Sterically Stabilised Cyclopropenonophanes and an Electronically Stabilised Cyclopropenethionophane

—

Syntheses, Structural Properties and Reactivity

Daniel B. Werz, Andreea Schuster, Rolf Gleiter,* Frank Rominger

Organisch-Chemisches Institut der Universität Heidelberg,

Im Neuenheimer Feld 270, D-69120 Heidelberg, Germany.

E-mail address: rolf.gleiter@urz.uni-heidelberg.de

Supporting Information

1.	Experimental Part	S2
1.1	General Experimental	S2
1.2	Preparation of Compounds and Analytical Data of Compounds 10, 11, 16-19, 5-7, 22 and 25	S2
2.	X-Ray Diffraction Analyses	S7
2.1	General	S7
2.2	Crystallographic Data of 6α	S7
2.3	Crystallographic Data of 6β	S14
2.4	Crystallographic Data of 7α	S21
2.5	Crystallographic Data of 7β	S24
2.6	Crystallographic Data of 19	S28
2.7	Crystallographic Data of 22	S32
2.8	Crystallographic Data of 23α	S35
2.9	Crystallographic Data of 23β	S38
2.10	Crystallographic Data of 24	S41

1. Experimental Part

1.1 General Experimental

All reactions were conducted (if not otherwise stated) in oven-dried glassware under an argon atmosphere with magnetic stirring. THF, diethylether and DME were dried with sodium/benzophenone and distilled under argon before use, CH₂Cl₂ was dried using CaH₂ and distilled before use. Melting points are uncorrected. Materials used for column chromatography: Silica gel 60 and alumina (Alox III). Alox III was obtained by adding 6% (Vol) of water to neutral alumina. ¹H NMR and ¹³C NMR were recorded either at 300 and 500 MHz (¹H NMR) or 75.5 and 125.8 MHz (¹³C NMR), respectively, using the solvent as internal standard. The IR spectra were recorded with a FT-IR instrument. The UV light absorption spectra were recorded using a FT-UV spectrometer. The high-resolution mass spectra (HRMS) were recorded in the EI (70 eV) mode, the CI mode and the positive ion FAB mode in *m*-nitrobenzylalcohol. Elemental analyses were carried out by the Mikroanalytisches Laboratorium der Universität Heidelberg.

1.2 Preparation and Analytical Data of Compounds **10**, **11**, **16-19**, **5-7**, **22**, and **25**

Preparation of 3,3,6,6-Tetramethylsuberinic acid dichloride (10). 4.48 g (20.0 mmol) of dicarboxylic acid **8** are dissolved in 12 ml (170 mmol) of thionylchloride and stirred for 6 h using a one-necked round-bottom flask with a reflux condenser and magnetic stirring. After the reaction is finished, excess thionylchloride is removed; the residue is distilled at 96 °C /0.3 mbar. 4.53 g (88%) of **10** are obtained as a slightly yellow oil with a characteristic smell: ¹H NMR (CDCl₃, 300 MHz) δ 1.01 (s, 12H, CH₃), 1.29 (m, 4H, CH₂) 2.82 (s, 4H, CH₂); ¹³C NMR (CDCl₃, 75 MHz) δ 26.6 (CH₃), 32.6 (C), 34.8 (CH₂), 57.7 (CH₂), 171.3 (COCl).

Preparation of 3,3,7,7-Tetramethylazelaic acid dichloride (11). 15.1 g (61.9 mmol) of dicarboxylic acid **9** are dissolved in 21 ml (300 mmol) of thionylchloride and stirred for 6 h using a one-necked round-bottom flask with a reflux condenser and magnetic stirring. After the reaction is finished, excess thionylchloride is removed; the residue is distilled at 113 °C /0.1 mbar, 16.3 g (94%) of **11** are obtained as a yellow oil with a characteristic smell: ¹H NMR (CDCl₃, 300 MHz) δ 1.05 (s, 12H, CH₃), 1.31 (m, 6H, CH₂) 2.84 (s, 4H, CH₂); ¹³C NMR (CDCl₃, 75 MHz) δ 18.5 (CH₂), 26.8 (CH₃), 34.6 (C), 42.1 (CH₂), 58.3 (CH₂), 171.5 (COCl).

Preparation of 3,3,6,6,10,10,13,13-octamethylcyclotetradecane-1,8-dione (16). 18 ml (130 mmol) of triethylamine in 150 ml of diethylether under an argon atmosphere were cooled to 0 °C. A solution of 4.52 g (17 mmol) of **10** in 15 ml of diethylether was added dropwise while a white solid precipitated. The suspension was stirred for 10 h at 0 °C and for 3 d at room temperature, it was filtered to remove the precipitate. The filtrate was washed twice with 5% hydrochloric acid, saturated NaHCO₃ solution and brine, dried over Na₂SO₄. The solvent was removed by rotary evaporation. The yellow residue was

dissolved in 65 ml of dioxane and added to a solution of 2.5 g (45 mmol) KOH in 10 ml of water. This mixture was refluxed for 2 d. Dioxane was removed by rotary evaporation, the viscous residue was diluted by water and extracted 5x with diethylether. The combined organic phases were dried over Na₂SO₄. The solvent was removed by rotary evaporation and the residue was purified by column chromatography on alumina (Alox III) using petroleum ether as eluent. A second column chromatography (silica gel, CCl₄/CHCl₃ 1:0 → 1:3) improved the purity of the compound. 66 mg (2.3%) of a colorless crystalline powder were obtained: mp 218 °C; ¹H NMR (CDCl₃, 300 MHz) δ 0.87 (s, 24H, CH₃), 1.36 (s, 8H, CH₂) 2.21 (s, 8H, CH₂); ¹³C NMR (CDCl₃, 75 MHz) δ 28.7 (CH₃), 32.3 (C), 32.3 (CH₂), 50.5 (CH₂), 209.2 (CO).

Preparation of 3,3,7,7,11,11,15,15-Octamethylcyclohexadecane-1,9-dione (17). 65 ml (470 mmol) of triethylamine in 500 ml of diethylether under an argon atmosphere were cooled to 0 °C. A solution of 16.3 g (58.2 mmol) of **11** in 50 ml of diethylether was added dropwise while a white solid precipitated. The suspension was stirred for 10 h at 0 °C and for 3 d at room temperature, it was filtered to remove the precipitate. The filtrate was washed twice with 5% hydrochloric acid, saturated NaHCO₃ solution and brine, dried over Na₂SO₄. The solvent was removed by rotary evaporation. The yellow residue was dissolved in 280 ml of dioxane and added to a solution of 11.4 g (200 mmol) KOH in 25 ml of water. This mixture was refluxed for 2 d. Dioxane was removed by rotary evaporation, the viscous residue was diluted by water and extracted 5x with diethylether. The combined organic phases were dried over Na₂SO₄. The solvent was removed by rotary evaporation and the residue was purified by column chromatography on alumina (Alox III) using petroleum ether as eluent. 618 mg (4.0%) of colorless crystals were obtained: mp 150 °C; ¹H NMR (CDCl₃, 300 MHz) δ 0.92 (s, 24H, CH₃), 1.01 (m, 4H, CH₂), 1.30 (m, 8H, CH₂) 2.21 (s, 8H, CH₂); ¹³C NMR (CDCl₃, 75 MHz) δ 19.5 (CH₂), 28.6 (CH₃), 32.9 (C), 40.9 (CH₂), 51.9 (CH₂), 209.0 (CO).

Preparation of 3,3,6,6,10,10,13,13-Octamethyl-2,7,9,14-tetrabromocyclotetradecane-1,8-dione (18). 66 mg (0.196 mmol) of diketone **16** were dissolved in 1.5 ml of CH₂Cl₂. 176 mg (1.1 mmol) of bromine dissolved in 1.5 ml of CH₂Cl₂ were added and stirred for 5 h at room temperature (Note: No Ar atmosphere!). After 10 minutes the formation of a gas was observed (HBr). For work-up the reaction mixture was washed subsequently with saturated Na₂SO₃ solution, then with 5% NaHCO₃ solution and brine. All aqueous phases were extracted with CH₂Cl₂. The combined organic phases were dried over Na₂SO₄, the solvent was removed by rotary evaporation. The crude product was purified by column chromatography on alumina (Alox III) using CCl₄/CHCl₃ (1:0 → 3:1) as eluent: 59 mg (46%) of **18** (mixture of stereoisomers!) were obtained as a colorless powder: ¹H NMR (CD₂Cl₂, 300 MHz) δ 1.08 (s, 12H, CH₃), 1.09 (s, 12H, CH₃), 1.16-1.26 (m, 6H, CH₂), 1.52 (b, 2H, CH₂), 4.46 (b, 4H, CHBr); ¹³C NMR (CD₂Cl₂, 75 MHz) δ 27.5-27.8 (CH₃), 28.4 (CH₃), 34.4 (CH₂), 38.7 (C), 62.8 (CHBr), 63.1-63.5 (CHBr), 198.5 (C=O); IR (CDCl₃) 2968, 2935, 1725, 1330, 1051 cm⁻¹; UV-vis (CH₂Cl₂) λ_{max}, nm (ε, M⁻¹ cm⁻¹) 232 (1550), 294 (260), 318 (310); HRMS (CI+) calcd for C₂₂H₃₇⁸¹Br₄O₂ 656.9445, found 656.9422.

Preparation of 3,3,7,7,11,11,15,15-Octamethyl-2,8,10,16-tetrabromocyclohexadecane-1,9-dione (19).

200 mg (0.549 mmol) of diketone **17** were dissolved in 1.5 ml of CH₂Cl₂. 492 mg (1.1 mmol) of bromine dissolved in 1.5 ml of CH₂Cl₂ were added and stirred for 5 h at room temperature (Note: No Ar atmosphere!). After 5 minutes the formation of a gas was observed (HBr), after 15 minutes the deep brown color disappeared. For work-up the reaction mixture was washed subsequently with saturated Na₂SO₃ solution, then with 5% NaHCO₃ solution and brine. All aqueous phases were extracted with CH₂Cl₂. The combined organic phases were dried over Na₂SO₄, the solvent was removed by rotary evaporation and 300 mg (80%) of pure **19** (only one stereoisomer!) were obtained as a colorless powder: mp 210 °C (dec.); ¹H NMR (CDCl₃, 300 MHz) δ 1.11 (s, 12H, CH₃), 1.15 (s, 12H, CH₃), 1.22-1.39 (m, 12H, CH₂), 4.48 (s, 4H, CHBr); ¹³C NMR (CDCl₃, 75 MHz) δ 19.2 (CH₃), 23.0 (CH₃), 27.6 (CH₃), 38.3 (C), 40.5 (CH₂), 62.3 (CHBr), 197.3 (C=O); IR (KBr) 2964, 1715, 1632, 1464, 1370 cm⁻¹; UV-vis (CH₂Cl₂) λ_{max}, nm (ε, M⁻¹ cm⁻¹) 232 (39800), 318 (320). Anal. Calcd for C₂₄H₄₀Br₄O₂: C, 42.38; H, 5.93. Found: C, 42.44; H, 5.95.

Preparation of Tricyclo[11.1.0.0^{6,8}]tetradeca-2,2,5,5,9,9,12,12-octamethyl-1(13),6(8)-diene-7,14-dione (5). 57 mg (0.509 mmol) of absolutely anhydrous KOtBu were dissolved in 1.5 ml of THF and cooled to -40 °C. 59 mg (0.091 mmol) of tetrabromide **18** dissolved in 1.5 ml of THF were added dropwise while the color of the reaction mixture turned immediately to brown. After stirring for 30 minutes the solution is added to 50 ml of cold (0 °C) 5% NaH₂PO₃ solution and extracted with cold CH₂Cl₂ (-20 °C). The organic phase was washed with brine and dried over Na₂SO₄. The solvent was removed by rotary evaporation and the residue separated by column chromatography on alumina (Alox III) using CCl₄ → CHCl₃ as eluent. 8 mg (27%) of **5** as a colorless crystalline solid were obtained: ¹H NMR (CD₂Cl₂, 500 MHz) δ 1.30 (s, 24H, CH₃), 1.50 (s, 8H, CH₂); ¹³C NMR (CDCl₃, 125 MHz) δ 26.9 (CH₃), 37.5 (C), 39.1 (CH₂), 160.8 (C=O), 167.8 (C=C); IR (KBr) 2969, 2931, 2873, 1854, 1814, 1618 cm⁻¹; UV-vis (CH₂Cl₂) λ_{max}, nm (ε, M⁻¹ cm⁻¹) 232 (1200), 260 (760), 276 (810). HRMS (FAB+) calcd for C₂₂H₃₃O₂ 329.2481, found 329.2482.

Preparation of Tricyclo[13.1.0.0^{7,9}]hexadeca-2,2,6,6,10,10,14,14-octamethyl-1(15),7(9)-diene-8,16-dione (6). 148 mg (1.32 mmol) of absolutely anhydrous KOtBu were dissolved in 1.5 ml of THF and cooled to -40 °C. 150 mg (0.221 mmol) of tetrabromide **19** dissolved in 1.5 ml of THF were added dropwise while the color of the reaction mixture turned immediately to brown. After stirring for 30 minutes the solution is added to 50 ml of cold (0 °C) 5% NaH₂PO₃ solution and extracted with cold CH₂Cl₂ (-20 °C). The organic phase was washed with brine and dried over Na₂SO₄. The solvent was removed by rotary evaporation at 10 °C and the residue separated by column chromatography on alumina (Alox III) using CHCl₃/EtOH (100:1) as eluent. 40 mg (51%) of **6** as a colorless crystalline solid were obtained: ¹H NMR (CDCl₃, 300 MHz) δ 1.11-1.13 (m, 4H, CH₂), 1.29 (s, 24H, CH₃), 1.52-1.59 (m, 8H, CH₂); ¹³C NMR (CDCl₃, 75 MHz) δ 22.4 (CH₂), 26.9 (CH₃), 37.5 (C), 41.9 (CH₂), 161.3 (C=O), 166.0 (C=C); IR (CHCl₃) 2966, 2871, 1832, 1614 cm⁻¹; UV-vis (CH₂Cl₂) λ_{max}, nm (ε, M⁻¹ cm⁻¹) 230 (480), 260

(250), 322 (150). HRMS (FAB+) calcd for $C_{24}H_{37}O_2$ 357.2794, found 357.2801. Anal. Calcd for $C_{24}H_{36}O_2$: C, 80.85; H, 10.18. Found: C, 79.90; H, 10.14.

Preparation of 1,1',7,7'-Tetrathia[7.7]cyclopropenethionophane (7). To a solution of 140 mg (0.38 mmol) of 1,1',7,7'-tetrathia[7.7]cyclopropenonophane (**20**) in 120 ml of DME under an argon atmosphere was added dropwise 386 mg (3.04 mmol) of oxalylchloride. The solution is stirred for 4 h in a water bath of about 35-40 °C. To protect the reaction from daylight the flask is wrapped by aluminium foil. After 4 h the reaction vessel is allowed to cool at room temperature, then cooled by an ice-bath to 0 °C. 500 mg (6.56 mmol) of thioacetic acid are added, the solution is stirred for 1 h at 0 °C. Solvent is evaporated in vacuo and the residue absorbed on celite. The pure product **7** (47 mg, 31%) is obtained by column chromatography on alumina (Alox III) using hexane / ethyl acetate (3:1) as eluent. For further purification the product is recrystallized from DME: Yellow solid; decomposition > 110 °C; 1H NMR (CD_2Cl_2 , 500 MHz) δ 1.52 (m, 4H, $SCH_2CH_2CH_2$), 1.84 (pquint., 8H, SCH_2CH_2), 3.18 (m, 8H, SCH_2); ^{13}C NMR (CD_2Cl_2 , 125 MHz) δ 25.0 ($SCH_2CH_2CH_2$), 30.4 (SCH_2CH_2), 34.0 (SCH_2), 155.8 ($C=C$), 172.3 (CS); IR (KBr) 2925, 2855, 1604, 1458, 1416, 1275, 1112, 1060 cm^{-1} ; UV-vis (CH_2Cl_2) λ_{max} , nm (ϵ , $M^{-1} cm^{-1}$) 270 (20900), 352 (2040); HRMS (FAB+) calcd for $C_{16}H_{20}S_6$ 404.9968, found 404.9990.

Preparation of Bis(methylthio)cyclopropenethione (22). To a solution of 56 mg (0.38 mmol) of bis(methylthio)cyclopropenone in 120 ml of DME under an argon atmosphere was added dropwise 97 mg (0.77 mmol) of oxalylchloride. The solution is stirred for 3 h in a water bath of about 35-40 °C. To protect the reaction from daylight the flask is wrapped by aluminium foil. After 3 h the reaction vessel is allowed to cool at room temperature, then cooled by an ice-bath to 0 °C. 250 mg (3.28 mmol) of thioacetic acid are added, the solution is stirred for 1 h at 0 °C. Solvent is evaporated in vacuo and the residue absorbed on celite. The pure product (28 mg, 45%) is obtained by column chromatography on alumina (Alox III) using hexane / ethyl acetate (5:1) as eluent: Yellow solid; mp 110 °C; 1H NMR ($CDCl_3$, 500 MHz) δ 2.87 (m, 6H, SCH_3); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 15.9 (SCH_3), 155.3 ($C=C$), 173.4 (CS); IR (KBr) 2995, 2920, 1680, 1633, 1422, 1327, 1285, 1131, 977 cm^{-1} ; UV-vis (CH_2Cl_2) λ_{max} , nm (ϵ , $M^{-1} cm^{-1}$) 270 (15100), 296 (7400), 416 (310); (EI, 70 eV) 162 (100), 147 (24), 115 (59), 100 (67), 88 (15); HRMS (EI+) calcd for $C_5H_6S_3$ 161.9632, found 161.9620.

Preparation of 3,3,7,7,10,10,14,14-Octamethylcyclotetradeca-1,8-diyne (25). A 10-ml-flask with 19 mg (0.053 mmol) of cyclopropenonophane **6** under argon was heated for 30 minutes to 240 °C and then allowed to cool to room temperature. The so obtained solid was dissolved in CCl_4 . GC and GC/MS experiments showed still starting material. The mixture is separated by column chromatography on alumina (Alox III) with petroleum ether as eluent. 3 mg (19%) of a colorless crystalline solid are obtained: 1H NMR (C_6D_6 , 300 MHz) δ 0.95 (m, 4H, CH_2), 1.29 (s, 24H, CH_3), 1.36-1.40 (m, 8H, CH_2); ^{13}C NMR (C_6D_6 , 75 MHz) δ 23.1 (CH_2), 30.2 (CH_3), 31.7 (C), 44.8 (CH_2), 87.7 (CC); IR ($CDCl_3$) 2964, 2926, 2854, 2235, 1262, 1042 cm^{-1} ; UV-vis (CH_2Cl_2) λ_{max} , nm (ϵ , $M^{-1} cm^{-1}$) 230 (600), 260 (390), 276

(430); (EI, 70 eV) 300 (46), 285 (100), 229 (43), 217 (85), 161 (79); HRMS (EI+) calcd for $C_{22}H_{36}$ 300.2817, found 300.2810.

2. X-Ray Diffraction Analyses

2.1 General

The reflections were collected with a CCD diffractometer (Mo- K_{α} radiation, graphite monochromator). Intensities were corrected for Lorentz and polarisation effects, an empirical absorption correction was applied using SADABS based on the Laue symmetry of the reciprocal space. The structures were solved by direct methods. The structural parameters of the non-hydrogen atoms were refined anisotropically according to a full-matrix least-squares technique (F^2). The hydrogen atoms were calculated according to stereochemical aspects. Structure solution and refinement were carried out with SHELXTL (5.10) software package. ORTEP drawings were obtained using the ORTEP-3 for Windows program by L. Farrugia.

2.2 Crystallographic Data for **6 α**

Table 1: Crystal data and structure refinement for **6 α** .

Empirical formula	C ₂₄ H ₃₆ O ₂	
Formula weight	356.53	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	orthorhombic	
Space group	Pna2 ₁	
Z	8	
Unit cell dimensions	a = 15.0286(3) Å	α = 90 deg.
	b = 7.4850(2) Å	β = 90 deg.
	c = 39.3182(8) Å	γ = 90 deg.
Volume	4422.87(17) Å ³	
Density (calculated)	1.07 Mg/m ³	
Absorption coefficient	0.07 mm ⁻¹	
Crystal size	.44 x .42 x .30 mm ³	
Theta range for data collection	2.1 to 25.6 deg.	
Index ranges	-18 ≤ h ≤ 17, -8 ≤ k ≤ 8, -46 ≤ l ≤ 45	
Reflections collected	23546	
Independent reflections	7583 (R(int) = 0.0215)	
Observed reflections	6695 (I > 2σ(I))	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	7583 / 1 / 757	
Goodness-of-fit on F ²	1.04	
Final R indices (I > 2σ(I))	R1 = 0.034, wR2 = 0.083	
Absolute structure parameter	1.2(10)	
Largest diff. peak and hole	0.14 and -0.18 eÅ ⁻³	

Table 2: Atomic coordinates and equivalent isotropic displacement parameters (Å²) for **6 α** . U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U _{eq}
O1	-0.5204(1)	-1.1552(2)	-0.3577(1)	0.0522(4)
O2	-0.4098(1)	-1.1851(2)	-0.4770(1)	0.0443(4)

O3	-0.3373(1)	-0.6813(2)	-0.5868(1)	0.0457(4)
O4	-0.2297(1)	-0.6777(2)	-0.7078(1)	0.0516(4)
C1	-0.4449(1)	-1.4595(2)	-0.3523(1)	0.0272(4)
C2	-0.4662(2)	-1.2754(2)	-0.3536(1)	0.0301(5)
C3	-0.3803(1)	-1.3377(2)	-0.3451(1)	0.0282(4)
C4	-0.2863(1)	-1.2981(3)	-0.3349(1)	0.0347(5)
C5	-0.2378(2)	-1.2077(3)	-0.3650(1)	0.0339(5)
H5A	-0.2690(16)	-1.099(3)	-0.3718(6)	0.052(7)
H5B	-0.1804(17)	-1.168(3)	-0.3559(6)	0.045(6)
C6	-0.2282(2)	-1.3213(3)	-0.3967(1)	0.0383(5)
H6A	-0.2847(17)	-1.353(3)	-0.4046(6)	0.045(6)
H6B	-0.1976(16)	-1.439(3)	-0.3908(6)	0.054(7)
C7	-0.1802(2)	-1.2236(3)	-0.4253(1)	0.0325(5)
H7A	-0.2094(14)	-1.110(3)	-0.4310(5)	0.037(5)
H7B	-0.1232(17)	-1.195(3)	-0.4197(6)	0.036(6)
C8	-0.1740(1)	-1.3283(2)	-0.4590(1)	0.0303(4)
C9	-0.2680(1)	-1.3678(2)	-0.4698(1)	0.0251(4)
C10	-0.3553(1)	-1.3061(3)	-0.4755(1)	0.0282(4)
C11	-0.3323(1)	-1.4892(2)	-0.4770(1)	0.0258(4)
C12	-0.3565(1)	-1.6834(2)	-0.4796(1)	0.0291(4)
C13	-0.3582(2)	-1.7603(2)	-0.4432(1)	0.0279(6)
H13A	-0.3753(16)	-1.885(3)	-0.4453(6)	0.046(6)
H13B	-0.302(2)	-1.747(3)	-0.4334(7)	0.028(6)
C14	-0.4241(1)	-1.6738(3)	-0.4185(1)	0.0300(4)
H14A	-0.4850(17)	-1.698(3)	-0.4265(6)	0.033(5)
H14B	-0.4175(12)	-1.547(3)	-0.4179(5)	0.024(5)
C15	-0.4114(2)	-1.7445(2)	-0.3823(1)	0.0297(6)
H15A	-0.4239(14)	-1.868(3)	-0.3815(5)	0.037(6)
H15B	-0.353(2)	-1.730(3)	-0.3758(7)	0.037(7)
C16	-0.4694(1)	-1.6539(2)	-0.3549(1)	0.0332(4)
C17	-0.2384(2)	-1.4686(3)	-0.3240(1)	0.0465(5)
H17A	-0.2423(14)	-1.566(3)	-0.3399(6)	0.040(6)
H17B	-0.2670(16)	-1.514(3)	-0.3039(7)	0.049(6)
H17C	-0.1732(19)	-1.437(3)	-0.3201(6)	0.062(7)
C18	-0.2893(2)	-1.1649(4)	-0.3050(1)	0.0512(6)
H18A	-0.327(2)	-1.214(3)	-0.2830(8)	0.063(8)
H18B	-0.230(2)	-1.127(4)	-0.2989(7)	0.069(8)
H18C	-0.322(2)	-1.055(4)	-0.3109(8)	0.074(9)
C19	-0.1195(2)	-1.4993(3)	-0.4548(1)	0.0413(5)
H19A	-0.058(2)	-1.475(3)	-0.4464(7)	0.066(8)
H19B	-0.1487(15)	-1.582(3)	-0.4391(6)	0.047(6)
H19C	-0.1148(15)	-1.557(3)	-0.4776(7)	0.053(7)
C20	-0.1311(2)	-1.2114(4)	-0.4868(1)	0.0430(6)
H20A	-0.1636(18)	-1.104(4)	-0.4902(7)	0.062(8)
H20B	-0.074(2)	-1.180(3)	-0.4814(7)	0.054(7)
H20C	-0.135(3)	-1.286(4)	-0.5100(10)	0.086(11)
C21	-0.2861(2)	-1.7823(3)	-0.5005(1)	0.0393(6)
H21A	-0.2226(18)	-1.769(3)	-0.4887(7)	0.038(7)
H21B	-0.2874(16)	-1.734(3)	-0.5258(7)	0.043(7)
H21C	-0.3021(15)	-1.912(3)	-0.5009(6)	0.050(6)
C22	-0.4472(2)	-1.7022(3)	-0.4973(1)	0.0374(5)
H22A	-0.4587(17)	-1.831(3)	-0.5014(6)	0.043(7)
H22B	-0.4960(15)	-1.642(3)	-0.4858(5)	0.033(6)
H22C	-0.4466(15)	-1.658(3)	-0.5210(6)	0.041(6)
C23	-0.4502(3)	-1.7431(3)	-0.3202(1)	0.0472(8)
H23A	-0.4702(18)	-1.866(4)	-0.3211(7)	0.058(7)
H23B	-0.394(3)	-1.736(4)	-0.3139(9)	0.055(11)
H23C	-0.4840(19)	-1.683(4)	-0.3031(8)	0.061(7)
C24	-0.5684(2)	-1.6729(3)	-0.3632(1)	0.0447(6)
H24A	-0.5824(19)	-1.805(3)	-0.3672(7)	0.050(8)
H24B	-0.6046(18)	-1.626(3)	-0.3440(7)	0.062(7)
H24C	-0.5862(16)	-1.608(3)	-0.3854(7)	0.054(7)
C25	-0.4146(1)	-0.9847(2)	-0.5879(1)	0.0263(4)
C26	-0.3916(1)	-0.8018(3)	-0.5890(1)	0.0275(4)
C27	-0.4789(1)	-0.8645(2)	-0.5957(1)	0.0272(4)

C28	-0.5719(1)	-0.8244(3)	-0.6068(1)	0.0310(4)
C29	-0.5652(2)	-0.7238(3)	-0.6407(1)	0.0344(6)
H29A	-0.5331(13)	-0.611(3)	-0.6376(5)	0.031(5)
H29B	-0.6290(17)	-0.691(3)	-0.6488(6)	0.039(6)
C30	-0.5184(2)	-0.8263(3)	-0.6691(1)	0.0373(5)
H30A	-0.4590(17)	-0.865(3)	-0.6599(6)	0.046(6)
H30B	-0.5538(18)	-0.937(4)	-0.6732(7)	0.068(8)
C31	-0.5087(2)	-0.7200(3)	-0.7018(1)	0.0336(5)
H31A	-0.4758(14)	-0.610(3)	-0.6972(5)	0.037(6)
H31B	-0.5679(16)	-0.686(3)	-0.7107(6)	0.040(6)
C32	-0.4635(1)	-0.8175(3)	-0.7315(1)	0.0324(4)
C33	-0.3688(1)	-0.8579(2)	-0.7216(1)	0.0271(4)
C34	-0.2823(1)	-0.7969(3)	-0.7128(1)	0.0316(5)
C35	-0.3055(1)	-0.9808(2)	-0.7139(1)	0.0280(4)
C36	-0.2807(1)	-1.1743(2)	-0.7098(1)	0.0311(4)
C37	-0.3374(2)	-1.2584(2)	-0.6811(1)	0.0309(6)
H37A	-0.3195(13)	-1.381(3)	-0.6801(5)	0.032(5)
H37B	-0.4051(18)	-1.242(3)	-0.6883(6)	0.028(6)
C38	-0.3251(2)	-1.1802(2)	-0.6458(1)	0.0291(4)
H38A	-0.2646(17)	-1.206(3)	-0.6378(6)	0.039(6)
H38B	-0.3352(14)	-1.046(3)	-0.6466(6)	0.040(6)
C39	-0.3906(2)	-1.2609(3)	-0.6207(1)	0.0326(6)
H39A	-0.454(2)	-1.256(3)	-0.6295(7)	0.036(7)
H39B	-0.3784(14)	-1.393(3)	-0.6180(5)	0.036(5)
C40	-0.3909(1)	-1.1795(2)	-0.5845(1)	0.0289(4)
C41	-0.6275(2)	-0.9952(3)	-0.6103(1)	0.0426(5)
H41A	-0.6868(17)	-0.962(3)	-0.6192(6)	0.045(6)
H41B	-0.6320(18)	-1.055(4)	-0.5885(8)	0.069(8)
H41C	-0.5976(15)	-1.083(3)	-0.6261(6)	0.045(6)
C42	-0.6147(2)	-0.7021(4)	-0.5799(1)	0.0442(6)
H42A	-0.6159(18)	-0.761(3)	-0.5572(7)	0.040(7)
H42B	-0.6769(19)	-0.682(3)	-0.5864(6)	0.049(6)
H42C	-0.5817(15)	-0.586(3)	-0.5791(5)	0.040(6)
C43	-0.5131(2)	-0.9893(3)	-0.7408(1)	0.0417(5)
H43A	-0.5067(14)	-1.085(3)	-0.7235(6)	0.044(6)
H43B	-0.4864(18)	-1.043(3)	-0.7622(7)	0.069(8)
H43C	-0.5755(16)	-0.971(3)	-0.7440(5)	0.036(5)
C44	-0.4610(2)	-0.6927(4)	-0.7627(1)	0.0470(6)
H44A	-0.5263(17)	-0.658(3)	-0.7676(6)	0.053(6)
H44B	-0.4290(16)	-0.573(3)	-0.7565(6)	0.051(6)
H44C	-0.440(2)	-0.762(3)	-0.7835(8)	0.061(8)
C45	-0.3014(3)	-1.2738(3)	-0.7430(1)	0.0498(8)
H45A	-0.367(3)	-1.260(3)	-0.7486(9)	0.061(10)
H45B	-0.2840(18)	-1.399(4)	-0.7386(7)	0.069(8)
H45C	-0.2640(19)	-1.223(4)	-0.7627(8)	0.064(8)
C46	-0.1818(2)	-1.1893(3)	-0.7020(1)	0.0447(6)
H46A	-0.1704(17)	-1.319(3)	-0.6984(6)	0.039(7)
H46B	-0.1465(16)	-1.146(3)	-0.7201(6)	0.045(6)
H46C	-0.1654(15)	-1.123(3)	-0.6807(6)	0.044(6)
C47	-0.4617(2)	-1.2744(3)	-0.5631(1)	0.0415(6)
H47A	-0.4758(17)	-1.221(3)	-0.5448(8)	0.044(7)
H47B	-0.4436(16)	-1.402(4)	-0.5599(6)	0.054(7)
H47C	-0.515(2)	-1.268(3)	-0.5734(8)	0.044(8)
C48	-0.3003(2)	-1.2001(3)	-0.5675(1)	0.0361(5)
H48A	-0.2830(16)	-1.325(3)	-0.5687(6)	0.036(6)
H48B	-0.3036(14)	-1.152(3)	-0.5446(6)	0.037(6)
H48C	-0.2545(16)	-1.134(3)	-0.5810(7)	0.048(6)

Table 3: Hydrogen coordinates and isotropic displacement parameters ($\text{\AA}^2$) for **6a**.

Atom	x	y	z	U_{eq}
------	---	---	---	-----------------

H5A	-0.2690(16)	-1.099(3)	-0.3718(6)	0.052(7)
H5B	-0.1804(17)	-1.168(3)	-0.3559(6)	0.045(6)
H6A	-0.2847(17)	-1.353(3)	-0.4046(6)	0.045(6)
H6B	-0.1976(16)	-1.439(3)	-0.3908(6)	0.054(7)
H7A	-0.2094(14)	-1.110(3)	-0.4310(5)	0.037(5)
H7B	-0.1232(17)	-1.195(3)	-0.4197(6)	0.036(6)
H13A	-0.3753(16)	-1.885(3)	-0.4453(6)	0.046(6)
H13B	-0.302(2)	-1.747(3)	-0.4334(7)	0.028(6)
H14A	-0.4850(17)	-1.698(3)	-0.4265(6)	0.033(5)
H14B	-0.4175(12)	-1.547(3)	-0.4179(5)	0.024(5)
H15A	-0.4239(14)	-1.868(3)	-0.3815(5)	0.037(6)
H15B	-0.353(2)	-1.730(3)	-0.3758(7)	0.037(7)
H17A	-0.2423(14)	-1.566(3)	-0.3399(6)	0.040(6)
H17B	-0.2670(16)	-1.514(3)	-0.3039(7)	0.049(6)
H17C	-0.1732(19)	-1.437(3)	-0.3201(6)	0.062(7)
H18A	-0.327(2)	-1.214(3)	-0.2830(8)	0.063(8)
H18B	-0.230(2)	-1.127(4)	-0.2989(7)	0.069(8)
H18C	-0.322(2)	-1.055(4)	-0.3109(8)	0.074(9)
H19A	-0.058(2)	-1.475(3)	-0.4464(7)	0.066(8)
H19B	-0.1487(15)	-1.582(3)	-0.4391(6)	0.047(6)
H19C	-0.1148(15)	-1.557(3)	-0.4776(7)	0.053(7)
H20A	-0.1636(18)	-1.104(4)	-0.4902(7)	0.062(8)
H20B	-0.074(2)	-1.180(3)	-0.4814(7)	0.054(7)
H20C	-0.135(3)	-1.286(4)	-0.5100(10)	0.086(11)
H21A	-0.2226(18)	-1.769(3)	-0.4887(7)	0.038(7)
H21B	-0.2874(16)	-1.734(3)	-0.5258(7)	0.043(7)
H21C	-0.3021(15)	-1.912(3)	-0.5009(6)	0.050(6)
H22A	-0.4587(17)	-1.831(3)	-0.5014(6)	0.043(7)
H22B	-0.4960(15)	-1.642(3)	-0.4858(5)	0.033(6)
H22C	-0.4466(15)	-1.658(3)	-0.5210(6)	0.041(6)
H23A	-0.4702(18)	-1.866(4)	-0.3211(7)	0.058(7)
H23B	-0.394(3)	-1.736(4)	-0.3139(9)	0.055(11)
H23C	-0.4840(19)	-1.683(4)	-0.3031(8)	0.061(7)
H24A	-0.5824(19)	-1.805(3)	-0.3672(7)	0.050(8)
H24B	-0.6046(18)	-1.626(3)	-0.3440(7)	0.062(7)
H24C	-0.5862(16)	-1.608(3)	-0.3854(7)	0.054(7)
H29A	-0.5331(13)	-0.611(3)	-0.6376(5)	0.031(5)
H29B	-0.6290(17)	-0.691(3)	-0.6488(6)	0.039(6)
H30A	-0.4590(17)	-0.865(3)	-0.6599(6)	0.046(6)
H30B	-0.5538(18)	-0.937(4)	-0.6732(7)	0.068(8)
H31A	-0.4758(14)	-0.610(3)	-0.6972(5)	0.037(6)
H31B	-0.5679(16)	-0.686(3)	-0.7107(6)	0.040(6)
H37A	-0.3195(13)	-1.381(3)	-0.6801(5)	0.032(5)
H37B	-0.4051(18)	-1.242(3)	-0.6883(6)	0.028(6)
H38A	-0.2646(17)	-1.206(3)	-0.6378(6)	0.039(6)
H38B	-0.3352(14)	-1.046(3)	-0.6466(6)	0.040(6)
H39A	-0.454(2)	-1.256(3)	-0.6295(7)	0.036(7)
H39B	-0.3784(14)	-1.393(3)	-0.6180(5)	0.036(5)
H41A	-0.6868(17)	-0.962(3)	-0.6192(6)	0.045(6)
H41B	-0.6320(18)	-1.055(4)	-0.5885(8)	0.069(8)
H41C	-0.5976(15)	-1.083(3)	-0.6261(6)	0.045(6)
H42A	-0.6159(18)	-0.761(3)	-0.5572(7)	0.040(7)
H42B	-0.6769(19)	-0.682(3)	-0.5864(6)	0.049(6)
H42C	-0.5817(15)	-0.586(3)	-0.5791(5)	0.040(6)
H43A	-0.5067(14)	-1.085(3)	-0.7235(6)	0.044(6)
H43B	-0.4864(18)	-1.043(3)	-0.7622(7)	0.069(8)
H43C	-0.5755(16)	-0.971(3)	-0.7440(5)	0.036(5)
H44A	-0.5263(17)	-0.658(3)	-0.7676(6)	0.053(6)
H44B	-0.4290(16)	-0.573(3)	-0.7565(6)	0.051(6)
H44C	-0.440(2)	-0.762(3)	-0.7835(8)	0.061(8)
H45A	-0.367(3)	-1.260(3)	-0.7486(9)	0.061(10)
H45B	-0.2840(18)	-1.399(4)	-0.7386(7)	0.069(8)
H45C	-0.2640(19)	-1.223(4)	-0.7627(8)	0.064(8)
H46A	-0.1704(17)	-1.319(3)	-0.6984(6)	0.039(7)

H46B	-0.1465(16)	-1.146(3)	-0.7201(6)	0.045(6)
H46C	-0.1654(15)	-1.123(3)	-0.6807(6)	0.044(6)
H47A	-0.4758(17)	-1.221(3)	-0.5448(8)	0.044(7)
H47B	-0.4436(16)	-1.402(4)	-0.5599(6)	0.054(7)
H47C	-0.515(2)	-1.268(3)	-0.5734(8)	0.044(8)
H48A	-0.2830(16)	-1.325(3)	-0.5687(6)	0.036(6)
H48B	-0.3036(14)	-1.152(3)	-0.5446(6)	0.037(6)
H48C	-0.2545(16)	-1.134(3)	-0.5810(7)	0.048(6)

Table 4: Anisotropic displacement parameters ($\text{\AA}^2$) for **6a**. The anisotropic displacement factor exponent takes the form: $-2 \pi^2 (h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12})$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0309(8)	0.0265(7)	0.0992(13)	0.0045(8)	-0.0126(9)	0.0054(6)
O2	0.0298(8)	0.0313(8)	0.0717(11)	-0.0062(8)	-0.0086(8)	0.0067(6)
O3	0.0314(9)	0.0291(7)	0.0766(12)	0.0083(8)	-0.0096(8)	-0.0062(7)
O4	0.0313(9)	0.0278(7)	0.0956(13)	0.0046(7)	-0.0140(8)	-0.0043(6)
C1	0.0302(10)	0.0264(8)	0.0251(8)	-0.0011(7)	0.0052(7)	-0.0016(8)
C2	0.0238(11)	0.0291(9)	0.0373(11)	0.0002(9)	0.0003(9)	-0.0014(8)
C3	0.0306(10)	0.0272(9)	0.0268(9)	-0.0021(7)	0.0016(7)	0.0035(8)
C4	0.0265(11)	0.0412(11)	0.0363(11)	-0.0047(9)	-0.0043(9)	0.0057(9)
C5	0.0237(12)	0.0342(10)	0.0438(13)	-0.0029(10)	-0.0044(10)	-0.0028(10)
C6	0.0366(13)	0.0363(12)	0.0421(12)	-0.0024(9)	-0.0008(10)	-0.0066(10)
C7	0.0217(13)	0.0308(11)	0.0450(14)	-0.0015(9)	-0.0032(10)	-0.0032(9)
C8	0.0197(10)	0.0332(11)	0.0379(10)	0.0025(9)	0.0019(8)	0.0003(8)
C9	0.0242(9)	0.0246(8)	0.0265(9)	0.0032(7)	0.0035(7)	0.0015(7)
C10	0.0281(11)	0.0260(10)	0.0306(10)	-0.0011(9)	0.0000(9)	-0.0016(8)
C11	0.0263(9)	0.0269(9)	0.0242(8)	0.0011(7)	0.0029(7)	0.0006(7)
C12	0.0327(11)	0.0227(10)	0.0319(10)	-0.0041(8)	0.0041(8)	-0.0040(8)
C13	0.0343(16)	0.0184(11)	0.0311(13)	-0.0010(7)	0.0028(11)	-0.0015(8)
C14	0.0317(11)	0.0221(11)	0.0363(11)	-0.0010(8)	0.0032(8)	-0.0014(8)
C15	0.0394(17)	0.0192(11)	0.0304(13)	0.0026(7)	0.0013(12)	-0.0003(8)
C16	0.0427(11)	0.0212(8)	0.0356(10)	0.0026(8)	0.0095(9)	-0.0005(8)
C17	0.0376(13)	0.0500(14)	0.0519(14)	0.0078(12)	-0.0081(10)	0.0108(10)
C18	0.0380(14)	0.0683(18)	0.0475(14)	-0.0216(13)	-0.0090(11)	0.0034(13)
C19	0.0264(11)	0.0413(13)	0.0563(14)	0.0014(11)	0.0016(10)	0.0085(9)
C20	0.0271(14)	0.0523(14)	0.0497(15)	0.0122(13)	0.0063(11)	-0.0055(12)
C21	0.0456(16)	0.0327(11)	0.0395(14)	-0.0088(10)	0.0106(12)	0.0028(11)
C22	0.0406(14)	0.0341(12)	0.0375(14)	-0.0032(11)	0.0004(10)	-0.0077(11)
C23	0.078(3)	0.0280(13)	0.0356(15)	0.0051(9)	0.0120(17)	0.0027(12)
C24	0.0404(14)	0.0302(12)	0.0636(16)	-0.0014(11)	0.0149(12)	-0.0076(9)
C25	0.0281(10)	0.0264(9)	0.0245(8)	-0.0012(7)	0.0025(7)	0.0004(7)
C26	0.0227(11)	0.0264(9)	0.0335(10)	0.0026(9)	0.0002(8)	-0.0004(8)
C27	0.0263(9)	0.0277(9)	0.0277(9)	-0.0025(7)	0.0040(7)	0.0006(8)
C28	0.0210(10)	0.0315(10)	0.0405(10)	-0.0064(9)	0.0020(8)	-0.0002(8)
C29	0.0233(13)	0.0324(11)	0.0476(15)	-0.0038(10)	-0.0051(11)	0.0047(9)
C30	0.0375(13)	0.0366(12)	0.0377(12)	0.0013(9)	-0.0026(9)	0.0064(10)
C31	0.0215(12)	0.0325(11)	0.0467(14)	0.0051(10)	-0.0054(10)	0.0004(9)
C32	0.0264(11)	0.0346(11)	0.0362(11)	0.0058(9)	-0.0073(8)	-0.0040(9)
C33	0.0286(10)	0.0245(8)	0.0283(9)	0.0010(7)	0.0017(7)	-0.0021(8)
C34	0.0270(12)	0.0251(9)	0.0428(12)	0.0038(9)	-0.0020(10)	0.0007(9)
C35	0.0301(10)	0.0249(9)	0.0291(9)	0.0014(7)	0.0049(7)	-0.0002(7)
C36	0.0407(11)	0.0177(8)	0.0350(10)	0.0002(7)	0.0055(9)	0.0038(7)
C37	0.0378(16)	0.0179(11)	0.0369(14)	0.0003(7)	0.0039(12)	0.0000(8)
C38	0.0351(12)	0.0218(10)	0.0304(10)	-0.0011(8)	-0.0004(8)	0.0015(8)
C39	0.0421(18)	0.0205(11)	0.0353(14)	0.0002(8)	0.0001(12)	-0.0006(8)
C40	0.0364(11)	0.0206(9)	0.0297(10)	0.0000(8)	0.0013(8)	-0.0007(8)
C41	0.0285(11)	0.0419(13)	0.0573(14)	-0.0046(12)	0.0023(10)	-0.0063(9)
C42	0.0246(13)	0.0525(14)	0.0555(17)	-0.0146(14)	0.0049(11)	0.0040(12)
C43	0.0396(13)	0.0462(13)	0.0392(12)	-0.0035(10)	-0.0075(10)	-0.0117(10)

C44	0.0383(13)	0.0554(15)	0.0474(14)	0.0176(12)	-0.0059(11)	-0.0039(11)
C45	0.088(3)	0.0279(13)	0.0340(14)	-0.0001(9)	0.0075(16)	0.0048(12)
C46	0.0416(14)	0.0328(12)	0.0597(16)	0.0073(11)	0.0168(12)	0.0104(10)
C47	0.0496(18)	0.0327(12)	0.0422(15)	0.0065(11)	0.0084(14)	-0.0033(11)
C48	0.0444(14)	0.0323(11)	0.0317(13)	0.0011(10)	-0.0045(10)	0.0091(11)

Table 5: Bond lengths (Å) and angles (deg) for **6a**.

O1-C2	1.224(3)	C40-C48	1.526(3)
O2-C10	1.222(2)	C40-C47	1.533(3)
O3-C26	1.219(3)	C3-C1-C2	61.11(14)
O4-C34	1.208(3)	C3-C1-C16	146.72(18)
C1-C3	1.362(3)	C2-C1-C16	152.11(18)
C1-C2	1.416(3)	O1-C2-C3	151.9(2)
C1-C16	1.505(2)	O1-C2-C1	150.5(2)
C2-C3	1.413(3)	C3-C2-C1	57.56(13)
C3-C4	1.497(3)	C1-C3-C2	61.33(14)
C4-C17	1.527(3)	C1-C3-C4	149.38(17)
C4-C18	1.543(3)	C2-C3-C4	149.27(17)
C4-C5	1.547(3)	C3-C4-C17	110.81(18)
C5-C6	1.515(3)	C3-C4-C18	107.77(18)
C6-C7	1.524(3)	C17-C4-C18	109.8(2)
C7-C8	1.542(3)	C3-C4-C5	109.01(17)
C8-C9	1.504(2)	C17-C4-C5	111.01(18)
C8-C19	1.529(3)	C18-C4-C5	108.3(2)
C8-C20	1.542(3)	C6-C5-C4	115.38(18)
C9-C11	1.357(3)	C5-C6-C7	112.52(18)
C9-C10	1.410(3)	C6-C7-C8	114.77(17)
C10-C11	1.415(3)	C9-C8-C19	111.63(16)
C11-C12	1.502(2)	C9-C8-C20	107.73(17)
C12-C21	1.530(3)	C19-C8-C20	109.15(18)
C12-C22	1.537(3)	C9-C8-C7	106.62(16)
C12-C13	1.543(3)	C19-C8-C7	111.37(18)
C13-C14	1.531(3)	C20-C8-C7	110.26(18)
C14-C15	1.531(3)	C11-C9-C10	61.47(13)
C15-C16	1.543(3)	C11-C9-C8	149.34(17)
C16-C24	1.530(3)	C10-C9-C8	148.72(17)
C16-C23	1.544(4)	O2-C10-C9	151.0(2)
C25-C27	1.356(3)	O2-C10-C11	151.6(2)
C25-C26	1.413(3)	C9-C10-C11	57.43(13)
C25-C40	1.506(2)	C9-C11-C10	61.10(13)
C26-C27	1.419(3)	C9-C11-C12	146.56(17)
C27-C28	1.493(3)	C10-C11-C12	151.82(18)
C28-C41	1.534(3)	C11-C12-C21	109.71(17)
C28-C29	1.537(3)	C11-C12-C22	109.51(17)
C28-C42	1.538(3)	C21-C12-C22	109.03(19)
C29-C30	1.525(3)	C11-C12-C13	107.61(15)
C30-C31	1.517(3)	C21-C12-C13	109.14(18)
C31-C32	1.537(3)	C22-C12-C13	111.81(19)
C32-C33	1.505(3)	C14-C13-C12	116.23(18)
C32-C43	1.531(3)	C13-C14-C15	111.35(18)
C32-C44	1.543(3)	C14-C15-C16	115.30(19)
C33-C35	1.357(3)	C1-C16-C24	110.02(16)
C33-C34	1.422(3)	C1-C16-C15	109.46(16)
C34-C35	1.421(3)	C24-C16-C15	111.0(2)
C35-C36	1.504(2)	C1-C16-C23	108.28(18)
C36-C46	1.522(3)	C24-C16-C23	109.3(2)
C36-C45	1.535(4)	C15-C16-C23	108.66(19)
C36-C37	1.550(3)	C27-C25-C26	61.63(14)
C37-C38	1.516(3)	C27-C25-C40	146.17(18)
C38-C39	1.521(3)	C26-C25-C40	151.91(18)
C39-C40	1.547(3)	O3-C26-C25	151.6(2)

O3-C26-C27	151.2(2)
C25-C26-C27	57.22(13)
C25-C27-C26	61.15(13)
C25-C27-C28	150.00(18)
C26-C27-C28	148.58(18)
C27-C28-C41	111.66(17)
C27-C28-C29	106.91(16)
C41-C28-C29	111.35(18)
C27-C28-C42	108.15(17)
C41-C28-C42	109.30(18)
C29-C28-C42	109.38(19)
C30-C29-C28	114.78(17)
C31-C30-C29	113.50(18)
C30-C31-C32	115.91(17)
C33-C32-C43	110.66(17)
C33-C32-C31	108.47(17)
C43-C32-C31	111.42(18)
C33-C32-C44	107.68(17)
C43-C32-C44	109.31(19)
C31-C32-C44	109.21(18)
C35-C33-C34	61.42(14)
C35-C33-C32	148.89(17)
C34-C33-C32	149.67(17)
O4-C34-C35	151.9(2)
O4-C34-C33	151.1(2)
C35-C34-C33	57.04(13)
C33-C35-C34	61.54(14)
C33-C35-C36	148.22(18)
C34-C35-C36	150.24(18)
C35-C36-C46	109.58(17)
C35-C36-C45	109.03(18)
C46-C36-C45	109.6(2)
C35-C36-C37	109.43(16)
C46-C36-C37	111.04(19)
C45-C36-C37	108.2(2)
C38-C37-C36	116.32(18)
C37-C38-C39	111.21(19)
C38-C39-C40	116.26(19)
C25-C40-C48	110.32(16)
C25-C40-C47	109.40(17)
C48-C40-C47	109.3(2)
C25-C40-C39	107.57(15)
C48-C40-C39	111.21(19)
C47-C40-C39	108.97(19)

2.3 Crystallographic Data for 6 β

Table 1: Crystal data and structure refinement for 6 β .

Empirical formula	$C_{24}H_{36}O_2$
Formula weight	356.53
Temperature	200(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1/c$
Z	8
Unit cell dimensions	$a = 40.0257(6)$ Å $\alpha = 90$ deg. $b = 7.4849(2)$ Å $\beta = 100.68$ deg. $c = 15.0438(3)$ Å $\gamma = 90$ deg.
Volume	$4428.90(16)$ Å ³
Density (calculated)	1.07 Mg/m ³
Absorption coefficient	0.07 mm ⁻¹
Crystal size	$.32 \times .32 \times .14$ mm ³
Theta range for data collection	1.4 to 25.6 deg.
Index ranges	$-46 \leq h \leq 48$, $-8 \leq k \leq 9$, $-17 \leq l \leq 17$
Reflections collected	33435
Independent reflections	7678 ($R(\text{int}) = 0.0757$)
Observed reflections	5090 ($I > 2\sigma(I)$)
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7678 / 0 / 758
Goodness-of-fit on F^2	1.08
Final R indices ($I > 2\sigma(I)$)	$R1 = 0.064$, $wR2 = 0.156$
Largest diff. peak and hole	0.32 and -0.27 eÅ ⁻³

Table 2: Atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$) for as2. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
O1	0.1759(1)	0.8231(3)	0.1931(1)	0.0529(7)
O2	0.0552(1)	0.8190(3)	0.2414(1)	0.0457(6)
C1	0.0635(1)	0.6358(4)	0.3872(2)	0.0266(6)
C2	0.0747(1)	0.6768(4)	0.4860(2)	0.0316(7)
C3	0.1086(1)	0.7758(5)	0.4961(2)	0.0337(7)
H3A	0.1161(8)	0.811(4)	0.561(2)	0.038(9)
H3B	0.1064(7)	0.883(4)	0.462(2)	0.033(8)
C4	0.1371(1)	0.6735(5)	0.4632(2)	0.0382(8)
H4A	0.1414(8)	0.557(5)	0.499(2)	0.049(10)
H4B	0.1296(11)	0.634(5)	0.400(3)	0.076(13)
C5	0.1697(1)	0.7806(4)	0.4698(2)	0.0339(7)
H5A	0.1651(9)	0.889(5)	0.433(2)	0.057(10)
H5B	0.1794(8)	0.812(4)	0.532(2)	0.046(9)
C6	0.1993(1)	0.6826(4)	0.4387(2)	0.0306(7)
C7	0.1894(1)	0.6425(4)	0.3393(2)	0.0286(7)
C8	0.1805(1)	0.7036(4)	0.2486(2)	0.0313(7)
C9	0.1818(1)	0.5197(4)	0.2724(2)	0.0276(7)
C10	0.1778(1)	0.3264(4)	0.2454(2)	0.0330(7)
C11	0.1490(1)	0.2423(4)	0.2885(2)	0.0304(7)
H11A	0.1465(9)	0.116(5)	0.269(2)	0.050(10)
H11B	0.1554(7)	0.250(4)	0.353(2)	0.036(8)
C12	0.1136(1)	0.3198(4)	0.2582(2)	0.0305(7)
H12A	0.1048(8)	0.297(4)	0.193(2)	0.048(10)
H12B	0.1153(8)	0.444(5)	0.268(2)	0.036(8)
C13	0.0884(1)	0.2398(4)	0.3112(2)	0.0304(7)
H13A	0.0848(8)	0.115(5)	0.295(2)	0.039(9)
H13B	0.0973(8)	0.246(4)	0.375(2)	0.036(8)
C14	0.0523(1)	0.3210(4)	0.2940(2)	0.0301(7)
C15	0.0555(1)	0.5153(4)	0.3192(2)	0.0270(7)
C16	0.0571(1)	0.6991(4)	0.2964(2)	0.0290(7)
C17	0.0780(1)	0.5054(5)	0.5430(2)	0.0413(8)
H17A	0.0870(8)	0.531(4)	0.607(2)	0.039(9)
H17B	0.0933(8)	0.418(4)	0.517(2)	0.041(9)
H17C	0.0534(10)	0.450(5)	0.535(2)	0.056(10)
C18	0.0475(1)	0.7982(6)	0.5149(3)	0.0458(9)
H18A	0.0524(7)	0.823(4)	0.578(2)	0.032(8)
H18B	0.0253(9)	0.730(5)	0.500(2)	0.049(10)
H18C	0.0436(9)	0.907(5)	0.477(2)	0.055(11)
C19	0.2084(1)	0.5110(5)	0.4927(3)	0.0436(9)
H19A	0.2298(12)	0.471(6)	0.474(3)	0.082(14)
H19B	0.2113(8)	0.539(4)	0.559(2)	0.048(9)
H19C	0.1914(9)	0.418(5)	0.479(2)	0.044(9)
C20	0.2306(1)	0.8071(6)	0.4516(3)	0.0468(9)
H20A	0.2360(10)	0.836(5)	0.517(3)	0.072(12)
H20B	0.2218(11)	0.926(6)	0.412(3)	0.086(14)
H20C	0.2528(12)	0.723(7)	0.436(3)	0.103(16)
C21	0.2108(1)	0.2267(5)	0.2820(4)	0.0490(10)
H21A	0.2050(9)	0.091(5)	0.265(2)	0.062(11)
H21B	0.2153(10)	0.237(6)	0.347(3)	0.064(13)
H21C	0.2293(12)	0.274(6)	0.263(3)	0.085(14)
C22	0.1694(1)	0.3119(5)	0.1419(3)	0.0470(9)
H22A	0.1677(7)	0.181(4)	0.1317(19)	0.030(8)
H22B	0.1901(9)	0.353(4)	0.116(2)	0.048(9)
H22C	0.1475(9)	0.390(5)	0.116(2)	0.050(10)
C23	0.0308(1)	0.2267(5)	0.3546(3)	0.0412(8)
H23A	0.0416(8)	0.239(4)	0.419(2)	0.040(9)
H23B	0.0290(11)	0.105(6)	0.336(3)	0.077(13)
H23C	0.0101(10)	0.276(5)	0.345(2)	0.047(10)

C24	0.0350(1)	0.3012(5)	0.1946(2)	0.0377(8)
H24A	0.0329(7)	0.175(4)	0.1802(19)	0.028(8)
H24B	0.0461(8)	0.362(4)	0.149(2)	0.040(9)
H24C	0.0122(9)	0.350(4)	0.185(2)	0.045(9)
O51	0.4453(1)	0.8135(3)	0.1867(1)	0.0445(6)
O52	0.3256(1)	0.8438(3)	0.0173(2)	0.0507(7)
C51	0.3130(1)	0.6619(4)	0.1511(2)	0.0271(7)
C52	0.3026(1)	0.7016(4)	0.2400(2)	0.0351(7)
C53	0.3329(1)	0.7912(4)	0.3027(2)	0.0335(7)
H53A	0.3388(8)	0.912(4)	0.277(2)	0.040(9)
H53B	0.3263(7)	0.830(4)	0.357(2)	0.033(8)
C54	0.3647(1)	0.6785(5)	0.3277(2)	0.0380(8)
H54A	0.3737(7)	0.630(4)	0.271(2)	0.035(8)
H54B	0.3589(10)	0.562(5)	0.359(3)	0.067(12)
C55	0.3932(1)	0.7753(4)	0.3903(2)	0.0325(7)
H55A	0.3982(7)	0.888(4)	0.364(2)	0.032(8)
H55B	0.3867(7)	0.809(4)	0.449(2)	0.036(8)
C56	0.4269(1)	0.6708(4)	0.4127(2)	0.0295(7)
C57	0.4376(1)	0.6312(4)	0.3247(2)	0.0241(6)
C58	0.4436(1)	0.6931(4)	0.2402(2)	0.0265(7)
C59	0.4446(1)	0.5104(4)	0.2639(2)	0.0242(6)
C60	0.4473(1)	0.3156(4)	0.2408(2)	0.0269(7)
C61	0.4109(1)	0.2386(4)	0.2212(2)	0.0277(7)
H61A	0.4129(8)	0.117(5)	0.204(2)	0.042(9)
H61B	0.4007(8)	0.242(5)	0.274(2)	0.042(9)
C62	0.3863(1)	0.3253(4)	0.1430(2)	0.0281(7)
H62A	0.3944(6)	0.309(3)	0.0837(18)	0.016(7)
H62B	0.3867(8)	0.458(5)	0.153(2)	0.041(9)
C63	0.3503(1)	0.2538(4)	0.1373(2)	0.0284(7)
H63A	0.3510(8)	0.134(4)	0.120(2)	0.035(8)
H63B	0.3436(8)	0.269(4)	0.197(2)	0.042(9)
C64	0.3226(1)	0.3454(4)	0.0663(2)	0.0314(7)
C65	0.3201(1)	0.5399(4)	0.0896(2)	0.0270(7)
C66	0.3214(1)	0.7243(4)	0.0685(2)	0.0289(7)
C67	0.2914(1)	0.5317(5)	0.2824(3)	0.0458(9)
H67A	0.2853(8)	0.556(4)	0.345(2)	0.047(9)
H67B	0.2721(10)	0.492(5)	0.239(2)	0.051(10)
H67C	0.3096(10)	0.434(5)	0.290(2)	0.064(11)
C68	0.2730(1)	0.8342(7)	0.2223(3)	0.0533(10)
H68A	0.2665(10)	0.870(5)	0.285(3)	0.070(12)
H68B	0.2793(9)	0.949(6)	0.191(3)	0.065(12)
H68C	0.2528(9)	0.777(5)	0.186(2)	0.054(11)
C69	0.4226(1)	0.4996(5)	0.4652(2)	0.0420(8)
H69A	0.4446(10)	0.430(5)	0.480(3)	0.067(12)
H69B	0.4070(8)	0.408(5)	0.429(2)	0.043(9)
H69C	0.4136(9)	0.530(5)	0.517(3)	0.056(11)
C70	0.4545(1)	0.7873(6)	0.4696(2)	0.0433(9)
H70A	0.4482(8)	0.818(4)	0.527(2)	0.042(9)
H70B	0.4544(12)	0.908(6)	0.437(3)	0.092(15)
H70C	0.4758(11)	0.729(6)	0.479(3)	0.072(13)
C71	0.4682(1)	0.2170(5)	0.3216(2)	0.0378(8)
H71A	0.4556(8)	0.225(4)	0.374(2)	0.041(9)
H71B	0.4705(10)	0.089(6)	0.302(2)	0.068(12)
H71C	0.4900(10)	0.265(5)	0.340(2)	0.055(11)
C72	0.4648(1)	0.2961(5)	0.1586(2)	0.0360(8)
H72A	0.4665(7)	0.178(4)	0.1473(18)	0.023(8)
H72B	0.4514(8)	0.362(4)	0.102(2)	0.034(8)
H72C	0.4887(8)	0.341(4)	0.1748(19)	0.032(8)
C73	0.2881(1)	0.2566(5)	0.0681(3)	0.0463(9)
H73A	0.2818(8)	0.267(5)	0.125(3)	0.045(10)
H73B	0.2891(9)	0.127(5)	0.046(2)	0.053(10)
H73C	0.2695(10)	0.318(4)	0.024(2)	0.050(10)
C74	0.3310(1)	0.3269(5)	-0.0283(2)	0.0422(9)
H74A	0.3330(8)	0.196(5)	-0.038(2)	0.039(9)
H74B	0.3143(9)	0.375(5)	-0.074(2)	0.050(10)

H74C	0.3535(9)	0.398(5)	-0.034(2)	0.051(10)
------	-----------	----------	-----------	-----------

Table 3: Hydrogen coordinates and isotropic displacement parameters ($\text{\AA}^2$) for **6P**.

Atom	x	y	z	U_{eq}
H3A	0.1161(8)	0.811(4)	0.561(2)	0.038(9)
H3B	0.1064(7)	0.883(4)	0.462(2)	0.033(8)
H4A	0.1414(8)	0.557(5)	0.499(2)	0.049(10)
H4B	0.1296(11)	0.634(5)	0.400(3)	0.076(13)
H5A	0.1651(9)	0.889(5)	0.433(2)	0.057(10)
H5B	0.1794(8)	0.812(4)	0.532(2)	0.046(9)
H11A	0.1465(9)	0.116(5)	0.269(2)	0.050(10)
H11B	0.1554(7)	0.250(4)	0.353(2)	0.036(8)
H12A	0.1048(8)	0.297(4)	0.193(2)	0.048(10)
H12B	0.1153(8)	0.444(5)	0.268(2)	0.036(8)
H13A	0.0848(8)	0.115(5)	0.295(2)	0.039(9)
H13B	0.0973(8)	0.246(4)	0.375(2)	0.036(8)
H17A	0.0870(8)	0.531(4)	0.607(2)	0.039(9)
H17B	0.0933(8)	0.418(4)	0.517(2)	0.041(9)
H17C	0.0534(10)	0.450(5)	0.535(2)	0.056(10)
H18A	0.0524(7)	0.823(4)	0.578(2)	0.032(8)
H18B	0.0253(9)	0.730(5)	0.500(2)	0.049(10)
H18C	0.0436(9)	0.907(5)	0.477(2)	0.055(11)
H19A	0.2298(12)	0.471(6)	0.474(3)	0.082(14)
H19B	0.2113(8)	0.539(4)	0.559(2)	0.048(9)
H19C	0.1914(9)	0.418(5)	0.479(2)	0.044(9)
H20A	0.2360(10)	0.836(5)	0.517(3)	0.072(12)
H20B	0.2218(11)	0.926(6)	0.412(3)	0.086(14)
H20C	0.2528(12)	0.723(7)	0.436(3)	0.103(16)
H21A	0.2050(9)	0.091(5)	0.265(2)	0.062(11)
H21B	0.2153(10)	0.237(6)	0.347(3)	0.064(13)
H21C	0.2293(12)	0.274(6)	0.263(3)	0.085(14)
H22A	0.1677(7)	0.181(4)	0.1317(19)	0.030(8)
H22B	0.1901(9)	0.353(4)	0.116(2)	0.048(9)
H22C	0.1475(9)	0.390(5)	0.116(2)	0.050(10)
H23A	0.0416(8)	0.239(4)	0.419(2)	0.040(9)
H23B	0.0290(11)	0.105(6)	0.336(3)	0.077(13)
H23C	0.0101(10)	0.276(5)	0.345(2)	0.047(10)
H24A	0.0329(7)	0.175(4)	0.1802(19)	0.028(8)
H24B	0.0461(8)	0.362(4)	0.149(2)	0.040(9)
H24C	0.0122(9)	0.350(4)	0.185(2)	0.045(9)
H53A	0.3388(8)	0.912(4)	0.277(2)	0.040(9)
H53B	0.3263(7)	0.830(4)	0.357(2)	0.033(8)
H54A	0.3737(7)	0.630(4)	0.271(2)	0.035(8)
H54B	0.3589(10)	0.562(5)	0.359(3)	0.067(12)
H55A	0.3982(7)	0.888(4)	0.364(2)	0.032(8)
H55B	0.3867(7)	0.809(4)	0.449(2)	0.036(8)
H61A	0.4129(8)	0.117(5)	0.204(2)	0.042(9)
H61B	0.4007(8)	0.242(5)	0.274(2)	0.042(9)
H62A	0.3944(6)	0.309(3)	0.0837(18)	0.016(7)
H62B	0.3867(8)	0.458(5)	0.153(2)	0.041(9)
H63A	0.3510(8)	0.134(4)	0.120(2)	0.035(8)
H63B	0.3436(8)	0.269(4)	0.197(2)	0.042(9)
H67A	0.2853(8)	0.556(4)	0.345(2)	0.047(9)
H67B	0.2721(10)	0.492(5)	0.239(2)	0.051(10)
H67C	0.3096(10)	0.434(5)	0.290(2)	0.064(11)
H68A	0.2665(10)	0.870(5)	0.285(3)	0.070(12)
H68B	0.2793(9)	0.949(6)	0.191(3)	0.065(12)
H68C	0.2528(9)	0.777(5)	0.186(2)	0.054(11)
H69A	0.4446(10)	0.430(5)	0.480(3)	0.067(12)

H69B	0.4070(8)	0.408(5)	0.429(2)	0.043(9)
H69C	0.4136(9)	0.530(5)	0.517(3)	0.056(11)
H70A	0.4482(8)	0.818(4)	0.527(2)	0.042(9)
H70B	0.4544(12)	0.908(6)	0.437(3)	0.092(15)
H70C	0.4758(11)	0.729(6)	0.479(3)	0.072(13)
H71A	0.4556(8)	0.225(4)	0.374(2)	0.041(9)
H71B	0.4705(10)	0.089(6)	0.302(2)	0.068(12)
H71C	0.4900(10)	0.265(5)	0.340(2)	0.055(11)
H72A	0.4665(7)	0.178(4)	0.1473(18)	0.023(8)
H72B	0.4514(8)	0.362(4)	0.102(2)	0.034(8)
H72C	0.4887(8)	0.341(4)	0.1748(19)	0.032(8)
H73A	0.2818(8)	0.267(5)	0.125(3)	0.045(10)
H73B	0.2891(9)	0.127(5)	0.046(2)	0.053(10)
H73C	0.2695(10)	0.318(4)	0.024(2)	0.050(10)
H74A	0.3330(8)	0.196(5)	-0.038(2)	0.039(9)
H74B	0.3143(9)	0.375(5)	-0.074(2)	0.050(10)
H74C	0.3535(9)	0.398(5)	-0.034(2)	0.051(10)

Table 4: Anisotropic displacement parameters ($\text{\AA}^2$) for **6 β** . The anisotropic displacement factor exponent takes the form: $-2\pi^2(h^2 a^{*2} U_{11} + \dots + 2hk a^* b^* U_{12})$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.097(2)	0.0286(13)	0.0289(12)	0.0034(10)	0.0019(12)	-0.0050(12)
O2	0.0737(17)	0.0317(13)	0.0298(12)	0.0067(10)	0.0047(11)	-0.0074(11)
C1	0.0256(15)	0.0271(16)	0.0286(16)	0.0013(13)	0.0088(12)	0.0051(12)
C2	0.0376(17)	0.0368(18)	0.0207(15)	0.0024(13)	0.0064(12)	0.0060(14)
C3	0.0438(19)	0.0349(19)	0.0215(16)	-0.0039(15)	0.0039(14)	0.0007(15)
C4	0.0364(18)	0.041(2)	0.0364(19)	-0.0085(16)	0.0041(15)	-0.0007(15)
C5	0.0440(19)	0.0348(19)	0.0211(16)	-0.0019(14)	0.0017(14)	-0.0042(15)
C6	0.0287(16)	0.0368(18)	0.0242(15)	0.0019(13)	-0.0005(12)	-0.0044(13)
C7	0.0277(16)	0.0291(16)	0.0294(16)	0.0021(13)	0.0063(12)	-0.0012(13)
C8	0.0410(18)	0.0267(17)	0.0261(16)	0.0002(14)	0.0058(13)	-0.0018(13)
C9	0.0250(15)	0.0256(16)	0.0326(16)	-0.0020(13)	0.0069(12)	-0.0009(12)
C10	0.0359(17)	0.0193(15)	0.0455(19)	-0.0008(13)	0.0126(14)	0.0003(13)
C11	0.0310(17)	0.0215(16)	0.0385(19)	0.0014(14)	0.0060(14)	0.0003(13)
C12	0.0332(17)	0.0235(18)	0.0352(18)	-0.0024(14)	0.0068(13)	-0.0004(13)
C13	0.0334(17)	0.0199(17)	0.0379(19)	0.0029(14)	0.0070(14)	-0.0006(13)
C14	0.0307(16)	0.0238(16)	0.0358(17)	-0.0017(13)	0.0064(13)	-0.0036(12)
C15	0.0265(15)	0.0272(16)	0.0286(15)	0.0001(13)	0.0085(12)	0.0006(12)
C16	0.0333(16)	0.0280(17)	0.0258(16)	0.0007(14)	0.0055(12)	-0.0004(13)
C17	0.053(2)	0.044(2)	0.0279(18)	0.0069(16)	0.0106(16)	0.0024(18)
C18	0.061(3)	0.051(2)	0.029(2)	-0.0043(18)	0.0163(17)	0.016(2)
C19	0.043(2)	0.045(2)	0.039(2)	0.0128(17)	-0.0002(16)	0.0029(19)
C20	0.045(2)	0.058(3)	0.034(2)	-0.0031(18)	-0.0013(16)	-0.0188(18)
C21	0.033(2)	0.026(2)	0.090(3)	-0.001(2)	0.017(2)	-0.0007(15)
C22	0.065(3)	0.032(2)	0.050(2)	-0.0138(17)	0.028(2)	-0.0067(19)
C23	0.041(2)	0.033(2)	0.053(2)	0.0019(17)	0.0172(18)	-0.0068(17)
C24	0.0337(19)	0.035(2)	0.042(2)	-0.0055(16)	0.0026(15)	-0.0017(16)
O51	0.0712(16)	0.0315(13)	0.0347(12)	0.0063(10)	0.0206(11)	0.0073(11)
O52	0.0924(19)	0.0266(12)	0.0394(13)	0.0032(11)	0.0291(13)	-0.0016(12)
C51	0.0237(15)	0.0285(16)	0.0274(15)	0.0048(13)	0.0008(12)	0.0042(12)
C52	0.0373(18)	0.0409(19)	0.0284(16)	0.0058(14)	0.0093(13)	0.0050(14)
C53	0.0443(19)	0.0351(19)	0.0228(16)	-0.0003(14)	0.0109(14)	0.0074(15)
C54	0.0382(18)	0.038(2)	0.0383(19)	-0.0060(16)	0.0073(15)	0.0033(15)
C55	0.0446(19)	0.0320(18)	0.0231(16)	-0.0023(14)	0.0120(14)	0.0004(15)
C56	0.0381(17)	0.0319(17)	0.0183(14)	-0.0006(12)	0.0049(12)	-0.0040(13)
C57	0.0203(14)	0.0287(16)	0.0218(14)	-0.0002(12)	-0.0001(11)	-0.0030(12)
C58	0.0287(16)	0.0243(16)	0.0258(16)	0.0006(13)	0.0029(12)	0.0023(12)
C59	0.0171(13)	0.0263(16)	0.0269(15)	-0.0008(12)	-0.0018(11)	-0.0038(11)
C60	0.0253(15)	0.0233(16)	0.0305(16)	-0.0021(12)	0.0014(12)	0.0032(12)

C61	0.0305(16)	0.0182(16)	0.0337(17)	-0.0008(13)	0.0037(13)	-0.0011(13)
C62	0.0298(16)	0.0237(17)	0.0296(17)	-0.0016(13)	0.0024(13)	-0.0007(13)
C63	0.0345(17)	0.0177(16)	0.0313(17)	0.0003(13)	0.0017(13)	-0.0018(13)
C64	0.0322(16)	0.0214(15)	0.0364(17)	-0.0015(13)	-0.0045(13)	-0.0017(13)
C65	0.0218(14)	0.0262(16)	0.0298(16)	0.0007(13)	-0.0035(12)	-0.0008(12)
C66	0.0354(17)	0.0270(16)	0.0245(15)	0.0017(14)	0.0059(12)	0.0005(13)
C67	0.043(2)	0.057(2)	0.038(2)	0.0120(18)	0.0121(17)	-0.0088(19)
C68	0.044(2)	0.075(3)	0.042(2)	0.006(2)	0.0115(18)	0.022(2)
C69	0.055(2)	0.042(2)	0.0299(18)	0.0069(16)	0.0106(17)	-0.0011(19)
C70	0.050(2)	0.057(3)	0.0215(18)	-0.0088(17)	0.0030(16)	-0.0105(19)
C71	0.036(2)	0.032(2)	0.041(2)	0.0033(15)	-0.0026(16)	0.0101(16)
C72	0.0341(19)	0.036(2)	0.0390(19)	-0.0101(16)	0.0085(15)	0.0011(15)
C73	0.0333(19)	0.033(2)	0.068(3)	0.0027(19)	-0.0026(19)	-0.0068(16)
C74	0.055(2)	0.031(2)	0.0357(19)	-0.0032(15)	-0.0053(17)	0.0023(17)

Table 5: Bond lengths (Å) and angles (deg) for **6β**.

O1-C8	1.214(3)	C59-C60	1.507(4)
O2-C16	1.213(3)	C60-C71	1.532(4)
C1-C15	1.357(4)	C60-C72	1.537(4)
C1-C16	1.423(4)	C60-C61	1.543(4)
C1-C2	1.502(4)	C61-C62	1.531(4)
C2-C3	1.531(4)	C62-C63	1.523(4)
C2-C17	1.536(4)	C63-C64	1.549(4)
C2-C18	1.539(4)	C64-C65	1.505(4)
C3-C4	1.529(4)	C64-C74	1.528(5)
C4-C5	1.521(4)	C64-C73	1.538(5)
C5-C6	1.538(4)	C65-C66	1.420(4)
C6-C7	1.504(4)	C15-C1-C16	61.5(2)
C6-C19	1.527(4)	C15-C1-C2	150.1(3)
C6-C20	1.544(4)	C16-C1-C2	148.1(3)
C7-C9	1.356(4)	C1-C2-C3	107.0(2)
C7-C8	1.422(4)	C1-C2-C17	111.2(3)
C8-C9	1.421(4)	C3-C2-C17	111.4(3)
C9-C10	1.503(4)	C1-C2-C18	107.7(3)
C10-C21	1.530(5)	C3-C2-C18	110.2(3)
C10-C22	1.534(5)	C17-C2-C18	109.2(3)
C10-C11	1.557(4)	C4-C3-C2	115.2(3)
C11-C12	1.521(4)	C5-C4-C3	113.4(3)
C12-C13	1.519(4)	C4-C5-C6	115.5(3)
C13-C14	1.546(4)	C7-C6-C19	110.7(3)
C14-C15	1.502(4)	C7-C6-C5	108.7(2)
C14-C24	1.534(4)	C19-C6-C5	111.1(3)
C14-C23	1.534(4)	C7-C6-C20	107.7(2)
C15-C16	1.422(4)	C19-C6-C20	109.5(3)
O51-C58	1.219(3)	C5-C6-C20	109.1(3)
O52-C66	1.213(3)	C9-C7-C8	61.5(2)
C51-C65	1.366(4)	C9-C7-C6	148.8(3)
C51-C66	1.424(4)	C8-C7-C6	149.7(3)
C51-C52	1.503(4)	O1-C8-C9	151.7(3)
C52-C67	1.527(5)	O1-C8-C7	151.3(3)
C52-C68	1.529(5)	C9-C8-C7	57.0(2)
C52-C53	1.544(4)	C7-C9-C8	61.5(2)
C53-C54	1.515(4)	C7-C9-C10	148.3(3)
C54-C55	1.523(4)	C8-C9-C10	150.2(3)
C55-C56	1.539(4)	C9-C10-C21	109.3(3)
C56-C57	1.495(4)	C9-C10-C22	109.5(3)
C56-C69	1.530(4)	C21-C10-C22	110.1(3)
C56-C70	1.538(4)	C9-C10-C11	109.1(2)
C57-C59	1.353(4)	C21-C10-C11	108.1(3)
C57-C58	1.414(4)	C22-C10-C11	110.7(3)
C58-C59	1.411(4)	C12-C11-C10	116.2(3)

C13-C12-C11	111.3(3)
C12-C13-C14	116.6(3)
C15-C14-C24	110.0(3)
C15-C14-C23	109.2(3)
C24-C14-C23	109.5(3)
C15-C14-C13	107.6(2)
C24-C14-C13	111.3(3)
C23-C14-C13	109.2(3)
C1-C15-C16	61.6(2)
C1-C15-C14	146.2(3)
C16-C15-C14	151.8(3)
O2-C16-C15	151.7(3)
O2-C16-C1	151.4(3)
C15-C16-C1	56.96(19)
C65-C51-C66	61.2(2)
C65-C51-C52	149.5(3)
C66-C51-C52	149.4(3)
C51-C52-C67	111.0(3)
C51-C52-C68	107.9(3)
C67-C52-C68	109.5(3)
C51-C52-C53	108.2(2)
C67-C52-C53	111.4(3)
C68-C52-C53	108.6(3)
C54-C53-C52	116.0(3)
C53-C54-C55	112.9(3)
C54-C55-C56	114.8(3)
C57-C56-C69	111.6(3)
C57-C56-C70	108.0(3)
C69-C56-C70	109.0(3)
C57-C56-C55	106.8(2)
C69-C56-C55	111.2(3)
C70-C56-C55	110.0(3)
C59-C57-C58	61.29(19)
C59-C57-C56	149.5(3)
C58-C57-C56	148.8(3)
O51-C58-C59	151.7(3)
O51-C58-C57	151.1(3)
C59-C58-C57	57.2(2)
C57-C59-C58	61.5(2)
C57-C59-C60	146.6(3)
C58-C59-C60	151.5(3)
C59-C60-C71	109.6(2)
C59-C60-C72	109.8(2)
C71-C60-C72	109.2(3)
C59-C60-C61	107.5(2)
C71-C60-C61	109.3(3)
C72-C60-C61	111.5(2)
C62-C61-C60	116.0(2)
C63-C62-C61	111.2(2)
C62-C63-C64	115.5(2)
C65-C64-C74	109.9(3)
C65-C64-C73	108.2(3)
C74-C64-C73	109.3(3)
C65-C64-C63	109.8(2)
C74-C64-C63	110.5(3)
C73-C64-C63	109.1(3)
C51-C65-C66	61.4(2)
C51-C65-C64	146.7(3)
C66-C65-C64	151.8(3)
O52-C66-C65	151.0(3)
O52-C66-C51	151.6(3)
C65-C66-C51	57.40(19)

2.4 Crystallographic Data for 7α

Table 1: Crystal data and structure refinement for 7α.

Identification	
Empirical formula	C ₁₆ H ₂₀ S ₆
Formula weight	404.68
Temperature	200(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2 ₁ /n
Z	2
Unit cell dimensions	a = 9.0844(3) Å α = 90 deg. b = 11.8406(3) Å β = 113.063(1) deg. c = 9.8502(3) Å γ = 90 deg.
Volume	974.85(5) Å ³
Density (calculated)	1.38 g/cm ³
Absorption coefficient	0.69 mm ⁻¹
Crystal shape	polyhedron
Crystal size	0.37 x 0.30 x 0.05 mm ³
Theta range for data collection	2.6 to 27.4 deg.
Index ranges	-11 ≤ h ≤ 10, -15 ≤ k ≤ 14, -10 ≤ l ≤ 12
Reflections collected	4668
Independent reflections	2093 (R(int) = 0.0244)
Observed reflections	1654 (I > 2σ(I))
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.97 and 0.78
Refinement method	Full-matrix least-squares on F ²
Data/restraints/parameters	2093 / 0 / 100
Goodness-of-fit on F ²	1.07
Final R indices (I > 2σ(I))	R1 = 0.031, wR2 = 0.074
Largest diff. peak and hole	0.27 and -0.24 eÅ ⁻³

Table 2: Atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$) for **7a**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
S1	0.5408(1)	0.0045(1)	0.2275(1)	0.0494(2)
S2	0.8928(1)	-0.1593(1)	0.6075(1)	0.0368(2)
S3	0.8491(1)	0.1978(1)	0.6043(1)	0.0342(1)
C1	0.6819(2)	0.0109(1)	0.3954(2)	0.0345(5)
C2	0.7955(2)	-0.0415(2)	0.5206(2)	0.0314(4)
C3	0.7808(2)	0.0728(2)	0.5191(2)	0.0317(4)
C4	0.7311(2)	0.2951(1)	0.4580(2)	0.0341(4)
C5	0.5971(2)	0.3513(1)	0.4890(2)	0.0329(4)
C6	0.4714(2)	0.2704(1)	0.4996(2)	0.0281(4)
C7	0.3268(2)	0.3361(1)	0.5004(2)	0.0332(4)
C8	0.7947(2)	-0.2655(1)	0.4660(2)	0.0345(4)

Table 3: Hydrogen coordinates and isotropic displacement parameters ($\text{\AA}^2$) for **7a**.

Atom	x	y	z	U_{eq}
H4A	0.8025	0.3542	0.4466	0.041
H4B	0.6845	0.2532	0.3637	0.041
H5A	0.5433	0.4067	0.4096	0.039
H5B	0.6447	0.3936	0.5828	0.039
H6A	0.5184	0.2252	0.5912	0.034
H6B	0.4368	0.2179	0.4147	0.034
H7A	0.3654	0.3972	0.5747	0.040
H7B	0.2722	0.3721	0.4027	0.040
H8A	0.7401	-0.2272	0.3699	0.041
H8B	0.8771	-0.3162	0.4570	0.041

Table 4: Anisotropic displacement parameters ($\text{\AA}^2$) for **7a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 (h^2 a^{*2} U_{11} + \dots + 2hk a^* b^* U_{12})$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	0.0545(3)	0.0378(3)	0.0354(3)	-0.0007(2)	-0.0045(3)	-0.0058(2)
S2	0.0318(2)	0.0386(3)	0.0337(3)	0.0027(2)	0.0061(2)	0.0003(2)
S3	0.0308(2)	0.0349(2)	0.0323(3)	-0.0080(2)	0.0076(2)	-0.0041(2)
C1	0.0359(10)	0.0315(9)	0.0341(12)	-0.0018(8)	0.0116(9)	-0.0064(7)
C2	0.0307(9)	0.0357(9)	0.0282(11)	-0.0006(8)	0.0121(8)	-0.0054(7)
C3	0.0287(9)	0.0367(9)	0.0305(11)	-0.0020(8)	0.0122(8)	-0.0045(7)
C4	0.0355(9)	0.0325(9)	0.0343(11)	-0.0004(8)	0.0136(8)	-0.0071(7)
C5	0.0389(10)	0.0230(8)	0.0353(11)	-0.0016(8)	0.0131(8)	-0.0044(7)
C6	0.0325(9)	0.0229(8)	0.0281(10)	0.0012(7)	0.0108(8)	-0.0006(6)
C7	0.0352(10)	0.0255(8)	0.0369(11)	0.0031(8)	0.0120(8)	0.0019(7)
C8	0.0363(10)	0.0305(9)	0.0373(11)	-0.0016(8)	0.0150(8)	0.0028(7)

Table 5: Bond lengths ($\text{\AA}$) and angles (deg) for **7a**.

S1-C1	1.650(2)	S2-C2	1.6932(18)
-------	----------	-------	------------

S2-C8	1.8295(18)
S3-C3	1.6949(17)
S3-C4	1.8290(18)
C1-C3	1.403(2)
C1-C2	1.405(3)
C2-C3	1.358(3)
C4-C5	1.519(2)
C5-C6	1.525(2)
C6-C7	1.530(2)
C7-C8#1	1.520(2)
C8-C7#1	1.520(2)
C2-S2-C8	100.34(8)
C3-S3-C4	99.92(8)
C3-C1-C2	57.84(13)
C3-C1-S1	151.16(15)
C2-C1-S1	150.99(15)
C3-C2-C1	61.01(12)
C3-C2-S2	148.81(15)
C1-C2-S2	149.99(15)
C2-C3-C1	61.15(12)
C2-C3-S3	148.04(15)
C1-C3-S3	150.59(15)
C5-C4-S3	112.74(13)
C4-C5-C6	114.79(14)
C5-C6-C7	110.44(13)
C8#1-C7-C6	114.84(14)
C7#1-C8-S2	113.01(13)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1

2.5 Crystallographic Data of 7 β

Table 1: Crystal data and structure refinement for 7 β .

Empirical formula	$C_{16}H_{20}S_6$	
Formula weight	404.68	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P \bar{1}$	
Z	2	
Unit cell dimensions	$a = 9.4288(3)$ Å	$\alpha = 107.530(1)$ deg.
	$b = 10.8171(3)$ Å	$\beta = 113.144(1)$ deg.
	$c = 11.4266(4)$ Å	$\gamma = 98.332(1)$ deg.
Volume	$974.70(5)$ Å ³	
Density (calculated)	1.38 g/cm ³	
Absorption coefficient	0.69 mm ⁻¹	
Crystal shape	polyhedron	
Crystal size	$.40 \times .18 \times .16$ mm ³	
Theta range for data collection	2.1 to 27.5 deg.	
Index ranges	$-12 \leq h \leq 12$, $-14 \leq k \leq 13$, $-14 \leq l \leq 14$	
Reflections collected	9197	
Independent reflections	4390 ($R(\text{int}) = 0.0255$)	
Observed reflections	3561 ($I > 2\sigma(I)$)	
Absorption correction	Semi-empirical from equivalents	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	4390 / 6 / 208	
Goodness-of-fit on F^2	1.04	
Final R indices ($I > 2\sigma(I)$)	$R1 = 0.034$, $wR2 = 0.082$	
Largest diff. peak and hole	0.88 and -0.58 eÅ ⁻³	

Table 2: Atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$) for **7 β** . U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
S1	-0.1876(1)	1.1545(1)	0.3659(1)	0.0385(1)
S2	0.0420(1)	0.8520(1)	0.2717(1)	0.0320(1)
S3	0.3257(1)	1.2343(1)	0.6135(1)	0.0360(1)
S11	0.3039(1)	1.5808(1)	0.0580(1)	0.0542(2)
S12	0.1169(1)	1.2768(1)	0.1804(1)	0.0497(2)
S13	0.3745(1)	1.1839(1)	-0.0469(1)	0.0388(1)
C1	-0.0234(2)	1.1060(2)	0.3998(2)	0.0289(4)
C2	0.0560(2)	1.0095(2)	0.3718(2)	0.0278(4)
C3	0.1432(2)	1.1322(2)	0.4797(2)	0.0287(4)
C4	0.2758(3)	1.3895(2)	0.6774(2)	0.0366(4)
C5	0.2051(3)	1.4505(2)	0.5713(2)	0.0349(6)
C6	0.3123(3)	1.4870(2)	0.5082(2)	0.0400(6)
C7	0.2300(3)	1.5344(2)	0.3923(2)	0.0492(6)
C8	0.0832(3)	1.4336(3)	0.2651(3)	0.0556(7)
C11	0.2833(2)	1.4304(2)	0.0661(2)	0.0364(4)
C12	0.2245(2)	1.3333(2)	0.1067(2)	0.0360(4)
C13	0.3094(2)	1.3031(2)	0.0354(2)	0.0330(4)
C14	0.2896(3)	1.0444(2)	-0.0136(2)	0.0340(4)
C15	0.3734(2)	1.0603(2)	0.1367(2)	0.0318(4)
C16	0.2825(3)	0.9488(2)	0.1581(2)	0.0340(4)
C17	0.3511(2)	0.9663(2)	0.3091(2)	0.0327(4)
C18	0.2543(2)	0.8598(2)	0.3326(2)	0.0349(4)
C5B	0.308(3)	1.492(2)	0.619(2)	0.028(7)
C6B	0.199(3)	1.438(2)	0.468(2)	0.034(8)

Table 3: Hydrogen coordinates and isotropic displacement parameters ($\text{\AA}^2$) for **7 β** .

Atom	x	y	z	U_{eq}
H4A	0.1972	1.3696	0.7116	0.044
H4B	0.3750	1.4583	0.7572	0.044
H4C	0.1599	1.3650	0.6541	0.044
H4D	0.3393	1.4320	0.7796	0.044
H5A	0.1825	1.5336	0.6162	0.042
H5B	0.1006	1.3849	0.4956	0.042
H6A	0.3440	1.4066	0.4710	0.048
H6B	0.4121	1.5600	0.5819	0.048
H7A	0.1989	1.6147	0.4309	0.059
H7B	0.3107	1.5648	0.3638	0.059
H7C	0.2176	1.6254	0.4285	0.059
H7D	0.3317	1.5407	0.3849	0.059
H8A	0.0442	1.4767	0.1981	0.067
H8B	-0.0031	1.4107	0.2906	0.067
H14A	0.1739	1.0357	-0.0413	0.041
H14B	0.2958	0.9591	-0.0725	0.041
H15A	0.4853	1.0565	0.1619	0.038
H15B	0.3787	1.1500	0.1977	0.038
H16A	0.2858	0.8601	0.1028	0.041
H16B	0.1678	0.9472	0.1235	0.041
H17A	0.4636	0.9619	0.3418	0.039
H17B	0.3545	1.0575	0.3653	0.039
H18A	0.3039	0.8795	0.4329	0.042
H18B	0.2612	0.7696	0.2848	0.042
H5B1	0.2906	1.5776	0.6657	0.034

H5B2	0.4223	1.5134	0.6365	0.034
H6B1	0.0861	1.4212	0.4529	0.041
H6B2	0.2120	1.3487	0.4242	0.041

Table 4: Anisotropic displacement parameters ($\text{\AA}^2$) for **7 β** . The anisotropic displacement factor exponent takes the form: $-2 \pi^2 (h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12})$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	0.0325(3)	0.0467(3)	0.0314(3)	0.0074(2)	0.0139(2)	0.0185(2)
S2	0.0362(3)	0.0274(2)	0.0314(2)	0.0090(2)	0.0170(2)	0.0086(2)
S3	0.0311(3)	0.0325(3)	0.0347(3)	0.0099(2)	0.0079(2)	0.0113(2)
S11	0.0773(4)	0.0331(3)	0.0517(4)	0.0191(3)	0.0260(3)	0.0218(3)
S12	0.0563(4)	0.0415(3)	0.0516(3)	0.0085(3)	0.0353(3)	0.0068(3)
S13	0.0547(3)	0.0361(3)	0.0456(3)	0.0214(2)	0.0362(3)	0.0190(2)
C1	0.0344(10)	0.0316(10)	0.0241(9)	0.0109(7)	0.0166(8)	0.0107(8)
C2	0.0315(9)	0.0303(10)	0.0269(9)	0.0138(7)	0.0162(8)	0.0107(8)
C3	0.0328(10)	0.0293(10)	0.0277(9)	0.0131(8)	0.0157(8)	0.0111(8)
C4	0.0421(11)	0.0321(11)	0.0335(10)	0.0081(8)	0.0192(9)	0.0111(9)
C5	0.0400(14)	0.0300(11)	0.0423(13)	0.0144(10)	0.0248(11)	0.0146(10)
C6	0.0422(14)	0.0350(12)	0.0427(13)	0.0113(10)	0.0241(11)	0.0068(10)
C7	0.0847(18)	0.0363(12)	0.0529(14)	0.0223(11)	0.0486(14)	0.0313(12)
C8	0.0646(16)	0.0725(18)	0.0504(14)	0.0261(13)	0.0369(13)	0.0435(14)
C11	0.0393(11)	0.0321(11)	0.0283(10)	0.0076(8)	0.0100(8)	0.0104(8)
C12	0.0369(11)	0.0316(10)	0.0310(10)	0.0061(8)	0.0135(8)	0.0074(8)
C13	0.0368(10)	0.0293(10)	0.0295(10)	0.0107(8)	0.0134(8)	0.0083(8)
C14	0.0471(12)	0.0283(10)	0.0333(10)	0.0130(8)	0.0240(9)	0.0120(8)
C15	0.0320(10)	0.0332(10)	0.0326(10)	0.0139(8)	0.0163(8)	0.0108(8)
C16	0.0424(11)	0.0310(10)	0.0316(10)	0.0136(8)	0.0189(9)	0.0113(8)
C17	0.0309(10)	0.0408(11)	0.0326(10)	0.0181(9)	0.0160(8)	0.0165(8)
C18	0.0427(11)	0.0395(11)	0.0354(10)	0.0222(9)	0.0212(9)	0.0229(9)

Table 5: Bond lengths ($\text{\AA}$) and angles (deg) for **7 β** .

S1-C1	1.6453(19)	C16-C17	1.523(3)
S2-C2	1.6946(19)	C17-C18	1.520(3)
S2-C18	1.822(2)	C5B-C6B	1.482(18)
S3-C3	1.6976(19)	C2-S2-C18	101.49(9)
S3-C4	1.825(2)	C3-S3-C4	100.99(10)
S11-C11	1.646(2)	C12-S12-C8	100.38(11)
S12-C12	1.707(2)	C13-S13-C14	97.74(9)
S12-C8	1.823(2)	C2-C1-C3	58.12(13)
S13-C13	1.696(2)	C2-C1-S1	151.37(16)
S13-C14	1.8159(19)	C3-C1-S1	150.51(16)
C1-C2	1.402(3)	C3-C2-C1	61.00(13)
C1-C3	1.404(3)	C3-C2-S2	151.38(15)
C2-C3	1.363(3)	C1-C2-S2	147.62(15)
C4-C5B	1.512(16)	C2-C3-C1	60.88(13)
C4-C5	1.525(3)	C2-C3-S3	147.62(15)
C5-C6	1.525(3)	C1-C3-S3	151.49(16)
C6-C7	1.528(3)	C5B-C4-C5	33.3(10)
C7-C8	1.497(4)	C5B-C4-S3	113.9(11)
C7-C6B	1.609(16)	C5-C4-S3	115.02(14)
C11-C12	1.397(3)	C4-C5-C6	115.03(19)
C11-C13	1.398(3)	C5-C6-C7	113.2(2)
C12-C13	1.356(3)	C8-C7-C6	117.2(2)
C14-C15	1.523(3)	C8-C7-C6B	86.2(8)
C15-C16	1.514(3)	C6-C7-C6B	35.8(9)

C7-C8-S12	114.25(17)
C12-C11-C13	58.06(14)
C12-C11-S11	151.10(18)
C13-C11-S11	150.83(18)
C13-C12-C11	61.03(15)
C13-C12-S12	146.63(17)
C11-C12-S12	152.24(17)
C12-C13-C11	60.92(15)
C12-C13-S13	147.51(17)
C11-C13-S13	151.07(17)
C15-C14-S13	114.29(14)
C16-C15-C14	110.56(16)
C15-C16-C17	113.18(17)
C18-C17-C16	113.49(17)
C17-C18-S2	113.81(13)
C6B-C5B-C4	109.9(15)
C5B-C6B-C7	114.6(15)

2.6 Crystallographic Data of 19

Table 1: Crystal data and structure refinement for **19**.

Identification code	as8	
Empirical formula	C ₂₄ H ₄₀ Br ₄ O ₂	
Formula weight	680.20	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	P2 ₁ /c	
Z	2	
Unit cell dimensions	a = 10.5953(6) Å	α = 90 deg.
	b = 11.4554(6) Å	β = 108.903(1) deg.
	c = 11.5796(7) Å	γ = 90 deg.
Volume	1329.66(13) Å ³	
Density (calculated)	1.70 Mg/m ³	
Absorption coefficient	6.07 mm ⁻¹	
Crystal shape	polyhedron	
Crystal size	.30 x .20 x .14 mm ³	
Theta range for data collection	2.0 to 25.6 deg.	
Index ranges	-12 ≤ h ≤ 12, -13 ≤ k ≤ 13, -13 ≤ l ≤ 13	

Reflections collected	9712
Independent reflections	2317 (R(int) = 0.0227)
Observed reflections	2093 (I > 2 σ (I))
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.54 and 0.32
Refinement method	Full-matrix least-squares on F ²
Data/restraints/parameters	2317 / 0 / 216
Goodness-of-fit on F ²	1.10
Final R indices (I > 2 σ (I))	R1 = 0.022, wR2 = 0.052
Largest diff. peak and hole	0.32 and -0.48 eÅ ⁻³

Table 2: Atomic coordinates and equivalent isotropic displacement parameters (Å²) for **19**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U _{eq}
Br4	0.4486(1)	0.9758(1)	0.1523(1)	0.0336(1)
Br6	0.8178(1)	0.9110(1)	0.2133(1)	0.0458(1)
C1	0.2435(2)	0.5408(2)	-0.0269(3)	0.0268(5)
H1A	0.194(3)	0.528(2)	0.029(2)	0.031(7)
H1B	0.180(3)	0.573(2)	-0.102(3)	0.033(7)
C2	0.3595(2)	0.6263(2)	0.0232(2)	0.0216(5)
H2A	0.424(2)	0.5902(18)	0.085(2)	0.006(5)
H2B	0.397(3)	0.640(2)	-0.040(2)	0.027(7)
C3	0.3309(2)	0.7454(2)	0.0716(2)	0.0209(5)
C4	0.4586(2)	0.8179(2)	0.0866(2)	0.0210(5)
H4	0.463(2)	0.836(2)	0.006(2)	0.016(6)
C5	0.5931(2)	0.7692(2)	0.1698(2)	0.0211(5)
O5	0.6046(2)	0.7275(1)	0.2683(1)	0.0267(4)
C6	0.7117(2)	0.7790(2)	0.1215(2)	0.0245(5)
H6	0.687(2)	0.806(2)	0.042(2)	0.017(6)
C7	0.7978(2)	0.6679(2)	0.1272(2)	0.0243(5)
C8	0.6990(2)	0.5749(2)	0.0540(2)	0.0253(5)
H8A	0.648(3)	0.609(2)	-0.022(2)	0.026(7)
H8B	0.636(3)	0.553(2)	0.103(2)	0.030(7)
C31	0.2125(3)	0.8056(2)	-0.0224(2)	0.0283(5)
H31A	0.224(3)	0.815(3)	-0.094(3)	0.035(8)
H31B	0.134(3)	0.759(2)	-0.034(2)	0.028(7)
H31C	0.195(3)	0.883(3)	0.006(3)	0.037(7)
C32	0.2992(3)	0.7309(2)	0.1911(2)	0.0258(5)
H32A	0.279(3)	0.808(3)	0.215(2)	0.032(7)
H32B	0.366(3)	0.693(3)	0.251(3)	0.034(8)
H32C	0.223(3)	0.685(3)	0.178(2)	0.031(7)
C71	0.9005(3)	0.6961(3)	0.0637(3)	0.0410(7)
H71A	0.956(3)	0.751(3)	0.104(3)	0.037(8)
H71B	0.952(3)	0.623(3)	0.064(3)	0.048(9)
H71C	0.855(3)	0.721(3)	-0.021(3)	0.051(9)
C72	0.8679(3)	0.6253(3)	0.2568(3)	0.0337(6)
H72A	0.922(3)	0.681(3)	0.302(3)	0.037(8)
H72B	0.814(3)	0.602(3)	0.296(3)	0.040(9)
H72C	0.926(3)	0.566(3)	0.253(3)	0.040(8)

Table 3: Hydrogen coordinates and isotropic displacement parameters (Å²) for **19**.

Atom	x	y	z	U _{eq}
H1A	0.194(3)	0.528(2)	0.029(2)	0.031(7)

H1B	0.180(3)	0.573(2)	-0.102(3)	0.033(7)
H2A	0.424(2)	0.5902(18)	0.085(2)	0.006(5)
H2B	0.397(3)	0.640(2)	-0.040(2)	0.027(7)
H4	0.463(2)	0.836(2)	0.006(2)	0.016(6)
H6	0.687(2)	0.806(2)	0.042(2)	0.017(6)
H8A	0.648(3)	0.609(2)	-0.022(2)	0.026(7)
H8B	0.636(3)	0.553(2)	0.103(2)	0.030(7)
H31A	0.224(3)	0.815(3)	-0.094(3)	0.035(8)
H31B	0.134(3)	0.759(2)	-0.034(2)	0.028(7)
H31C	0.195(3)	0.883(3)	0.006(3)	0.037(7)
H32A	0.279(3)	0.808(3)	0.215(2)	0.032(7)
H32B	0.366(3)	0.693(3)	0.251(3)	0.034(8)
H32C	0.223(3)	0.685(3)	0.178(2)	0.031(7)
H71A	0.956(3)	0.751(3)	0.104(3)	0.037(8)
H71B	0.952(3)	0.623(3)	0.064(3)	0.048(9)
H71C	0.855(3)	0.721(3)	-0.021(3)	0.051(9)
H72A	0.922(3)	0.681(3)	0.302(3)	0.037(8)
H72B	0.814(3)	0.602(3)	0.296(3)	0.040(9)
H72C	0.926(3)	0.566(3)	0.253(3)	0.040(8)

Table 4: Anisotropic displacement parameters ($\text{\AA}^2$) for **19**. The anisotropic displacement factor exponent takes the form: $-2\pi^2(h^2 a^{*2} U_{11} + \dots + 2hk a^* b^* U_{12})$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br4	0.0415(2)	0.0186(1)	0.0396(2)	-0.0042(1)	0.0116(1)	0.0018(1)
Br6	0.0404(2)	0.0264(2)	0.0705(2)	-0.0121(1)	0.0179(2)	-0.0153(1)
C1	0.0192(12)	0.0271(13)	0.0335(14)	-0.0086(11)	0.0076(11)	-0.0026(10)
C2	0.0167(11)	0.0240(12)	0.0216(12)	-0.0024(10)	0.0027(11)	0.0011(10)
C3	0.0215(11)	0.0215(11)	0.0202(12)	-0.0006(9)	0.0075(10)	0.0013(9)
C4	0.0266(12)	0.0167(11)	0.0202(12)	-0.0006(9)	0.0084(10)	0.0013(9)
C5	0.0263(12)	0.0130(11)	0.0224(13)	-0.0047(9)	0.0056(10)	-0.0030(9)
O5	0.0269(9)	0.0281(9)	0.0221(9)	0.0020(7)	0.0037(7)	-0.0004(7)
C6	0.0250(12)	0.0212(12)	0.0256(13)	-0.0018(10)	0.0058(11)	-0.0058(10)
C7	0.0162(11)	0.0250(12)	0.0302(13)	-0.0059(10)	0.0055(10)	-0.0029(9)
C8	0.0176(11)	0.0268(13)	0.0284(13)	-0.0053(10)	0.0031(11)	-0.0008(10)
C31	0.0274(14)	0.0324(15)	0.0240(14)	0.0033(11)	0.0069(11)	0.0081(11)
C32	0.0264(13)	0.0292(14)	0.0226(13)	0.0004(11)	0.0093(11)	0.0006(11)
C71	0.0304(15)	0.0416(17)	0.058(2)	-0.0106(16)	0.0235(15)	-0.0104(14)
C72	0.0249(14)	0.0346(15)	0.0333(15)	-0.0077(12)	-0.0023(13)	0.0043(13)

Table 5: Bond lengths ($\text{\AA}$) and angles (deg) for **19**.

Br4-C4	1.978(2)	C8-C1#1	1.533(3)
Br6-C6	1.976(2)	C2-C1-C8#1	107.95(19)
C1-C2	1.531(3)	C1-C2-C3	118.46(19)
C1-C8#1	1.533(3)	C31-C3-C32	108.74(19)
C2-C3	1.541(3)	C31-C3-C2	110.59(19)
C3-C31	1.533(3)	C32-C3-C2	110.96(19)
C3-C32	1.535(3)	C31-C3-C4	108.8(2)
C3-C4	1.549(3)	C32-C3-C4	113.31(19)
C4-C5	1.542(3)	C2-C3-C4	104.37(17)
C5-O5	1.207(3)	C5-C4-C3	118.20(19)
C5-C6	1.536(3)	C5-C4-Br4	104.15(14)
C6-C7	1.555(3)	C3-C4-Br4	112.28(15)
C7-C72	1.523(4)	O5-C5-C6	121.9(2)
C7-C71	1.531(4)	O5-C5-C4	121.6(2)
C7-C8	1.541(3)	C6-C5-C4	116.44(19)

C5-C6-C7	117.59(19)
C5-C6-Br6	104.51(15)
C7-C6-Br6	112.27(15)
C72-C7-C71	110.2(2)
C72-C7-C8	110.3(2)
C71-C7-C8	110.1(2)
C72-C7-C6	113.4(2)
C71-C7-C6	107.8(2)
C8-C7-C6	104.93(18)
C1#1-C8-C7	117.82(19)

Symmetry transformations used to generate equivalent atoms:

#1 $-x+1, -y+1, -z$

2.7 Crystallographic Data of 22

Table 1: Crystal data and structure refinement for **22**.

Empirical formula
Formula weight

C₅H₆S₃
162.28

Temperature	200(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	Pnma
Z	4
Unit cell dimensions	a = 10.2496(11) Å $\alpha = 90$ deg. b = 6.7971(7) Å $\beta = 90$ deg. c = 10.6545(11) Å $\gamma = 90$ deg.
Volume	742.27(13) Å ³
Density (calculated)	1.45 g/cm ³
Absorption coefficient	0.89 mm ⁻¹
Crystal shape	polyhedron
Crystal size	0.34 x 0.11 x 0.09 mm ³
Theta range for data collection	2.8 to 27.5 deg.
Index ranges	-13 ≤ h ≤ 9, -8 ≤ k ≤ 8, -13 ≤ l ≤ 12
Reflections collected	4249
Independent reflections	917 (R(int) = 0.0375)
Observed reflections	724 (I > 2σ(I))
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.92 and 0.75
Refinement method	Full-matrix least-squares on F ²
Data/restraints/parameters	917 / 0 / 52
Goodness-of-fit on F ²	1.12
Final R indices (I > 2σ(I))	R1 = 0.024, wR2 = 0.054
Largest diff. peak and hole	0.31 and -0.22 eÅ ⁻³

Table 2: Atomic coordinates and equivalent isotropic displacement parameters (Å²) for **22**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U _{eq}
S1	0.3946(1)	0.2500	0.2837(1)	0.0345(2)
S2	0.3736(1)	0.2500	0.6876(1)	0.0329(2)
S3	0.7447(1)	0.2500	0.5158(1)	0.0354(2)
C1	0.4720(2)	0.2500	0.4249(2)	0.0264(5)
C2	0.4601(2)	0.2500	0.5520(2)	0.0271(5)
C3	0.5845(2)	0.2500	0.4987(2)	0.0264(5)
C4	0.2081(2)	0.2500	0.6309(2)	0.0362(6)
C5	0.5332(2)	0.2500	0.1782(2)	0.0352(6)

Table 3: Hydrogen coordinates and isotropic displacement parameters (Å²) for **22**.

Atom	x	y	z	U _{eq}
H4A	0.1854	0.3818	0.6007	0.054
H4B	0.1489	0.2128	0.6992	0.054
H4C	0.1997	0.1554	0.5619	0.054
H5A	0.6021	0.3342	0.2126	0.053
H5B	0.5063	0.3003	0.0960	0.053
H5C	0.5663	0.1155	0.1690	0.053

Table 4: Anisotropic displacement parameters (Å²) for **22**. The anisotropic displacement factor exponent takes the form: $-2\pi^2(h^2 a^{*2} U_{11} + \dots + 2hk a^* b^* U_{12})$

Atom	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
S1	0.0243(3)	0.0551(4)	0.0241(3)	0.000	0.0002(2)	0.000
S2	0.0296(3)	0.0464(4)	0.0228(3)	0.000	0.0040(2)	0.000
S3	0.0269(3)	0.0527(4)	0.0266(3)	0.000	-0.0003(2)	0.000
C1	0.0264(12)	0.0266(12)	0.0262(10)	0.000	0.0027(8)	0.000
C2	0.0284(12)	0.0271(12)	0.0258(10)	0.000	0.0006(9)	0.000
C3	0.0316(13)	0.0260(11)	0.0216(10)	0.000	0.0017(8)	0.000
C4	0.0270(13)	0.0451(15)	0.0366(12)	0.000	0.0023(9)	0.000
C5	0.0286(13)	0.0519(16)	0.0252(10)	0.000	0.0026(9)	0.000

Table 5: Bond lengths (Å) and angles (deg) for **22**.

S1-C1	1.701(2)
S1-C5	1.812(2)
S2-C2	1.695(2)
S2-C4	1.801(2)
S3-C3	1.652(2)
C1-C2	1.360(3)
C1-C3	1.395(3)
C2-C3	1.396(3)
C1-S1-C5	100.53(10)
C2-S2-C4	101.93(10)
C2-C1-C3	60.89(14)
C2-C1-S1	147.02(17)
C3-C1-S1	152.09(17)
C1-C2-C3	60.82(14)
C1-C2-S2	153.63(18)
C3-C2-S2	145.55(17)
C1-C3-C2	58.29(14)
C1-C3-S3	152.09(16)
C2-C3-S3	149.62(16)

2.8 Crystallographic Data of 23 α

Table 1: Crystal data and structure refinement for 23 α .

Empirical formula	C ₆ H ₉ IS ₃
Formula weight	304.21
Temperature	200(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P $\bar{1}$
Z	2
Unit cell dimensions	a = 6.5122(4) Å α = 74.596(2) deg. b = 7.7517(5) Å β = 83.282(2) deg. c = 11.2286(8) Å γ = 74.962(2) deg.
Volume	527.05(6) Å ³
Density (calculated)	1.92 g/cm ³
Absorption coefficient	3.57 mm ⁻¹
Crystal shape	polyhedron
Crystal size	0.11 x 0.10 x 0.02 mm ³
Theta range for data collection	1.9 to 26.4 deg.
Index ranges	-8 ≤ h ≤ 8, -9 ≤ k ≤ 9, -14 ≤ l ≤ 14
Reflections collected	5065
Independent reflections	2134 (R(int) = 0.0383)
Observed reflections	1755 (I > 2σ(I))
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.93 and 0.69
Refinement method	Full-matrix least-squares on F ²
Data/restraints/parameters	2134 / 0 / 94
Goodness-of-fit on F ²	1.00

Final R indices ($I > 2\sigma(I)$)
Largest diff. peak and hole

R1 = 0.029, wR2 = 0.050
0.44 and -0.50 eÅ⁻³

Table 2: Atomic coordinates and equivalent isotropic displacement parameters (Å²) for **23α**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
I1	0.7084(1)	0.6497(1)	0.7141(1)	0.0330(1)
S1	1.0566(2)	0.9740(2)	0.8138(1)	0.0461(3)
S2	0.5110(2)	1.2487(1)	0.9897(1)	0.0355(2)
S3	0.5173(2)	1.2361(1)	0.5984(1)	0.0323(2)
C1	0.7992(6)	1.0948(5)	0.8032(4)	0.0314(9)
C2	0.6216(6)	1.1883(5)	0.8589(3)	0.0306(9)
C3	0.6232(6)	1.1826(5)	0.7377(3)	0.0279(8)
C4	1.1194(7)	0.9511(6)	0.6575(4)	0.0509(12)
C5	0.2563(6)	1.3895(6)	0.9394(4)	0.0438(11)
C6	0.2719(6)	1.3992(5)	0.6223(3)	0.0325(9)

Table 3: Hydrogen coordinates and isotropic displacement parameters (Å²) for **23α**.

Atom	x	y	z	U_{eq}
H4A	1.0295	0.8796	0.6381	0.076
H4B	1.2695	0.8877	0.6489	0.076
H4C	1.0934	1.0736	0.6004	0.076
H5A	0.2762	1.4915	0.8693	0.066
H5B	0.1765	1.4389	1.0075	0.066
H5C	0.1768	1.3150	0.9139	0.066
H6A	0.1818	1.3376	0.6869	0.049
H6B	0.1972	1.4481	0.5451	0.049
H6C	0.3034	1.5006	0.6480	0.049

Table 4: Anisotropic displacement parameters (Å²) for **23α**. The anisotropic displacement factor exponent takes the form: $-2\pi^2(h^2 a^{*2} U_{11} + \dots + 2hk a^* b^* U_{12})$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I1	0.0372(2)	0.0360(2)	0.0295(2)	-0.0113(1)	-0.0005(1)	-0.0123(1)
S1	0.0326(6)	0.0391(6)	0.0644(8)	-0.0141(6)	-0.0144(6)	0.0021(5)
S2	0.0414(6)	0.0363(6)	0.0277(5)	-0.0088(4)	-0.0032(5)	-0.0059(5)
S3	0.0303(6)	0.0375(5)	0.0293(5)	-0.0131(4)	-0.0005(4)	-0.0041(5)
C1	0.031(2)	0.028(2)	0.035(2)	-0.0064(17)	-0.0062(18)	-0.0068(18)
C2	0.034(2)	0.0225(19)	0.034(2)	-0.0037(17)	-0.0022(18)	-0.0081(18)
C3	0.025(2)	0.0238(18)	0.036(2)	-0.0071(16)	-0.0010(16)	-0.0077(16)
C4	0.031(2)	0.047(3)	0.082(4)	-0.035(2)	0.007(2)	-0.006(2)
C5	0.033(2)	0.054(3)	0.044(3)	-0.022(2)	-0.002(2)	0.000(2)
C6	0.032(2)	0.033(2)	0.029(2)	-0.0092(17)	-0.0014(17)	-0.0006(18)

Table 5: Bond lengths (Å) and angles (deg) for **23α**.

S1-C1	1.696(4)
S1-C4	1.800(5)
S2-C2	1.685(4)
S2-C5	1.793(4)
S3-C3	1.688(4)
S3-C6	1.804(4)
C1-C3	1.358(5)
C1-C2	1.375(5)
C2-C3	1.372(5)
C1-S1-C4	100.6(2)
C2-S2-C5	101.23(19)
C3-S3-C6	101.05(18)
C3-C1-C2	60.3(3)
C3-C1-S1	151.7(3)
C2-C1-S1	147.8(3)
C3-C2-C1	59.3(3)
C3-C2-S2	154.6(3)
C1-C2-S2	146.2(3)
C1-C3-C2	60.5(3)
C1-C3-S3	145.2(3)
C2-C3-S3	154.3(3)

2.9 Crystallographic Data of 23 β

Table 1: Crystal data and structure refinement for 23 β .

Empirical formula	$C_7H_{11}Cl_2IS_3$	
Formula weight	389.14	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pnma	
Z	4	
Unit cell dimensions	$a = 17.2213(7)$ Å	$\alpha = 90$ deg.
	$b = 7.0050(3)$ Å	$\beta = 90$ deg.
	$c = 11.4894(5)$ Å	$\gamma = 90$ deg.
Volume	$1386.03(10)$ Å ³	
Density (calculated)	1.87 g/cm ³	
Absorption coefficient	3.11 mm ⁻¹	
Crystal shape	needle	
Crystal size	$0.68 \times 0.06 \times 0.04$ mm ³	
Theta range for data collection	2.1 to 27.5 deg.	
Index ranges	$-22 \leq h \leq 22, -9 \leq k \leq 9, -14 \leq l \leq 14$	
Reflections collected	11445	
Independent reflections	1718 ($R(\text{int}) = 0.0444$)	
Observed reflections	1326 ($I > 2\sigma(I)$)	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.89 and 0.23	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	1718 / 7 / 88	
Goodness-of-fit on F^2	1.06	
Final R indices ($I > 2\sigma(I)$)	$R1 = 0.030, wR2 = 0.060$	

Table 2: Atomic coordinates and equivalent isotropic displacement parameters (Å²) for **23β**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
I1	0.2573(1)	0.2500	0.0196(1)	0.0351(1)
S1	-0.0284(1)	0.2500	0.5214(1)	0.0354(3)
S3	0.1811(1)	0.2500	0.7168(1)	0.0341(3)
S2	0.1787(1)	0.2500	0.3299(1)	0.0375(3)
C1	0.0699(3)	0.2500	0.5247(4)	0.0303(10)
C2	0.1385(3)	0.2500	0.4638(4)	0.0303(11)
C3	0.1389(3)	0.2500	0.5838(4)	0.0290(11)
C4	-0.0406(3)	0.2500	0.3648(5)	0.0501(16)
C5	0.2811(3)	0.2500	0.3637(5)	0.0408(14)
C6	0.2829(3)	0.2500	0.6797(4)	0.0380(13)
C10	0.5403(6)	0.2500	0.5555(7)	0.223(11)
Cl1	0.4865(2)	0.2500	0.6815(2)	0.1240(19)
Cl2	0.4838(3)	0.2500	0.4310(3)	0.199(3)
Cl1B	0.4802(16)	0.403(4)	0.620(3)	0.180(12)
Cl2B	0.5719(12)	0.068(3)	0.4865(16)	0.117(8)

Table 3: Hydrogen coordinates and isotropic displacement parameters (Å²) for **23β**.

Atom	x	y	z	U_{eq}
H4A	-0.0961	0.2500	0.3459	0.075
H4B	-0.0162	0.1358	0.3319	0.075
H4C	-0.0162	0.3642	0.3319	0.075
H5A	0.3111	0.2500	0.2912	0.061
H5B	0.2939	0.1358	0.4090	0.061
H5C	0.2939	0.3642	0.4090	0.061
H6A	0.3140	0.2500	0.7512	0.057
H6B	0.2951	0.3642	0.6339	0.057
H6C	0.2951	0.1358	0.6339	0.057
H10A	0.5742	0.1358	0.5548	0.268
H10B	0.5742	0.3642	0.5548	0.268

Table 4: Anisotropic displacement parameters (Å²) for **23β**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 (h^2 a^{*2} U_{11} + \dots + 2hk a^* b^* U_{12})$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I1	0.0455(2)	0.0340(2)	0.0258(2)	0.000	-0.0039(2)	0.000
S1	0.0342(6)	0.0419(7)	0.0301(6)	0.000	-0.0006(5)	0.000
S3	0.0385(7)	0.0403(8)	0.0234(6)	0.000	-0.0012(5)	0.000
S2	0.0464(8)	0.0439(8)	0.0223(6)	0.000	0.0019(5)	0.000
C1	0.039(3)	0.026(2)	0.026(2)	0.000	-0.004(2)	0.000
C2	0.038(3)	0.026(3)	0.027(3)	0.000	-0.002(2)	0.000
C3	0.036(3)	0.023(3)	0.028(3)	0.000	0.000(2)	0.000
C4	0.042(3)	0.077(5)	0.031(3)	0.000	-0.010(2)	0.000

C5	0.044(3)	0.043(3)	0.036(3)	0.000	0.009(2)	0.000
C6	0.038(3)	0.045(3)	0.031(3)	0.000	-0.004(2)	0.000
C10	0.068(6)	0.54(3)	0.061(6)	0.000	0.006(5)	0.000
Cl1	0.0564(16)	0.261(6)	0.0550(16)	0.000	0.0045(12)	0.000
Cl2	0.160(4)	0.379(9)	0.056(2)	0.000	-0.039(2)	0.000

Table 5: Bond lengths (Å) and angles (deg) for **23β**.

S1-C1	1.694(5)	C10-Cl2B-Cl1B#1	48.6(8)
S1-C4	1.812(5)	Cl1B#3-Cl2B-Cl1B#1	99.6(16)
S3-C3	1.691(5)		
S3-C6	1.805(5)		
S2-C2	1.687(5)		
S2-C5	1.806(5)		
C1-C3	1.369(7)		
C1-C2	1.374(7)		
C2-C3	1.379(6)		
C10-Cl2B#1	1.596(13)		
C10-Cl2B	1.596(13)		
C10-Cl1B	1.664(15)		
C10-Cl1B#1	1.664(15)		
C10-Cl1	1.719(9)		
C10-Cl2	1.730(9)		
Cl1B-Cl2B#2	1.91(4)		
Cl1B-Cl1B#1	2.14(5)		
Cl1B-Cl2B#1	2.21(3)		
Cl2B-Cl1B#3	1.91(4)		
Cl2B-Cl1B#1	2.21(3)		
C1-S1-C4	97.9(3)		
C3-S3-C6	101.8(2)		
C2-S2-C5	101.8(2)		
C3-C1-C2	60.4(3)		
C3-C1-S1	151.5(4)		
C2-C1-S1	148.1(4)		
C1-C2-C3	59.6(3)		
C1-C2-S2	144.8(4)		
C3-C2-S2	155.6(4)		
C1-C3-C2	60.0(4)		
C1-C3-S3	145.2(4)		
C2-C3-S3	154.8(4)		
Cl2B#1-C10-Cl2B	106.0(16)		
Cl2B#1-C10-Cl1B	85.4(14)		
Cl2B-C10-Cl1B	161.3(15)		
Cl2B#1-C10-Cl1B#1	161.3(15)		
Cl2B-C10-Cl1B#1	85.4(14)		
Cl1B-C10-Cl1B#1	80(2)		
Cl2B#1-C10-Cl1	127.0(8)		
Cl2B-C10-Cl1	127.0(8)		
Cl1B-C10-Cl1	44.7(12)		
Cl1B#1-C10-Cl1	44.7(12)		
Cl2B#1-C10-Cl2	77.4(8)		
Cl2B-C10-Cl2	77.4(8)		
Cl1B-C10-Cl2	91.1(13)		
Cl1B#1-C10-Cl2	91.1(13)		
Cl1-C10-Cl2	113.1(6)		
C10-Cl1B-Cl2B#2	113.3(19)		
C10-Cl1B-Cl1B#1	50.0(12)		
Cl2B#2-Cl1B-Cl1B#1	127.3(10)		
C10-Cl1B-Cl2B#1	46.0(8)		
Cl2B#2-Cl1B-Cl2B#1	80.5(16)		
Cl1B#1-Cl1B-Cl2B#1	95.3(8)		
C10-Cl2B-Cl1B#3	130.0(15)		

Symmetry transformations used to generate equivalent atoms:

#1 $x, -y+1/2, z$

#2 $-x+1, y+1/2, -z+1$

#3 $-x+1, y-1/2, -z+1$

2.10 Crystallographic Data of 24

Table 1: Crystal data and structure refinement for **24**.

Empirical formula	$C_6H_9I_3S_3$	
Formula weight	558.01	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Z	4	
Unit cell dimensions	$a = 9.6693(5)$ Å $b = 14.1562(3)$ Å $c = 10.6917(5)$ Å	$\alpha = 90$ deg. $\beta = 104.057(2)$ deg. $\gamma = 90$ deg.
Volume	$1419.66(10)$ Å ³	
Density (calculated)	2.61 g/cm ³	
Absorption coefficient	7.01 mm ⁻¹	

Crystal shape	polyhedron
Crystal size	.40 x .18 x .15 mm ³
Theta range for data collection	2.4 to 27.4 deg.
Index ranges	-8≤h≤12, -18≤k≤9, -11≤l≤13
Reflections collected	4332
Independent reflections	2868 (R(int) = 0.0246)
Observed reflections	2105 (I > 2σ(I))
Absorption correction	Semi-empirical from equivalents
Refinement method	Full-matrix least-squares on F ²
Data/restraints/parameters	2868 / 0 / 112
Goodness-of-fit on F ²	1.13
Final R indices (I > 2σ(I))	R1 = 0.033, wR2 = 0.081
Largest diff. peak and hole	1.35 and -1.01 eÅ ⁻³

Table 2: Atomic coordinates and equivalent isotropic displacement parameters (Å²) for **24**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U _{eq}
S1	-0.3841(2)	0.3815(1)	0.5089(2)	0.0314(4)
S2	-0.5817(2)	0.3696(1)	0.0904(2)	0.0345(4)
S3	-0.8311(2)	0.3801(1)	0.3630(2)	0.0336(4)
C1	-0.5219(7)	0.3795(4)	0.3779(6)	0.0234(14)
C2	-0.5833(7)	0.3758(4)	0.2483(7)	0.0264(14)
C3	-0.6689(7)	0.3792(5)	0.3332(6)	0.0269(14)
C4	-0.2316(7)	0.3774(5)	0.4396(7)	0.0320(16)
C5	-0.3927(8)	0.3648(6)	0.0999(7)	0.0382(19)
C6	-0.9372(8)	0.3631(5)	0.2002(7)	0.0371(18)
I1	0.0994(1)	0.3821(1)	0.7860(1)	0.0254(1)
I2	-0.1776(1)	0.3801(1)	0.8431(1)	0.0340(1)
I3	0.3846(1)	0.3780(1)	0.7319(1)	0.0318(1)

Table 3: Hydrogen coordinates and isotropic displacement parameters (Å²) for **24**.

Atom	x	y	z	U _{eq}
H4A	-0.2376	0.3211	0.3853	0.048
H4B	-0.1441	0.3749	0.5088	0.048
H4C	-0.2304	0.4340	0.3872	0.048
H5A	-0.3451	0.4182	0.1511	0.057
H5B	-0.3770	0.3682	0.0128	0.057
H5C	-0.3535	0.3055	0.1408	0.057
H6A	-0.9194	0.4151	0.1455	0.056
H6B	-1.0384	0.3621	0.2003	0.056
H6C	-0.9111	0.3031	0.1664	0.056

Table 4: Anisotropic displacement parameters (Å²) for **24**. The anisotropic displacement factor exponent takes the form: $-2\pi^2(h^2 a^{*2} U_{11} + \dots + 2hk a^* b^* U_{12})$

Atom	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
S1	0.0258(9)	0.0415(10)	0.0260(8)	0.0013(8)	0.0047(8)	0.0003(9)

S2	0.0290(10)	0.0473(12)	0.0272(9)	-0.0023(8)	0.0068(8)	0.0009(9)
S3	0.0244(9)	0.0368(10)	0.0433(10)	0.0001(9)	0.0155(9)	0.0026(9)
C1	0.024(3)	0.025(3)	0.020(3)	0.004(3)	0.004(3)	0.002(3)
C2	0.023(3)	0.021(3)	0.035(4)	0.000(3)	0.008(3)	0.001(3)
C3	0.022(3)	0.022(3)	0.037(4)	-0.001(3)	0.008(3)	-0.004(3)
C4	0.022(3)	0.037(4)	0.036(4)	0.007(3)	0.005(3)	0.004(3)
C5	0.033(4)	0.047(5)	0.040(4)	0.002(3)	0.019(4)	-0.005(4)
C6	0.021(4)	0.031(4)	0.058(5)	-0.003(3)	0.005(4)	-0.004(3)
I1	0.0219(2)	0.0236(2)	0.0304(2)	0.0002(2)	0.0054(2)	0.0018(2)
I2	0.0239(2)	0.0359(3)	0.0448(3)	0.0043(2)	0.0131(2)	-0.0005(2)
I3	0.0268(3)	0.0339(3)	0.0392(3)	0.0010(2)	0.0164(2)	0.0039(2)

Table 5: Bond lengths (Å) and angles (deg) for **24**.

S1-C1	1.683(7)
S1-C4	1.804(7)
S2-C2	1.694(7)
S2-C5	1.807(8)
S3-C3	1.675(7)
S3-C6	1.809(8)
C1-C2	1.370(10)
C1-C3	1.385(9)
C2-C3	1.369(9)
I1-I2	2.8877(6)
I1-I3	2.9525(6)
C1-S1-C4	102.6(3)
C2-S2-C5	101.6(3)
C3-S3-C6	98.9(4)
C2-C1-C3	59.6(5)
C2-C1-S1	154.6(6)
C3-C1-S1	145.7(5)
C3-C2-C1	60.7(5)
C3-C2-S2	144.6(6)
C1-C2-S2	154.6(6)
C2-C3-C1	59.6(5)
C2-C3-S3	150.6(6)
C1-C3-S3	149.8(5)
I2-I1-I3	178.11(2)