Crystal structure of 4,4'-(propane-1,3-diyl)bis(pyridin-1-ium)[4-(3,5-dicarboxyphenoxy)-3-nitrobenzoate], $(C_{13}H_{14}N_2)(C_{15}H_9NO_9)_2$, $C_{43}H_{32}N_4O_{18}$

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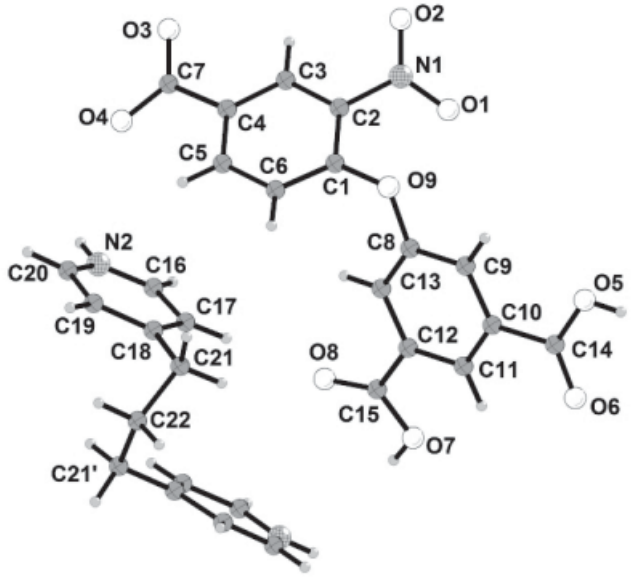

Abstract

$C_{43}H_{32}N_4O_{18}$, monoclinic, *C*2/*c* (o. 15), a = 22.409(3) Å, b = 8.280(1) Å, c = 22.146(3) Å, β = 105.242(1)°, V = 3964.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.0482, $wR_{ref}(F^2)$ = 0.1411, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.19×0.29×0.36 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.19 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14574, 3695
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2804
$N(param)_{refined}$:	296
Programs:	SHELX [6]

Source of material

The mixture of 5-(4-carboxy-2-nitrophenoxy)isophthalic acid (34.7 mg, 0.1 mmol), 1,3-di(4-pyridyl)propane (19.8 mg, 0.1 mmol) and NaOH (8.2 mg, 0.2 mmol) were added to a H_2O solution (15 ml) in a Teflon-lined stainless steel reactor. The mixture was heated at 433 K for 3 days, and then slowly cooled down to room temperature over 24 hours. Yellow block-shaped crystals of the title compound were obtained. **Elemental Analysis** found (/%): C,57.81; H,3.60; N,6.22; calcd. for $C_{43}H_{32}N_4O_{18}$ (/%): C,57.85; H,3.61; N,6.28.

Discussion

The design and synthesis of supramolecular coordination polymeric networks, especially those constructed by hydrogen bonding and intermolecular weak interactions with ligands containing O or N donors have been a field of rapid growth due to their special physical properties and potential application in functional material [1-5]. We report here the synthesis and structure of supramolecular hydrogen bonded polymeric networks of an organic salt. The crystal structure of the title compound comprises one half of a 4,4'-(propane-1,3-diyl)bis(pyridin-1-ium) cation and one 4-(3,5-dicarboxyphenoxy)-3-nitrobenzoate per asymmetric unit. The two N atoms of the cation are protonated, one carboxylate group is deprotonated (C7). In the complex, there exist inter-molecular hydrogen bonds: d(O7–H7···O3) = 2.608(2) Å and d(O5–H5A···O4) = 2.513(2) Å between two carboxyl, and hydrogen bonds between carboxylate oxygen atoms and nitrogen atoms of 1,3-di(4-pyridyl)propane: d(N2–H2···O3) = 2.968(4) Å and d(N2–H2···O6) = 2.822(3) Å, respectively. A three-dimensional supramolecular network is formed. The hydrogen bonds play an important role for the self-assembly and the stabilisation of the supramolecular structure.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(3)	8*f*		0.6932	0.0635	0.5118	0.044
H(5)	8*f*		0.5823	0.3638	0.5735	0.044
H(6)	8*f*		0.6614	0.4235	0.6604	0.047
H(9)	8*f*		0.8552	0.3595	0.7890	0.039
H(11)	8*f*		0.7733	0.1893	0.9174	0.044
H(13)	8*f*		0.6829	0.1777	0.7347	0.042
H(16)	8*f*		0.5015	0.0801	0.6397	0.080
H(17)	8*f*		0.5571	0.2528	0.7207	0.082
H(19)	8*f*		0.4644	0.6173	0.6189	0.069
H(20)	8*f*		0.4189	0.4404	0.5446	0.084
H(21A)	8*f*		0.5719	0.6538	0.7093	0.113
H(21B)	8*f*		0.5736	0.5273	0.7624	0.113
H(22A)	8*f*	0.5	0.4778	0.7527	0.7162	0.157
H(22B)	8*f*	0.5	0.5222	0.7527	0.7838	0.157
H(2)	8*f*		0.4396	0.1880	0.5592	0.083
H(5A)	8*f*		0.9597	0.3396	0.9248	0.076
H(7)	8*f*		0.6449	–0.0010	0.9059	0.084

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*f*	0.7240(1)	0.2697(3)	0.64145(9)	0.030(1)	0.049(1)	0.023(1)	–0.010(1)	0.0010(8)	0.0041(9)
C(2)	8*f*	0.7329(1)	0.1643(3)	0.5959(1)	0.024(1)	0.050(1)	0.032(1)	–0.000(1)	0.0018(9)	0.005(1)
C(3)	8*f*	0.6861(1)	0.1324(3)	0.5424(1)	0.029(1)	0.051(1)	0.028(1)	0.002(1)	0.0034(9)	–0.004(1)
C(4)	8*f*	0.62863(9)	0.2038(3)	0.53465(9)	0.025(1)	0.048(1)	0.025(1)	–0.0004(9)	0.0024(8)	0.0018(9)
C(5)	8*f*	0.6203(1)	0.3129(3)	0.5791(1)	0.028(1)	0.048(1)	0.032(1)	0.003(1)	0.0054(9)	0.001(1)
C(6)	8*f*	0.6675(1)	0.3477(3)	0.6316(1)	0.037(1)	0.050(1)	0.029(1)	–0.004(1)	0.0071(9)	–0.005(1)
C(7)	8*f*	0.57504(9)	0.1605(3)	0.48007(9)	0.028(1)	0.051(1)	0.026(1)	0.004(1)	0.0016(9)	–0.001(1)
C(8)	8*f*	0.7684(1)	0.2738(3)	0.75234(9)	0.030(1)	0.042(1)	0.024(1)	–0.0014(9)	0.0018(8)	0.0013(9)
C(9)	8*f*	0.82131(9)	0.3124(3)	0.7991(1)	0.026(1)	0.040(1)	0.030(1)	–0.0017(9)	0.0038(8)	–0.0005(9)
C(10)	8*f*	0.82281(9)	0.2797(3)	0.86090(9)	0.027(1)	0.042(1)	0.026(1)	0.0025(9)	–0.0006(8)	–0.0014(9)
C(11)	8*f*	0.7721(1)	0.2104(3)	0.8759(1)	0.031(1)	0.051(1)	0.027(1)	0.002(1)	0.0037(9)	0.002(1)
C(12)	8*f*	0.7197(1)	0.1727(3)	0.8286(1)	0.028(1)	0.044(1)	0.033(1)	0.0017(9)	0.0058(9)	0.003(1)
C(13)	8*f*	0.7179(1)	0.2038(3)	0.7665(1)	0.026(1)	0.046(1)	0.029(1)	–0.0038(9)	–0.0012(8)	–0.000(1)
C(14)	8*f*	0.8803(1)	0.3054(3)	0.9129(1)	0.032(1)	0.047(1)	0.028(1)	0.001(1)	0.0012(9)	0.000(1)
C(15)	8*f*	0.6649(1)	0.0922(3)	0.8422(1)	0.031(1)	0.054(2)	0.035(1)	0.002(1)	0.0064(9)	0.007(1)
C(16)	8*f*	0.4957(2)	0.1913(4)	0.6366(2)	0.078(2)	0.053(2)	0.084(2)	0.002(2)	0.049(2)	–0.004(2)
C(17)	8*f*	0.5287(1)	0.2944(5)	0.6858(1)	0.046(2)	0.106(3)	0.058(2)	0.018(2)	0.024(1)	0.038(2)
C(18)	8*f*	0.5167(1)	0.4615(3)	0.6793(1)	0.037(1)	0.069(2)	0.032(1)	–0.011(1)	0.008(1)	0.007(1)
C(19)	8*f*	0.4742(1)	0.5083(4)	0.6250(1)	0.060(2)	0.071(2)	0.038(1)	0.006(1)	0.004(1)	–0.001(1)
C(20)	8*f*	0.4467(2)	0.4038(5)	0.5810(1)	0.055(2)	0.100(3)	0.050(2)	0.006(2)	0.004(1)	–0.014(2)
C(21)	8*f*	0.5466(2)	0.5826(5)	0.7270(1)	0.111(3)	0.127(3)	0.040(2)	–0.083(3)	0.014(2)	–0.007(2)
C(22)	4*e*	$\frac{1}{2}$	0.6836(6)	$\frac{3}{4}$	0.29(1)	0.045(3)	0.045(3)	0	0.016(4)	0
N(1)	8*f*	0.79214(9)	0.0813(3)	0.6028(1)	0.028(1)	0.071(2)	0.054(1)	0.006(1)	–0.004(1)	–0.001(1)
N(2)	8*f*	0.4583(1)	0.2525(4)	0.5885(1)	0.054(2)	0.096(2)	0.060(2)	–0.011(2)	0.020(1)	–0.018(2)
O(1)	8*f*	0.8217(1)	0.0432(3)	0.6553(1)	0.057(1)	0.102(2)	0.073(1)	0.024(1)	–0.025(1)	0.000(1)
O(2)	8*f*	0.8068(1)	0.0466(4)	0.5560(1)	0.055(1)	0.180(3)	0.075(2)	0.048(2)	0.019(1)	–0.009(2)
O(3)	8*f*	0.58379(7)	0.0873(2)	0.43417(7)	0.0314(9)	0.085(1)	0.0350(9)	0.0066(8)	0.0008(7)	–0.0192(9)
O(4)	8*f*	0.52271(7)	0.2036(3)	0.48462(7)	0.0241(8)	0.093(2)	0.0373(9)	0.0098(8)	0.0002(7)	–0.0152(9)
O(5)	8*f*	0.92910(7)	0.3434(3)	0.89479(7)	0.0285(8)	0.087(1)	0.0312(8)	–0.0098(9)	–0.0022(7)	0.0035(9)
O(6)	8*f*	0.87971(8)	0.2884(3)	0.96703(7)	0.0378(9)	0.103(2)	0.0257(9)	–0.006(1)	–0.0012(7)	0.0043(9)
O(7)	8*f*	0.67647(8)	0.0386(3)	0.89995(7)	0.0358(9)	0.095(2)	0.0354(9)	–0.0109(9)	0.0060(7)	0.0173(9)
O(8)	8*f*	0.61579(7)	0.0777(3)	0.80383(8)	0.0265(8)	0.089(1)	0.046(1)	–0.0075(9)	–0.0010(7)	0.019(1)
O(9)	8*f*	0.77260(7)	0.3068(2)	0.69212(7)	0.0329(8)	0.069(1)	0.0234(7)	–0.0178(8)	0.0005(6)	–0.0001(7)

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