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N-[2-(4-Methoxyphenoxy)ethyl]-acetamide

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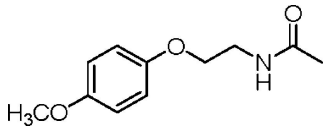
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.056; wR factor = 0.090; data-to-parameter ratio = 16.7.

The title compound, $C_{11}H_{15}NO_3$, was synthesized from 4-methoxyphenol and 2-methyl-4,5-dihydrooxazole under nitrogen. There are two independent molecules in the asymmetric unit, which are connected into one-dimensional chains *via* intermolecular N—H···O hydrogen bonds.

Related literature

Our interest in nefazodone, which is a type of antidepressant (Madding, 1986), prompted us to synthesize and determine the crystal structure of the title compound.



Experimental

Crystal data

$C_{11}H_{15}NO_3$
M_r = 209.24
Monoclinic, $P2_1/c$
a = 6.0861 (13) Å
b = 49.810 (10) Å
c = 7.4043 (15) Å
β = 90.386 (4)°
V = 2244.6 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.977, T_{max} = 0.982
12862 measured reflections
4592 independent reflections
2442 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.090
S = 1.03
4592 reflections
275 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 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
N1—H1···O6^{i}	0.86	2.00	2.863 (3)	175
N2—H2···O3^{ii}	0.86	2.00	2.861 (3)	174

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); 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: LH2385).

References

Bruker (1997). *SADABS* (Version 2.0), *SMART* (Version 5.611), *SAINT* (Version 6.0) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Madding, G. D. (1986). US Patent No. 4 596 884.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

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N-[2-(4-Methoxyphenoxy)ethyl]acetamide

Z.-H. Shang, H.-J. Lv and J.-L. Gao

Comment

Nefazodone is a type of antidepressive drug (Madding, 1986). The title compound, which can be hydrolized to 2-(4-methoxyphenoxy)ethanamine, was obtained in order to attempt to synthesize compounds analogous to Nefazodone. *N*-(2-(4-methoxyphenoxy)ethyl)acetamide was synthesized from 4-methoxyphenol and 2-methyl-4,5-dihydrooxazole under reflux under N_2. The crystal structure is stabilized mainly through intermolecular N—H···O hydrogen bonds.

Experimental

The mixture of 4-bromophenol 12.4 g (0.10 mol) and 2-methyl-4,5-dihydrooxazole 1.2 g(0.12 mol) was slowly heated to reflux under nitrogen and maintained for 7 h while monitored by TLC. After completion of the reaction, excess 2-methyl-4,5-dihydrooxazole was recovered by distillation and the residue was cooled to room temperature and added 50 ml NaOH solution(10%). The product was filtered and purifed by by column chromatography. 50 mg of the title compound was dissolved in 20 ml me thanol; the solution was kept at room temperature for 20 d by natural evaporation to give colorless single crystals of (I), suitable for X-Ray analysis.

Refinement

H atoms were positioned geometrically, with C—H = 0.93–0.97Å and N—H = 0.86 Å. They were refined in a riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(C) for methyl H atoms.

Figures

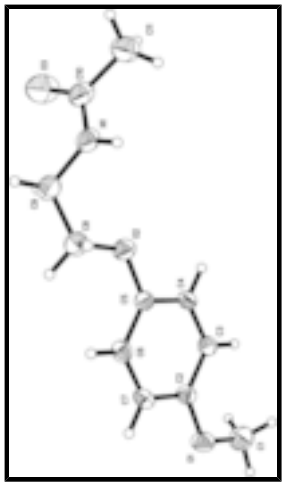

Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids.

Fig. 2. The reaction scheme for the formation of the title compound.

N-[2-(4-Methoxyphenoxy)ethyl]acetamide

Crystal data

$C_{11}H_{15}NO_3$	F_{000} = 896
M_r = 209.24	D_x = 1.238 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 496-498 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 6.0861 (13) Å	Cell parameters from 2481 reflections
b = 49.810 (10) Å	θ = 2045–23.0º
c = 7.4043 (15) Å	μ = 0.09 mm^{-1}
β = 90.386 (4)º	T = 294 (2) K
V = 2244.6 (8) Å^3	Block, colorless
Z = 8	0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer	4592 independent reflections
Radiation source: fine-focus sealed tube	2442 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.043
T = 294(2) K	θ_{max} = 26.4º
φ and ω scans	θ_{min} = 0.8º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	h = −7→7
T_{min} = 0.977, T_{max} = 0.982	k = −41→62
12862 measured reflections	l = −8→9

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.056	$w = 1/[\sigma^2(F_o^2) + (0.0796P)^2 + 0.1165P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.090	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.03	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
4592 reflections	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
275 parameters	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	0.1801 (3)	0.21713 (3)	0.4769 (2)	0.0560 (5)
O2	0.4970 (3)	0.11353 (3)	0.4256 (2)	0.0540 (5)
O3	0.7789 (4)	0.04205 (5)	0.1509 (3)	0.1111 (10)
O4	0.6768 (3)	0.21834 (3)	0.9432 (2)	0.0559 (5)
O5	0.9966 (3)	0.11483 (3)	0.9755 (2)	0.0490 (5)
O6	1.3017 (4)	0.05180 (5)	0.6685 (3)	0.1050 (9)
N1	0.6282 (4)	0.05968 (4)	0.3971 (3)	0.0567 (6)
H1	0.5282	0.0583	0.4781	0.068*
N2	1.1293 (4)	0.06004 (4)	0.9255 (3)	0.0544 (6)
H2	1.0231	0.0558	0.9959	0.065*
C1	−0.0118 (5)	0.22455 (6)	0.3806 (5)	0.0744 (9)
H1A	0.0125	0.2224	0.2535	0.112*
H1B	−0.0464	0.2430	0.4058	0.112*
H1C	−0.1318	0.2133	0.4171	0.112*
C2	0.2497 (4)	0.19099 (5)	0.4623 (3)	0.0396 (6)
C3	0.1409 (4)	0.17102 (5)	0.3673 (3)	0.0430 (6)
H3	0.0100	0.1748	0.3070	0.052*
C4	0.2280 (4)	0.14543 (5)	0.3624 (3)	0.0415 (6)
H4	0.1543	0.1320	0.2988	0.050*
C5	0.4219 (4)	0.13936 (5)	0.4501 (3)	0.0387 (6)
C6	0.5288 (4)	0.15904 (5)	0.5493 (3)	0.0435 (6)
H6	0.6575	0.1551	0.6121	0.052*
C7	0.4420 (4)	0.18470 (5)	0.5540 (3)	0.0435 (6)
H7	0.5141	0.1980	0.6200	0.052*
C8	0.6947 (4)	0.10513 (5)	0.5113 (4)	0.0567 (7)
H8A	0.6660	0.1000	0.6352	0.068*
H8B	0.8006	0.1197	0.5122	0.068*
C9	0.7836 (5)	0.08171 (5)	0.4081 (5)	0.0672 (9)
H9A	0.8207	0.0875	0.2870	0.081*
H9B	0.9174	0.0755	0.4664	0.081*
C10	0.6358 (5)	0.04167 (6)	0.2659 (4)	0.0626 (8)
C11	0.4603 (5)	0.02066 (6)	0.2641 (4)	0.0761 (9)
H11A	0.3950	0.0197	0.1458	0.114*
H11B	0.3498	0.0252	0.3508	0.114*
H11C	0.5235	0.0036	0.2944	0.114*
C12	0.4858 (5)	0.22556 (6)	1.0413 (5)	0.0761 (10)
H12A	0.3651	0.2144	1.0039	0.114*
H12B	0.4507	0.2440	1.0181	0.114*

H12C	0.5127	0.2231	1.1681	0.114*
C13	0.7487 (4)	0.19226 (5)	0.9541 (3)	0.0403 (6)
C14	0.6415 (4)	0.17188 (5)	1.0475 (3)	0.0417 (6)
H14	0.5115	0.1755	1.1083	0.050*
C15	0.7294 (4)	0.14637 (5)	1.0494 (3)	0.0409 (6)
H15	0.6585	0.1328	1.1128	0.049*
C16	0.9212 (4)	0.14067 (5)	0.9585 (3)	0.0362 (6)
C17	1.0268 (4)	0.16081 (5)	0.8639 (3)	0.0425 (6)
H17	1.1557	0.1571	0.8018	0.051*
C18	0.9391 (4)	0.18638 (5)	0.8624 (3)	0.0430 (6)
H18	1.0099	0.1999	0.7985	0.052*
C19	1.2056 (4)	0.10835 (5)	0.9008 (4)	0.0509 (7)
H19A	1.3123	0.1221	0.9320	0.061*
H19B	1.1950	0.1073	0.7702	0.061*
C20	1.2753 (5)	0.08168 (5)	0.9779 (4)	0.0595 (8)
H20A	1.4227	0.0776	0.9372	0.071*
H20B	1.2794	0.0829	1.1086	0.071*
C21	1.1539 (5)	0.04652 (6)	0.7719 (4)	0.0614 (8)
C22	0.9902 (5)	0.02469 (6)	0.7364 (4)	0.0813 (10)
H22A	0.9622	0.0234	0.6089	0.122*
H22B	0.8558	0.0287	0.7978	0.122*
H22C	1.0477	0.0079	0.7796	0.122*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0594 (12)	0.0379 (10)	0.0706 (13)	0.0045 (9)	−0.0100 (10)	−0.0026 (9)
O2	0.0517 (12)	0.0447 (11)	0.0654 (12)	0.0068 (9)	−0.0082 (9)	−0.0054 (9)
O3	0.131 (2)	0.0875 (18)	0.116 (2)	−0.0183 (15)	0.0837 (19)	−0.0161 (15)
O4	0.0582 (12)	0.0401 (11)	0.0696 (12)	0.0060 (9)	0.0087 (10)	0.0064 (9)
O5	0.0467 (11)	0.0416 (11)	0.0587 (11)	0.0002 (8)	0.0091 (8)	0.0024 (8)
O6	0.122 (2)	0.1002 (19)	0.0933 (18)	−0.0285 (15)	0.0700 (17)	−0.0220 (14)
N1	0.0571 (15)	0.0468 (14)	0.0665 (16)	0.0042 (11)	0.0239 (12)	0.0071 (12)
N2	0.0595 (15)	0.0455 (13)	0.0585 (15)	0.0018 (11)	0.0219 (11)	0.0006 (12)
C1	0.066 (2)	0.059 (2)	0.098 (2)	0.0161 (16)	−0.0107 (19)	−0.0002 (18)
C2	0.0437 (15)	0.0364 (14)	0.0388 (14)	−0.0035 (11)	0.0023 (11)	0.0026 (11)
C3	0.0379 (15)	0.0461 (16)	0.0448 (15)	0.0005 (12)	−0.0062 (11)	−0.0030 (12)
C4	0.0387 (14)	0.0426 (15)	0.0432 (15)	−0.0080 (11)	−0.0006 (11)	−0.0080 (11)
C5	0.0393 (15)	0.0356 (14)	0.0412 (14)	−0.0017 (11)	0.0051 (11)	0.0003 (11)
C6	0.0399 (15)	0.0464 (16)	0.0439 (15)	−0.0019 (12)	−0.0074 (11)	0.0023 (12)
C7	0.0435 (15)	0.0437 (15)	0.0431 (15)	−0.0102 (12)	−0.0050 (12)	−0.0028 (12)
C8	0.0513 (17)	0.0485 (17)	0.0701 (19)	0.0042 (14)	−0.0048 (14)	0.0037 (14)
C9	0.0546 (18)	0.0528 (18)	0.095 (2)	0.0047 (15)	0.0157 (16)	0.0065 (17)
C10	0.071 (2)	0.0522 (18)	0.0645 (19)	0.0085 (15)	0.0244 (16)	0.0095 (16)
C11	0.088 (2)	0.064 (2)	0.076 (2)	−0.0072 (18)	0.0158 (18)	−0.0042 (17)
C12	0.067 (2)	0.0541 (19)	0.107 (3)	0.0160 (16)	0.0101 (19)	0.0039 (18)
C13	0.0418 (15)	0.0380 (14)	0.0410 (14)	−0.0040 (11)	−0.0065 (11)	0.0020 (11)
C14	0.0365 (14)	0.0447 (16)	0.0438 (15)	−0.0014 (11)	0.0039 (11)	0.0019 (12)

C15	0.0384 (14)	0.0415 (14)	0.0429 (14)	−0.0084 (11)	0.0022 (11)	0.0063 (11)
C16	0.0361 (14)	0.0357 (14)	0.0366 (13)	−0.0033 (11)	−0.0021 (11)	−0.0003 (11)
C17	0.0360 (14)	0.0510 (16)	0.0407 (14)	−0.0059 (12)	0.0062 (11)	0.0011 (12)
C18	0.0446 (15)	0.0425 (15)	0.0419 (14)	−0.0068 (12)	0.0024 (12)	0.0044 (12)
C19	0.0412 (15)	0.0460 (15)	0.0654 (17)	−0.0024 (12)	0.0050 (13)	−0.0037 (14)
C20	0.0557 (18)	0.0529 (18)	0.0700 (19)	0.0070 (15)	−0.0026 (15)	−0.0022 (15)
C21	0.075 (2)	0.0455 (17)	0.0634 (19)	0.0015 (15)	0.0222 (16)	−0.0028 (15)
C22	0.098 (3)	0.056 (2)	0.089 (2)	−0.0116 (18)	0.013 (2)	−0.0147 (18)

Geometric parameters (Å, °)

O1—C2	1.374 (3)	C8—H8A	0.9700
O1—C1	1.413 (3)	C8—H8B	0.9700
O2—C5	1.378 (3)	C9—H9A	0.9700
O2—C8	1.420 (3)	C9—H9B	0.9700
O3—C10	1.222 (3)	C10—C11	1.496 (4)
O4—C13	1.373 (3)	C11—H11A	0.9600
O4—C12	1.421 (3)	C11—H11B	0.9600
O5—C16	1.372 (3)	C11—H11C	0.9600
O5—C19	1.428 (3)	C12—H12A	0.9600
O6—C21	1.214 (3)	C12—H12B	0.9600
N1—C10	1.323 (4)	C12—H12C	0.9600
N1—C9	1.451 (3)	C13—C18	1.379 (3)
N1—H1	0.8600	C13—C14	1.393 (3)
N2—C21	1.331 (3)	C14—C15	1.378 (3)
N2—C20	1.448 (3)	C14—H14	0.9300
N2—H2	0.8600	C15—C16	1.381 (3)
C1—H1A	0.9600	C15—H15	0.9300
C1—H1B	0.9600	C16—C17	1.384 (3)
C1—H1C	0.9600	C17—C18	1.381 (3)
C2—C3	1.385 (3)	C17—H17	0.9300
C2—C7	1.385 (3)	C18—H18	0.9300
C3—C4	1.381 (3)	C19—C20	1.506 (3)
C3—H3	0.9300	C19—H19A	0.9700
C4—C5	1.376 (3)	C19—H19B	0.9700
C4—H4	0.9300	C20—H20A	0.9700
C5—C6	1.385 (3)	C20—H20B	0.9700
C6—C7	1.384 (3)	C21—C22	1.497 (4)
C6—H6	0.9300	C22—H22A	0.9600
C7—H7	0.9300	C22—H22B	0.9600
C8—C9	1.497 (4)	C22—H22C	0.9600
C2—O1—C1	117.6 (2)	C10—C11—H11B	109.5
C5—O2—C8	119.83 (19)	H11A—C11—H11B	109.5
C13—O4—C12	118.1 (2)	C10—C11—H11C	109.5
C16—O5—C19	118.35 (18)	H11A—C11—H11C	109.5
C10—N1—C9	121.9 (2)	H11B—C11—H11C	109.5
C10—N1—H1	119.1	O4—C12—H12A	109.5
C9—N1—H1	119.1	O4—C12—H12B	109.5
C21—N2—C20	122.2 (2)	H12A—C12—H12B	109.5

C21—N2—H2	118.9	O4—C12—H12C	109.5
C20—N2—H2	118.9	H12A—C12—H12C	109.5
O1—C1—H1A	109.5	H12B—C12—H12C	109.5
O1—C1—H1B	109.5	O4—C13—C18	116.2 (2)
H1A—C1—H1B	109.5	O4—C13—C14	124.7 (2)
O1—C1—H1C	109.5	C18—C13—C14	119.2 (2)
H1A—C1—H1C	109.5	C15—C14—C13	119.6 (2)
H1B—C1—H1C	109.5	C15—C14—H14	120.2
O1—C2—C3	125.0 (2)	C13—C14—H14	120.2
O1—C2—C7	115.8 (2)	C14—C15—C16	120.9 (2)
C3—C2—C7	119.1 (2)	C14—C15—H15	119.6
C4—C3—C2	119.6 (2)	C16—C15—H15	119.6
C4—C3—H3	120.2	O5—C16—C15	115.6 (2)
C2—C3—H3	120.2	O5—C16—C17	124.7 (2)
C5—C4—C3	121.2 (2)	C15—C16—C17	119.7 (2)
C5—C4—H4	119.4	C18—C17—C16	119.4 (2)
C3—C4—H4	119.4	C18—C17—H17	120.3
C4—C5—O2	115.3 (2)	C16—C17—H17	120.3
C4—C5—C6	119.5 (2)	C13—C18—C17	121.2 (2)
O2—C5—C6	125.2 (2)	C13—C18—H18	119.4
C7—C6—C5	119.3 (2)	C17—C18—H18	119.4
C7—C6—H6	120.3	O5—C19—C20	107.6 (2)
C5—C6—H6	120.3	O5—C19—H19A	110.2
C6—C7—C2	121.2 (2)	C20—C19—H19A	110.2
C6—C7—H7	119.4	O5—C19—H19B	110.2
C2—C7—H7	119.4	C20—C19—H19B	110.2
O2—C8—C9	108.0 (2)	H19A—C19—H19B	108.5
O2—C8—H8A	110.1	N2—C20—C19	112.6 (2)
C9—C8—H8A	110.1	N2—C20—H20A	109.1
O2—C8—H8B	110.1	C19—C20—H20A	109.1
C9—C8—H8B	110.1	N2—C20—H20B	109.1
H8A—C8—H8B	108.4	C19—C20—H20B	109.1
N1—C9—C8	112.4 (2)	H20A—C20—H20B	107.8
N1—C9—H9A	109.1	O6—C21—N2	121.1 (3)
C8—C9—H9A	109.1	O6—C21—C22	122.8 (3)
N1—C9—H9B	109.1	N2—C21—C22	116.1 (3)
C8—C9—H9B	109.1	C21—C22—H22A	109.5
H9A—C9—H9B	107.9	C21—C22—H22B	109.5
O3—C10—N1	122.0 (3)	H22A—C22—H22B	109.5
O3—C10—C11	121.1 (3)	C21—C22—H22C	109.5
N1—C10—C11	116.9 (3)	H22A—C22—H22C	109.5
C10—C11—H11A	109.5	H22B—C22—H22C	109.5
C1—O1—C2—C3	−3.2 (3)	C12—O4—C13—C18	−177.6 (2)
C1—O1—C2—C7	178.0 (2)	C12—O4—C13—C14	3.4 (4)
O1—C2—C3—C4	179.9 (2)	O4—C13—C14—C15	−179.9 (2)
C7—C2—C3—C4	−1.3 (3)	C18—C13—C14—C15	1.1 (4)
C2—C3—C4—C5	−0.2 (4)	C13—C14—C15—C16	−0.6 (4)
C3—C4—C5—O2	−176.3 (2)	C19—O5—C16—C15	−173.5 (2)
C3—C4—C5—C6	1.9 (4)	C19—O5—C16—C17	4.2 (3)

C8—O2—C5—C4	−179.5 (2)	C14—C15—C16—O5	177.8 (2)
C8—O2—C5—C6	2.5 (3)	C14—C15—C16—C17	−0.1 (4)
C4—C5—C6—C7	−1.9 (3)	O5—C16—C17—C18	−177.4 (2)
O2—C5—C6—C7	176.0 (2)	C15—C16—C17—C18	0.3 (3)
C5—C6—C7—C2	0.4 (4)	O4—C13—C18—C17	180.0 (2)
O1—C2—C7—C6	−179.9 (2)	C14—C13—C18—C17	−0.9 (4)
C3—C2—C7—C6	1.2 (4)	C16—C17—C18—C13	0.2 (4)
C5—O2—C8—C9	−157.5 (2)	C16—O5—C19—C20	165.5 (2)
C10—N1—C9—C8	155.8 (3)	C21—N2—C20—C19	86.4 (3)
O2—C8—C9—N1	−57.7 (3)	O5—C19—C20—N2	63.7 (3)
C9—N1—C10—O3	2.8 (5)	C20—N2—C21—O6	0.1 (4)
C9—N1—C10—C11	−177.2 (3)	C20—N2—C21—C22	180.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O6^{i}	0.86	2.00	2.863 (3)	175
N2—H2···O3^{ii}	0.86	2.00	2.861 (3)	174

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

Fig. 1

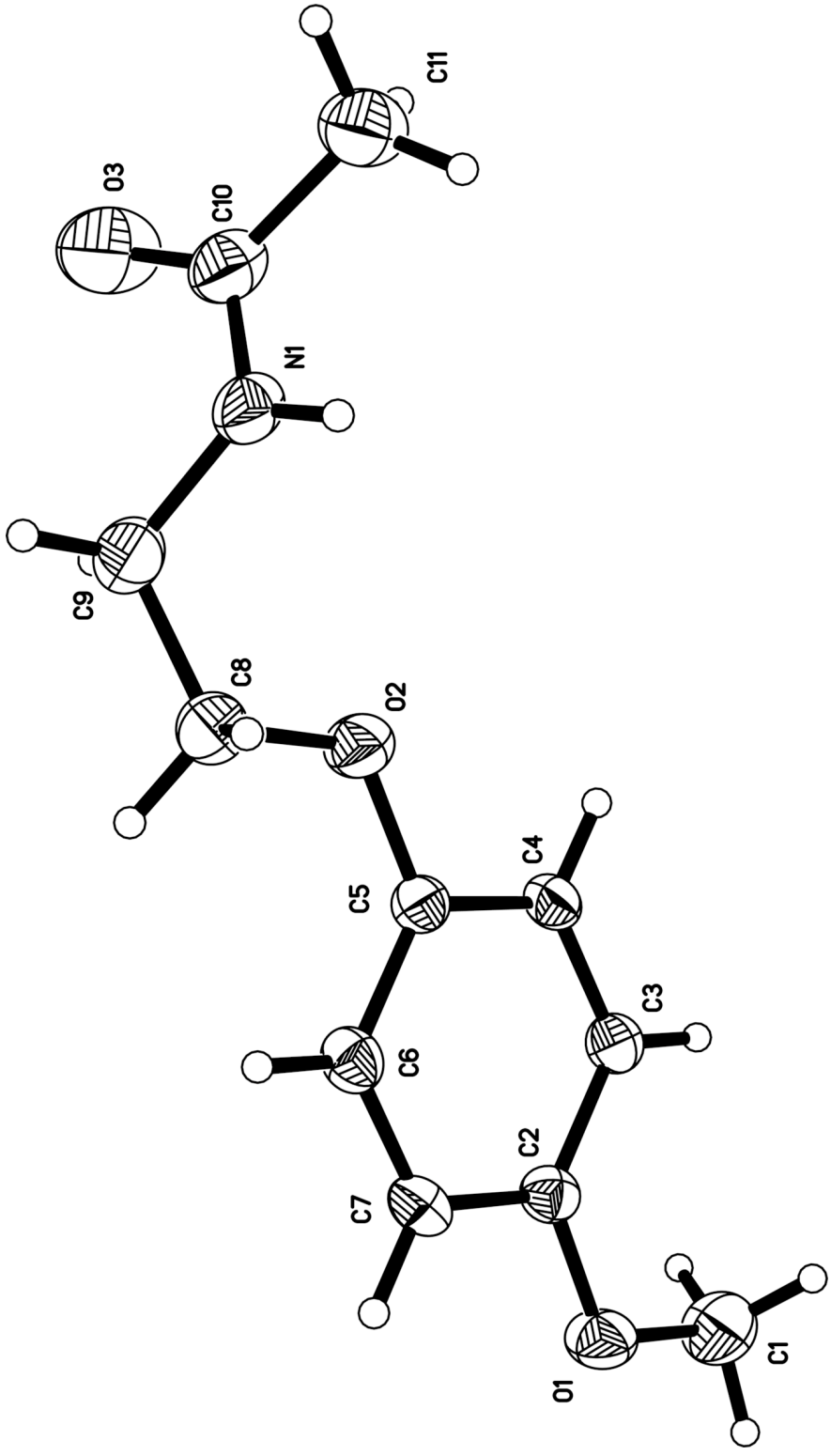

Fig. 2