THE HEAT OF FUSION OF CaSiO₃

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By combining calorimetric measurements of the heats of solution ΔH_{sol} and of enthalpy of cooling ΔH_{cool} the temperature dependence of relative enthalpy of pseudowollastonite and that of CaSiO₃ melt was determined in the temperature intervals of 1506—1780 K and 1829—1961 K. The following values were determined on the basis of these relationships: heat of fusion of CaSiO₃ at 1817 K $\Delta H_{fus,CS,\varphi'} = 57.3 \pm 2.9$ [kJ mole⁻¹], entropy of fusion of CaSiO₃ $\Delta S_{fus,CS,\varphi'} = 31.5 \pm 1.6$ [J mole⁻¹ K⁻¹], temperature dependence of the molar heat of pseudowollastonite $C_{CS,C,\varphi} = 6.358 \times 10^{-2} + 5.724 \times 10^{-5}T$ [kJ mole⁻¹ K⁻¹] and molar heat of CaSiO₃ melt $C_{CS,m,\varphi'} = 1.4985 \times 10^{-1}$ [kJ mole⁻¹ K⁻¹] in the temperature intervals mentioned above.

INTRODUCTION

Wollastonite-CaSiO₃ (CS) arises in two modifications: α -wollastonite(pseudowollastonite) and β -wollastonite. The temperature of transformation from β -wollastonite to α -wollastonite is specified within a relatively wide range of 1393-1463 K [1] \div [3]. CaSiO₃ melts congruently at 1817 K.

The temperature dependences of the molar heat of both wollastonite modifications [3], [4] and the molar heat of $CaSiO_3$ melt are specified in literature. However, the latter quantity seems to require verification [3], [4]. The heat of fusion of $CaSiO_3$ [3] \div [7], established in the indirect way, show a considerable dispersion. For instance, in study [7] where the values of the heats of fusion of a number of silicates are discussed, six values of this quantity are listed for $CaSiO_3$, while the highest and the lowest one differ by 200%. The heat of fusion of $CaSiO_3$ has not so far been measured calorimetrically.

The present study was aimed at determining the temperature dependence of the heat content of $CaSiO_3$ melt within the 1829-1961 K temperature range and at measuring the dependence of enthalpy of pseudowollastonite within the 1506-1780 K temperature interval. The determination of the heat of fusion of $CaSiO_3$ on the basis of these measurements at an equilibrium temperature of 1817 K and that of the heat of fusion of $Ca_2MgSi_2O_7$ [8] is a contribution to the enthalpic analysis of the system $Ca_2MgSi_2O_7-CaSiO_3$.

EXPERIMENTAL

For the reasons mentioned in [8] and [9] the heat of fusion of $CaSiO_3$ was determined from the temperature dependence of the relative enthalpy of $CaSiO_3$

melt and that of pseudowollastonite. The relative enthalpy values [8], [9] of the phase in question — $H_{\rm rel}$ — at the given temperatures were determined by the ,,single sample double calorimetry" on the basis of the relationship

$$H_{\rm rel} = - \left(\Delta H_{\rm cool} + \Delta H_{\rm sol} \right),\tag{1}$$

where ΔH_{cool} is the change in enthalpy due to cooling 1 mole of the respective phase from a chosen temperature T to the temperature of 298 K, measured in a drop calorimeter [10],

 $\Delta H_{\rm sol}$ is the heat of solution of 1 mole of the respective phase in a mixture of concentrated HF and HNO₃ (2:1) at 298 K, measured in a solution calorimeter [11].

Apart from the description of the calorimeters employed, studies [10] and [11] also present a description of the measuring procedures on both types of calorimeters.

In the determination of the error involved in the measuring of relative enthalpy of pseudowollastonite and the CaSiO₃ melt it was necessary to employ a technique based on the different characters of $\Delta H_{\rm cool}$, or $\Delta H_{\rm sol}$ of the two phases. Whereas with pseudowollastonite the $\Delta H_{\rm cool}$ value depends solely on the temperature up to which the sample has been heated in the drop calorimeter furnace, in the case of the CaSiO₃ melt the value of this quantity depends with each sample in another way on sample temperature in the furnace, as well as on the structure of glass formed from the melt in the course of its cooling in the drop calorimeter. For this reason the error involved in the determination of $\Delta H_{\rm cool}$ and $\Delta H_{\rm sol}$ can therefore be determined for pseudowollastonite only.

Within the framework of the present study the error of ΔH_{cool} , cs, c, φ determination (c designates a crystalline phase) averaged by a regressive function (designated φ), $\delta(\Delta H_{\text{cool}}, \text{cs}, \text{c}, \varphi)$ according to the equation was calculated:

$$\delta(\Delta H_{\rm cool, CS, c, \varphi}) = \frac{1}{\sqrt{n}} \cdot \sqrt{\frac{S_0}{n-p}} \cdot t, \qquad (2)$$

where n is the number of enthalpy change values measured,

 $S_{\rm o}$ is the residual sum of squares,

- p is the number of independent coefficients in the regressive function,
- t is the Student's coefficient read from tables for the respective number of measurements and for the reliability coefficient $(1 \alpha) = 0.95$.

The error determined in this way corresponds to one half of the reliability interval width [12] of the physical quantity determination for the given reliability coefficient and is approximately equal to double the value of standard deviation of the Gauss' normal distribution of errors.

When determining the error involved in the determination of the heat of solution of pseudowollastonite, use was made of experience gained in the measuring of the heat of solution in the solution calorimeter. The mean relative error of one solution heat measurement thus amounted to 0.014 (the Student's coefficient t, determined from tables for the respective numbers of measurements and for the reliability coefficient $(1 - \alpha) = 0.95$ was likewise employed in the calculation). By multiplying this value by that of the arithmetic mean of the heat of solution of pseudowollastonite and by dividing by |/n, the error of the arithmetic mean of the heat of solution of pseudowollastonite was determined $\delta(\Delta H_{sol}, cs, c, \phi)$, where Φ designates the arithmetic mean.

In view of the impossibility of determining the individual errors of $\Delta H_{\rm cool}$ and $\Delta H_{\rm sol}$ for the CaSiO₃ melt, the error involved in the determination of $H_{\rm rel}$ was established from the relationship analogous to (2), where the residual sum of squares is a sum of squares of differences between the $H_{\rm rel}$ values measured and those calculated from the regressive function. The error of the determination of CaSiO₃ heat of fusion was calculated from the error of $H_{\rm rel}$ of both CaSiO₃ phases according to the "Gauss' error distribution law" (all the calculated values the error of which had been established in this way are designated with index φ').

The number of valid decimal places in the values of coefficients in the regressive functions has been chosen with respect to their considerable effect on the last significant place of the respective quantities and their errors.

Both pseudowollastonite and the CaSiO₃ glass, which was a precursor phase for the formation of the CaSiO₃ melt, were prepared by the precursor method [13]. Pseudowollastonite was prepared from a precursor with a molar ratio of CaO:SiO₂ = = 1:1, which was heated for 8 hours at about 1670 K. The presence of pure pseudowollastonite was proved by X-ray phase analysis as well as by IR spectroscopy. The CaSiO₃ glass was obtained by quenching the CaSiO₃ melt prepared by fusing pseudowollastonite and heating it for 4 hours at about 1870 K.

In the case of pseudowollastonite the measurement of one sample in the drop calorimeter yielded a change in enthalpy listed in Table I for its cooling from the temperature range of 1506–1780 K down to 298 K ($\Delta H_{\rm cool}$, cs, c). X-ray phase analysis of the sample after these measurements indicated that no transformation of pseudowollastonite to β -wollastonite had taken place in the drop calorimeter.

<i>Т</i> [К]	$\Delta H_{cool, CS, c}$ [kJ mole ⁻¹]	$\Delta H_{ m cool, CS, c, \varphi}$ [kJ mole ⁻¹]	Hrel,CS,c [kJ mole ⁻¹]	H _{rel,CS,c,φ'} [kJ mole ⁻¹]
1506	139.6	139.2	448.8	448.4
1576	150.6	149.8	459.8	459.0
1630	157.1	158.2	466.3	467.4
1678	165.0	165.8	474.2	475.0
1730	173.9	174.2	483.1	483.4
1780	183.3	182.4	492.5	491.6

Table I Pseudowollastonite

From the data measured and from the condition that the value $\Delta H_{\text{cool, CS, c}}$ is equal to zero at 298 K, the regressive function $\Delta H_{\text{cool, CS, c}} = f(T)$ was determined by the least square method,

$$-\Delta H_{\text{cool, CS, c, }\varphi} = -2.15 \times 10^{1} + 6.358 \times 10^{-2} T + 2.862 \times 10^{-5} T^{2} \qquad \text{[kJ mole^{-1}]}$$
(3)

with an error $\delta(\Delta H_{\text{cool, CS, c}}, \varphi) = 1.1 \text{ kJ mole}^{-1}$. The values of $(-\Delta H_{\text{cool, CS, c}}, \varphi)$, calculated according to this equation are listed in Table I.

Equation (3) was used for determining the temperature dependence of the molar heat of pseudowollastonite,

$$C_{\text{CS, c}, \varphi} = 6.358 \times 10^{-2} + 5.724 \times 10^{-5} T$$
 [kJ mole⁻¹ K⁻¹]. (4)

The same pseudowollastonite sample which had been used for measuring the changes in the enthalpy of cooling in the drop calorimeter was employed for the determination of the heat of solution $(\Delta H_{\rm sol, CS, c})$ in the solution calorimeter, and are listed in Table II.

Table II

 $\Delta H_{sol, CS, c}$ [kJ m ble⁻¹] 311.1 310.4 308.3 307.6 310.7 307.3

The results of the six measurements were used for determining the arithmetic mean of the heat of solution of pseudowollastonite

$$-\Delta H_{\text{sol, CS, c, } \phi} = 309.2 + 1.8$$
 [kJ mole⁻¹]. (5)

By adding the heat of solution $(-\Delta H_{\text{sol, CS, c}, \phi})$ to the data measured $(-\Delta H_{\text{cool,CS,c}})$ listed in Table I, the values $H_{\text{rel,CS,c}}$ were determined (Table I). By adding equations (3) and (5) the temperature dependence of the relative enthalpy of pseudowollastonite was determined:

$$H_{\text{rel, CS, c. }, \varphi'} = 2.877 \times 10^2 + 6.358 \times 10^{-2} T + 2.862 \times 10^{-5} T^2 \qquad \text{[kJ mole^{-1}]}$$
(6)

with an error $\delta(H_{\text{rel, CS, c, }\varphi'}) = 2.1 \text{ kJ mole}^{-1}$. The values $H_{\text{rel, CS, c, }\varphi'}$ calculated for the respective temperatures according to this equation are listed in Table I.

In the case of the CaSiO₃ melt it was necessary to determine also the value of the heat of solution on each sample for which the change of enthalpy between the chosen temperature and 298 K had been determined by the drop calorimeter. The values of relative enthalpy were therefore determined for each temperature chosen as a sum of the change in enthalpy of the CaSiO₃ melt measured in the drop calorimeter, and the heat of solution. The values of the heat of solution of each sample were determined as mean values of there measurements. Table III lists the values of the change in enthalpy due to cooling down the CaSiO₃ melt ($\Delta H_{cool, CS, m}$), the mean values of heat of solution ($\Delta H_{sol, CS, m, \sigma}$) and the values of relative enthalpy $H_{rel, CS, m}$ for the temperature interval 1829—1961 K (m — melt designation).

<i>T</i>	ΔH _{cool} , cs, m	$\frac{-\Delta H_{sol, CS, m, \phi}}{[kJ mole^{-1}]}$	H _{rel, CS, m}	H _{rel, CS, m,} φ
[K]	[kJ mole ⁻¹]		[kJ mole ⁻¹]	[kJ mole ⁻¹]
1829	217.1	340.5	557.6	556.8
1848	219.9	339.5	559.4	559.6
1859	222.2	339.0	561.2	561.3
1915	230.5	337.3	567.8	569.7
1961	238.9	339.0	577.9	576.6

Table III CaSiO₃ melt

The experimental relative enthalpy data established $(H_{rel, CS, m})$ were used for determining the temperature dependence of the relative enthalpy of the CaSiO₃ melt,

$$H_{\rm rel, CS, m, \phi} = 2.827 \times 10^2 + 1.4985 \times 10^{-1} T$$
 [kJ mole⁻¹] (7)

with an error $\delta(H_{\text{rel, CS, m, }\varphi}) = 2.0 \text{ kJ mole}^{-1}$. The relative enthalpy values for the CaSiO₃ melt calculated for the respective temperatures according to relationship (7) are listed in Table III ($H_{\text{rel, CS, m, }\varphi}$).



Fig. 1. The temperature dependence of $H_{rel,CS,\varphi}$; 1 — pseudowollastonite, 2 — CaSiO₃ melt.

Fig. 1 shows a plot of the relative enthalpies of pseudowollastonite and those of the $CaSiO_3$ melt together with the curves defined by equations (6) and (7). The value of the heat of fusion of $CaSiO_3$ at 1817 K was determined from the difference between the relative enthalpy of the $CaSiO_3$ melt and that of pseudowollastonite calculated for the melting temperature of $CaSiO_3$ (1817 K) (Fig. 1):

$$\Delta H_{\rm fus, CS, \varphi'} (1817 \text{ K}) = (57.3 \pm 2.9) \text{ kJ mole}^{-1}$$
(8)

as well as the value of the entropy of fusion of $CaSiO_3$,

$$\Delta S_{\text{fus, CS, }\phi'} (1817 \text{ K}) = (31.5 \pm 1.6) \text{ J mole}^{-1} \text{ K}^{-1}.$$
(9)

DISCUSSION

The temperature dependence of the enthalpic content of crystalline substances is most frequently described in literature by the function $-\Delta H_{\text{cool}, c, \varphi} = f_1(T) =$ $= a + bT + cT^2 + dT^{-1}$. With respect to the narrow temperature interval in which the change in enthalpy is measured during the cooling of pseudowollastonite (1506-1780 K) as well as with that to the errors involved in the measurement of this quantity, in the present case the function has been proved unsuitable for describing the relationship $-\Delta H_{\text{cool}, \text{ CS}, \text{ c}, \varphi} = f(T)$. When considering the zero value of the enthalpy of pseudowollastonite at 298 K, the relationship of this type exhibits an inflexion point. For this reason a second order polynomial was employed for describing the change in enthalpy during the cooling of pseudowollastonite — relationship (3). The course of the enthalpy of pseudowollastonite calculated from the regressive second order polynomial and from function $f_1(T)$ is compared in Fig. 2 with the values determined from tables [4] for the temperature interval of 1506-1780 K.



Fig. 2. The relationship $-\Delta H_{cool,CS,c,\phi} = f(T)$; 1 — the present study: second order polynomial — relationship (3), 2 — the present study: the regressive function has the form $-\Delta H_{cool,CS,c,\phi} = f_1(T) = a + bT + cT^2 + dT^{-1}$, 3 — according to [4].

Within the investigated temperature interval of 1829-1961 K the molar heat of the melt is constant within the framework of the errors. Its value $C_{\text{CS, m, }\varphi} = 1.4985 \times 10^{-1} \text{ kJ mole}^{-1} \text{ K}^{-1}$ is close to the value $C_{\text{CS, m, }\varphi} = 1.5073 \times 10^{-1} \text{ kJ mole}^{-1} \text{ K}^{-1}$ [4] where, however, the value $C_{\text{CS, m, }\varphi} = 1.4654 \times 10^{-1} \text{ kJ mole}^{-1} \text{ K}^{-1}$ is also specified.

Whereas the differences between the regressive functions employed for describing the temperature dependence of the enthalpy of pseudowollastonite again lie within the framework of the error involved in the method, the values as well as the temperature course of their derivations (and thus also $C_{\text{CS}, c, \varphi}$) show differences (Fig. 3). The course of the curves in Figs. 2 and 3 indicates that the suitable descriptive functions for the smoothing of the experimental values should always be chosen with utmost care.

The Heat of Fusion of CaSiO₃

The value of the heat of fusion of $\operatorname{CaSiO}_3 \Delta H_{\operatorname{fus}, \operatorname{CS}, \varphi'} = 57.3 \text{ kJ mole}^{-1}$ is in a very satisfactory agreement with the value $\Delta H_{\operatorname{fus}, \operatorname{CS}, \varphi'} = 56.1 \text{ kJ mole}^{-1}$ given in the tables [3]÷[6]. It is reported in tables [6] that the latter value has been calculated by Kelley from the data by Allen and White for the system $\operatorname{CaSiO}_3 -$ MgSiO₃. A survey of the values of the heat of fusion for CaSiO_3 has been worked out by Bottinga and Richet [7]. Among the data reported in the study the heat of fusion of CaSiO_3 measured in the present study is similar to that established by Adams and Cohen [14], ($\Delta H_{\operatorname{fus}, \operatorname{CS}, \varphi' = 50.1 \text{ kJ mole}^{-1}$) from the decrease of CaSiO_3 solidification point in the system CaSiO_3 -BaSiO₃, and to the value $\Delta H_{\operatorname{fus}, \operatorname{CS}, \varphi' = 59.0 \text{ kJ mole}^{-1}$ calculated by Bottinga and Richet [7] from the heat



Fig. 3. Temperature dependence of the molar heat of pseudowollastonite; 1 — the present study: a plot of the relation $C_{CS,c,\varphi} = b + 2cT$, 2 — the present study: $C_{CS,c,\varphi} = b + 2cT - dT^{-2}$, 3 — according to [4].

of devitrification of CaSiO₃ at 298 K, from the calculated value of the molar heat of the CaSiO₃ melt, from the data on the molar heat of pseudowollastonite [4] and from the molar heat of CaSiO₃ glass. Bottinga and Richet also report the value $\Delta H_{\text{fus, CS, }\varphi'} = 68.2 \text{ kJ mole}^{-1}$ calculated on the basis of the Clapeyron's equation. The two heat of fusion values reported for CaSiO₃ in [7], $\Delta H_{\text{fus, CS, }\varphi'} =$ $= 27.6 \text{ kJ mole}^{-1}$ and $\Delta H_{\text{fus, CS, }\varphi'} = 82.9 \text{ kJ mole}^{-1}$, however, differ quite considerably from the value established in the present study and moreover differ mutually by as much as 200%. The differences between the calculated values as well as the differences between the calculated and the experimentally established values of the thermodynamic quantities of silicates represent significant justification for their further calorimetric study.

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TEPLO TOPENIA CaSiO₃

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Z hodnôt zmien entalpie pri ochladzovaní pseudowollastonitu a taveniny CaSiO₃ medzí zvolenými teplotami v intervale 1506—1780 K, 1829—1961K a teplotou 298 K a z hodnôt príslušných rozpúšťacích tepiel pri teplote 298 K sa vypočítala závislosť relatívnej entalpie pseudowollastonitu a taveniny CaSiO₃ od teploty:

> $H_{\rm rel, CS, c, w'} = 2,877 \cdot 10^2 + 6,358 \cdot 10^{-2}T + 2,862 \cdot 10^{-5}T^2$ [kJ mol-1]

s chybou $\delta(H_{\text{rel,CS,c},\varphi'}) = 2,1 \text{ kJ mol}^{-1}$ a

 $H_{\rm rel, CS, m, \varphi} = 2,827.10^2 + 1,4985.10^{-1}T \, [\rm kJ \, mol^{-1}]$

s chybou $\delta(H_{\rm rel,CS,m,\varphi})=2,0$ kJ mol⁻¹. Z týchto vzťahov sa stanovila teplotná závislosť mólového tepla pseudowollastonitu $C_{\rm CS,c,\varphi}=6,358\cdot10^{-2}+6.724\cdot10^{-5}T$ [kJ mol⁻¹ K⁻¹] a hodnota mólového tepla taveniny $CaSiO_3$: $C_{CS,m,\varphi} = 1,4985 \cdot 10^{-1} \text{ kJ mol}^{-1} \text{ K}^{-1}$ vo vyššie uvedených teplotných intervaloch. Teplo topenia CaSiO₃ sa určilo z rozdielu relatívnych entalpií taveniny CaSiO₃ a pseudowollastonitu pri teplote 1817 K $\Delta H_{tus, CS, \varphi} = (57, 3 \pm 2, 9)$ kJ mol⁻¹ a porovnalo sa s hodnotami tejto veličiny, uvedenými v [3]-[7]. Entropia topenia CaSiO₃ $\Delta S_{\text{fus,CS}, \varphi'} = (31,5 \pm 1,6) \text{ J mol}^{-1} \text{ K}^{-1}.$

- Obr. 1. Znázornenie závislosti H_{rel,CS, φ} od teploty; 1 — pseudowollastonit. $2 - tavenina CaSiO_3$.
- Obr. 2. Závislosť — $\Delta H_{cool,CS,c,\varphi} = f(T);$ 1 — táto práca: polynóm druhého stupňa — vzťah (3), 2 — táto práca: regresná funkcia má tvar $-\Delta H_{cool, CS, c, \varphi} = f_1(T) = a + bT + cT^2 + cT^2$ $+ dT^{-1}$, 3 — podľa [4]

Obr. 3. Teplotná závislosť mólového tepla pseudowollastonitu;

- 1 táto práca: znázornenie vzťahu $C_{\text{CS,c},\varphi} = b + 2cT$,
- 2 táto práca: $C_{CS,c,\sigma} = b + 2cT dT^{-2}$,

3 - podľa [4].

ТЕПЛОТА ПЛАВЛЕНИЯ CaSiO

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На основании величин изменений энтальнии при охлаждении исевловоиластонита и расплава CaSiO₃ между подобранными температурами в интервале 1506—1780 К, 1829—1961 К и температурой 298 К и на осповании соответствующих теплот растворения при температуре 298 К расчитали зависимость относительной энтальний псевдоволластонита и расплава CaSiO₃ от температуры:

 $H_{\rm rel,CS,c,\phi'} = 2,877.10^2 + 6,358. \times 10^{-2}T + 2,862.10^{-5}T^2 \, [{\rm kmmom^{-1}}]$

с погрешностью $\delta(H_{\text{rel.CS.c.}e'}) = 2,1$ кДж.мол⁻¹ и

 $H_{\rm rel,CS,m,\sigma} = 2,827.10^2 + 1,4985.10^{-1}T$ [KI[2K.MOJI⁻¹]

с погрешностью ($H_{rel,cs,m,\varphi}$) = 2,0 кДж.мол⁻¹. Из этих отнопений установили темиературную зависимость мольной теплоты псевдоволластонита $C_{\rm CS,c,\phi} = 6,358 \cdot 10^{-2} +$ нендрагурную замления собласт теплоты песеногы песеновыме объявление соблеко – 5,000 г. (в. 5,000 – 5,724, 10-57 [кДж.мол-1, К-1] и величину мольной теплоты расплава CaSiO₃ : $C_{\text{Cs},m,\varphi} = 1,4985$. 10-1 кДж. × мол-1. К-1 в сверх приведенных температурных интер валах. Теплоту плавления CaSiO₃ и псевдоволластонита определили при температуре 1817 К $\Delta H_{\text{fus,cs},\phi}$ = (57,3 ± 2,9) кДж.мол⁻¹ и сопоставили с величинами, приводимыми в [3]-[7]. Энтропия плавления CaSiO₃∆S_{fus,CS,φ}' = (31,5±1,6) Дж.мол⁻¹. К⁻¹.

Рис. 1. Изображение зависимости H_{rel.CS.}, от температуры; 1 — псевдоволластонит,

2 — расплав СаSiO3.

Puc. 2. Зависимость — $\Delta H_{\text{cool},CS,c,q} = f(T);$

1 — приводимая работа: полином второй степени — отношение (3),

2-приводимая работа: регрессивная функция имеет вид — $\Delta H_{cool,CS,c,\varphi} =$ $f_1(T) = a + bT + cT^2 + dT^{-1}$

3 — согласно [4].

Рис. 3. Температурная зависимость мольной теплоты псевдоволластонита;

- 1 приводимая работа: изображение отношения $C_{CS,c,\varphi} = b + 2 cT$, 2 приводимая работа: $C_{CS,c,\varphi} = b + 2 cT dT^{-2}$,
- 3 согласно [4].