ELSEVIER

Thermochimica Acta 288 (1996) 63–71

thermochimica acta

Thermochemical investigations of the systems useful for lithium battery construction

Part 2. Solution enthalpies of $LiClO_4$ in 1,2–dimethoxyethane + γ-butyrolactone mixtures and of $LiClO_4$, NaI and N,N-dimethylacetamide in 1,2-dimethoxyethane + propylene carbonate mixtures at 298.15 K[1]

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Received 27 December 1995; accepted 12 June 1996

Abstract

Solution enthalpies of $LiClO_4$ in 1,2-dimethoxyethane + γ-butyrolactone (1,2-DME + γBL) mixtures and of $LiClO_4$, NaI and N,N-dimethylacetamide in 1,2-DME + propylene carbonate (PC) mixtures have been measured. The molar enthalpies of solution, $\Delta_{sol}H_m$ of the examined electrolytes as a function of the mixed solvent composition exhibit a distinct minimum within the range of high 1,2-DME content, similar to those observed earlier for $LiBF_4$ and $LiPF_6$ solutions in 1,2-DME + γ-BL mixtures while the dissolution enthalpy of DMA runs monotonously within the same range of the mixed solvent compositions. The presence of the $\Delta_{sol}H_m$ minimum of the salts is explained as a resultant of energetic effects of interactions in the solution, connected with the ionic association of the salt, the 1,2-DME + cation complex formation and changes in a composition of the ionic solvation shells

Keywords: $LiClO_4$; NaI, 1,2-dimethoxyethane mixtures; γ-butyrolactone; propylene carbonate; dissolution enthalpies.

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[1] Part 1: ref. [1].

PII: S0040-6031(96)03044-4

1. Introduction

Recently, the thermochemical investigations of the systems useful for lithium battery construction have been undertaken in our laboratory [1]. As is known, these batteries contain, in most cases a lithium salt with a large complex anion dissolved in a liquid mixture that consist of a low viscosity solvent and a high dielectric solvent. The most frequently used salts are: $LiBF_4$, $LiPF_6$, $LiClO_4$ and $LiAsF_6$. In our previous paper [1] the results of dissolution enthalpies measurements for $LiBF_4$ and $LiPF_6$ in γ-butyrolactone + 1,2-dimethoxyethane mixtures at 298.15 K were reported and a dependence of the measured enthalpies on the mixed solvent composition was discussed. As an extension of these investigations, in this paper we describe a thermochemical studies of lithium perchlorate ($LiClO_4$) solutions in the mixtures of 1,2-dimethoxyethane with γ-butyrolactone and with propylene carbonate. For the sake of comparison the results of the dissolution enthalpy measurements of a simple inorganic salt, sodium iodide (NaI) and a non-ionic substance, that is neither complexed by the 1,2-dimethoxyethane nor H-bonded to the mixed solvent components, namely N,N-dimethylacetamide in selected mixtures of 1,2-DME + PC are also given.

Experimental

Lithium perchlorate, Aldrich p.a. and sodium iodide, Merck p.a. were dried for several days, until constant weight, under reduced pressure at ca. 420 K ($LiClO_4$) or ca. 333 K (NaI). Both salts were next stored under a dry nitrogen.

γ-Butyrolactone (γ-BL), propylene carbonate (PC), N,N-dimethylacetamide (DMA) and 1,2-dimethoxyethane (1,2-DME) all Fluka AG were dried with 0.4 nm molecular sieves and distilled in a water-free atmosphere (γ-BL, PC and DMA under reduced pressure).

The mixed solvents were prepared by weight in a dry box. Their water content was determined by the modified Karl Fischer procedure [2]. In all cases the volume fraction of water in the solvents, before the experiment, was less than 10^{-5}.

Dissolution enthalpies were measured with a non-isothermal-non-adiabatic type calorimeter. The tightly closed glass calorimetric vessel, capacity ca. 100 cm^3 was equipped with a calibration heater, a thermistor and a stirrer-ampoule holder. A thin-walled glass ampoule, capacity ca. 0.5 cm^3, with the sample of salt to be dissolved was attached to the ampoule holder and crushed against the bottom of the calorimetric vessel during the experiment. The 10 KΩ thermistor was connected to a Wheatstone bridge. A temperature change of approximately $3 \cdot 10^{-5}$ K could be detected. The ampoule breaking-heat effect in the reaction vessel was negligible. The use of ampoules filled incompletely made it possible the evaporation of some amount of the solvent into an empty space released after the ampoule breaking. The calculated endothermic effect of the evaporation for the most volatile solvent (1,2-DME) was less than $2 \cdot 10^{-3}$ J and could be neglected in comparison with the measured heat effect which was 5 to 20 J.

The calorimeter was placed in a hermetically closed brass jacket with a capacity of about 1 dm^3 and immersed into a water thermostat. The temperature stability of the thermostat was better than $1 \cdot 10^{-3}$ K. The error in the calorimetric measurements was estimated to be ± 0.5%.

Results and discussion

The measured enthalpies of solution, $\Delta_{sol}H_m$ of $LiClO_4$ in the mixtures of 1,2-dimethoxyethane with γ-butyrolactone and with propylene carbonate are given in Tables 1 and 2 as a function both of the electrolyte concentration and the mixed solvents composition. The analogous data for NaI in 1,2-DME + PC mixtures are given in Table 3. Figure 1 presents the molar enthalpies of solution of $LiClO_4$ at the concentration $m = 0.003$ mol·kg^{-1} (values interpolated from the experimental data) as a function of the mixed solvent composition, $\Delta_{sol}H_m(m = 0.003) = f(x)$ in the mixtures of

Table 1
Molar enthalpy of solution, $\Delta_{sol}H_m$ of $LiClO_4$ in the mixtures of 1,2-dimethoxyethane (1,2-DME) with γ-butyrolactone (γ-BL) at 298.15 K. (m/mol·kg^{-1}, $\Delta_{sol}H_m$/kJ·mol^{-1}).

m	$\Delta_{sol}H_m$	m	$\Delta_{sol}H_m$	m	$\Delta_{sol}H_m$
DME		10 mol% γ-BL		20 mol% γ-BL	
0.00282	−56.80	0.00283	−62.11	0.00289	−64.56
0.00342	−56.70	0.00411	−62.09	0.00343	−64.55
0.00471	−56.63	0.00588	−62.06	0.00508	−64.51
0.00571	−56.52	0.00743	−62.03	0.00679	−64.47
0.00785	−56.33	0.00918	−61.99	0.00883	−64.42
30 mol% γ-BL		40 mol% γ-BL		50 mol% γ-BL	
0.00275	−64.24	0.00296	−63.11	0.00282	−61.41
0.00338	−64.22	0.00365	−63.09	0.00363	−61.39
0.00490	−64.18	0.00507	−63.06	0.00555	−61.34
0.00603	−64.15	0.00666	−63.01	0.00638	−61.32
0.00835	−64.09	0.00874	−62.96	0.00718	−61.29
0.00977	−64.05	0.01103	−62.90	0.01083	−61.20
60 mol% γ-BL		70 mol% γ-BL		80 mol% γ-BL	
0.00283	−59.39	0.00290	−56.62	0.00288	−53.47
0.00325	−59.38	0.00393	−56.60	0.00375	−53.45
0.00507	−59.33	0.00542	−56.56	0.00531	−53.42
0.00684	−59.29	0.00684	−56.53	0.00707	−53.38
0.00803	−59.26	0.00880	−56.48	0.00883	−53.35
0.01041	−59.20			0.01012	−53.32
90 mol% γ-BL		100 mol% γ-BL			
0.00297	−50.58	0.00279	−47.12		
0.00380	−50.57	0.00312	−47.12		
0.00475	−50.55	0.00425	−46.54		
0.00667	−50.52	0.00541	−46.47		
0.00843	−50.50	0.00776	−46.33		
0.00996	−50.47	0.01031	−46.20		

Table 2
Molar enthalpy of solution, $\Delta_{sol}H_m$ of $LiClO_4$ in the mixtures of 1,2-dimethoxyethane (1,2-DME) with propylene carbonate (PC) at 298.15 K. (m/mol·kg^{-1}, $\Delta_{sol}H_m$/kJ·mol^{-1}).

m	$\Delta_{sol}H_m$	m	$\Delta_{sol}H_m$	m	$\Delta_{sol}H_m$
10 mol% PC		20 mol% PC		30 mol% PC	
0.00282	−64.75	0.00293	−67.03	0.00266	−66.19
0.00327	−64.70	0.00308	−66.99	0.00312	−66.13
0.00433	−64.65	0.00473	−66.87	0.00445	−65.93
0.00609	−64.53	0.00654	−66.66	0.00688	−65.66
0.00786	−64.44	0.00718	−66.64	0.00813	−65.48
0.00940	−64.37	0.00927	−66.49	0.00965	−65.38
40 mol% PC		50 mol% PC		60 mol% PC	
0.00287	−64.99	0.00285	−62.35	0.00268	−59.27
0.00363	−64.84	0.00408	−62.28	0.00346	−59.21
0.00555	−64.71	0.00483	−62.22	0.00507	−59.15
0.00679	−64.62	0.00705	−62.11	0.00668	−59.07
0.00818	−64.48	0.00824	−62.06	0.00745	−59.05
0.00972	−64.41	0.00977	−62.00	0.00973	−58.98
70 mol% PC		80 mol% PC		90 mol% PC	
0.00281	−54.84	0.00264	−50.64	0.00287	−45.77
0.00345	−54.81	0.00307	−50.62	0.00341	−45.76
0.00455	−54.78	0.00444	−50.60	0.00423	−45.75
0.00637	−54.74	0.00640	−50.58	0.00662	−45.73
0.00810	−54.70	0.00812	−50.55	0.00845	−45.70
0.01004	−54.65	0.00965	−50.52	0.01037	−45.68
100 mol% PC					
0.00272	−39.69				
0.00330	−39.67				
0.00461	−39.66				
0.00582	−39.64				
0.00775	−39.64				
0.00918	−39.62				

1,2-DME with γ-BL, and Fig. 2 gives the analogous data for $LiClO_4$ and NaI in the mixtures of 1,2-DME with PC. For the sake of comparison the $\Delta_{sol}H_m(m = 0.003) = f(x)$ curves illustrating investigated earlier dissolution enthalpies of $LiBF_4$ and $LiPF_6$ in 1,2-DME + γ-BL mixtures are also shown in Fig. 1. As is seen from these data the $\Delta_{sol}H_m(m = 0.003) = f(x)$ plots for all three systems examined here have very similar shape with a distinct minimum within the high 1,2-DME content in the mixed solvent. In addition, these curves are similar to those for mentioned above $LiBF_4$ and $LiPF_6$ solutions, although their $\Delta_{sol}H_m$ values are different [1]. Therefore, the course of the examined function seems to be not too sensitive to the change of the complex inorganic anion of the lithium salt. The replacement of the γ-butyrolactone by the propylene carbonate in the mixture with 1,2-dimethoxyethane also does not alter

Table 3
Molar enthalpy of solution, $\Delta_{sol}H_m$ of NaI in the mixtures of 1,2-dimethoxyethane (1,2-DME) with propylene carbonate (PC) at 298.15 K. (m/mol·kg^{-1}, $\Delta_{sol}H_m$/kJ·mol^{-1}).

m	$\Delta_{sol}H_m$	m	$\Delta_{sol}H_m$	m	$\Delta_{sol}H_m$
DME		10 mol% PC		20 mol% PC	
0.00269	−41.66	0.00275	−47.40	0.00282	−50.44
0.00345	−41.61	0.00387	−47.29	0.00375	−50.37
0.00471	−41.55	0.00477	−47.23	0.00465	−50.25
0.00588	−41.51	0.00658	−47.18	0.00613	−50.12
0.00733	−41.46	0.00809	−47.04	0.00708	−50.05
0.00925	−41.40	0.00935	−47.00	0.00887	−49.92
30 mol% PC		40 mol% PC		50 mol% PC	
0.00278	−49.63	0.00294	−47.77	0.00288	−46.10
0.00322	−49.54	0.00385	−47.66	0.00385	−46.04
0.00485	−49.31	0.00427	−47.65	0.00417	−46.03
0.00638	−49.17	0.00532	−47.53	0.00603	−45.90
0.00745	−49.04	0.00672	−47.40	0.00704	−45.87
0.00913	−48.85	0.00806	−47.31	0.00948	−45.76
60 mol% PC		70 mol% Pc		80 mol% PC	
0.00275	−44.05	0.00298	−41.89	0.00294	−39.10
0.00331	−44.02	0.00316	−41.88	0.00356	−39.08
0.00507	−43.93	0.00465	−41.83	0.00498	−39.06
0.00586	−43.91	0.00606	−41.79	0.00573	−39.05
0.00725	−43.85	0.00821	−41.75	0.00723	−39.02
0.00976	−43.77	0.00989	−41.71	0.00886	−39.00
90 mol% PC		95 mol% PC		100 mol% PC	
0.00277	−35.02	0.00278	−31.05	0.00285	−20.98
0.00365	−35.01	0.00356	−31.04	0.00374	−20.97
0.00481	−34.99	0.00512	−31.02	0.00569	−20.95
0.00634	−34.97	0.00689	−31.00	0.00788	−20.93
0.00808	−34.95	0.00775	−31.00	0.00835	−20.93
0.00954	−34.94	0.00915	−30.98	0.00963	−20.92

significantly the shape of the $LiClO_4$ dissolution enthalpy function. Both, propylene carbonate and γ-butyrolactone are aprotic solvents and have relatively high electric permittivities (PC: $\varepsilon = 64.92$ [3], γ-BL: $\varepsilon = 41.77$ [3]). Density, viscosity and electric permittivity of the mixtures of 1,2-DME with PC and with γ-BL vary in similar, monotonous way along with the change of the mixture composition [1,3–8]. Moreover, dependences of electric conductance and ionic association constant on the mixed solvent composition for $LiClO_4$ solutions in both investigated mixtures [3,7] and for $LiBF_4$ and $LiPF_6$ solutions in 1,2-DME + γ-BL mixtures run in like manner [4]. It can be therefore concluded, that similarly as in the examined earlier systems [1], the observed course of the lithium salt molar solution enthalpy as a function of the mixed solvent composition is a resultant of the three essential effects. They are: 1) the decrease

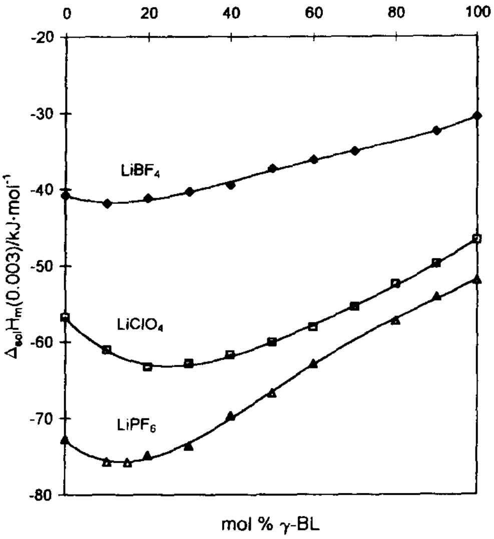

Fig. 1. Molar enthalpies of solution, $\Delta_{sol}H_m$ for lithium salts in the mixtures of 1,2-dimethoxyethane with γ-butyrolactone at 298.15 K. The salt concentration, m = 0.003 mol·kg^{-1} of solvent.

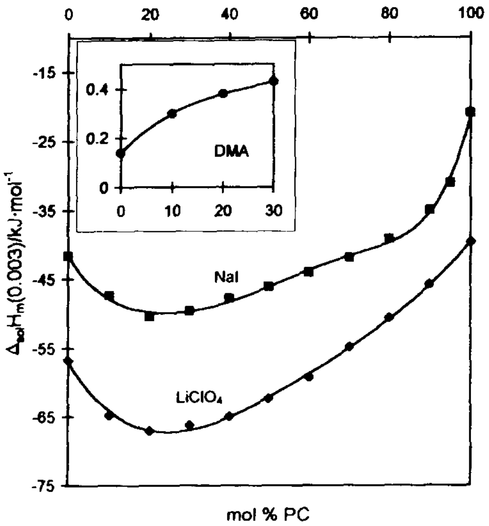

Fig. 2. Molar enthalpies of solution, $\Delta_{sol}H_m$ (m = 0.003 mol·kg^{-1}) for LiClO$_4$, NaI and N,N-dimethylacetamide in the mixtures of 1,2-dimethoxyethane with propylene carbonate at 298.15 K.

of the ionic association of the salt along with the decrease of the 1,2-DME content in the mixed solvent, 2) 1,2-dimethoxyethane-lithium cation complex formation [5,9–13], and 3) changes in the composition of the ionic solvation shells. Since NaI in the solvents with the low electric permittivity forms ionic associates, and sodium cation can be also complexed by the 1,2-DME molecule [5,9–13] the reasons of the presence of the $\Delta_{sol}H_m$ minimum for the NaI in 1,2-DME + PC mixtures are possibly the same as for the discussed lithium salts.

The deepest minimum of the $\Delta_{sol}H_m$ ($m = 0.003$) = f(x) function is observed for $LiClO_4$ solution in 1,2-dimethoxyethane + propylene carbonate mixtures. It is probably due to the fact that the decrease of the ionic association, caused by the introduction of the polar cosolvent into 1,2-dimethoxyethane is sharpest in this system. The ion association constant, K_A for $LiClO_4$ in the mixture of 1,2-DME + PC containing 10 mol% of PC is as much as 732 times smaller than that in pure, 1,2-DME, while in 1,2-DME + γ-BL mixture the analogous K_A(DME)/K_A(10% γ-BL) ratios for the investigated lithium salts are equal: 127 for $LiClO_4$, 100 for $LiBF_4$ and only 11 for $LiAsF_6$ (see Table 4 and ref. [1]). Since the ion association enthalpies for the lithium salts in these solutions are positive, as calculated from the appropriate literature data [7,8,14], the dissociation of the ion pairs gives the exothermic impact into the $\Delta_{sol}H_m$ that is the greatest for $LiClO_4$ solutions in the 1,2-DME + PC mixtures.

The dissolution enthalpies of the both investigated here salts, particularly of the NaI in the 1,2-dimethoxyethane + propylene carbonate mixtures decrease rapidly when small amounts of 1,2-DME are added to PC (Fig. 2). It is known that the ability of PC to interact with alkali ions is relatively poor (the Gutman donor number, DN = 15.1) while 1,2-DME has stronger electron-donor properites (DN = 23.9) and, as was mentioned earlier it can form complexes with small alkali cations [5,9–13]. Moreover the enthalpic effect of the electrolyte solvation in 1,2-DME is more exothermic than that in PC (Fig. 2). Therefore, the observed course of the $\Delta_{sol}H_m$ ($m = 0.003$) = f(x) function within the low 1,2-DME content suggests that the addition of even small amount of the 1,2-DME to PC causes that a significant number of the PC molecules is

Table 4
Relative permittivity, ε and equilibrium constants of the ion pair formation, K_A for $LiC1O_4$ in the mixtures of 1,2-dimethoxyethane (1,2-DME) with γ-butyrolactone (γ-BL) and propylene carbonate (PC) at 298.15 K.

1,2-DME + γ-BL			1,2-DME + PC		
[γ-BL] mol%	ε	K_a	[PC] mol%	ε	K_a
0.0	7.20 [3]	$3.5 \cdot 10^6$ [7,8]	0.0	7.20 [3]	$3.5 \cdot 10^6$ [7,8]
10.3	10.20 [7]	27540*	9.7	11.73 [3]	4781 [3]
13.1	10.93 [3]	19727 [3]	14.3	14.48 [3]	1206 [3]
20.5	12.80 [7]	2500*	20.0	16.60*	447*
25.3	14.67 [3]	1237 [3]	23.3	18.89 [3]	269 [3]
30.2	16.10 [7]	700*	30.0	21.70*	90*
36.6	18.47 [3]	299 [3]	40.0	27.50*	30*
50.2	22.80 [7]	120*	54.7	36.29 [3]	11*
70.6	29.80 [7]	32*	70.0	45.00*	6*
89.7	36.80 [7]		90.0	58.30*	
100.	41.77 [3]	4.4 [3]	100.	64.9 [3]	1.3 [3]

*interpolated values, assuming linear dependences of the log K_A *vs.* $1/\varepsilon$ [3,7] and of ε *vs.* volumetric ratio of the mixture composition [3–5].

removed from the cationic solvation shells and they are replaced by the 1,2-DME ones. The above conclusion can be confirmed by the results of investigations of lithium salt solutions in the mixtures of propylene carbonate with tetraethylene glycol dimethyl ether (TEGDME) [15]. As was found from spectroscopic (NMR, IR, VIS) and conductometric studies only few if any PC molecules are complexed to the Li^+ ions in the examined mixture containing 70 mol%, of PC. Even if the TEGDME content amounts to ca. 6 mol% only, most of the cations remain bond to the ether molecules [15].

In order to eliminate the solute-solute interactions it is necessary to extrapolate the measured enthalpies of solution as a function of the salt concentration to infinitely diluted solution (m = 0) and determine the standard solution enthalpies, $\Delta_{sol}H^{\infty}$. Different extrapolation methods are proposed to this goal, most of them are based on the first or the second approximation of the Debye-Hückel theory. These relatively simple methods can be used only for the solutions in which the ion association process is negligible small, i.e. when the ion association constant, K_A is less than 10 [16], or the solvent electric permittivity is relatively high ($\varepsilon > 30$). From the systems investigated here it concerns only the solutions in pure propylene carbonate, pure γ-butyrolactone and the mixtures containing more than 50 mol% PC (Table 4).

The standard solution enthalpies in propylene carbonate for $LiClO_4$, $\Delta_{sol}H^{\infty} = -39.75\,kJ{\cdot}mol^{-1}$, and for NaI in PC $\Delta_{sol}H^{\infty} = -21.05\,kJ{\cdot}mol^{-1}$ determined from our data by means of the method proposed by Criss and Cobble [17] are in a good agreement with the appropriate literature data. They are: $\Delta_{sol}H^{\infty} = -39.79\,kJ{\cdot}mol^{-1}$ [18] for $LiClO_4$ and $-21.09\,kJ{\cdot}mol^{-1}$ [18] or $-21.51\,kJ{\cdot}mol^{-1}$ [19] for NaI. The $\Delta_{sol}H^{\infty}$ values for $LiClO_4$ and NaI in PC + 1,2-DME mixtures within the high PC content range, determined by using the same method are presented in Table 5. For other investigated mixtures an extrapolation method taking into account the ionic association process has to be employed. One of them is based on an expression proposed by Barthel and co-workers [6]:

$$\Delta L_{\phi} = \alpha L_{\phi}(\mathrm{FI}) - \alpha' L_{\phi}(\mathrm{FI}) + (\alpha' - \alpha)\Delta H_A \tag{1}$$

Where $L_{\phi}(\mathrm{FI})$ denotes the relative apparent molal heat content of a solution with "free" ions, which can be calculated from Debye-Hückel theory [6]. ΔH_A is the enthalpy of

Table 5
Standard dissolution enthalpies, $\Delta_{sol}H^{\infty}$ of $LiClO_4$ and NaI in the 1,2-dimethoxyethane (1,2-DME) + propylene carbonate (PC) mixtures at 298.15 K.

mol %	$\Delta_{sol}H^{\infty}/kJ{\cdot}mol^{-1}$	
PC	$LiClO_4$	NaI
50	−62.77	−46.53
60	−59.58	−44.37
70	−55.04	−42.11
80	−50.76	−39.23
90	−45.87	−35.12
95		−31.13
100	−39.75	−21.05

ionic association and α and α' are the dissociation degrees at the molality of m and m' respectively. However, such procedure requires the determination of the L_ϕ values with the accuracy accesible only in very precise dilution experiment but not in dissolution enthalpy measurements. Therefore, the determination of the standard dissolution enthalpies from the data obtained in this work was not possible within the range of high 1,2-DME content.

If the possibility of the ionic association and the solute-solute complex formation (e.g. 1,2-DME + cation complex) processes were eliminated then, in the light of the discussion presented in our previous paper [1] the minimum of the $\Delta_{sol}H^\infty$ should not be observed within the range of high 1,2-DME content. In order to check this supposition, the dissolution enthalpies of N,N-dimethylacetamide, a non-ionic substance, that is neither complexed by the 1,2-DME molecules nor hydrogen bonded to the mixed solvent components were measured within the diether-rich region. As it was expected, the obtained $\Delta_{sol}H$ (DMA) = f(x) function exhibits a monotonous course (Fig. 2). This observation seems to confirm a rightness of the proposed model of interactions in the examined systems.

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