

Adducts of 1,1,1-tris(4-hydroxyphenyl)ethane with diamines: three-dimensional hydrogen-bonded frameworks formed with 1,6-diaminohexane and 2,2'-bipyridyl

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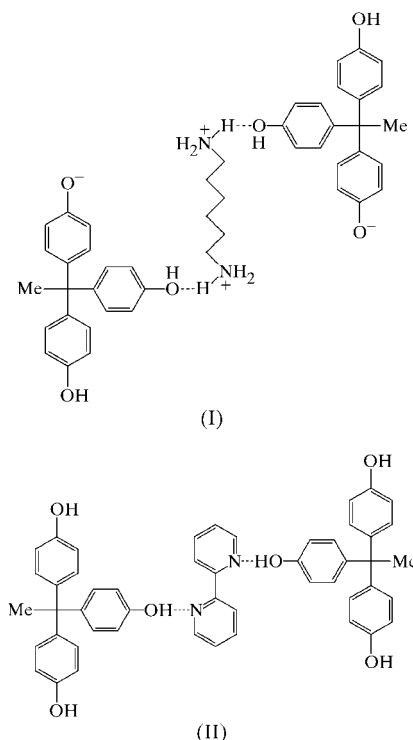
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The adduct 1,6-diaminohexane–1,1,1-tris(4-hydroxyphenyl)ethane (1/2) is a salt [hexane-1,6-diyl diammonium–4-[1,1-bis(4-hydroxyphenyl)ethyl]phenolate (1/2)], $C_6H_{18}N_2^{2+} \cdot 2C_{20}H_{17}O_3^-$, in which the cation lies across a centre of inversion in space group $P\bar{1}$. The anions are linked by two short O–H...O hydrogen bonds [H...O 1.74 and 1.76 Å, O...O 2.5702 (12) and 2.5855 (12) Å, and O–H...O 168 and 169°] into a chain containing two types of $R_2^2(24)$ ring. Each cation is linked to four different anion chains by three N–H...O hydrogen bonds [H...O 1.76–2.06 Å, N...O 2.6749 (14)–2.9159 (14) Å and N–H...O 156–172°]. In the adduct 2,2'-bipyridyl–1,1,1-tris(4-hydroxyphenyl)ethane (1/2), $C_{10}H_8N_2 \cdot 2C_{20}H_{18}O_3$, the neutral diamine lies across a centre of inversion in space group $P2_1/n$. The tris(phenol) molecules are linked by two O–H...O hydrogen bonds [H...O both 1.90 Å, O...O 2.7303 (14) and 2.7415 (15) Å, and O–H...O 173 and 176°] into sheets built from $R_4^4(38)$ rings. Pairs of tris(phenol) sheets are linked *via* the diamine by means of a single O–H...N hydrogen bond [H...N 1.97 Å, O...N 2.7833 (16) Å and O–H...N 163°].

Comment

When cocrystallized with 4,4'-bipyridyl, the threefold symmetric tris(phenol) 1,1,1-tris(4-hydroxyphenyl)ethane, $CH_3C(C_6H_4OH)_3$, forms a hydrogen-bonded adduct of 3:2 stoichiometry, $3C_{10}H_8N_2 \cdot 2C_{20}H_{18}O_3$, in which all the hydrogen bonds are of the O–H...N type and the supramolecular structure of which consists of tenfold interwoven nets built

from $R_{12}^{12}(126)$ rings (Bényei *et al.*, 1998). In a similar way, 4,4'-bipyridyl forms a 3:2 adduct with 1,3,5-benzenetricarboxylic acid, where the supramolecular structure consists of threefold interwoven sheets of $R_{12}^{12}(102)$ rings (Sharma & Zaworotko, 1996). In the expectation of forming nets built from still larger rings, we recently investigated the adducts formed by 1,1,1-tris(4-hydroxyphenyl)ethane with the extended bipyridyl 1,2-bis(4-bipyridyl)ethane, but this yielded not a 3:2 adduct but, instead, a 1:2 adduct (Zakaria *et al.*, 2002). Continuing our study of the adducts formed by 1,1,1-tris(4-hydroxyphenyl)ethane and diamines, we have now synthesized and characterized the adducts (I) and (II), formed with 1,6-diaminohexane and 2,2'-bipyridyl, respectively.



The tris(phenol) 1,1,1-tris(4-hydroxyphenyl)ethane forms a 1:2 adduct, (I), with 1,6-diaminohexane. The constitution of (I) is that of a salt, $C_6H_{18}N_2^{2+} \cdot 2C_{20}H_{17}O_3^-$, in which one hydroxyl H atom has been transferred from each molecule of the tris(phenol) to an amine group (Fig. 1). The constitution of (I) may be contrasted with that of the corresponding 1:2 adduct formed with 1,2-diaminoethane (Ferguson *et al.*, 1998b), in which there is no transfer of an H atom from O to N, so that both components are neutral overall.

The cation in (I) lies across a centre of inversion in space group $P\bar{1}$, chosen, for the sake of convenience, as that at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, and the anion occupies a general position. Five hydrogen bonds, two of the O–H...O type and three of the N–H...O type (Table 2), link the ions into a three-dimensional framework, which can readily be analysed using the substructure approach (Gregson *et al.*, 2000). The anions form a one-dimensional substructure in the form of a chain of fused rings, and these chains are linked by the cations into the overall three-dimensional structure, where each cation is linked to four anion chains.

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The hydroxyl atom O2 in the anion at (x, y, z) acts as a hydrogen-bond donor to the charged atom O1 in the anion at $(1 - x, 1 - y, 2 - z)$, while atom O2 at $(1 - x, 1 - y, 2 - z)$ in turn acts as a donor to atom O1 at (x, y, z) , so generating a centrosymmetric $R_2^2(24)$ ring, centred at $(\frac{1}{2}, \frac{1}{2}, 1)$. Similarly, hydroxyl atom O3 at (x, y, z) acts as a hydrogen-bond donor to atom O1 at $(1 - x, -y, 2 - z)$, so producing a second $R_2^2(24)$ ring, centred at $(\frac{1}{2}, 0, 1)$. In this manner, a chain containing two types of $R_2^2(24)$ ring is generated, running along the line $(\frac{1}{2}, y, 1)$ (Fig. 2). There is just one chain of this type per unit cell.

The ammonium N4 atoms at (x, y, z) and $(1 - x, 1 - y, 1 - z)$ both lie in the cation centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, and each forms three N—H···O hydrogen bonds (Table 2). Atom N4 at (x, y, z) acts as a hydrogen-bond donor *via* atoms H4A, H4B and H4C, respectively, to atom O2 at $(2 - x, 1 - y, 1 - z)$, O1 at $(x, y, z - 1)$ and O3 at (x, y, z) . These O atoms lie in [010] chains along $(\frac{3}{2}, y, 0)$, $(\frac{1}{2}, y, 0)$ and $(\frac{1}{2}, y, 1)$, respectively. The corresponding O-atom acceptors from N4 at $(1 - x, 1 - y, 1 - z)$ are at $(x - 1, y, z)$, $(1 - x, 1 - y, 2 - z)$ and $(1 - x, 1 - y, 1 - z)$, and these acceptors lie in the anion chains along $(-\frac{1}{2}, y, 1)$, $(\frac{1}{2}, y, 1)$ and $(\frac{1}{2}, y, 0)$, respectively. Hence, each cation is directly hydrogen-bonded to four different [010] chains, and propagation of the N—H···O hydrogen bonds by the space group links all the chains into a single framework (Fig. 3).

The 1:2 adduct formed with 2,2'-bipyridyl, (II), differs from (I) in that both components in (II) are neutral overall, with no transfer of an H atom from O to N, so that the formula is $C_{10}H_8N_2 \cdot 2C_{20}H_{18}O_3$ (Fig. 4). The diamine lies across a centre of inversion in space group $P2_1/n$, chosen as that at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, and it thus has parallel rings, with the two N atoms on opposite edges of the essentially planar molecule.

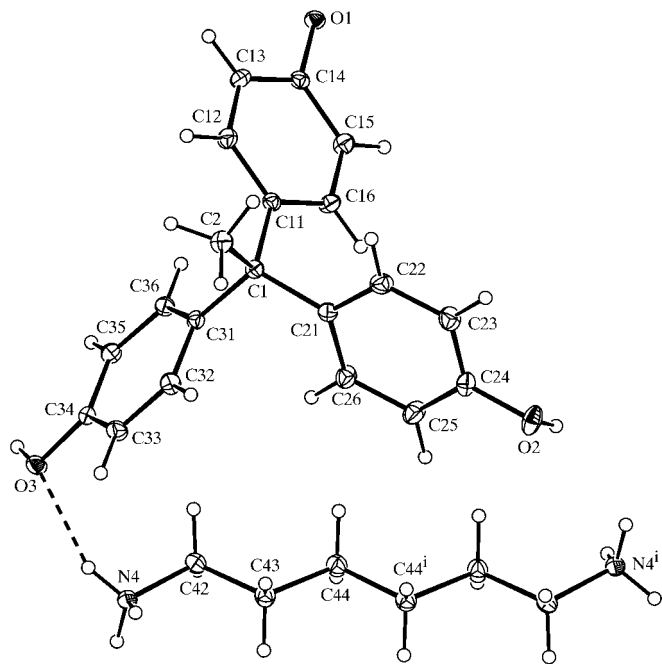


Figure 1
The molecular components of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level [symmetry code: (i) $1 - x, 1 - y, 1 - z$].

Three hydrogen bonds (Table 4) link the components of (II) into a three-dimensional framework, and again this is most readily analysed in terms of the tris(phenol) substructure, which is two-dimensional in (II), as opposed to one-dimensional in (I). Atom O3 at (x, y, z) acts as a hydrogen-bond donor to atom O2 at $(x, 1 + y, z)$, so generating by translation a $C(12)$ chain running parallel to the [010] direction (Fig. 5). Atom O2 at (x, y, z) itself acts as a donor to atom O1 at $(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$, while atom O2 at $(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$ in turn acts as a donor to O1 at $(1 + x, y, 1 + z)$, so producing a second $C(12)$ chain, this time parallel to [101] and generated by the n -glide plane at $y = \frac{1}{4}$ (Fig. 5). The combination of these two chain motifs generates a $(10\bar{1})$ sheet built from a single type of $R_4^4(38)$ ring.

Centrosymmetrically related pairs of $(10\bar{1})$ sheets in (II) are linked by the diamines (Fig. 6) to form centrosymmetric bilayers. Atom O1 at (x, y, z) is part of the reference $(10\bar{1})$ sheet (Fig. 5), and it acts as a hydrogen-bond donor to atom N42, also at (x, y, z) , part of the diamine centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. The symmetry-related N42 atom in the same diamine is at $(1 - x, 1 - y, 1 - z)$, and it accepts a hydrogen bond from atom O1 at $(1 - x, 1 - y, 1 - z)$, which lies in an adjacent inversion-related sheet. Within this second sheet, atom O2 at $(1 - x, 1 - y, 1 - z)$ acts as a hydrogen-bond donor to atom O1 at $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$, which in turn acts as a donor to atom N42 at $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$. This N42 is a component of the diamine

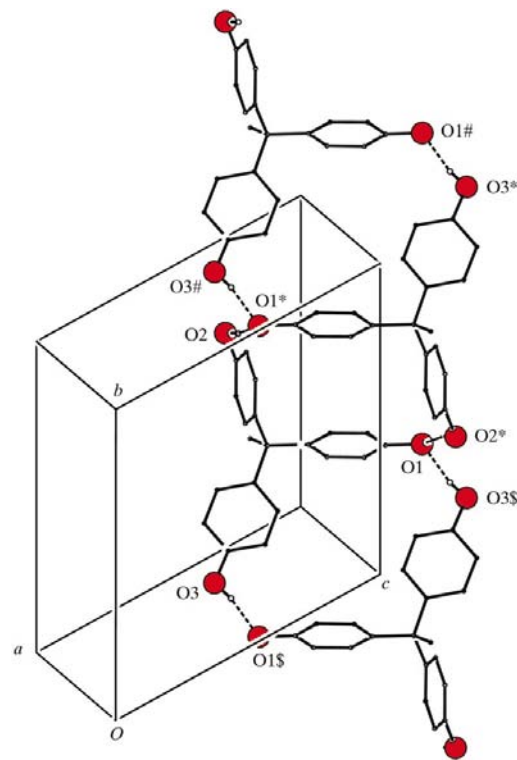


Figure 2
Part of the crystal structure of (I), showing the formation of a chain of fused $R_2^2(24)$ rings along [010]. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*), hash (#) or dollar sign (\$) are at the symmetry positions $(1 - x, 1 - y, 2 - z)$, $(x, 1 + y, z)$ and $(1 - x, -y, 2 - z)$, respectively.

centred at (0,1,0), with the symmetry-related N42 atom in the (0,1,0) diamine at $(x - \frac{1}{2}, \frac{3}{2} - y, -\frac{1}{2})$, and it accepts a hydrogen bond from atom O1 at $(x - \frac{1}{2}, \frac{3}{2} - y, -\frac{1}{2})$, which itself lies in the original (10 $\bar{1}$) sheet. In each bilayer, each diamine passes through an $R_4^4(38)$ ring in an adjacent bilayer (Fig. 6), so that each bilayer is interlinked with its two immediate neighbours, so generating a single three-dimensional structure. The overall supramolecular topology in (II) is thus analogous to that in the adduct formed between the tris(phenol) and 1,2-bis(4-pyridyl)ethane (Zakaria *et al.*, 2002).

All of the hydrogen bonds in compounds (I) and (II) have $H \cdots A$ and $D \cdots A$ distances which are fairly short for their types, and the $D-H \cdots A$ angles all exceed 160° (Tables 2 and 4). It is notable that the $O \cdots O$ distances for the hydrogen bonds in (I), where the acceptor in both is the negatively charged atom O1, are significantly shorter than those in (II), where both acceptors still carry an H atom. Similarly, of the three $N-H \cdots O$ hydrogen bonds in (I), that involving the charged atom O1 is significantly shorter than the other two, even though O1 acts here as a triple acceptor, whereas atoms O2 and O3 are both single acceptors.

In each of (I) and (II), the heavy-atom skeleton of the tris(phenol) component adopts a conformation close to C_3 rotational symmetry (Tables 1 and 3), while the centrosymmetric cation in (I) adopts the all-*trans* extended-chain conformation, with the two independent torsion angles both

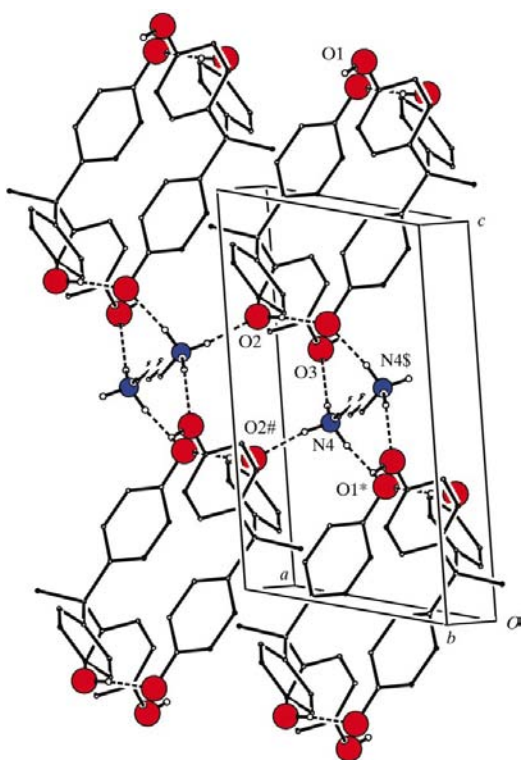


Figure 3
Part of the crystal structure of (I), showing the linking by the cations of the [010] chains. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*), hash (#) or dollar sign (\$) are at the symmetry positions $(x, y, z - 1)$, $(2 - x, 1 - y, 1 - z)$ and $(1 - x, 1 - y, 1 - z)$, respectively.

close to 180° (Table 1). The bond lengths and angles present no unexpected features.

It is appropriate to summarize here the general structural features of the adducts formed by 1,1,1-tris(4-hydroxyphenyl)ethane with diamines. In the 1:1 and 1:2 adducts formed with hexamethylenetetramine, $(CH_2)_6N_4$, the amine and the tris(phenol) units alternate in sheets and chains, respectively, with additional amine units pendant from the chains in the 1:2 adduct, so that no tris(phenol) substructure can be identified in either adduct (Coupar, Ferguson *et al.*, 1997; Coupar, Glidewell & Ferguson, 1997). Similarly, there is no tris(phenol) substructure present in the 3:2 adduct formed with 4,4'-bipyridyl (Bényei *et al.*, 1998). In all of the other adducts we have studied (Ferguson *et al.*, 1997, 1998*a,b*, 1999; Glidewell *et al.*, 2000; Zakaria *et al.*, 2002), there are two motifs formed by the tris(phenol) molecules which are consistently present. The predominant motif is a $C(12)$ chain, often generated by translation and formed when the hydroxyl O atom in one molecule acts as a hydrogen-bond donor to an O atom in another molecule. This motif is present in the adducts with 1,4-diazabicyclo[2.2.2]octane (Ferguson *et al.*, 1997), 1,2-diaminoethane (Ferguson *et al.*, 1998*b*), bis(2-aminoethyl)amine (Glidewell *et al.*, 2000), 1,10-phenanthroline (Ferguson *et al.*, 1999), and 1,2-bis(4-pyridyl)ethane and 1,2-bis(4-pyridyl)ethene (Zakaria *et al.*, 2002), as well as in (I) and (II). When there are two independent molecules of the tris(phenol), the $C(12)$ motif can be replaced by a $C_2^2(24)$ chain, as in the adducts formed with piperazine (Ferguson *et al.*, 1997) and 1,4,8,11-tetraazacyclotetradecane (Ferguson *et al.*, 1998*a*).

Pairs of these $C(12)$ or $C_2^2(24)$ chains can either combine to form chains of fused rings, comprising $R_2^2(24)$ rings, as in (I), or $R_4^4(38)$ rings, as in the adduct formed with bis(2-aminoethyl)amine, or they can combine to form sheets built from $R_4^4(38)$ rings, as in the adducts with 1,4-diazabicyclo[2.2.2]octane, piperazine, 1,2-diaminoethane, 1,2-bis(4-pyridyl)-

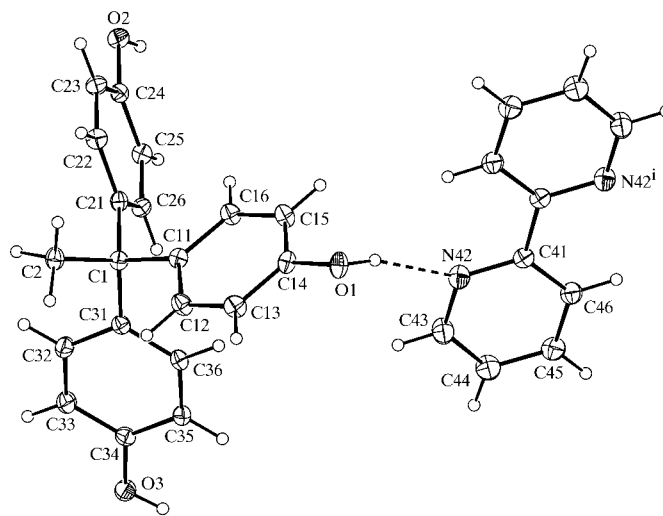


Figure 4
The molecular components of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level [symmetry code: (i) $1 - x, 1 - y, 1 - z$].

ethane or 2,2-bipyridyl, as described here for (II). A substructure comprising sheets of $R_4^4(38)$ rings can also be identified in the three-dimensional framework structure of 1,1,1-tris(4-hydroxyphenyl)ethane itself (Ferguson *et al.*, 1997), while in the methanol solvate formed with 1,10-phenanthroline, $C(12)$ chains formed by the tris(phenol) alone combine with $C(14)$ chains formed from alternating tris(phenol) and methanol units to generate sheets built from $R_6^6(42)$ rings (Ferguson *et al.*, 1999). Where sheets of $R_4^4(38)$ rings are formed, they are sometimes interwoven in pairs; this occurs in the tris(phenol) itself and in its adduct with 1,2-diaminoethane. Likewise, the sheets of $R_6^6(42)$ rings in the 1,10-phenanthroline adduct are pairwise interwoven. However, in the 1,2-bis(4-pyridyl)ethane adduct (Zakaria *et al.*, 2002), the sheets of $R_4^4(38)$ rings are generated wholly by translation and no mutual interweaving is possible.

In view of the striking difference in stoichiometry between the adduct formed with 4,4'-bipyridyl on the one hand, where the amine–tris(phenol) ratio is 3:2 (Bényei *et al.*, 1998), and, on the other hand, the adducts formed with 1,2-bis(4-pyridyl)ethane (Zakaria *et al.*, 2002) and 2,2'-bipyridyl, (II), where this ratio is 1:2, we have briefly re-investigated the crystallization characteristics of mixtures of the tris(phenol) with both 4,4'-bipyridyl and 1,2-bis(4-pyridyl)ethane. In each system, mixtures in methanol containing molar ratios of the amine to tris(phenol) of 1:2, 1:1, 3:2 and 2:1 (*i.e.* ratios of n :2

for $n = 1$ to 4) were crystallized at ambient temperature. Each such solution containing 4,4'-bipyridyl yielded the analytically pure 3:2 adduct, and each solution containing 1,2-bis(4-pyridyl)ethane yielded the analytically pure 1:2 adduct. It must be concluded that these compositions represent very well defined energy minima in their respective phase systems, but it is by no means clear why the compositions and structures of these stable phases should be so different.

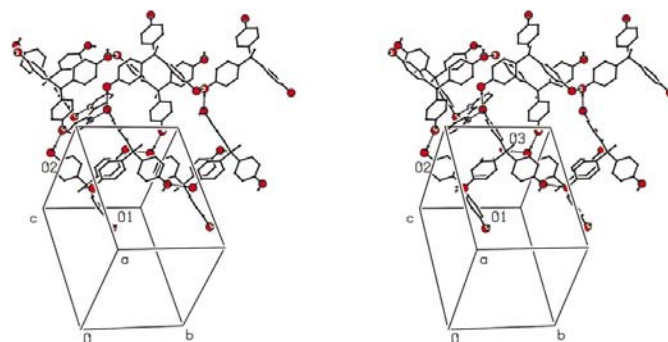


Figure 6

A stereoview of part of the crystal structure of (II), showing the linking of two $(10\bar{1})$ sheets by means of a diamine passing through an $R_4^4(38)$ ring, forming part of a third such sheet. For the sake of clarity, H atoms bonded to C atoms have been omitted.

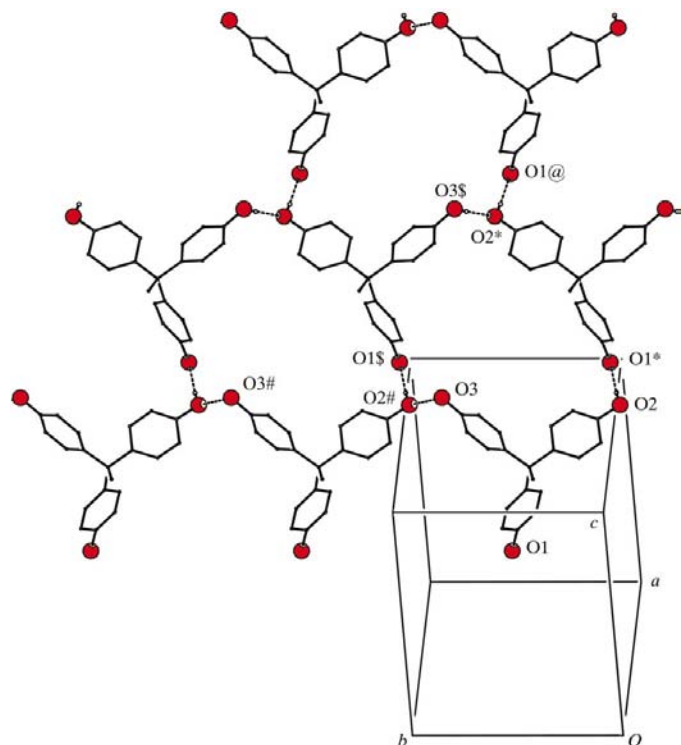


Figure 5

Part of the crystal structure of (II), showing the formation of a $(10\bar{1})$ sheet built from $R_4^4(38)$ rings. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*), hash (#), dollar sign (\$) or 'at' sign (@) are at the symmetry positions $(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$, $(x, 1 + y, z)$, $(\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z)$ and $(1 + x, y, 1 + z)$, respectively.

Experimental

For (I) and (II), equimolar quantities of the tris(phenol) and the appropriate diamine were separately dissolved in methanol. These component solutions were mixed and the mixtures set aside to crystallize, producing (I) and (II). Analysis for (I), found: C 75.3, H 7.3, N 3.9%; $C_{46}H_{52}N_2O_6$ requires C 75.8, H 7.2, N 3.8%. Analysis for (II), found: C 77.5, H 5.7, N 3.7%; $C_{50}H_{44}N_2O_6$ requires C 78.1, H 5.8, N 3.6%. Crystals suitable for single-crystal X-ray diffraction were selected directly from the analytical samples.

Compound (I)

Crystal data

$C_6H_{18}N_2^{2+} \cdot 2C_{20}H_{17}O_3^-$
 $M_r = 728.9$
 Triclinic, $P\bar{1}$
 $a = 6.47150$ (10) Å
 $b = 12.2447$ (3) Å
 $c = 13.0098$ (4) Å
 $\alpha = 77.2052$ (10)°
 $\beta = 76.6823$ (10)°
 $\gamma = 84.3491$ (16)°
 $V = 977.06$ (4) Å³

$Z = 1$
 $D_x = 1.239$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 8773 reflections
 $\theta = 2.6$ – 27.5°
 $\mu = 0.08$ mm⁻¹
 $T = 150$ (2) K
 Needle, colourless
 $0.32 \times 0.18 \times 0.16$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
 φ scans, and ω scans with κ offsets
 14 162 measured reflections
 4471 independent reflections
 3584 reflections with $I > 2\sigma(I)$

$R_{int} = 0.047$
 $\theta_{max} = 27.5^\circ$
 $h = -8 \rightarrow 8$
 $k = -15 \rightarrow 15$
 $l = -16 \rightarrow 16$

Table 1

Selected torsion angles (°) for (I).

C2—C1—C11—C12	−34.16 (15)	N4—C42—C43—C44	174.00 (11)
C2—C1—C21—C22	−45.77 (15)	C42—C43—C44—C44 ⁱ	−179.02 (14)
C2—C1—C31—C32	−58.15 (14)		

Symmetry code: (i) 1 − x, 1 − y, 1 − z.

Table 2

Hydrogen-bonding geometry (Å, °) for (I).

D—H...A	D—H	H...A	D...A	D—H...A
O2—H2...O1 ⁱ	0.84	1.74	2.5702 (12)	168
O3—H3...O1 ⁱⁱ	0.84	1.76	2.5855 (12)	169
N4—H4A...O2 ⁱⁱⁱ	0.91	1.92	2.8227 (14)	172
N4—H4B...O1 ^{iv}	0.91	1.79	2.6749 (14)	164
N4—H4C...O3	0.91	2.06	2.9159 (14)	156

Symmetry codes: (i) 1 − x, 1 − y, 2 − z; (ii) 1 − x, −y, 2 − z; (iii) 2 − x, 1 − y, 1 − z; (iv) x, y, z − 1.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.2476P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.109$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.05$	$\Delta\rho_{\max} = 0.22 \text{ e } \text{Å}^{-3}$
4471 reflections	$\Delta\rho_{\min} = -0.20 \text{ e } \text{Å}^{-3}$
249 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	(Sheldrick, 1997)
	Extinction coefficient: 0.027 (4)

Compound (II)

Crystal data

$\text{C}_{10}\text{H}_{18}\text{N}_2 \cdot 2\text{C}_{20}\text{H}_{18}\text{O}_3$	$D_x = 1.288 \text{ Mg m}^{-3}$
$M_r = 768.88$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 8697 reflections
$a = 12.1021 (4) \text{ Å}$	$\theta = 2.7\text{--}27.5^\circ$
$b = 10.7710 (4) \text{ Å}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 15.2142 (6) \text{ Å}$	$T = 150 (2) \text{ K}$
$\beta = 90.2800 (17)^\circ$	Block, colourless
$V = 1983.17 (13) \text{ Å}^3$	$0.32 \times 0.30 \times 0.16 \text{ mm}$
$Z = 2$	

Data collection

Nonius KappaCCD area-detector diffractometer	$R_{\text{int}} = 0.051$
φ scans, and ω scans with κ offsets	$\theta_{\max} = 27.5^\circ$
12 870 measured reflections	$h = -15 \rightarrow 15$
4511 independent reflections	$k = -13 \rightarrow 13$
3423 reflections with $I > 2\sigma(I)$	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.4013P]$
$R[F^2 > 2\sigma(F^2)] = 0.044$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.117$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.04$	$\Delta\rho_{\max} = 0.24 \text{ e } \text{Å}^{-3}$
4511 reflections	$\Delta\rho_{\min} = -0.19 \text{ e } \text{Å}^{-3}$
267 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	(Sheldrick, 1997)
	Extinction coefficient: 0.017 (3)

Compound (I) crystallized in the triclinic system; space group $P\bar{1}$ was assumed and confirmed by the analysis. For compound (II), space group $P2_1/n$ was uniquely assigned from the systematic absences. H atoms were treated as riding, with C—H = 0.95 (aromatic), 0.98 (CH₃) or 0.99 Å (CH₂), N—H = 0.91 Å and O—H = 0.84 Å.

Table 3

Selected torsion angles (°) for (II).

C2—C1—C11—C12	53.23 (16)	C2—C1—C31—C32	44.22 (17)
C2—C1—C21—C22	39.62 (17)		

Table 4

Hydrogen-bonding geometry (Å, °) for (II).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...N42	0.84	1.97	2.7833 (16)	163
O2—H2...O1 ⁱ	0.84	1.90	2.7303 (14)	173
O3—H3...O2 ⁱⁱ	0.84	1.90	2.7415 (15)	176

Symmetry codes: (i) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (ii) x, 1 + y, z.

For both compounds, data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2001); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the University of Toronto using a Nonius KappaCCD diffractometer purchased with funds from NSERC, Canada. CMZ thanks the Association of Commonwealth Universities for the award of a Commonwealth Fellowship 2000–2001.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1534). Services for accessing these data are described at the back of the journal.

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