Journal of Thermal Analysis and Calorimetry, Vol. 74 (2003) 843–852

THERMAL BEHAVIOR OF VARIOUS SULFONE-BASED DIIMID-DIACIDS SOLVATED WITH POLAR ORGANIC SOLVENTS

C.-P. Yang [1]*, *E. M. Woo* [2] *and G.-L. Jou* [1]

[1]Department of Chemical Engineering, Tatung University, 40 Chungshan North Rd., 3rd Sec., Taipei 104, Taiwan
[2]Department of Chemical Engineering, National Cheng Kung University, Tainan 701, Taiwan

(Received February 24, 2003; in revised form April 3, 2003)

Abstract

Thermal analysis techniques were performed to reveal ‘crystalline solvate’ behavior between organic compounds and polar solvents. Diimide-dicarboxylic acid (DIDA) was formed by reacting 3,3’-diaminodiphenylsulfone (3,3’-DPS) or 4,4’-diaminodiphenylsulfone (4,4’-DPS) with trimellitic anhydride (TMA) in some polar solvents (PSv). The products could crystallize upon cooling in a polar solvent media to form a solvate containing a finite quantity of solvents, leading to what can be termed as ‘crystalline solvates’ (CS). This study has demonstrated that sampling techniques in TG and DSC must be kept to be as similar as possible, which is a critical point in practices of thermal analysis techniques. DSC analysis revealed that there are two endothermic peaks in the CS, with the lower one being the de-solvate temperature of CS (T_d) at which the solvated solvent molecules were removed, and the higher peak being the melting point of the de-solvated DIDA (T_m). T_d was found to vary with the types of polar solvents and structures of DIDA. The TG result indicated that most of the sulfone-based DIDA-CS contained 2 moles of solvent per mole of solvate. X-ray analysis revealed that different crystalline structures were found for DIDA-CS solvated with different solvent molecules, but all de-solvated DIDA possessed the same crystal unit.

Keywords: 2,2-bis(3-trimellitimidophenyl)sulfone, 2,2- bis(4-trimellitimidophenyl)sulfone, crystalline solvate, diimide-diacid, DSC, polar solvents, TG

Introduction

Many inorganic salts can crystallize from aqueous solutions, and the salt crystals always contain a fixed number of water molecules in their crystal units. The water-salt crystals are called crystalline hydrate [1]. Examples are seen in $CuSO_4·5H_2O$, $Na_2SO_4·10H_2O$ and $CaSO_4·2H_2O$. Some of these dehydrating salts possess excellent hydrating capability that they are used as desiccants. In addition, some organic-inorganic complexes are also known to crystallize with a certain number of crystalline water molecules, such as $Lu(NO_3)_3·N_4(CH_2)_6$, which contains one mole of H_2O [2].

---

* Author for correspondence: E-mail: cpyang@ttu.edu.tw.

1388–6150/2003/ $ 20.00

Akadémiai Kiadó, Budapest
Kluwer Academic Publishers, Dordrecht

Furthermore, some organo-metallic complexes can crystallize from solutions of organic solvents, and these complexes may contain a fixed number of solvents molecules in their crystal units [3–6]. These solvents are called ‘crystalline solvents’, and the crystals of complexes with solvents are called crystalline solvates (CS). Examples are seen in $Mg(OCH_3)_2$ containing CH_3OH [3], $Co_3O(CH_3COO)_6(C_5H_5N)_3$ containing $CHCl_3$ and H_2O [4], $Pd(Hdpf\text{-}P)_2Cl_2$ containing AcOH [5] and $Ni(dipdtc)(PPh_3)_2{\cdot}ClO_4$ containing $CHCl_3$ [6]. Recently, C_{60} has also been found to form crystalline solvates with solvents such as: CCl_4 [7], 1,2-bromobenzene [8], benzene [9] and 1,3,5-trimethylbenzene [10].

There have been some interesting reports demonstrating unique thermal analysis in evaluating thermal behavior of organo-metal salts or organo-ligand complexes [11–15]. But it has been rarely reported that solid organic compounds in solvents would be packed into crystals in the form of crystalline solvates. A recent investigation has reported that diamantine dicarboxylic acid [16] may crystallize from cooled solutions in DMF and X-ray analysis revealed that the crystals contain two molecules of DMF for each crystallized molecule. We recently discovered that the imide-based dicarboxylic acid exhibited a behavior of solvating with polar solvent molecules. As aromatic diamines were reacted with TMA in polar solvents to form DIDAs [17–20], some of the DIDAs crystallized with a phenomenon of CS. This study dealt with synthesis of 3,3’-diaminodiphenyl sulfone or 4,4’-diaminodiphenyl sulfone reacted with TMA to product DIDA 2,2-bis(3-trimellitimidophenyl)sulfone or 2,2-bis(4-trimellitimidophenyl)sulfone, respectively, in several polar solvents. Crystalline solvate products with a fixed number of solvent molecules were found to form in the polar solvent-product solutions. Confirmation of such crystalline solvates was characterized using thermal analysis techniques. A special sampling method for ensuring comparability between different thermal analysis techniques is also discussed.

Experimental

Materials

Trimellitic anhydride (TMA, from Wako), 3,3’-diaminodiphenylsulfone (3,3’-DPS, from ACROS) (*m.p.* 167°C), and 4,4’-diaminodiphenylsulfone (4,4’-DPS, from ACROS) (*m.p.* 175–177°C) were used in as-received states. N,N-dimethylformamide (DMF, from Fluka), N,N-dimethylacetamide (DMAc, from TCI), N-methyl-2-pyrrolidone (NMP, from Fluka), 1,3-dimethyl-2-imidazolidone (DMI, from Fluka), and dimethylsulfoxide (DMSO, from Fluka) were also used as received.

*Synthesis of diimide-dicarboxylic acid (DIDA, **II**)*

Synthesis of 2,2-bis(3-trimellitimidophenyl)sulfone (DIDA-A, $\mathbf{II_A}$) was proceeded as following. 4.97 g (20 mmol) of 3,3’-DPS was dissolved in 30 mL of NMP, and 7.60 g (40 mmol) of TMA was added, to be followed with stirring. Then, 15 mL of toluene was added, and the mixture was heated with reflux for 4 h until about 0.72 mL of water was distilled off azeotropically. After crystallization, methanol was used for washing the products. Then, the products were dried at 100°C in vacuum. The mass of crystals (II'_A-NMP) thus obtained weighed 12.58 g. The de-solvated products $\mathbf{II_A}$ was obtained by heating II'_A -NMP at 200°C for 2 h in vacuum to give 11.84 g of white powder (yield 99.5%, *m.p.* 358–359°C). The DSC and TG analysis spectrum and analysis data are shown in Fig. 1 and Table 1.

Table 1 Thermal properties of II'_A[a]

CS[b]	T_d[c]/°C	T_m[d]/°C	W[e]/%	m[f]	Note	
					PSv[g]	
					b.p./°C	M_w
$\mathbf{II'_A}$-DMF	189.0	358.6	19.9	2	151	73
$\mathbf{II'_A}$-DMAc	152.6	355.5	19.1	2	163	87
$\mathbf{II'_A}$-NMP	170.3	361.0	24.8	2	202	99
$\mathbf{II'_A}$-DMSO	190.6	359.5	21.0	2	189	78
$\mathbf{II'_A}$-DMI	196.6	358.8	19.6	1	226	114
$\mathbf{II_A^h}$		358.8				

[a]$\mathbf{II'_A}$– DIDA-3,3'-DPS-CS; [b]CS – crystalline solvate; [c]T_d – decomposition temperature of CS; [d]T_m – Melting point; [e]ΔW – mass loss; [f]m – mole of PSv, $m=$ **(596×** W)/(M_w of PSv)×(1–ΔW); [g]PSv – polar solvent; [h]$\mathbf{II_A}$ – DIDA-3,3'-DPS (M_w: 596)

Synthesis of 2,2-bis(4-trimellitimidophenyl)sulfone (DIDA-B, $\mathbf{II_B}$) was similarly carried out. 4.97 g (20 mmol) of 4,4'-DPS was dissolved in 30 mL of NMP, and 7.60 g (40 mmol) of TMA was added, stirred, and dissolved. Then, 15 mL of toluene was added, and the mixture was heated with reflux for 4 h until about 0.72 mL of water and toluene was distilled off. After the products were crystallized and precipitated in the solutions, methanol was used for washing. The products were dried at 100°C in vacuum, and the mass of crystal ($\mathbf{II'_B}$-NMP) thus obtained weighed 12.58 g. The de-solvated products $\mathbf{II_B}$ was obtained by heating $\mathbf{II'_B}$-NMP at 200°C for 2 h in vac-

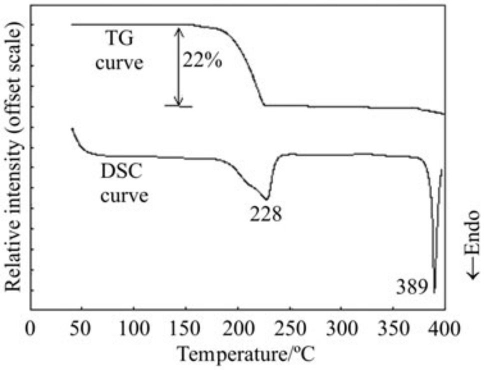


Fig. 1 TG and DSC curves of $\mathbf{II'_B}$-NMP at a heating rate of 15°C min^{-1} under nitrogen

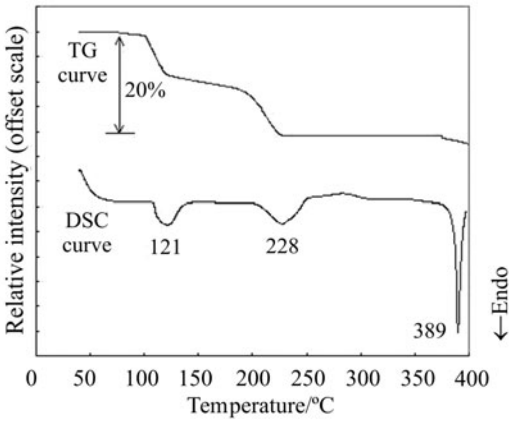


Fig. 2 TG and DSC curves of $\mathbf{II'_B}$-DMF at a heating rate of 15°C min^{-1} under nitrogen

uum to give 11.84 g of white powder (yield 99.5%, T_m 388–389°C). The DSC and TG traces are shown in Fig. 2 and the numerical data are shown in Table 2.

Table 2 Thermal properties of $\mathbf{II'_B}$[a]

CS^b	T_d^c/°C	T_m^d/°C	W^e/%	m^f	Note	
					PSv^g	
					b.p./°C	M_w
$\mathbf{II'_B}$-DMF	121 228	389.0	10 10	2	151	73
$\mathbf{II'_B}$-DMAc	189.1	389.1	19.2	2	163	87
$\mathbf{II'_B}$-NMP	228.0	388.7	22.0	2	202	99
$\mathbf{II'_B}$-DMSO	228.4	381.7	11.8	1	189	78
$\mathbf{II_B^h}$		389.1				

[a] $\mathbf{II'_B}$– DIDA-4,4'-DPS-CS; [b] CS – crystalline solvate; [c] T_d – decomposition temperature of CS; [d] T_m – melting point; [e] ΔW – mass loss; [f] m – mole of PSv, $m=(596\times\Delta W)/(M_w \text{ of PSv})\times(1-\Delta W)$; [g] PSv – polar solvent; [h] $\mathbf{II_B}$– DIDA-4,4'-DPS (M_w: 596)

Preparation of crystalline solvates (DIDA-CS, ***II'****)*

Several polar solvents were used, such as N-methyl-2-pyrrolidinone (NMP), N,N-dimethylforamide (DMF), N,N-dimethylacetamide (DMAc), 1,3-dimethyl-2-imidazolidinone (DMI), or dimethylsulfoxide (DMSO), for forming possible crystalline solvates with the products. After high-temperature drying of **II'**, de-solvated DIDA (**II**) was added into suitable polar solvents (e. g. NMP, DMF, DMAc, DMI, and DMSO), and heated with stirring until dissolving. The mixture was left to cooling to induce crystallization. After crystallization, it was washed thoroughly several times with methanol. The purified products collected by filtration were dried at 80°C for several hours in vacuum and appeared as mass of white powder. These crystalline solvates were designated as **II'**-NMP, **II'**-DMF, **II'**-DMAc, **II'**-DMI and **II'**-DMSO, respectively. The thermal-decomposing temperature (T_d), melting point (T_m), and mass loss (ΔW) of those crystalline solvates are listed in Table 1 and Table 2.

Measurements and apparatus

Wide-angle X-ray measurements were performed with powder specimens on a SIEMENS X-ray diffractometer with Ni-filtered CuK$_\alpha$ radiation (40 kV, 15 mA) at a scanning rate of 3° min^{-1}.

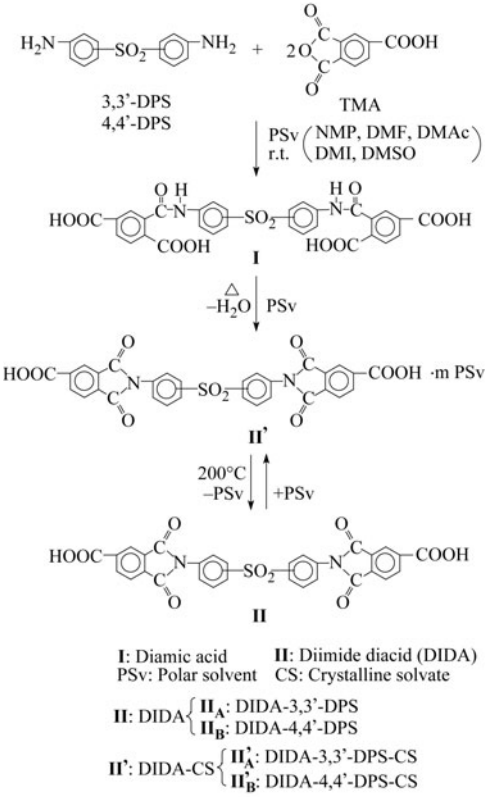
Two thermal analysis techniques were used for studying the de-solvating behavior. Differential scanning calorimetry (DSC) was measured on TA Instrument DSC 2010, in flowing nitrogen 40 cm^3 min^{-1} at a heating rate of 15°C min^{-1}. Thermogravimetery analysis (TG) was conducted with a TA Instrument TGA-2050, where experiments were carried out on 10±2 mg samples in flowing nitrogen of 90 cm^3 min^{-1} at a heating rate of 15°C min^{-1}. A critical point must be noted here, which is especially important as the transition temperatures as revealed by DSC and TG characterization are to be directly compared. Preliminary thermal analysis investigation revealed that substantial differences between TG and DSC results might be caused by different ways of sample loading and/or packing in the TG and DSC characterizations. In this study, a special sampling method the TG and DSC analysis was used to ensure that the thermal events leading to signals in these two techniques were kept to be as similar as possible. To ensure that the sampling was as identical as possible between DSC and TG characterizations, samples for the TG characterization were first loaded and sealed into aluminum pans, which were punctured with small holes for vapor de-solvating. The aluminum pan-sealed samples were then placed onto the platinum tray for TG characterization.

Results and discussion

Diimide-diacid (DIDA) and its crystalline solvate (DIDA-CS)

DIDA and its derivatives were first produced by the ring-opening addition of 1 mole 3,3'-DPS or 4,4'-DPS and 2 mole TMA to form diamide-tetracid **I** in polar solvents such as NMP at room temperature, followed by cyclodehydration to the diimide-dicarboxylic acid **II** by means of toluene-water azeotropic distillation. After distillation of toluene, crystals of the **II**' series were obtained. The crystalline products were washed with methanol, and then vacuum-dried (at 100°C). The white powder was termed as '**II'**-NMP'. DSC and TG characterization revealed that these crystalline powders contained a definite quantity of solvent, indicating that it is a form of crystalline solvate and was termed as 'DIDA-CS-NMP'.

If the NMP solvent was replaced with DMAc, and similar procedures were used for producing DIDA, crystalline solvates were obtained and were termed as



Scheme 1 Formation of DIDA (**II**) and DIDA-CS (**II'**)

DIDA-CS-DMAc (abbr.: **II'**-DMAc). If the **II'** series crystalline products were vacumed-dried at 200°C for 1 h, a white powder was obtained, which was proven by the DSC result that it was de-solvated DIDA. The de-solvated series was termed as '**II**-series'. Reversibility of crystalline solvates was observed in DIDA-CS (**II'**) and DIDA (**II**). **II'** was treated at high temperatures to produce solvent-free **II**. If the solvent-free **II**-series was added into polar solvents (such as NMP, DMF, DMAc, DMI, or DMSO), dissolved with heating, and left to cool to induce re-crystallization. Crystalline solvate, DIDA-CS, was found to form in the solutions. Scheme 1 shows the routes of reversibility.

Thermal properties of DIDA-CS

The quantities of solvated solvents in DIDA-3,3'-DPS-CS ($\mathbf{II}'_{\mathbf{A}}$) and DIDA-4,4'-DPS-CS ($\mathbf{II}'_{\mathbf{B}}$) could be characterized by DSC and TG. Figure 1 shows the DSC and TG results for the NMP-solvated compounds upon heating scans (both at 15°C min^{-1}). On the DSC traces for the DIDA-CS samples, there are two endothermic peaks. The higher one is T_{m} for de-solvated DIDA, while the lower one near 200°C was determined to be the de-solvating temperature for DIDA-CS, at which solvent molecules were deabsorbed from the DIDA molecules. This lower peak is termed as T_d. TG was used for measuring the mass loss at this temperature (ΔW). From the quantitative ΔW, one may estimate the number of solvent molecules in each of the solvated DIDA molecules. One may suspect that the T_d is quite close to the boiling temperature of NMP. But the samples were dried at 100°C under vacuum for sufficient time, and any residual NMP should have been removed. We thus ruled out the possibility of T_d being attributed to evaporation of NMP. Rather, it may be that the observed endothermic peaks at 200°C is the de-solvating temperature of crystalline solvates of $\mathbf{II}'_\mathbf{B}$-NMP. As mentioned earlier in the Experimental section, it is critical that the way of sampling must be as identical as possible between TG and DSC characterizations. The figure shows that the de-solvating temperatures as revealed by the DSC and TG results are completely consistent.

In additions, Tables 1 and 2 show the numerical results of thermal analysis for the crystalline solvates of the DIDA $\mathbf{II}_\mathbf{A}$ and $\mathbf{II}_\mathbf{B}$ series in polar solvents of DMF, DMAc, NMP, and DMSO. The T_m values of $\mathbf{II}_\mathbf{A}$ and $\mathbf{II}_\mathbf{B}$ are 358.8 and 389.1°C, respectively. The T_d of $\mathbf{II}'_\mathbf{A}$-NMP is 170°C, while the T_d of $\mathbf{II}'_\mathbf{B}$-NMP is 228°C. The mass losses of these two series are 24.8 and 22%, respectively. The estimated quantities are 2 moles NMP for each mole of DIDA in the solvated compound. Table 1 shows that the T_d of 189.8°C for the crystalline solvate (CS) of $\mathbf{II}_\mathbf{A}$ and DMF, e. g. $\mathbf{II}'_\mathbf{A}$-DMF, is much higher than the boiling point of DMF (151–156°C). The second endothermic peak in DSC traces (358.6°C) is the melting point of the de-solvated $\mathbf{II}_\mathbf{A}$. The mass loss at T_d was found to be 20%, which is translated to 2 moles of DMF for each mole of $\mathbf{II}_\mathbf{A}$ solvated. If the solvent was replaced with DMAc, a different CS was formed, which is $\mathbf{II}'_\mathbf{A}$-DMAc. It shows a T_d of 152.6°C, which is lower than that for $\mathbf{II}'_\mathbf{A}$-DMF. This T_d is also lower than the boiling point of DMAc (163–166°C). Polar solvent such as DMSO could also form crystalline solvate with $\mathbf{II}_\mathbf{A}$, which is labeled as $\mathbf{II}'_\mathbf{A}$-DMSO. The de-solvating temperature (T_d 190.6°C) is higher than the boiling point of DMSO (189°C). In addition, $\mathbf{II}_\mathbf{A}$ and DMI could also form crystalline solvates, labeled as $\mathbf{II}'_\mathbf{A}$-DMI. Its T_d is 196.6°C, and the CS contained 1 mole of DMI for each mole of solvated compound. Table 2 lists the numerical results for the $\mathbf{II}'_\mathbf{B}$, which was formed by reacting 4,4'-DPS with TMA and polar solvent. The T_d for the $\mathbf{II}'_\mathbf{B}$-DMAc is 189.1°C, which is higher than the boiling point of DMAc. In comparison with $\mathbf{II}'_\mathbf{A}$-DMAc, $\mathbf{II}'_\mathbf{B}$-DMAc is more stable. $\mathbf{II}_\mathbf{B}$ could also form CS with DMSO, and the T_d (228.4°C) is much higher than the boiling point of DMSO. TG results revealed that $\mathbf{II}'_\mathbf{B}$-DMSO contained only 1 mole of DMSO for each mole of solvate.

An occasional exception was noted. Figure 2 shows the DSC and TG curves for the crystalline solvate of $\mathbf{II}_\mathbf{B}$ and DMF ($\mathbf{II}'_\mathbf{B}$-DMF). In this figure, the DSC traces show 3 endothermic peaks. The highest one at 389°C is the melting point of the de-solvated $\mathbf{II}_\mathbf{B}$, while the two lower peaks are both associated with the de-solvating temperatures at which the solvated solvent molecules were driven from the crystals. The first T_d for $\mathbf{II}'_\mathbf{B}$-DMF is about 121°C, with a mass loss of 10%, to be followed by the second peak of 10% mass loss at 228°C. From the TG mass losses, the solvated solvent was estimated to be just 2 moles of solvent per mole of solvate.

Discussion on sampling techniques for DSC and TG

For escape of volatiles during heat scans, DSC samples (5–10 mg) are usually loaded in aluminum DSC pans whose top cover are punctured with small holes for gas evaporating/de-solvating. By contrast, typically, TG samples are directly loaded (with no pan-sealing) in an open platinum tray. Assuming that heat transfer and temperature schemes are identical between DSC and TG, the sealed DSC pans may still make it more difficult for the de-solvating species to escape from the DSC pans than from the open (unsealed) platinum tray (TG). As a result, differences in DSC and TG may yield. Therefore, in this study, sampling techniques in TG and DSC were kept to be as similar as possible. The DSC samples pans were prepared by loading the samples into DSC aluminum pans and sealed, with the top pan cover being punctured with a small hole for breathing. But for TG, samples were not loaded directly into the platinum tray. To ensure that the sampling was as identical as possible between DSC and TG characterizations, samples for the TG characterization were first loaded and sealed into aluminum pans, which were punctured with small holes for vapor de-solvating. The pan-sealed samples were then placed onto the platinum tray for TG characterization. In this way, de-solvating of the samples would similarly go through holes on the aluminum pan during the heating scans for both DSC and TG experiments. Using such techniques, the peak temperatures in DSC traces and transition temperatures in TG curves were found to be more reasonable and comparable.

Crystal structures of DIDA-CS

X-ray crystallography was performed on the crystalline solvates of the $\mathbf{II'_A}$ and $\mathbf{II'_B}$ series and their de-solvated $\mathbf{II_A}$ and $\mathbf{II_B}$ series, whose results are shown in Figs 3 and 4, respectively. Figure 3 shows the result of the $\mathbf{II_A}$ and $\mathbf{II'_A}$ series. The $\mathbf{II'_A}$ series displayed different crystal structures for the solvates by different solvents. If the $\mathbf{II'_A}$ series were de-

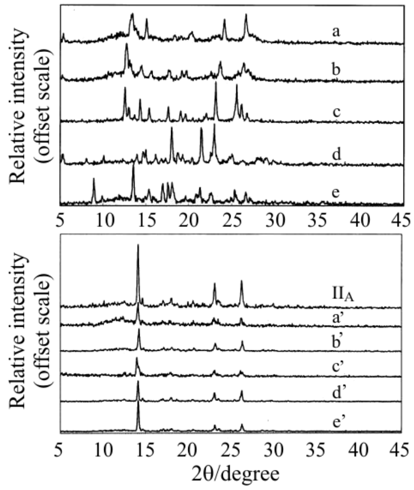


Fig. 3 Wide-angle X-ray diffractograms of $\mathbf{II'_A}$ and $\mathbf{II_A}$. Abbreviations: a – $\mathbf{II'_A}$-DMF; b – $\mathbf{II'_A}$-DMAc; c – $\mathbf{II'_A}$-NMP; d – $\mathbf{II'_A}$-DMSO; e – $\mathbf{II'_A}$-DMI; a' –, b' –, c' –, d' – and e' – are de-solvated. a –, b –, c –, d – and e – under 200°C for 1 h

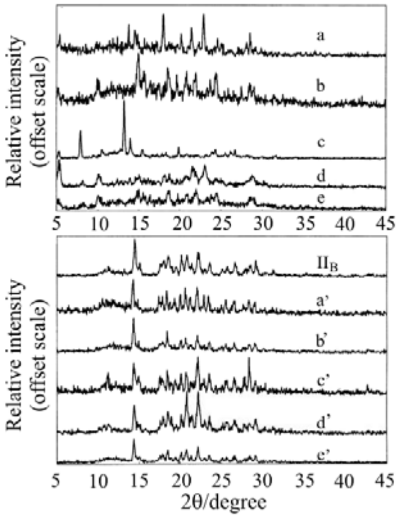


Fig. 4 Wide-angle X-ray diffractograms of $\mathbf{II}'_\mathbf{B}$ and $\mathbf{II_B}$. Abbreviations: a – $\mathbf{II}'_\mathbf{B}$-DMF; b – $\mathbf{II}'_\mathbf{B}$-DMAc; c – $\mathbf{II}'_\mathbf{B}$-NMP; d – $\mathbf{II}'_\mathbf{B}$-DMSO; e – $\mathbf{II}'_\mathbf{B}$-DMI; a' –, b' –, c' –, d' – and e' – are de-solvated. a –, b –, c –, d – and e – under 200°C for 1 h

solvated at temperature higher than T_d, e. g. 200°C for a period of time of 0.5–1 h, it was found that all de-solvated compounds (a', b', c', d', and e') are same as $\mathbf{II_A}$ as demonstrated. $\mathbf{II}'_\mathbf{A}$ apparently is a result of physical bonding between $\mathbf{II_A}$ and polar solvents (PSv), whose structures could be changed to $\mathbf{II_A}$ as the polar solvents were de-solvated. Similar discussions could be offered for the $\mathbf{II}'_\mathbf{B}$ and $\mathbf{II_B}$ series as shown in Fig. 4, which shows the crystallographs for the various crystalline solvates of $\mathbf{II_B}$ series (e. g. CS-$\mathbf{II}'_\mathbf{B}$).

Conclusions

The sulfone-based DIDA, owing to strong interactions between the sulfone-imide containing carboxylic acid groups, easily formed crystalline solvates (CS) with polar solvent molecules. High-temperature (e. g., 200°C) treatment on these crystalline solvates led to de-solvated DIDA, which may be returned into reversible crystalline solvates if they were re-dissolved into the polar solvents and re-crystallized. Most crystalline solvates (DIDA-CS) exhibited a T_d that is near the boiling point of the polar solvent. In some cases, T_d is much higher than the boiling point of the polar solvents, owing to strong interactions between solute and solvent. Such cases included $\mathbf{II}'_\mathbf{A}$-DMSO, $\mathbf{II}'_\mathbf{B}$-DMAc, $\mathbf{II}'_\mathbf{B}$-DMSO, and $\mathbf{II}'_\mathbf{B}$-DMF. Most crystalline solvates of DIDA-CS, the solute/solvent ratio is 1/2. In some other cases the ratios are near 2. For the crystalline solvate of DIDA-CS, the values of T_d are proportional to the boiling point of the polar solvents, in the order of DMI>DMSO>NMP>DMAc. The only exception is DMF. The crystalline solvates based on the same DIDA exhibited different characteristic X-ray diffraction peaks that varied with the type of solvents. However, the various DIDA-CS could be de-solvated at high temperatures, and all de-solvated DIDA exhibited the same X-ray diffraction peaks.

In addition, this study has demonstrated a critical point in thermal analysis techniques. This is especially important as the transition temperatures as revealed by DSC and TG characterizations are to be directly compared. Differences in DSC and TG curves may yield owing to sampling ways are, by instrument designs, different between these two techniques. This study has demonstrated that the sampling techniques in TG and DSC must be kept to be as similar as possible, which is discussed fully in the Experimental section.

* * *

The authors are grateful to the National Science Council of the Republic of China for the support of this work (Grant NSC 90-2216-E-036-016). The referees' critical and constructive comments have helped a great deal in enhancing the technical accuracy of this paper.

References

1 G.-L. Miessler and D.-A. Tarr, Inorganic Chemistry 2nd Edition, Prentice Hall, New Jersey 1998, p. 297.
2 M.-J. Alewicz, Mol. Struct., 407 (1997) 63.
3 Z.-A. Starikova, A.-I. Yanovsky, E.-P. Turevskaya and N.-Y. Turova, Polyhedron, 16 (1997) 967.
4 J.-K. Beatti, T.-W. Hambley, J.-A. Klepetko, A.-F. Masters and P. Turner, Polyhedron, 15 (1996) 2141.
5 G.-L. Perlovich, W. Zielenkiewicz, Z. Kaszkur, E. Utzig and O.-A. Golubchikov, Thermochim. Acta, 311 (1998) 163.
6 J.-K. Beatti, T.-W. Hambley, J.-A. Klepetko, A.-F. Masters and P. Turner, Polyhedron, 17 (1998) 1343.
7 M.-B. Ferrari, S. Capacchi, G. Pelosi, G. Reffo, P. Tarasconi, R. Albertini, S. Pinelli and P. Lunghi, Inorganica Chimica Acta, 286 (1999) 134.
8 N.-V. Avramenko, E.-B. Stukalin, M.-V. Korobov, I.-S. Neretin and Y.-L. Slovokhotov, Thermochim. Acta, 370 (2001) 21.
9 P. Tekely, P. Palmas, P. Mutzenhardt, F. M. A.–S. Grell, I. Messari and M. Gelbcke, Solid State Commun., 106 (1998) 391.
10 N.-V. Avramenko, A.-V. Mirakyan, I.-S. Neretin, Y.-L. Slovokhotov and M.-V. Korobov, Thermochim. Acta, 344 (2000) 23.
11 M. Murai, H. Nakayama and K. Ishii, J. Therm. Anal. Cal., 69 (2002) 953.
12 B. B. V. Sailaja, T. Kebede and M. S. Prasada Rao, J. Therm. Anal. Cal., 68 (2002) 841.
13 H.-D. Wang, Y.-T. Li, P.-H. Ma and X.-C. Zeng, J. Therm. Anal. Cal., 69 (2002) 575.
14 A. I. El-Said, J. Therm. Anal. Cal., 68 (2002) 917.
15 F. Chehimi-Moumen, D. Ben Hassen-Chehimi, M. Ferid and M. Trabelsi-Ayadi, J. Therm. Anal. Cal., 65 (2001) 87.
16 Y.-T. Chern and W.-L. Wang, Macromolecules, 28 (1995) 5554.
17 C.-P. Yang, S.-H. Hsiao and H.-C. Hsiao, J. Polym. Sci. Part A: Polym. Chem., 37 (1999) 69.
18 C.-P. Yang, R.-S. Chen and C.-C. Huang, J. Polym. Sci. Part A: Polym. Chem., 37 (1999) 2421.
19 C.-P. Yang and R.-S. Chen, Polymer, 40 (1999) 1025.
20 C.-P. Yang, S.-H. Hsiao and J.-H. Lin, U.S. Patent 5,414,070 (1995).