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3-Bromo-4-methylbicyclo[6.3.0]undecane-2,6-dione, (I), 5-bromo-4-methylbicyclo[6.3.0]undecane-2,6-dione, (II), 3,5-dibromo-4-methylbicyclo[6.3.0]undecane-2,6-dione, (III), and 1,7-dibromo-4-methylbicyclo[6.3.0]undecane-2,6-dione, (IV)

M. Umehara, H. Hosomi and S. Ohba

Abstract

Structures of title four compounds, $C_{12}H_{17}BrO_2$, (I), $C_{12}H_{17}BrO_2$, (II), $C_{12}H_{16}Br_2O_2$, (III), $C_{12}H_{16}Br_2O_2$, (IV), which were obtained in the bromination of *trans*-4-methylbicyclo[6.3.0]undecane-2,6-dione were determined. The eight-membered rings in (I)-(IV) take boat-chair form.

Experimental

The bromination of *trans*-4-methylbicyclo[6.3.0]undecane-2,6-dione with pyridinium bromide perbromide in acetic acid gave 3-bromide (I), 5-bromide (II), 7-bromide, and 3,5-dibromide (III). On the other hand, the bromination in solid phases by same reagent yielded the three monobromides and 1,7-dibromide (IV). The crystal structure of 7-bromide will be reported separately together with those of 7-acetonyl derivative and C_5—C_8—C_5 fused-ring compound of a Basmane-like carbon skeletone (Umehara *et al.* 1999). The crystals were grown from an ether solution for (I) and (II), a hexane/ether solution for (III), and an ether/acetone solution for (IV). Melting points are (I) 382, (II) 396, (III) 397, and (IV) 424 K.

Refinement

The refinement were carried out based on all the reflections with $|F_o| \neq 0$. The threshold, $I > 2\sigma(I)$, was used only for calculation of the *R*-factor. The positions of all the H atoms were calculated geometrically, and a riding model was used in their refinement [C—H 0.96 Å]. U_{iso}(H) values were refined.

Computing details

For all compounds, data collection: AFC/MSC Diffractometer Control System (Rigaku Corporation, 1993); cell refinement: AFC/MSC Diffractometer Control System; data reduction: local programs; program(s) used to solve structure: *CRYSTAN*GM (Edwards *et al.*, 1996); program(s) used to refine structure: *CRYSTAN*GM; molecular graphics: *CRYSTAN*GM; software used to prepare material for publication: *CRYSTAN*GM

3-Bromo-4-methylbicyclo[6.3.0]undecane-2,6-dione

Crystal data

$C_{12}H_{17}Br_1O_2$	$V = 1212.7\ (5)$ Å^3
$M_r = 273.17$	$Z = 4$

CIF access

Monoclinic, $P2_1/n$	Mo $K\alpha$
a = 10.994 (2) Å	μ = 3.37 mm^{-1}
b = 6.718 (2) Å	T = 297 K
c = 16.824 (2) Å	0.6 × 0.6 × 0.5 mm
β = 102.59 (1)°	

Data collection

Rigaku AFC-5 diffractometer	1530 reflections with $I > 2\sigma(I)$
Absorption correction: integration (Coppens et al., 1965)	R_{int} = 0.019
T_{min} = 0.198, T_{max} = 0.279	3 standard reflections
2929 measured reflections	every 100 reflections
2788 independent reflections	intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054	153 parameters
$wR(F^2)$ = 0.069	H-atom parameters constrained
S = 1.31	$\Delta\rho_{max}$ = 0.67 e Å^{-3}
2341 reflections	$\Delta\rho_{min}$ = −0.99 e Å^{-3}

Table 1
Selected geometric parameters (Å)

Br1—C4	1.978 (5)	C7—C8	1.517 (7)
O2—C7	1.212 (7)	C8—C9	1.513 (8)
O3—C14	1.201 (6)	C9—C10	1.530 (8)
C4—C5	1.525 (8)	C9—C13	1.524 (7)
C4—C14	1.530 (7)	C10—C11	1.523 (9)
C5—C6	1.531 (8)	C11—C12	1.532 (8)
C5—C15	1.519 (7)	C12—C13	1.527 (8)
C6—C7	1.509 (8)	C13—C14	1.501 (7)

5-Bromo-4-methylbicyclo[6.3.0]undecane-2,6-dione

Crystal data

$C_{12}H_{17}Br_1O_2$	V = 1234.7 (3) Å^3
M_r = 273.17	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$
a = 7.186 (1) Å	μ = 3.31 mm^{-1}
b = 18.104 (2) Å	T = 295 K
c = 9.569 (2) Å	0.6 × 0.6 × 0.3 mm
β = 97.34 (2)°	

Data collection

Rigaku AFC-5 diffractometer	1257 reflections with $I > 2\sigma(I)$
Absorption correction: integration (Coppens et al, 1965)	$R_{int} = 0.029$
$T_{min} = 0.166$, $T_{max} = 0.389$	3 standard reflections
3063 measured reflections	every 100 reflections
2845 independent reflections	intensity decay: 4.7%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$	153 parameters
$wR(F^2) = 0.074$	H-atom parameters constrained
$S = 1.31$	$\Delta\rho_{max} = 0.57$ e Å^{-3}
2199 reflections	$\Delta\rho_{min} = -0.68$ e Å^{-3}

Table 2
Selected geometric parameters (Å)

Br1—C4	1.960 (6)	C7—C11	1.538 (8)
O2—C5	1.211 (9)	C8—C9	1.541 (10)
O3—C12	1.210 (8)	C9—C10	1.518 (10)
C4—C5	1.514 (8)	C10—C11	1.516 (8)
C4—C14	1.522 (8)	C11—C12	1.501 (8)
C5—C6	1.493 (8)	C12—C13	1.485 (8)
C6—C7	1.528 (8)	C13—C14	1.546 (8)
C7—C8	1.531 (8)	C14—C15	1.511 (10)

3,5-Dibromo-4-methylbicyclo[6.3.0]undecane-2,6-dione

Crystal data

$C_{12}H_{16}Br_2O_2$	$V = 2659.0$ (12) Å^3
$M_r = 352.07$	$Z = 8$
Monoclinic, $C2/c$	Mo $K\alpha$
$a = 16.027$ (3) Å	$\mu = 6.08$ mm^{-1}
$b = 11.741$ (4) Å	$T = 298$ K
$c = 14.477$ (3) Å	0.5 × 0.5 × 0.4 mm
$\beta = 102.56$ (1)°	

Data collection

Rigaku AFC-5 diffractometer	1313 reflections with $I > 2\sigma(I)$
Absorption correction: integration (Coppens et al., 1965)	$R_{int} = 0.020$

T_{min} = 0.050, T_{max} = 0.178
2424 measured reflections
2337 independent reflections

3 standard reflections
every 100 reflections
intensity decay: 2.6%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.085
S = 1.88
1975 reflections

161 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.90 e Å^{-3}
$\Delta\rho_{min}$ = −0.88 e Å^{-3}

Table 3
Selected geometric parameters (Å)

Br1—C5	1.98 (2)	C8—C9	1.51 (3)
Br2—C7	1.97 (1)	C9—C10	1.52 (3)
O3—C15	1.22 (2)	C10—C11	1.53 (3)
O4—C8	1.22 (3)	C10—C14	1.53 (3)
C5—C6	1.52 (3)	C11—C12	1.54 (4)
C5—C15	1.51 (2)	C12—C13	1.55 (3)
C6—C7	1.52 (3)	C13—C14	1.54 (3)
C6—C16	1.53 (3)	C14—C15	1.51 (2)
C7—C8	1.51 (3)		

1,7-Dibromo-4-methylbicyclo[6.3.0]undecane-2,6-dione

Crystal data

$C_{12}H_{16}Br_2O_2$
M_r = 352.07
Triclinic, $P\overline{1}$
a = 7.228 (2) Å
b = 15.875 (3) Å
c = 5.851 (2) Å
α = 99.57 (3)°
β = 98.31 (3)°
γ = 77.20 (2)°
V = 641.5 (3) Å^3
Z = 2
Mo $K\alpha$
μ = 6.30 mm^{-1}
T = 298 K
0.6 × 0.4 × 0.3 mm

Data collection

Rigaku AFC-5 diffractometer
Absorption correction: integration (Coppens et al., 1965)
T_{min} = 0.092, T_{max} = 0.204
2729 measured reflections
2515 independent reflections

1842 reflections with $I > 2\sigma(I)$
R_{int} = 0.011
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$	161 parameters
$wR(F^2) = 0.055$	H-atom parameters constrained
$S = 1.23$	$\Delta\rho_{max} = 0.89$ e Å^{-3}
2287 reflections	$\Delta\rho_{min} = -1.04$ e Å^{-3}

Table 4
Selected geometric parameters (Å)

Br1—C5	1.961 (5)	C8—C9	1.530 (7)
Br2—C10	1.997 (5)	C9—C10	1.514 (6)
O3—C15	1.204 (6)	C10—C11	1.521 (7)
O4—C11	1.217 (5)	C11—C12	1.505 (7)
C5—C6	1.532 (7)	C12—C13	1.536 (7)
C5—C15	1.532 (7)	C13—C14	1.521 (7)
C6—C7	1.537 (7)	C13—C16	1.528 (7)
C6—C10	1.535 (6)	C14—C15	1.499 (7)
C7—C8	1.521 (7)		

Acknowledgements

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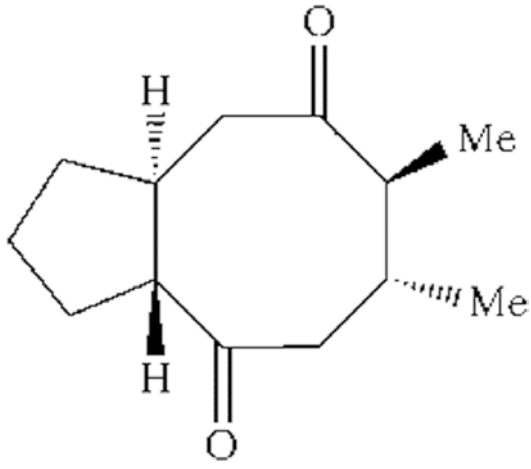
CIF access

Scheme 1



supplementary materials

3-Bromo-4-methylbicyclo[6.3.0]undecane-2,6-dione

Crystal data

$C_{12}H_{17}Br_1O_2$	F_{000} = 560
M_r = 273.17	D_x = 1.496 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.994 (2) Å	Cell parameters from 25 reflections
b = 6.718 (2) Å	θ = 14.7–15.0º
c = 16.824 (2) Å	μ = 3.37 mm^{-1}
β = 102.59 (1)º	T = 297 K
V = 1212.7 (5) Å^3	Prism, colourless
Z = 4	0.6 × 0.6 × 0.5 mm

Data collection

Rigaku AFC-5 diffractometer	θ_{max} = 27.5º
θ–2θ scans	h = 0→14
Absorption correction: integration (Coppens et al., 1965)	k = 0→9
T_{min} = 0.198, T_{max} = 0.279	l = −22→22
2929 measured reflections	3 standard reflections
2788 independent reflections	every 100 reflections
1530 reflections with $I > 2\sigma(I)$	intensity decay: none
R_{int} = 0.019	

Refinement

Refinement on F	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.054	$w = 1/[\sigma^2(F) + 0.0009F^2]$
$wR(F^2)$ = 0.069	$(\Delta/\sigma)_{max}$ = 0.003
S = 1.31	$\Delta\rho_{max}$ = 0.67 e Å^{-3}
2341 reflections	$\Delta\rho_{min}$ = −0.99 e Å^{-3}
153 parameters	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.73790 (5)	0.3148 (1)	0.65150 (4)	0.0817 (3)
O2	1.1213 (3)	−0.1893 (6)	0.5871 (2)	0.085 (2)
O3	1.0545 (3)	0.2850 (5)	0.6473 (2)	0.065 (1)
C4	0.8539 (4)	0.1437 (7)	0.6073 (3)	0.048 (2)
C5	0.8074 (4)	−0.0707 (8)	0.5975 (3)	0.050 (2)
C6	0.9010 (5)	−0.1994 (8)	0.5653 (3)	0.062 (2)

C7	1.0315 (5)	−0.2173 (7)	0.6166 (3)	0.062 (2)
C8	1.0491 (5)	−0.2653 (8)	0.7065 (3)	0.059 (2)
C9	1.0921 (4)	−0.0847 (8)	0.7588 (3)	0.053 (2)
C10	1.1238 (5)	−0.125 (1)	0.8505 (3)	0.068 (2)
C11	1.1036 (6)	0.071 (1)	0.8912 (3)	0.081 (3)
C12	1.0543 (5)	0.2188 (9)	0.8224 (3)	0.073 (2)
C13	0.9986 (4)	0.0856 (7)	0.7501 (3)	0.048 (2)
C14	0.9772 (4)	0.1825 (7)	0.6678 (3)	0.049 (2)
C15	0.6821 (5)	−0.085 (1)	0.5380 (3)	0.076 (3)
H4	0.85870	0.18480	0.55350	0.05 (1)*
H5	0.80290	−0.11920	0.65050	0.03 (1)*
H6A	0.90740	−0.14270	0.51400	0.06 (1)*
H6B	0.86650	−0.33090	0.55620	0.07 (2)*
H8A	0.97250	−0.31100	0.71870	0.06 (1)*
H8B	1.11060	−0.36850	0.71980	0.07 (2)*
H9	1.16400	−0.03370	0.74190	0.05 (1)*
H10A	1.20830	−0.16960	0.86840	0.09 (2)*
H10B	1.06860	−0.22500	0.86310	0.04 (1)*
H11A	1.04390	0.05470	0.92470	0.10 (2)*
H11B	1.18080	0.11670	0.92430	0.12 (2)*
H12A	0.99230	0.30530	0.83600	0.08 (2)*
H12B	1.12050	0.29820	0.81020	0.08 (2)*
H13	0.92120	0.03110	0.75760	0.03 (1)*
H15A	0.65250	−0.21960	0.53390	0.06 (2)*
H15B	0.69160	−0.04010	0.48550	0.05 (1)*
H15C	0.62260	−0.00110	0.55600	0.11 (2)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0673 (4)	0.0905 (5)	0.0784 (4)	0.0287 (3)	0.0283 (3)	−0.0035 (4)
O2	0.072 (2)	0.113 (3)	0.070 (2)	0.020 (2)	0.037 (2)	0.014 (2)
O3	0.065 (2)	0.066 (2)	0.061 (2)	−0.019 (2)	0.023 (2)	0.006 (2)
C4	0.048 (3)	0.060 (3)	0.035 (3)	0.009 (2)	0.019 (2)	0.005 (2)
C5	0.050 (3)	0.061 (3)	0.032 (2)	−0.007 (2)	0.009 (2)	−0.003 (2)
C6	0.075 (3)	0.054 (3)	0.047 (3)	−0.003 (3)	0.020 (3)	−0.005 (3)
C7	0.072 (4)	0.052 (3)	0.057 (3)	0.009 (3)	0.026 (3)	0.000 (3)
C8	0.066 (3)	0.055 (3)	0.058 (3)	0.015 (3)	0.020 (3)	0.015 (3)
C9	0.040 (2)	0.073 (4)	0.044 (3)	−0.001 (2)	0.011 (2)	0.007 (3)
C10	0.049 (3)	0.105 (5)	0.053 (3)	0.000 (3)	0.006 (2)	0.017 (3)
C11	0.083 (4)	0.113 (5)	0.044 (3)	−0.035 (4)	0.004 (3)	0.006 (4)
C12	0.071 (4)	0.084 (4)	0.053 (3)	−0.012 (4)	0.009 (3)	−0.011 (3)
C13	0.042 (2)	0.057 (3)	0.040 (3)	−0.008 (2)	0.013 (2)	−0.003 (2)
C14	0.049 (3)	0.044 (3)	0.043 (3)	−0.005 (2)	0.019 (2)	−0.010 (2)
C15	0.062 (4)	0.102 (5)	0.058 (4)	−0.015 (4)	0.005 (3)	−0.001 (4)

Geometric parameters (Å, °)

Br1—C4	1.978 (5)	C5—H5	0.961

O2—C7	1.212 (7)	C6—H6A	0.960
O3—C14	1.201 (6)	C6—H6B	0.960
C4—C5	1.525 (8)	C8—H8A	0.960
C4—C14	1.530 (7)	C8—H8B	0.960
C5—C6	1.531 (8)	C9—H9	0.960
C5—C15	1.519 (7)	C10—H10A	0.960
C6—C7	1.509 (8)	C10—H10B	0.959
C7—C8	1.517 (7)	C11—H11A	0.960
C8—C9	1.513 (8)	C11—H11B	0.958
C9—C10	1.530 (8)	C12—H12A	0.961
C9—C13	1.524 (7)	C12—H12B	0.960
C10—C11	1.523 (9)	C13—H13	0.960
C11—C12	1.532 (8)	C15—H15A	0.958
C12—C13	1.527 (8)	C15—H15B	0.960
C13—C14	1.501 (7)	C15—H15C	0.961
C4—H4	0.959		
Br1—C4—C5	111.2 (3)	H6A—C6—H6B	109.0
Br1—C4—C14	101.6 (4)	C7—C8—H8A	110.7
C5—C4—C14	117.4 (4)	C7—C8—H8B	108.4
C4—C5—C6	109.7 (4)	C9—C8—H8A	108.0
C4—C5—C15	111.4 (5)	C9—C8—H8B	109.2
C6—C5—C15	108.3 (5)	H8A—C8—H8B	109.0
C5—C6—C7	118.1 (5)	C8—C9—H9	106.7
O2—C7—C6	120.7 (5)	C10—C9—H9	109.9
O2—C7—C8	120.2 (5)	C13—C9—H9	106.6
C6—C7—C8	119.1 (5)	C9—C10—H10A	111.3
C7—C8—C9	111.4 (5)	C9—C10—H10B	109.3
C8—C9—C10	114.7 (5)	C11—C10—H10A	110.8
C8—C9—C13	115.3 (4)	C11—C10—H10B	110.2
C10—C9—C13	103.3 (5)	H10A—C10—H10B	109.1
C9—C10—C11	106.1 (5)	C10—C11—H11A	110.5
C10—C11—C12	106.4 (5)	C10—C11—H11B	109.9
C11—C12—C13	103.7 (5)	C12—C11—H11A	110.1
C9—C13—C12	102.6 (4)	C12—C11—H11B	110.7
C9—C13—C14	112.4 (4)	H11A—C11—H11B	109.1
C12—C13—C14	115.8 (5)	C11—C12—H12A	111.4
O3—C14—C4	119.1 (5)	C11—C12—H12B	111.2
O3—C14—C13	122.3 (5)	C13—C12—H12A	111.2
C4—C14—C13	118.5 (4)	C13—C12—H12B	110.3
Br1—C4—H4	111.1	H12A—C12—H12B	109.0
C5—C4—H4	104.8	C9—C13—H13	107.5
C14—C4—H4	110.9	C12—C13—H13	109.4
C4—C5—H5	107.7	C14—C13—H13	108.8
C6—C5—H5	108.2	C5—C15—H15A	110.6
C15—C5—H5	111.6	C5—C15—H15B	108.7
C5—C6—H6A	106.3	C5—C15—H15C	109.7
C5—C6—H6B	107.7	H15A—C15—H15B	109.5
C7—C6—H6A	107.4	H15A—C15—H15C	109.4
C7—C6—H6B	108.1	H15B—C15—H15C	108.9

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T_{min} = 0.166, T_{max} = 0.389	l = −12→12
3063 measured reflections	3 standard reflections
2845 independent reflections	every 100 reflections
1257 reflections with $I > 2\sigma(I)$	intensity decay: 4.7%
R_{int} = 0.029	

Refinement

Refinement on F	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.057	$w = 1/[\sigma^2(F) + 0.0009F^2]$
$wR(F^2)$ = 0.074	$(\Delta/\sigma)_{max}$ = 0.002
S = 1.31	$\Delta\rho_{max}$ = 0.57 e Å^{-3}
2199 reflections	$\Delta\rho_{min}$ = −0.68 e Å^{-3}
153 parameters	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	1.2535 (1)	0.57710 (4)	0.6034 (1)	0.0902 (3)
O2	1.0556 (6)	0.7026 (3)	0.3330 (5)	0.076 (2)
O3	0.6435 (5)	0.7860 (3)	0.4319 (5)	0.079 (2)
C4	1.0230 (7)	0.6300 (3)	0.5317 (6)	0.053 (2)
C5	1.0902 (7)	0.6977 (3)	0.4599 (7)	0.053 (2)
C6	1.1866 (7)	0.7582 (3)	0.5472 (6)	0.053 (2)

C7	1.0618 (7)	0.8265 (3)	0.5480 (6)	0.045 (2)
C8	1.1559 (9)	0.8933 (3)	0.6257 (7)	0.064 (2)
C9	0.995 (1)	0.9391 (3)	0.6740 (9)	0.077 (3)
C10	0.8158 (9)	0.8965 (3)	0.6273 (7)	0.067 (3)
C11	0.8809 (7)	0.8173 (3)	0.6166 (6)	0.045 (2)
C12	0.7432 (7)	0.7656 (3)	0.5362 (6)	0.050 (2)
C13	0.7248 (7)	0.6886 (3)	0.5862 (6)	0.059 (2)
C14	0.9007 (7)	0.6445 (3)	0.6475 (6)	0.050 (2)
C15	0.844 (1)	0.5743 (4)	0.7164 (9)	0.087 (3)
H4	0.94840	0.60040	0.46260	0.04 (1)*
H6A	1.21770	0.74210	0.64290	0.05 (2)*
H6B	1.30000	0.77080	0.50950	0.07 (2)*
H7	1.02590	0.84050	0.45160	0.04 (1)*
H8A	1.24060	0.87660	0.70520	0.12 (3)*
H8B	1.22450	0.92210	0.56530	0.02 (1)*
H9A	1.01120	0.94380	0.77480	0.14 (4)*
H9B	0.99080	0.98750	0.63280	0.06 (2)*
H10A	0.72530	0.90140	0.69240	0.09 (2)*
H10B	0.76060	0.91300	0.53600	0.06 (2)*
H11	0.91150	0.79710	0.70950	0.02 (1)*
H13A	0.64050	0.69000	0.65640	0.06 (2)*
H13B	0.66780	0.66090	0.50660	0.12 (3)*
H14	0.97270	0.67430	0.71770	0.05 (2)*
H15A	0.77220	0.58450	0.79220	0.14 (4)*
H15B	0.76930	0.54520	0.64630	0.10 (3)*
H15C	0.95400	0.54680	0.75200	0.09 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0691 (5)	0.0649 (5)	0.1331 (8)	0.0171 (4)	−0.0042 (4)	−0.0002 (6)
O2	0.081 (3)	0.097 (4)	0.048 (3)	−0.009 (3)	0.022 (2)	−0.006 (3)
O3	0.054 (2)	0.121 (4)	0.066 (3)	−0.007 (3)	−0.018 (2)	0.022 (3)
C4	0.045 (3)	0.052 (4)	0.059 (4)	0.000 (3)	−0.005 (3)	−0.005 (3)
C5	0.040 (3)	0.059 (4)	0.058 (4)	0.008 (3)	0.014 (3)	−0.002 (4)
C6	0.033 (3)	0.065 (4)	0.057 (4)	0.000 (3)	0.007 (3)	−0.002 (3)
C7	0.043 (3)	0.051 (3)	0.043 (4)	0.000 (3)	0.009 (3)	0.008 (3)
C8	0.065 (4)	0.056 (4)	0.069 (5)	−0.014 (3)	0.004 (4)	0.003 (4)
C9	0.098 (5)	0.042 (4)	0.090 (6)	−0.002 (3)	0.018 (4)	0.002 (4)
C10	0.061 (4)	0.065 (4)	0.073 (5)	0.010 (3)	0.021 (4)	0.004 (4)
C11	0.044 (3)	0.051 (3)	0.039 (3)	0.003 (3)	0.006 (3)	0.005 (3)
C12	0.034 (3)	0.081 (4)	0.034 (3)	0.001 (3)	0.011 (3)	−0.004 (3)
C13	0.040 (3)	0.073 (4)	0.058 (4)	−0.013 (3)	0.015 (3)	−0.012 (4)
C14	0.052 (3)	0.054 (4)	0.041 (4)	−0.013 (3)	0.007 (3)	−0.003 (3)
C15	0.101 (6)	0.070 (5)	0.089 (6)	−0.020 (5)	0.027 (5)	0.008 (5)

Geometric parameters (Å, °)

Br1—C4	1.960 (6)	C6—H6A	0.960

O2—C5	1.211 (9)	C6—H6B	0.960
O3—C12	1.210 (8)	C7—H7	0.960
C4—C5	1.514 (8)	C8—H8A	0.960
C4—C14	1.522 (8)	C8—H8B	0.960
C5—C6	1.493 (8)	C9—H9A	0.960
C6—C7	1.528 (8)	C9—H9B	0.960
C7—C8	1.531 (8)	C10—H10A	0.960
C7—C11	1.538 (8)	C10—H10B	0.960
C8—C9	1.541 (10)	C11—H11	0.960
C9—C10	1.518 (10)	C13—H13A	0.961
C10—C11	1.516 (8)	C13—H13B	0.959
C11—C12	1.501 (8)	C14—H14	0.960
C12—C13	1.485 (8)	C15—H15A	0.960
C13—C14	1.546 (8)	C15—H15B	0.961
C14—C15	1.511 (10)	C15—H15C	0.959
C4—H4	0.960		
Br1—C4—C5	104.5 (4)	C11—C7—H7	107.5
Br1—C4—C14	111.7 (4)	C7—C8—H8A	109.4
C5—C4—C14	116.0 (5)	C7—C8—H8B	111.4
O2—C5—C4	118.5 (6)	C9—C8—H8A	110.8
O2—C5—C6	121.9 (6)	C9—C8—H8B	110.5
C4—C5—C6	119.5 (6)	H8A—C8—H8B	109.0
C5—C6—C7	111.5 (5)	C8—C9—H9A	110.5
C6—C7—C8	114.7 (5)	C8—C9—H9B	110.7
C6—C7—C11	116.3 (5)	C10—C9—H9A	109.3
C8—C7—C11	102.9 (5)	C10—C9—H9B	111.2
C7—C8—C9	105.6 (5)	H9A—C9—H9B	109.0
C8—C9—C10	106.2 (5)	C9—C10—H10A	112.4
C9—C10—C11	104.1 (5)	C9—C10—H10B	110.2
C7—C11—C10	102.2 (5)	C11—C10—H10A	111.8
C7—C11—C12	112.7 (5)	C11—C10—H10B	109.2
C10—C11—C12	115.8 (5)	H10A—C10—H10B	109.0
O3—C12—C11	121.0 (6)	C7—C11—H11	109.3
O3—C12—C13	119.0 (6)	C10—C11—H11	109.5
C11—C12—C13	120.0 (5)	C12—C11—H11	107.2
C12—C13—C14	120.3 (5)	C12—C13—H13A	106.9
C4—C14—C13	109.4 (5)	C12—C13—H13B	106.4
C4—C14—C15	112.7 (5)	C14—C13—H13A	107.8
C13—C14—C15	110.3 (5)	C14—C13—H13B	106.1
Br1—C4—H4	109.9	H13A—C13—H13B	109.0
C5—C4—H4	108.8	C4—C14—H14	107.3
C14—C4—H4	105.9	C13—C14—H14	108.5
C5—C6—H6A	110.2	C15—C14—H14	108.6
C5—C6—H6B	108.6	C14—C15—H15A	111.6
C7—C6—H6A	107.8	C14—C15—H15B	108.3
C7—C6—H6B	109.7	C14—C15—H15C	109.6
H6A—C6—H6B	109.0	H15A—C15—H15B	109.1
C6—C7—H7	107.1	H15A—C15—H15C	109.2
C8—C7—H7	107.8	H15B—C15—H15C	109.0

3,5-Dibromo-4-methylbicyclo[6.3.0]undecane-2,6-dione

Crystal data

$C_{12}H_{16}Br_2O_2$	F_{000} = 1392
M_r = 352.07	D_x = 1.759 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
a = 16.027 (3) Å	Cell parameters from 25 reflections
b = 11.741 (4) Å	θ = 14.4–14.9º
c = 14.477 (3) Å	μ = 6.08 mm^{-1}
β = 102.56 (1)º	*T* = 298 K
V = 2659.0 (12) Å^3	Prism, colourless
Z = 8	0.5 × 0.5 × 0.4 mm

Data collection

Rigaku AFC-5 diffractometer	θ_{max} = 25.0º
θ–2θ scans	*h* = 0→19
Absorption correction: integration (Coppens et al., 1965)	*k* = 0→14
T_{min} = 0.050, T_{max} = 0.178	*l* = −17→17
2424 measured reflections	3 standard reflections
2337 independent reflections	every 100 reflections
1313 reflections with $I > 2\sigma(I)$	intensity decay: 2.6%
R_{int} = 0.020	

Refinement

Refinement on *F*	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.055	$w = 1/[\sigma^2(F) + 0.0009F^2]$
$wR(F^2)$ = 0.085	$(\Delta/\sigma)_{max}$ = 0.003
S = 1.88	$\Delta\rho_{max}$ = 0.90 e Å^{-3}
1975 reflections	$\Delta\rho_{min}$ = −0.88 e Å^{-3}
161 parameters	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.7533 (1)	0.0300 (2)	0.5073 (2)	0.0698 (8)
Br2	0.4886 (2)	0.3617 (2)	0.3931 (2)	0.083 (1)
O3	0.6047 (9)	−0.001 (1)	0.6521 (8)	0.077 (6)
O4	0.4446 (8)	0.186 (1)	0.5798 (9)	0.090 (6)
C5	0.634 (1)	0.072 (1)	0.510 (1)	0.050 (6)
C6	0.608 (1)	0.178 (2)	0.451 (1)	0.052 (7)

C7	0.519 (1)	0.216 (1)	0.459 (1)	0.051 (7)
C8	0.502 (1)	0.239 (2)	0.556 (1)	0.065 (7)
C9	0.558 (1)	0.320 (2)	0.623 (1)	0.063 (7)
C10	0.614 (1)	0.254 (2)	0.703 (1)	0.059 (6)
C11	0.671 (1)	0.330 (2)	0.777 (1)	0.070 (8)
C12	0.742 (2)	0.250 (2)	0.829 (1)	0.09 (1)
C13	0.740 (2)	0.143 (2)	0.766 (1)	0.073 (9)
C14	0.679 (1)	0.176 (1)	0.672 (1)	0.052 (6)
C15	0.637 (1)	0.076 (1)	0.615 (1)	0.051 (6)
C16	0.609 (1)	0.156 (2)	0.347 (1)	0.068 (8)
H5	0.59670	0.01170	0.48190	0.04 (4)*
H6	0.64790	0.23770	0.47570	0.08 (6)*
H7	0.47880	0.15910	0.42970	0.07 (6)*
H9A	0.52270	0.37250	0.64810	0.05 (5)*
H9B	0.59380	0.36170	0.58980	0.04 (4)*
H10	0.57790	0.21160	0.73450	0.06 (5)*
H11A	0.64020	0.36270	0.82040	0.14 (11)*
H11B	0.69610	0.38940	0.74710	0.07 (6)*
H12A	0.73050	0.22770	0.88860	0.08 (6)*
H12B	0.79750	0.28540	0.83960	0.18 (13)*
H13A	0.71720	0.07820	0.79200	0.15 (11)*
H13B	0.79640	0.12500	0.75740	0.14 (11)*
H14	0.71030	0.21910	0.63400	0.03 (4)*
H16A	0.59540	0.22510	0.31130	0.08 (6)*
H16B	0.56850	0.09840	0.32280	0.04 (5)*
H16C	0.66530	0.13100	0.34220	0.08 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.072 (1)	0.060 (1)	0.070 (1)	0.007 (1)	0.021 (1)	0.000 (1)
Br2	0.090 (2)	0.072 (2)	0.080 (2)	0.014 (1)	−0.002 (1)	0.005 (1)
O3	0.12 (1)	0.05 (1)	0.05 (1)	−0.03 (1)	0.03 (1)	0.00 (1)
O4	0.065 (9)	0.091 (11)	0.092 (10)	−0.037 (9)	0.034 (7)	−0.036 (9)
C5	0.06 (1)	0.03 (1)	0.05 (1)	−0.01 (1)	0.01 (1)	0.00 (1)
C6	0.06 (1)	0.05 (1)	0.04 (1)	−0.01 (1)	0.01 (1)	0.00 (1)
C7	0.06 (1)	0.04 (1)	0.05 (1)	−0.01 (1)	0.01 (1)	0.00 (1)
C8	0.05 (1)	0.05 (1)	0.08 (1)	−0.01 (1)	0.01 (1)	−0.02 (1)
C9	0.07 (1)	0.05 (1)	0.06 (1)	−0.01 (1)	0.02 (1)	−0.01 (1)
C10	0.05 (1)	0.05 (1)	0.06 (1)	−0.02 (1)	0.02 (1)	−0.02 (1)
C11	0.08 (1)	0.05 (1)	0.07 (1)	−0.03 (1)	0.02 (1)	−0.01 (1)
C12	0.11 (2)	0.08 (2)	0.06 (1)	−0.01 (2)	0.02 (1)	−0.02 (1)
C13	0.10 (2)	0.04 (1)	0.07 (1)	−0.02 (1)	0.01 (1)	−0.01 (1)
C14	0.06 (1)	0.04 (1)	0.04 (1)	−0.02 (1)	0.01 (1)	0.00 (1)
C15	0.06 (1)	0.03 (1)	0.05 (1)	0.00 (1)	0.01 (1)	0.00 (1)
C16	0.07 (1)	0.09 (2)	0.05 (1)	−0.01 (1)	0.01 (1)	0.01 (1)

Geometric parameters (Å, °)

Br1—C5	1.98 (2)	C5—H5	0.96
Br2—C7	1.97 (1)	C6—H6	0.96
O3—C15	1.22 (2)	C7—H7	0.96
O4—C8	1.22 (3)	C9—H9A	0.96
C5—C6	1.52 (3)	C9—H9B	0.96
C5—C15	1.51 (2)	C10—H10	0.95
C6—C7	1.52 (3)	C11—H11A	0.96
C6—C16	1.53 (3)	C11—H11B	0.95
C7—C8	1.51 (3)	C12—H12A	0.96
C8—C9	1.51 (3)	C12—H12B	0.96
C9—C10	1.52 (3)	C13—H13A	0.96
C10—C11	1.53 (3)	C13—H13B	0.96
C10—C14	1.53 (3)	C14—H14	0.96
C11—C12	1.54 (4)	C16—H16A	0.96
C12—C13	1.55 (3)	C16—H16B	0.95
C13—C14	1.54 (3)	C16—H16C	0.97
C14—C15	1.51 (2)		
Br1—C5—C6	109.9 (10)	C8—C7—H7	107.2
Br1—C5—C15	102.0 (10)	C8—C9—H9A	109.3
C6—C5—C15	119.1 (12)	C8—C9—H9B	109.7
C5—C6—C7	110.2 (14)	C10—C9—H9A	109.8
C5—C6—C16	110.8 (17)	C10—C9—H9B	108.9
C7—C6—C16	109.5 (13)	H9A—C9—H9B	109.1
Br2—C7—C6	110.8 (12)	C9—C10—H10	108.2
Br2—C7—C8	102.7 (11)	C11—C10—H10	107.5
C6—C7—C8	118.9 (13)	C14—C10—H10	110.6
O4—C8—C7	118.0 (16)	C10—C11—H11A	112.1
O4—C8—C9	121.3 (14)	C10—C11—H11B	110.7
C7—C8—C9	120.5 (14)	C12—C11—H11A	110.8
C8—C9—C10	109.9 (19)	C12—C11—H11B	109.1
C9—C10—C11	113.6 (19)	H11A—C11—H11B	109.4
C9—C10—C14	114.3 (13)	C11—C12—H12A	109.9
C11—C10—C14	102.3 (13)	C11—C12—H12B	111.9
C10—C11—C12	104.5 (18)	C13—C12—H12A	109.0
C11—C12—C13	106.8 (16)	C13—C12—H12B	110.1
C12—C13—C14	104.1 (18)	H12A—C12—H12B	109.1
C10—C14—C13	103.5 (14)	C12—C13—H13A	112.0
C10—C14—C15	112.1 (14)	C12—C13—H13B	110.8
C13—C14—C15	114.2 (13)	C14—C13—H13A	108.9
O3—C15—C5	119.7 (12)	C14—C13—H13B	112.0
O3—C15—C14	121.1 (13)	H13A—C13—H13B	109.0
C5—C15—C14	119.2 (12)	C10—C14—H14	109.0
Br1—C5—H5	108.8	C13—C14—H14	109.1
C6—C5—H5	107.3	C15—C14—H14	108.8
C15—C5—H5	109.3	C6—C16—H16A	109.7
C5—C6—H6	108.0	C6—C16—H16B	109.3

C7—C6—H6	108.4	C6—C16—H16C	109.4
C16—C6—H6	109.9	H16A—C16—H16B	110.2
Br2—C7—H7	108.6	H16A—C16—H16C	108.9
C6—C7—H7	108.2	H16B—C16—H16C	109.3

1,7-Dibromo-4-methylbicyclo[6.3.0]undecane-2,6-dione

Crystal data

$C_{12}H_{16}Br_2O_2$	$Z = 2$
$M_r = 352.07$	$F_{000} = 348$
Triclinic, $P\overline{1}$	$D_x = 1.823$ Mg m^{-3}
$a = 7.228$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 15.875$ (3) Å	Cell parameters from 25 reflections
$c = 5.851$ (2) Å	$\theta = 14.8$–15.0°
$\alpha = 99.57$ (3)°	$\mu = 6.30$ mm^{-1}
$\beta = 98.31$ (3)°	$T = 298$ K
$\gamma = 77.20$ (2)°	Prism, colourless
$V = 641.5$ (3) Å^3	0.6 × 0.4 × 0.3 mm

Data collection

Rigaku AFC-5 diffractometer	$\theta_{max} = 26.0^\circ$
θ–2θ scans	$h = 0 \rightarrow 9$
Absorption correction: integration (Coppens et al., 1965)	$k = -20 \rightarrow 20$
$T_{min} = 0.092$, $T_{max} = 0.204$	$l = -7 \rightarrow 7$
2729 measured reflections	3 standard reflections
2515 independent reflections	every 100 reflections
1842 reflections with $I > 2\sigma(I)$	intensity decay: none
$R_{int} = 0.011$	

Refinement

Refinement on F	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F) + 0.0009F^2]$
$wR(F^2) = 0.055$	$(\Delta/\sigma)_{max} = 0.0004$
$S = 1.23$	$\Delta\rho_{max} = 0.89$ e Å^{-3}
2287 reflections	$\Delta\rho_{min} = -1.04$ e Å^{-3}
161 parameters	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	−0.19210 (7)	0.76010 (4)	0.40240 (9)	0.0605 (2)

Br2	0.56250 (7)	0.61370 (3)	0.7544 (1)	0.0558 (2)
O3	−0.0880 (5)	0.8768 (2)	0.9618 (6)	0.061 (1)
O4	0.3430 (5)	0.7843 (2)	1.1903 (5)	0.053 (1)
C5	−0.0188 (6)	0.7499 (3)	0.6916 (7)	0.035 (1)
C6	0.1666 (6)	0.6854 (3)	0.6382 (7)	0.031 (1)
C7	0.1301 (7)	0.5920 (3)	0.5823 (8)	0.046 (2)
C8	0.1561 (8)	0.5579 (3)	0.8154 (9)	0.054 (2)
C9	0.2406 (7)	0.6242 (3)	0.9976 (7)	0.045 (2)
C10	0.3123 (6)	0.6796 (3)	0.8564 (7)	0.034 (1)
C11	0.3604 (6)	0.7651 (3)	0.9832 (7)	0.036 (1)
C12	0.4274 (6)	0.8237 (3)	0.8493 (8)	0.040 (2)
C13	0.2840 (7)	0.9094 (3)	0.8188 (8)	0.040 (2)
C14	0.0976 (6)	0.8942 (3)	0.6773 (8)	0.041 (2)
C15	−0.0084 (6)	0.8445 (3)	0.7916 (8)	0.039 (2)
C16	0.3732 (8)	0.9669 (3)	0.7000 (9)	0.055 (2)
H5	−0.07740	0.72750	0.79930	0.013 (8)*
H6	0.21960	0.70620	0.52220	0.018 (9)*
H7A	0.00250	0.59280	0.50760	0.04 (1)*
H7B	0.21880	0.55630	0.48100	0.05 (1)*
H8A	0.24210	0.50220	0.80890	0.06 (2)*
H8B	0.03570	0.55190	0.85480	0.05 (1)*
H9A	0.34350	0.59630	1.09990	0.04 (1)*
H9B	0.14440	0.65980	1.08890	0.04 (1)*
H12A	0.54420	0.83780	0.93090	0.04 (1)*
H12B	0.45170	0.79280	0.69760	0.04 (1)*
H13	0.25540	0.93860	0.97070	0.08 (2)*
H14A	0.01720	0.94980	0.65600	0.04 (1)*
H14B	0.12570	0.86260	0.52750	0.04 (1)*
H16A	0.49030	0.97580	0.79190	0.10 (2)*
H16B	0.39850	0.93810	0.54670	0.06 (2)*
H16C	0.28750	1.02190	0.68710	0.08 (2)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0449 (3)	0.0805 (4)	0.0530 (3)	−0.0137 (3)	−0.0128 (2)	0.0195 (3)
Br2	0.0457 (3)	0.0409 (3)	0.0763 (4)	0.0090 (2)	0.0162 (3)	0.0108 (3)
O3	0.067 (2)	0.047 (2)	0.075 (3)	−0.006 (2)	0.041 (2)	−0.007 (2)
O4	0.071 (2)	0.053 (2)	0.033 (2)	−0.020 (2)	0.001 (2)	−0.001 (2)
C5	0.039 (2)	0.040 (2)	0.030 (2)	−0.010 (2)	0.004 (2)	0.011 (2)
C6	0.037 (2)	0.030 (2)	0.026 (2)	−0.006 (2)	0.004 (2)	0.005 (2)
C7	0.065 (3)	0.034 (2)	0.039 (3)	−0.014 (2)	0.007 (2)	−0.002 (2)
C8	0.071 (4)	0.043 (3)	0.052 (3)	−0.021 (3)	0.003 (3)	0.012 (2)
C9	0.058 (3)	0.043 (3)	0.034 (2)	−0.009 (2)	0.003 (2)	0.013 (2)
C10	0.039 (2)	0.027 (2)	0.031 (2)	0.001 (2)	0.006 (2)	0.003 (2)
C11	0.034 (2)	0.037 (2)	0.032 (2)	−0.004 (2)	−0.002 (2)	0.003 (2)
C12	0.037 (2)	0.041 (2)	0.043 (3)	−0.011 (2)	0.007 (2)	0.000 (2)
C13	0.054 (3)	0.029 (2)	0.041 (3)	−0.012 (2)	0.018 (2)	−0.002 (2)

C14	0.045 (3)	0.031 (2)	0.050 (3)	−0.006 (2)	0.009 (2)	0.013 (2)
C15	0.038 (2)	0.036 (2)	0.042 (3)	0.001 (2)	0.008 (2)	0.012 (2)
C16	0.071 (4)	0.043 (3)	0.061 (3)	−0.021 (3)	0.020 (3)	0.007 (2)

Geometric parameters (Å, °)

Br1—C5	1.961 (5)	C5—H5	0.960
Br2—C10	1.997 (5)	C6—H6	0.960
O3—C15	1.204 (6)	C7—H7A	0.960
O4—C11	1.217 (5)	C7—H7B	0.960
C5—C6	1.532 (7)	C8—H8A	0.960
C5—C15	1.532 (7)	C8—H8B	0.960
C6—C7	1.537 (7)	C9—H9A	0.960
C6—C10	1.535 (6)	C9—H9B	0.960
C7—C8	1.521 (7)	C12—H12A	0.960
C8—C9	1.530 (7)	C12—H12B	0.960
C9—C10	1.514 (6)	C13—H13	0.960
C10—C11	1.521 (7)	C14—H14A	0.960
C11—C12	1.505 (7)	C14—H14B	0.960
C12—C13	1.536 (7)	C16—H16A	0.960
C13—C14	1.521 (7)	C16—H16B	0.960
C13—C16	1.528 (7)	C16—H16C	0.959
C14—C15	1.499 (7)		
Br1—C5—C6	109.3 (3)	C6—C7—H7B	110.6
Br1—C5—C15	103.9 (3)	C8—C7—H7A	110.6
C6—C5—C15	118.6 (4)	C8—C7—H7B	110.7
C5—C6—C7	110.0 (4)	H7A—C7—H7B	109.0
C5—C6—C10	111.3 (4)	C7—C8—H8A	110.3
C7—C6—C10	102.0 (4)	C7—C8—H8B	110.5
C6—C7—C8	106.0 (4)	C9—C8—H8A	109.7
C7—C8—C9	106.9 (4)	C9—C8—H8B	110.4
C8—C9—C10	104.6 (4)	H8A—C8—H8B	109.0
Br2—C10—C6	107.6 (3)	C8—C9—H9A	111.8
Br2—C10—C9	108.2 (4)	C8—C9—H9B	110.8
Br2—C10—C11	102.8 (3)	C10—C9—H9A	110.5
C6—C10—C9	104.1 (4)	C10—C9—H9B	110.1
C6—C10—C11	116.6 (4)	H9A—C9—H9B	109.0
C9—C10—C11	117.1 (4)	C11—C12—H12A	108.5
O4—C11—C10	119.0 (4)	C11—C12—H12B	108.7
O4—C11—C12	121.9 (5)	C13—C12—H12A	108.2
C10—C11—C12	119.2 (4)	C13—C12—H12B	108.5
C11—C12—C13	113.9 (4)	H12A—C12—H12B	109.0
C12—C13—C14	112.3 (4)	C12—C13—H13	108.1
C12—C13—C16	109.4 (4)	C14—C13—H13	107.9
C14—C13—C16	109.6 (4)	C16—C13—H13	109.5
C13—C14—C15	113.3 (4)	C13—C14—H14A	108.5
O3—C15—C5	118.0 (4)	C13—C14—H14B	108.4
O3—C15—C14	121.9 (5)	C15—C14—H14A	108.4
C5—C15—C14	120.1 (4)	C15—C14—H14B	109.2

Br1—C5—H5	109.1	H14A—C14—H14B	108.9
C6—C5—H5	108.3	C13—C16—H16A	108.4
C15—C5—H5	107.3	C13—C16—H16B	109.2
C5—C6—H6	107.8	C13—C16—H16C	110.2
C7—C6—H6	117.0	H16A—C16—H16B	110.0
C10—C6—H6	108.7	H16A—C16—H16C	110.1
C6—C7—H7A	109.9	H16B—C16—H16C	109.0