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(*E*)-Ethyl 2-[2-(bromomethyl)phenyl]-2-(methoxyimino)acetate

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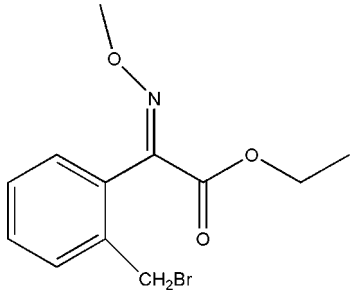
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.009 Å; disorder in main residue; R factor = 0.061; wR factor = 0.189; data-to-parameter ratio = 11.6.

In the title compound, $C_{12}H_{14}BrNO_3$, the ethyl acetate and CH_2Br groups each display rotational disorder, with occupancies of 0.7:0.3 and 0.8:0.2, respectively. The crystal packing is stabilized by intermolecular C—H···O hydrogen bonds and van der Waals forces.

Related literature

For the crystal structures of related compounds, see: Laurent *et al.* (1981). For details of the biological activities of strobilurin compounds, see: Bartlett *et al.* (2002); Sauter *et al.* (1999).



Experimental

Crystal data

$C_{12}H_{14}BrNO_3$
M_r = 300.15
Triclinic, $P\overline{1}$
a = 7.773 (2) Å
b = 8.443 (2) Å
c = 10.472 (3) Å
α = 78.982 (5)°
β = 85.344 (5)°
γ = 86.869 (6)°
V = 671.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.06 mm^{-1}
T = 294 (2) K
0.24 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.528, T_{max} = 0.609
(expected range = 0.499–0.577)
3405 measured reflections
2324 independent reflections
1097 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.189
S = 1.03
2324 reflections
200 parameters
102 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9C···O2^{i}	0.96	2.59	3.264 (10)	127

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2343).

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supplementary materials

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(*E*)-Ethyl 2-[2-(bromomethyl)phenyl]-2-(methoxyimino)acetate

L. Xu, W.-Z. Bi and K. Zhou

Comment

Strobilurin compounds are given more attention because their characteristics of high-efficient, low toxicity, broad-spectrum, security and environmental protection (Bartlett *et al.*, 2002; Sauter *et al.*, 1999). In a search for new strobilurin compounds with better biological activity, the title compound, (I), was synthesized. We report here the crystal structure of (I).

Bond lengths and angles are in agreement with those in previous reports (Laurent *et al.*, 1981). The benzene ring (C2—C7) make a dihedral angle of 73.97 (2)° with the plane N1/O1/C8/C9/C10. The crystal packing is stabilized by intermolecular C—H···O hydrogen bonds and van der Waals forces.

Experimental

To a 100-ml flask, 12 mmol of 1-bromopyrrolidine-2,5-dione, 10 mmol of ethyl 2-methoxyimino-2-*o*-tolylacetate in 30 ml carbon tetrachloride, and a little azodiisobutyronitrile were refluxed for 12 h. The resulting product was washed with carbon tetrachloride and dried *in vacuo*. The residue was purified by silica gel flash chromatography using a gradient from a 30:1 mixture of petroleum ether:ethyl acetate as eluent.(52% yield). The crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from hexane at room temperature for one week.

Refinement

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2 (1.5 for methyl and ethyl groups) times U_{eq}(C). The ethyl acetate group and atom Br were found to be disordered. The ethyl acetate group (atoms O2, O3, C11 and C12 with their attached H atoms) was treated over two orientations, with refined occupncies of 0.700 for the primed and 0.300 for the unprimed atoms. Atoms Br1 was refined over two positions [occupancies 0.800 for the primed and 0.200 for the unprimed atoms].

Figures

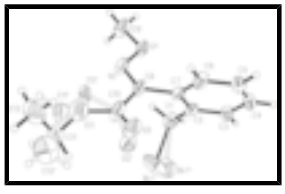


Fig. 1. View of the title compound (I), with displacement ellipsoids drawn at the 40% probability level.

(*E*)-Ethyl 2-[2-(bromomethyl)phenyl]-2-(methoxyimino)acetate

Crystal data

$C_{12}H_{14}BrNO_3$	$Z = 2$
$M_r = 300.15$	$F_{000} = 304$
Triclinic, $P\overline{1}$	$D_x = 1.484$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.773$ (2) Å	Cell parameters from 757 reflections
$b = 8.443$ (2) Å	$\theta = 2.6$–20.5°
$c = 10.472$ (3) Å	$\mu = 3.06$ mm^{-1}
$\alpha = 78.982$ (5)°	$T = 294$ (2) K
$\beta = 85.344$ (5)°	Block, colorless
$\gamma = 86.869$ (6)°	0.24 × 0.20 × 0.18 mm
$V = 671.8$ (3) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2324 independent reflections
Radiation source: fine-focus sealed tube	1097 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.028$
$T = 294$(2) K	$\theta_{max} = 25.0^\circ$
phi and ω scans	$\theta_{min} = 2.0^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -8 \rightarrow 9$
$T_{min} = 0.528$, $T_{max} = 0.609$	$k = -5 \rightarrow 10$
3405 measured reflections	$l = -11 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.061$	H-atom parameters constrained
$wR(F^2) = 0.189$	$w = 1/[\sigma^2(F_o^2) + (0.090P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.004$
2324 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
200 parameters	$\Delta\rho_{min} = -0.32$ e Å^{-3}
102 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}	Occ. (<1)
Br1	0.2222 (5)	0.9312 (3)	0.5699 (3)	0.1277 (12)	0.80
Br1'	0.2145 (16)	0.9244 (16)	0.5155 (13)	0.127 (5)	0.20
O1	0.6797 (4)	0.5967 (5)	0.7615 (4)	0.0626 (12)	
N1	0.5775 (6)	0.6726 (7)	0.8473 (5)	0.0570 (13)	
C1	0.3974 (7)	0.7607 (7)	0.5621 (6)	0.0660 (18)	
H1A	0.4331	0.7571	0.4717	0.079*	
H1B	0.4977	0.7828	0.6043	0.079*	
C2	0.3315 (6)	0.6064 (7)	0.6261 (5)	0.0448 (14)	
C3	0.2597 (7)	0.5061 (8)	0.5532 (6)	0.0583 (16)	
H3	0.2522	0.5401	0.4639	0.070*	
C4	0.2012 (8)	0.3596 (8)	0.6131 (8)	0.0663 (18)	
H4	0.1535	0.2953	0.5633	0.080*	
C5	0.2102 (8)	0.3038 (8)	0.7431 (8)	0.0686 (18)	
H5	0.1709	0.2025	0.7816	0.082*	
C6	0.2796 (8)	0.4016 (8)	0.8176 (6)	0.0631 (17)	
H6	0.2849	0.3660	0.9070	0.076*	
C7	0.3404 (6)	0.5502 (7)	0.7600 (6)	0.0458 (14)	
C8	0.4170 (7)	0.6517 (7)	0.8421 (5)	0.0516 (15)	
C9	0.8561 (7)	0.6317 (10)	0.7687 (7)	0.087 (2)	
H9A	0.8699	0.7460	0.7439	0.130*	
H9B	0.9284	0.5769	0.7107	0.130*	
H9C	0.8885	0.5956	0.8564	0.130*	
C10	0.2964 (9)	0.7311 (11)	0.9314 (7)	0.080 (2)	
O2	0.1487 (10)	0.6847 (11)	0.9616 (8)	0.074 (2)	0.70
O3	0.3666 (9)	0.8488 (10)	0.9785 (8)	0.068 (2)	0.70
C11	0.2622 (13)	0.9412 (14)	1.0607 (9)	0.074 (3)	0.70
H11A	0.1412	0.9382	1.0451	0.089*	0.70
H11B	0.2951	1.0529	1.0414	0.089*	0.70
C12	0.2900 (18)	0.8691 (17)	1.2004 (10)	0.113 (4)	0.70
H12A	0.2222	0.9298	1.2565	0.170*	0.70
H12B	0.4100	0.8722	1.2149	0.170*	0.70
H12C	0.2554	0.7591	1.2190	0.170*	0.70
O2'	0.143 (3)	0.773 (2)	0.904 (2)	0.076 (6)	0.30

O3'	0.382 (2)	0.760 (2)	1.0313 (15)	0.054 (4)	0.30
C11'	0.271 (3)	0.813 (3)	1.135 (2)	0.067 (6)	0.30
H11C	0.3363	0.8111	1.2103	0.081*	0.30
H11D	0.1766	0.7407	1.1605	0.081*	0.30
C12'	0.199 (6)	0.985 (3)	1.087 (4)	0.149 (15)	0.30
H12D	0.1258	1.0196	1.1564	0.223*	0.30
H12E	0.1324	0.9867	1.0137	0.223*	0.30
H12F	0.2923	1.0569	1.0626	0.223*	0.30

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.155 (2)	0.0519 (8)	0.161 (2)	0.0117 (9)	0.0467 (15)	−0.0106 (11)
Br1'	0.104 (5)	0.091 (5)	0.178 (10)	−0.005 (3)	−0.012 (6)	−0.010 (5)
O1	0.034 (2)	0.097 (3)	0.068 (3)	−0.004 (2)	−0.006 (2)	−0.044 (2)
N1	0.040 (3)	0.087 (4)	0.052 (3)	−0.008 (2)	−0.007 (2)	−0.032 (3)
C1	0.071 (4)	0.073 (4)	0.052 (4)	−0.013 (3)	−0.008 (3)	−0.003 (3)
C2	0.037 (3)	0.061 (4)	0.039 (4)	−0.001 (3)	−0.006 (3)	−0.014 (3)
C3	0.061 (4)	0.075 (5)	0.045 (4)	0.003 (3)	−0.011 (3)	−0.026 (4)
C4	0.072 (5)	0.058 (4)	0.081 (6)	0.002 (3)	−0.018 (4)	−0.038 (4)
C5	0.072 (4)	0.050 (4)	0.088 (6)	−0.007 (3)	−0.020 (4)	−0.016 (4)
C6	0.067 (4)	0.079 (5)	0.045 (4)	−0.012 (4)	−0.018 (3)	−0.008 (3)
C7	0.035 (3)	0.063 (4)	0.044 (4)	0.001 (3)	−0.008 (3)	−0.020 (3)
C8	0.040 (4)	0.082 (4)	0.040 (3)	−0.010 (3)	−0.006 (3)	−0.028 (3)
C9	0.039 (4)	0.134 (7)	0.098 (6)	−0.008 (4)	−0.003 (4)	−0.050 (5)
C10	0.049 (4)	0.140 (7)	0.069 (5)	−0.017 (4)	−0.003 (4)	−0.064 (5)
O2	0.048 (4)	0.108 (6)	0.074 (5)	−0.014 (4)	0.003 (4)	−0.037 (4)
O3	0.062 (4)	0.081 (5)	0.074 (5)	−0.008 (4)	−0.003 (4)	−0.042 (4)
C11	0.072 (6)	0.082 (6)	0.078 (7)	0.006 (5)	−0.007 (5)	−0.036 (6)
C12	0.135 (8)	0.121 (8)	0.086 (7)	0.019 (7)	0.009 (6)	−0.039 (7)
O2'	0.059 (9)	0.088 (9)	0.090 (10)	−0.001 (7)	−0.013 (7)	−0.037 (8)
O3'	0.064 (8)	0.064 (8)	0.043 (8)	−0.002 (7)	−0.001 (6)	−0.030 (6)
C11'	0.067 (9)	0.074 (10)	0.066 (10)	−0.009 (8)	−0.004 (8)	−0.026 (8)
C12'	0.154 (17)	0.147 (17)	0.148 (17)	−0.013 (10)	−0.001 (10)	−0.034 (10)

Geometric parameters (Å, °)

Br1—C1	1.936 (6)	C9—H9B	0.9600
Br1'—C1	1.952 (9)	C9—H9C	0.9600
O1—N1	1.377 (6)	C10—O2	1.231 (10)
O1—C9	1.429 (7)	C10—O2'	1.268 (19)
N1—C8	1.276 (6)	C10—O3	1.349 (10)
C1—C2	1.446 (8)	C10—O3'	1.353 (16)
C1—H1A	0.9700	O3—C11	1.443 (8)
C1—H1B	0.9700	C11—C12	1.501 (9)
C2—C7	1.398 (7)	C11—H11A	0.9700
C2—C3	1.407 (7)	C11—H11B	0.9700
C3—C4	1.360 (9)	C12—H12A	0.9600
C3—H3	0.9300	C12—H12B	0.9600

C4—C5	1.360 (9)	C12—H12C	0.9600
C4—H4	0.9300	O3'—C11'	1.454 (10)
C5—C6	1.395 (8)	C11'—C12'	1.532 (11)
C5—H5	0.9300	C11'—H11C	0.9700
C6—C7	1.375 (8)	C11'—H11D	0.9700
C6—H6	0.9300	C12'—H12D	0.9600
C7—C8	1.499 (7)	C12'—H12E	0.9600
C8—C10	1.494 (8)	C12'—H12F	0.9600
C9—H9A	0.9600		
N1—O1—C9	109.0 (4)	H9A—C9—H9C	109.5
C8—N1—O1	112.3 (4)	H9B—C9—H9C	109.5
C2—C1—Br1	110.5 (4)	O2—C10—O3	124.6 (7)
C2—C1—Br1'	112.8 (6)	O2'—C10—O3	110.0 (11)
C2—C1—H1A	109.6	O2—C10—O3'	115.1 (9)
Br1—C1—H1A	109.6	O2'—C10—O3'	129.0 (12)
Br1'—C1—H1A	93.0	O2—C10—C8	121.0 (7)
C2—C1—H1B	109.6	O2'—C10—C8	121.1 (10)
Br1—C1—H1B	109.6	O3—C10—C8	114.3 (6)
Br1'—C1—H1B	122.0	O3'—C10—C8	109.7 (8)
H1A—C1—H1B	108.1	C10—O3—C11	120.2 (8)
C7—C2—C3	117.9 (6)	O3—C11—C12	108.3 (9)
C7—C2—C1	121.8 (5)	O3—C11—H11A	110.0
C3—C2—C1	120.2 (5)	C12—C11—H11A	110.0
C4—C3—C2	120.1 (6)	O3—C11—H11B	110.0
C4—C3—H3	119.9	C12—C11—H11B	110.0
C2—C3—H3	119.9	H11A—C11—H11B	108.4
C5—C4—C3	122.3 (6)	C11—C12—H12A	109.5
C5—C4—H4	118.9	C11—C12—H12B	109.5
C3—C4—H4	118.9	H12A—C12—H12B	109.5
C4—C5—C6	118.7 (6)	C11—C12—H12C	109.5
C4—C5—H5	120.7	H12A—C12—H12C	109.5
C6—C5—H5	120.7	H12B—C12—H12C	109.5
C7—C6—C5	120.5 (6)	C10—O3'—C11'	113.9 (15)
C7—C6—H6	119.8	O3'—C11'—C12'	110 (2)
C5—C6—H6	119.8	O3'—C11'—H11C	109.7
C6—C7—C2	120.5 (5)	C12'—C11'—H11C	109.7
C6—C7—C8	119.4 (5)	O3'—C11'—H11D	109.7
C2—C7—C8	120.2 (5)	C12'—C11'—H11D	109.7
N1—C8—C10	115.9 (5)	H11C—C11'—H11D	108.2
N1—C8—C7	126.3 (5)	C11'—C12'—H12D	109.5
C10—C8—C7	117.7 (5)	C11'—C12'—H12E	109.5
O1—C9—H9A	109.5	H12D—C12'—H12E	109.5
O1—C9—H9B	109.5	C11'—C12'—H12F	109.5
H9A—C9—H9B	109.5	H12D—C12'—H12F	109.5
O1—C9—H9C	109.5	H12E—C12'—H12F	109.5
C9—O1—N1—C8	177.4 (5)	C6—C7—C8—C10	−73.4 (8)
Br1—C1—C2—C7	−87.3 (6)	C2—C7—C8—C10	106.8 (7)
Br1'—C1—C2—C7	−106.0 (7)	N1—C8—C10—O2	−159.6 (8)

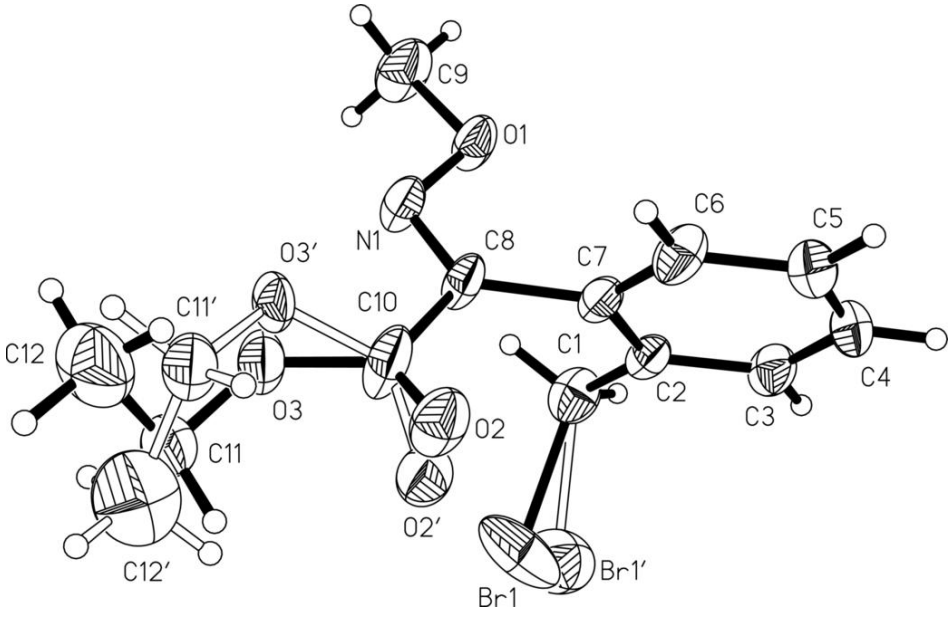
Br1—C1—C2—C3	94.3 (5)	C7—C8—C10—O2	17.8 (12)
Br1'—C1—C2—C3	75.6 (7)	N1—C8—C10—O2'	152.8 (13)
C7—C2—C3—C4	0.1 (8)	C7—C8—C10—O2'	−29.8 (16)
C1—C2—C3—C4	178.5 (5)	N1—C8—C10—O3	17.4 (10)
C2—C3—C4—C5	−0.4 (10)	C7—C8—C10—O3	−165.1 (7)
C3—C4—C5—C6	0.9 (10)	N1—C8—C10—O3'	−21.9 (12)
C4—C5—C6—C7	−1.1 (10)	C7—C8—C10—O3'	155.5 (10)
C5—C6—C7—C2	0.8 (9)	O2—C10—O3—C11	−6.2 (15)
C5—C6—C7—C8	−179.0 (5)	O2'—C10—O3—C11	36.7 (16)
C3—C2—C7—C6	−0.3 (8)	O3'—C10—O3—C11	−92.5 (15)
C1—C2—C7—C6	−178.7 (5)	C8—C10—O3—C11	176.9 (8)
C3—C2—C7—C8	179.5 (5)	C10—O3—C11—C12	96.9 (12)
C1—C2—C7—C8	1.1 (7)	O2—C10—O3'—C11'	−30 (2)
O1—N1—C8—C10	−179.5 (6)	O2'—C10—O3'—C11'	15 (3)
O1—N1—C8—C7	3.3 (8)	O3—C10—O3'—C11'	84.8 (19)
C6—C7—C8—N1	103.7 (7)	C8—C10—O3'—C11'	−170.6 (14)
C2—C7—C8—N1	−76.1 (7)	C10—O3'—C11'—C12'	−71 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9C···O2^{i}	0.96	2.59	3.264 (10)	127

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

Fig. 1


C9
O1
N1
C8
C7
C6
C5
C4
C3
C2
C1
O3′
C11′
C10
C12
O3
C11
O2
O2′
C12′
Br1′
Br1