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4-(4-Aminophenoxy)anilinium 2-hydroxy-2,2-diphenylacetate

Qiuxia Han and Jie Li*

Basic Experiment Teaching Center, Henan University, Kaifeng 475001, People's Republic of China
Correspondence e-mail: lijiehd@163.com

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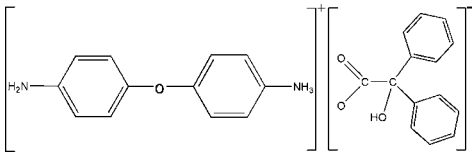
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; R factor = 0.058; wR factor = 0.154; data-to-parameter ratio = 8.3.

The asymmetric unit of the title salt, $C_{12}H_{13}N_2O^+\cdot C_{14}H_{11}O_3^-$, contains two cations and two anions, in which cation–anion pairs are linked together by an N—H···O hydrogen bond. An intramolecular O—H···O hydrogen bond occurs within each anion and forms an S(5) ring. These component ions are organized through further hydrogen bonds into layers parallel to (001).

Related literature

For related literature, see: Li (2007).



Experimental

Crystal data

$C_{12}H_{13}N_2O^+\cdot C_{14}H_{11}O_3^-$
M_r = 428.47
Monoclinic, $P2_1$
a = 14.619 (6) Å
b = 6.442 (3) Å
c = 24.938 (10) Å
β = 105.499 (7)°
V = 2263.2 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.15 × 0.10 × 0.08 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.987, T_{max} = 0.993
12318 measured reflections
4840 independent reflections
2576 reflections with $I > 2\sigma(I)$
R_{int} = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.154
S = 0.84
4840 reflections
582 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6A···O4	0.82	2.10	2.576 (5)	117
O3—H3···O2	0.82	2.08	2.589 (5)	120
N4—H4C···O5^{i}	0.89	1.81	2.683 (5)	168
N4—H4B···O1^{ii}	0.89	1.89	2.761 (5)	166
N4—H4A···O2	0.89	1.90	2.790 (5)	179
N3—H3A···O6^{iii}	0.86	2.33	3.076 (6)	146
N1—H1C···O5	0.89	1.89	2.777 (5)	174
N1—H1B···O4^{iii}	0.89	1.88	2.762 (5)	173
N1—H1A···O1^{iv}	0.89	1.81	2.685 (5)	165

Symmetry codes: (i) $x-1, y, z$; (ii) $x, y-1, z$; (iii) $x, y+1, z$; (iv) $x+1, y, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2584).

References

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supplementary materials

Acta Cryst. (2007). E**63**, o4380 [**doi:10.1107/S1600536807051057**]

4-(4-Aminophenoxy)anilinium 2-hydroxy-2,2-diphenylacetate

Q. Han and J. Li

Comment

This work continues our previous synthetic and structural studies of the hydrogen bonding interactions between the 2-hydroxy-2,2-diphenylacetate anion and substituted pyridinium cations (Li, 2007).

Compound (I) is composed of two pairs of 4,4'-oxydianilinium cations and 2-hydroxy-2,2-diphenylacetate anions, in which each cation interacts with its neighbouring anion by an intermolecular N—H···O hydrogen bond (Fig. 1 and Table 1). In addition, an intramolecular O—H···O bond also occurs in each anion and constructs an S(5) ring. Moreover, adjacent ion pairs are organized into an infinite two-dimensional network running parallel to the plane (001) by further intermolecular N—H···O hydrogen bonds (Fig. 2).

Experimental

A 5-ml ethanol solution of 4,4'-oxydianiline (1.0 mmol, 0.20 g) added to 20 ml hot aqueous solution of 2-hydroxy-2,2-diphenylacetic acid (1.0 mmol, 0.23 g) and the mixture was stirred for 15 minutes at 373 K. Then the solution was filtered, and the filtrate was kept at the room temperature. After 5 d, colorless crystals of (I) were obtained.

Refinement

Anomalous dispersion was negligible and Friedel pairs were merged before refinement.

All the H atoms were placed in calculated positions, with C—H = 0.93 Å, O—H = 0.82 Å, N—H = 0.86 and 0.89 Å for $–NH_2$and $–NH_3$ H atoms, respectively, and were refined as riding with U_{iso}(H) = 1.2U_{eq}(C or $N_{amine\ group}$) or 1.5U_{eq}(O or $N_{protonated}$).

Figures

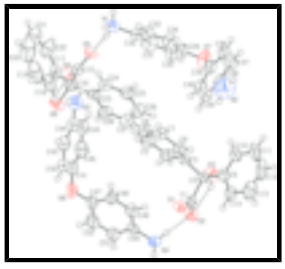


Fig. 1. The molecular structure of (I). Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

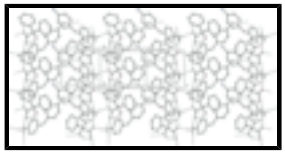


Fig. 2. Par of an (001) hydrogen bonded sheet in (I) Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

4-(4-Aminophenoxy)anilinium 2-hydroxy-2,2-diphenylacetate

Crystal data

$C_{12}H_{13}N_2O^+ \cdot C_{14}H_{11}O_3^-$	$F_{000} = 904$
$M_r = 428.47$	$D_x = 1.258$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 5100 reflections
$a = 14.619$ (6) Å	$\theta = 2.1$–28.3º
$b = 6.442$ (3) Å	$\mu = 0.09$ mm^{-1}
$c = 24.938$ (10) Å	$T = 298$ (2) K
$\beta = 105.499$ (7)º	Blade, colourless
$V = 2263.2$ (16) Å^3	0.15 × 0.10 × 0.08 mm
$Z = 4$	

Data collection

Bruker SMART APEX CCD diffractometer	4840 independent reflections
Radiation source: fine-focus sealed tube	2576 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.068$
$T = 298$(2) K	$\theta_{max} = 26.0$º
ω scans	$\theta_{min} = 1.9$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$h = -18 \rightarrow 17$
$T_{min} = 0.987$, $T_{max} = 0.993$	$k = -7 \rightarrow 4$
12318 measured reflections	$l = -29 \rightarrow 30$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.058$	H-atom parameters constrained
$wR(F^2) = 0.154$	$w = 1/[\sigma^2(F_o^2) + (0.0797P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.84$	$(\Delta/\sigma)_{max} < 0.001$
4840 reflections	$\Delta\rho_{max} = 0.20$ e Å^{-3}
582 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2825 (3)	0.3643 (8)	0.32307 (18)	0.0382 (11)
C2	0.3730 (3)	0.4688 (7)	0.35928 (17)	0.0395 (11)
C3	0.4293 (3)	0.5421 (7)	0.31939 (19)	0.0397 (12)
C4	0.4765 (3)	0.3916 (9)	0.2954 (2)	0.0556 (14)
H4	0.4791	0.2547	0.3076	0.067*
C5	0.5184 (4)	0.4447 (10)	0.2545 (2)	0.0651 (16)
H5	0.5490	0.3437	0.2389	0.078*
C6	0.5156 (4)	0.6472 (11)	0.2360 (2)	0.0630 (16)
H6	0.5425	0.6828	0.2074	0.076*
C7	0.4726 (3)	0.7946 (10)	0.2603 (2)	0.0580 (15)
H7	0.4730	0.9321	0.2490	0.070*
C8	0.4288 (3)	0.7449 (8)	0.30121 (19)	0.0460 (12)
H8	0.3989	0.8477	0.3165	0.055*
C9	0.3496 (4)	0.6422 (7)	0.39551 (18)	0.0448 (12)
C10	0.4236 (4)	0.7485 (8)	0.42978 (19)	0.0568 (14)
H10	0.4855	0.7172	0.4292	0.068*
C11	0.4081 (6)	0.9000 (10)	0.4648 (2)	0.082 (2)
H11	0.4594	0.9669	0.4887	0.098*
C12	0.3177 (8)	0.9535 (11)	0.4649 (3)	0.095 (3)
H12A	0.3073	1.0618	0.4873	0.114*
C13	0.2429 (6)	0.8474 (10)	0.4322 (3)	0.084 (2)
H13A	0.1814	0.8793	0.4332	0.101*
C14	0.2584 (4)	0.6913 (8)	0.3970 (2)	0.0576 (15)
H14	0.2072	0.6198	0.3745	0.069*
C15	0.9588 (3)	−0.1653 (8)	0.18057 (19)	0.0411 (12)
C16	0.8636 (3)	−0.0693 (7)	0.14776 (18)	0.0423 (12)
C17	0.8775 (4)	0.0975 (8)	0.10815 (19)	0.0493 (13)
C18	0.7976 (5)	0.2000 (9)	0.0766 (2)	0.0727 (18)
H18	0.7385	0.1672	0.0816	0.087*
C19	0.8036 (7)	0.3481 (12)	0.0384 (3)	0.102 (3)
H19	0.7494	0.4161	0.0179	0.123*
C20	0.8907 (9)	0.3945 (13)	0.0308 (3)	0.121 (4)
H20A	0.8948	0.4956	0.0050	0.145*

C21	0.9723 (7)	0.2965 (14)	0.0602 (3)	0.113 (3)
H21A	1.0308	0.3286	0.0542	0.136*
C22	0.9648 (5)	0.1483 (10)	0.0992 (3)	0.0794 (19)
H22	1.0193	0.0814	0.1198	0.095*
C23	0.8138 (3)	0.0002 (7)	0.19063 (19)	0.0403 (12)
C24	0.8126 (3)	0.2028 (8)	0.2080 (2)	0.0467 (13)
H24	0.8383	0.3060	0.1904	0.056*
C25	0.7739 (4)	0.2567 (9)	0.2512 (2)	0.0600 (15)
H25	0.7739	0.3948	0.2621	0.072*
C26	0.7355 (4)	0.1064 (11)	0.2779 (2)	0.0666 (16)
H26	0.7093	0.1414	0.3068	0.080*
C27	0.7365 (4)	−0.0954 (10)	0.2609 (3)	0.0679 (17)
H27	0.7110	−0.1984	0.2788	0.081*
C28	0.7747 (4)	−0.1489 (9)	0.2179 (2)	0.0612 (15)
H28	0.7742	−0.2870	0.2070	0.073*
C29	0.9439 (4)	0.4522 (10)	0.3931 (2)	0.0604 (15)
C30	0.9793 (4)	0.2606 (10)	0.3844 (2)	0.0672 (17)
H30	0.9791	0.1513	0.4087	0.081*
C31	1.0150 (4)	0.2339 (9)	0.3388 (2)	0.0645 (16)
H31	1.0415	0.1068	0.3334	0.077*
C32	1.0121 (3)	0.3899 (8)	0.3017 (2)	0.0423 (12)
C33	0.9784 (3)	0.5809 (8)	0.3111 (2)	0.0527 (14)
H33	0.9782	0.6897	0.2866	0.063*
C34	0.9447 (4)	0.6110 (9)	0.3571 (2)	0.0588 (15)
H34	0.9222	0.7410	0.3636	0.071*
C35	0.8408 (4)	0.6235 (10)	0.4356 (2)	0.0581 (15)
C36	0.8631 (4)	0.8080 (11)	0.4649 (2)	0.0716 (18)
H36	0.9241	0.8303	0.4876	0.086*
C37	0.7951 (4)	0.9557 (12)	0.4603 (2)	0.0790 (19)
H37	0.8101	1.0779	0.4806	0.095*
C38	0.7034 (4)	0.9295 (11)	0.4260 (2)	0.0700 (18)
C39	0.6839 (4)	0.7431 (12)	0.3972 (2)	0.0719 (19)
H39	0.6234	0.7199	0.3741	0.086*
C40	0.7516 (4)	0.5932 (10)	0.4020 (2)	0.0651 (16)
H40	0.7368	0.4698	0.3823	0.078*
C41	0.4180 (4)	0.2114 (9)	0.0745 (2)	0.0587 (15)
C42	0.3848 (4)	0.3786 (10)	0.0423 (2)	0.0637 (16)
H42	0.3231	0.3793	0.0194	0.076*
C43	0.4434 (4)	0.5486 (10)	0.0436 (2)	0.0638 (16)
H43	0.4211	0.6628	0.0211	0.077*
C44	0.5346 (4)	0.5498 (10)	0.0780 (2)	0.0583 (15)
C45	0.5667 (4)	0.3820 (11)	0.1109 (2)	0.0656 (16)
H45	0.6274	0.3830	0.1352	0.079*
C46	0.5084 (5)	0.2087 (10)	0.1081 (2)	0.0687 (17)
H46	0.5311	0.0913	0.1292	0.082*
C47	0.3195 (4)	0.0073 (9)	0.1157 (2)	0.0606 (16)
C48	0.3129 (4)	0.1615 (8)	0.1527 (2)	0.0537 (14)
H48	0.3351	0.2940	0.1483	0.064*
C49	0.2740 (4)	0.1215 (8)	0.1956 (2)	0.0518 (13)

H49	0.2703	0.2261	0.2206	0.062*
C50	0.2403 (3)	−0.0742 (7)	0.20173 (19)	0.0416 (12)
C51	0.2414 (4)	−0.2238 (9)	0.1636 (2)	0.0589 (15)
H51	0.2153	−0.3534	0.1666	0.071*
C52	0.2815 (5)	−0.1844 (9)	0.1201 (2)	0.0701 (17)
H52	0.2826	−0.2873	0.0941	0.084*
N1	1.0460 (3)	0.3568 (6)	0.25262 (16)	0.0466 (10)
H1A	1.1086	0.3742	0.2614	0.070*
H1B	1.0184	0.4476	0.2264	0.070*
H1C	1.0317	0.2284	0.2400	0.070*
N2	0.6361 (4)	1.0889 (11)	0.4196 (3)	0.119 (2)
H2A	0.6511	1.2045	0.4370	0.143*
H2B	0.5797	1.0711	0.3983	0.143*
N3	0.5907 (4)	0.7274 (9)	0.0817 (2)	0.0833 (16)
H3A	0.6461	0.7316	0.1049	0.100*
H3B	0.5699	0.8327	0.0607	0.100*
N4	0.2020 (2)	−0.1196 (6)	0.24908 (15)	0.0420 (10)
H4A	0.2232	−0.0258	0.2757	0.063*
H4B	0.2207	−0.2456	0.2622	0.063*
H4C	0.1389	−0.1152	0.2381	0.063*
O1	0.2287 (2)	0.4743 (5)	0.28538 (12)	0.0421 (8)
O2	0.2690 (2)	0.1789 (5)	0.33123 (14)	0.0567 (9)
O3	0.4277 (2)	0.3158 (6)	0.39477 (15)	0.0605 (10)
H3	0.3970	0.2079	0.3921	0.091*
O4	0.9744 (3)	−0.3488 (5)	0.17179 (14)	0.0592 (10)
O5	1.0144 (2)	−0.0501 (5)	0.21549 (13)	0.0469 (8)
O6	0.8082 (2)	−0.2314 (6)	0.11509 (16)	0.0624 (10)
H6A	0.8432	−0.3276	0.1119	0.094*
O7	0.9104 (3)	0.4712 (7)	0.43911 (15)	0.0776 (12)
O8	0.3592 (3)	0.0372 (7)	0.07231 (16)	0.0867 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.044 (3)	0.036 (3)	0.039 (3)	0.001 (2)	0.019 (2)	−0.002 (2)
C2	0.043 (3)	0.031 (3)	0.040 (2)	−0.005 (2)	0.003 (2)	0.007 (2)
C3	0.034 (3)	0.038 (3)	0.044 (3)	−0.002 (2)	0.005 (2)	0.001 (2)
C4	0.051 (3)	0.043 (3)	0.074 (4)	−0.002 (3)	0.020 (3)	−0.001 (3)
C5	0.055 (4)	0.066 (5)	0.080 (4)	0.004 (3)	0.027 (3)	−0.007 (4)
C6	0.048 (3)	0.082 (5)	0.065 (4)	0.001 (3)	0.027 (3)	−0.001 (3)
C7	0.052 (3)	0.059 (4)	0.066 (3)	−0.003 (3)	0.021 (3)	0.008 (3)
C8	0.039 (3)	0.048 (3)	0.049 (3)	−0.001 (2)	0.007 (2)	−0.001 (3)
C9	0.064 (3)	0.033 (3)	0.036 (3)	−0.011 (3)	0.011 (3)	0.003 (2)
C10	0.079 (4)	0.046 (4)	0.040 (3)	−0.015 (3)	0.007 (3)	0.000 (3)
C11	0.146 (7)	0.058 (5)	0.041 (3)	−0.025 (4)	0.025 (4)	−0.014 (3)
C12	0.191 (9)	0.053 (4)	0.057 (4)	0.000 (5)	0.063 (5)	−0.014 (4)
C13	0.129 (6)	0.059 (5)	0.082 (4)	0.009 (4)	0.059 (5)	−0.009 (4)
C14	0.082 (4)	0.044 (3)	0.053 (3)	−0.006 (3)	0.030 (3)	−0.010 (3)

C15	0.050 (3)	0.031 (3)	0.044 (3)	0.002 (2)	0.016 (2)	−0.001 (2)
C16	0.045 (3)	0.036 (3)	0.044 (3)	−0.002 (2)	0.008 (2)	0.000 (2)
C17	0.071 (4)	0.035 (3)	0.038 (3)	0.009 (3)	0.008 (3)	0.000 (2)
C18	0.101 (5)	0.055 (4)	0.049 (3)	0.012 (4)	−0.004 (3)	−0.001 (3)
C19	0.166 (8)	0.071 (6)	0.055 (4)	0.026 (6)	0.004 (5)	0.009 (4)
C20	0.228 (12)	0.065 (6)	0.076 (5)	0.018 (7)	0.053 (7)	0.029 (4)
C21	0.166 (8)	0.093 (6)	0.103 (6)	−0.001 (6)	0.076 (6)	0.016 (5)
C22	0.100 (5)	0.073 (5)	0.076 (4)	0.012 (4)	0.042 (4)	0.016 (4)
C23	0.031 (3)	0.036 (3)	0.052 (3)	0.004 (2)	0.008 (2)	0.001 (2)
C24	0.040 (3)	0.044 (3)	0.053 (3)	−0.004 (2)	0.008 (2)	0.000 (3)
C25	0.060 (4)	0.054 (4)	0.067 (4)	−0.002 (3)	0.018 (3)	−0.006 (3)
C26	0.058 (4)	0.077 (5)	0.071 (4)	0.005 (3)	0.028 (3)	−0.004 (4)
C27	0.061 (4)	0.065 (5)	0.085 (4)	−0.004 (3)	0.034 (3)	0.013 (4)
C28	0.060 (4)	0.050 (4)	0.076 (4)	−0.009 (3)	0.024 (3)	−0.002 (3)
C29	0.064 (4)	0.070 (4)	0.050 (3)	0.006 (3)	0.020 (3)	0.007 (3)
C30	0.093 (5)	0.049 (4)	0.060 (4)	0.015 (3)	0.020 (3)	0.021 (3)
C31	0.073 (4)	0.047 (4)	0.073 (4)	0.019 (3)	0.019 (3)	0.012 (3)
C32	0.036 (3)	0.035 (3)	0.054 (3)	0.001 (2)	0.007 (2)	0.002 (3)
C33	0.059 (3)	0.035 (3)	0.066 (4)	0.001 (3)	0.020 (3)	0.006 (3)
C34	0.073 (4)	0.041 (3)	0.067 (4)	0.007 (3)	0.027 (3)	−0.001 (3)
C35	0.060 (4)	0.071 (4)	0.046 (3)	0.005 (3)	0.018 (3)	0.006 (3)
C36	0.051 (4)	0.092 (5)	0.065 (4)	0.003 (4)	0.005 (3)	−0.017 (4)
C37	0.074 (5)	0.081 (5)	0.079 (4)	0.001 (4)	0.015 (4)	−0.029 (4)
C38	0.062 (4)	0.090 (5)	0.065 (4)	0.015 (4)	0.028 (3)	0.003 (4)
C39	0.056 (4)	0.103 (6)	0.057 (4)	−0.019 (4)	0.015 (3)	−0.019 (4)
C40	0.074 (4)	0.070 (4)	0.059 (4)	−0.010 (4)	0.032 (3)	−0.008 (3)
C41	0.081 (4)	0.058 (4)	0.041 (3)	−0.011 (3)	0.025 (3)	−0.007 (3)
C42	0.061 (4)	0.081 (5)	0.048 (3)	−0.009 (3)	0.013 (3)	−0.004 (3)
C43	0.069 (4)	0.071 (4)	0.050 (3)	−0.001 (3)	0.013 (3)	0.012 (3)
C44	0.065 (4)	0.067 (4)	0.047 (3)	−0.001 (3)	0.020 (3)	−0.005 (3)
C45	0.063 (4)	0.076 (5)	0.056 (3)	0.009 (4)	0.013 (3)	−0.003 (3)
C46	0.101 (5)	0.056 (4)	0.050 (3)	0.018 (4)	0.021 (4)	0.009 (3)
C47	0.087 (4)	0.056 (4)	0.044 (3)	−0.021 (3)	0.027 (3)	−0.014 (3)
C48	0.079 (4)	0.041 (3)	0.048 (3)	−0.012 (3)	0.028 (3)	0.002 (3)
C49	0.071 (4)	0.029 (3)	0.057 (3)	−0.001 (3)	0.020 (3)	−0.009 (3)
C50	0.045 (3)	0.032 (3)	0.045 (3)	−0.004 (2)	0.005 (2)	−0.004 (2)
C51	0.078 (4)	0.043 (3)	0.060 (3)	−0.021 (3)	0.025 (3)	−0.011 (3)
C52	0.108 (5)	0.043 (4)	0.063 (4)	−0.017 (3)	0.029 (4)	−0.020 (3)
N1	0.041 (2)	0.036 (2)	0.063 (3)	0.0039 (19)	0.013 (2)	−0.003 (2)
N2	0.100 (5)	0.104 (5)	0.158 (6)	0.039 (4)	0.044 (4)	0.001 (5)
N3	0.078 (4)	0.082 (4)	0.087 (4)	−0.025 (3)	0.016 (3)	−0.001 (3)
N4	0.038 (2)	0.034 (2)	0.055 (2)	−0.0003 (17)	0.0143 (18)	0.003 (2)
O1	0.0451 (19)	0.0297 (19)	0.0488 (18)	−0.0006 (15)	0.0081 (16)	0.0045 (16)
O2	0.075 (2)	0.030 (2)	0.061 (2)	−0.0153 (17)	0.0114 (19)	0.0040 (18)
O3	0.060 (2)	0.044 (2)	0.065 (2)	0.0040 (18)	−0.0052 (19)	0.016 (2)
O4	0.078 (3)	0.027 (2)	0.068 (2)	0.0149 (18)	0.012 (2)	−0.0037 (18)
O5	0.0371 (18)	0.043 (2)	0.057 (2)	−0.0013 (16)	0.0064 (16)	−0.0092 (18)
O6	0.056 (2)	0.047 (2)	0.075 (2)	−0.0053 (18)	0.0023 (19)	−0.023 (2)
O7	0.098 (3)	0.082 (3)	0.060 (2)	0.027 (3)	0.034 (2)	0.019 (2)

O8	0.140 (4)	0.076 (3)	0.059 (2)	−0.042 (3)	0.052 (3)	−0.026 (2)

Geometric parameters (Å, °)

C1—O2	1.236 (5)	C29—C30	1.378 (8)
C1—O1	1.269 (5)	C30—C31	1.383 (7)
C1—C2	1.543 (6)	C30—H30	0.9300
C2—O3	1.420 (5)	C31—C32	1.358 (7)
C2—C3	1.525 (6)	C31—H31	0.9300
C2—C9	1.532 (7)	C32—C33	1.369 (7)
C3—C8	1.382 (6)	C32—N1	1.454 (6)
C3—C4	1.412 (7)	C33—C34	1.376 (7)
C4—C5	1.367 (7)	C33—H33	0.9300
C4—H4	0.9300	C34—H34	0.9300
C5—C6	1.381 (9)	C35—C40	1.362 (7)
C5—H5	0.9300	C35—C36	1.388 (8)
C6—C7	1.367 (8)	C35—O7	1.398 (7)
C6—H6	0.9300	C36—C37	1.359 (8)
C7—C8	1.380 (7)	C36—H36	0.9300
C7—H7	0.9300	C37—C38	1.394 (8)
C8—H8	0.9300	C37—H37	0.9300
C9—C10	1.370 (7)	C38—N2	1.402 (8)
C9—C14	1.380 (7)	C38—C39	1.389 (9)
C10—C11	1.368 (8)	C39—C40	1.365 (8)
C10—H10	0.9300	C39—H39	0.9300
C11—C12	1.367 (10)	C40—H40	0.9300
C11—H11	0.9300	C41—C42	1.353 (8)
C12—C13	1.361 (10)	C41—C46	1.362 (8)
C12—H12A	0.9300	C41—O8	1.407 (7)
C13—C14	1.392 (7)	C42—C43	1.386 (8)
C13—H13A	0.9300	C42—H42	0.9300
C14—H14	0.9300	C43—C44	1.380 (7)
C15—O4	1.235 (5)	C43—H43	0.9300
C15—O5	1.262 (6)	C44—C45	1.362 (8)
C15—C16	1.542 (6)	C44—N3	1.397 (7)
C16—O6	1.434 (6)	C45—C46	1.396 (8)
C16—C17	1.510 (7)	C45—H45	0.9300
C16—C23	1.514 (6)	C46—H46	0.9300
C17—C18	1.388 (7)	C47—O8	1.371 (6)
C17—C22	1.392 (8)	C47—C52	1.371 (8)
C18—C19	1.367 (9)	C47—C48	1.376 (7)
C18—H18	0.9300	C48—C49	1.364 (6)
C19—C20	1.369 (11)	C48—H48	0.9300
C19—H19	0.9300	C49—C50	1.377 (7)
C20—C21	1.376 (12)	C49—H49	0.9300
C20—H20A	0.9300	C50—C51	1.358 (7)
C21—C22	1.387 (9)	C50—N4	1.465 (5)
C21—H21A	0.9300	C51—C52	1.387 (7)
C22—H22	0.9300	C51—H51	0.9300

C23—C24	1.377 (7)	C52—H52	0.9300
C23—C28	1.384 (7)	N1—H1A	0.8900
C24—C25	1.387 (7)	N1—H1B	0.8900
C24—H24	0.9300	N1—H1C	0.8900
C25—C26	1.377 (8)	N2—H2A	0.8600
C25—H25	0.9300	N2—H2B	0.8600
C26—C27	1.368 (8)	N3—H3A	0.8600
C26—H26	0.9300	N3—H3B	0.8600
C27—C28	1.380 (7)	N4—H4A	0.8900
C27—H27	0.9300	N4—H4B	0.8900
C28—H28	0.9300	N4—H4C	0.8900
C29—C34	1.364 (8)	O3—H3	0.8200
C29—O7	1.368 (6)	O6—H6A	0.8200
O2—C1—O1	124.5 (4)	O7—C29—C30	116.5 (5)
O2—C1—C2	118.4 (4)	C29—C30—C31	118.8 (5)
O1—C1—C2	117.0 (4)	C29—C30—H30	120.6
O3—C2—C3	108.1 (4)	C31—C30—H30	120.6
O3—C2—C9	108.4 (4)	C32—C31—C30	121.1 (5)
C3—C2—C9	113.8 (4)	C32—C31—H31	119.4
O3—C2—C1	108.2 (4)	C30—C31—H31	119.4
C3—C2—C1	106.3 (3)	C31—C32—C33	119.7 (5)
C9—C2—C1	111.8 (4)	C31—C32—N1	120.6 (4)
C8—C3—C4	118.1 (4)	C33—C32—N1	119.7 (4)
C8—C3—C2	123.3 (4)	C34—C33—C32	119.7 (5)
C4—C3—C2	118.2 (4)	C34—C33—H33	120.2
C5—C4—C3	120.7 (5)	C32—C33—H33	120.2
C5—C4—H4	119.6	C29—C34—C33	120.7 (5)
C3—C4—H4	119.6	C29—C34—H34	119.6
C4—C5—C6	120.4 (6)	C33—C34—H34	119.6
C4—C5—H5	119.8	C40—C35—C36	119.9 (6)
C6—C5—H5	119.8	C40—C35—O7	119.9 (6)
C7—C6—C5	119.0 (5)	C36—C35—O7	120.2 (5)
C7—C6—H6	120.5	C37—C36—C35	119.4 (5)
C5—C6—H6	120.5	C37—C36—H36	120.3
C6—C7—C8	121.8 (6)	C35—C36—H36	120.3
C6—C7—H7	119.1	C36—C37—C38	121.9 (6)
C8—C7—H7	119.1	C36—C37—H37	119.0
C7—C8—C3	119.9 (5)	C38—C37—H37	119.0
C7—C8—H8	120.0	C37—C38—N2	120.8 (7)
C3—C8—H8	120.0	C37—C38—C39	116.9 (6)
C10—C9—C14	118.4 (5)	N2—C38—C39	122.3 (6)
C10—C9—C2	117.9 (5)	C40—C39—C38	121.4 (6)
C14—C9—C2	123.6 (4)	C40—C39—H39	119.3
C9—C10—C11	121.1 (6)	C38—C39—H39	119.3
C9—C10—H10	119.4	C35—C40—C39	120.4 (6)
C11—C10—H10	119.4	C35—C40—H40	119.8
C12—C11—C10	120.4 (6)	C39—C40—H40	119.8
C12—C11—H11	119.8	C42—C41—C46	120.7 (6)
C10—C11—H11	119.8	C42—C41—O8	119.6 (6)

C11—C12—C13	119.6 (6)	C46—C41—O8	119.7 (6)
C11—C12—H12A	120.2	C41—C42—C43	119.6 (6)
C13—C12—H12A	120.2	C41—C42—H42	120.2
C12—C13—C14	120.1 (7)	C43—C42—H42	120.2
C12—C13—H13A	120.0	C44—C43—C42	120.5 (6)
C14—C13—H13A	120.0	C44—C43—H43	119.8
C9—C14—C13	120.3 (6)	C42—C43—H43	119.8
C9—C14—H14	119.9	C45—C44—C43	119.4 (6)
C13—C14—H14	119.9	C45—C44—N3	120.6 (5)
O4—C15—O5	124.8 (5)	C43—C44—N3	119.9 (6)
O4—C15—C16	118.2 (5)	C44—C45—C46	119.8 (6)
O5—C15—C16	117.0 (4)	C44—C45—H45	120.1
O6—C16—C17	107.5 (4)	C46—C45—H45	120.1
O6—C16—C23	108.2 (4)	C41—C46—C45	120.0 (6)
C17—C16—C23	115.2 (4)	C41—C46—H46	120.0
O6—C16—C15	107.6 (4)	C45—C46—H46	120.0
C17—C16—C15	111.9 (4)	O8—C47—C52	117.1 (5)
C23—C16—C15	106.3 (3)	O8—C47—C48	123.2 (5)
C18—C17—C22	117.8 (5)	C52—C47—C48	119.6 (5)
C18—C17—C16	118.0 (5)	C49—C48—C47	120.6 (5)
C22—C17—C16	124.2 (5)	C49—C48—H48	119.7
C19—C18—C17	121.8 (7)	C47—C48—H48	119.7
C19—C18—H18	119.1	C48—C49—C50	119.8 (5)
C17—C18—H18	119.1	C48—C49—H49	120.1
C20—C19—C18	118.9 (8)	C50—C49—H49	120.1
C20—C19—H19	120.5	C51—C50—C49	120.0 (5)
C18—C19—H19	120.5	C51—C50—N4	120.1 (4)
C19—C20—C21	122.2 (8)	C49—C50—N4	119.9 (4)
C19—C20—H20A	118.9	C50—C51—C52	120.3 (5)
C21—C20—H20A	118.9	C50—C51—H51	119.8
C20—C21—C22	118.0 (8)	C52—C51—H51	119.8
C20—C21—H21A	121.0	C47—C52—C51	119.5 (5)
C22—C21—H21A	121.0	C47—C52—H52	120.3
C17—C22—C21	121.4 (7)	C51—C52—H52	120.3
C17—C22—H22	119.3	C32—N1—H1A	109.5
C21—C22—H22	119.3	C32—N1—H1B	109.5
C24—C23—C28	117.5 (5)	H1A—N1—H1B	109.5
C24—C23—C16	123.5 (4)	C32—N1—H1C	109.5
C28—C23—C16	118.7 (4)	H1A—N1—H1C	109.5
C23—C24—C25	121.6 (5)	H1B—N1—H1C	109.5
C23—C24—H24	119.2	C38—N2—H2A	120.0
C25—C24—H24	119.2	C38—N2—H2B	120.0
C26—C25—C24	120.1 (6)	H2A—N2—H2B	120.0
C26—C25—H25	119.9	C44—N3—H3A	120.0
C24—C25—H25	119.9	C44—N3—H3B	120.0
C27—C26—C25	118.7 (5)	H3A—N3—H3B	120.0
C27—C26—H26	120.7	C50—N4—H4A	109.5
C25—C26—H26	120.7	C50—N4—H4B	109.5
C26—C27—C28	121.1 (5)	H4A—N4—H4B	109.5

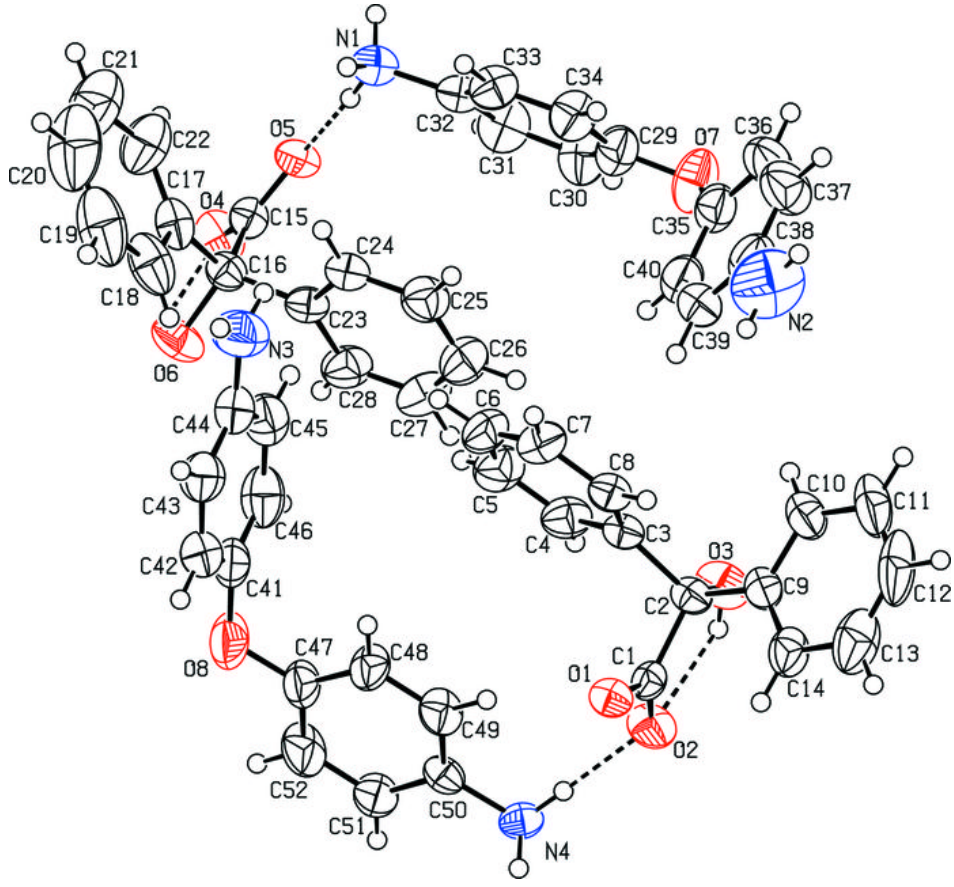
C26—C27—H27	119.4	C50—N4—H4C	109.5
C28—C27—H27	119.4	H4A—N4—H4C	109.5
C27—C28—C23	121.0 (5)	H4B—N4—H4C	109.5
C27—C28—H28	119.5	C2—O3—H3	109.5
C23—C28—H28	119.5	C16—O6—H6A	109.5
C34—C29—O7	123.6 (6)	C29—O7—C35	115.3 (4)
C34—C29—C30	119.9 (5)	C47—O8—C41	117.7 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6A···O4	0.82	2.10	2.576 (5)	117
O3—H3···O2	0.82	2.08	2.589 (5)	120
N4—H4C···O5^{i}	0.89	1.81	2.683 (5)	168
N4—H4B···O1^{ii}	0.89	1.89	2.761 (5)	166
N4—H4A···O2	0.89	1.90	2.790 (5)	179
N3—H3A···O6^{iii}	0.86	2.33	3.076 (6)	146
N1—H1C···O5	0.89	1.89	2.777 (5)	174
N1—H1B···O4^{iii}	0.89	1.88	2.762 (5)	173
N1—H1A···O1^{iv}	0.89	1.81	2.685 (5)	165

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

Fig. 1


N1
C21
C33
C34
C32
C22
O5
C29
O7
C36
C20
C17
C31
C30
C37
O4
C15
C35
C19
C24
C38
C16
C40
C18
C25
C23
N2
N3
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O6
C26
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C44
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C27
C7
C8
C10
C43
C5
C11
C46
C4
C3
O3
C42
C41
C9
C12
C2
C47
C48
C1
C13
O8
O1
C14
C49
O2
C52
C50
C51
N4

Fig. 2