Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,3-Dibromo-1-(4-methylphenyl)-3-(5-nitrofuran-2-yl)propan-1-one

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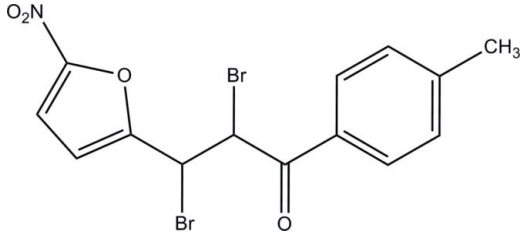
Received 25 November 2010; accepted 2 December 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ() = 0.000 Å; disorder in main residue; R factor = 0.041; wR factor = 0.103; data-to-parameter ratio = 12.6.

In the title compound, $C_{14}H_{11}Br_2NO_4$, the whole molecule is disordered over two positions with a refined occupancy ratio of 0.539 (9):0.461 (9). The 2-nitrofuran and toluene groups are approximately planar, with maximum deviations of 0.176 (11) and 0.121 (14) Å, respectively, in the major component and 0.208 (11) and 0.30 (17) Å in the minor component. The dihedral angles between the 2-nitrofuran and toluene groups are 8.7 (5) and 8.0 (9)° for the major and minor components, respectively. In the crystal, weak intermolecular C—H···O interactions connect molecules into a three-dimensional network, generating $R_2^1(6)$ ring motifs.

Related literature

For the biological activity of nitrofurans, see: Holla *et al.* (1986, 1987, 1992); Hegde *et al.* (2006); Rai *et al.* (2008). For a related structure, see: Fun *et al.* (2010). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986). For standard bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{14}H_{11}Br_2NO_4$ M_r = 417.06
Triclinic, $P\overline{1}$
a = 8.7766 (3) Å
b = 9.0386 (3) Å
c = 10.4841 (3) Å
α = 87.601 (2)°
β = 75.505 (2)°
γ = 69.554 (2)°
V = 753.53 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 5.39 mm^{-1}
T = 100 K
0.47 × 0.21 × 0.13 mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.184, T_{max} = 0.550
10357 measured reflections
3465 independent reflections
2729 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 1.19
3465 reflections
274 parameters
658 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2A—H2AA···O3A^{i}	0.93	2.53	3.210 (15)	131
C3A—H3AA···O2A^{ii}	0.93	2.51	3.216 (12)	133
C6A—H6AA···O2A^{ii}	0.98	2.33	3.217 (10)	151
C13A—H13A···O3A^{iii}	0.93	2.55	3.434 (13)	158

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

HKF and TSH thank Universiti Sains Malaysia (USM) for the Research University Grant (1001/PFIZIK/811160). TSH also thanks USM for the award of a research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5178).

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‡ Thomson Reuters ResearcherID: A-3561-2009.

supplementary materials

Acta Cryst. (2011). E**67**, o79 [**doi:10.1107/S1600536810050488**]

2,3-Dibromo-1-(4-methylphenyl)-3-(5-nitrofuran-2-yl)propan-1-one

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Comment

Nitrofurans are a class of synthetic compounds characterized by the presence of 5-nitro-2-furyl group. The presence of nitro group in position-5 of the molecule conferred antibacterial activity (Holla *et al.*1986). A number of nitrofurans have attained commercial utility as antibacterial agents in humans and in veterinary medicine because of their broad spectrum of activities (Holla & Kalluraya *et al.*, 1992; Holla *et al.*, 1987). The incorporation of 5-nitrofuran or 5-nitrothiophene moiety into various heterocyclic systems has found to increase their biological activities. We have reported few heterocyclic systems carrying a 5-nitrofuran moiety as potent antimicrobial agents (Hegde *et al.*, 2006). During the synthetic procedures, the dibromopropanones were obtained by the bromination of 1-aryl-3-(5-nitro-2-furyl)-2-propen-1-ones. Acid-catalysed condensation of acetophenones with nitrofural diacetate in acetic acid yielded the required 1-aryl-3-(5-nitro-2-furyl)-2-propen-1-ones (chalcones) (Rai *et al.*, 2008).

In the title compound (Fig. 1), the whole molecule is disordered over two positions with a refined occupancy ratio of 0539 (9):0.461 (9) The molecule consists of a 2-nitrofuran (C1–C3/C5/N1/O1/O3/O4) group, a toluene group which (C9–C15) and one 2, 3-dibromopropanal (C6–C9/Br1/Br2/O2) moiety. Both ring groups are essentially planar (maximum deviation of 0.176 (11) and 0.121 (14) Å in the major component and 0.208 (11) and 0.30 (17) Å in the minor component for the 2-nitrofuran and toluene groups respectively). The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable to a closely related structure (Fun *et al.*, 2010).

In the crystal packing (Fig. 2), intermolecular C3A—H3A···O2A^{ii} and C6A—H6A···O2A^{ii} hydrogen bonds connect neighbouring molecules generating $R_2^1(6)$ ring motifs (Bernstein *et al.*, 1995) (Table 1). These dimers are linked into a three-dimensional network by intermolecular C2A—H2AA···O3A^{i} and C13A—H13A···O3A^{iii} hydrogen bonds (Table 1).

Experimental

1-(4-Methylphenyl)-3-(5-nitro-2-furyl)-2-propen-1-one (0.01 mol) was dissolved in glacial acetic acid (25 ml) by gentle warming. A solution of bromine in glacial acetic acid (30%*w*/*v*) was added to it with constant stirring till the yellow color of the bromine persisted. The reaction mixture was kept aside at room temperature for overnight. Crystals of dibromopropanones which separated out were collected by filtration and washed with ethanol and dried and then recrystallized from glacial acetic acid. Crystals suitable for X-ray analysis were obtained from 1:2 mixtures of DMF and ethanol by slow evaporation.

Refinement

All the H atoms were positioned geometrically [C–H = 0.93 to 0.98 Å] and were refined using a riding model, with U_{iso}(H) = 1.2 U_{eq} (C). The whole molecule is disordered over two positions with a refined ratio of 0539 (9):0.461 (9) Initially rigid, similarity and simulation restraints were applied. After steady state has been reached, rigid restraints were removed for the final refinement.

Figures

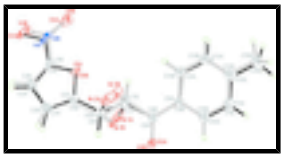

Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. The minor component of disorder is shown with open bonds.

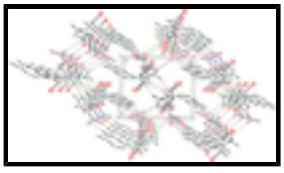

Fig. 2. The crystal packing of the title compound, viewed along *a* axis. Only the major disordered component is shown. Hydrogen atoms not involved in hydrogen boding are omitted for clarity.

2,3-Dibromo-1-(4-methylphenyl)-3-(5-nitrofuran-2-yl)propan-1-one

Crystal data

$C_{14}H_{11}Br_2NO_4$	$Z = 2$
$M_r = 417.06$	$F(000) = 408$
Triclinic, $P\overline{1}$	$D_x = 1.838$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.7766$ (3) Å	Cell parameters from 4461 reflections
$b = 9.0386$ (3) Å	$\theta = 2.8$–$29.8°$
$c = 10.4841$ (3) Å	$\mu = 5.39$ mm^{-1}
$\alpha = 87.601$ (2)°	$T = 100$ K
$\beta = 75.505$ (2)°	Block, colourless
$\gamma = 69.554$ (2)°	0.47 × 0.21 × 0.13 mm
$V = 753.53$ (4) Å^3	

Data collection

Bruker APEXII DUO CCD area-detector diffractometer	3465 independent reflections
Radiation source: fine-focus sealed tube	2729 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.026$
φ and ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)	$h = -11 \rightarrow 11$
$T_{min} = 0.184$, $T_{max} = 0.550$	$k = -11 \rightarrow 11$
10357 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.103$	H-atom parameters constrained
$S = 1.19$	$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 1.118P]$ where $P = (F_o^2 + 2F_c^2)/3$
3465 reflections	$(\Delta/\sigma)_{max} < 0.001$
274 parameters	$\Delta\rho_{max} = 0.88$ e Å^{-3}
658 restraints	$\Delta\rho_{min} = -0.40$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1A	0.3252 (8)	0.1907 (8)	0.2855 (6)	0.0247 (12)	0.539 (9)
O2A	0.7200 (11)	0.4559 (10)	0.3552 (8)	0.0263 (15)	0.539 (9)
O3A	0.0607 (10)	−0.0275 (11)	0.3127 (8)	0.0353 (18)	0.539 (9)
O4A	0.2823 (12)	−0.0290 (13)	0.1606 (10)	0.029 (2)	0.539 (9)
N1A	0.188 (3)	0.009 (3)	0.2706 (15)	0.025 (3)	0.539 (9)
C1A	0.1954 (16)	0.1367 (19)	0.3437 (13)	0.024 (2)	0.539 (9)
C2A	0.0942 (13)	0.2160 (16)	0.4548 (12)	0.024 (2)	0.539 (9)
H2AA	−0.0059	0.2062	0.5033	0.029*	0.539 (9)
C3A	0.1735 (15)	0.3187 (18)	0.4824 (14)	0.026 (3)	0.539 (9)
H3AA	0.1409	0.3841	0.5579	0.031*	0.539 (9)
C5A	0.3058 (10)	0.3035 (9)	0.3781 (8)	0.0256 (15)	0.539 (9)
C8A	0.7314 (16)	0.3690 (17)	0.2649 (12)	0.028 (2)	0.539 (9)
C9A	0.8760 (16)	0.322 (2)	0.1492 (12)	0.024 (3)	0.539 (9)
C10A	1.009 (2)	0.374 (4)	0.148 (2)	0.023 (3)	0.539 (9)
H10A	1.0027	0.4368	0.2193	0.028*	0.539 (9)
C11A	1.1495 (18)	0.334 (2)	0.0434 (16)	0.027 (2)	0.539 (9)
H11A	1.2350	0.3720	0.0449	0.032*	0.539 (9)
C12A	1.1681 (14)	0.2394 (19)	−0.0645 (13)	0.027 (2)	0.539 (9)
C13A	1.0316 (14)	0.1904 (19)	−0.0648 (12)	0.028 (3)	0.539 (9)
H13A	1.0379	0.1293	−0.1364	0.033*	0.539 (9)
C14A	0.8896 (14)	0.2312 (18)	0.0384 (11)	0.031 (3)	0.539 (9)
H14A	0.8015	0.1982	0.0348	0.037*	0.539 (9)
C15A	1.3292 (18)	0.177 (2)	−0.1713 (17)	0.049 (4)	0.539 (9)

H15A	1.4031	0.2314	−0.1629	0.073*	0.539 (9)
H15B	1.3048	0.1948	−0.2561	0.073*	0.539 (9)
H15C	1.3823	0.0660	−0.1629	0.073*	0.539 (9)
Br1A	0.6688 (5)	0.1302 (5)	0.4219 (5)	0.0395 (7)	0.539 (9)
Br2A	0.3626 (5)	0.5504 (4)	0.2175 (5)	0.0414 (6)	0.539 (9)
C6A	0.4234 (8)	0.3896 (8)	0.3487 (7)	0.0309 (16)	0.539 (9)
H6AA	0.4189	0.4424	0.4301	0.037*	0.539 (9)
C7A	0.5995 (9)	0.2863 (9)	0.2889 (7)	0.0312 (15)	0.539 (9)
H7AA	0.6066	0.2322	0.2074	0.037*	0.539 (9)
O1B	0.3468 (10)	0.1563 (9)	0.3082 (8)	0.024 (2)*	0.461 (9)
O2B	0.6979 (14)	0.4869 (11)	0.3410 (10)	0.022 (2)*	0.461 (9)
O3B	0.1011 (13)	−0.0594 (12)	0.2947 (11)	0.037 (3)*	0.461 (9)
O4B	0.3032 (19)	−0.0313 (19)	0.1354 (13)	0.037 (3)*	0.461 (9)
N1B	0.197 (4)	0.012 (4)	0.241 (2)	0.025 (3)	0.461 (9)
C1B	0.206 (2)	0.122 (2)	0.3321 (16)	0.025 (3)*	0.461 (9)
C2B	0.1002 (19)	0.194 (2)	0.4467 (16)	0.030 (3)*	0.461 (9)
H2BA	0.0011	0.1790	0.4910	0.036*	0.461 (9)
C3B	0.171 (2)	0.298 (2)	0.4858 (17)	0.031 (4)*	0.461 (9)
H3BA	0.1208	0.3734	0.5552	0.037*	0.461 (9)
C5B	0.3271 (12)	0.2658 (10)	0.4029 (9)	0.021 (2)*	0.461 (9)
C8B	0.713 (2)	0.393 (2)	0.2559 (14)	0.025 (3)*	0.461 (9)
C9B	0.860 (2)	0.340 (3)	0.1418 (16)	0.025 (4)*	0.461 (9)
C10B	1.003 (3)	0.374 (5)	0.145 (3)	0.030 (5)*	0.461 (9)
H10B	1.0002	0.4344	0.2156	0.036*	0.461 (9)
C11B	1.149 (2)	0.320 (3)	0.0433 (19)	0.029 (3)*	0.461 (9)
H11B	1.2444	0.3393	0.0480	0.035*	0.461 (9)
C12B	1.1500 (17)	0.235 (2)	−0.0665 (15)	0.026 (3)*	0.461 (9)
C13B	1.0072 (18)	0.203 (2)	−0.0705 (16)	0.029 (4)*	0.461 (9)
H13B	1.0071	0.1493	−0.1440	0.035*	0.461 (9)
C14B	0.8655 (18)	0.252 (2)	0.0331 (14)	0.028 (3)*	0.461 (9)
H14B	0.7728	0.2254	0.0308	0.033*	0.461 (9)
C15B	1.306 (2)	0.188 (2)	−0.1803 (17)	0.028 (3)*	0.461 (9)
H15D	1.4029	0.1346	−0.1477	0.043*	0.461 (9)
H15E	1.3172	0.2816	−0.2225	0.043*	0.461 (9)
H15F	1.2962	0.1196	−0.2428	0.043*	0.461 (9)
Br1B	0.6416 (5)	0.1406 (5)	0.4607 (4)	0.0297 (5)	0.461 (9)
Br2B	0.4087 (6)	0.5267 (6)	0.1895 (6)	0.0438 (8)	0.461 (9)
C6B	0.4744 (8)	0.3165 (8)	0.3932 (6)	0.0174 (16)*	0.461 (9)
H6BA	0.4387	0.4117	0.4505	0.021*	0.461 (9)
C7B	0.5614 (9)	0.3472 (9)	0.2554 (7)	0.0208 (17)*	0.461 (9)
H7BA	0.5963	0.2535	0.1965	0.025*	0.461 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.025 (3)	0.024 (3)	0.035 (3)	−0.015 (2)	−0.015 (2)	0.000 (2)
O2A	0.030 (4)	0.024 (3)	0.033 (3)	−0.015 (3)	−0.014 (3)	0.002 (3)
O3A	0.028 (4)	0.044 (4)	0.044 (4)	−0.029 (4)	0.000 (3)	−0.015 (3)

O4A	0.025 (4)	0.040 (4)	0.024 (5)	−0.018 (3)	0.000 (3)	−0.017 (3)
N1A	0.035 (3)	0.0307 (16)	0.021 (7)	−0.0187 (17)	−0.020 (4)	0.008 (5)
C1A	0.021 (3)	0.025 (4)	0.038 (4)	−0.015 (3)	−0.017 (2)	−0.001 (2)
C2A	0.021 (3)	0.025 (4)	0.038 (4)	−0.015 (3)	−0.017 (2)	−0.001 (2)
C3A	0.024 (4)	0.021 (5)	0.041 (4)	−0.016 (3)	−0.013 (3)	−0.004 (3)
C5A	0.030 (3)	0.022 (3)	0.033 (4)	−0.014 (3)	−0.014 (3)	0.003 (3)
C8A	0.024 (4)	0.028 (5)	0.043 (4)	−0.016 (4)	−0.016 (3)	0.000 (4)
C9A	0.025 (4)	0.025 (5)	0.029 (4)	−0.014 (4)	−0.014 (3)	0.001 (3)
C10A	0.030 (4)	0.027 (4)	0.026 (4)	−0.018 (3)	−0.018 (3)	0.004 (2)
C11A	0.025 (3)	0.026 (3)	0.037 (3)	−0.013 (2)	−0.015 (2)	0.005 (2)
C12A	0.025 (3)	0.026 (3)	0.037 (3)	−0.013 (2)	−0.015 (2)	0.005 (2)
C13A	0.027 (4)	0.030 (5)	0.030 (4)	−0.008 (4)	−0.016 (3)	−0.009 (3)
C14A	0.025 (4)	0.037 (6)	0.044 (4)	−0.017 (4)	−0.021 (3)	−0.005 (4)
C15A	0.033 (6)	0.059 (7)	0.044 (6)	−0.010 (5)	0.001 (5)	−0.020 (5)
Br1A	0.0302 (12)	0.0365 (8)	0.0632 (18)	−0.0172 (8)	−0.0264 (13)	0.0195 (12)
Br2A	0.0509 (16)	0.0327 (8)	0.0597 (17)	−0.0246 (10)	−0.0357 (13)	0.0189 (8)
C6A	0.033 (3)	0.031 (3)	0.037 (4)	−0.017 (3)	−0.017 (3)	0.002 (3)
C7A	0.031 (3)	0.030 (3)	0.039 (4)	−0.017 (3)	−0.012 (3)	0.001 (3)
N1B	0.035 (3)	0.0307 (16)	0.021 (7)	−0.0187 (17)	−0.020 (4)	0.008 (5)
Br1B	0.0228 (10)	0.0363 (6)	0.0371 (12)	−0.0145 (7)	−0.0151 (9)	0.0096 (9)
Br2B	0.0455 (16)	0.0513 (16)	0.0555 (18)	−0.0310 (13)	−0.0328 (14)	0.0264 (12)

Geometric parameters (Å, °)

O1A—C5A	1.375 (7)	O1B—C1B	1.343 (10)
O1A—C1A	1.387 (8)	O1B—C5B	1.368 (9)
O2A—C8A	1.218 (8)	O2B—C8B	1.207 (11)
O3A—N1A	1.243 (10)	O3B—N1B	1.251 (12)
O4A—N1A	1.222 (9)	O4B—N1B	1.228 (11)
N1A—C1A	1.439 (9)	N1B—C1B	1.436 (11)
C1A—C2A	1.329 (9)	C1B—C2B	1.347 (10)
C2A—C3A	1.416 (9)	C2B—C3B	1.413 (11)
C2A—H2AA	0.9300	C2B—H2BA	0.9300
C3A—C5A	1.352 (9)	C3B—C5B	1.364 (11)
C3A—H3AA	0.9300	C3B—H3BA	0.9300
C5A—C6A	1.465 (9)	C5B—C6B	1.496 (11)
C8A—C9A	1.469 (9)	C8B—C9B	1.472 (10)
C8A—C7A	1.550 (13)	C8B—C7B	1.527 (18)
C9A—C10A	1.405 (9)	C9B—C10B	1.399 (11)
C9A—C14A	1.406 (8)	C9B—C14B	1.401 (11)
C10A—C11A	1.380 (9)	C10B—C11B	1.391 (11)
C10A—H10A	0.9300	C10B—H10B	0.9300
C11A—C12A	1.390 (9)	C11B—C12B	1.406 (10)
C11A—H11A	0.9300	C11B—H11B	0.9300
C12A—C13A	1.416 (10)	C12B—C13B	1.388 (11)
C12A—C15A	1.508 (9)	C12B—C15B	1.515 (10)
C13A—C14A	1.377 (9)	C13B—C14B	1.379 (11)
C13A—H13A	0.9300	C13B—H13B	0.9300
C14A—H14A	0.9300	C14B—H14B	0.9300

C15A—H15A	0.9600	C15B—H15D	0.9600
C15A—H15B	0.9600	C15B—H15E	0.9600
C15A—H15C	0.9600	C15B—H15F	0.9600
Br1A—C7A	1.992 (10)	Br1B—C6B	1.994 (8)
Br2A—C6A	1.987 (8)	Br2B—C7B	1.938 (10)
C6A—C7A	1.486 (10)	C6B—C7B	1.520 (10)
C6A—H6AA	0.9800	C6B—H6BA	0.9800
C7A—H7AA	0.9800	C7B—H7BA	0.9800
C5A—O1A—C1A	101.7 (6)	O3B—N1B—C1B	111.7 (12)
O4A—N1A—O3A	124.4 (10)	O1B—C1B—C2B	110.1 (8)
O4A—N1A—C1A	118.0 (9)	O1B—C1B—N1B	117.5 (10)
O3A—N1A—C1A	115.5 (10)	C2B—C1B—N1B	132.3 (10)
C2A—C1A—O1A	114.5 (7)	C1B—C2B—C3B	105.8 (9)
C2A—C1A—N1A	130.6 (8)	C1B—C2B—H2BA	127.1
O1A—C1A—N1A	114.8 (8)	C3B—C2B—H2BA	127.1
C1A—C2A—C3A	104.2 (7)	C5B—C3B—C2B	107.5 (10)
C1A—C2A—H2AA	127.9	C5B—C3B—H3BA	126.3
C3A—C2A—H2AA	127.9	C2B—C3B—H3BA	126.3
C5A—C3A—C2A	106.9 (7)	C3B—C5B—O1B	107.6 (8)
C5A—C3A—H3AA	126.5	C3B—C5B—C6B	136.7 (9)
C2A—C3A—H3AA	126.5	O1B—C5B—C6B	115.8 (7)
C3A—C5A—O1A	112.1 (6)	O2B—C8B—C9B	123.2 (13)
C3A—C5A—C6A	130.6 (7)	O2B—C8B—C7B	118.0 (12)
O1A—C5A—C6A	117.3 (6)	C9B—C8B—C7B	118.1 (10)
O2A—C8A—C9A	122.9 (9)	C10B—C9B—C14B	118.6 (10)
O2A—C8A—C7A	115.9 (9)	C10B—C9B—C8B	118.5 (11)
C9A—C8A—C7A	120.4 (8)	C14B—C9B—C8B	122.9 (11)
C10A—C9A—C14A	117.7 (8)	C11B—C10B—C9B	121.1 (12)
C10A—C9A—C8A	117.3 (8)	C11B—C10B—H10B	119.5
C14A—C9A—C8A	125.0 (9)	C9B—C10B—H10B	119.5
C11A—C10A—C9A	120.5 (9)	C10B—C11B—C12B	119.3 (12)
C11A—C10A—H10A	119.7	C10B—C11B—H11B	120.3
C9A—C10A—H10A	119.7	C12B—C11B—H11B	120.3
C10A—C11A—C12A	122.6 (9)	C13B—C12B—C11B	119.5 (10)
C10A—C11A—H11A	118.7	C13B—C12B—C15B	122.3 (11)
C12A—C11A—H11A	118.7	C11B—C12B—C15B	118.1 (10)
C11A—C12A—C13A	116.6 (8)	C14B—C13B—C12B	120.8 (12)
C11A—C12A—C15A	123.1 (10)	C14B—C13B—H13B	119.6
C13A—C12A—C15A	120.1 (10)	C12B—C13B—H13B	119.6
C14A—C13A—C12A	121.6 (8)	C13B—C14B—C9B	120.6 (12)
C14A—C13A—H13A	119.2	C13B—C14B—H14B	119.7
C12A—C13A—H13A	119.2	C9B—C14B—H14B	119.7
C13A—C14A—C9A	120.9 (8)	C12B—C15B—H15D	109.5
C13A—C14A—H14A	119.5	C12B—C15B—H15E	109.5
C9A—C14A—H14A	119.5	H15D—C15B—H15E	109.5
C5A—C6A—C7A	113.1 (6)	C12B—C15B—H15F	109.5
C5A—C6A—Br2A	110.2 (5)	H15D—C15B—H15F	109.5
C7A—C6A—Br2A	105.6 (6)	H15E—C15B—H15F	109.5
C5A—C6A—H6AA	109.3	C5B—C6B—C7B	115.9 (6)

C7A—C6A—H6AA	109.3	C5B—C6B—Br1B	106.1 (5)
Br2A—C6A—H6AA	109.3	C7B—C6B—Br1B	107.6 (5)
C6A—C7A—C8A	115.3 (7)	C5B—C6B—H6BA	109.0
C6A—C7A—Br1A	106.7 (5)	C7B—C6B—H6BA	109.0
C8A—C7A—Br1A	102.9 (7)	Br1B—C6B—H6BA	109.0
C6A—C7A—H7AA	110.5	C6B—C7B—C8B	111.7 (8)
C8A—C7A—H7AA	110.5	C6B—C7B—Br2B	109.3 (6)
Br1A—C7A—H7AA	110.5	C8B—C7B—Br2B	105.6 (8)
C1B—O1B—C5B	108.3 (8)	C6B—C7B—H7BA	110.0
O4B—N1B—O3B	124.1 (14)	C8B—C7B—H7BA	110.0
O4B—N1B—C1B	121.7 (12)	Br2B—C7B—H7BA	110.0
C5A—O1A—C1A—C2A	5.5 (14)	C5B—O1B—C1B—C2B	−5.7 (17)
C5A—O1A—C1A—N1A	−177.3 (19)	C5B—O1B—C1B—N1B	178 (2)
O4A—N1A—C1A—C2A	169 (2)	O4B—N1B—C1B—O1B	−10 (5)
O3A—N1A—C1A—C2A	5(4)	O3B—N1B—C1B—O1B	153 (2)
O4A—N1A—C1A—O1A	−7(4)	O4B—N1B—C1B—C2B	175 (2)
O3A—N1A—C1A—O1A	−172 (2)	O3B—N1B—C1B—C2B	−23 (5)
O1A—C1A—C2A—C3A	−7.5 (16)	O1B—C1B—C2B—C3B	8.7 (19)
N1A—C1A—C2A—C3A	176 (3)	N1B—C1B—C2B—C3B	−175 (3)
C1A—C2A—C3A—C5A	6.3 (17)	C1B—C2B—C3B—C5B	−8(2)
C2A—C3A—C5A—O1A	−3.3 (17)	C2B—C3B—C5B—O1B	5(2)
C2A—C3A—C5A—C6A	174.7 (11)	C2B—C3B—C5B—C6B	−173.2 (13)
C1A—O1A—C5A—C3A	−1.1 (14)	C1B—O1B—C5B—C3B	0.1 (17)
C1A—O1A—C5A—C6A	−179.3 (11)	C1B—O1B—C5B—C6B	178.9 (13)
O2A—C8A—C9A—C10A	3(3)	O2B—C8B—C9B—C10B	13 (4)
C7A—C8A—C9A—C10A	−166 (2)	C7B—C8B—C9B—C10B	−176 (3)
O2A—C8A—C9A—C14A	−175.3 (16)	O2B—C8B—C9B—C14B	−170 (2)
C7A—C8A—C9A—C14A	15 (3)	C7B—C8B—C9B—C14B	1(3)
C14A—C9A—C10A—C11A	−2(4)	C14B—C9B—C10B—C11B	−1(5)
C8A—C9A—C10A—C11A	180 (2)	C8B—C9B—C10B—C11B	176 (3)
C9A—C10A—C11A—C12A	−1(4)	C9B—C10B—C11B—C12B	3(5)
C10A—C11A—C12A—C13A	3(3)	C10B—C11B—C12B—C13B	−1(4)
C10A—C11A—C12A—C15A	−172 (3)	C10B—C11B—C12B—C15B	175 (3)
C11A—C12A—C13A—C14A	−2(2)	C11B—C12B—C13B—C14B	−1(3)
C15A—C12A—C13A—C14A	172.9 (17)	C15B—C12B—C13B—C14B	−177.8 (19)
C12A—C13A—C14A—C9A	−1(3)	C12B—C13B—C14B—C9B	3(3)
C10A—C9A—C14A—C13A	2(3)	C10B—C9B—C14B—C13B	−2(4)
C8A—C9A—C14A—C13A	−178.9 (17)	C8B—C9B—C14B—C13B	−179 (2)
C3A—C5A—C6A—C7A	140.5 (14)	C3B—C5B—C6B—C7B	−138.9 (18)
O1A—C5A—C6A—C7A	−41.6 (10)	O1B—C5B—C6B—C7B	42.8 (10)
C3A—C5A—C6A—Br2A	−101.6 (14)	C3B—C5B—C6B—Br1B	101.8 (18)
O1A—C5A—C6A—Br2A	76.3 (8)	O1B—C5B—C6B—Br1B	−76.5 (8)
C5A—C6A—C7A—C8A	−175.9 (8)	C5B—C6B—C7B—C8B	−178.2 (9)
Br2A—C6A—C7A—C8A	63.6 (8)	Br1B—C6B—C7B—C8B	−59.7 (9)
C5A—C6A—C7A—Br1A	−62.3 (6)	C5B—C6B—C7B—Br2B	65.3 (7)
Br2A—C6A—C7A—Br1A	177.1 (3)	Br1B—C6B—C7B—Br2B	−176.3 (4)
O2A—C8A—C7A—C6A	45.1 (16)	O2B—C8B—C7B—C6B	−43.9 (18)
C9A—C8A—C7A—C6A	−144.9 (13)	C9B—C8B—C7B—C6B	145.4 (16)
O2A—C8A—C7A—Br1A	−70.7 (13)	O2B—C8B—C7B—Br2B	74.8 (16)

C9A—C8A—C7A—Br1A	99.4 (14)	C9B—C8B—C7B—Br2B	−95.9 (17)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2A—H2AA···O3A^{i}	0.93	2.53	3.210 (15)	131
C3A—H3AA···O2A^{ii}	0.93	2.51	3.216 (12)	133
C6A—H6AA···O2A^{ii}	0.98	2.33	3.217 (10)	151
C13A—H13A···O3A^{iii}	0.93	2.55	3.434 (13)	158

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

Fig. 1

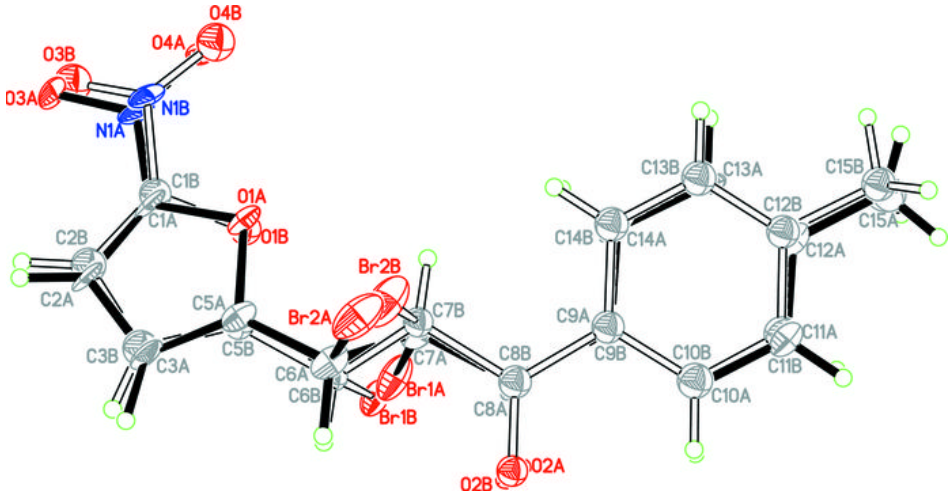
O4B
O4A
O3B
O3A
N1B
N1A
C1B
C1A
O1A
O1B
C2B
C2A
C5A
C5B
C3B
C3A
C6A
C6B
Br2B
Br2A
C7B
C7A
Br1A
Br1B
C8B
C8A
O2B
O2A
C9A
C9B
C13B
C13A
C14B
C14A
C12B
C12A
C15B
C15A
C10B
C10A
C11A
C11B

Fig. 2