Research Article

Heat Capacities and Thermodynamic Properties of Pinnoite and Inderite

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In this paper, in order to understand the thermodynamic properties of natural minerals of pinnoite (MgB$_2$O$_4$·3H$_2$O, Pin) and inderite (Mg$_2$B$_6$O$_{11}$·15H$_2$O, Ind) deposited in salt lakes, heat capacities of two minerals were measured using a precision calorimeter at temperatures from 306.15 to 355.15 K after the high purity was synthesized. It was found that there are no phase transitions and thermal anomalies for the two minerals, and the molar heat capacities against temperature for Pin and Ind were fitted as $C_{p,m,Pin} = -2029.47058 + 16.94666 \times T - 0.04396 \times T^2 + 3.89409 \times 10^{-5} \times T^3$ and $C_{p,m,Ind} = -30814.43795 + 282.68108 \times T - 0.85605 \times T^2 + 8.70708 \times 10^{-4} \times T^3$, respectively. On the basis of molar heat capacities ($C_{p,m}$) of Pin and Ind, the thermodynamic functions of entropy, enthalpy, and Gibbs free energy at the temperature of 1 K interval for the two minerals were obtained for the first time.

1. Introduction

Magnesium borates are widely used in many fields such as chemical industry, glass and ceramics industries, and superconducting materials because of the excellent performance [1–4], and the extensive application of magnesium borate also makes it a great resource to be developed and utilized in the world today. Fortunately, six natural hydrated magnesium borate minerals 2MgO·3B$_2$O$_3$·15H$_2$O (inderite), 2MgO·3B$_2$O$_3$·15H$_2$O (kurnakovite), 2MgO·2B$_2$O$_3$·MgCl$_2$·14H$_2$O (chloropinnoite), MgO·B$_2$O$_3$·3H$_2$O (pinnoite), MgO·2B$_2$O$_3$·9H$_2$O (hungchaoite), and MgO·3B$_2$O$_3$·7·5H$_2$O (mcallisterite) have been found in the Qaidam Basin, China [5, 6]. It is well known that thermodynamic properties are the theoretical foundation for the exploitation of brine resources; hence, in order to provide useful information for synthesizing materials and extracting magnesium borates in salt lake brines, the study of thermodynamic properties for magnesium borates is necessary.

Heat capacity which refers to the energy absorbed or released when the unit mass changes per unit temperature [7] is one of the important thermodynamic properties for optimizing the production process, and it can be used for reliable calculation of enthalpy, entropy, and Gibbs free energy [8, 9]. In addition, heat capacity is the inherent characteristic of crystals, which is closely related to its unique composition and crystal structure. In order to understand the thermodynamic properties of natural minerals of pinnoite (MgB$_2$O$_4$·3H$_2$O, Pin) and inderite (Mg$_2$B$_6$O$_{11}$·15H$_2$O, Ind) deposited in salt lakes, the heat capacities of minerals pinnoite and inderite were measured using the calorimeter at temperatures from 306.15 to 355.15 K after the high purity was synthesized [10], and the relative thermodynamic functions of entropy, enthalpy, and Gibbs free energy were also carried out according to the thermodynamic equations for the first time.

2. Experimental

2.1. Recrystallizations or Synthesizations and Characterizations for Pinnoite and Inderite. For pinnoite purification, 30.00 g of the MgB$_2$O$_4$·H$_2$O purchased products was taken and dissolved in 100.00 g of fresh CO$_2$-free deionized distilled water (DDW), and the pH was adjusted to 8 with 50% (v/v) hydrochloric acid, and then, the solution was stirred at 200 rpm for about one week at room temperature until the solution reaches equilibrium. Next, the precipitates were filtered and washed with DDW three times as well as once with anhydrous...
ethanol and then dried at 25°C in a vacuum drying box and characterized before use. The synthesis of inderite was done as described previously [11, 12]. Briefly, 40.00 g borax was dissolved in 400.00 g of fresh CO2-free deionized distilled water (DDW) under the condition of stirring at 343.15K. After the borax dissolved completely, the temperature was decreased to 323.15K to add 40.00 g of MgSO4·7H2O, and then the solution was stirred at 200 rpm for 12 hours. The precipitate was filtered in an attemperator and then recrystallized in a thermostat water bath at 303K. Similarly, it was filtered in an attemperator and washed three times with DDW and once with anhydrous ethanol and then dried in a desiccator before use.

The pinnoite and inderite were identified by X-ray diffraction analysis (MSAL XD-3, Beijing Purkinje General Instrument Co. Ltd., China), and the results are shown in Figure 1, and it showed that the diffraction peaks on patterns correspond well with the standard Atlas database. Figure 2 shows the method for the identification of pinnoite and inderite by TG-DSC (Labsys, Setaram, France), respectively. The thermal analysis curve results of pinnoite showed that the total weight loss rate of the sample is 33.21% between 378.15 K and 673.15 K, and the total weight loss rate was 48.31%, which was basically consistent with the theoretical water loss rate of 48.29% of Mg2B6O11·15H2O from hot loss of crystalline water to Mg2B6O11. The concentration of borate expressed as B2O3 was analyzed by the gravimetric method of mannitol with an uncertainty of ±0.0005 in mass fraction [14]. The content of magnesium expressed as MgO in the sample was determined by EDTA complexometry with an uncertainty of ±0.0051 in mass fraction, and the content of H2O can be calculated by subtraction. The analytical results of MgB2O4·3H2O and Mg2B6O11·15H2O were compared with the theoretical values and are shown in Table 1, and it was shown that the purities of the synthesized pinnoite and inderite were higher than 99.9%.

2.2. Experimental Method. The high-precision calorimeter (Labsys, Setaram, France) was used for heat capacity experiment, which requires three groups of minerals, namely, blank, reference, and sample experiments, and alumina as a reference experiment. To verify the performance, the heat capacity of KCl was measured, and the average experimental value for five times of 0.6860 J g⁻¹ K⁻¹ is in accordance with 0.6879 J g⁻¹ K⁻¹ reported in the literature [15], and the deviation is 0.0028. The
heat capacities of the samples were carried out in the range of 306.15 K to 355.15 K with a heating rate of 2 K/min, putting about 15 mg of samples in the crucible weighted with an accuracy of 0.00001 g, and the flow rate of N₂ is 20 mL/min.

3. Results and Discussion

3.1. Measurement of the Heat Capacity. The values for the molar heat capacities of pinnoite and inderite were measured by the calorimeter at temperature from 306.15 to 355.15 K with the standard uncertainty of 0.05 J·mol⁻¹·K⁻¹ are listed in Table 2 and plotted in Figure 3 [16]. As shown in Figure 3, the heat capacities of pinnoite and inderite are all increased with the rise of temperature from 306.15 to 355.15 K, which shows that the structures of pinnoite and inderite are stable in these temperature regions, that is, neither phase transition nor other thermal anomalies take place within the temperature range of the experiment.

To determine the molar heat capacities, the sample weights for pinnoite and inderite were 15.92 and 15.27 mg, respectively. The temperature heating rate was 2 K·min⁻¹. The standard uncertainty \( u \) is \( u (C_{p,m}) = 0.05 \text{ J·mol}^{-1}·\text{K}^{-1} \).

### Table 1: Chemical analytical results of pinnoite (MgB₂O₄·3H₂O) and inderite (Mg₂B₆O₁₁·15H₂O)².

| Component content | Purity | \( w \) (MgO) | \( w \) (B₂O₃) | \( w \) (H₂O) | \( n \) (MgO: B₂O₃: H₂O) |
|-------------------|--------|---------------|---------------|---------------|-----------------|
| Pinnoite, MgB₂O₄·3H₂O | Theoretical | 1.0000 | 0.2459 | 0.4247 | 0.3274 | 1.00:1.00:3.00 |
| | Experimental | 0.9900 | 0.2447 | 0.4243 | 0.3300 | 1.00:1.00:3.01 |
| Inderite, Mg₂B₆O₁₁·15H₂O | Theoretical | 1.0000 | 0.1440 | 0.3732 | 0.4828 | 2.00:3.00:15.00 |
| | Experimental | 0.9990 | 0.1439 | 0.3734 | 0.4827 | 2.00:3.00:15.02 |

²Standard uncertainties \( u \) are \( u(\text{MgO}) = 0.0051, u(\text{B₂O₃}) = 0.0005, \) and \( u(\text{H₂O}) = 0.0079 \) in mass fraction.

| Table 2: The molar heat capacities of pinnoite and inderite at 306.15–355.15 K². |
|--------------------------|-----------------|-----------------|-----------------|
| \( T \) (K) | \( C_{p,m} \) (J·mol⁻¹·K⁻¹) | \( T \) (K) | \( C_{p,m} \) (J·mol⁻¹·K⁻¹) | \( T \) (K) | \( C_{p,m} \) (J·mol⁻¹·K⁻¹) |
| Pinnoite, MgB₂O₄·3H₂O | 306.15 | 154.85 | 323.15 | 169.84 | 340.15 | 181.16 |
| | 307.15 | 155.94 | 324.15 | 170.55 | 341.15 | 181.66 |
| | 308.15 | 157.45 | 325.15 | 171.22 | 342.15 | 182.19 |
| | 309.15 | 158.71 | 326.15 | 171.93 | 343.15 | 182.68 |
| | 310.15 | 160.76 | 328.15 | 173.30 | 345.15 | 183.63 |
| | 311.15 | 161.68 | 329.15 | 173.96 | 346.15 | 184.09 |
| | 312.15 | 162.60 | 330.15 | 174.65 | 347.15 | 184.53 |
| | 313.15 | 163.39 | 331.15 | 175.37 | 348.15 | 184.96 |
| | 314.15 | 164.14 | 332.15 | 176.07 | 349.15 | 185.40 |
| | 315.15 | 164.88 | 333.15 | 176.80 | 350.15 | 185.86 |
| | 317.15 | 165.52 | 334.15 | 177.57 | 351.15 | 186.34 |
| | 318.15 | 166.17 | 335.15 | 178.27 | 352.15 | 186.86 |
| | 319.15 | 166.84 | 336.15 | 178.91 | 353.15 | 187.43 |
| | 320.15 | 167.57 | 337.15 | 179.53 | 354.15 | 188.04 |
| | 321.15 | 168.32 | 338.15 | 180.09 | 355.15 | 188.70 |
| | 322.15 | 169.07 | 339.15 | 180.63 | | |
| Inderite, Mg₂B₆O₁₁·15H₂O | 306.15 | 474.94 | 323.15 | 521.82 | 340.15 | 560.43 |
| | 307.15 | 479.69 | 324.15 | 524.12 | 341.15 | 562.89 |
| | 308.15 | 484.73 | 325.15 | 526.36 | 342.15 | 565.29 |
| | 309.15 | 488.20 | 326.15 | 528.59 | 343.15 | 567.98 |
| | 310.15 | 490.94 | 327.15 | 530.77 | 344.15 | 570.78 |
| | 311.15 | 493.51 | 328.15 | 533.01 | 345.15 | 573.30 |
| | 312.15 | 495.98 | 329.15 | 535.19 | 346.15 | 576.09 |
| | 313.15 | 498.44 | 330.15 | 537.38 | 347.15 | 578.95 |
| | 314.15 | 500.45 | 331.15 | 539.50 | 348.15 | 581.91 |
| | 315.15 | 502.52 | 332.15 | 541.70 | 349.15 | 584.65 |
| | 316.15 | 504.70 | 333.15 | 543.75 | 350.15 | 587.56 |
| | 317.15 | 506.89 | 334.15 | 545.82 | 351.15 | 590.97 |
| | 318.15 | 509.24 | 335.15 | 548.06 | 352.15 | 595.06 |
| | 319.15 | 511.70 | 336.15 | 550.52 | 353.15 | 600.09 |
| | 320.15 | 514.21 | 337.15 | 552.93 | 354.15 | 605.63 |
| | 321.15 | 516.73 | 338.15 | 555.50 | 355.15 | 611.12 |
| | 322.15 | 519.25 | 339.15 | 557.97 | | |
Figure 3: Molar heat capacities versus temperature in the range of 306.15 K to 355.15 K for pinnoite (a) and inderite (b).

Table 3: Polynomial coefficients of equation (1) for minerals of pinnoite and inderite at 306.15 ~355.15 K.

| Minerals                  | A₀       | A₁       | A₂       | A₃       | r        |
|---------------------------|----------|----------|----------|----------|----------|
| MgB₂O₃·3H₂O               | -2029.47 | 16.9466  | -0.04396 | 3.89409 × 10⁻⁵ | 0.99905  |
| Mg₂B₆O₁₁·15H₂O            | -30814.4 | 282.6810 | -0.85605 | 8.70708 × 10⁻⁴ | 0.99924  |

Table 4: Molar heat capacities and thermodynamic functions (H_T−H_{298.15}), (S_T−S_{298.15}), and (G_T−G_{298.15}) of pinnoite and inderite.

| T (K) | C_{p,m} (J·mol⁻¹·K⁻¹) | H_T−H_{298.15} (kJ·mol⁻¹) | S_T−S_{298.15} (J·mol⁻¹·K⁻¹) | G_T−G_{298.15} (kJ·mol⁻¹) |
|-------|------------------------|---------------------------|-------------------------------|--------------------------|
| Pinnoite, MgB₂O₄·3H₂O | 298.15 | 147.49 | 0.0000 | 0.0000 | 0.0000 |
|       | 306.15 | 154.85 | 1.2142 | 4.0183 | 0.0160 |
|       | 307.15 | 155.94 | 1.3706 | 4.5282 | 0.0203 |
|       | 308.15 | 157.45 | 1.5279 | 5.0396 | 0.0251 |
|       | 309.15 | 158.71 | 1.6862 | 5.5524 | 0.0303 |
|       | 310.15 | 159.85 | 1.8454 | 6.0665 | 0.0362 |
|       | 311.15 | 160.76 | 2.0055 | 6.5820 | 0.0425 |
|       | 312.15 | 161.68 | 2.1665 | 7.0986 | 0.0493 |
|       | 313.15 | 162.60 | 2.3284 | 7.6165 | 0.0567 |
|       | 314.15 | 163.39 | 2.4912 | 8.1355 | 0.0646 |
|       | 315.15 | 164.14 | 2.6548 | 8.6555 | 0.0730 |
|       | 316.15 | 164.88 | 2.8193 | 9.1766 | 0.0819 |
|       | 317.15 | 165.52 | 2.9846 | 9.6986 | 0.0913 |
|       | 318.15 | 166.17 | 3.1507 | 10.2215 | 0.1013 |
|       | 319.15 | 166.84 | 3.3176 | 10.7453 | 0.1117 |
|       | 320.15 | 167.57 | 3.4853 | 11.2700 | 0.1228 |
|       | 321.15 | 168.32 | 3.6538 | 11.7954 | 0.1343 |
|       | 322.15 | 169.07 | 3.8230 | 12.3215 | 0.1463 |
|       | 323.15 | 169.84 | 3.9930 | 12.8483 | 0.1589 |
|       | 324.15 | 170.55 | 4.1637 | 13.3758 | 0.1720 |
|       | 325.15 | 171.22 | 4.3352 | 13.9039 | 0.1857 |
|       | 326.15 | 171.93 | 4.5073 | 14.4325 | 0.1999 |
|       | 327.15 | 172.60 | 4.6802 | 14.9616 | 0.2145 |
|       | 328.15 | 173.30 | 4.8537 | 15.4913 | 0.2298 |
|       | 329.15 | 173.96 | 5.0279 | 16.0213 | 0.2455 |
|       | 330.15 | 174.65 | 5.2028 | 16.5518 | 0.2618 |
|       | 331.15 | 175.37 | 5.3783 | 17.0827 | 0.2786 |
|       | 332.15 | 176.07 | 5.5545 | 17.6139 | 0.2960 |
|       | 333.15 | 176.80 | 5.7313 | 18.1453 | 0.3139 |
### Table 4: Continued.

| $T$ (K) | $C_{p,m}^\alpha$ (J·mol$^{-1}$·K$^{-1}$) | $H_T - H_{298.15}$ (kJ·mol$^{-1}$) | $S_T - S_{298.15}$ (J·mol$^{-1}$·K$^{-1}$) | $G_T - G_{298.15}$ (kJ·mol$^{-1}$) |
|---------|---------------------------------|--------------------------------------|---------------------------------|----------------------------------|
| 334.15  | 177.57                          | 5.9087                               | 18.6771                         | −0.3323                         |
| 335.15  | 178.27                          | 6.0867                               | 19.2091                         | −0.3512                         |
| 336.15  | 178.91                          | 6.2654                               | 19.7413                         | −0.3707                         |
| 337.15  | 179.53                          | 6.4446                               | 20.2737                         | −0.3907                         |
| 338.15  | 180.09                          | 6.6244                               | 20.8063                         | −0.4112                         |
| 339.15  | 180.63                          | 6.8048                               | 21.3389                         | −0.4323                         |
| 340.15  | 181.16                          | 6.9857                               | 21.8717                         | −0.4539                         |
| 341.15  | 181.66                          | 7.1673                               | 22.4045                         | −0.4761                         |
| 342.15  | 182.19                          | 7.3493                               | 22.9374                         | −0.4987                         |
| 343.15  | 182.68                          | 7.5319                               | 23.4703                         | −0.5219                         |
| 344.15  | 183.16                          | 7.7151                               | 24.0033                         | −0.5457                         |
| 345.15  | 183.63                          | 7.8987                               | 24.5361                         | −0.5699                         |
| 346.15  | 184.09                          | 8.0829                               | 25.0690                         | −0.5947                         |
| 347.15  | 184.53                          | 8.2676                               | 25.6018                         | −0.6201                         |
| 348.15  | 184.96                          | 8.4528                               | 26.1345                         | −0.6459                         |
| 349.15  | 185.40                          | 8.6385                               | 26.6671                         | −0.6723                         |
| 350.15  | 185.86                          | 8.8246                               | 27.1995                         | −0.6993                         |
| 351.15  | 186.34                          | 9.0113                               | 27.7319                         | −0.7267                         |
| 352.15  | 186.86                          | 9.1985                               | 28.2641                         | −0.7547                         |
| 353.15  | 187.43                          | 9.3861                               | 28.7961                         | −0.7833                         |
| 354.15  | 188.04                          | 9.5742                               | 29.3279                         | −0.8123                         |
| 355.15  | 188.70                          | 9.7627                               | 29.8596                         | −0.8419                         |

*Inderite, Mg$_3$B$_6$O$_{11}$-15H$_2$O*
In order to obtain the heat capacity quickly at a certain temperature, the molar heat capacities of pinnoite and indirite determined in this work have been fitted as equation (1) by means of a least-squares method with the correlation coefficients $r = 0.99905$ for pinnoite and $0.99924$ for indirite listed in Table 3. On the basis of polynomial equations, the molar heat capacities of pinnoite and indirite have been fitted as equation (1) by means of a least-squares method with the correlation coefficients $r = 0.99905$ for pinnoite and $0.99924$ for indirite listed in Table 3. On the basis of polynomial equations, the

| $T$ (K) | $C_{p,m}$ (J·mol$^{-1}$·K$^{-1}$) | $H_T - H_{298.15}$ (kJ·mol$^{-1}$) | $S_T - S_{298.15}$ (J·mol$^{-1}$·K$^{-1}$) | $G_T - G_{298.15}$ (kJ·mol$^{-1}$) |
|--------|-------------------------------|-----------------------------------|----------------------------------------|-----------------------------------|
| 342.15 | 565.29                        | 22.5500                           | 70.3701                                | -1.5271                           |
| 343.15 | 567.98                        | 23.1170                           | 72.0248                                | -1.5983                           |
| 344.15 | 570.78                        | 23.6867                           | 73.6828                                | -1.6712                           |
| 345.15 | 573.30                        | 24.2593                           | 75.3442                                | -1.7457                           |
| 346.15 | 576.09                        | 24.8349                           | 77.0092                                | -1.8219                           |
| 347.15 | 578.95                        | 25.4134                           | 78.6782                                | -1.8997                           |
| 348.15 | 581.91                        | 25.9951                           | 80.3514                                | -1.9792                           |
| 349.15 | 584.65                        | 26.5800                           | 82.0290                                | -2.0604                           |
| 350.15 | 587.56                        | 27.1682                           | 83.7113                                | -2.1433                           |
| 351.15 | 590.97                        | 27.7599                           | 85.3987                                | -2.2278                           |
| 352.15 | 595.06                        | 28.3552                           | 87.0914                                | -2.3141                           |
| 353.15 | 600.09                        | 28.9541                           | 88.7898                                | -2.4020                           |
| 354.15 | 605.63                        | 29.5569                           | 90.4942                                | -2.4917                           |
| 355.15 | 611.12                        | 30.1636                           | 92.2050                                | -2.5830                           |

*The standard uncertainties $u$ are $u(C_{p,m}) = 0.05$ J·mol$^{-1}$·K$^{-1}$, $u(H_T - H_{298.15}) = 0.0056$, $u(S_T - S_{298.15}) = 0.0080$, and $u(G_T - G_{298.15}) = 0.0010$. 

Figure 4: Variations of (a) $(H_T - H_{298.15})$, (b) $(S_T - S_{298.15})$, and (c) $(G_T - G_{298.15})$ with $T$ for pinnoite and indirite.
molar heat capacities of pinnoite and inderite at 298.15 K can be calculated as 147.49 and 446.59 J·mol⁻¹·K⁻¹, and the standard deviations between experimental values $C_{p, exp}$ and fitted values $C_{p, fit}$ through the polynomial equation are within 0.02:

$$C_{p,m}(J \cdot mol^{-1} \cdot K^{-1}) = A_0 + A_1 T + A_2 T^2 + A_3 T^3.$$  \hspace{1cm} (1)

3.2. Enthalpy, Entropy, and Gibbs Free Energy. The thermodynamic functions of the relative standard states of pinnoite and inderite at 298.15 K can be derived from the following thermodynamic equations:

$$H_T - H_{298.15} = \int_{298.15}^{T} C_{p,m}dT,$$

$$S_T - S_{298.15} = \int_{298.15}^{T} \frac{C_{p,m}}{T}dT,$$

$$G_T - G_{298.15} = \int_{298.15}^{T} C_{p,m}dT - T \int_{298.15}^{T} \frac{C_{p,m}}{T}dT.$$  \hspace{1cm} (2)

Using the formula above, the thermodynamic functions $(H_T - H_{298.15})$, $(S_T - S_{298.15})$, and $(G_T - G_{298.15})$ of pinnoite and inderite are calculated on the basis of the obtained heat capacity values and are listed in Table 4 with a temperature of 1 K interval.

From the thermodynamic function data listed in Table 4, it is shown that with the increase of temperature from 298.15 K to 355.15 K, the molar heat capacity, enthalpy $(H_T - H_{298.15})$, and entropy $(S_T - S_{298.15})$ increase with the increase of temperature, while the change of free energy $(G_T - G_{298.15})$ is just the opposite. The variations of $(H_T - H_{298.15})$, $(S_T - S_{298.15})$, and $(G_T - G_{298.15})$ with $T$ are plotted in Figure 4.

4. Conclusions

In the work, pinnoite and inderite have been successfully synthesized and characterized, and then the solid molar heat capacities of $\text{MgB}_2\text{O}_4$·$3\text{H}_2\text{O}$ and $\text{Mg}_2\text{B}_6\text{O}_{11}$·$15\text{H}_2\text{O}$ were measured by differential scanning calorimetry. The experimental results showed that the molar heat capacities of the two magnesium borates are increased with the increase of temperature. No phase transitions or other thermal anomalies occurred. At the same time, heat capacity at various temperatures was fitted by the least-square method, and the polynomial equation of the change of the molar heat capacity with the temperature is obtained. The fitting results are in good agreement with the experimental data and can be used for the exploitation and utilization of magnesium and boron resources in salt lakes.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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The authors declare that they have no conflicts of interest.

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