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An easy way to achieve three-dimensional metal–organic coordination polymers: synthesis and crystal structure of dizinc diisophthalate bis-dimethylsulfoxide monohydrate: $[Zn_2(ip)_4(DMSO)_2(H_2O){\cdot}3\ DMSO]_n$

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An easy way of producing three-dimensional metal–organic coordination polymers involving zinc(II) benzene-dicarboxylates is reported. The reaction of zinc oxide with benzene dicarboxylic acids in water yielded the expected hydrated zinc dicarboxylates. These zinc compounds were then suspended in dimethylsulfoxide and heated to above 100 °C for a couple of hours; the solutions were allowed after filtration to cool down to eventually deliver crystalline compounds displaying complex zeotype structures. The crystal structure of the title compound, $[Zn_2(ip)_4(DMSO)_2(H_2O){\cdot}3\ DMSO]_n$ (ipH_2 = isophthalic acid = 1,3-benzenedicarboxylic acid, DMSO = dimethylsulfoxide), is reported for the first time and shows a three-dimensional network where octahedrally and tetrahedrally coordinated zinc atoms (present in a 1:1 ratio) are linked by bridging isophthalate ligands. The complex coordination network exhibits a remarkable channel structure along the *z*-axis. The related zinc terephthalate–DMSO complex was similarly prepared and the crystal structure determination revealed an already documented zeotypic structure: $[\{Zn_4(OH)_2(tp)_3(DMSO)_4\}\ 2H_2O]_n$ (tpH_2 = terephthalic acid = 1,4-benzenedicarboxylic acid). Weak interactions as well as hydrogen bonds involving water molecules and carboxy groups play a major role in the formation of these complex three-dimensional networks. In comparison, the zinc 1,2-benzene-dicarboxylate–DMSO complex could not be isolated, even under more drastic conditions. The higher symmetry of the coordination network found in the zinc terephthalate–DMSO complexes was incidentally corroborated by ^{13}C CP/MAS spectroscopy. Copyright © 2007 John Wiley & Sons, Ltd.

KEYWORDS: zinc; benzene-dicarboxylate; X-ray structure determination; DMSO; three-dimensional network; hydrogen bond; MOF; ^{13}C CP/MAS

INTRODUCTION

Metal–organic frameworks (MOF) attract an ever-growing interest across a broad range of technological areas ranging from the manufacture of new photoluminescent materials to the development of new hydrogen storage materials and new catalytic systems.[1–7] The use of adequate polydentate ligands displaying a constrained geometry combined with effective hydrogen bonded networks allows definite three-dimensional-networks to be defined and, in particular cases, better understanding of some important structure–reactivity relationships. This approach might be very useful in the copolymerization of CO_2 and epoxides where zinc dicarboxylates play an important role as effective heterogeneous catalysts.[8–10] The most efficient

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dicarboxylates are obtained from glutaric and adipic acid [$HO_2C\text{-}(CH_2)_n\text{-}CO_2H$, with $n = 3$ or 4] and display complex polymeric structures. Despite comprehensive studies on the reactivity of the Zn carboxylate–CO_2–epoxide system,[11–14] the overall mechanism and the nature of the active site in this catalysis remain a subject of discussion. Dicarboxylic acids presenting a steric restriction (e.g. an aromatic or a bicyclic spacer) are in this regard an interesting tool for the directed construction of extended networks[15–17] and should play an important role in the development of new copolymerization catalysts. We report here the X-ray structural characterization of a new polymeric three-dimensional network obtained from zinc isophthalate hydrate[18] via a surprisingly easy synthetic method. This method involves the synthesis of the hydrated zinc carboxylate according to 'the oxide way' (zinc oxide reacted with a given carboxylic acid) followed by the dissolution in DMSO above 100 °C. This method of synthesis is noteworthy if one considers that most other reported metal–organic coordination polymers including zinc dicarboxylate fragments have been generated via more complex synthetic methods. These syntheses are mainly based on ligand exchanges between a carboxylic acid or its sodium salt and a suitable zinc salt (nitrate, chloride or perchlorate) in the presence of a solvent as organic template and a base.[18–22] We extended the synthesis to zinc 1,2-benzene dicarboxylate (phthalate) and zinc 1,4-benzene dicarboxylate (terephthalate). Unfortunately owing to the very poor solubility of zinc phthalate in DMSO (also at 200 °C in stainless steel autoclave), we were not able to isolate a crystalline zinc phthalate-DMSO compound. Also, running the synthesis directly in DMSO, from ZnO and phthalic acid, yielded a polycrystalline, needle-formed compound without DMSO included in the metalorganic network, as confirmed by ^{13}C-CP/MAS spectroscopy. In comparison we were able to isolate single crystals and perform an X-ray structure determination in the case of the zinc terephtalate–DMSO compound. This compound was revealed to be similar to the metalorganic network reported by Wang *et al.*,[23] confirming thus the applicability of the synthetic method.

RESULTS AND DISCUSSION

Tetrakis (μ-1,3-benzenedicarboxylate)bis (dimethyl sulfoxide) dizinc monohydrate DMSO trisolvate [$\{Zn_2(ip)_4(DMSO)_2(H_2O)\}\cdot 3$ DMSO]$_n$

Zinc oxide was added to an aqueous solution of 1,3-benzene dicarboxylic acid (1 : 1 molar ratio). The resulting cloudy suspension was stirred under reflux for ca. 4 h; the remaining fine particles were then filtered and the solution was eventually concentrated *in vacuuo* to yield a crystalline white solid of general form [$(ip)Zn(H_2O)_x]_n$, barely soluble in common solvents (yield >95%). The isolated polycrystalline powder was suspended in hot DMSO (ca. 120 °C) and stirred until complete dissolution; the solution was then concentrated

Table 1. Selected geometric parameters (bonds, Å; angles, deg) for compound **1**

Zn(1)–O(1)	2.0725(19)
Zn(1)–O(11)	2.081(2)
Zn(1)–O(5)	2.0963(19)
Zn(1)–O(10)	2.1069(19)
Zn(1)–O(7)	2.1216(19)
Zn(1)–O(9)	2.131(2)
Zn(2)–O(2)	1.9329(19)
Zn(2)–O(6)	1.9427(19)
Zn(2)–O(3)[a]	1.9550(19)
Zn(2)–O(8)	1.9666(18)
O11···O12: 2.72 Å	2.72 Å
O(1)–Zn(1)–O(11)	88.17(9)
O(1)–Zn(1)–O(5)	94.09(8)
O(11)–Zn(1)–O(5)	93.53(8)
O(1)–Zn(1)–O(10)	177.42(8)
O(11)–Zn(1)–O(10)	89.84(9)
O(5)–Zn(1)–O(10)	87.68(8)
O(1)–Zn(1)–O(7)	95.00(8)
O(11)–Zn(1)–O(7)	170.66(9)
O(5)–Zn(1)–O(7)	94.99(7)
O(10)–Zn(1)–O(7)	86.71(8)
O(1)–Zn(1)–O(9)	91.94(8)
O(11)–Zn(1)–O(9))	82.69(9)
O(5)–Zn(1)–O(9)	172.79(8)
O(10)–Zn(1)–O(9)	86.17(8)
O(7)–Zn(1)–O(9)	88.42(8)
O(2)–Zn(2)–O(6)	114.35(9)
O(2)–Zn(2)–O(3)[a]	115.75(9)
O(6)–Zn(2)–O(3)[a]	105.93(9)
O(2)–Zn(2)–O(8)	108.79(9)
O(6)–Zn(2)–O(8)	113.02(9)
O(3)[a]–Zn(2)–O(8)	97.94(8)

Symmetry transformations used to generate equivalent atoms: [a] $x + 1/2, -y + 3/2, -z + 1$; [b] $x - 1/2, -y + 3/2, -z + 1$; [c] $-x + 2, y - 1/2, -z + 3/2$; [d] $-x + 2, y + 1/2, -z + 3/2$.

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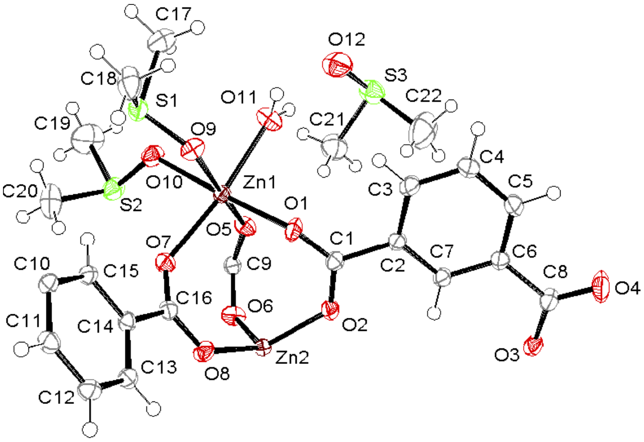

Figure 1. ORTEP drawings (30% probability) with atomic numbering of the basic fragment found in [{$Zn_2(ip)_4(DMSO)_2(H_2O)$}·3 DMSO]$_n$. (noncoordinated DMSO molecule omitted for sake of clarity). This figure is available in colour online at www.interscience.wiley.com/AOC.

and allowed to stand for ca. 4 weeks at room temperature to eventually afford small prismatic single-crystals suitable for an X-ray structural characterization (the procedure can be shortened by using an oven at 100 °C for a couple of days, although this yields only microcrystalline material). The crystals of the title compound were orthorhombic, space group $P2_12_12_1$ (no. 19). Experimental data for the crystal structures of compound **1** are listed in Table 1. (Figures were prepared using Winray32 (R. Soltek, WinRay32, University of Heidelberg, 2000) and ORTEP-3 for Windows version 1.08 (L. J. Farrugia, University of Glasgow, 2005). Crystallographic data of the structures have been deposited at the Cambridge Crystallographic Database Centre, supplementary publication no. CCDC 634460. Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033, e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk.) Three solvated DMSO molecules stabilize the crystal structure, one of them being disordered. The molecular structure of the elementary motif is given in Fig. 1 with atomic labeling, whereas selected bond lengths and angles are listed in Table 2. Compound **1** displays a metalorganic coordination network that consists of repeating [$Zn_2(ip)_4(DMSO)_2(H_2O)$] units containing two different zinc atoms, one being octahedrally coordinated while the other displays a tetrahedral coordination geometry. Within this unit the octahedral Zn coordinates one water molecule, two DMSO molecules and three bridging carboxylate groups whereas the tetrahedral zinc atom bears four carboxylates groups. Three of them act as bidentate (O,O′.) bridging ligands between octahedral and tetrahedral zinc atoms, whereas the fourth carboxylate is monodentate, bonded only via one oxygen atom (see Fig. 2 for details). The isophthalate ligands can be ordered into two groups according to their bridging coordination mode: two isophthalates exhibit strictly bidentate carboxylate groups (μ_2-O5,O6 and μ_2-O6,O8), while the other ones display one bidendate (μ_2-O1,O2) and one monodentate (O3) carboxylate. Both isophthalate types connect tetrahedral and octahedral zinc centers to generate a complex three-dimensional architecture displaying a remarkable channel structure along the Z axis (Fig. 3). The presence of free DMSO molecules in the crystal structure seems to play a major role in the formation of this channelled structure. One of these free DMSO molecules interacts with the water molecule coordinated to Zn2; the resulting hydrogen bond is quite strong (O11· · ·O12, 2.72 Å) and might also contribute to the edification of the complex three-dimensional-network. In comparison, the structure of the related zinc isophthalate monohydrate displays a more regular three-dimensional structure with monodentate isophthalate's carboxy groups and tetrahedral zinc atoms (one water molecule and three carboxylate O atoms).[18] The presence of DMSO in zinc-containing three-dimensional

Table 2. Experimental data for the crystal structure of **1**

Crystal data	Compound
Empirical formula	C_{26} H_{40} O_{14} S_5 Zn_2
Molecular mass	867.62
Crystal color	colorless
Crystal size, mm	0.5 × 0.5 × 0.5
Crystal system	Orthorhombic
Space group	$P2_12_12_1$ (no.19)
a (´Å)	12.6977(16)
b (´Å)	15.1033(19)
c (´Å)	18.903(2)
α (deg)	90
β (deg)	90
γ (deg)	90
V (´Å^3)	3625.2(8)
Z	4
D calcd (g cm^{-3})	1.590
μ (mm^{-1})	1.673
F(000)	1792
Wavelength (´Å)	0.71073
T (K)	200(2) K
θ−range (deg)	1.73 to 28.32
Index ranges	$-16 \le h \le 16$
	$-20 \le k \le 19$
	$-25 \le l \le 25$
Solution and refinement	
Number of reflections measured	42703
Number of independent reflections	8857
GOF	1.071
R [$I.2\theta(I)$]	0.0293
$wR2$ [$I.2\theta(I)$]	0.0767
Largest e_{max}, e_{min} (e´Å^{-3})	0.760 and −0.548

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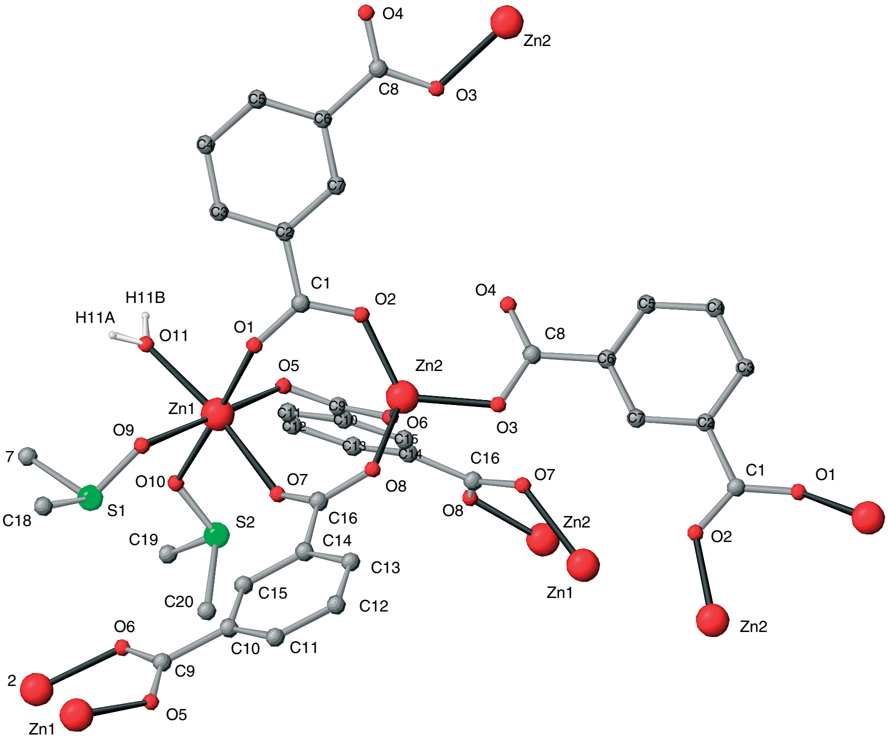

Figure 2. Winray32 view with atomic numbering showing different coordination modes of the isophthalate and the two different coordination environments around the zinc atoms in $[\{Zn_2(ip)_4(DMSO)_2(H_2O)\}{\cdot}3\ DMSO]_n$. This figure is available in colour online at www.interscience.wiley.com/AOC.

metalorganic structures has been rarely reported;[24–26] unfortunately, studies on its influence in the edification of zeotypic structures remain scarce. To illustrate this, two zinc terephthalate–DMSO complexes prepared according to a similar procedure and displaying two completely distinct zeotypic structures have been reported: $[\{Zn_2(tp)_2(DMSO)_2\}{\cdot}5DMSO]_n$ obtained from $Zn(NO_3)_2{\cdot}6H_2O$–terephthalic acid[25] and $[\{Zn_4(OH)_2(tp)_3(DMSO)_4\}{\cdot}2H_2O]_n$[23] prepared from $Zn(ClO_4)_2{\cdot}6H_2O$–sodium terephthalate.

Tris (μ-1,4-benzenedicarboxylate)tetrakis (dimethyl sulfoxide)di-μ_3-hydroxo-tetrazinc dihydrate $[\{Zn_4(OH)_2(tp)_3(DMSO)_4\}{\cdot}2H_2O]_n$

The synthesis was similar to that used for the zinc isophthalate derivative. An aqueous suspension of zinc oxide–1,4-benzene dicarboxylic acid (1 : 1 molar ratio) was stirred under reflux for ca. 4 h; after filtration the solution was eventually concentrated *in vacuuo* to quantitatively yield a white amorphous solid of general form $[(tp)Zn(H_2O)_x]_n$ ($x = 1$ or 2).[27] The solid was suspended in hot DMSO (ca. 110 °C) and stirred for ca. 4 h; the suspension was then hot filtered and allowed to stand for ca. 4 weeks at room temperature to eventually yield small prismatic single crystals suitable for an X-ray structural characterization. Crystals of compound **2** are orthorhombic, space group Pbca (no. 61), with $a = 14.4669(17)$, $b = 17.046(2)$, $c = 18.020(2)$; $Z = 8$, $Dc = 1.674$; $R = 0.0455$, $Rw(F2) = 0.0787$ for 5564 reflections. (Crystallographic data of the structures have been deposited at the Cambridge Crystallographic Database Centre, supplementary publication no. CCDC 649474. Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk; or www: http://www.ccdc.cam.ac.uk).

One solvated water molecule, slightly disordered, stabilizes the crystal structure via a hydrogen bond with a carboxy oxygen atom of a terephthalate ligand. Compound **2** displays a crystal structure similar to that reported for the zinc terephthalate–DMSO complex prepared from $Zn(ClO_4)_2.6H_2O$ and sodium terephthalate,[23] with slightly better structural parameters (R factor of 0.0455 against 0058). This species displays a metal–organic coordination network consisting of repeating $\{Zn_4(OH)_2(tp)_3(dmso)_4\}$ units with two different coordination environments around the zinc atoms (octahedral and tetrahedral). These characteristic $Zn_4(OH)_2(tp)_3$ clusters are regularly found in zinc terephthalates obtained following other synthesis methods.[29–32] The zinc atoms in the $Zn_4(OH)_2(tp)_3$ clusters are connected by two hydroxide groups to form Zn_2O_2 rhombuses with are further linked via two terephthalate ligand types (Fig. 4). Similarly to the zinc isophthalate–DMSO derivative, one ligand type exhibits strictly bidentate carboxylate groups (μ_2-O11,O12 and μ_2-O13,O14) while the other displays two monodentate (O21 and

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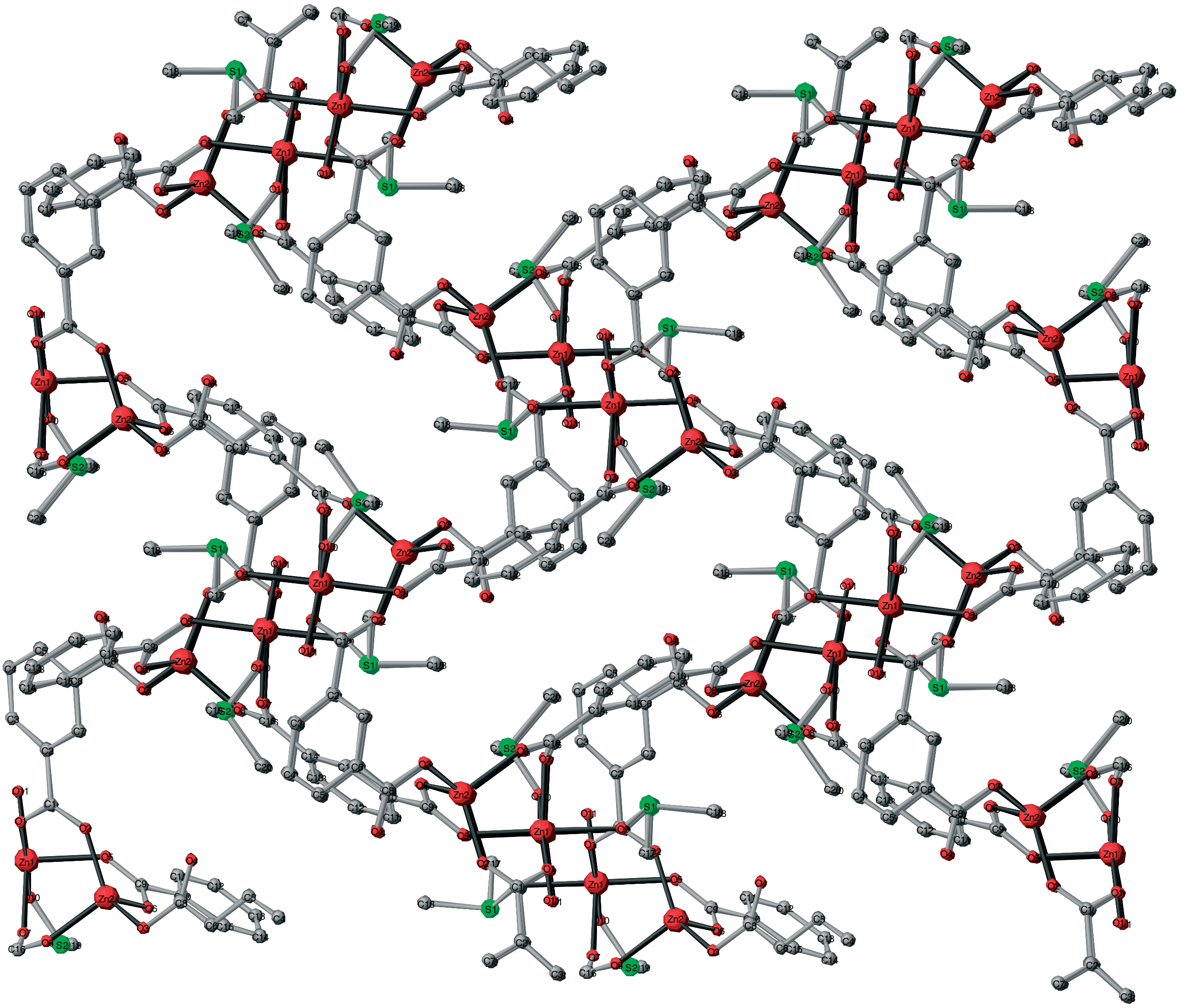

Figure 3. Winray32 view showing the characteristic channelled structure along the *z*-axis found in $[\{Zn_2(ip)_4(DMSO)_2(H_2O)\}\cdot 3\ DMSO]_n$ (free DMSO molecules are omitted for clarity). This figure is available in colour online at www.interscience.wiley.com/AOC.

O22) carboxylate. The crystal structure of the zinc terephthalate–DMSO complex shows a zeolite-like channeled structure along the *y* and *z* axes with an overall higher symmetry than in the case of the zinc isophthalate–DMSO (Fig. 5). This was confirmed indirectly with ^{13}C CP/MAS spectroscopy.[†] Compounds **1** and **2** were measured as polycrystalline powders at different rotation frequencies. The $^{13}C\{^1H\}$ NMR spectra recorded at 6 kHz are displayed in Fig. 6. In compound **2**, which displays the higher symmetry, several discrete signals belonging to different types of DMSO molecules ($\delta = 42.6$, 44.0, 44.9, 45.7 ppm) and carboxylate groups (177.0, 178.0, 179.0 ppm) present in the crystal structure can be clearly seen. The remaining aromatic carbons are found as two sets of signals: at 143.8, 144.4, 146.4 ppm and at 135.7, 136.7, 137.7 ppm for quaternary and tertiary carbons, respectively. In comparison, compound **1** delivered at 6 kHz ^{13}C spectra with a lower resolution: carboxylates, aromatic carbons and methyl groups of the DMSO molecules were found as broad signals ($\Delta W_{1/2} \approx 400$–$500$ Hz) at 178.5, 138.1 (supplementary shoulder at 133.6 ppm) and 44.1 ppm, respectively. Interestingly, zinc phthalate prepared in DMSO from zinc oxide and phthalic acid, once washed with methanol to eliminate the excess of dimethylsulfoxide, delivered ^{13}C CP/MAS

[†] Solid-state $^{13}C\{^1H\}$ NMR characterization was performed on a Varian Inova 400 spectrometer with an 9.4 T Oxford Magnet (^{13}C 100.567 MHz) equipped with a broadband double-bearing MAS probehead (rotors: ZrO_2/5 mm diameter). The zinc carboxylates were densely packed into the zirconia rotors. The rotors were spun at 3, 5 and 6 kHz in order to determine the isotropic lines. Cross polarization and magic angle spinning were applied for all the spectra. The contact time was 3 ms and the recycle delay was 5 s. The sweep width, pulse duration and number of scans were 50 kHz, 5.0 µs and ca. 10 000 scans. All spectra were recorded at room temperature (298 K) and externally referenced with respect to hexamethyl benzene (132.1 ppm for the aromatic line) whereas the Hartmann-Hahn match was optimized using adamantane. The final FIDs were put to an exponential multiplication with a line-broadening factor ranging from 10 to 20 Hz.

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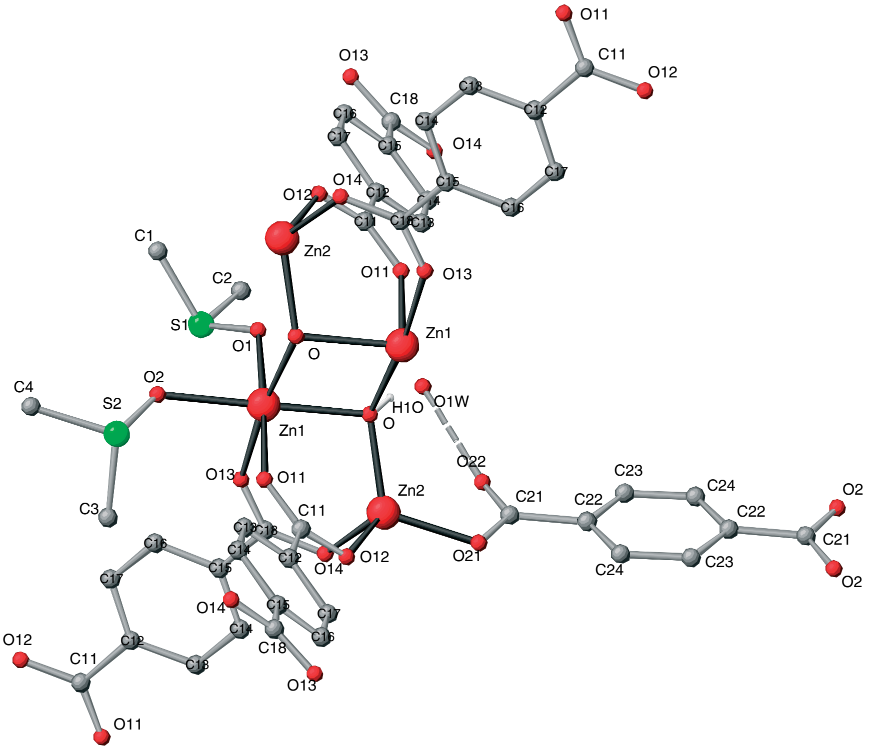

Figure 4. Winray32 view with atomic numbering showing coordination modes of the terephthalates and coordination environments around the zinc atoms in $[\{Zn_4(OH)_2(tp)_3(DMSO)_4\}\cdot 2H_2O]_n$. This figure is available in colour online at www.interscience.wiley.com/AOC.

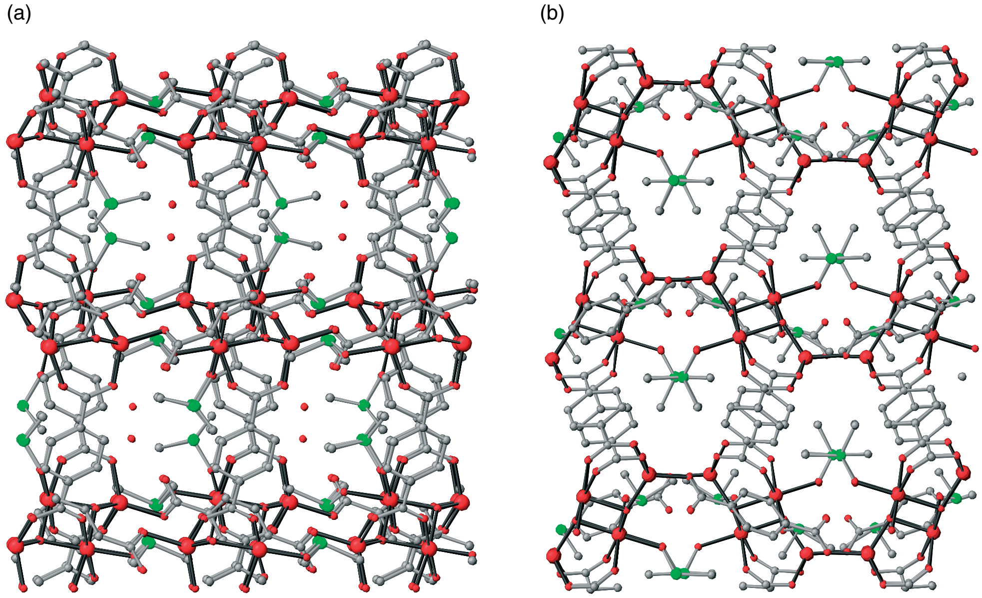

Figure 5. Winray32 view showing the characteristic channeled structure along the *z* (a) and *y* (b) axis found in $[\{Zn_4(OH)_2(tp)_3(DMSO)_4\}\cdot 2H_2O]_n$. This figure is available in colour online at www.interscience.wiley.com/AOC.

spectra without the signal at ca. 44 ppm characteristic of the DMSO's methyl groups (see supplementary materials). These spectra were comparable to the spectra recorded in the case of an amorphous zinc phthalate obtained from zinc oxide and phthalic acid in water, although showing a significantly better resolution owing to a higher crystallinity. This suggests

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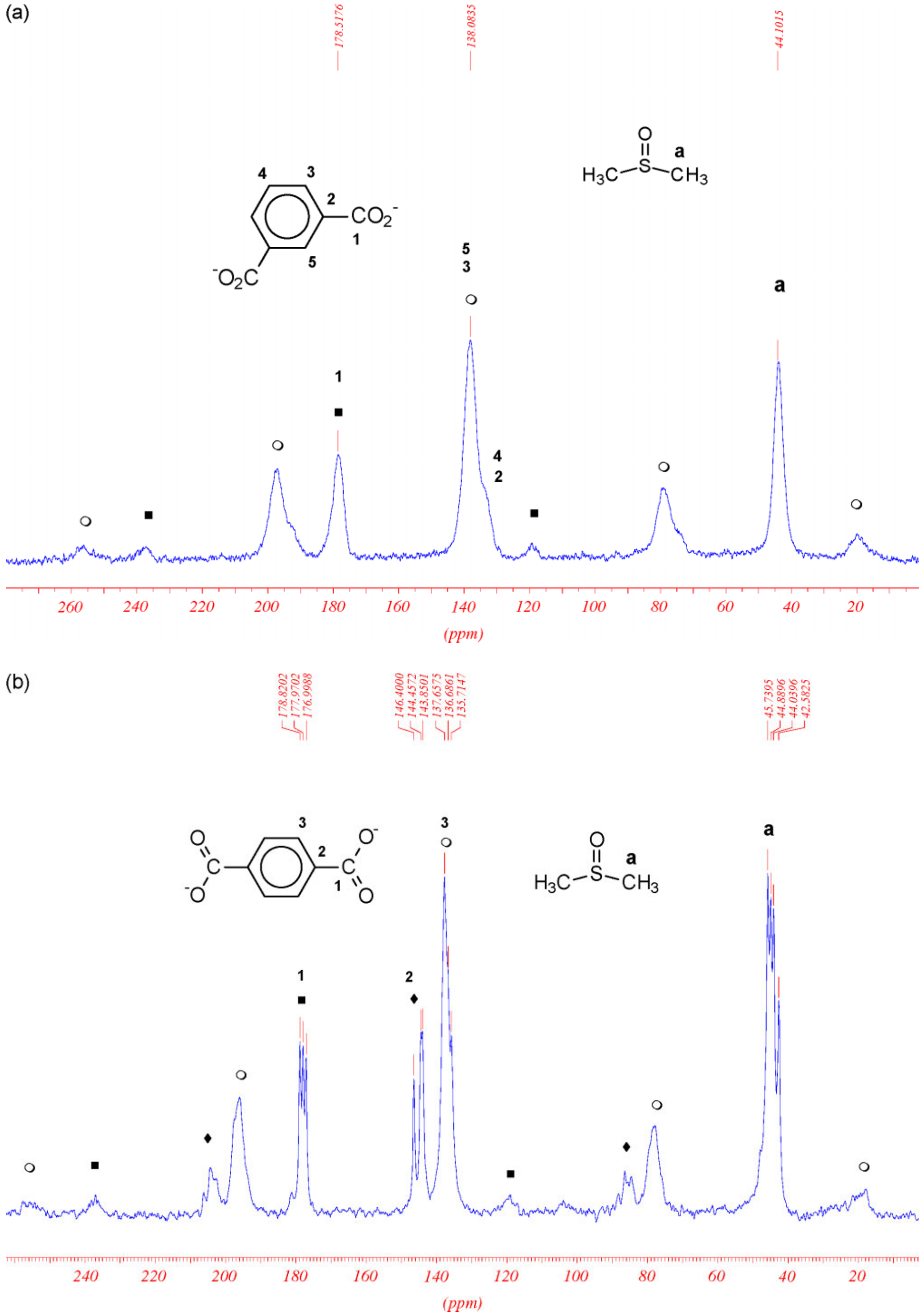

Figure 6. CP/MAS ^{13}C NMR spectra of zinc isophthalate–DMSO (a) zinc terephthalate–DMSO (b) compounds measured at 6 kHz (interrelated spinning bands are indicated). This figure is available in colour online at www.interscience.wiley.com/AOC.

that the coordination of an O-donor ligand, like, for example, DMSO, to the zinc atom in zinc phthalate derivatives is not favorable for electronic and/or steric reasons. Supporting this, the only X-ray crystal structures involving zinc phthalate reported in the literature clearly show the preferred role of nitrogen-donor bases as stabilizing ligands like 2,2-bipyridine,[37] 1,4,8,11-tetraazacyclotetradecane (cyclam)[35] or imidazole- and pyridine derivatives[33,34] and, in some rare cases, the role of sulfur-donor molecules like thiourea.[19]

More work is currently in progress to better understand the influence of the substitution at the aromatic ring on the final zeotype three-dimensional-stucture of the zinc benzene-dicarboxylates and, within this context, to

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structurally characterize the 'missing link' in this study, zinc 1,2-benzenedicarboxylate (DMSO-solvated or not).

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