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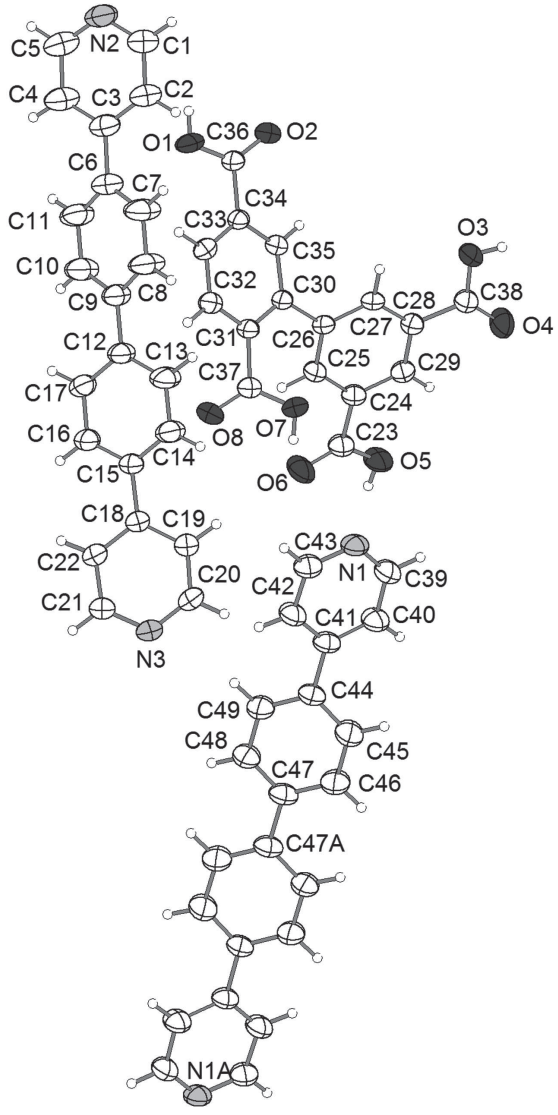
Crystal structure of biphenyl-2,3′,5,5′-tetracarboxylic acid – 4,4′-biphenyl-4,4′-diyldipyridine (3/2), $C_{49}H_{34}N_3O_8$



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Abstract

$C_{49}H_{34}N_3O_8$, triclinic, $P\bar{1}$ (no. 2), $a = 10.0660(3)$ Å, $b = 14.0604(2)$ Å, $c = 14.8204(3)$ Å, $\alpha = 106.697(1)°$, $\beta = 100.308(1)°$, $\gamma = 102.378(1)°$, $V = 1896.46(7)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0541$, $wR_{ref}(F^2) = 0.1258$, $T = 296(2)$ K.

CCDC no.: 1472352

Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	colourless, block, size 0.29×0.38×0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.95 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω scans
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	28129, 7050
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5130
$N(param)_{refined}$:	545
Programs:	Bruker programs [15], SHELX [16]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{iso}
H(1D)	2*i*	0.7379	1.0852	0.1180	0.112
H(3)	2*i*	1.0260	0.9132	0.6412	0.079
H(5)	2*i*	0.3060	0.5348	0.5335	0.083
H(7)	2*i*	0.5474	0.4772	0.1891	0.080
H(1)	2*i*	0.3743	−0.3286	0.7452	0.080
H(2)	2*i*	0.4733	−0.1606	0.7614	0.078
H(4)	2*i*	0.8409	−0.1794	0.8952	0.073
H(5A)	2*i*	0.7301	−0.3475	0.8796	0.075
H(7A)	2*i*	0.5564	0.0012	0.8232	0.080
H(8)	2*i*	0.6519	0.1666	0.8300	0.080
H(10)	2*i*	1.0315	0.1229	0.867	0.075

Table 2 (continued)

Atom	Site	x	y	z	U_{iso}
H(11)	2*i*	0.9359	−0.0429	0.8597	0.077
H(13)	2*i*	0.7364	0.2956	0.7926	0.081
H(14)	2*i*	0.8274	0.4625	0.8020	0.075
H(16)	2*i*	1.2068	0.4569	0.9312	0.065
H(17)	2*i*	1.1139	0.2899	0.9214	0.068
H(19)	2*i*	0.9299	0.5907	0.7672	0.057
H(20)	2*i*	1.0241	0.7571	0.7755	0.060
H(21)	2*i*	1.3519	0.7888	0.9778	0.069
H(22)	2*i*	1.2654	0.6234	0.9752	0.061
H(25)	2*i*	0.4297	0.6407	0.3051	0.045
H(27)	2*i*	0.8089	0.8459	0.4304	0.041
H(29)	2*i*	0.6198	0.6890	0.5822	0.046
H(32)	2*i*	0.5429	0.6647	0.0225	0.047
H(33)	2*i*	0.6319	0.8332	0.0328	0.047
H(35)	2*i*	0.7123	0.9210	0.3236	0.042
H(39)	2*i*	−0.2054	0.5484	0.3229	0.059
H(40)	2*i*	−0.0156	0.6750	0.3291	0.058
H(42)	2*i*	0.1299	0.7130	0.6090	0.057
H(43)	2*i*	−0.0651	0.5857	0.5951	0.060
H(45)	2*i*	0.1097	0.8473	0.3787	0.089
H(46)	2*i*	0.2998	0.9796	0.3931	0.093
H(48)	2*i*	0.5271	0.9093	0.5992	0.067
H(49)	2*i*	0.3368	0.7786	0.5856	0.066

Source of material

2,3′,5,5′-Biphenyltetracarboxylic acid (0.07 g, 0.2 mmol) and 4,4′-bis(pyrid-4-yl)biphenyl (0.06 g, 0.2 mmol) were added to water (10 mL) in a Teflon-lined stainless steel reactor. The mixture was heated at 393 K for 3 d, and then slowly cooled down to room temperature. Colorless block crystals of the title compound were obtained.

Experimental details

The hydrogen atoms were placed at calculated positions with the SHELX program (AFIX options: 43 and 147) [16].

Discussion

The synthetic development of coordination polymers (CPs) continues to attract much attention because of their intriguing structural topologies and potential applications in gas storage, separation, sensing and heterogeneous catalysis [1–8]. In the construction of CPs, multidentate organic ligands play a vital role since the structural integrity of these ligands in most cases remains unaltered throughout the The mixed-ligands strategy incorporating N-donor co-linkers with different lengths and rigidness/flexibility has been proved successful to create various CPs. With the

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*i*	−0.1544(2)	0.5550(1)	0.4581(2)	0.037(1)	0.039(1)	0.059(1)	0.0020(9)	0.015(1)	0.021(1)
N(2)	2*i*	0.5419(3)	−0.3531(2)	0.8124(2)	0.080(2)	0.035(1)	0.060(1)	0.009(1)	0.021(1)	0.021(1)
N(3)	2*i*	1.1957(2)	0.7886(2)	0.8778(2)	0.055(1)	0.037(1)	0.055(1)	0.002(1)	0.006(1)	0.025(1)
O(1)	2*i*	0.7053(2)	1.0235(1)	0.1084(1)	0.104(2)	0.038(1)	0.061(1)	−0.014(1)	−0.012(1)	0.0314(9)
O(2)	2*i*	0.8117(2)	1.0707(1)	0.2641(1)	0.062(1)	0.0351(9)	0.054(1)	−0.0075(8)	−0.0073(9)	0.0186(8)
O(3)	2*i*	0.9554(2)	0.8969(1)	0.5964(1)	0.044(1)	0.049(1)	0.051(1)	−0.0101(8)	−0.0061(8)	0.0226(8)
O(4)	2*i*	0.8615(2)	0.8023(1)	0.6791(1)	0.048(1)	0.073(1)	0.0385(9)	0.0038(9)	0.0061(8)	0.0260(9)
O(5)	2*i*	0.3803(2)	0.5737(1)	0.5349(1)	0.047(1)	0.060(1)	0.052(1)	−0.0135(9)	0.0131(8)	0.0277(9)
O(6)	2*i*	0.2860(2)	0.5235(2)	0.3751(1)	0.051(1)	0.077(1)	0.054(1)	−0.024(1)	−0.0022(9)	0.031(1)
O(7)	2*i*	0.5906(2)	0.5369(1)	0.1980(1)	0.057(1)	0.0311(8)	0.065(1)	0.0033(8)	−0.0014(9)	0.0244(8)
O(8)	2*i*	0.4008(2)	0.5284(1)	0.0908(1)	0.053(1)	0.0369(9)	0.073(1)	−0.0085(8)	−0.013(1)	0.0180(9)
C(1)	2*i*	0.4691(3)	−0.2980(2)	0.7771(2)	0.072(2)	0.041(2)	0.077(2)	0.000(1)	0.005(2)	0.025(1)
C(2)	2*i*	0.5287(3)	−0.1970(2)	0.7861(2)	0.076(2)	0.038(1)	0.077(2)	0.003(1)	0.009(2)	0.028(1)
C(3)	2*i*	0.6685(3)	−0.1495(2)	0.8307(2)	0.062(2)	0.035(1)	0.049(1)	0.010(1)	0.024(1)	0.018(1)
C(4)	2*i*	0.7451(3)	−0.2076(2)	0.8653(2)	0.064(2)	0.043(1)	0.085(2)	0.012(1)	0.032(2)	0.031(1)
C(5)	2*i*	0.6777(3)	−0.3091(2)	0.8553(2)	0.075(2)	0.043(2)	0.082(2)	0.019(2)	0.033(2)	0.031(1)
C(6)	2*i*	0.7346(3)	−0.0403(2)	0.8399(2)	0.060(2)	0.032(1)	0.060(2)	0.008(1)	0.023(1)	0.019(1)
C(7)	2*i*	0.6530(3)	0.0250(2)	0.8319(2)	0.048(2)	0.042(2)	0.119(3)	0.008(1)	0.030(2)	0.037(2)
C(8)	2*i*	0.7108(3)	0.1249(2)	0.8364(2)	0.048(2)	0.040(1)	0.126(3)	0.012(1)	0.028(2)	0.043(2)
C(9)	2*i*	0.8534(3)	0.1648(2)	0.8502(2)	0.044(1)	0.035(1)	0.064(2)	0.008(1)	0.017(1)	0.022(1)
C(10)	2*i*	0.9349(3)	0.0992(2)	0.8582(2)	0.045(2)	0.039(1)	0.108(2)	0.009(1)	0.020(2)	0.032(2)
C(11)	2*i*	0.8772(3)	−0.0010(2)	0.8535(2)	0.056(2)	0.036(1)	0.108(2)	0.014(1)	0.020(2)	0.034(2)
C(12)	2*i*	0.9140(2)	0.2727(2)	0.8557(2)	0.040(1)	0.034(1)	0.062(2)	0.006(1)	0.014(1)	0.022(1)
C(13)	2*i*	0.8320(3)	0.3276(2)	0.8208(2)	0.033(1)	0.047(2)	0.123(3)	0.001(1)	0.004(2)	0.047(2)
C(14)	2*i*	0.8866(3)	0.4281(2)	0.8262(2)	0.039(2)	0.044(1)	0.108(2)	0.007(1)	0.006(2)	0.042(2)
C(15)	2*i*	1.0276(2)	0.4786(2)	0.8668(2)	0.037(1)	0.032(1)	0.046(1)	0.003(1)	0.009(1)	0.018(1)
C(16)	2*i*	1.1113(3)	0.4248(2)	0.9027(2)	0.038(1)	0.041(1)	0.080(2)	0.001(1)	−0.001(1)	0.033(1)

Table 3 (continued)

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(17)	2*i*	1.0549(3)	0.3243(2)	0.8968(2)	0.044(2)	0.042(1)	0.085(2)	0.007(1)	0.002(1)	0.036(1)
C(18)	2*i*	1.0866(2)	0.5866(2)	0.8706(2)	0.040(1)	0.033(1)	0.041(1)	0.006(1)	0.009(1)	0.017(1)
C(19)	2*i*	1.0161(3)	0.6296(2)	0.8110(2)	0.046(2)	0.040(1)	0.051(1)	0.001(1)	0.001(1)	0.022(1)
C(20)	2*i*	1.0731(3)	0.7297(2)	0.8163(2)	0.053(2)	0.044(1)	0.058(2)	0.009(1)	0.007(1)	0.031(1)
C(21)	2*i*	1.2654(3)	0.7483(2)	0.9352(2)	0.054(2)	0.042(1)	0.065(2)	−0.005(1)	−0.005(1)	0.028(1)
C(22)	2*i*	1.2141(3)	0.6488(2)	0.9336(2)	0.048(2)	0.041(1)	0.062(2)	0.004(1)	0.001(1)	0.030(1)
C(23)	2*i*	0.3792(3)	0.5762(2)	0.4469(2)	0.040(1)	0.045(1)	0.049(2)	−0.002(1)	0.010(1)	0.024(1)
C(24)	2*i*	0.5056(2)	0.6530(2)	0.4441(2)	0.034(1)	0.037(1)	0.043(1)	0.002(1)	0.010(1)	0.019(1)
C(25)	2*i*	0.5069(2)	0.6745(2)	0.3588(2)	0.032(1)	0.036(1)	0.040(1)	−0.000(1)	0.004(1)	0.016(1)
C(26)	2*i*	0.6203(2)	0.7454(2)	0.3511(2)	0.032(1)	0.027(1)	0.038(1)	0.0038(9)	0.0078(9)	0.0151(9)
C(27)	2*i*	0.7327(2)	0.7969(2)	0.4333(2)	0.032(1)	0.028(1)	0.040(1)	0.0008(9)	0.008(1)	0.0150(9)
C(28)	2*i*	0.7329(2)	0.7763(2)	0.5195(2)	0.032(1)	0.033(1)	0.038(1)	0.0058(9)	0.0082(9)	0.0131(9)
C(29)	2*i*	0.6194(2)	0.7036(2)	0.5249(2)	0.038(1)	0.040(1)	0.040(1)	0.006(1)	0.011(1)	0.020(1)
C(30)	2*i*	0.6237(2)	0.7670(2)	0.2586(2)	0.027(1)	0.030(1)	0.038(1)	0.0027(9)	0.0046(9)	0.0163(9)
C(31)	2*i*	0.5734(2)	0.6897(2)	0.1661(2)	0.029(1)	0.028(1)	0.043(1)	0.0027(9)	0.006(1)	0.0158(9)
C(32)	2*i*	0.5771(2)	0.7162(2)	0.0834(2)	0.044(1)	0.032(1)	0.036(1)	0.004(1)	0.006(1)	0.0114(9)
C(33)	2*i*	0.6303(2)	0.8171(2)	0.0893(2)	0.044(1)	0.038(1)	0.038(1)	0.006(1)	0.008(1)	0.021(1)
C(34)	2*i*	0.6817(2)	0.8946(2)	0.1799(2)	0.027(1)	0.031(1)	0.044(1)	0.0034(9)	0.0066(9)	0.019(1)
C(35)	2*i*	0.6778(2)	0.8689(2)	0.2631(2)	0.033(1)	0.030(1)	0.037(1)	0.0017(9)	0.0047(9)	0.0124(9)
C(36)	2*i*	0.7392(2)	1.0052(2)	0.1878(2)	0.034(1)	0.033(1)	0.047(1)	0.002(1)	0.006(1)	0.020(1)
C(37)	2*i*	0.5131(2)	0.5767(2)	0.1488(2)	0.039(1)	0.031(1)	0.041(1)	0.003(1)	0.007(1)	0.014(1)
C(38)	2*i*	0.8548(2)	0.8259(2)	0.6068(2)	0.035(1)	0.038(1)	0.040(1)	0.007(1)	0.009(1)	0.015(1)
C(39)	2*i*	−0.1371(3)	0.5824(2)	0.3816(2)	0.039(1)	0.047(1)	0.052(2)	−0.003(1)	0.005(1)	0.018(1)
C(40)	2*i*	−0.0231(3)	0.6587(2)	0.3849(2)	0.044(2)	0.049(1)	0.049(1)	−0.003(1)	0.013(1)	0.020(1)
C(41)	2*i*	0.0801(2)	0.7110(2)	0.4707(2)	0.033(1)	0.034(1)	0.051(1)	0.004(1)	0.016(1)	0.014(1)
C(42)	2*i*	0.0625(3)	0.6812(2)	0.5496(2)	0.037(1)	0.047(1)	0.052(2)	−0.004(1)	0.009(1)	0.018(1)
C(43)	2*i*	−0.0551(3)	0.6041(2)	0.5407(2)	0.046(2)	0.049(1)	0.056(2)	0.002(1)	0.016(1)	0.025(1)
C(44)	2*i*	0.2032(2)	0.7963(2)	0.4794(2)	0.034(1)	0.036(1)	0.049(1)	0.001(1)	0.014(1)	0.013(1)
C(45)	2*i*	0.1944(3)	0.8588(2)	0.4231(2)	0.046(2)	0.078(2)	0.089(2)	−0.016(2)	−0.004(2)	0.051(2)
C(46)	2*i*	0.3092(3)	0.9383(2)	0.4315(2)	0.052(2)	0.077(2)	0.098(2)	−0.017(2)	−0.002(2)	0.060(2)
C(47)	2*i*	0.4377(2)	0.9577(2)	0.4959(2)	0.036(1)	0.036(1)	0.058(2)	0.002(1)	0.017(1)	0.015(1)
C(48)	2*i*	0.4435(3)	0.8970(2)	0.5534(2)	0.035(1)	0.055(2)	0.075(2)	−0.003(1)	0.007(1)	0.033(1)
C(49)	2*i*	0.3285(3)	0.8180(2)	0.5452(2)	0.041(2)	0.053(2)	0.071(2)	−0.001(1)	0.009(1)	0.035(1)

assembly process, which helps to realize predesigned structural topology. Different factors such as coordination geometry of the metal atom, flexibility of the ligand, solvent system, pH value, temperature, template, ligand-to-metal ratios and counter ions can potentially influence the construction of the coordination networks. Subtle modification to any of these parameters can significantly alter the structure of the complexes produced, generating, for example, classical monomeric mononuclear species, linear chain polymers or extended three-dimensional networks. In particular, different conformations of the ligand often play an important role in the self-assembly processes to form metal-organic coordination polymers with different structures. The organic carboxylate ligands have been proven to be good candidates for the construction of CPs [9–11]. For example, polycarboxylates present versatile coordination modes, can yield predetermined networks and have been widely utilized to construct coordination polymers [12–14]. Mixed multicarboxylate and nitrogen donor linkers have been widely utilized to construct CPs. aim of preparing new materials with interesting structural topologies and physicochemical properties, we chose 2,3′,5,5′-biphenyltetracarboxylic acid and 4,4′-bis(pyrid-4-yl)biphenyl to construct a new compound. In the course of this study the pure organic title compound was surprisingly obtained.

The crystal structure of the title compound comprises one 2,3′,5,5′-biphenyltetracarboxylic acid (bita) ligand and one and a half 4,4′-bis(pyrid-4-yl)biphenyl (bpp) ligand per asymmetric unit (*cf*. the figure). All of the carboxylate groups of the acid remain protonated. Hydrogen bonding and intermolecular weak interactions play an important role in the structure of the title compound. A short intermolecular distance between the adjacent bita O atoms with an O1—N3B distance of 2.551(2) Å, O3—O2B distance of 2.692(2) Å, O5—N1C distance of 2.624(2) Å and O7—N2D distance of 2.590(3) Å are found, corresponding to a strong intermolecular hydrogen

bond. Bita and bpp molecules are assembled by hydrogen bonds to give a three-dimensional supramolecular structure.

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