

2-Phenylbiguanidinium hydrogen succinate methanol monosolvate

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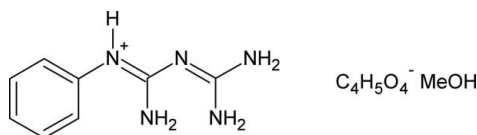
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; some non-H atoms missing; R factor = 0.056; wR factor = 0.174; data-to-parameter ratio = 18.7.

In the crystal of the title compound, $\text{C}_8\text{H}_{12}\text{N}_5^+ \cdot \text{C}_4\text{H}_5\text{O}_4^- \cdot \text{CH}_3\text{OH}$, the hydrogen succinate anions form infinite [010] chains *via* short, almost symmetrical, $\text{O} \cdots \text{H} \cdots \text{O}$ hydrogen bonds. The 2-phenylbiguanidinium cations interconnect these chains into layers lying parallel to the bc plane by way of $\text{N}-\text{H} \cdots \text{O}$ links. These planes only weakly interact in the direction of the a axis *via* $\text{C}-\text{H} \cdots \pi$ contacts between offset phenyl rings, leaving as much as 17% of the unit-cell volume accessible for the solvent. However, the methanol solvent molecules could not be resolved due to extensive disorder and their assumed presence was removed from the overall scattering by the *PLATON SQUEEZE* procedure.

Related literature

Biguanides forms stable complexes, see: Marchi *et al.* (1999); Ray (1961); Anderson *et al.* (1995) and also have applications in medicine, see: Sirtori & Pasik (1994); Clement & Girreser (1999); Thompson *et al.* (1999); Ross *et al.* (2004); Woo *et al.* (1999); Watkins *et al.* (1987); Morain *et al.* (1994); Shapiro *et al.* (1959*a,b*). Ionic crystal structures containing biguanide cations are formed by relatively strong hydrogen bonds, see: Martin *et al.* (1996); Martin & Pinkerton (1996); Pinkerton *et al.* (1978); Matulková *et al.* (2008, 2010). For the *SQUEEZE* method used to solve the structure, see: van der Sluis & Spek (1990).



Experimental

Crystal data

$\text{C}_8\text{H}_{12}\text{N}_5^+ \cdot \text{C}_4\text{H}_5\text{O}_4^- \cdot \text{CH}_4\text{O}$
 $M_r = 327.35$
 Monoclinic, $P2_1/c$
 $a = 10.3280$ (3) Å
 $b = 6.4590$ (1) Å
 $c = 24.6770$ (6) Å
 $\beta = 94.0480$ (13)°
 $V = 1642.06$ (7) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 293$ K
 $0.45 \times 0.4 \times 0.18$ mm

Data collection

Nonius KappaCCD diffractometer
 18123 measured reflections
 3568 independent reflections
 2613 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.174$
 $S = 1.07$
 3568 reflections
 191 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C3–C8 ring.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1} \cdots \text{O3}^{\text{i}}$	0.93	2.18	3.021 (2)	149
$\text{N1}-\text{H1} \cdots \text{O4}^{\text{i}}$	0.93	2.64	3.5217 (17)	158
$\text{N2}-\text{H2A} \cdots \text{O3}^{\text{i}}$	0.87	2.09	2.8855 (17)	152
$\text{N2}-\text{H2B} \cdots \text{O4}^{\text{ii}}$	0.92	2.14	3.0248 (19)	162
$\text{N4}-\text{H4A} \cdots \text{O1}^{\text{iii}}$	0.93	2.13	3.0273 (18)	161
$\text{N4}-\text{H4B} \cdots \text{O2}^{\text{iv}}$	0.96	1.90	2.8605 (16)	180
$\text{N5}-\text{H5B} \cdots \text{O1}^{\text{iv}}$	0.90	2.15	3.0440 (17)	170
$\text{O2}-\text{H2} \cdots \text{O4}^{\text{ii}}$	1.20	1.25	2.4500 (16)	173
$\text{C6}-\text{H6} \cdots \text{Cg1}^{\text{v}}$	0.93	3.10	3.676 (2)	122

Symmetry codes: (i) $-x, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x, y - 1, z$; (iii) $-x, -y, -z + 2$; (iv) $-x, -y - 1, -z + 2$; $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *COLLECT* (Hooft, 1998) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *COLLECT* and *DENZO*; data reduction: *COLLECT* and *DENZO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5725).

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supplementary materials

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2-Phenylbiguanidinium hydrogen succinate methanol monosolvate

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Comment

Biguanides are strong σ - and π -donating ligands, which form stable complexes (Marchi *et al.*, 1999; Ray, 1961) with transition metal ions in high or unusual oxidation states. Biguanide complexes of boron have also been investigated as potential compounds for wood conservation (Anderson *et al.*, 1995).

Another application of biguanides lies in the field of medicine (Sirtori & Pasik, 1994; Clement & Girreser, 1999). *N*-dimethylbiguanide and *N*-phenylethylbiguanide are used for the treatment of diabetes mellitus (Thompson *et al.*, 1999; Ross *et al.*, 2004; Woo *et al.*, 1999), therapeutic treatment of pain, anxiety, memory disorders (Morain *et al.*, 1994). Biguanide and its derivatives are also produced as antimalarial drugs (Watkins *et al.*, 1987) and drugs with hypoglycaemic activity (Marchi *et al.*, 1999; Sirtori & Pasik, 1994; Shapiro *et al.*, 1959*a,b*).

We have prepared and discussed *N*-phenylbiguanide compounds within our project of searching for new materials with nonlinear optical properties (Matulková *et al.*, 2010; Matulková *et al.*, 2008), where *N*-phenylbiguanidinium(1+) cations can act as a polarizable compound with delocalized π -electron. The molecular conformation of title compound, (I), is illustrated in Fig. 1.

The hydrogen-bonding geometries in title compound are listed in list of hydrogen bonds and illustrated in Fig. 2. A number of intra- and intermolecular hydrogen bonds stabilize the molecular conformation. The crystal structure is built up chains (along the axis **b**) of hydrogen succinate anions with the shared hydrogen atoms with occupancy 0.5 (hydrogen bond O2 - H2...O4 with D...A distances of 2.451 (2) Å). These chains are interconnected by 2-phenylbiguanidinium cations to form a three-dimensional network. A residue electron density of disordered molecules of methanol was found on the differential Fourier map and the crystal structure was solved by a SQUEEZE method (van der Sluis & Spek, 1990). Free cavities of maximum on the Fourier map are indicated by blue spheres and are located in the 30% of crystal structure. The cavities can be filled by spheres of two types with the radii 2.247 Å and 2.076 Å (see Fig. 3). The unit cell contains two spheres of each size.

Experimental

The crystals of the title compound, were obtained from solution of 0.2 g of *N*-phenylbiguanide (98%, Aldrich) and 0.14 g of succinic acid (p.a., Lachema) in 10 ml of water. The solution was left to crystallize at room temperature for several weeks. The colourless crystals obtained were filtered off, washed with methanol and dried in vacuum desiccator over KOH. The melting point ranges 410–412 K.

The infrared spectra were recorded at room temperature using DRIFTS and the nujol or fluorolube mull techniques on a Nicolet Magna 760 FTIR spectrometer with 2 cm⁻¹ resolution (4 cm⁻¹ resolution in FAR IR region) and Happ-Genzel apodization in the 85–4000 cm⁻¹ region.

supplementary materials

FTIR spectrum (cm^{-1}): 3451 m; 3350 m; 3296 m; 3184 m; 2938 w; 2725 mb; 2660 mb; 2550 mb; 1712 m; 1674 m; 1640 vs; 1605 m; 1583 m; 1538 vs; 1497 s; 1455 m; 1429 m; 1419 m; 1334 w; 1310 w; 1294 w; 1256 m; 1198 m; 1176 m; 1074 w; 1054 wb; 1031 w; 957 mb; 842 w; 818 w; 804 w; 772 w; 747 m; 722 w; 698 m; 638 m; 579 m; 549 mb; 528 sh; 498 vw; 485 vw; 446 w; 414 vw; 367 wb; 261 mb; 221 w; 179 w; 143 w.

The Raman spectra of polycrystalline samples were recorded at room temperature on a Nicolet Magna 760 FTIR spectrometer equipped with the Nicolet Nexus FT Raman module (2 cm^{-1} resolution, Happ–Genzel apodization, 1064 nm Nd:YVO4 laser excitation, 450 mW power at the sample) in the $100\text{--}3700 \text{ cm}^{-1}$ region.

FT Raman spectrum (cm^{-1}): 3452 vw; 3335 vwb; 3200 vw; 3079 sh; 3066 m; 2966 m; 2953 m; 2918 m; 1668 w; 1639 w; 1604 vs; 1591 m; 1563 m; 1546 m; 1499 m; 1450 w; 1437 w; 1428 w; 1418 m; 1408 m; 1379 vw; 1341 vw; 1327 vw; 1295 s; 1255 s; 1178 m; 1156 m; 1095 w; 1079 w; 1054 w; 1030 m; 1002 vs; 952 w; 937 w; 923 m; 909 vw; 844 m; 767 w; 748 w; 722 w; 681 w; 633 w; 615 m; 572 vw; 538 w; 502 w; 488 w; 414 wb; 387 w; 374 w; 340 w; 283 w; 265 m; 220 m; 188 m; 141 vs; 124 m.

Refinement

H atoms attached to C and N atoms were calculated in geometrically idealized positions, with $C_{sp^3} - H = 0.97 \text{ \AA}$ and $C_{sp^2} - H = 0.93 \text{ \AA}$, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.5 U_{eq}(C)$. The positions of H atoms attached to O and N atoms were localized in difference Fourier maps, and refined isotropically.

Figures

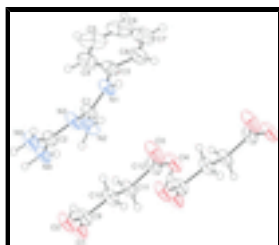


Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

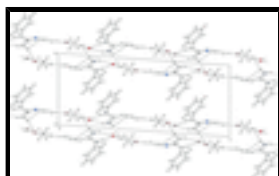


Fig. 2. Part of the crystal structure of (I) showing formation of cation layers along [010].

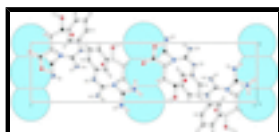


Fig. 3. Part of the crystal structure of (I) with blue sphere filling the cavity.

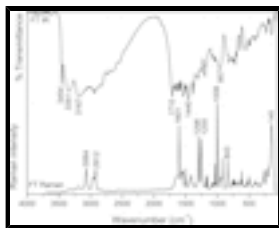
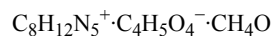


Fig. 4. FTIR (compiled from nujol and fluorolube mulls) and FT Raman spectra of (I).

2-Phenylbiguanidinium hydrogen succinate methanol monosolvate

Crystal data



$$M_r = 327.35$$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$$a = 10.3280 (3) \text{ \AA}$$

$$b = 6.4590 (1) \text{ \AA}$$

$$c = 24.6770 (6) \text{ \AA}$$

$$\beta = 94.0480 (13)^\circ$$

$$V = 1642.06 (7) \text{ \AA}^3$$

$$Z = 4$$

$$F(000) = 696$$

$$D_x = 1.324 \text{ Mg m}^{-3}$$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3693 reflections

$$\theta = 1-27.1^\circ$$

$$\mu = 0.10 \text{ mm}^{-1}$$

$$T = 293 \text{ K}$$

Plate, colourless

$$0.45 \times 0.4 \times 0.18 \text{ mm}$$

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube
horizontally mounted graphite crystal

Detector resolution: $9.091 \text{ pixels mm}^{-1}$

ω and π scans to fill the Ewald sphere

18123 measured reflections

3568 independent reflections

2613 reflections with $I > 2\sigma(I)$

$$R_{\text{int}} = 0.030$$

$$\theta_{\text{max}} = 27.1^\circ, \theta_{\text{min}} = 1.7^\circ$$

$$h = -13 \rightarrow 13$$

$$k = -8 \rightarrow 8$$

$$l = -31 \rightarrow 31$$

Refinement

Refinement on F^2

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.056$$

$$wR(F^2) = 0.174$$

$$S = 1.07$$

3568 reflections

191 parameters

0 restraints

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.1087P)^2 + 0.1338P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\text{max}} = 0.001$$

$$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$$

Extinction correction: *SHELXL97* (Sheldrick, 1997),

$$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

supplementary materials

Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.055 (7)

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.20697 (15)	-0.1226 (2)	0.77926 (5)	0.0515 (4)
H1	0.1757	-0.1468	0.7435	0.062*
N2	0.07708 (16)	-0.3948 (2)	0.79668 (5)	0.0570 (4)
H2A	0.0599	-0.4106	0.7620	0.068*
H2B	0.0280	-0.4630	0.8208	0.068*
N3	0.18707 (15)	-0.2116 (2)	0.86872 (5)	0.0514 (4)
N4	0.16449 (16)	-0.2862 (2)	0.95713 (5)	0.0537 (4)
H4A	0.1524	-0.1453	0.9625	0.064*
H4B	0.1638	-0.3785	0.9874	0.064*
N5	0.18754 (16)	-0.5585 (2)	0.90047 (5)	0.0575 (4)
H5A	0.2005	-0.6075	0.8702	0.069*
H5B	0.1909	-0.6467	0.9288	0.069*
C1	0.15804 (17)	-0.2481 (2)	0.81647 (6)	0.0464 (4)
C2	0.17836 (16)	-0.3547 (2)	0.90737 (6)	0.0448 (4)
O1	-0.16551 (14)	-0.15632 (17)	1.00242 (4)	0.0577 (4)
O2	-0.16362 (15)	-0.43722 (16)	0.95237 (5)	0.0616 (4)
H2	-0.1523	-0.4961	0.9068	0.074*
O3	-0.10001 (17)	0.1366 (2)	0.81889 (5)	0.0738 (5)
O4	-0.13315 (14)	0.42420 (17)	0.86173 (4)	0.0598 (4)
C3	0.29311 (18)	0.0463 (2)	0.78891 (6)	0.0519 (4)
C4	0.3920 (2)	0.0464 (3)	0.82884 (8)	0.0687 (6)
H4	0.4044	-0.0663	0.8521	0.082*
C5	0.4738 (3)	0.2177 (5)	0.83414 (11)	0.0929 (9)
H5	0.5407	0.2191	0.8614	0.112*
C6	0.4578 (3)	0.3832 (4)	0.80008 (14)	0.0964 (10)
H6	0.5130	0.4967	0.8042	0.116*
C7	0.3612 (3)	0.3805 (4)	0.76041 (14)	0.0911 (9)
H7	0.3508	0.4928	0.7369	0.109*
C8	0.2768 (2)	0.2135 (3)	0.75394 (9)	0.0694 (6)
H8	0.2103	0.2139	0.7265	0.083*
C9	-0.16134 (17)	-0.2385 (2)	0.95785 (6)	0.0464 (4)

C10	-0.15527 (19)	-0.1185 (2)	0.90552 (6)	0.0488 (4)
H10A	-0.0830	-0.1701	0.8864	0.059*
H10B	-0.2341	-0.1447	0.8828	0.059*
C11	-0.13977 (19)	0.1122 (2)	0.91303 (6)	0.0462 (4)
H11A	-0.2155	0.1663	0.9293	0.055*
H11B	-0.0647	0.1387	0.9379	0.055*
C12	-0.12325 (17)	0.2254 (2)	0.86057 (6)	0.0462 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0785 (10)	0.0498 (8)	0.0273 (6)	-0.0067 (6)	0.0109 (6)	0.0028 (5)
N2	0.0825 (11)	0.0601 (9)	0.0291 (7)	-0.0165 (7)	0.0088 (6)	-0.0007 (6)
N3	0.0828 (10)	0.0438 (7)	0.0288 (7)	-0.0077 (7)	0.0109 (6)	0.0018 (5)
N4	0.0905 (11)	0.0458 (8)	0.0258 (6)	-0.0013 (7)	0.0104 (6)	0.0035 (5)
N5	0.0926 (12)	0.0439 (8)	0.0368 (7)	0.0057 (7)	0.0099 (7)	0.0031 (6)
C1	0.0672 (10)	0.0430 (8)	0.0302 (7)	0.0002 (7)	0.0121 (7)	0.0014 (6)
C2	0.0593 (10)	0.0457 (8)	0.0300 (7)	-0.0025 (7)	0.0065 (6)	0.0021 (6)
O1	0.1019 (10)	0.0397 (6)	0.0324 (6)	-0.0008 (6)	0.0104 (6)	0.0029 (4)
O2	0.1145 (11)	0.0315 (6)	0.0402 (6)	-0.0014 (6)	0.0168 (6)	0.0058 (4)
O3	0.1439 (14)	0.0514 (7)	0.0279 (6)	-0.0011 (7)	0.0179 (7)	0.0000 (5)
O4	0.1054 (10)	0.0345 (6)	0.0410 (6)	-0.0023 (6)	0.0155 (6)	0.0084 (4)
C3	0.0733 (11)	0.0461 (9)	0.0391 (8)	-0.0031 (8)	0.0240 (8)	-0.0013 (6)
C4	0.0815 (14)	0.0792 (14)	0.0468 (10)	-0.0154 (10)	0.0140 (10)	0.0015 (9)
C5	0.0854 (16)	0.119 (2)	0.0775 (16)	-0.0400 (15)	0.0293 (13)	-0.0254 (15)
C6	0.105 (2)	0.0770 (16)	0.114 (2)	-0.0389 (14)	0.0597 (19)	-0.0213 (15)
C7	0.110 (2)	0.0545 (12)	0.116 (2)	-0.0070 (12)	0.0548 (19)	0.0143 (13)
C8	0.0849 (14)	0.0561 (11)	0.0711 (13)	0.0021 (9)	0.0319 (11)	0.0170 (9)
C9	0.0715 (11)	0.0329 (7)	0.0357 (8)	0.0001 (7)	0.0097 (7)	0.0041 (6)
C10	0.0817 (12)	0.0329 (8)	0.0325 (7)	-0.0001 (7)	0.0086 (7)	0.0027 (5)
C11	0.0779 (11)	0.0335 (7)	0.0281 (7)	-0.0031 (7)	0.0087 (7)	0.0026 (5)
C12	0.0741 (11)	0.0366 (8)	0.0281 (7)	-0.0040 (7)	0.0050 (7)	0.0029 (5)

Geometric parameters (\AA , $^\circ$)

N1—C1	1.3495 (19)	C3—C4	1.368 (3)
N1—C3	1.417 (2)	C3—C8	1.385 (2)
N1—H1	0.9313	C4—C5	1.393 (3)
N2—C1	1.333 (2)	C4—H4	0.9300
N2—H2A	0.8679	C5—C6	1.363 (4)
N2—H2B	0.9206	C5—H5	0.9300
N3—C1	1.3242 (19)	C6—C7	1.348 (5)
N3—C2	1.3358 (19)	C6—H6	0.9300
N4—C2	1.3223 (18)	C7—C8	1.389 (4)
N4—H4A	0.9291	C7—H7	0.9300
N4—H4B	0.9561	C8—H8	0.9300
N5—C2	1.331 (2)	C9—C10	1.511 (2)
N5—H5A	0.8301	C10—C11	1.509 (2)
N5—H5B	0.9016	C10—H10A	0.9700

supplementary materials

O1—C9	1.2245 (18)	C10—H10B	0.9700
O2—C9	1.2909 (19)	C11—C12	1.5069 (19)
O2—H2	1.2016	C11—H11A	0.9700
O3—C12	1.2164 (18)	C11—H11B	0.9700
O4—C12	1.2884 (19)		
C1—N1—C3	127.49 (14)	C4—C5—H5	119.4
C1—N1—H1	115.0	C7—C6—C5	119.3 (2)
C3—N1—H1	117.4	C7—C6—H6	120.3
C1—N2—H2A	121.6	C5—C6—H6	120.3
C1—N2—H2B	117.6	C6—C7—C8	121.3 (2)
H2A—N2—H2B	119.8	C6—C7—H7	119.4
C1—N3—C2	123.36 (14)	C8—C7—H7	119.4
C2—N4—H4A	119.0	C3—C8—C7	119.1 (2)
C2—N4—H4B	121.6	C3—C8—H8	120.4
H4A—N4—H4B	119.4	C7—C8—H8	120.4
C2—N5—H5A	120.7	O1—C9—O2	121.57 (13)
C2—N5—H5B	121.7	O1—C9—C10	123.48 (13)
H5A—N5—H5B	117.2	O2—C9—C10	114.95 (13)
N3—C1—N2	125.19 (14)	C11—C10—C9	114.32 (12)
N3—C1—N1	119.06 (15)	C11—C10—H10A	108.7
N2—C1—N1	115.64 (14)	C9—C10—H10A	108.7
N4—C2—N5	117.58 (14)	C11—C10—H10B	108.7
N4—C2—N3	116.65 (14)	C9—C10—H10B	108.7
N5—C2—N3	125.71 (13)	H10A—C10—H10B	107.6
C9—O2—H2	114.2	C12—C11—C10	113.04 (12)
C4—C3—C8	119.99 (19)	C12—C11—H11A	109.0
C4—C3—N1	123.32 (16)	C10—C11—H11A	109.0
C8—C3—N1	116.63 (18)	C12—C11—H11B	109.0
C3—C4—C5	119.0 (2)	C10—C11—H11B	109.0
C3—C4—H4	120.5	H11A—C11—H11B	107.8
C5—C4—H4	120.5	O3—C12—O4	120.62 (13)
C6—C5—C4	121.2 (3)	O3—C12—C11	122.59 (14)
C6—C5—H5	119.4	O4—C12—C11	116.78 (12)

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 is the centroid of the C3–C8 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O3 ⁱ	0.93	2.18	3.021 (2)	149
N1—H1 \cdots O4 ⁱ	0.93	2.64	3.5217 (17)	158
N2—H2A \cdots O3 ⁱ	0.87	2.09	2.8855 (17)	152
N2—H2B \cdots O4 ⁱⁱ	0.92	2.14	3.0248 (19)	162
N4—H4A \cdots O1 ⁱⁱⁱ	0.93	2.13	3.0273 (18)	161
N4—H4B \cdots O2 ^{iv}	0.96	1.90	2.8605 (16)	180
N5—H5B \cdots O1 ^{iv}	0.90	2.15	3.0440 (17)	170
O2—H2 \cdots O4 ⁱⁱ	1.20	1.25	2.4500 (16)	173
C6—H6 \cdots Cg1 ^v	0.93	3.10	3.676 (2)	122

Symmetry codes: (i) $-x, y-1/2, -z+3/2$; (ii) $x, y-1, z$; (iii) $-x, -y, -z+2$; (iv) $-x, -y-1, -z+2$; (v) $-x+1, y+1/2, -z+3/2$.

Fig. 1

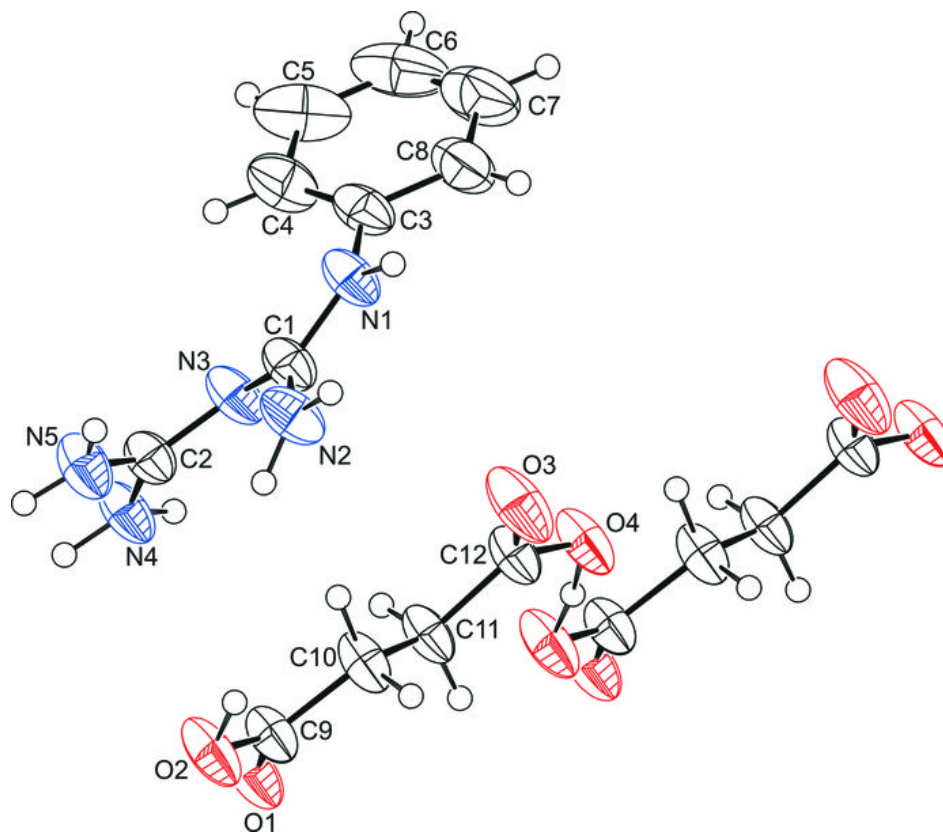


Fig. 2

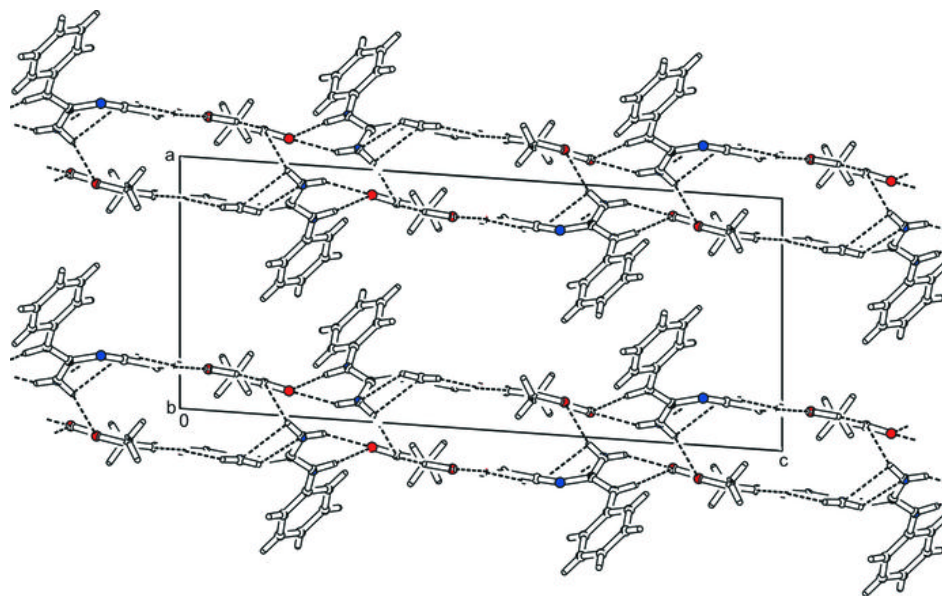


Fig. 3

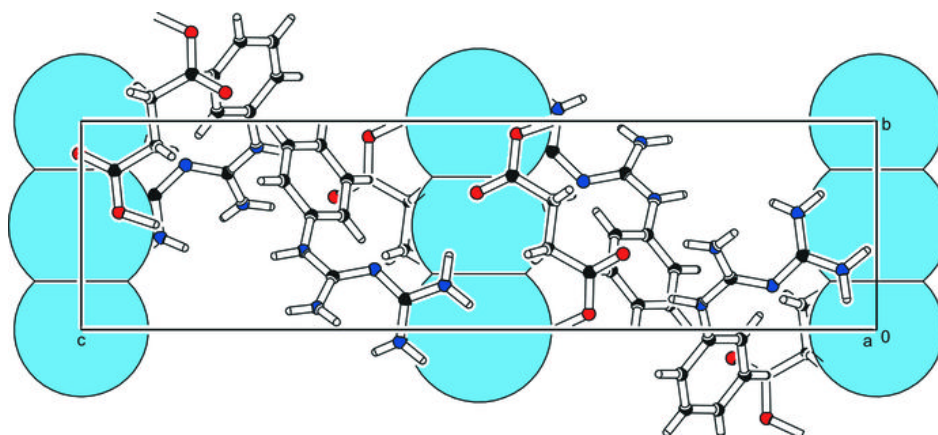


Fig. 4

