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5-Ethyl-2-methyl-3-methylsulfinyl-1-benzofuran

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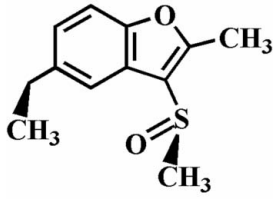
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.107; data-to-parameter ratio = 15.3.

The title compound, $C_{12}H_{14}O_2S$, was prepared by the oxidation of 5-ethyl-2-methyl-3-methylsulfanyl-1-benzofuran using 3-chloroperbenzoic acid. The O atom and the methyl group of the methylsulfinyl substituent lie on opposite sides of the plane of the benzofuran unit. The methyl group of the ethyl substituent is rotated out of the benzofuran plane by 113.5 (2)°. The crystal structure is stabilized by aromatic π–π stacking interactions, with centroid–centroid distances of 3.639 (3) and 3.604 (3) Å between benzene/furan and furan/furan rings, respectively, and (2-methyl group)CH_2—H$\cdots\pi$(benzene ring) interactions.

Related literature

For crystal structures of isomeric compounds, see: Choi *et al.* (2007*a*,*b*).



Experimental

Crystal data

$C_{12}H_{14}O_2S$
M_r = 222.29
Triclinic, $P\overline{1}$
a = 8.0314 (6) Å
b = 8.7673 (7) Å
c = 8.9017 (7) Å
α = 97.740 (1)°
β = 108.721 (1)°
γ = 107.289 (1)°
V = 548.17 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 173 (2) K
0.52 × 0.43 × 0.25 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
3074 measured reflections
2107 independent reflections
2009 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.107
S = 1.06
2107 reflections
138 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.83 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

Cg is the centroid of the C2–C7 benzene ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C11—H11$B\cdots Cg^{i}$	0.98	2.75	3.532 (3)	137

Symmetry code: (i) $-x+1, -y+1, -z+2$.

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: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2078).

References

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supplementary materials

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5-Ethyl-2-methyl-3-methylsulfinyl-1-benzofuran

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Comment

As part of our continuing work related to the synthesis and structure of 2-methyl-3-methylsulfinyl-1-benzofuran derivatives, the crystal structures of 5-bromo-2-methyl-3-methylsulfinyl-1-benzofuran (Choi *et al.*, 2007*a*) and 2,5-dimethyl-3-methylsulfinyl-1-benzofuran (Choi *et al.*, 2007*b*) have been reported. Here we present the molecular and crystal structure of the title compound (Fig. 1).

The benzofuran ring system is essentially planar, with a mean deviation of 0.003Å from the least-squares plane defined by the nine constituent atoms. The molecular packing (Fig. 2) is stabilized by two different π···π interactions within each stack of molecules; one between the furan ring (*Cg*1) and an adjacent benzene ring(*Cg*2^{ii}) {distance; 3.639 (3) Å} of benzofuran unit, and a second between the furan ring (*Cg*1) and an adjacent furan ring (*Cg*1^{i}) of benzofuran unit {distance; 3.604 (3) Å}. The crystal packing (Fig. 2) is further stabilized by CH_2—H···π interactions between the 2-methyl group and the benzene ring of benzofuran unit, with a C11—H11B···*Cg*2^{i} separation of 2.75 Å (Fig. 2 and Table 1; *Cg*1 and *Cg*2 are the centroids of the C1/C2/C7/O1/C8 furan ring and the C2—C7 benzene ring, respectively; symmetry codes as in Fig. 2).

Experimental

3-Chloroperbenzoic acid (77%, 471 mg, 2.1 mmol) was added in small portions to a stirred solution of 5-ethyl-2-methyl-3-methylsulfanyl-1-benzofuran (412 mg, 2.0 mmol) in dichloromethane (30 ml) at 273 K. After being stirred at room temperature for 2hrs, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (ethyl acetate) to afford the title compound as a colorless solid [yield 92%, m.p. 361–362 K; R_f = 0.30 (ethyl acetate)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a dilute solution of title compound (I) in acetone at room temperature.

Refinement

All H atoms were geometrically located in ideal positions and refined using a riding model, with C—H = 0.93 Å for aromatic H atoms and C—H = 0.96 Å for methyl H atoms, and with U_{iso}(H) = 1.2Ueq(C) for aromatic H atoms, and U_{iso}(H) = 1.5Ueq(C) for methyl H atoms.

Figures

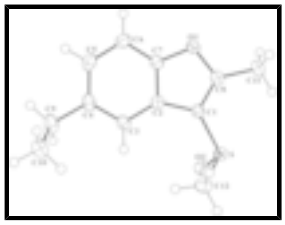


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoides drawn at the 50% probability level.

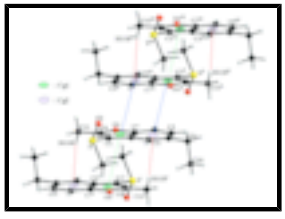


Fig. 2. π···π and —CH_2—H···π interactions (dasded lines). *Cg* denotes ring centroids. [Symmetry codes: (i) 1 − *x*, 1 − *y*, 2 − *z*; (ii) −*x*, 1 − *y*, 2 − *z*; (iii) *x* − 1, *y*., *z*.]

5-Ethyl-2-methyl-3-methylsulfinyl-1-benzofuran

Crystal data

$C_{12}H_{14}O_2S$	$Z = 2$
$M_r = 222.29$	$F_{000} = 236$
Triclinic, $P\bar{1}$	$D_x = 1.347$ Mg m^{-3}
Hall symbol: -P_1	Mo $K\alpha$ radiation λ = 0.71073 Å
$a = 8.0314$ (6) Å	Cell parameters from 2853 reflections
$b = 8.7673$ (7) Å	θ = 2.5–28.2º
$c = 8.9017$ (7) Å	μ = 0.27 mm^{-1}
α = 97.740 (1)º	$T = 173$ (2) K
β = 108.721 (1)º	Block, colorless
γ = 107.289 (1)º	0.52 × 0.43 × 0.25 mm
$V = 548.17$ (7) Å^3	

Data collection

Bruker SMART CCD diffractometer	2107 independent reflections
Radiation source: fine-focus sealed tube	2009 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
Detector resolution: 10.00 pixels mm^{-1}	$\theta_{max} = 26.0$º
$T = 173(2)$ K	$\theta_{min} = 2.5$º
φ and ω scans	$h = -9\rightarrow6$
Absorption correction: none	$k = -10\rightarrow10$
3074 measured reflections	$l = -8\rightarrow10$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.4774P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2107 reflections	$\Delta\rho_{max} = 0.83$ e Å^{-3}
138 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2$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}
S	0.31899 (6)	0.70982 (6)	0.72234 (5)	0.02583 (17)
O1	0.38885 (18)	0.59991 (16)	1.14540 (15)	0.0242 (3)
O2	0.1366 (2)	0.73454 (19)	0.64848 (18)	0.0352 (4)
C1	0.3101 (2)	0.6141 (2)	0.8823 (2)	0.0205 (4)
C2	0.1751 (2)	0.4623 (2)	0.8850 (2)	0.0207 (4)
C3	0.0173 (2)	0.3325 (2)	0.7693 (2)	0.0239 (4)
H3	−0.0237	0.3312	0.6563	0.029*
C4	−0.0790 (3)	0.2043 (2)	0.8232 (2)	0.0268 (4)
C5	−0.0168 (3)	0.2090 (2)	0.9904 (3)	0.0300 (4)
H5	−0.0840	0.1211	1.0249	0.036*
C6	0.1394 (3)	0.3372 (2)	1.1079 (2)	0.0290 (4)
H6	0.1804	0.3395	1.2212	0.035*
C7	0.2314 (3)	0.4610 (2)	1.0499 (2)	0.0229 (4)
C8	0.4327 (2)	0.6902 (2)	1.0395 (2)	0.0217 (4)
C9	−0.2488 (3)	0.0602 (3)	0.7007 (3)	0.0362 (5)
H9A	−0.3121	−0.0072	0.7612	0.043*
H9B	−0.3392	0.1034	0.6329	0.043*
C10	−0.1996 (3)	−0.0496 (3)	0.5889 (3)	0.0372 (5)
H10A	−0.1121	−0.0949	0.6549	0.045*
H10B	−0.1401	0.0154	0.5261	0.045*
H10C	−0.3148	−0.1403	0.5132	0.045*
C11	0.5995 (3)	0.8462 (2)	1.1159 (2)	0.0280 (4)

H11A	0.6144	0.9046	1.0319	0.042*
H11B	0.7127	0.8208	1.1659	0.042*
H11C	0.5816	0.9160	1.2005	0.042*
C12	0.3148 (3)	0.5425 (3)	0.5772 (2)	0.0316 (4)
H12A	0.1921	0.4523	0.5380	0.047*
H12B	0.4148	0.5027	0.6306	0.047*
H12C	0.3351	0.5818	0.4842	0.047*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0286 (3)	0.0238 (3)	0.0230 (3)	0.00662 (19)	0.00919 (19)	0.00760 (18)
O1	0.0256 (7)	0.0238 (7)	0.0184 (6)	0.0071 (5)	0.0049 (5)	0.0038 (5)
O2	0.0410 (8)	0.0373 (8)	0.0302 (7)	0.0218 (7)	0.0088 (6)	0.0119 (6)
C1	0.0197 (8)	0.0210 (8)	0.0207 (8)	0.0071 (7)	0.0075 (7)	0.0054 (7)
C2	0.0195 (8)	0.0210 (8)	0.0230 (9)	0.0090 (7)	0.0081 (7)	0.0062 (7)
C3	0.0198 (8)	0.0252 (9)	0.0240 (9)	0.0072 (7)	0.0064 (7)	0.0044 (7)
C4	0.0202 (9)	0.0229 (9)	0.0358 (10)	0.0060 (7)	0.0124 (8)	0.0029 (8)
C5	0.0331 (10)	0.0240 (9)	0.0391 (11)	0.0087 (8)	0.0221 (9)	0.0103 (8)
C6	0.0378 (11)	0.0285 (10)	0.0255 (9)	0.0125 (8)	0.0163 (8)	0.0104 (8)
C7	0.0227 (9)	0.0212 (9)	0.0238 (9)	0.0081 (7)	0.0084 (7)	0.0036 (7)
C8	0.0203 (8)	0.0215 (9)	0.0236 (9)	0.0085 (7)	0.0083 (7)	0.0045 (7)
C9	0.0252 (10)	0.0297 (10)	0.0444 (12)	0.0011 (8)	0.0127 (9)	0.0018 (9)
C10	0.0373 (12)	0.0297 (10)	0.0337 (11)	0.0051 (9)	0.0091 (9)	0.0011 (9)
C11	0.0232 (9)	0.0232 (9)	0.0287 (10)	0.0048 (8)	0.0045 (8)	0.0007 (7)
C12	0.0353 (11)	0.0366 (11)	0.0245 (10)	0.0148 (9)	0.0126 (8)	0.0062 (8)

Geometric parameters (Å, °)

S—O2	1.495 (2)	C6—C7	1.383 (3)
S—C1	1.758 (2)	C6—H6	0.9500
S—C12	1.804 (2)	C8—C11	1.484 (3)
O1—C8	1.374 (2)	C9—C10	1.514 (3)
O1—C7	1.385 (2)	C9—H9A	0.9900
C1—C8	1.359 (3)	C9—H9B	0.9900
C1—C2	1.454 (2)	C10—H10A	0.9800
C2—C7	1.393 (3)	C10—H10B	0.9800
C2—C3	1.397 (3)	C10—H10C	0.9800
C3—C4	1.400 (3)	C11—H11A	0.9800
C3—H3	0.9500	C11—H11B	0.9800
C4—C5	1.400 (3)	C11—H11C	0.9800
C4—C9	1.515 (3)	C12—H12A	0.9800
C5—C6	1.391 (3)	C12—H12B	0.9800
C5—H5	0.9500	C12—H12C	0.9800
O2—S—C1	108.54 (9)	C1—C8—C11	133.31 (17)
O2—S—C12	106.41 (9)	O1—C8—C11	115.67 (15)
C1—S—C12	99.54 (9)	C10—C9—C4	113.45 (17)
C8—O1—C7	106.28 (13)	C10—C9—H9A	108.9

C8—C1—C2	107.40 (16)	C4—C9—H9A	108.9
C8—C1—S	121.09 (14)	C10—C9—H9B	108.9
C2—C1—S	131.20 (14)	C4—C9—H9B	108.9
C7—C2—C3	119.36 (17)	H9A—C9—H9B	107.7
C7—C2—C1	104.43 (16)	C9—C10—H10A	109.5
C3—C2—C1	136.21 (17)	C9—C10—H10B	109.5
C2—C3—C4	118.64 (17)	H10A—C10—H10B	109.5
C2—C3—H3	120.7	C9—C10—H10C	109.5
C4—C3—H3	120.7	H10A—C10—H10C	109.5
C3—C4—C5	119.79 (17)	H10B—C10—H10C	109.5
C3—C4—C9	120.04 (18)	C8—C11—H11A	109.5
C5—C4—C9	120.17 (18)	C8—C11—H11B	109.5
C6—C5—C4	122.62 (17)	H11A—C11—H11B	109.5
C6—C5—H5	118.7	C8—C11—H11C	109.5
C4—C5—H5	118.7	H11A—C11—H11C	109.5
C7—C6—C5	115.92 (18)	H11B—C11—H11C	109.5
C7—C6—H6	122.0	S—C12—H12A	109.5
C5—C6—H6	122.0	S—C12—H12B	109.5
C6—C7—O1	125.44 (17)	H12A—C12—H12B	109.5
C6—C7—C2	123.67 (18)	S—C12—H12C	109.5
O1—C7—C2	110.88 (15)	H12A—C12—H12C	109.5
C1—C8—O1	111.02 (16)	H12B—C12—H12C	109.5
O2—S—C1—C8	−117.99 (16)	C5—C6—C7—C2	0.3 (3)
C12—S—C1—C8	130.99 (16)	C8—O1—C7—C6	−179.47 (18)
O2—S—C1—C2	54.83 (19)	C8—O1—C7—C2	0.01 (19)
C12—S—C1—C2	−56.18 (19)	C3—C2—C7—C6	0.0 (3)
C8—C1—C2—C7	−0.05 (19)	C1—C2—C7—C6	179.51 (17)
S—C1—C2—C7	−173.62 (14)	C3—C2—C7—O1	−179.53 (15)
C8—C1—C2—C3	179.4 (2)	C1—C2—C7—O1	0.03 (19)
S—C1—C2—C3	5.8 (3)	C2—C1—C8—O1	0.1 (2)
C7—C2—C3—C4	−0.3 (3)	S—C1—C8—O1	174.41 (12)
C1—C2—C3—C4	−179.69 (19)	C2—C1—C8—C11	179.73 (19)
C2—C3—C4—C5	0.4 (3)	S—C1—C8—C11	−5.9 (3)
C2—C3—C4—C9	−179.01 (17)	C7—O1—C8—C1	−0.04 (19)
C3—C4—C5—C6	−0.2 (3)	C7—O1—C8—C11	−179.78 (15)
C9—C4—C5—C6	179.24 (18)	C3—C4—C9—C10	68.7 (3)
C4—C5—C6—C7	−0.2 (3)	C5—C4—C9—C10	−110.7 (2)
C5—C6—C7—O1	179.69 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11B···Cg^{i}	0.98	2.75	3.532 (3)	137

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

Fig. 1

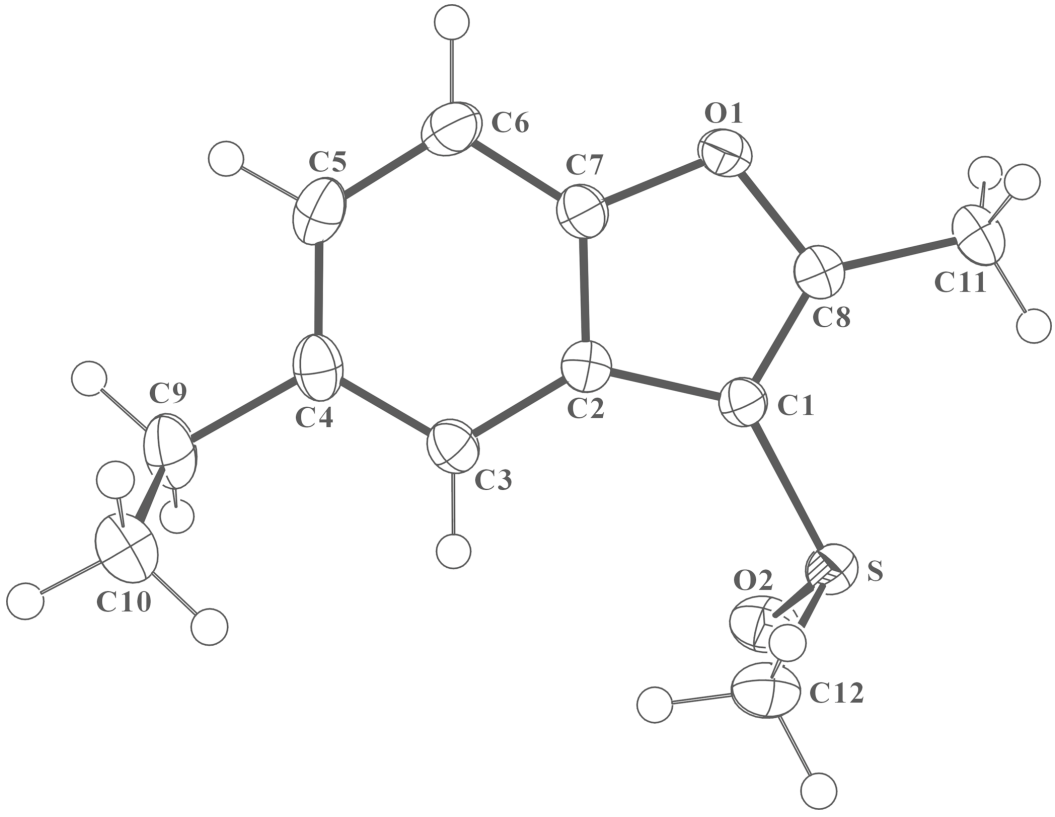
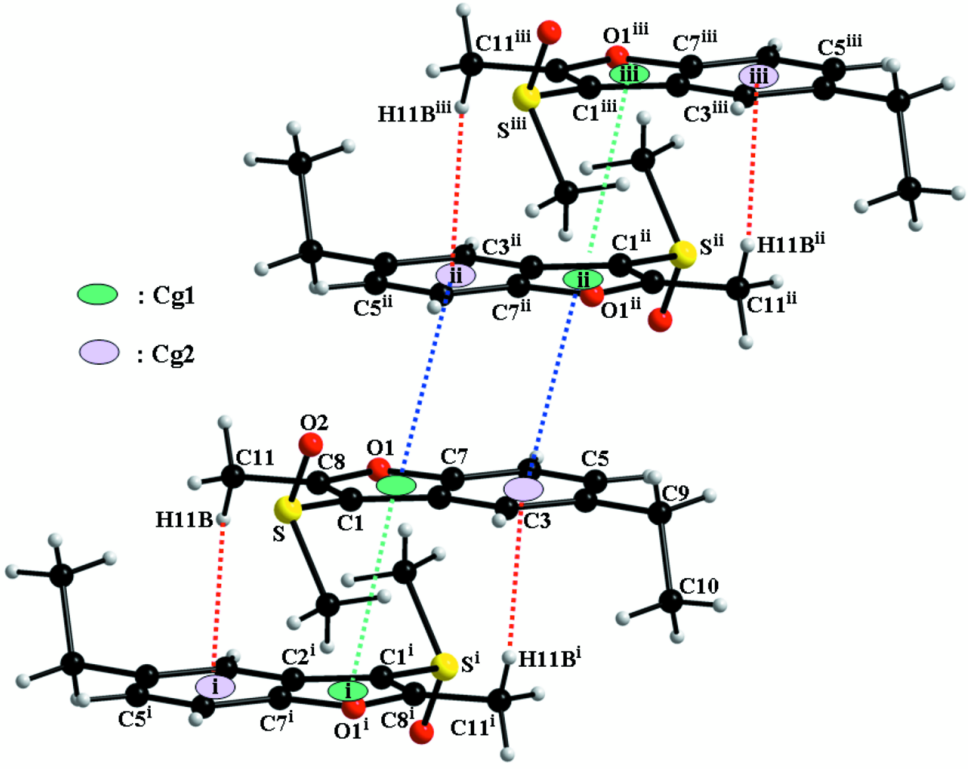


Fig. 2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C11ii
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C8
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H11B
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C1
C3
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C1i
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