

## SOLUBILITY OF CO<sub>2</sub> IN MOLTEN Li<sub>2</sub>CO<sub>3</sub>-LiCl

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### Abstract

Solubility of CO<sub>2</sub> in molten Li<sub>2</sub>CO<sub>3</sub>-LiCl was measured by a pressure differential method, and the enthalpy change of the solution was calculated on that basis. The relationships between the solubility and the enthalpy change, and the temperature and the composition of the melts were discussed. The results showed that when the temperature was 873–923 K and the Li<sub>2</sub>CO<sub>3</sub> content was 10–50 mol%, the solubility of CO<sub>2</sub> increased with decreasing temperature and/or increasing Li<sub>2</sub>CO<sub>3</sub> content. The maximum solubility was  $3.965 \times 10^{-7} \text{ g}_{\text{CO}_2}/\text{g}_{\text{melt}}$  at 873 K when the content of Li<sub>2</sub>CO<sub>3</sub> was 50 mol%. The solution of CO<sub>2</sub> was exothermic. With increasing temperature and Li<sub>2</sub>CO<sub>3</sub> content, more enthalpy was needed for CO<sub>2</sub> solution.

**Keywords:** MSCC-ET; Li<sub>2</sub>CO<sub>3</sub>-LiCl melts; CO<sub>2</sub> solubility; Pressure differential method; Solution enthalpy change

### 1. Introduction

Carbon fuels have been exploited rapidly as human industrialisation has progressed. This has resulted in large emissions of CO<sub>2</sub>, the most concerning greenhouse gas. Accordingly, there has been much attention on how to effectively decrease CO<sub>2</sub> emissions and carbon fuel use.

Electrochemical transformation is an important method for CO<sub>2</sub> utilisation. A higher temperature is more helpful for the electrochemical reduction of CO<sub>2</sub> from the viewpoint of both thermodynamics and kinetics. Therefore, molten salt is a better medium for the electrochemical transformation of CO<sub>2</sub> than an aqueous solution. Furthermore, molten salt often has a wider electrochemical potential window. As a result, the molten salt CO<sub>2</sub> capture and electrochemical transformation (MSCC-ET) process has become a research hotspot. This process produces carbon and oxygen, with high value-added carbon materials obtained by improving the electrode, electrolyte, and/or process conditions [1-3], and oxygen generation achieved from utilising solar energy, enabling humans to breathe and survive in outer space [4-5].

For the MSCC-ET process, molten salts that have been reported to date include carbonate [1, 6-10], chloride-oxide [5, 11], chloride-carbonate [3, 12-14], fluoride-carbonate [4, 15], and carbonate-oxide [16].

In this process, it is important to understand the solubility of CO<sub>2</sub> in the melt, because this affects the electrical conductivity of the electrolyte, as well as the current efficiency during the electrolysis process [17].

Clase et al. measured the solubility of CO<sub>2</sub> in a molten Na<sub>2</sub>CO<sub>3</sub>-K<sub>2</sub>CO<sub>3</sub> eutectic mixture at 1073 K and Henry's constant was determined as  $1.83 \times 10^{-1} \text{ mol}\cdot\text{L}^{-1}\cdot\text{atm}^{-1}$  [18]. Clase et al. also measured CO<sub>2</sub> solubility in molten Li<sub>2</sub>CO<sub>3</sub>-Na<sub>2</sub>CO<sub>3</sub>-K<sub>2</sub>CO<sub>3</sub> (43.5, 31.5 and 25.0 mol%) at 973 K, and the solubility was determined as  $9.5 \times 10^{-2} \text{ mol}\cdot\text{L}^{-1}$  under a CO<sub>2</sub> pressure of 1 atm [19].

Wakamatsu et al. studied the solubility of CO<sub>2</sub> in molten LiCl-Li<sub>2</sub>O using the mass measurement technique, reporting a 95% solution of the molar quantity of Li<sub>2</sub>O into the molten salts in the range of 0–60 mol% Li<sub>2</sub>O at 923 K [20]. Our previous study on the same melts using Raman spectroscopy indicated that the solubility of CO<sub>2</sub> was  $0.1105 \text{ g}_{\text{CO}_2}/\text{g}_{\text{melt}}$  for the melts containing 8 wt% of Li<sub>2</sub>O at 923 K and the conversion rate of Li<sub>2</sub>O to Li<sub>2</sub>CO<sub>3</sub> was 94.19% [17].

Shi et al. studied the solubility of CO<sub>2</sub> in molten LiF-Li<sub>2</sub>CO<sub>3</sub>. The maximum solubility was  $6.8 \times 10^{-4} \text{ mol}_{\text{CO}_2}/\text{mol}_{\text{melt}}$  at 913 K when the mole fraction of LiF was 50% [21]. Deng found the absorption of CO<sub>2</sub> in molten LiCl-KCl to be negligible; however, CO<sub>2</sub> could be rapidly captured when Li<sub>2</sub>O or CaO was added into molten LiCl-KCl, with a conversion efficiency of Li<sub>2</sub>O to Li<sub>2</sub>CO<sub>3</sub> of around 94%. CO<sub>2</sub>

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solubility in molten Li-Na-K carbonates was much higher than in molten LiCl-KCl, with approximately 6 mmol CO<sub>2</sub> absorbed by 100 g Li-Na-K carbonate. More time was required to reach the absorption equilibrium in molten carbonates with CaO or Li<sub>2</sub>O, and the conversion efficiency of Li<sub>2</sub>O was approximately 45% [22].

On the other hand, considering the laborious experimental conditions to determine the CO<sub>2</sub> solubility, some theoretical models were developed and used to model the CO<sub>2</sub> solubility in the electrolyte, such as Statistical Associating Fluid Theory (SAFT) [23, 24], Cubic-Plus-Association (e-CPA) [25, 26], and Mixed-Solvent Electrolyte (MSE) [27]. However, these models have all been applied for the aqueous systems or ionic liquids but few models were focused on the molten salts systems.

Compared with other molten salts systems used in MSCC-ET process, molten chloride-carbonates are getting more attention for their lower operating temperature and cost. However, there have been few research publications on the solubility of CO<sub>2</sub> in molten chloride-carbonates. In the present study, the solubility of CO<sub>2</sub> in molten Li<sub>2</sub>CO<sub>3</sub>-LiCl was studied using the pressure differential method, and the effects of temperature and Li<sub>2</sub>CO<sub>3</sub> content were comprehensively determined. Data on the thermodynamics of CO<sub>2</sub> solution was also calculated.

## 2. Experimental

The purity and company of the reagents are listed in Table S1. All solid chemicals were dried at 423 K for 24 h to remove water and then stored in an argon-filled glove box with water and oxygen contents less than 1 ppm.

The solubility of CO<sub>2</sub> in molten Li<sub>2</sub>CO<sub>3</sub>-LiCl was measured using a pressure differential method. The apparatus used for the experiment is shown in Figure 1. The same apparatus and method were used to measure the solubility of CO<sub>2</sub> in molten Li<sub>2</sub>CO<sub>3</sub>-LiF [21]. The molten salts sample was held in a corundum crucible which was placed in a stainless steel container. The container was put in a furnace connecting to a temperature controller for heating to a certain temperature. The valves were used to control gas flows, the two pumps were for evacuating the air, and the digital pressure gauge was for reading the pressure.

**Table S1.** Information on the reagents

Regent	Purity	Company
Li <sub>2</sub> CO <sub>3</sub>	>99%	Alfa
LiCl	>99.9%	Alfa
CO <sub>2</sub>	>99.5%	Sifang(Shenyang, China)

The volume ( $V_m$ ) of the container and connecting tube was measured using CO<sub>2</sub> according to the following procedure. First, the volume ( $V_1$ ) in the bellow was measured by the water displacement method. Second, the air in the whole apparatus was evacuated and the container and connecting tube were filled with CO<sub>2</sub>, giving a pressure reading of  $P_0$ . Third, Valve 8 was opened to allow CO<sub>2</sub> to flow into the bellow, giving a pressure reading of  $P_0'$ . Finally, the  $V_m$  value was calculated using equation (1).

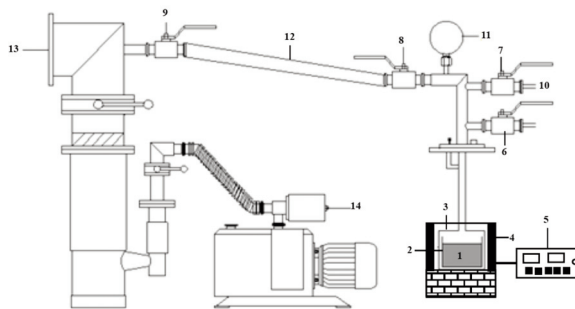
$$P_0'V_m = P_0(V_m + V_1) \quad (1)$$

With  $V_m$  thus obtained, the concentration of CO<sub>2</sub> in the melts could be measured. First, a sample with a certain mass ( $m$ ) was placed in the container and heated to the target temperature. The volume of the melt was calculated by knowing the value of  $m$  and the density, which was measured using the Archimedes' principle [28]. Second, CO<sub>2</sub> was allowed to flow in by opening Valve 7. Finally, the Soave-Redlich-Kwong equation [29] of state was used to calculate the number of moles of CO<sub>2</sub> dissolved into the melt. Then, the number of moles was converted to the mass of CO<sub>2</sub>.

## 3. Results and discussions

### 3.1. Determination of dissolution equilibrium duration time

To determine the dissolution equilibrium duration, the pressure changes in the container for the Li<sub>2</sub>CO<sub>3</sub>-LiCl melts with different compositions were measured at 873 K, as shown in Figure 2 (The related data were listed in Table S2). When the pressure in the container remained constant, CO<sub>2</sub> dissolution had reached equilibrium. The reaction between CO<sub>2</sub> and the melts is given in equation (2) [30]. The amount of CO<sub>2</sub> dissolved in the melts increased with increasing time. However, after 60–80 minutes there was little



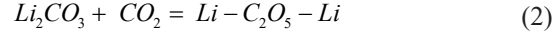
**Figure 1.** The apparatus for measuring the solubility of CO<sub>2</sub>. 1- Molten salts; 2-Corundum crucible 3- Container; 3 Furnace; 4-Temperature controller; 5, 6, 7, 8, 9-Valves; 10-CO<sub>2</sub> let; 11-Pressure gage; 12-Bellow; 13-Diffusion pump; 14-Vacuum pump



**Table S2.** Values of pressure and dissolution during time of CO<sub>2</sub> in molten Li<sub>2</sub>CO<sub>3</sub>-LiCl systems at 873K

$x_{Li_2CO_3}$ /mol %	Dissolution during time/min	Pressure of CO <sub>2</sub> /MPa
10	10	0.0918
	20	0.0927
	30	0.0935
	40	0.0937
	50	0.0940
	60	0.0942
	70	0.0943
	80	0.0945
	90	0.0946
	100	0.0945
	110	0.0945
20	10	0.0933
	20	0.0954
	30	0.0982
	40	0.0997
	50	0.1021
	60	0.1025
	70	0.1026
	80	0.1027
	90	0.1027
	100	0.1027
	110	0.1027
30	10	0.0948
	20	0.0973
	30	0.9998
	40	0.1015
	50	0.1019
	60	0.1024
	70	0.1025
	80	0.1025
	90	0.1025
	100	0.1025
	110	0.1025
40	10	0.0946
	20	0.0954
	30	0.0970
	40	0.0990
	50	0.1002
	60	0.1015
	70	0.1018
	80	0.1023
	90	0.1023
	100	0.1023
	110	0.1023

further change, indicating that dissolution equilibrium had been reached. In the following solubility measurements, the pressure was read after 80 minutes.



### 3.2. Solubility of CO<sub>2</sub> in molten Li<sub>2</sub>CO<sub>3</sub>-LiCl

The relationship between the solubility of CO<sub>2</sub> in the melts with various compositions and temperature is shown in Figure 3 (The related data were listed in Table S3). Solubility decreased with increasing temperature, which indicated the equilibrium between Li<sub>2</sub>CO<sub>3</sub> and Li-C<sub>2</sub>O<sub>5</sub>-Li in equation (2) shifted left. Therefore, the temperature must not be set too high when Li<sub>2</sub>CO<sub>3</sub>-LiCl melts are used as the medium for CO<sub>2</sub> electrolysis. Additionally, solubility increased as Li<sub>2</sub>CO<sub>3</sub> content increased, indicating the very low solubility of CO<sub>2</sub> in the molten chloride, whereas the molten carbonate allowed CO<sub>2</sub> dissolution. The effect of LiCl in the melts was only to decrease the liquidus temperature and thereby decrease the working temperature.

Overall, the solubility of CO<sub>2</sub> in the Li<sub>2</sub>CO<sub>3</sub>-LiCl melts was much lower than that in the Li<sub>2</sub>O-LiCl melts (approximately 10<sup>-5</sup> times lower), in the studied ranges of temperature and sample compositions. The maximum solubility of CO<sub>2</sub> was 3.965 × 10<sup>-7</sup> g<sub>CO<sub>2</sub></sub>/g<sub>melt</sub> at 873 K when the content of Li<sub>2</sub>CO<sub>3</sub> was 50 mol%.

### 3.3. The enthalpy change of the solution of CO<sub>2</sub>

The enthalpy change  $\Delta_{sol}H$  of CO<sub>2</sub> solution in the Li<sub>2</sub>CO<sub>3</sub>-LiCl system was expressed according to equations (3) [31].

$$\Delta_{sol}H = R \left( \frac{\partial \ln(K_H(T, P) / P^0)}{\partial (1/T)} \right)_p \quad (3)$$

where,  $K_H(T, P)$  is effective Henry constant (L·mol<sup>-1</sup>Pa<sup>-1</sup>), which can be expressed according to equation (4).

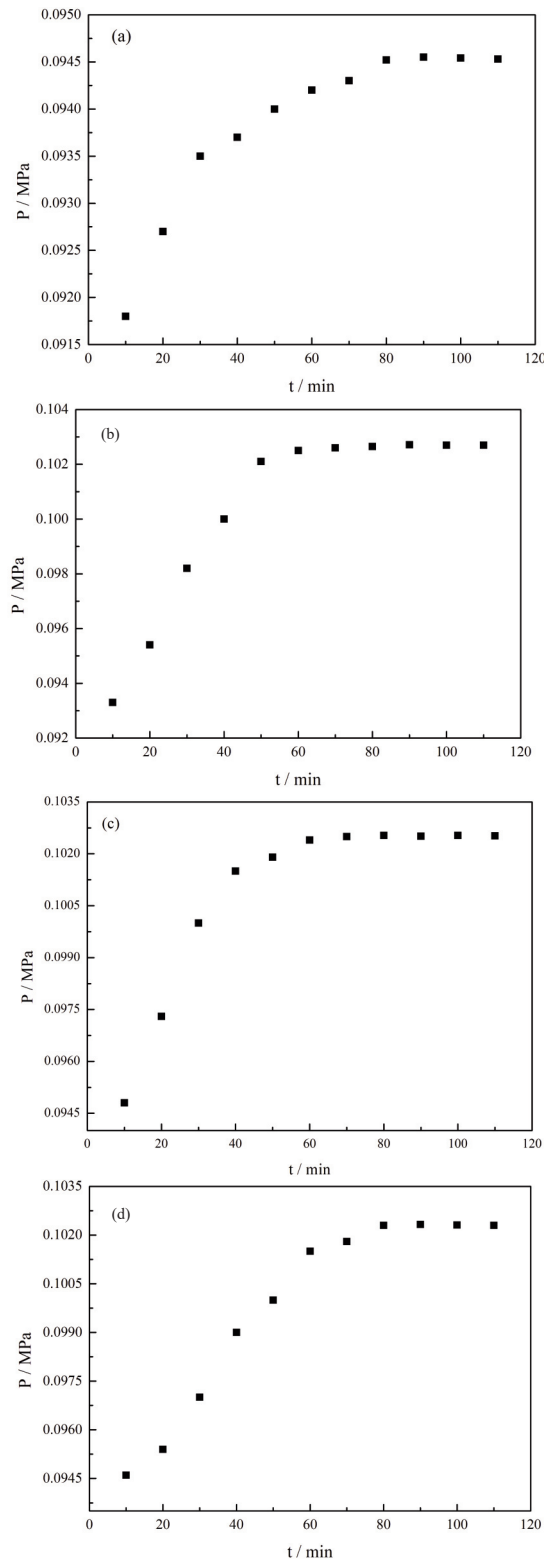
$$K_H(T, P) = \phi_{CO_2}(T, P) P_{CO_2}^m / C_{(CO_2)} \quad (4)$$

where,  $P_{CO_2}^m$  is the equilibrium pressure of CO<sub>2</sub> (MPa) and  $\phi_{CO_2}(T, P)$  is the fugacity coefficient of CO<sub>2</sub>, which can be calculated using the Soave method [31].

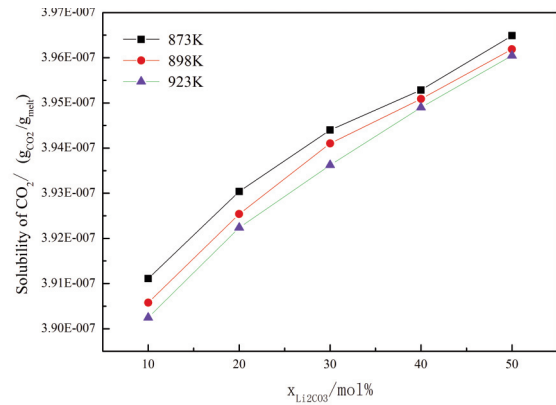
The relationship between  $\Delta_{sol}H$  and Li<sub>2</sub>CO<sub>3</sub> content in the melts at different temperatures is shown in Figure 4 (The related data were listed in Table S4).

From Figure 4 it can be seen that the value of  $\Delta_{sol}H$  was negative; therefore, CO<sub>2</sub> dissolution was an exothermic process. The value of  $\Delta_{sol}H$  decreased with increasing temperature and Li<sub>2</sub>CO<sub>3</sub> content, which indicated more enthalpy was needed for CO<sub>2</sub> solution.

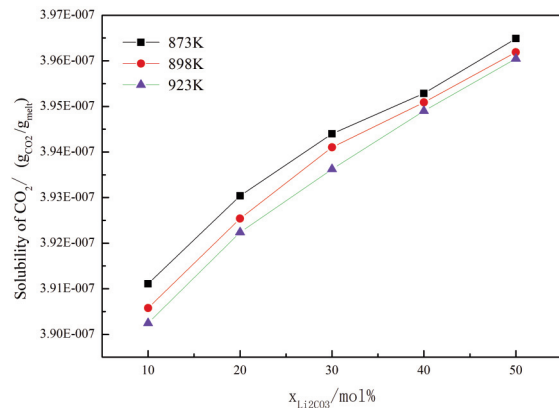




**Figure 2.** Relationship between pressure and dissolution during time of  $\text{CO}_2$  in molten  $\text{Li}_2\text{CO}_3$ - $\text{LiCl}$  systems at 873K; (a)  $\text{LiCl}$ -10mol% $\text{Li}_2\text{CO}_3$ ; (b)  $\text{LiCl}$ -20mol% $\text{Li}_2\text{CO}_3$ ; (c)  $\text{LiCl}$ -30mol% $\text{Li}_2\text{CO}_3$ ; (d)  $\text{LiCl}$ -40mol% $\text{Li}_2\text{CO}_3$



**Figure 3.** Solubility curves of  $\text{CO}_2$  in  $\text{Li}_2\text{CO}_3$ - $\text{LiCl}$  melts for different compositions of the melts



**Figure 4.** The enthalpy change of  $\text{CO}_2$  in  $\text{Li}_2\text{CO}_3$ - $\text{LiCl}$  melts for different compositions of the melts

**Table S3.** Values of equilibrium pressure and solubility of  $\text{CO}_2$  in  $\text{Li}_2\text{CO}_3$ - $\text{LiCl}$  melts

$x_{\text{Li}_2\text{CO}_3}$ /mol %	T/K	Equilibrium Pressure of $\text{CO}_2$ /MPa	Solubility of $\text{CO}_2$ / $\text{g}_{\text{CO}_2}/\text{g}_{\text{melt}}$
10	873	$9.46 \times 10^{-2}$	$3.911 \times 10^{-7}$
	898	$9.82 \times 10^{-2}$	$3.906 \times 10^{-7}$
	923	$1.015 \times 10^{-1}$	$3.902 \times 10^{-7}$
20	873	$1.027 \times 10^{-1}$	$3.934 \times 10^{-7}$
	898	$1.063 \times 10^{-1}$	$3.930 \times 10^{-7}$
	923	$1.102 \times 10^{-1}$	$3.925 \times 10^{-7}$
30	873	$1.016 \times 10^{-1}$	$3.948 \times 10^{-7}$
	898	$1.051 \times 10^{-1}$	$3.946 \times 10^{-7}$
	923	$1.085 \times 10^{-1}$	$3.944 \times 10^{-7}$
40	873	$1.011 \times 10^{-1}$	$3.956 \times 10^{-7}$
	898	$1.047 \times 10^{-1}$	$3.953 \times 10^{-7}$
	923	$1.081 \times 10^{-1}$	$3.951 \times 10^{-7}$
50	873	$1.006 \times 10^{-1}$	$3.965 \times 10^{-7}$
	898	$1.041 \times 10^{-1}$	$3.962 \times 10^{-7}$
	923	$1.073 \times 10^{-1}$	$3.960 \times 10^{-7}$



**Table S4.** Values of  $K_H$  and  $\Delta_{sol}H$  of  $CO_2$  in  $Li_2CO_3$ - $LiCl$  melts

$x_{Li_2CO_3}$ /mol %	T/K	$K_H$ /mol L <sup>-1</sup> MPa	$\Delta H$ /kJ mol <sup>-1</sup>
10	873	0.164	-4.449
	898	0.158	-4.577
	923	0.153	-4.704
20	873	0.152	-4.924
	898	0.147	-5.069
	923	0.142	-5.214
30	873	0.155	-5.055
	898	0.149	-5.209
	923	0.145	-5.362
40	873	0.156	-5.375
	898	0.150	-5.533
	923	0.145	-5.692
50	873	0.157	-5.641
	898	0.151	-5.803
	923	0.147	-5.964

#### 4. Conclusions

The solubility of  $CO_2$  in molten  $Li_2CO_3$ - $LiCl$  with 10–50 mol%  $Li_2CO_3$  content at 873–923 K was measured using a pressure differential method. This also enabled the enthalpy change of solution to be calculated. The following additional conclusions were drawn:

When the temperature decreased and the concentration of  $Li_2CO_3$  increased, the solubility of  $CO_2$  increased, with a maximum solubility of  $3.965 \times 10^{-7}$  g $_{CO_2}$ /g $_{melt}$  at 873 K when the content of  $Li_2CO_3$  was 50 mol%.

$CO_2$  dissolution in the  $Li_2CO_3$ - $LiCl$  melts was an exothermic process. The enthalpy change of  $CO_2$  solution decreased with increasing temperature and  $Li_2CO_3$  content.

#### Conflicts of interest

There are no conflicts of interest to declare.

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## RASTVORLJIVOST CO<sub>2</sub> U RASTOPLJENOM Li<sub>2</sub>CO<sub>3</sub>-LiCl SISTEMU

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### Apstrakt

Rastvorljivost CO<sub>2</sub> u rastopljenom Li<sub>2</sub>CO<sub>3</sub>-LiCl sistemu je izmerena metodom diferencijalnog pritiska i na osnovu toga je izračunata promena entalpije rastvora. Razmatrani su odnosi između rastvorljivosti i entalpije, kao i temperature i sastava rastopa. Rezultati su pokazali da se, kada je temperatura bila između 873 i 923 K, a sadržaj Li<sub>2</sub>CO<sub>3</sub> između 10–50 mol%, rastvorljivost CO<sub>2</sub> povećala sa smanjenjem temperature i/ili povećanjem sadržaja Li<sub>2</sub>CO<sub>3</sub>. Maksimalna rastvorljivost je iznosila  $3,965 \times 10^{-7} \text{ g}_{\text{CO}_2}/\text{g}_{\text{melt}}$  na temperaturi od 873 K kada je sadržaj Li<sub>2</sub>CO<sub>3</sub> iznosio 50 mol%. Rastvor CO<sub>2</sub> je bilo egzotermno. Sa porastom temperature i sadržaja Li<sub>2</sub>CO<sub>3</sub> bila je potrebna veća entalpija za rastvor CO<sub>2</sub>.

**Ključne reči:** MSCC-ET; Li<sub>2</sub>CO<sub>3</sub>-LiCl rastop; Rastvorljivost CO<sub>2</sub>; Metoda diferencijalnog pritiska; Promena entalpije rastvora

