^{3+} . Data are taken from Deer et al. (1963).

iron decreases and vice versa. From this fact this author concluded that there is substitution of Fe^{3+} for Al in natural nepheline. In their study of iron in the sodalite group, BROUSSE et al. (1969) have proved that the iron is substituted into the structure of feldspathoids instead of Al, and that Fe^{3+} is enriched with decreasing Al and also increasing Si. These studies may support the validity of the two types of replacement in iron-bearing nephelines, proposed on the basis of the experimental data in the present study.

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