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2-(3-Oxocyclohex-1-enyl)benzoic acid

Flaviana T. Vieira,[a] Daniele C. Menezes,[a] Geraldo M. de Lima[a] and Nivaldo L. Speziali[b]*

[a]Departamento de Química, ICEx–UFMG, Brazil, and [b]Departamento de Física, ICEx–UFMG, Brazil
Correspondence e-mail: nspezial@fisica.ufmg.br

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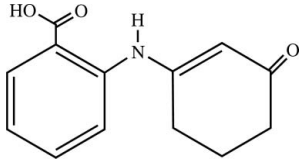
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.046; wR factor = 0.132; data-to-parameter ratio = 7.4.

The title compound, $C_{13}H_{13}NO_3$, crystallizes with two molecules in the asymmetric unit. The values of the relevant C—C, C═C and C—N bond lengths indicate that the molecules exist in the enamine tautomeric form. In each molecule, the cyclohexene ring adopts a slightly distorted envelope conformation. Molecules are linked by intermolecular O—H···O hydrogen bonds between carboxyl and C═O groups. There is also an intramolecular N—H···O hydrogen bond in each molecule.

Related literature

For related literature, see: Allen (2002); Strozhev & Lielbriedis (1990).



Experimental

Crystal data

$C_{13}H_{13}NO_3$
M_r = 231.24
Orthorhombic, $Pna2_1$
a = 11.3867 (11) Å
b = 13.0719 (9) Å
c = 15.3389 (14) Å
V = 2283.2 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.2 × 0.2 × 0.2 mm

Data collection

Siemens $P4$ diffractometer
Absorption correction: none
2549 measured reflections
2266 independent reflections
1800 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
3 standard reflections
every 247 reflections
intensity decay: 4%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.132
S = 0.93
2266 reflections
307 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected bond lengths (Å).

C1—N	1.407 (5)	C1′—N′	1.396 (5)
N—C7	1.359 (5)	N′—C7′	1.366 (5)
C7—C12	1.364 (5)	C7′—C12′	1.358 (5)
C11—C12	1.433 (5)	C11′—C12′	1.425 (5)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*O*···O3′	0.82	1.81	2.562 (4)	151
N—H1···O1	0.86	1.98	2.665 (4)	136
O2′—H2*O*′···O3^{i}	0.82	1.78	2.562 (4)	160
N′—H1′···O1′	0.86	1.98	2.659 (4)	135

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

Data collection: *XSCANS* (Siemens, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); 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: WN2148).

References

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  doi:10.1107/S1600536807029959

supplementary materials

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2-(3-Oxocyclohex-1-enyl)benzoic acid

F. T. Vieira, D. C. Menezes, G. M. de Lima and N. L. Speziali

Comment

The preparation of the title compound has been reported in the literature in a condensation reaction with isopropylidene malonate (Strozhev & Lielbriedis, 1990). The compound was obtained in 37% yield and no X-ray crystallographic determination has hitherto been performed. A search of the Cambridge Structural Database (Version 5.28; Allen, 2002) yielded no hits.

The title compound crystallizes with two molecules per asymmetric unit. In each molecule the cyclohexene ring adopts a slightly distorted envelope conformation, with C9 and C9' as flap atoms. The bond distances C1—N, N—C7, C7═C12, C12—C11, C1'—N,' N'—C7', C7'═C12' and C12'—C11' (Table 1 and Fig. 1) clearly indicate that the enamine tautomer is present in the crystal structure, rather than the imine normally expected from a Schiff base reaction. The C—N—C bond angles, 131.6 (3)° and 130.7 (3)°, are typical of Nsp^3.

The X-ray crystallographic determination has revealed that the title compound exists in the solid state in a pseudo-polymeric arrangement, held together by intermolecular O—H···O hydrogen bonds. These connect carboxyl and C═O groups of neighbouring molecules.

Experimental

To a round-bottomed flask charged with 2-aminobenzoic acid (2.74 g, 0.02 mol) dissolved in methanol (20 ml) was slowly added a solution of 1,3-cyclohexadione (2.24 g, 0.02 mol) in 20 ml of methanol. The mixture was refluxed and stirred for 4 h; it was observed that the colour changed from colourless to yellow. The solution was cooled to room temperature and the solvent was removed in vacuum; the yellow solid was washed with diethyl ether. X-ray quality crystals were obtained after slow evaporation of a methanol/water (9:1) solution. Yield 75%. IR (ν/cm^{-1}): 1376 ($\nu_{C—N}$). ^{1}H-NMR: (δ): 7.97–7.92 d (C3), 7.4 m (C5, C6), 7.09 t (C4), 2.51–2.48 m (C11), 2.26–2.27 m (C9), 1.95–1.92 m (C10). ^{13}C-NMR (δ): 201. 9 (C12), 170.4 (C1), 165.4 (C8), 141.9 (C7), 134.6 (C3), 133.6 (C5), 125.5 (C6), 124.64 (C4), 122.58 (C2), 100.94 (C13), 37.12 (C11), 31.07 (C9), 22.92 (C10). Elemental analysis(%) for $C_{13}H_{13}NO_3$ found (calc.): C 67.21 (67.52), H 5.43 (5.66), N 5.96 (6.05).

Refinement

Most H atoms were detected in a Fourier difference map; nevertheless, their positions were subsequently calculated and they were constrained to ride on their parent atoms, with O—H = 0.82 Å, N—H = 0.86 Å, C—H = 0.93 Å for Csp^2 and C—H = 0.97Å for methylene. U_{iso}(H) = xU_{eq}(carrier atom), where x = 1.2 for C, N and 1.5 for O. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Figures

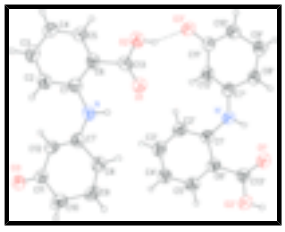


Fig. 1. Molecular structure of the title compound, showing the atom-numbering scheme and the intermolecular hydrogen bond (dashed line) Displacement ellipsoids are drawn at the 50% probability level.

2-(3-Oxocyclohex-1-enyl)benzoic acid

Crystal data

$C_{13}H_{13}NO_3$	$D_x = 1.345$ Mg m^{-3}
$M_r = 231.24$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, $Pna2_1$	Cell parameters from 38 reflections
$a = 11.3867$ (11) Å	$\theta = 4.8$–$12.5°$
$b = 13.0719$ (9) Å	$\mu = 0.10$ mm^{-1}
$c = 15.3389$ (14) Å	$T = 293$ (2) K
$V = 2283.2$ (3) Å^3	Prismatic, colorless
$Z = 8$	0.2 × 0.2 × 0.2 mm
$F_{000} = 976$	

Data collection

Siemens P4 diffractometer	$R_{int} = 0.015$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.5°$
Monochromator: graphite	$\theta_{min} = 2.1°$
$T = 293(2)$ K	$h = -1 \rightarrow 13$
ω / 2θ scans	$k = -3 \rightarrow 15$
Absorption correction: none	$l = -4 \rightarrow 18$
2549 measured reflections	3 standard reflections
2266 independent reflections	every 247 reflections
1800 reflections with $I > 2\sigma(I)$	intensity decay: 4%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.132$	$(\Delta/\sigma)_{max} = 0.018$
$S = 0.93$	$\Delta\rho_{max} = 0.18$ e Å^{-3}

2266 reflections
307 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: none

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}
O1	0.5835 (3)	0.4550 (2)	0.7105 (2)	0.0678 (9)
O2	0.4210 (2)	0.4172 (2)	0.7836 (2)	0.0644 (9)
H2O	0.4113	0.4792	0.7804	0.097*
C13	0.5183 (3)	0.3924 (3)	0.7441 (3)	0.0452 (9)
C6	0.5421 (3)	0.2797 (3)	0.7469 (2)	0.0402 (8)
C5	0.4676 (3)	0.2177 (3)	0.7959 (3)	0.0465 (9)
H5	0.4034	0.2471	0.8238	0.056*
C4	0.4865 (3)	0.1147 (3)	0.8043 (3)	0.0502 (10)
H4	0.4359	0.0743	0.8373	0.060*
C3	0.5822 (3)	0.0721 (3)	0.7627 (3)	0.0499 (10)
H3	0.5964	0.0024	0.7688	0.060*
C2	0.6568 (3)	0.1297 (3)	0.7128 (2)	0.0426 (8)
H2	0.7204	0.0990	0.6852	0.051*
C1	0.6372 (3)	0.2350 (3)	0.7033 (2)	0.0376 (8)
N	0.7126 (3)	0.2982 (2)	0.6544 (2)	0.0455 (8)
H1	0.7075	0.3621	0.6671	0.055*
C7	0.7918 (3)	0.2764 (3)	0.5909 (2)	0.0403 (8)
C8	0.8719 (3)	0.3646 (3)	0.5703 (3)	0.0443 (9)
H8A	0.9332	0.3678	0.6142	0.053*
H8B	0.8273	0.4277	0.5736	0.053*
C9	0.9286 (4)	0.3574 (3)	0.4812 (3)	0.0530 (11)
H9A	0.9899	0.4086	0.4760	0.064*
H9B	0.8704	0.3704	0.4364	0.064*
C10	0.9808 (4)	0.2511 (3)	0.4687 (3)	0.0519 (11)
H10A	1.0105	0.2450	0.4096	0.062*
H10B	1.0464	0.2424	0.5083	0.062*
C11	0.8926 (3)	0.1686 (3)	0.4848 (3)	0.0394 (8)

C12	0.8009 (3)	0.1860 (3)	0.5470 (3)	0.0400 (8)
H12	0.7463	0.1346	0.5577	0.048*
O3	0.9019 (2)	0.08747 (19)	0.4442 (2)	0.0554 (7)
O1'	0.6639 (3)	0.9504 (2)	0.5383 (2)	0.0723 (10)
O2'	0.8124 (2)	0.9080 (2)	0.4533 (2)	0.0649 (9)
H2O'	0.8278	0.9690	0.4585	0.097*
C13'	0.7225 (3)	0.8857 (3)	0.5014 (3)	0.0429 (9)
C6'	0.6979 (3)	0.7738 (3)	0.5042 (2)	0.0382 (8)
C5'	0.7693 (3)	0.7071 (3)	0.4574 (2)	0.0453 (9)
H5'	0.8350	0.7327	0.4288	0.054*
C4'	0.7451 (3)	0.6040 (3)	0.4525 (3)	0.0484 (9)
H4'	0.7951	0.5602	0.4223	0.058*
C3'	0.6461 (4)	0.5666 (3)	0.4926 (3)	0.0505 (10)
H3'	0.6276	0.4976	0.4874	0.061*
C2'	0.5738 (3)	0.6299 (3)	0.5405 (3)	0.0483 (10)
H2'	0.5075	0.6029	0.5675	0.058*
C1'	0.5988 (3)	0.7339 (3)	0.5491 (3)	0.0393 (8)
N'	0.5268 (2)	0.8004 (2)	0.5962 (2)	0.0455 (8)
H1'	0.5340	0.8638	0.5824	0.055*
C7'	0.4468 (3)	0.7814 (3)	0.6606 (2)	0.0382 (8)
C8'	0.3695 (3)	0.8713 (3)	0.6803 (3)	0.0442 (9)
H8'A	0.3073	0.8746	0.6372	0.053*
H8'B	0.4155	0.9336	0.6755	0.053*
C9'	0.3153 (4)	0.8663 (3)	0.7703 (3)	0.0522 (10)
H9'A	0.2570	0.9199	0.7766	0.063*
H9'B	0.3754	0.8766	0.8142	0.063*
C10'	0.2581 (3)	0.7627 (3)	0.7830 (3)	0.0554 (12)
H10C	0.2285	0.7577	0.8421	0.066*
H10D	0.1918	0.7566	0.7436	0.066*
C11'	0.3422 (3)	0.6767 (3)	0.7664 (2)	0.0408 (8)
C12'	0.4342 (3)	0.6921 (3)	0.7048 (3)	0.0414 (8)
H12'	0.4872	0.6393	0.6946	0.050*
O3'	0.3271 (2)	0.5937 (2)	0.8042 (2)	0.0561 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0716 (18)	0.0402 (15)	0.092 (2)	0.0011 (13)	0.0372 (18)	0.0043 (16)
O2	0.0588 (17)	0.0435 (16)	0.091 (2)	0.0050 (13)	0.0285 (17)	−0.0018 (16)
C13	0.0461 (19)	0.045 (2)	0.045 (2)	0.0024 (17)	0.0071 (18)	−0.0055 (18)
C6	0.0461 (19)	0.0359 (18)	0.038 (2)	−0.0023 (17)	−0.0044 (17)	−0.0062 (17)
C5	0.049 (2)	0.041 (2)	0.049 (2)	−0.0028 (17)	0.0085 (19)	0.0007 (18)
C4	0.060 (2)	0.044 (2)	0.046 (2)	−0.0035 (18)	0.008 (2)	0.0064 (18)
C3	0.063 (2)	0.0385 (19)	0.048 (2)	0.0012 (17)	−0.007 (2)	0.0031 (18)
C2	0.0444 (19)	0.0402 (19)	0.043 (2)	0.0068 (15)	−0.0017 (17)	−0.0013 (17)
C1	0.0384 (17)	0.0371 (17)	0.037 (2)	−0.0025 (14)	−0.0024 (17)	−0.0030 (16)
N	0.0464 (17)	0.0346 (15)	0.0555 (19)	−0.0008 (13)	0.0066 (15)	−0.0099 (15)
C7	0.0339 (17)	0.0378 (17)	0.049 (2)	−0.0029 (14)	−0.0030 (16)	−0.0041 (18)

C8	0.0448 (19)	0.0363 (18)	0.052 (2)	−0.0058 (15)	−0.0018 (17)	−0.0042 (17)
C9	0.065 (2)	0.042 (2)	0.052 (3)	−0.0191 (19)	0.008 (2)	−0.0043 (19)
C10	0.049 (2)	0.047 (2)	0.059 (3)	−0.0076 (18)	0.015 (2)	−0.0035 (19)
C11	0.0401 (17)	0.0319 (18)	0.046 (2)	−0.0004 (14)	0.0026 (16)	0.0001 (16)
C12	0.0359 (16)	0.0380 (17)	0.046 (2)	−0.0066 (14)	0.0041 (17)	0.0014 (17)
O3	0.0650 (17)	0.0355 (14)	0.0656 (18)	0.0000 (12)	0.0215 (15)	−0.0052 (14)
O1'	0.0770 (19)	0.0384 (15)	0.101 (3)	−0.0051 (14)	0.037 (2)	−0.0025 (17)
O2'	0.0670 (18)	0.0403 (15)	0.087 (2)	−0.0120 (13)	0.0299 (18)	−0.0014 (15)
C13'	0.0424 (19)	0.041 (2)	0.045 (2)	0.0014 (16)	0.0018 (18)	0.0038 (17)
C6'	0.0377 (17)	0.0398 (18)	0.037 (2)	0.0003 (15)	−0.0006 (16)	0.0032 (17)
C5'	0.049 (2)	0.048 (2)	0.038 (2)	−0.0001 (17)	0.0035 (18)	0.0046 (17)
C4'	0.063 (2)	0.0412 (18)	0.041 (2)	0.0035 (17)	0.004 (2)	−0.0007 (17)
C3'	0.066 (2)	0.0405 (19)	0.045 (2)	−0.0042 (17)	−0.001 (2)	−0.0029 (18)
C2'	0.046 (2)	0.049 (2)	0.050 (2)	−0.0092 (16)	0.0038 (18)	0.001 (2)
C1'	0.0395 (17)	0.0369 (17)	0.041 (2)	−0.0013 (14)	−0.0009 (18)	0.0038 (17)
N'	0.0452 (16)	0.0347 (15)	0.057 (2)	0.0013 (13)	0.0130 (16)	0.0070 (14)
C7'	0.0365 (17)	0.0368 (18)	0.041 (2)	−0.0046 (15)	−0.0048 (16)	−0.0014 (17)
C8'	0.0465 (19)	0.0352 (18)	0.051 (2)	0.0062 (15)	−0.0039 (17)	0.0052 (17)
C9'	0.063 (2)	0.036 (2)	0.058 (3)	0.0126 (18)	0.008 (2)	0.0006 (19)
C10'	0.051 (2)	0.048 (2)	0.067 (3)	0.0101 (18)	0.020 (2)	0.003 (2)
C11'	0.0426 (19)	0.0365 (19)	0.043 (2)	−0.0018 (15)	−0.0009 (17)	0.0003 (17)
C12'	0.0393 (17)	0.0324 (17)	0.053 (2)	0.0041 (14)	0.0028 (18)	0.0022 (17)
O3'	0.0614 (17)	0.0407 (15)	0.0662 (19)	0.0029 (12)	0.0232 (16)	0.0095 (13)

Geometric parameters (Å, °)

O1—C13	1.219 (5)	O1'—C13'	1.216 (4)
O2—C13	1.304 (4)	O2'—C13'	1.295 (4)
O2—H2O	0.8200	O2'—H2O'	0.8200
C13—C6	1.498 (5)	C13'—C6'	1.490 (5)
C6—C5	1.393 (5)	C6'—C5'	1.392 (5)
C6—C1	1.401 (5)	C6'—C1'	1.421 (5)
C5—C4	1.370 (6)	C5'—C4'	1.378 (5)
C5—H5	0.9300	C5'—H5'	0.9300
C4—C3	1.379 (6)	C4'—C3'	1.374 (6)
C4—H4	0.9300	C4'—H4'	0.9300
C3—C2	1.370 (5)	C3'—C2'	1.380 (6)
C3—H3	0.9300	C3'—H3'	0.9300
C2—C1	1.402 (5)	C2'—C1'	1.395 (5)
C2—H2	0.9300	C2'—H2'	0.9300
C1—N	1.407 (5)	C1'—N'	1.396 (5)
N—C7	1.359 (5)	N'—C7'	1.366 (5)
N—H1	0.8600	N'—H1'	0.8600
C7—C12	1.364 (5)	C7'—C12'	1.358 (5)
C7—C8	1.504 (5)	C7'—C8'	1.500 (5)
C8—C9	1.514 (6)	C8'—C9'	1.513 (6)
C8—H8A	0.9700	C8'—H8'A	0.9700
C8—H8B	0.9700	C8'—H8'B	0.9700
C9—C10	1.524 (6)	C9'—C10'	1.515 (5)

C9—H9A	0.9700	C9'—H9'A	0.9700
C9—H9B	0.9700	C9'—H9'B	0.9700
C10—C11	1.494 (5)	C10'—C11'	1.499 (5)
C10—H10A	0.9700	C10'—H10C	0.9700
C10—H10B	0.9700	C10'—H10D	0.9700
C11—O3	1.234 (4)	C11'—O3'	1.242 (4)
C11—C12	1.433 (5)	C11'—C12'	1.425 (5)
C12—H12	0.9300	C12'—H12'	0.9300
C13—O2—H2O	109.5	C13'—O2'—H2O'	109.5
O1—C13—O2	123.1 (4)	O1'—C13'—O2'	122.8 (3)
O1—C13—C6	124.3 (4)	O1'—C13'—C6'	124.5 (3)
O2—C13—C6	112.6 (3)	O2'—C13'—C6'	112.7 (3)
C5—C6—C1	119.0 (4)	C5'—C6'—C1'	119.0 (3)
C5—C6—C13	118.5 (3)	C5'—C6'—C13'	119.4 (3)
C1—C6—C13	122.4 (3)	C1'—C6'—C13'	121.6 (3)
C4—C5—C6	121.8 (4)	C4'—C5'—C6'	121.6 (4)
C4—C5—H5	119.1	C4'—C5'—H5'	119.2
C6—C5—H5	119.1	C6'—C5'—H5'	119.2
C5—C4—C3	118.5 (4)	C3'—C4'—C5'	119.1 (4)
C5—C4—H4	120.8	C3'—C4'—H4'	120.4
C3—C4—H4	120.8	C5'—C4'—H4'	120.4
C2—C3—C4	121.8 (4)	C4'—C3'—C2'	121.0 (4)
C2—C3—H3	119.1	C4'—C3'—H3'	119.5
C4—C3—H3	119.1	C2'—C3'—H3'	119.5
C3—C2—C1	119.9 (4)	C3'—C2'—C1'	120.8 (4)
C3—C2—H2	120.0	C3'—C2'—H2'	119.6
C1—C2—H2	120.0	C1'—C2'—H2'	119.6
C6—C1—C2	118.9 (4)	C2'—C1'—N'	122.4 (3)
C6—C1—N	118.7 (3)	C2'—C1'—C6'	118.3 (3)
C2—C1—N	122.3 (3)	N'—C1'—C6'	119.2 (3)
C7—N—C1	131.6 (3)	C7'—N'—C1'	130.7 (3)
C7—N—H1	114.2	C7'—N'—H1'	114.6
C1—N—H1	114.2	C1'—N'—H1'	114.6
N—C7—C12	125.9 (3)	C12'—C7'—N'	126.0 (3)
N—C7—C8	113.2 (3)	C12'—C7'—C8'	120.7 (3)
C12—C7—C8	121.0 (3)	N'—C7'—C8'	113.3 (3)
C7—C8—C9	113.6 (3)	C7'—C8'—C9'	112.9 (3)
C7—C8—H8A	108.8	C7'—C8'—H8'A	109.0
C9—C8—H8A	108.8	C9'—C8'—H8'A	109.0
C7—C8—H8B	108.8	C7'—C8'—H8'B	109.0
C9—C8—H8B	108.8	C9'—C8'—H8'B	109.0
H8A—C8—H8B	107.7	H8'A—C8'—H8'B	107.8
C8—C9—C10	109.7 (4)	C8'—C9'—C10'	109.3 (4)
C8—C9—H9A	109.7	C8'—C9'—H9'A	109.8
C10—C9—H9A	109.7	C10'—C9'—H9'A	109.8
C8—C9—H9B	109.7	C8'—C9'—H9'B	109.8
C10—C9—H9B	109.7	C10'—C9'—H9'B	109.8
H9A—C9—H9B	108.2	H9'A—C9'—H9'B	108.3
C11—C10—C9	112.0 (3)	C11'—C10'—C9'	112.0 (3)

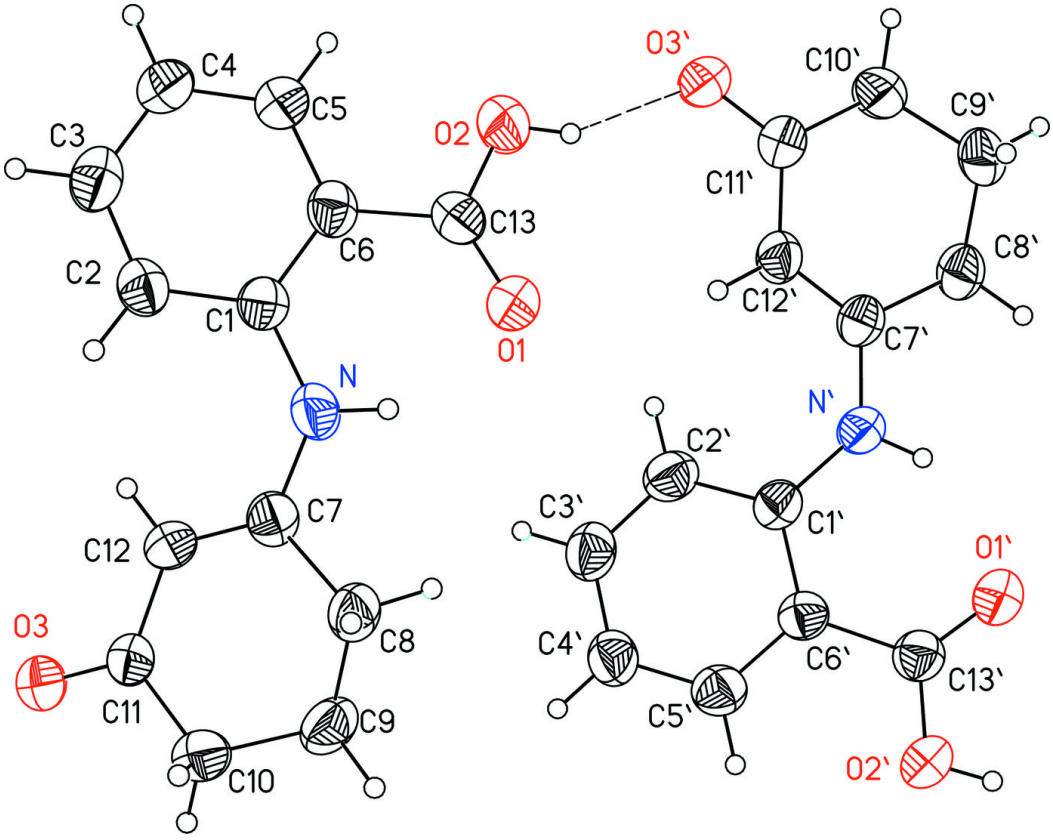
C11—C10—H10A	109.2	C11'—C10'—H10C	109.2
C9—C10—H10A	109.2	C9'—C10'—H10C	109.2
C11—C10—H10B	109.2	C11'—C10'—H10D	109.2
C9—C10—H10B	109.2	C9'—C10'—H10D	109.2
H10A—C10—H10B	107.9	H10C—C10'—H10D	107.9
O3—C11—C12	122.3 (3)	O3'—C11'—C12'	122.3 (3)
O3—C11—C10	118.6 (3)	O3'—C11'—C10'	119.2 (3)
C12—C11—C10	119.0 (3)	C12'—C11'—C10'	118.4 (3)
C7—C12—C11	121.4 (3)	C7'—C12'—C11'	122.0 (3)
C7—C12—H12	119.3	C7'—C12'—H12'	119.0
C11—C12—H12	119.3	C11'—C12'—H12'	119.0

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2O···O3'	0.82	1.81	2.562 (4)	151
N—H1···O1	0.86	1.98	2.665 (4)	136
O2'—H2O'···O3^{i}	0.82	1.78	2.562 (4)	160
N'—H1'···O1'	0.86	1.98	2.659 (4)	135

Symmetry codes: (i) *x*, *y*+1, *z*.

Fig. 1


C4
C5
C3
O2
O3'
C10'
C9'
C11'
C13
C6
C2
C1
C12'
C8'
O1
C7'
N
N'
C2'
C7
C3'
C1'
C12
O1'
O3
C8
C4'
C6'
C13'
C11
C9
C5'
C10
O2'