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TABLES

UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

| | | | |
|---|---|---|---|
| cal | calorie | kcal/mol | kilocalorie per mole |
| g | gram | mol | mole |
| g/mol | gram per mole | μm | micrometer |
| J | joule | pct | percent |
| kcal | kilocalorie | wt-pct | weight-percent |

# THE ENTHALPY OF FORMATION OF SYNTHETIC CANCRINITE $[Na_{7.68}(Al_6Si_6O_{24})(NO_3)_{1.68}(H_2O)_{4.1}]$

By K. O. Bennington[1] and R. R. Brown[1]

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## ABSTRACT

Cancrinite is one constituent of "red muds," a high-volume waste product of alumina production by the Bayer process. The Bureau of Mines determined the thermochemical properties of cancrinite as part of a program to provide such data for minerals of industrial importance. Cancrinite $[Na_{7.68}(Al_6Si_6O_{24})(NO_3)_{1.68}(H_2O)_{4.1}]$ was synthesized by reacting aluminum silicate gel with a solution of NaOH and $NaNO_3$, and the composition was determined by chemical analysis, differential scanning calorimetry, and thermogravimetric analysis. The heat of solution was determined by hydrofluoric acid solution calorimetry. The standard enthalpy of formation at 298.15 K, $\Delta Hf^\circ_{298}$, is

$$\Delta Hf^\circ_{298} = -3{,}487.97 \pm 2.6 \text{ kcal/mol},$$

and the standard enthalpy of formation from the oxides and $NaNO_3$, $\Delta H^\circ_{298}$, is

$$\Delta H^\circ_{298} = -215.20 \pm 1.8 \text{ kcal/mol}.$$


## INTRODUCTION

Cancrinite is a complex aluminosilicate that occurs in "red muds," which are a high-volume waste product of alumina production by the Bayer process. These muds constitute a major waste management problem for the aluminum industry. Because no heat-of-solution data for cancrinite have been reported previously in the literature, the Bureau of Mines undertook the study of this mineral as part of a program to provide thermochemical data for minerals of industrial importance. Cancrinite has a three-dimensional framework of $(Si, Al)O_4$ tetrahedra, forming a system of cages and channels. The channels contain salts such as sodium or calcium carbonate, or other substitute compounds, and the cages contain the remaining cations as well as water molecules. A comprehensive discussion of cancrinite including its molecular structure is given by Deer (6)[2] and Barrer (1-2).

The synthetic nitrate cancrinite used in this study had the composition $Na_{7.68}(Al_6Si_6O_{24})(NO_3)_{1.68}(H_2O)_{4.1}$. Experiments showed this cancrinite may be compelled to encapsulate and release foreign materials, particularly inert gases (1-2).

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[2]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

The heat of solution was determined experimentally by solution calorimetry using aqueous hydrofluoric acid as the reaction medium. No similar data have been previously reported in the literature.

## MATERIALS

### Acids

The hydrofluoric and hydrochloric acids were reagent-grade products that were used without treatment, except dilution with distilled water to the proper strength.

### Quartz ($SiO_2$)

The $SiO_2$ used was from an exceedingly clear and pure single quartz crystal. The crystal was sawed, crushed, ground to pass a 400-mesh screen, and elutriated in distilled water. The size fraction ranging between 10 and 20 μm was retained for the heat-of-solution measurements. This fraction was repeatedly leached with hydrochloric acid until the solution remained clear; it was then digested with hydrogen peroxide and dried. Each sample was heated through the alpha-beta transition immediately before solution measurements were made. No impurities were detected spectrographically, and the X-ray diffraction pattern matched that given in the Powder Diffraction File (PDF), card 5-490.

### Aluminum Chloride Hexahydrate ($AlCl_3 \cdot 6H_2O$)

The $AlCl_3 \cdot 6H_2O$ was reagent-grade material that showed spectrographic traces of Ba, Ca, and Mg, and approximately 0.05 wt-pct Na and 0.15 wt-pct Si. No corrections were made for impurities. It was stored over sulfuric acid in a desiccator until stable with 6 mol of $H_2O$. The final analysis showed 11.20 pct Al, 44.00 pct $Cl_2$, and 44.80 pct $H_2O$, as compared with the theoretical composition of 11.176 pct Al, 44.053 pct Cl, and 44.771 pct $H_2O$.

### Sodium Chloride (NaCl) and Sodium Nitrate ($NaNO_3$)

The NaCl and $NaNO_3$ were reagent-grade products that were dried at 400° and 200° C, respectively, before use.

### Cancrinite [$Na_{7.68}(Al_6Si_6O_{24})(NO_3)_{1.68}(H_2O)_{4.1}$]

### Preparation and Analysis

The cancrinite compound, supplied by Dr. Scott Barney of Atlantic Richfield Hanford Co., was synthesized by reacting aluminum silicate gel with a solution containing NaOH (sodium hydroxide) and $NaNO_3$. The results of the chemical analysis are given in table 1. The chemical composition of the cancrinite sample was calculated from the analysis and is represented by the extended formula,

$$Na_{7.681}(AlSiO_4)_6(NO_3)_{1.681}(H_2O)_{4.087}(SiO_2)_{.024},$$

giving a formula weight of 1,070.2733. The 0.024 mol $SiO_2$, equivalent to 0.135 wt-pct, was considered to be present as an impurity. The adjusted formula weight (minus the $SiO_2$ impurity) is 1,068.98, and the adjusted formula is

$$Na_{7.680}(AlSiO_4)_6(NO_3)_{1.680}(H_2O)_{4.1}.$$

This composition agrees favorably with those of nitrate cancrinites as reported by Barrer (2). The X-ray diffraction pattern matches the pattern given by PDF card 25-776 for cancrinite with ($CO_3$). Optical emission spectrographic analysis (table 1) showed traces of Cu, Fe, Ga, Mg, and Ti metallic impurities, which totaled less than 0.05 pct.

TABLE 1. - Cancrinite analysis

| Compound | wt-pct | Impurity | wt-pct |
|---|---|---|---|
| $Na_2O$..... | 22.24 | Cu........ | 0.00003-0.0003 |
| $Al_2O_3$.... | 28.58 | Fe........ | .001 - .01 |
| $SiO_2$..... | 33.82 | Ga........ | .003 - .03 |
| $N_2O_5$..... | 8.48 | Mg........ | .0001 - .001 |
| $H_2O$*..... | 6.88 | Ti........ | .001 - .01 |

*$H_2O$ content by difference.

Differential scanning calorimetry and simultaneous thermogravimetric analysis provided additional confirmation of the chemical analysis. These results were similar to those obtained by Barrer (2). In the temperature range of 25° to 548° C, only a minor continuous endothermic effect was observed, with a simultaneous weight loss of 6.81 pct, which is attributed to the evolution of the combined $H_2O$. In the temperature range of 548° to 1,150° C, the decomposition of the $NaNO_3$ occurs with a weight loss of 8.43 pct, with a primary endothermic effect observed at a peak temperature of 784° C. The results of the thermogravimetric analysis are in good agreement with the chemical analyses for $H_2O$ and $N_2O_5$ (table 1).

## EXPERIMENTAL DETERMINATIONS

### Heats of Solution at 298.15 K

The heat of formation of cancrinite was determined by hydrofluoric acid solution calorimetry. The apparatus used has been described in earlier publications (3, 8, 11). The solvent used was 948.7 g of 20.1-wt-pct hydrofluoric acid. The quantities of all reacting substances were stoichiometric with 0.742 g of $\alpha$-$SiO_2$, which when substituted into reaction 1 of the reaction scheme in table 2, provided the stoichiometric proportions for all subsequent reactions.

Weighed amounts of solid or liquid substances to be dissolved were placed in paraffin-sealed Teflon[3] tape capsules and dropped at the appropriate time, at 25° C, into the calorimeter, which was operated at 73.7° C. Each measurement resulted from a process of converting the pure reacting substance at 25° C plus the solvent at 73.7° C to a solution product at 73.7° C. Corrections were applied for the heat effects of the paraffin, Teflon, and a gold ballast. Electrical calibrations of the calorimeter were made over the temperature range of measurement.

Throughout this report uncertainties were assigned to measured and derived heat values as follows: (1) When several individual heat values were measured for a reaction, the precision uncertainty was taken as $2\sqrt{\Sigma d_i^2/n(n-1)}$, where $\Sigma d_i^2$ is the sum of the squares of the deviations from the mean value, and n is the number of determinations; (2) when the heats of two or more reactions were combined, the uncertainty was taken as the square root of the sum of the squares of the uncertainties for the individual reactions. These procedures followed the recommendations of Rossini (10).

[3]Reference to specific trade names does not imply endorsement by the Bureau of Mines.

TABLE 2. - Reaction scheme for cancrinite

| Reaction[1] | ΔH, kcal | Uncertainty, kcal |
|---|---|---|
| (1) $6SiO_2(c)$[2] + $36HF(sol)$ = $6H_2SiF_6(sol)$ + $12H_2O(sol)$.................... | -197.56 | 0.240 |
| (2) $1.68NaNO_3(c)$ = $1.68Na^+(sol)$ + $1.68NO_3^-(sol)$............................ | 6.046 | .012 |
| (3) $6NaCl(c)$ = $6Na^+(sol)$ + $6Cl^-(sol)$.................................... | -1.392 | .084 |
| (4) $285.644H_2O(\ell)$ = $285.644H_2O(sol)$.................................... | 233.657 | .857 |
| (5) $6(AlCl_3 \cdot 6H_2O)(c)$ = $6Al^{+++}(sol)$ + $18Cl^-(sol)$ + $36H_2O(sol)$.............. | -109.344 | .288 |
| (6) $24(HCl \cdot 12.731H_2O)(\ell)$ = $24H^+(sol)$ + $24Cl^-(sol)$ + $305.544H_2O(sol)$........ | 267.000 | .624 |
| (7) $Na_{7.68}(Al_6Si_6O_{24})(NO_3)_{1.68}(H_2O)_{4.1}(c)$ + $36HF(sol)$ + $24H^+(sol)$ = $6H_2SiF_6(sol)$ + $6Al^{+++}(sol)$ + $7.68Na^+(sol)$ + $1.68NO_3^-(sol)$ + $28.1H_2O(sol)$........................................ | [3]-499.583 | .205 |

$$\Delta H_8 = \Delta H_1 + \Delta H_2 + \Delta H_3 + \Delta H_4 + \Delta H_5 - \Delta H_6 - \Delta H_7$$

(8) $6SiO_2(c) + 1.68NaNO_3(c) + 6NaCl(c) + 285.644H_2O(\ell) + 6(AlCl_3 \cdot 6H_2O)(s)$
$= Na_{7.68}(Al_6Si_6O_{24})(NO_3)_{1.68}(H_2O)_{4.1}(c) + 24(HCl \cdot 12.731H_2O)(\ell)$

$$\Delta H_8 = 163.99 \pm 1.15 \text{ kcal}$$

[1]For reactions 1 through 7, reactants are at 25° C, reaction products are at 73.7° C, and solvent as indicated.

[2]For all reactions, state is designated by c, ℓ, g, and sol, indicating substances are crystalline, liquid, gas, and in solution, respectively.

[3]Corrected value.

All energy units are expressed in terms of the defined calorie (1 cal = 4.1840 J). All weighings were corrected to vacuum, and molecular weights are in accordance with the 1979 table of atomic weights (7). Final values are rounded to 10 cal. All calibrations are traceable to the National Bureau of Standards (NBS), and sample temperatures are based on the International Practical Temperature Scale of 1968 (IPTS-68) (4).

The reaction scheme for the solution calorimetric investigation is given in table 2. The symbols c, ℓ, g, and sol are used to denote substances in all reactions that are crystalline, liquid, gas, and in solution, respectively. The reactions are written in an abbreviated form sufficient to show that stoichiometry was maintained to permit cancellation of the reaction products. The table also contains the average measured heat values and their precision uncertainties.

The experimentally determined heat-of-solution values, together with the mean and the precision uncertainties that were used for the reactions presented in table 2, are listed in table 3. The heat of solution of synthetic cancrinite shown in table 3, reaction 7, was calculated for an actual weight of 1,070.27 g/mol, which included 0.024 mol of excess $SiO_2$. The formula weight was calculated to be 1,068.98 g. A thermal correction based on the heats of solution of $SiO_2$ and of the synthetic cancrinite provided a net change of +0.118 kcal. The correction to the adjusted formula weight provided a final heat of solution of -499.583 kcal/mol, which was used in the reaction scheme of table 2. The uncertainty was doubled to account for potential errors in the corrections procedure.

TABLE 3. - Experimental heats of solution, kilocalories per mole

| $SiO_2$, reaction 1 | $NaNO_3$, reaction 2 | NaCl, reaction 3 | $H_2O$, reaction 4 | $AlCl_3 \cdot 6H_2O$, reaction 5 | $HCl \cdot 12.731H_2O$, reaction 6 | $Na_{7.68}(Al_6Si_6O_{24})(NO_3)_{1.68}(H_2O)_{4.1}$, reaction 7 |
|---|---|---|---|---|---|---|
| -33.007 | +3.602 | +0.223 | 0.822 | -18.130 | 11.142 | -500.390 |
| -32.902 | 3.587 | .229 | .822 | -18.202 | 11.155 | -500.339 |
| -32.913 | 3.602 | .230 | .819 | -18.245 | 11.095 | -500.146 |
| -32.897 | 3.598 | .260 | .814 | -18.254 | 11.143 | -500.305 |
| -33.010 | 3.608 | .220 | .814 | -18.208 | 11.092 | |
| -32.849 | | | .815 | -18.305 | | |
| -32.930 | | | | | | |
| -32.897 | | | | | | |
| -32.926 ±.040 | 3.599 ±.007 | +0.232 ±.014 | 0.818 ±.003 | -18.224 ±.048 | 11.125 ±.026 | -500.295 ±.105 |

The heats of solution for $SiO_2$, $NaNO_3$, NaCl, $H_2O$, and $AlCl_3 \cdot 6H_2O$ were determined according to reactions 1 through 5, respectively, and for $HCl \cdot 12.731H_2O$ and the synthetic cancrinite, according to reactions 6 and 7, respectively. The final solutions, after conducting reactions 1 through 5 consecutively in the initial charge of acid and reactions 6 and 7 consecutively in a change of acid, are identical. Consequently, the reactions and their heats may be combined ($\Delta H_8 = \Delta H_1 + \Delta H_2 + \Delta H_3 + \Delta H_4 + \Delta H_5 - \Delta H_6 - \Delta H_7$) to give $\Delta H_8$ = 163.99 ±1.15 kcal for the overall calorimetric reaction.

## Standard Enthalpy of Formation

Calorimetric reaction 8 may be combined with auxiliary data from the literature to provide the enthalpy of formation from the component elements and from $NaNO_3$ and the component oxides. Table 4 gives the literature values of the enthalpies of formation and the uncertainty intervals for NaCl, $NaNO_3$, $Na_2O$, $H_2O$, $HCl \cdot 12.731H_2O$, $Al_2O_3$, $AlCl_3 \cdot 6H_2O$, and $SiO_2$. The sources of the values used for these compounds are as follows: NaCl, $NaNO_3$, and $Na_2O$ from Wagman (13); $H_2O$, $HCl \cdot 12.731H_2O$, and $Al_2O_3$ from Wagman (12); $AlCl_3 \cdot 6H_2O$ from Coughlin (5); and $SiO_2$ from Wise (14).

The standard enthalpy of formation, $\Delta Hf^\circ_{298}$ at 298.15 K, of synthetic cancrinite is obtained by the combination

$$\Delta H_{18} = \Delta H_8 + \Delta H_9 + \Delta H_{10} + \Delta H_{11} + \Delta H_{12} + \Delta H_{13} - \Delta H_{14},$$

and the result is

$$\Delta Hf^\circ_{298} = -3,487.97 \pm 2.6 \text{ kcal/mol}.$$

The formation from the constituent oxides and $NaNO_3$ is given by the combination

$$\Delta H_{19} = \Delta H_8 + \Delta H_9 + \Delta H_{10} - \Delta H_{14} - \Delta H_{15} - \Delta H_{16} + \Delta H_{17},$$

TABLE 4. - Enthalpy of formation of cancrinite

| | Reaction | $\Delta H_{298.15}$, kcal |
|---|---|---|
| (9) | $6Na(c) + 3Cl_2(g) = 6NaCl(c)$ | 6(-98.268±0.08) |
| (10) | $6Al(c) + 18(HCl \cdot 12.731H_2O)(\ell) = 6(AlCl_3 \cdot 6H_2O)(c) + 193.158H_2O(\ell) + 9H_2(g)$ | 6(-116.87± .13) |
| (11) | $6Si(c) + 6\ O_2(g) = 6SiO_2(c)$ | 6(-217.72± .34) |
| (12) | $1.68Na(c) + 0.84N_2(g) + 2.52\ O_2(g) = 1.68NaNO_3(c)$ | 1.68(-111.82± .20) |
| (13) | $16.1H_2(g) + 8.05\ O_2(g) = 16.1H_2O(\ell)$ | 16.1(-68.315± .01) |
| (14) | $3H_2(g) + 3Cl_2(g) + 76.386H_2O(\ell) = 6(HCl \cdot 12.731H_2O)(\ell)$ | 6(-38.82± .05) |
| (15) | $6Na(c) + 1.50\ O_2(g) = 3Na_2O(c)$ | 3(-99.00± .07) |
| (16) | $6Al(c) + 4.5\ O_2(g) = 3Al_2O_3(c)$ | 3(-400.5± .30) |
| (17) | $12H_2(g) + 6\ O_2(g) = 12H_2O(\ell)$ | 12(-68.315± .01) |

$$\Delta H_{18} = \Delta H_8 + \Delta H_9 + \Delta H_{10} + \Delta H_{11} + \Delta H_{12} + \Delta H_{13} - \Delta H_{14}$$

(18) $7.68Na(c) + 6Al(c) + 6Si(c) + 0.84N_2(g) + 4.1H_2(g) + 16.57\ O_2(g)$
$= Na_{7.68}(Al_6Si_6O_{24})(NO_3)_{1.68}(H_2O)_{4.1}(c)$

$$\Delta H_{18} = -3{,}487.967 \pm 2.560 \text{ kcal}$$

$$\Delta H_{19} = \Delta H_8 + \Delta H_9 + \Delta H_{10} - \Delta H_{14} - \Delta H_{15} - \Delta H_{16} + \Delta H_{17}$$

(19) $3Na_2O(c) + 3Al_2O_3(c) + 6SiO_2(c) + 1.68NaNO_3(c) + 4.1H_2O(\ell) = Na_{7.68}(Al_6Si_6O_{24})(NO_3)_{1.68}(H_2O)_{4.1}(c)$

$$\Delta H_{19} = -215.198 \pm 1.766 \text{ kcal}$$

and the result is

$$\Delta H^\circ_{298} \text{ (oxides)} = -215.20 \pm 1.8 \text{ kcal/mol.}$$

## DISCUSSION

Cancrinite may be considered in relation to nepheline, with which it is closely related in nature. Taking the enthalpy of formation of nepheline given by Robie (9) ($\Delta Hf^\circ_{298}$ = -500.03 kcal/mol), the reaction may be written

$$NaAlSiO_4 \text{ (nepheline)} + 0.683H_2O(\ell) + 0.28NaNO_3(c)$$
$$= 1/6[Na_{7.68}Al_6Si_6O_{24}(NO_3)_{1.68}(H_2O)_{4.1}(c)],$$

for which $\Delta H^\circ_{298}$ = -3.33 kcal. The enthalpy for the above reaction makes it appear plausible that the formation enthalpy of nitrate cancrinite may be similar to that of nepheline on a per-mol- $SiO_2$ basis.

Barrer (1) states that the saturation amount of entrained nitrate is about 1.9 mol. Barrer synthesized cancrinites with water contents varying between zero and 2.5 mol. The synthetic cancrinite used in this work had a nitrate content of 1.7 mol and a water content of 4.1 mol. Therefore, the enthalpy of formation given here is for a substance near nitrate saturation and with one of a wide range of possible water contents.

Natural cancrinites are commonly $CO_2$-rich or $SO_3$-rich. The $CO_2$-rich variety may be formed by a reaction such as nepheline + calcite → cancrinite. The cancrinites may also either be deposited with, or replace, nepheline. Excellent discussions are presented by Deer (6) and Barrer (1-2).

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