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Tegaserod maleate

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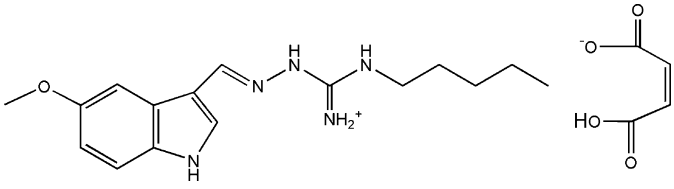
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.049; wR factor = 0.084; data-to-parameter ratio = 15.1.

The title compound, [(5-methoxyindol-3-yl)methylenehydrazino]-(pentylamino)methaniminium maleate, $C_{16}H_{24}N_5O^+\cdot C_4H_3O_4^-$, was synthesized by the reaction of tegaserod and maleic acid in anhydrous ethanol. In the tegaserod cation, the guanidyl group is protonated. A number of intermolecular N—H···O hydrogen bonds stabilize the crystal packing.

Related literature

For related crystal structures, see: Su & Zhu (2004); Sun *et al.* (2007). For biological activities of tegaserod maleate, see: Satish *et al.* (2001); Tack *et al.* (2001); Schikowski *et al.* (2002); Kamm *et al.* (2005).



Experimental

Crystal data

$C_{16}H_{24}N_5O^+\cdot C_4H_3O_4^-$
M_r = 417.47
Monoclinic, $C2/c$
a = 24.873 (3) Å
b = 8.4962 (16) Å
c = 22.294 (2) Å
β = 93.732 (2)°
V = 4701.3 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.55 × 0.42 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.954, T_{max} = 0.985
9394 measured reflections
4110 independent reflections
1423 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.084
S = 1.00
4110 reflections
273 parameters
12 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.01	2.829 (3)	159
N3—H3···O2^{ii}	0.86	1.97	2.809 (3)	164
N5—H5*B*···O1^{ii}	0.86	2.03	2.885 (3)	171
N5—H5*A*···O4	0.86	2.08	2.915 (4)	165
O3—H3*A*···O1	0.82	1.61	2.432 (3)	175

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2304).

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supplementary materials

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Comment

Tegaserod maleate is effective in the initial and retreatment of irritable bowel syndrome(IBS) and gastroesophageal reflux disease (GERO) as 5-HT4 receptor agonist (Satish *et al.*, 2001; Schikowski *et al.*, 2002). The study about tegaserod salts concentrates mainly in the clinical practice (Tack *et al.*, 2001; Kamm *et al.*, 2005), while structural studies of them have been rarely reported (Su & Zhu, 2004). As a part of our investigation on tegaserod salts, we present here the structure of the title compound, (I) (Fig. 1).

In (I), the guanidyl group is protonated. The C—N bond lengths in the guanidyl group show that the C—N bonds are conjugated, providing the planarity of guanidyl group. Similar result has been observed in other complex containing this group (Sun *et al.*, 2007).

A number of intermolecular N—H···O hydrogen bonds (Table 1) stabilize the crystal packing.

Experimental

To a solution of tegaserod (9.2 mmol) in 40 ml of anhydrous ethanol a solution of maleate(10 mmol) in 10 ml of anhydrous ethanol was added in 5 min at room temperature. The mixture was stirred for 1 h. The solvent was then evaporated and washed with anhydrous ethanol. The crude product was recrystallized from anhydrous ethanol to afford the desired product as colourless solid (m.p. 462 K). Single crystal of (I) were obtained by slow evaporation of aqueous ethanol(95%) solution at ambient temperature after 12 d.

Refinement

All hydrogen atoms were geometrically fixed at calculated positions (C—H 0.93–0.97 Å, N—H 0.86 Å, O—H 0.82 Å), and refined as riding with U_{iso}(H) = 1.2–1.5 U_{eq}(parent atom). The crystal packing contains the voids of 120 Å^3 centered at (0, 0.37, 1/4) and equivalent positions. Because of weak process of crystallization, all obtained single-crystals were of poor quality resulting in poor ratio observed/unique reflections of 0.35.

Figures

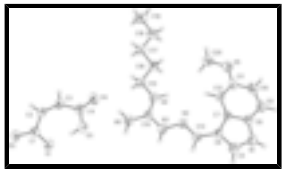


Fig. 1. The molecular structure of (I) showing atomic numbering. Displacement ellipsoids are drawn at the 30% probability level.

[(5-methoxyindol-3-yl)methylenehydrazino](pentylamino)methaniminium maleate

Crystal data

$C_{16}H_{24}N_5O^+ \cdot C_4H_3O_4^-$	F_{000} = 1776
M_r = 417.47	D_x = 1.180 Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point: 462 K
Hall symbol: -C 2yc	Mo *K*α radiation λ = 0.71073 Å
a = 24.873 (3) Å	Cell parameters from 919 reflections
b = 8.4962 (16) Å	θ = 2.4–18.6º
c = 22.294 (2) Å	μ = 0.09 mm^{-1}
β = 93.732 (2)º	*T* = 298 (2) K
V = 4701.3 (11) Å^3	Plate, white
Z = 8	0.55 × 0.42 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4110 independent reflections
Radiation source: fine-focus sealed tube	1423 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.058
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −29→29
T_{min} = 0.954, T_{max} = 0.985	*k* = −9→9
9394 measured reflections	*l* = −12→26

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.049	H-atom parameters constrained
$wR(F^2)$ = 0.084	$w = 1/[\sigma^2(F_o^2) + (0.0053P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max} < 0.001$
4110 reflections	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
273 parameters	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
12 restraints	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}
N1	0.18528 (10)	0.3231 (4)	0.26144 (12)	0.0815 (9)
H1	0.1625	0.3205	0.2308	0.098*
N2	0.31664 (11)	0.4735 (3)	0.40465 (12)	0.0720 (8)
N3	0.35352 (11)	0.5854 (3)	0.42702 (12)	0.0842 (9)
H3	0.3558	0.6767	0.4107	0.101*
N4	0.37930 (10)	0.4053 (4)	0.50059 (12)	0.0812 (9)
H4	0.3547	0.3445	0.4846	0.097*
N5	0.42242 (10)	0.6447 (3)	0.49566 (10)	0.0934 (10)
H5A	0.4434	0.6202	0.5264	0.112*
H5B	0.4257	0.7345	0.4785	0.112*
O1	0.57296 (9)	1.0683 (3)	0.57298 (9)	0.0937 (8)
O2	0.63215 (9)	1.1500 (3)	0.64577 (9)	0.0990 (9)
O3	0.51639 (8)	0.8387 (3)	0.55244 (9)	0.0880 (8)
H3A	0.5364	0.9147	0.5576	0.132*
O4	0.50672 (10)	0.6058 (3)	0.59162 (11)	0.1094 (10)
O5	0.24227 (10)	−0.1033 (3)	0.43832 (12)	0.1085 (9)
C1	0.60303 (14)	1.0445 (5)	0.62047 (16)	0.0758 (11)
C2	0.60531 (14)	0.8864 (5)	0.64804 (14)	0.0783 (11)
H2	0.6320	0.8736	0.6789	0.094*
C3	0.57583 (14)	0.7612 (5)	0.63601 (15)	0.0853 (12)
H3B	0.5854	0.6755	0.6603	0.102*
C4	0.53053 (16)	0.7283 (5)	0.59181 (18)	0.0809 (12)
C5	0.21768 (13)	0.4462 (4)	0.27763 (15)	0.0801 (10)
H5	0.2187	0.5411	0.2570	0.096*
C6	0.24901 (13)	0.4099 (5)	0.32940 (14)	0.0663 (10)
C7	0.23417 (13)	0.2540 (4)	0.34535 (15)	0.0621 (10)
C8	0.19506 (14)	0.2007 (5)	0.30291 (15)	0.0685 (10)
C9	0.17191 (14)	0.0541 (5)	0.30518 (17)	0.0864 (12)
H9	0.1454	0.0217	0.2765	0.104*
C10	0.18961 (14)	−0.0418 (5)	0.35149 (18)	0.0922 (12)
H10	0.1753	−0.1425	0.3540	0.111*
C11	0.22844 (15)	0.0072 (5)	0.39489 (17)	0.0801 (12)
C12	0.25123 (12)	0.1520 (5)	0.39251 (14)	0.0670 (10)

H12	0.2776	0.1831	0.4215	0.080*
C13	0.28742 (13)	0.5141 (4)	0.35782 (15)	0.0769 (11)
H13	0.2914	0.6144	0.3420	0.092*
C14	0.38510 (15)	0.5443 (5)	0.47484 (17)	0.0736 (11)
C15	0.41036 (12)	0.3455 (4)	0.55299 (13)	0.0786 (10)
H15A	0.4057	0.4135	0.5872	0.094*
H15B	0.4483	0.3435	0.5455	0.094*
C16	0.39137 (12)	0.1818 (4)	0.56628 (13)	0.0759 (10)
H16A	0.3963	0.1153	0.5317	0.091*
H16B	0.3531	0.1851	0.5725	0.091*
C17	0.42096 (12)	0.1093 (4)	0.62119 (14)	0.0813 (11)
H17A	0.4592	0.1057	0.6151	0.098*
H17B	0.4160	0.1754	0.6559	0.098*
C18	0.40137 (13)	−0.0553 (5)	0.63385 (15)	0.0973 (12)
H18A	0.4080	−0.1226	0.6000	0.117*
H18B	0.3628	−0.0525	0.6380	0.117*
C19	0.42889 (14)	−0.1251 (4)	0.69043 (15)	0.1351 (16)
H19A	0.4671	−0.1275	0.6867	0.203*
H19B	0.4159	−0.2302	0.6960	0.203*
H19C	0.4211	−0.0617	0.7244	0.203*
C20	0.28068 (13)	−0.0593 (4)	0.48471 (15)	0.1021 (13)
H20A	0.3133	−0.0271	0.4675	0.153*
H20B	0.2880	−0.1473	0.5110	0.153*
H20C	0.2668	0.0264	0.5071	0.153*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.066 (2)	0.108 (3)	0.0679 (19)	−0.0014 (18)	−0.0122 (16)	−0.0043 (18)
N2	0.075 (2)	0.072 (2)	0.0670 (19)	−0.0053 (18)	−0.0096 (17)	0.0091 (18)
N3	0.098 (2)	0.070 (2)	0.081 (2)	−0.014 (2)	−0.0259 (18)	0.0077 (18)
N4	0.098 (2)	0.073 (3)	0.070 (2)	−0.022 (2)	−0.0145 (18)	0.0087 (18)
N5	0.120 (2)	0.080 (2)	0.078 (2)	−0.024 (2)	−0.0150 (18)	0.0037 (18)
O1	0.1099 (19)	0.096 (2)	0.0706 (15)	−0.0253 (16)	−0.0285 (14)	0.0151 (14)
O2	0.113 (2)	0.095 (2)	0.0846 (17)	−0.0356 (16)	−0.0278 (15)	0.0050 (16)
O3	0.0922 (18)	0.098 (2)	0.0719 (16)	−0.0168 (16)	−0.0102 (14)	−0.0025 (15)
O4	0.122 (2)	0.087 (2)	0.116 (2)	−0.0410 (18)	−0.0185 (17)	−0.0034 (18)
O5	0.115 (2)	0.092 (2)	0.114 (2)	−0.0311 (18)	−0.0276 (18)	0.0317 (19)
C1	0.073 (3)	0.090 (4)	0.064 (3)	−0.015 (3)	0.005 (2)	0.003 (3)
C2	0.086 (3)	0.087 (3)	0.059 (2)	−0.009 (3)	−0.017 (2)	0.008 (2)
C3	0.090 (3)	0.085 (3)	0.078 (3)	−0.007 (3)	−0.018 (2)	0.009 (2)
C4	0.082 (3)	0.088 (4)	0.072 (3)	−0.009 (3)	0.000 (3)	−0.009 (3)
C5	0.074 (3)	0.098 (3)	0.067 (2)	0.000 (2)	0.0006 (17)	0.012 (2)
C6	0.061 (2)	0.081 (3)	0.055 (2)	−0.011 (2)	−0.0014 (16)	0.008 (2)
C7	0.049 (2)	0.074 (3)	0.064 (2)	−0.001 (2)	0.005 (2)	0.003 (2)
C8	0.054 (2)	0.090 (3)	0.061 (2)	−0.001 (2)	0.000 (2)	−0.005 (2)
C9	0.071 (3)	0.103 (4)	0.084 (3)	−0.016 (3)	−0.011 (2)	−0.010 (3)
C10	0.078 (3)	0.088 (3)	0.109 (3)	−0.026 (3)	−0.005 (3)	−0.008 (3)

C11	0.074 (3)	0.081 (4)	0.084 (3)	−0.021 (3)	−0.004 (2)	0.007 (3)
C12	0.057 (2)	0.082 (3)	0.061 (2)	−0.007 (2)	−0.0043 (19)	0.002 (2)
C13	0.076 (3)	0.079 (3)	0.074 (2)	−0.009 (2)	−0.0054 (19)	0.018 (2)
C14	0.077 (3)	0.077 (3)	0.065 (3)	−0.016 (3)	−0.008 (2)	−0.010 (3)
C15	0.090 (3)	0.087 (3)	0.056 (2)	−0.001 (2)	−0.013 (2)	0.004 (2)
C16	0.077 (2)	0.085 (3)	0.066 (2)	−0.005 (2)	0.008 (2)	0.003 (2)
C17	0.087 (3)	0.094 (3)	0.062 (2)	0.016 (2)	0.003 (2)	0.007 (2)
C18	0.096 (3)	0.107 (4)	0.090 (3)	0.011 (3)	0.017 (2)	0.024 (3)
C19	0.144 (4)	0.158 (4)	0.104 (3)	0.032 (3)	0.010 (3)	0.043 (3)
C20	0.106 (3)	0.105 (3)	0.094 (3)	−0.012 (3)	−0.004 (3)	0.037 (3)

Geometric parameters (Å, °)

N1—C5	1.355 (4)	C7—C8	1.388 (4)
N1—C8	1.402 (4)	C7—C12	1.407 (4)
N1—H1	0.8600	C8—C9	1.374 (4)
N2—C13	1.280 (3)	C9—C10	1.365 (4)
N2—N3	1.391 (3)	C9—H9	0.9300
N3—C14	1.329 (4)	C10—C11	1.386 (4)
N3—H3	0.8600	C10—H10	0.9300
N4—C14	1.325 (4)	C11—C12	1.356 (4)
N4—C15	1.450 (3)	C12—H12	0.9300
N4—H4	0.8600	C13—H13	0.9300
N5—C14	1.322 (4)	C15—C16	1.504 (4)
N5—H5A	0.8600	C15—H15A	0.9700
N5—H5B	0.8600	C15—H15B	0.9700
O1—C1	1.272 (3)	C16—C17	1.518 (3)
O2—C1	1.262 (4)	C16—H16A	0.9700
O3—C4	1.316 (4)	C16—H16B	0.9700
O3—H3A	0.8200	C17—C18	1.513 (4)
O4—C4	1.198 (4)	C17—H17A	0.9700
O5—C11	1.376 (4)	C17—H17B	0.9700
O5—C20	1.412 (3)	C18—C19	1.516 (4)
C1—C2	1.477 (4)	C18—H18A	0.9700
C2—C3	1.309 (4)	C18—H18B	0.9700
C2—H2	0.9300	C19—H19A	0.9600
C3—C4	1.475 (4)	C19—H19B	0.9600
C3—H3B	0.9300	C19—H19C	0.9600
C5—C6	1.385 (4)	C20—H20A	0.9600
C5—H5	0.9300	C20—H20B	0.9600
C6—C13	1.421 (4)	C20—H20C	0.9600
C6—C7	1.426 (4)		
C5—N1—C8	108.8 (3)	O5—C11—C10	114.6 (4)
C5—N1—H1	125.6	C11—C12—C7	118.8 (3)
C8—N1—H1	125.6	C11—C12—H12	120.6
C13—N2—N3	115.8 (3)	C7—C12—H12	120.6
C14—N3—N2	116.7 (3)	N2—C13—C6	121.7 (3)
C14—N3—H3	121.6	N2—C13—H13	119.2
N2—N3—H3	121.6	C6—C13—H13	119.2

C14—N4—C15	126.4 (3)	N5—C14—N4	121.1 (4)
C14—N4—H4	116.8	N5—C14—N3	118.5 (4)
C15—N4—H4	116.8	N4—C14—N3	120.4 (4)
C14—N5—H5A	120.0	N4—C15—C16	109.0 (3)
C14—N5—H5B	120.0	N4—C15—H15A	109.9
H5A—N5—H5B	120.0	C16—C15—H15A	109.9
C4—O3—H3A	109.5	N4—C15—H15B	109.9
C11—O5—C20	117.4 (3)	C16—C15—H15B	109.9
O2—C1—O1	123.2 (4)	H15A—C15—H15B	108.3
O2—C1—C2	117.0 (4)	C15—C16—C17	113.1 (3)
O1—C1—C2	119.7 (4)	C15—C16—H16A	109.0
C3—C2—C1	130.4 (4)	C17—C16—H16A	109.0
C3—C2—H2	114.8	C15—C16—H16B	109.0
C1—C2—H2	114.8	C17—C16—H16B	109.0
C2—C3—C4	133.2 (4)	H16A—C16—H16B	107.8
C2—C3—H3B	113.4	C18—C17—C16	112.4 (3)
C4—C3—H3B	113.4	C18—C17—H17A	109.1
O4—C4—O3	120.4 (4)	C16—C17—H17A	109.1
O4—C4—C3	121.6 (4)	C18—C17—H17B	109.1
O3—C4—C3	118.0 (4)	C16—C17—H17B	109.1
N1—C5—C6	110.1 (3)	H17A—C17—H17B	107.9
N1—C5—H5	125.0	C17—C18—C19	112.5 (3)
C6—C5—H5	125.0	C17—C18—H18A	109.1
C5—C6—C13	124.0 (4)	C19—C18—H18A	109.1
C5—C6—C7	105.9 (3)	C17—C18—H18B	109.1
C13—C6—C7	130.2 (3)	C19—C18—H18B	109.1
C8—C7—C12	118.2 (4)	H18A—C18—H18B	107.8
C8—C7—C6	108.3 (4)	C18—C19—H19A	109.5
C12—C7—C6	133.5 (4)	C18—C19—H19B	109.5
C9—C8—C7	123.2 (4)	H19A—C19—H19B	109.5
C9—C8—N1	129.9 (4)	C18—C19—H19C	109.5
C7—C8—N1	106.9 (4)	H19A—C19—H19C	109.5
C10—C9—C8	117.0 (4)	H19B—C19—H19C	109.5
C10—C9—H9	121.5	O5—C20—H20A	109.5
C8—C9—H9	121.5	O5—C20—H20B	109.5
C9—C10—C11	121.6 (4)	H20A—C20—H20B	109.5
C9—C10—H10	119.2	O5—C20—H20C	109.5
C11—C10—H10	119.2	H20A—C20—H20C	109.5
C12—C11—O5	124.1 (4)	H20B—C20—H20C	109.5
C12—C11—C10	121.3 (4)		
C13—N2—N3—C14	−178.5 (3)	C8—C9—C10—C11	1.1 (5)
O2—C1—C2—C3	171.5 (4)	C20—O5—C11—C12	3.3 (5)
O1—C1—C2—C3	−9.2 (6)	C20—O5—C11—C10	−178.5 (3)
C1—C2—C3—C4	0.3 (7)	C9—C10—C11—C12	−1.2 (6)
C2—C3—C4—O4	−174.7 (4)	C9—C10—C11—O5	−179.5 (3)
C2—C3—C4—O3	4.7 (6)	O5—C11—C12—C7	179.1 (3)
C8—N1—C5—C6	−0.3 (4)	C10—C11—C12—C7	1.0 (5)
N1—C5—C6—C13	179.7 (3)	C8—C7—C12—C11	−0.7 (5)
N1—C5—C6—C7	−0.1 (4)	C6—C7—C12—C11	−180.0 (3)

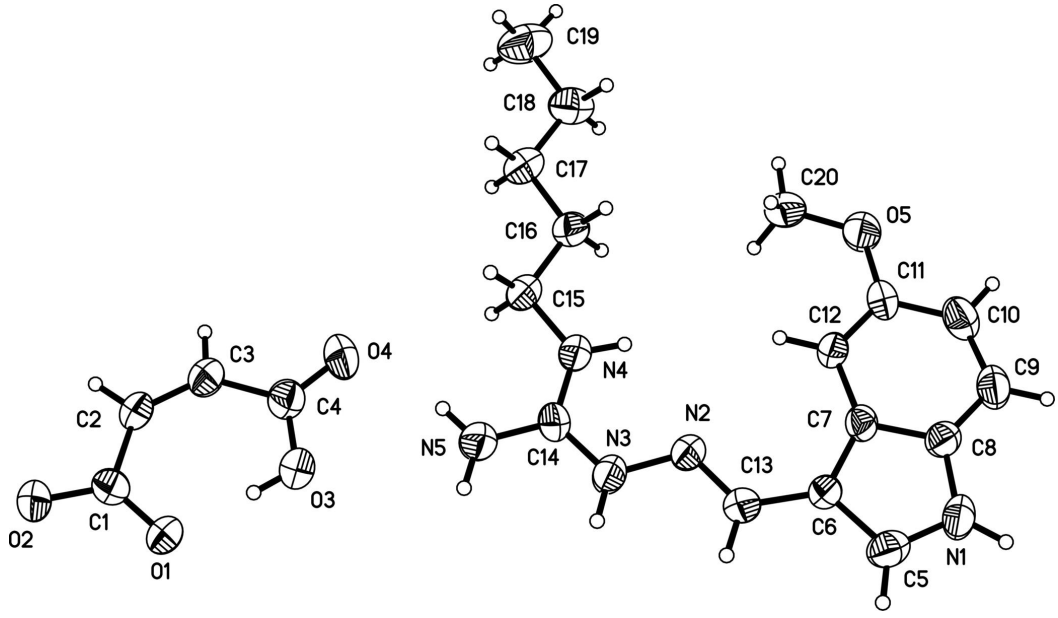
C5—C6—C7—C8	0.5 (3)	N3—N2—C13—C6	178.5 (3)
C13—C6—C7—C8	−179.3 (3)	C5—C6—C13—N2	−177.9 (3)
C5—C6—C7—C12	179.8 (3)	C7—C6—C13—N2	1.9 (5)
C13—C6—C7—C12	0.0 (6)	C15—N4—C14—N5	1.0 (5)
C12—C7—C8—C9	0.7 (5)	C15—N4—C14—N3	−179.4 (3)
C6—C7—C8—C9	−179.9 (3)	N2—N3—C14—N5	176.8 (3)
C12—C7—C8—N1	179.9 (3)	N2—N3—C14—N4	−2.8 (5)
C6—C7—C8—N1	−0.7 (4)	C14—N4—C15—C16	−178.8 (3)
C5—N1—C8—C9	179.7 (3)	N4—C15—C16—C17	−179.1 (2)
C5—N1—C8—C7	0.6 (4)	C15—C16—C17—C18	−180.0 (3)
C7—C8—C9—C10	−0.9 (5)	C16—C17—C18—C19	−177.2 (3)
N1—C8—C9—C10	−179.9 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.01	2.829 (3)	159
N3—H3···O2^{ii}	0.86	1.97	2.809 (3)	164
N5—H5B···O1^{ii}	0.86	2.03	2.885 (3)	171
N5—H5A···O4	0.86	2.08	2.915 (4)	165
O3—H3A···O1	0.82	1.61	2.432 (3)	175

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*−1/2; (ii) −*x*+1, −*y*+2, −*z*+1.

Fig. 1


C19
C18
C17
C16
C15
N4
C14
N5
N3
N2
C13
C6
C5
N1
C7
C8
C9
C10
C12
C11
O5
C20
C3
O4
C4
C2
C1
O2
O1
O3