Supporting Information

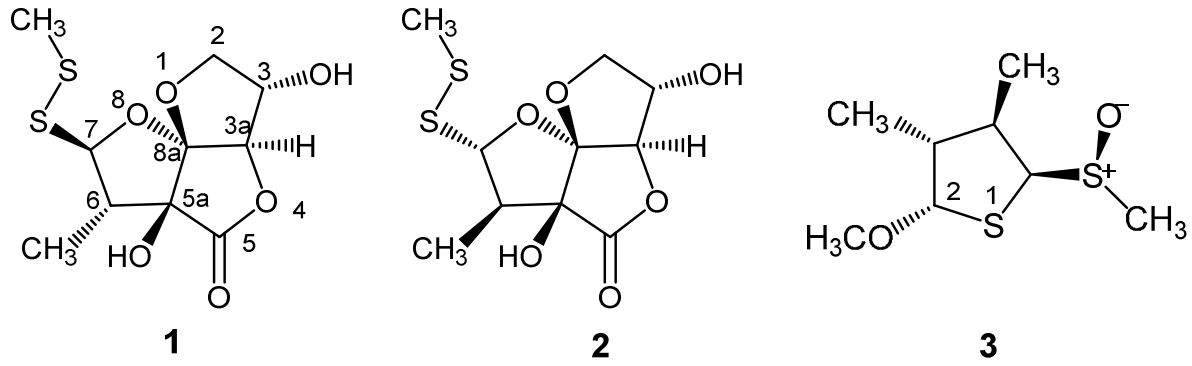
Rare Sulfur-containing Compounds, Kujounins A_1 and A_2 and Alliumsulfoxide A_1, from *Allium fistulosum* '*Kujou*'

Masashi Fukaya,[†] Seikou Nakamura,[†] Ryota Nakagawa, Souichi Nakashima, Masayuki Yamashita, and Hisashi Matsuda*

†Kyoto Pharmaceutical University, Misasagi, Yamashina-ku, Kyoto 607-8412, Japan

∗Corresponding author. Tel.: +81 75 959 4633; Fax: +81 75 595 4768; E-mail address: matsuda@mb.kyoto-phu.ac.jp.

List of Supporting Information



S1. Structure of compounds isolated from the white parts of *Allium fistulosum* 'Kujou'.

S2. Experimental Section.

General Experimental Procedures

The following instruments were used to obtain physical data: specific rotations, a Horiba SEPA-300 digital polarimeter (l = 5 cm); IR spectra, JASCO FT/IR-4600 Fourier Transform Infrared Spectrometer; ESIMS, Agilent Technologies Quadrupole LC/MS 6130; HRESIMS, SHIMADZU LCMS-IT-TOF; ^{1}H-NMR spectra, JEOL JNM-LA 500 (500 MHz) spectrometer; ^{13}C-NMR spectra, JEOL JNM-LA 500 (125 MHz) spectrometer; NOESY spectra, JNM-ECA 600 (600MHz) spectrometer; HPLC, a Shimadzu SPD-20AVP UV-VIS detector. YMC-triart PFP (250 × 4.6 mm i.d. and 250 × 10 mm i.d.) column was used for analytical and preparative purposes. The following experimental materials were used for chromatography: normal-phase silica gel column chromatography, Silica gel BW-200 (Fuji Silysia Chemical, Ltd., 150-350 mesh); reversed-phase silica gel column chromatography, Cosmosil 140C_{18}-OPN (nacalai tesque); TLC, precoated TLC plates with Silica gel 60F254 (Merck, 0.25 mm) (ordinary phase) and Silica gel RP-18 F_{254S} (Merck, 0.25 mm) (reversed phase); reversed-phase HPTLC, precoated TLC plates with Silica gel RP-18 WF_{254S} (Merck, 0.25 mm). Detection was achieved by spraying with 1% Ce(SO4)2– 10% aqueous H_2SO_4 followed by heating.

Plant Material

Kujou negi (*Allium fistulosum* 'Kujou') was cultivated in Kyoto, obtained from KOTO KYOTO Co., Ltd. (Kyoto, Japan) in 2016.

Extraction and Isolation

The white parts of fresh leaves of *Allium fistulosum* 'Kujou' (10.6 kg) were chopped and blended in a mixer with acetone. The mixture was soaked in acetone for 3 days at room temperature. Evaporation of the filtrate under reduced pressure provided acetone extract (676.8 g, 6.38%). The extract was partitioned between EtOAc and H_2O (1:1, v/v) to obtained EtOAc fraction (19.27 g, 0.18%) and aqueous phase. The EtOAc-soluble fraction (19.0g) was subjected to nomal phase silica gel column chromatography [600g, $CHCl_3$-MeOH (1:0→200:1→100:1→50:1→0:1, v/v)] to give eight fractions. Fr. 5 and 6 (total 1.6 g) was subjected to nomal phase silica gel column chromatography [100g, Hexane-Acetone (1:0→6:1→4:1→3:1→2:1→0:1)] to give nine fractions. Fr. 5, 6-5 (663.4 mg) was further separated by reversed phase silica gel column chromatography [30g, MeOH-H_2O (6:4→7:3→8:2→9:1→1:0, v/v)] to give 17 fractions. Fr. 5, 6-5-1 was purified by HPLC {mobile phase: H_2O-MeOH (7:3, v/v) [YMC-triart PFP (250 × 10 mm i.d.)]} to give **1** (14.3 mg), **2** (18.0 mg) and **3** (3.3 mg).

*Kujounin A_1 (**1**)*:

Colorless crystal; $[\alpha]^{25}_D$ +61.1 (MeOH); IR (ATR) 3437, 1785 and 1219 cm^{-1}; for ^1H and ^{13}C NMR data, see table. 1; ESIMS *m/z* 317$[M+Na]^+$ HRESIMS: *m/z* 317.0120 (Calcd for $C_{10}H_{14}O_6S_2$)

*Kujounin A_2 (**2**)*:

Colorless oil; $[\alpha]^{25}_D$ -74.1 (MeOH); IR (ATR) 3436, 1786, 1192 cm^{-1}; for ^1H and ^{13}C NMR data, see table. 2; ESIMS *m/z* 317$[M+Na]^+$ HRESIMS: *m/z* 317.0118 (Calcd for $C_{10}H_{14}O_6S_2$)

*Alliumsulfoxide A_1 (**3**)*:

Colorless crystal; $[\alpha]^{25}_D$ +5.4 (MeOH); IR (ATR) 2961, 1218, 1099 cm^{-1}; for ^1H and ^{13}C NMR data, see table. 3; ESIMS *m/z* 231$[M+Na]^+$ HRESIMS: *m/z* 231.0484 (Calcd for $C_8H_{16}O_2S_2$)

*X-ray crystallography data for Kujounin A_1 (**1**)*:

A colorless block crystal of $C_{10}H_{16}O_7S_2$ having approximate dimensions of 0.300 x 0.250 x 0.250 mm was mounted on a glass fiber. All measurements were made on a Rigaku R-AXIS RAPID diffractometer using graphite monochromated Cu-Kα radiation. The crystal-to-detector distance was 127.40 mm. MW 312.35, T = 296 K, λ = 1.54187 Å, monoclinic, space group P21 (#4), a = 8.4175(2) Å, b = 9.8388(3) Å, c = 9.2758(2) Å, β = 117.5162(14) o, V = 681.31(3) Å 3 , Z = 2, D_{calcd} = 1.522 g/cm ^{3} , μ(Mo Kα) = 38.108 cm $^{-1}$, F(000) = 328.00, $_{crystal\ size}$. No. of reflections measured: total, 7148; unique, 2358 (R_{int} = 0.0815); Parsons quotients (Flack x parameter). Refinement method: full-matrix least-squares on F ^{2}, goodness of fit indicator 1.152, flack parameter -0.00(2), final R1[I > 2.00σ(I)] = 0.0462, maximum peak in final Diff. Map and minimum peak 0.22 e $^-$ /Å ^{3} and –0.29 e $^-$ /Å ^{3} , Max Shift/Error in Final $_{Cycle}$ 0.002.

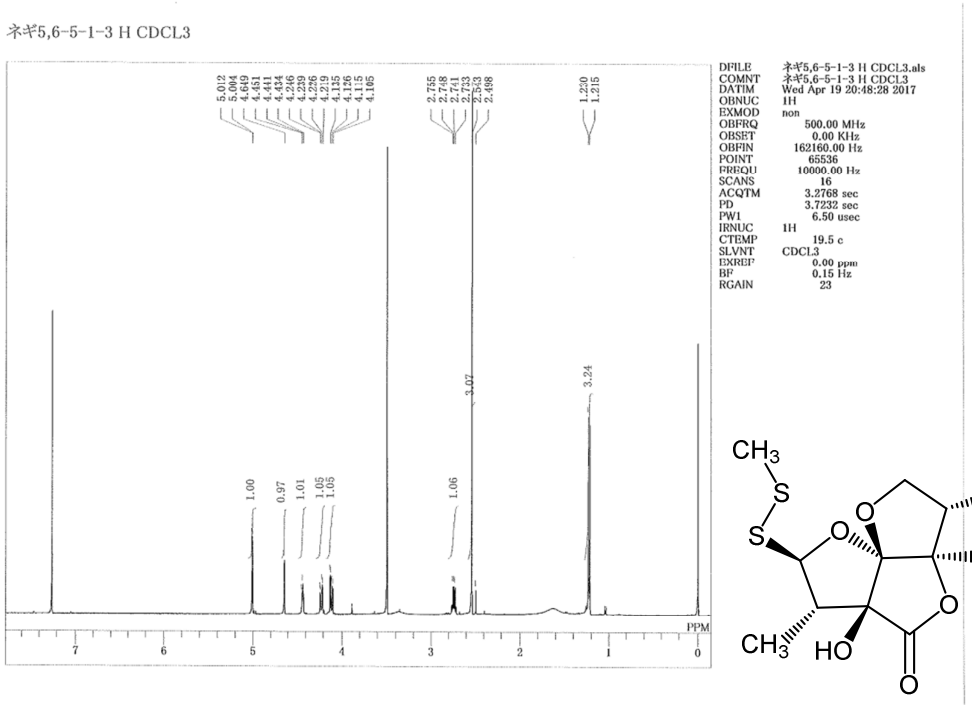
*X-ray crystallography data for Alliumsulfoxide A_1 (**3**)*

A colorless block crystal of $C_8H_{16}O_2S_2$ having approximate dimensions of 0.300 x 0.250 x 0.150 mm was mounted on a glass fiber. All measurements were made on a Rigaku R-AXIS RAPID diffractometer using graphite monochromated Cu-Kα radiation. The crystal-to-detector distance was 127.40 mm. MW 208.33 T = 296 K, λ = 1.54187 Å, monoclinic, space group P21 (#4), a = 16.4838(7) Å, b = 7.4579(3) Å, c = 9.0767(4) Å, β = 102.915(7) o, V =1087.61(9) Å 3 , Z = 4, D_{calcd} = 1.272 g/cm ^{3} , μ(CuKα) = 41.464 cm $^{-1}$, F(000) = 448.00, $_{crystal\ size}$. No. of reflections measured: total, 5844; unique, 1793 (R_{int} = 0.0580); Parsons quotients (Flack x parameter). Refinement method: full-matrix least-squares on F ^{2}, goodness of fit indicator 1.151, flack parameter0.064(17), final R1[I > 2.00σ(I)] = 0.0343, maximum peak in final Diff. Map and minimum peak 0.22 e $^-$ /Å ^{3} and –0.23 e $^-$ /Å ^{3} , Max Shift/Error in Final $_{Cycle}$ 0.001.

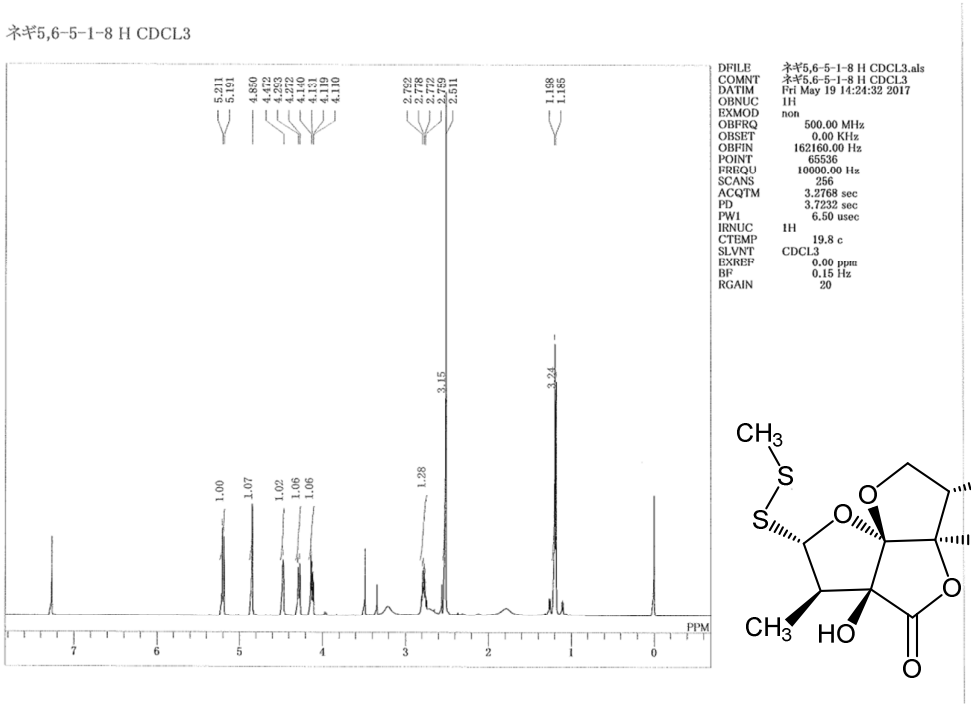
*HPLC Profile Comparison of alliumsulfoxide A_1 (**3**)*

In this study, we have isolated alliumsulfoxide A_1 (**3**) including a methyl ether, from the acetone extract of fresh white parts of *A. fistulosum* 'Kujou'. When we subjected normal and reversed phase column chromatography, we used methanol. In order to establish whether this methyl ether is artifact generated during the separation procedure, we examined the HPLC profile of the EtOAc soluble fractions from the acetone extract. The EtOAc soluble fraction was prepared without using methanol. A portion of the EtOAc soluble fraction and authentic sample were dissolved in MeCN and subjected to reversed-phase HPLC [column: YMC triart C18, 250 × 4.6 mm i.d.; mobile phase A: H_2O, B: MeCN, gradient: mobile phase A–B (0.0-60.0 min: linear gradient with 90–10→70–30, 60-70.0min: linear gradient with 70–30→0–100); detection: UV (210 nm); flow rate: 1.0 ml/min; column temperature: 25°C]. Compound **3** was detected in the EtOAc fraction by comparing the retention time with standard sample (t_R **3**: 28.1 min). Based on these results, the methyl ether derivative, which were isolated from acetone extract of fresh white parts of *A. fistulosum* 'Kujou', suggested genuine natural product.

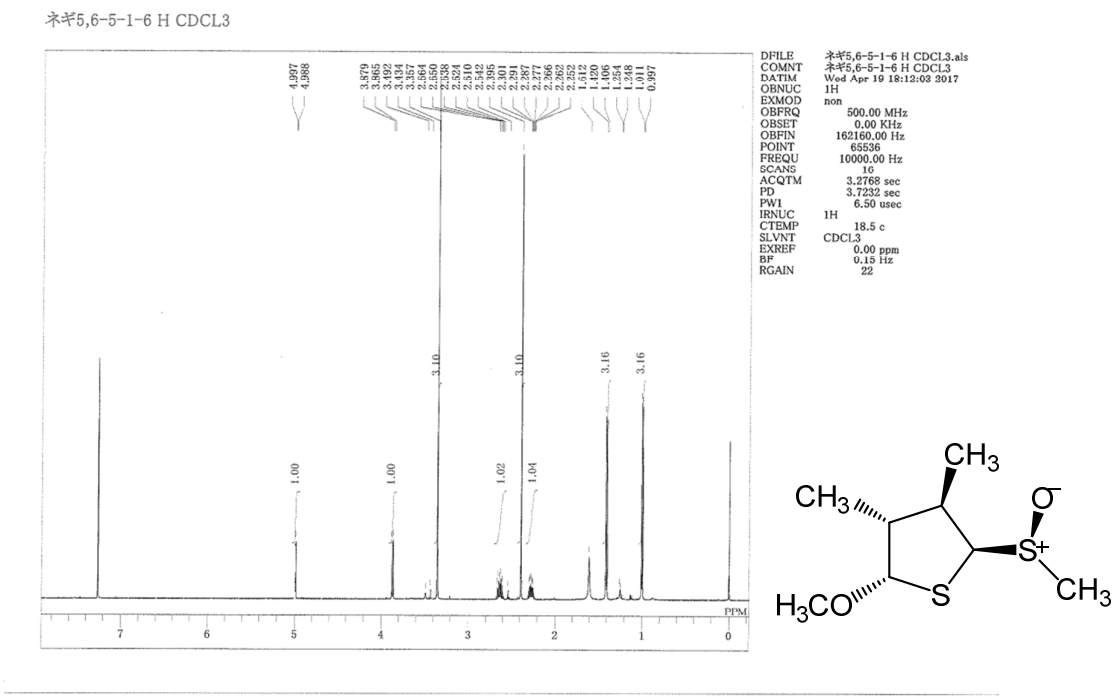
S3. ^{1}H NMR spectra of compounds 1—3.



^{1}H-NMR spectrum of compound **1**. Measured in $CDCl_3$.

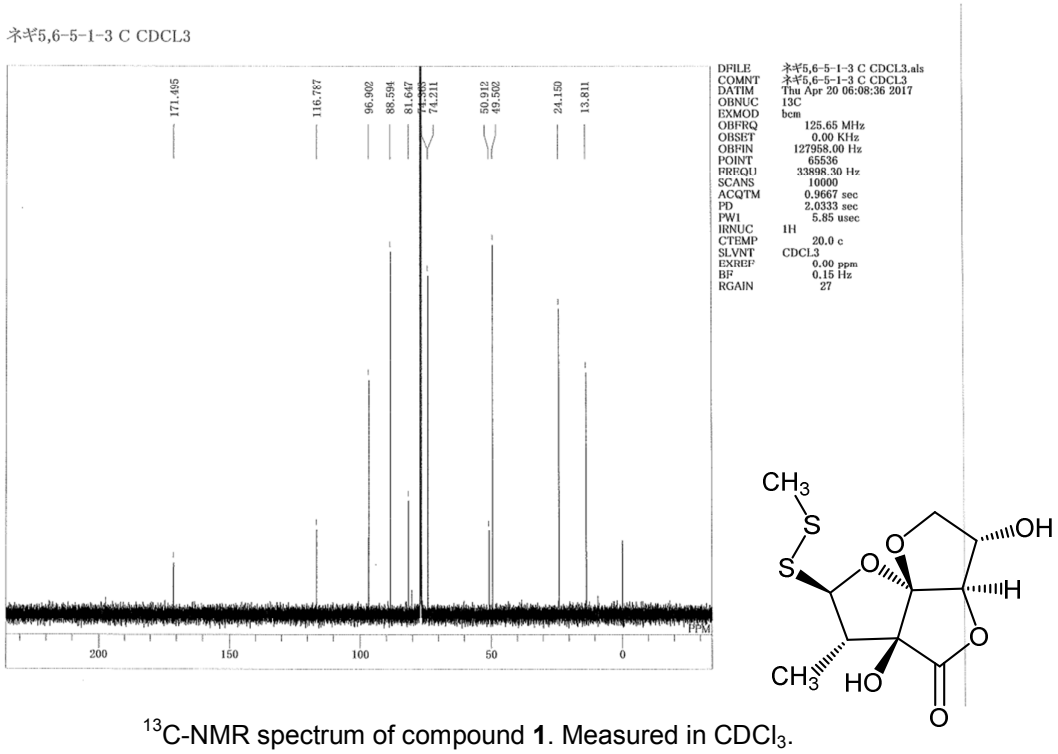


^{1}H-NMR spectrum of compound **2**. Measured in $CDCl_3$.

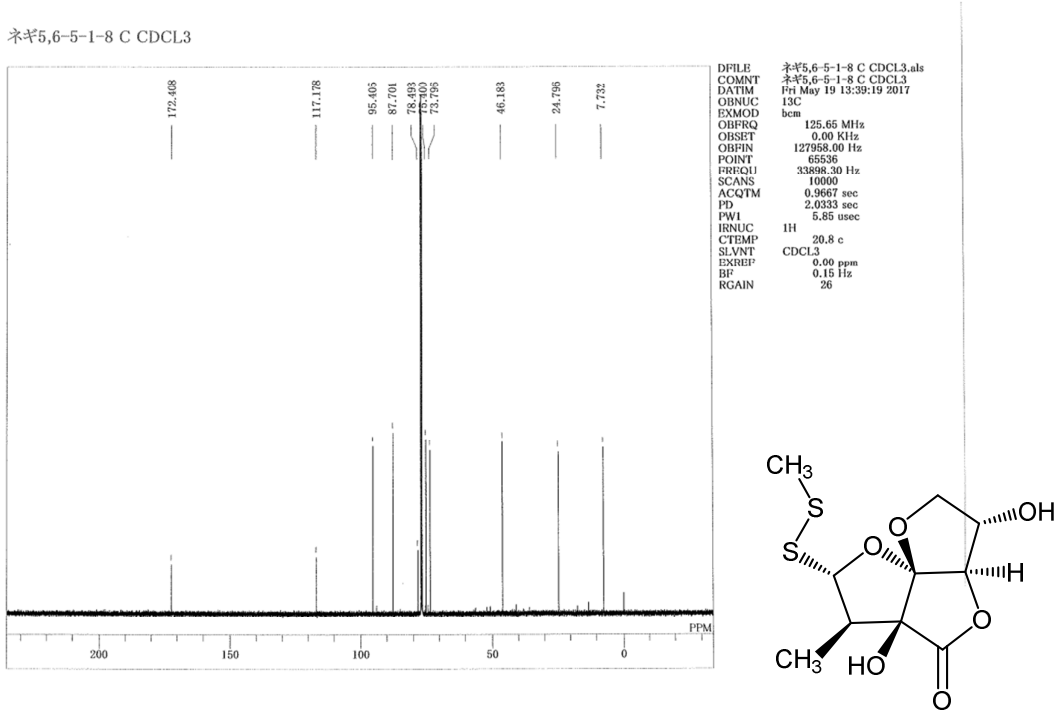


^{1}H-NMR spectrum of compound **3**. Measured in $CDCl_3$.

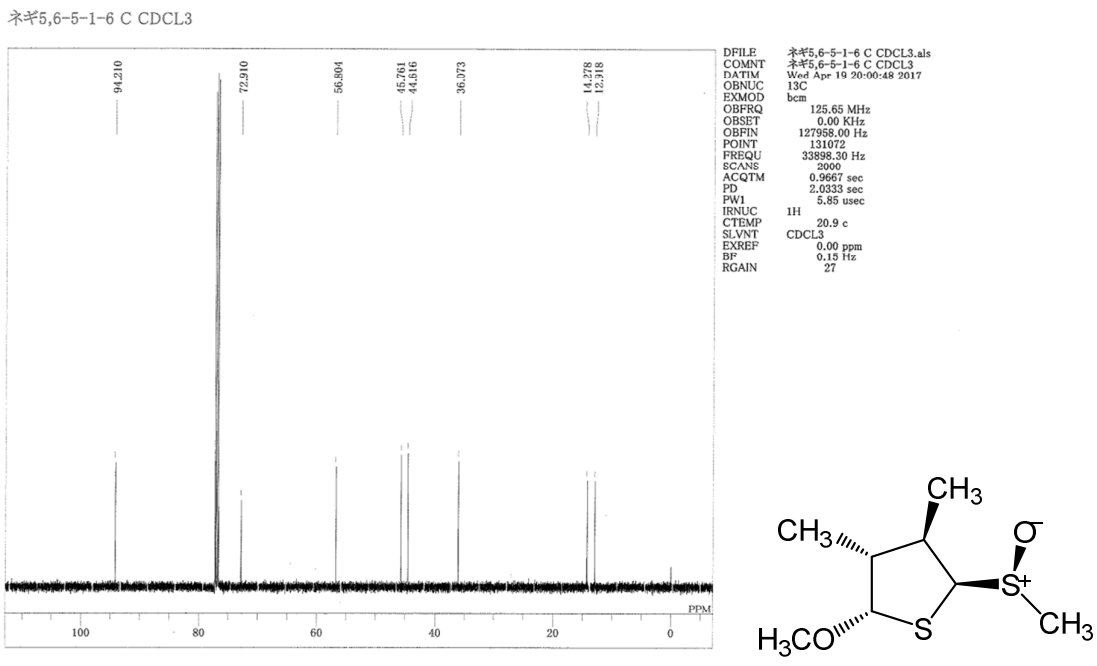
S4. ^{13}C NMR spectra of compounds 1—3.



^{13}C-NMR spectrum of compound **1**. Measured in $CDCl_3$.

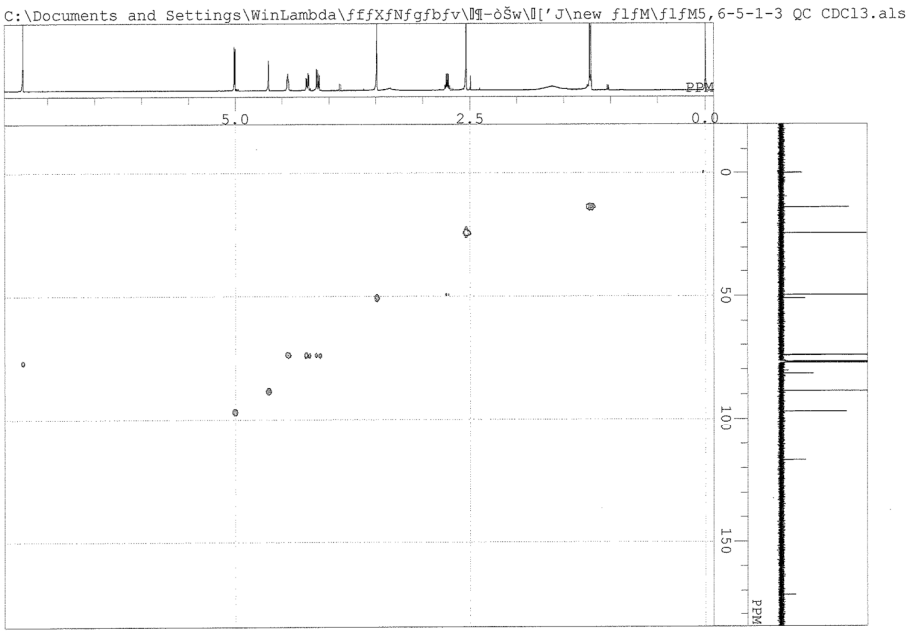


^{13}C-NMR spectrum of compound **2**. Measured in $CDCl_3$.

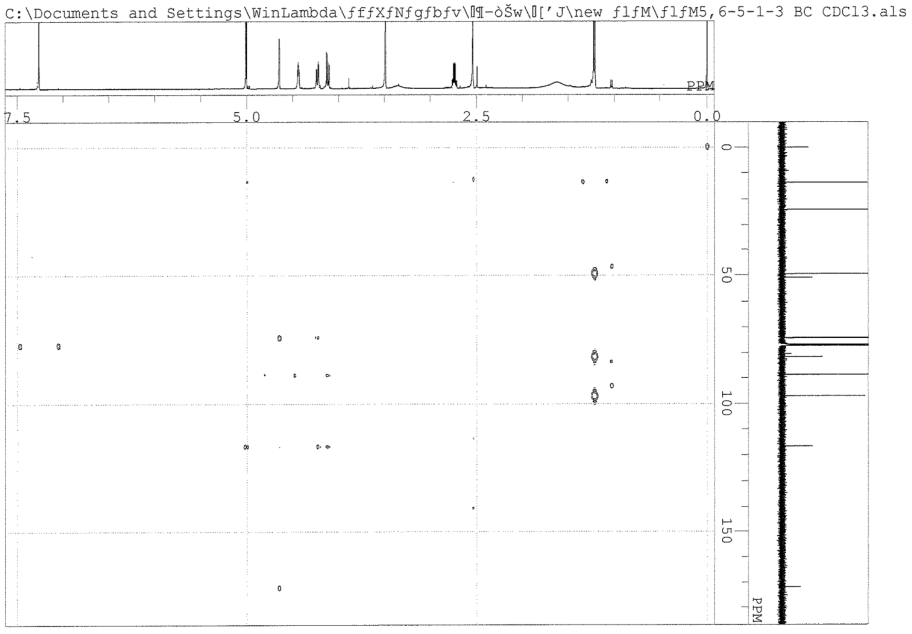


^{13}C-NMR spectrum of compound **3**. Measured in $CDCl_3$.

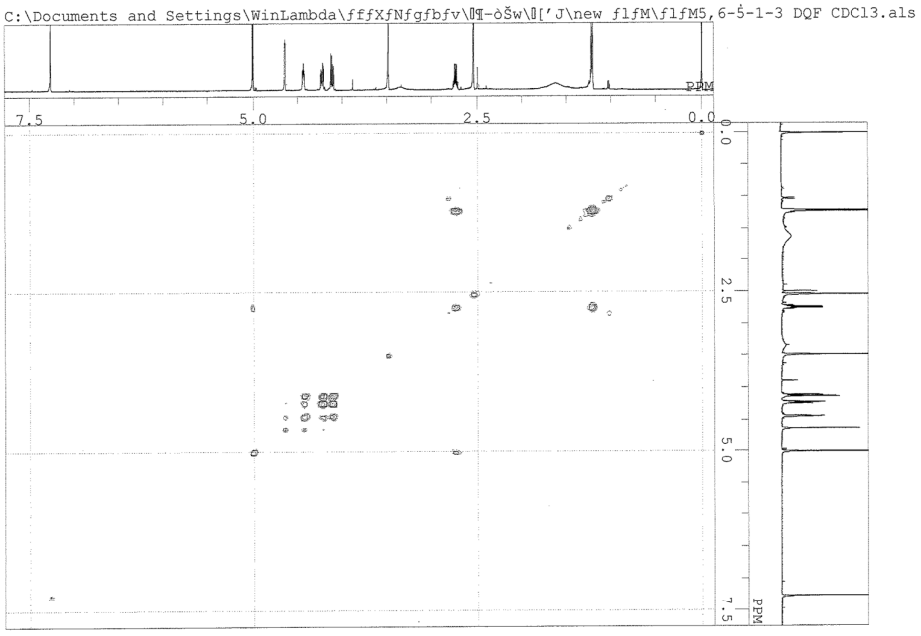
S5. 2D NMR spectra of compounds 1—3.



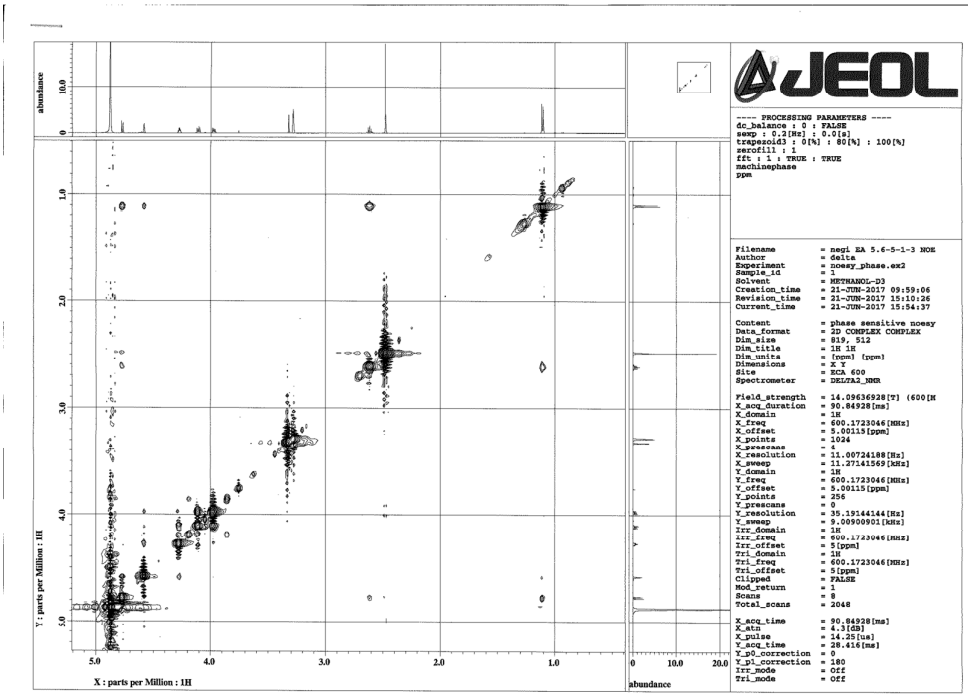
HMQC spectrum of compound **1**. Measured in $CDCl_3$.



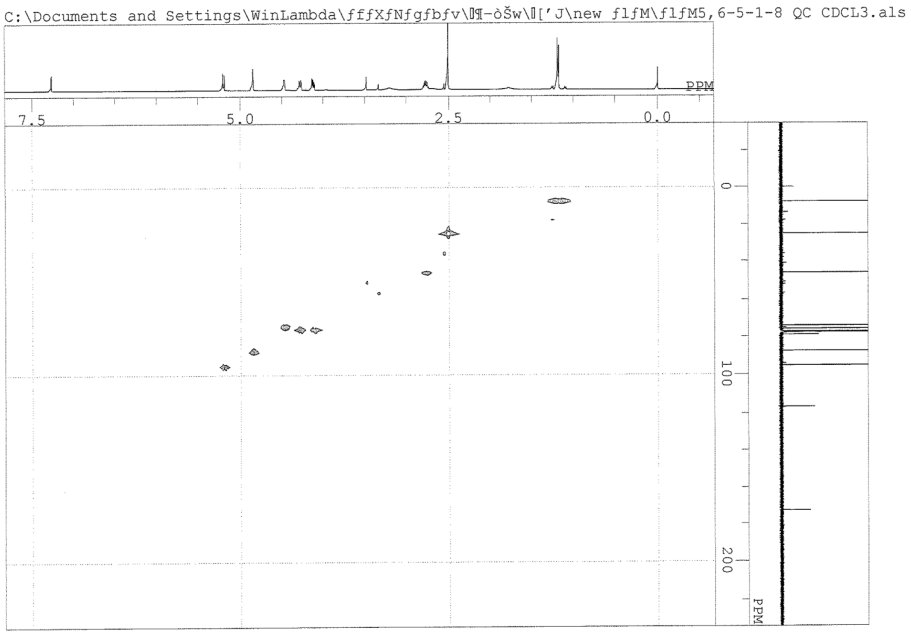
HMBC spectrum of compound **1**. Measured in $CDCl_3$.



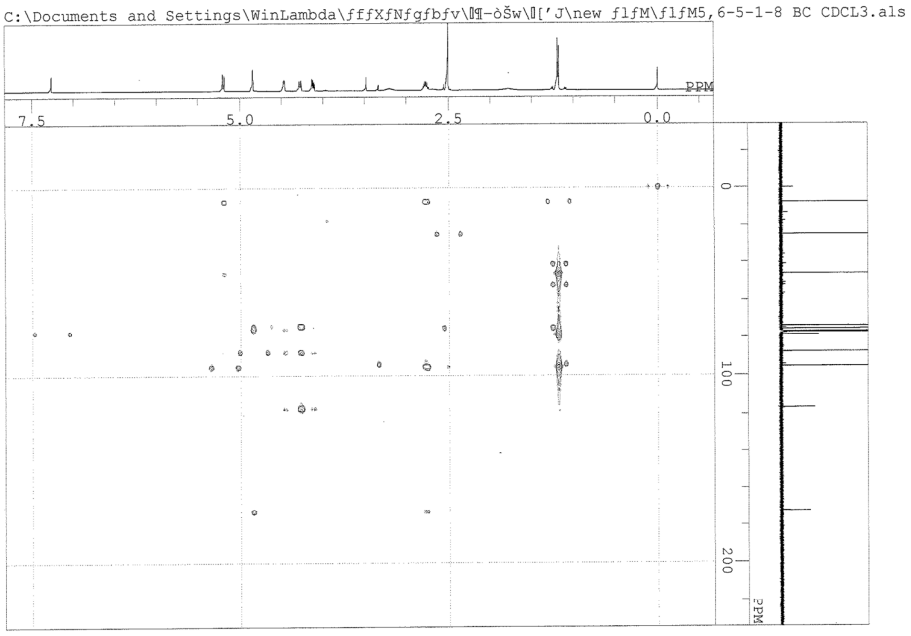
DQF-COSY spectrum of compound **1**. Measured in $CDCl_3$.



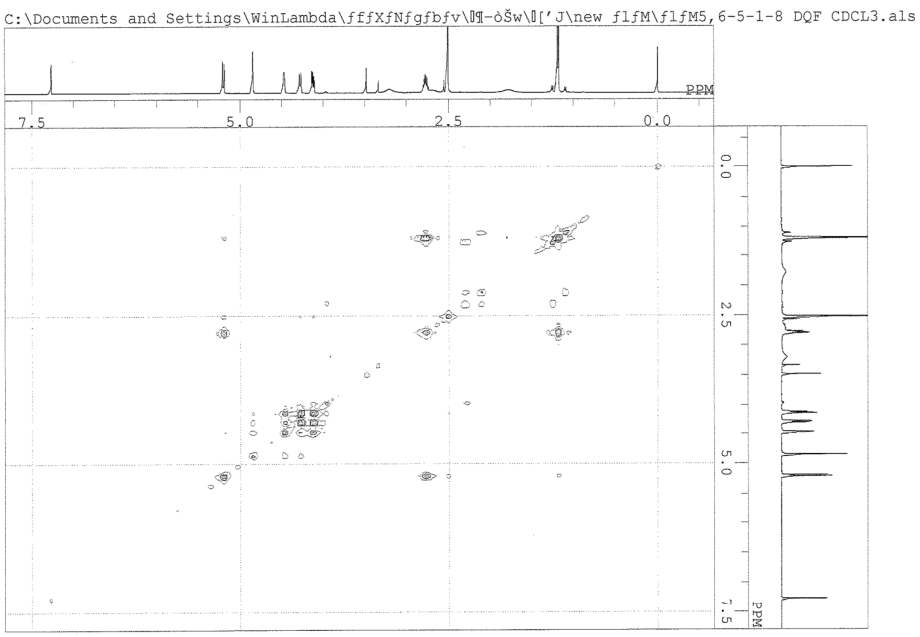
NOESY spectrum of compound **1**. Measured in $CDCl_3$.



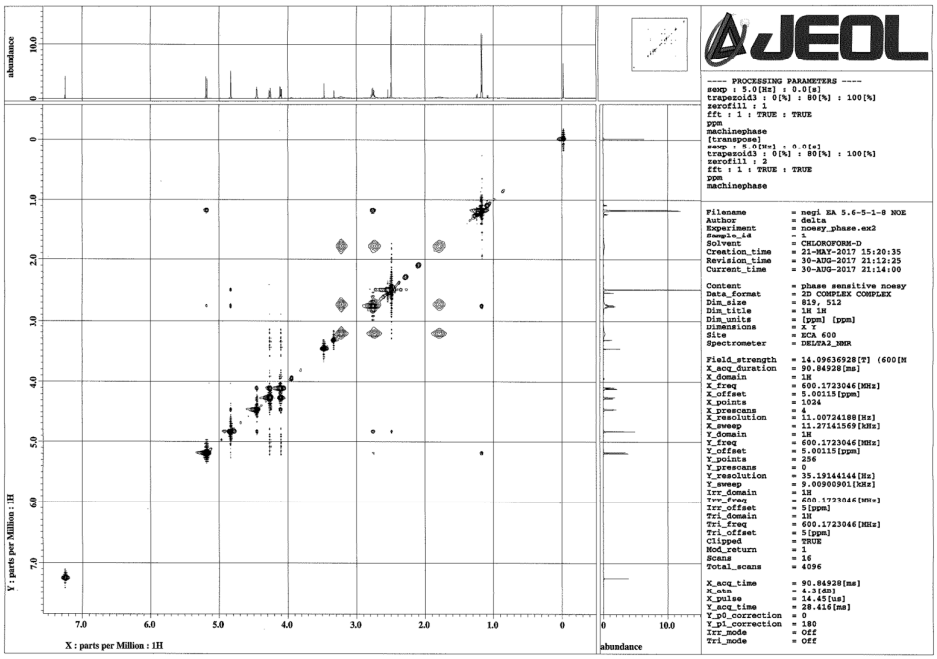
HMQC spectrum of compound **2**. Measured in $CDCl_3$.



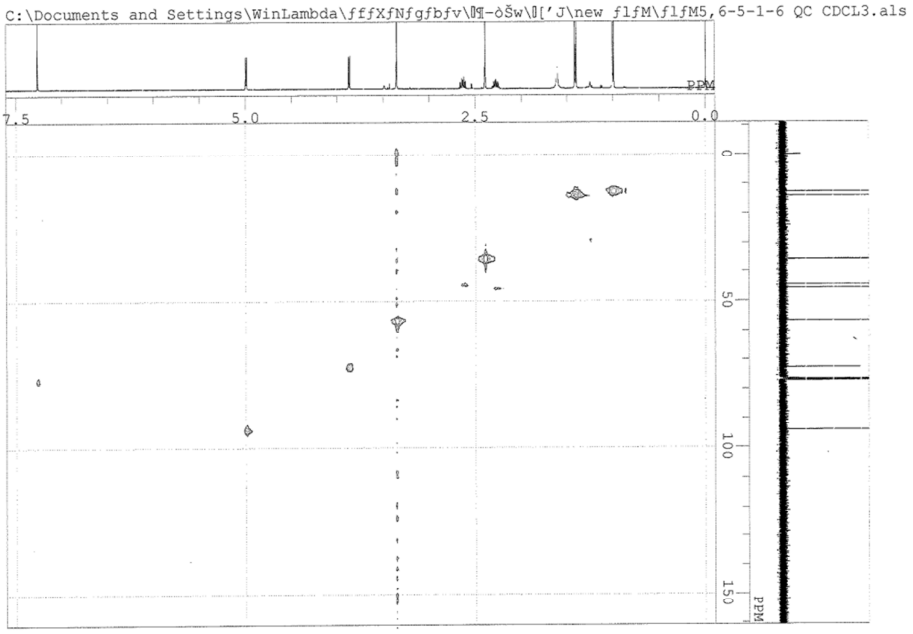
HMBC spectrum of compound **2**. Measured in $CDCl_3$.



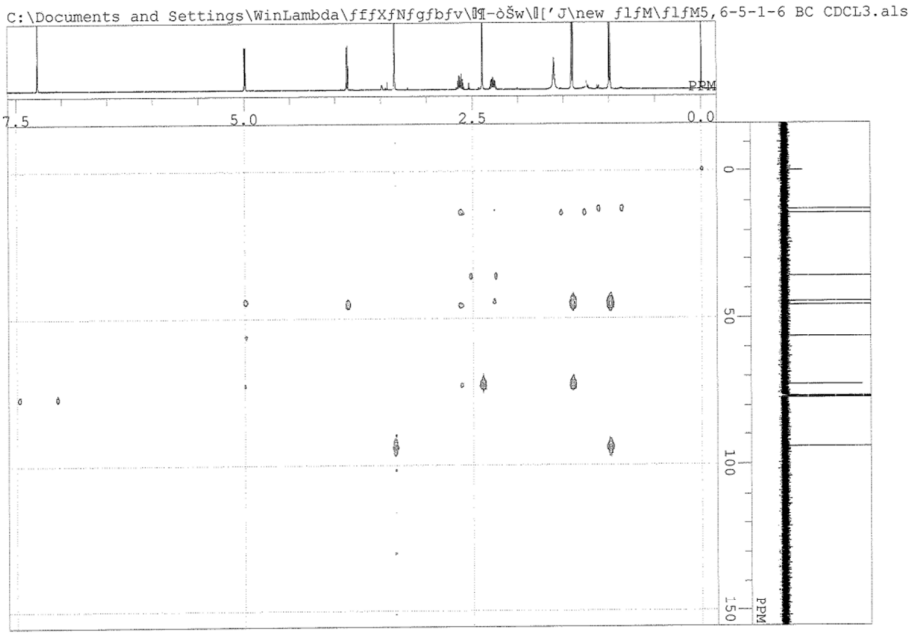
DQF-COSY spectrum of compound **2**. Measured in $CDCl_3$.



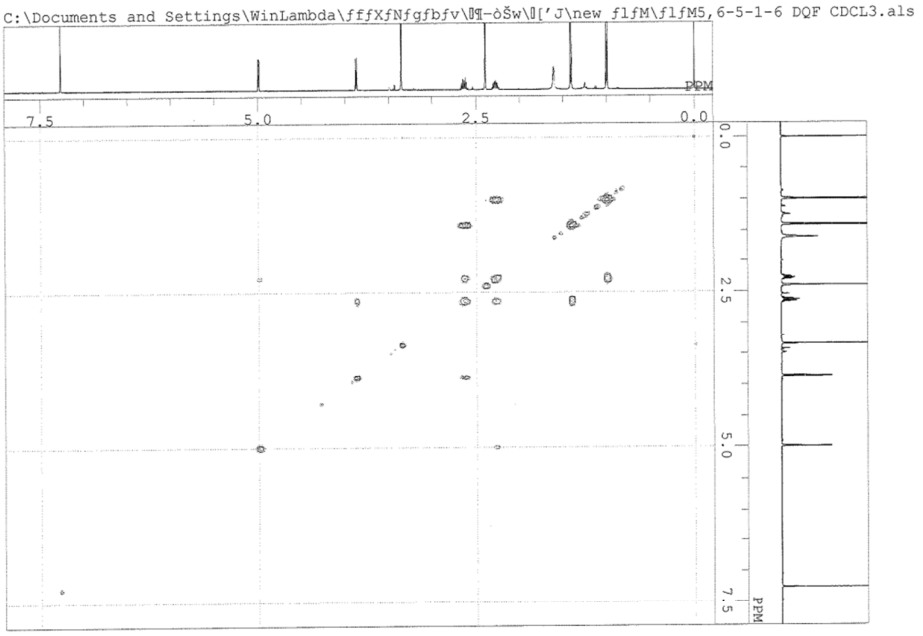
NOESY spectrum of compound **2**. Measured in $CDCl_3$.



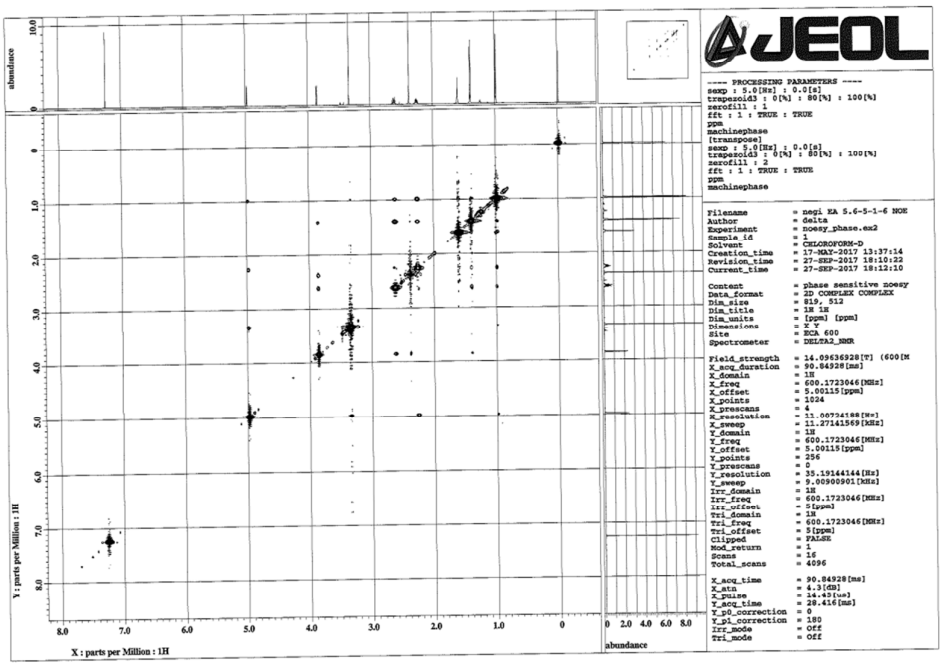
HMQC spectrum of compound **3**. Measured in $CDCl_3$.



HMBC spectrum of compound **3**. Measured in $CDCl_3$.



DQF-COSY spectrum of compound **3**. Measured in $CDCl_3$.



NOESY spectrum of compound **3**. Measured in $CDCl_3$.

S6. HRESIMS spectra of compounds 1—3.

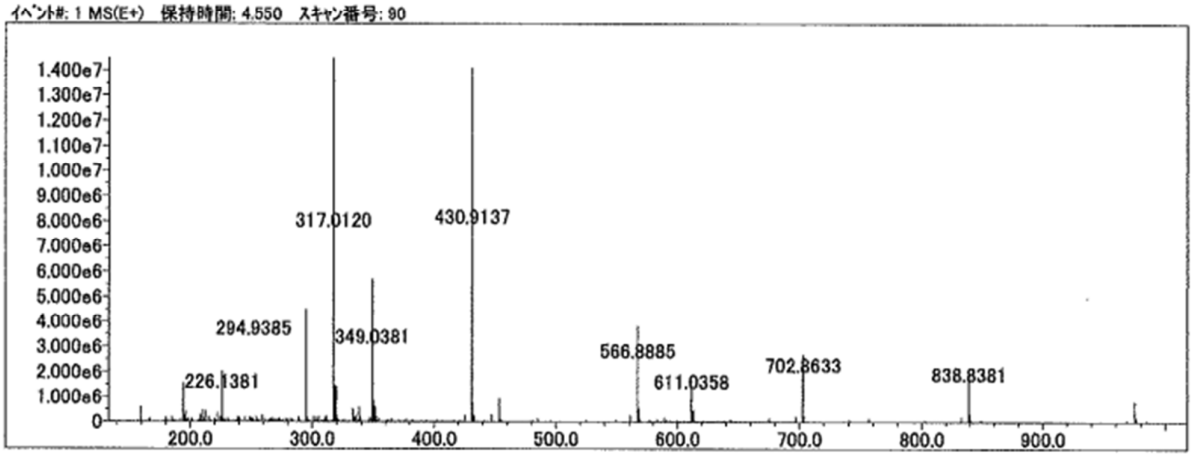
HRESIMS spectrum of compound 1

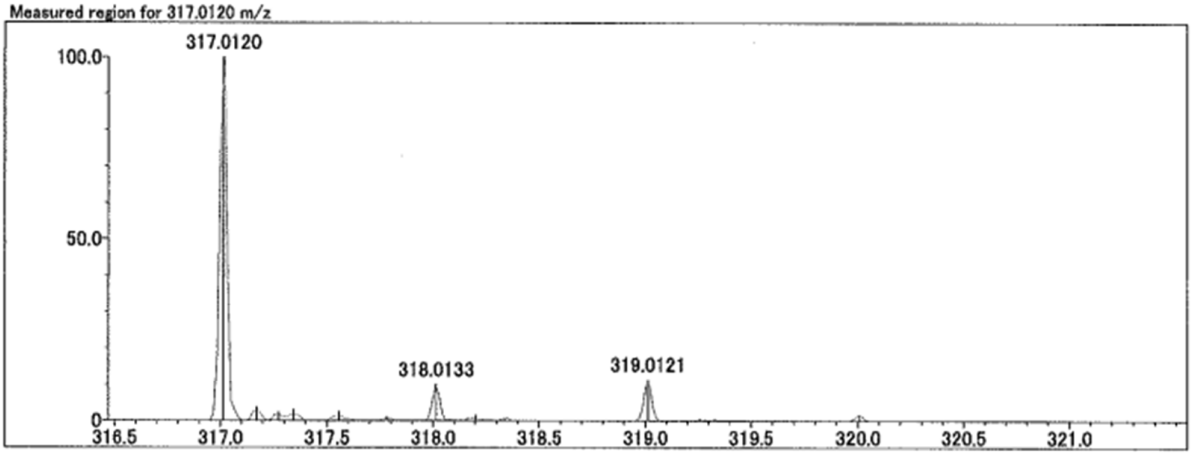
Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Use Adduct
H	1	10	20	N	3	0	0	Si	4	0	0	Br	1	0	0	Na
B	3	0	0	O	2	1	10	S	2	1	5	I	3	0	0	
C	4	5	15	F	1	0	0	Cl	1	0	0					

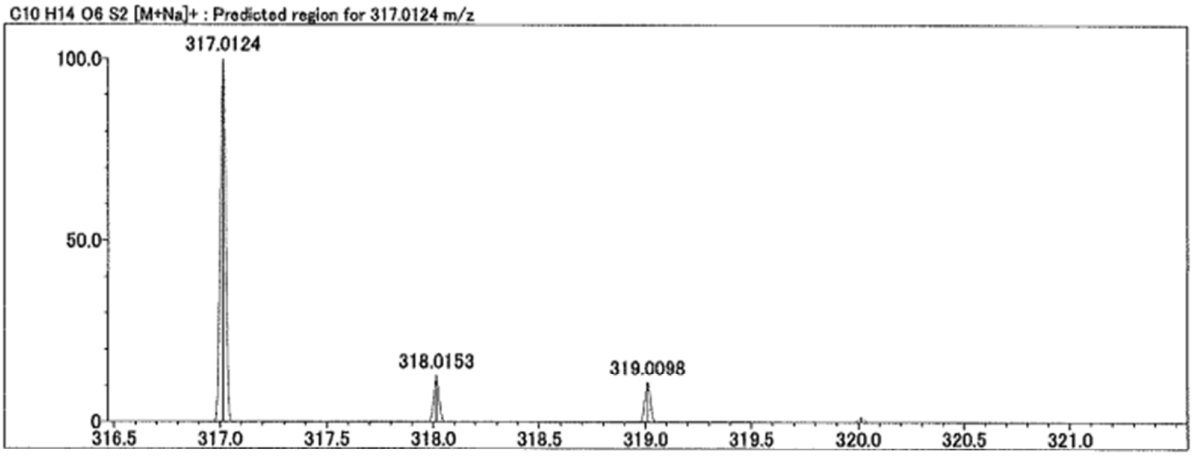
Error Margin (mDa): 3.0
HC Ratio: unlimited
Max Isotopes: all
MSn Iso RI (%): 75.00

DBE Range: not fixed
Apply N Rule: yes
Isotope RI (%): 1.00
MSn Logic Mode: AND

Electron Ions: both
Use MSn Info: no
Isotope Res: 10000
Max Results: 50







Rank	Score	Formula (M)	Ion	Meas. m/z	Pred. m/z	Df. (mDa)	Df. (ppm)	Iso	DBE
1	74.55	C10 H14 O6 S2	[M+Na]+	317.0120	317.0124	-0.4	-1.26	75.03	4.0

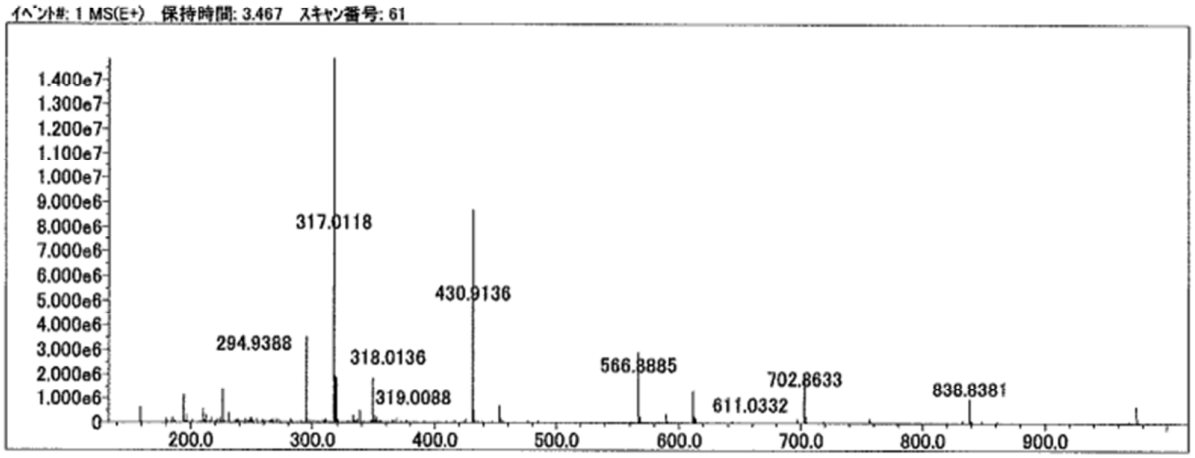
HRESIMS spectrum of compound 2

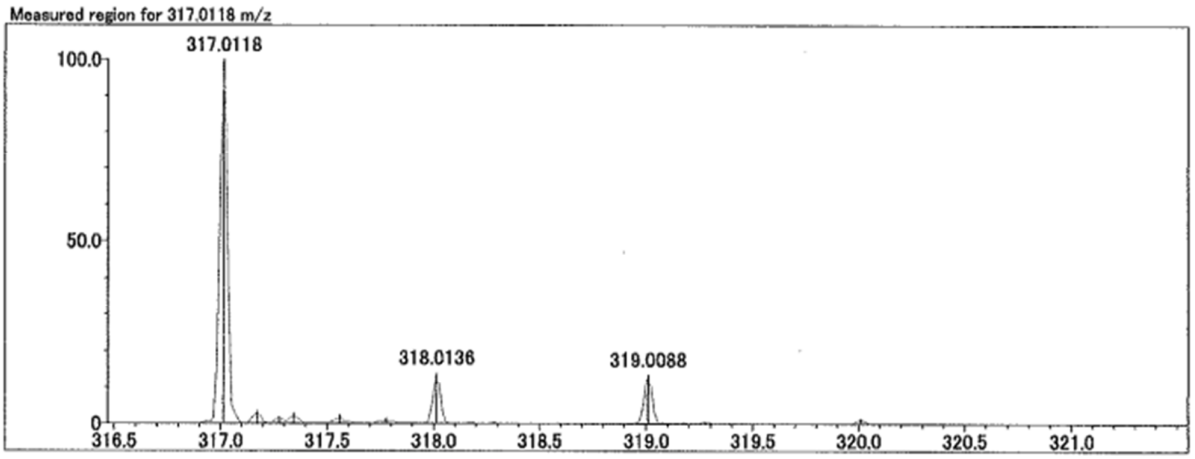
Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Use Adduct
H	1	10	20	N	3	0	0	Si	4	0	0	Br	1	0	0	Na
B	3	0	0	O	2	1	10	S	2	1	5	I	3	0	0	
C	4	5	15	F	1	0	0	Cl	1	0	0					

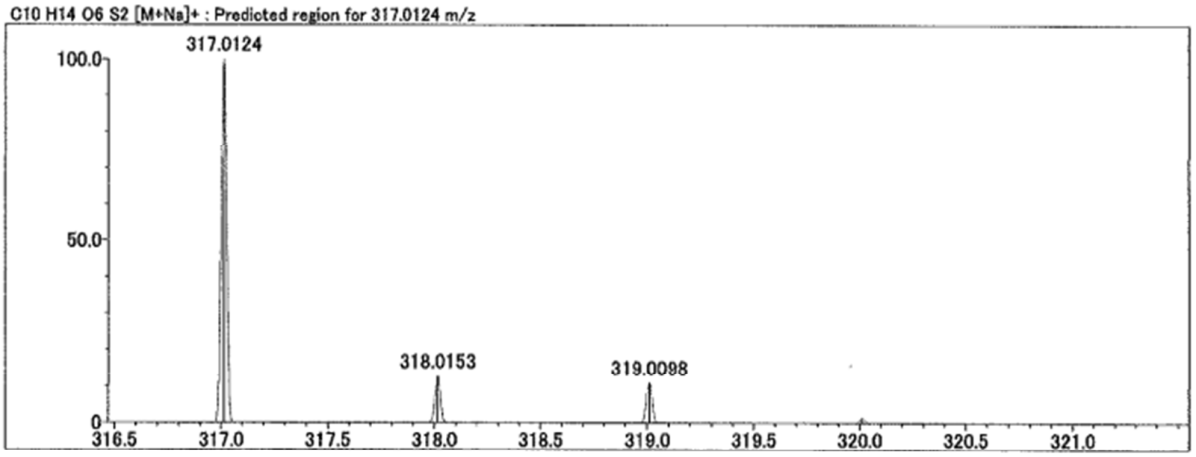
Error Margin (mDa): 3.0
HC Ratio: unlimited
Max Isotopes: all
MSn Iso RI (%): 75.00

DBE Range: not fixed
Apply N Rule: yes
Isotope RI (%): 1.00
MSn Logic Mode: AND

Electron Ions: both
Use MSn Info: no
Isotope Res: 10000
Max Results: 50







Rank	Score	Formula (M)	Ion	Meas. m/z	Pred. m/z	Df. (mDa)	Df. (ppm)	Iso	DBE
1	88.25	C10 H14 O6 S2	[M+Na]+	317.0118	317.0124	-0.6	-1.89	90.26	4.0

HRESIMS spectrum of compound 3

Data File: E:¥生薬¥negi EA 5,6-5-1-6-HR1.lcd

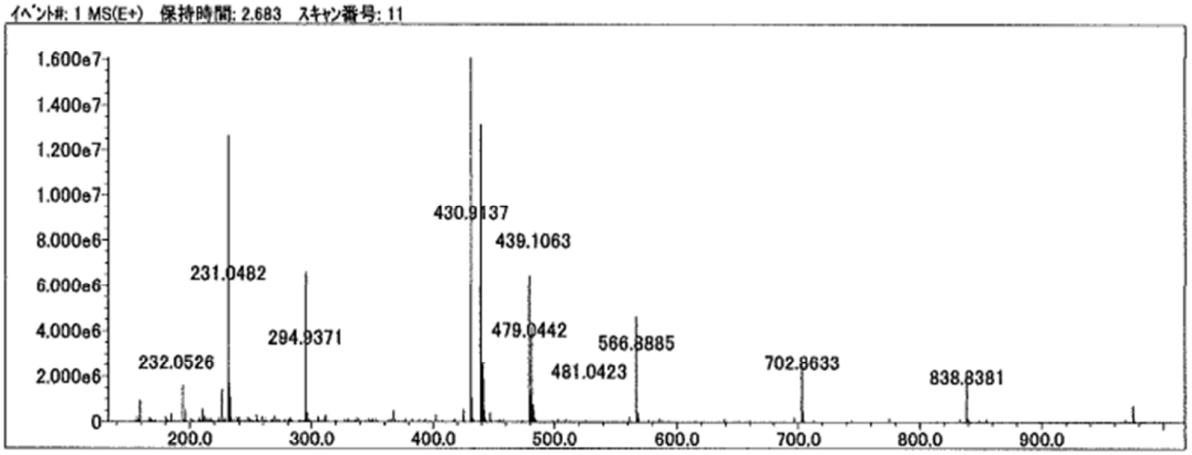
Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Elmt	Val.	Min	Max	Use Adduct
H	1	10	20	N	3	0	0	Si	4	0	0	Br	1	0	0	Na
B	3	0	0	O	2	1	3	S	2	1	3	I	3	0	0	
C	4	5	15	F	1	0	0	Cl	1	0	0					

Error Margin (mDa): 3.0
HC Ratio: unlimited
Max Isotopes: all
MSn Iso RI (%): 75.00

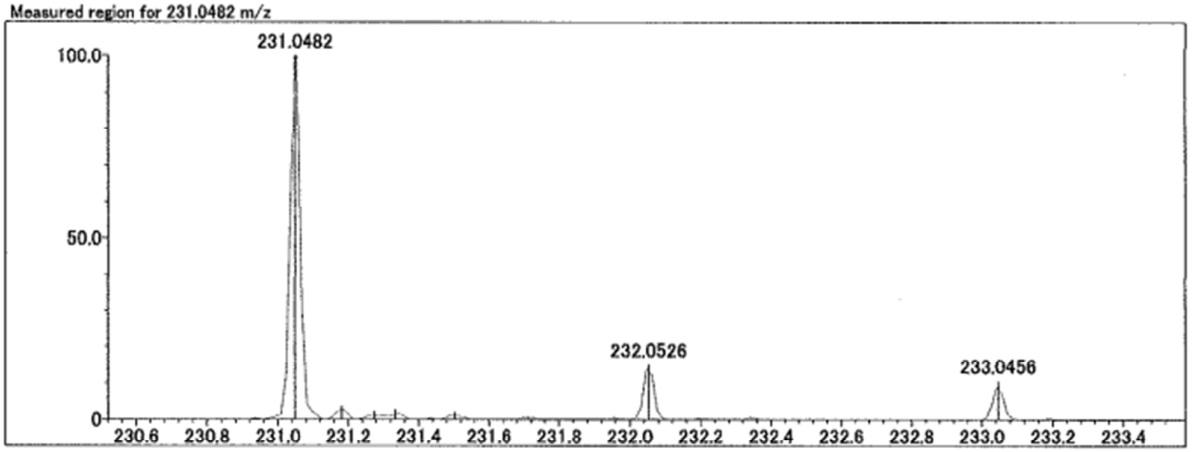
DBE Range: not fixed
Apply N Rule: yes
Isotope RI (%): 1.00
MSn Logic Mode: AND

Electron Ions: both
Use MSn Info: no
Isotope Res: 10000
Max Results: 50

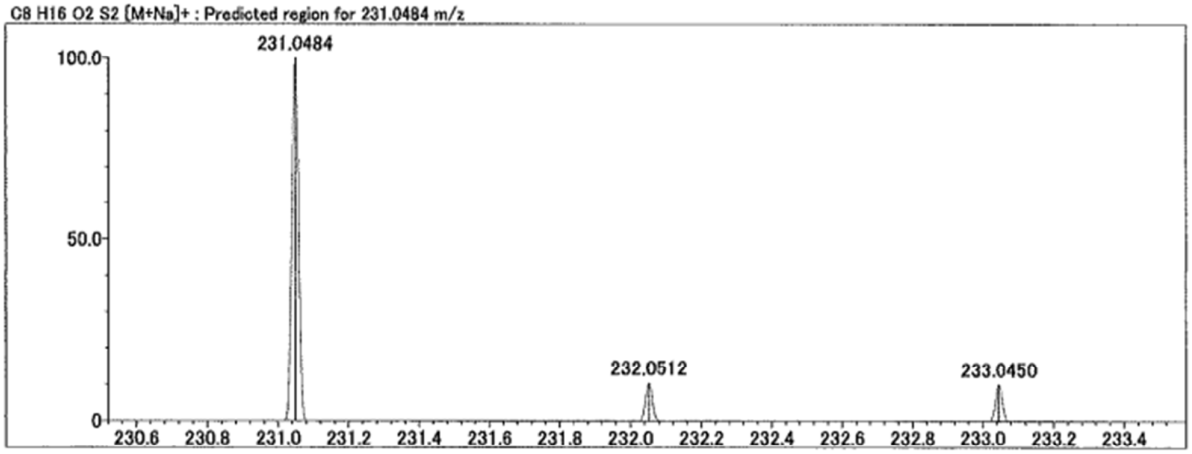
イベント#: 1 MS(E+) 保持時間: 2.683 スキャン番号: 11



Measured region for 231.0482 m/z



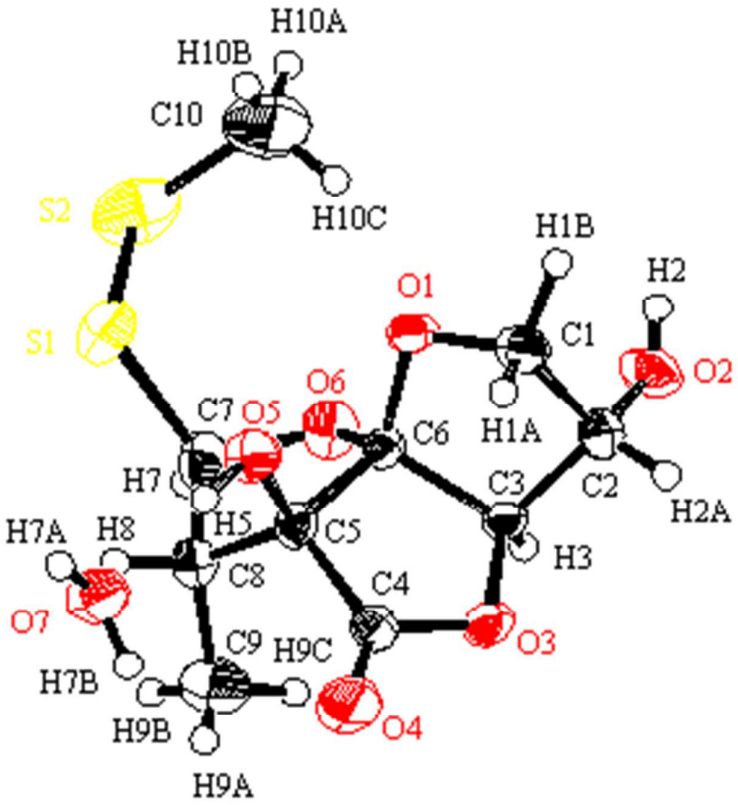
C8 H16 O2 S2 [M+Na]+ : Predicted region for 231.0484 m/z



Rank	Score	Formula (M)	Ion	Meas. m/z	Pred. m/z	Df. (mDa)	Df. (ppm)	Iso	DBE
1	79.48	C8 H16 O2 S2	[M+Na]+	231.0482	231.0484	-0.2	-0.87	79.48	1.0

S7. X-ray experimental details of compound 1 and 3.

X-ray experimental details of 1.



ORTEP drawing of **1**

Experimental

Data Collection

A colorless block crystal of $C_{10}H_{16}O_7S_2$ having approximate dimensions of 0.300 x 0.250 x 0.250 mm was mounted on a glass fiber. All measurements were made on a Rigaku R-AXIS RAPID diffractometer using graphite monochromated Cu-Kα radiation.

The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 8.4175(2) Å

b = 9.8388(3) Å β = 117.5162(14)^o

c = 9.2758(2) Å

V = 681.31(3) Å^3

For Z = 2 and F.W. = 312.35, the calculated density is 1.522 g/cm^3. Based on the reflection conditions of:

0k0: k = 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$P2_1$ (#4)

The data were collected at a temperature of 23 $\pm$ 1^{o}C to a maximum 2θ value of 136.3^o. A total of 30 oscillation images were collected. A sweep of data was done using ω scans from 80.0 to 260.0^o in 30.00^o step, at χ=54.0^o and ϕ = 0.0^o. The exposure rate was 60.0 [sec./^{o}]. A second sweep was performed using ω scans from 80.0 to 260.0^o in 30.00^o step, at χ=54.0^o and ϕ = 90.0^o. The exposure rate was 60.0 [sec./^{o}]. Another sweep was

performed using ω scans from 80.0 to 260.0^{o} in 30.00^{o} step, at χ=54.0^{o} and ϕ = 180.0^{o}. The exposure rate was 60.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^{o} in 30.00^{o} step, at χ=54.0^{o} and ϕ = 270.0^{o}. The exposure rate was 60.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^{o} in 30.00^{o} step, at χ=0.0^{o} and ϕ = 0.0^{o}. The exposure rate was 60.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 7148 reflections were collected, where 2358 were unique (R_{int} = 0.0815); equivalent reflections were merged.

The linear absorption coefficient, μ, for Cu-Kα radiation is 38.108 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.252 to 0.386. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction[1] was applied (coefficient = 0.005460).

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^{2} was based on 2358 observed reflections and 225 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0462$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1116$$

The goodness of fit[4] was 1.15. Unit weights were used. The maximum and

minimum peaks on the final difference Fourier map corresponded to 0.22 and -0.29 $e^-/Å^3$, respectively. The final Flack parameter [5] was -0.00(2), indicating that the present absolute structure is correct. [6]

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [7]. Anomalous dispersion effects were included in Fcalc[8]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[9]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[10]. All calculations were performed using the CrystalStructure[11] crystallographic software package except for refinement, which was performed using SHELXL Version 2016/6[12].

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{10}H_{16}O_7S_2$
Formula Weight	312.35
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.300 X 0.250 X 0.250 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 8.4175(2) Å b = 9.8388(3) Å c = 9.2758(2) Å β = 117.5162(14) ^{o} V = 681.31(3) Å^3
Space Group	$P2_1$ (#4)
Z value	2
D_{calc}	1.522 g/cm^3
F_{000}	328.00
μ(CuKα)	38.108 cm^{-1}

B. Intensity Measurements

Diffractometer	R-AXIS RAPID
Radiation	CuKα (λ = 1.54187 Å) graphite monochromated
Voltage, Current	40kV, 100mA
Temperature	23.0^{o}C
Detector Aperture	460.0 x 256.0 mm
Data Images	30 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	60.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=90.0)	80.0 - 260.0^{o}
Exposure Rate	60.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=180.0)	80.0 - 260.0^{o}
Exposure Rate	60.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=270.0)	80.0 - 260.0^{o}
Exposure Rate	60.0 sec./^{o}
ω oscillation Range (χ=0.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	60.0 sec./^{o}

Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	136.3^{o}
No. of Reflections Measured	Total: 7148 Unique: 2358 (R_{int} = 0.0815) Parsons quotients (Flack x parameter): 552
Corrections	Lorentz-polarization Absorption (trans. factors: 0.252 - 0.386) Secondary Extinction (coefficient: 5.46000e-003)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXT Version 2014/5)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w (Fo^2 - Fc^2)^2
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + (0.0000 · P)^2 + 0.4813 · P] where P = (Max(Fo^2,0) + 2Fc^2)/3
$2\theta_{max}$ cutoff	136.3^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2358
No. Variables	225
Reflection/Parameter Ratio	10.48
Residuals: R1 (I>2.00σ(I))	0.0462
Residuals: R (All reflections)	0.0695
Residuals: wR2 (All reflections)	0.1116
Goodness of Fit Indicator	1.152
Flack parameter (Parsons' quotients = 552)	-0.00(2)
Max Shift/Error in Final Cycle	0.002
Maximum peak in Final Diff. Map	0.22 e^-/Å^3
Minimum peak in Final Diff. Map	-0.29 e^-/Å^3

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
S1	0.9608(2)	0.5885(2)	0.7319(2)	3.81(4)
S2	1.0662(3)	0.4535(3)	0.9176(2)	5.47(6)
O1	0.5714(5)	0.3940(4)	0.4784(4)	2.38(7)
O2	0.5155(7)	0.1175(4)	0.3558(6)	3.53(10)
O3	0.5611(6)	0.3876(4)	0.1178(5)	3.02(8)
O4	0.5513(7)	0.6042(5)	0.0482(5)	4.37(11)
O5	0.5886(6)	0.6363(4)	0.3776(5)	2.76(8)
O6	0.8630(5)	0.3738(4)	0.5243(5)	2.88(8)
O7	0.6051(7)	0.8772(5)	0.2542(6)	3.47(9)
C1	0.4019(8)	0.3452(7)	0.3541(8)	2.71(11)
C2	0.4520(9)	0.2386(6)	0.2645(8)	2.55(11)
C3	0.6147(8)	0.3041(6)	0.2615(7)	2.09(10)
C4	0.5958(9)	0.5219(6)	0.1542(7)	2.61(11)
C5	0.6886(8)	0.5444(5)	0.3365(7)	2.10(10)
C6	0.6885(7)	0.4038(6)	0.4078(6)	1.88(9)
C7	0.9688(8)	0.4945(7)	0.5651(8)	2.97(12)
C8	0.8897(8)	0.5808(8)	0.4095(7)	2.78(10)
C9	0.9788(13)	0.5423(11)	0.3050(12)	4.73(19)
C10	0.8744(13)	0.3589(10)	0.8979(10)	6.2(2)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1A	0.34118	0.41790	0.27955	1.487
H1B	0.33812	0.30751	0.41725	2.782
H2	0.47490	0.11242	0.40992	7.518
H2A	0.35831	0.21545	0.15783	5.006
H3	0.70854	0.23573	0.27291	2.918
H5	0.59135	0.71144	0.33899	2.673
H7A	0.55795	0.94401	0.27520	4.799
H7B	0.57576	0.87418	0.15289	6.325
H7	1.08874	0.45915	0.60324	3.159
H8	0.90130	0.68026	0.43273	4.577
H9A	0.93402	0.59833	0.22182	4.688
H9B	1.11560	0.55447	0.37634	5.773
H9C	0.97382	0.44065	0.27774	5.594
H10A	0.91179	0.29144	0.98200	7.423
H10B	0.79044	0.41954	0.90742	7.423
H10C	0.81856	0.31513	0.79358	7.423

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S1	0.0525(10)	0.0487(10)	0.0365(9)	-0.0016(9)	0.0146(8)	-0.0094(9)
S2	0.0584(12)	0.0974(18)	0.0367(10)	0.0174(12)	0.0090(9)	0.0107(11)
O1	0.040(2)	0.031(2)	0.025(2)	-0.0012(18)	0.0209(18)	-0.0003(18)
O2	0.073(3)	0.025(3)	0.055(3)	0.002(2)	0.046(3)	0.007(2)
O3	0.062(3)	0.031(2)	0.025(2)	-0.007(2)	0.023(2)	-0.0012(19)
O4	0.090(4)	0.040(3)	0.033(3)	-0.005(3)	0.025(3)	0.009(2)
O5	0.051(3)	0.021(2)	0.040(2)	0.0094(18)	0.026(2)	0.0045(18)
O6	0.032(2)	0.035(2)	0.035(2)	0.0059(18)	0.0090(19)	0.004(2)
O7	0.060(3)	0.035(3)	0.040(3)	0.008(2)	0.026(3)	0.005(2)
C1	0.038(3)	0.035(4)	0.035(3)	0.001(3)	0.021(3)	0.002(3)
C2	0.039(3)	0.025(3)	0.034(3)	-0.004(3)	0.018(3)	-0.001(3)
C3	0.040(3)	0.022(3)	0.021(3)	-0.001(3)	0.017(3)	0.001(2)
C4	0.049(4)	0.024(3)	0.029(3)	-0.004(3)	0.020(3)	0.002(3)
C5	0.033(3)	0.020(3)	0.026(3)	-0.001(2)	0.013(3)	-0.001(2)
C6	0.030(3)	0.021(3)	0.023(3)	-0.000(2)	0.015(2)	-0.002(2)
C7	0.028(3)	0.038(4)	0.041(4)	-0.003(3)	0.012(3)	-0.007(3)
C8	0.040(3)	0.033(3)	0.036(3)	-0.009(3)	0.021(3)	-0.006(3)
C9	0.063(6)	0.073(7)	0.060(6)	-0.008(5)	0.043(5)	-0.005(5)
C10	0.099(7)	0.086(7)	0.051(5)	0.003(6)	0.036(5)	0.021(5)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Fragment Analysis

fragment: 1

S(1)	S(2)	O(1)	O(2)	O(3)
O(4)	O(5)	O(6)	C(1)	C(2)
C(3)	C(4)	C(5)	C(6)	C(7)
C(8)	C(9)	C(10)		

fragment: 2

O(7)

Table 5. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
S1	S2	2.026(3)	S1	C7	1.831(8)
S2	C10	1.799(11)	O1	C1	1.441(6)
O1	C6	1.417(8)	O2	C2	1.415(8)
O3	C3	1.448(7)	O3	C4	1.362(7)
O4	C4	1.193(8)	O5	C5	1.403(9)
O6	C6	1.395(6)	O6	C7	1.426(8)
C1	C2	1.514(11)	C2	C3	1.526(10)
C3	C6	1.553(8)	C4	C5	1.516(8)
C5	C6	1.534(8)	C5	C8	1.547(9)
C7	C8	1.536(9)	C8	C9	1.522(16)

Table 6. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
O2	H2	0.728	O5	H5	0.826
O7	H7A	0.836	O7	H7B	0.855
C1	H1A	0.961	C1	H1B	1.030
C2	H2A	0.965	C3	H3	1.005
C7	H7	0.967	C8	H8	0.997
C9	H9A	0.880	C9	H9B	1.036
C9	H9C	1.027	C10	H10A	0.960
C10	H10B	0.960	C10	H10C	0.960

Table 7. Bond angles (^{o})

atom	atom	atom	angle	atom	atom	atom	angle
S2	S1	C7	102.4(2)	S1	S2	C10	103.5(3)
C1	O1	C6	107.2(5)	C3	O3	C4	112.5(4)
C6	O6	C7	109.5(4)	O1	C1	C2	104.3(5)
O2	C2	C1	112.3(6)	O2	C2	C3	105.8(5)
C1	C2	C3	101.4(5)	O3	C3	C2	110.8(4)
O3	C3	C6	106.0(4)	C2	C3	C6	104.5(6)
O3	C4	O4	120.3(5)	O3	C4	C5	111.0(5)
O4	C4	C5	128.7(5)	O5	C5	C4	109.4(4)
O5	C5	C6	110.1(6)	O5	C5	C8	113.9(5)
C4	C5	C6	104.3(4)	C4	C5	C8	114.7(6)
C6	C5	C8	103.8(4)	O1	C6	O6	109.6(4)
O1	C6	C3	106.2(5)	O1	C6	C5	113.5(5)
O6	C6	C3	113.4(5)	O6	C6	C5	108.3(4)
C3	C6	C5	105.8(5)	S1	C7	O6	112.5(6)
S1	C7	C8	110.2(5)	O6	C7	C8	105.4(4)
C5	C8	C7	101.2(6)	C5	C8	C9	115.2(6)
C7	C8	C9	109.5(6)				

Table 8. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C2	O2	H2	107.6	C5	O5	H5	108.6
H7A	O7	H7B	110.8	O1	C1	H1A	109.2
O1	C1	H1B	104.4	C2	C1	H1A	108.1
C2	C1	H1B	114.4	H1A	C1	H1B	115.7
O2	C2	H2A	108.6	C1	C2	H2A	115.0
C3	C2	H2A	113.3	O3	C3	H3	111.9
C2	C3	H3	112.5	C6	C3	H3	110.7
S1	C7	H7	108.2	O6	C7	H7	102.5
C8	C7	H7	117.9	C5	C8	H8	107.4
C7	C8	H8	112.5	C9	C8	H8	110.8
C8	C9	H9A	105.4	C8	C9	H9B	107.1
C8	C9	H9C	115.0	H9A	C9	H9B	112.8
H9A	C9	H9C	116.2	H9B	C9	H9C	100.2
S2	C10	H10A	109.5	S2	C10	H10B	109.5
S2	C10	H10C	109.5	H10A	C10	H10B	109.5
H10A	C10	H10C	109.5	H10B	C10	H10C	109.5

Table 9. Torsion Angles(°)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
S2	S1	C7	O6	61.7(4)	S2	S1	C7	C8	179.0(3)
C7	S1	S2	C10	-89.0(3)	C1	O1	C6	O6	-146.4(4)
C1	O1	C6	C3	-23.5(5)	C1	O1	C6	C5	92.3(4)
C6	O1	C1	C2	40.0(5)	C3	O3	C4	O4	-176.4(6)
C3	O3	C4	C5	2.5(8)	C4	O3	C3	C2	114.7(5)
C4	O3	C3	C6	1.9(7)	C6	O6	C7	S1	88.9(5)
C6	O6	C7	C8	-31.2(7)	C7	O6	C6	O1	-110.0(5)
C7	O6	C6	C3	131.5(5)	C7	O6	C6	C5	14.4(7)
O1	C1	C2	O2	73.7(6)	O1	C1	C2	C3	-38.9(6)
O2	C2	C3	O3	153.1(5)	O2	C2	C3	C6	-93.2(5)
C1	C2	C3	O3	-89.6(5)	C1	C2	C3	C6	24.1(5)
O3	C3	C6	O1	115.6(5)	O3	C3	C6	O6	-124.0(5)
O3	C3	C6	C5	-5.4(6)	C2	C3	C6	O1	-1.5(5)
C2	C3	C6	O6	118.9(5)	C2	C3	C6	C5	-122.5(4)
O3	C4	C5	O5	-123.6(6)	O3	C4	C5	C6	-5.8(8)
O3	C4	C5	C8	107.1(6)	O4	C4	C5	O5	55.2(10)
O4	C4	C5	C6	173.0(8)	O4	C4	C5	C8	-74.1(10)
O5	C5	C6	O1	7.8(5)	O5	C5	C6	O6	-114.3(5)
O5	C5	C6	C3	123.8(4)	O5	C5	C8	C7	94.9(6)
O5	C5	C8	C9	-147.2(5)	C4	C5	C6	O1	-109.5(5)
C4	C5	C6	O6	128.4(5)	C4	C5	C6	C3	6.5(6)
C4	C5	C8	C7	-138.0(5)	C4	C5	C8	C9	-20.1(7)
C6	C5	C8	C7	-24.9(6)	C6	C5	C8	C9	93.0(6)
C8	C5	C6	O1	130.0(5)	C8	C5	C6	O6	8.0(7)
C8	C5	C6	C3	-113.9(5)	S1	C7	C8	C5	-87.5(5)
S1	C7	C8	C9	150.5(4)	O6	C7	C8	C5	34.1(7)
O6	C7	C8	C9	-87.9(6)					

Table 10. Possible hydrogen bonds

Donor	H	Acceptor	D...A	D-H	H...A	D-H...A
O2	H2	O1[1]	2.962(7)	0.73	2.49	123.99
O2	H2	O5[1]	2.987(9)	0.73	2.28	164.03
O5	H5	O7	2.663(7)	0.83	1.84	178.06
O7	H7A	O2[2]	2.776(8)	0.84	1.96	164.32

Symmetry Operators:

(1) -X+1,Y+1/2-1,-Z+1 (2) X,Y+1,Z

Table 11. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
S1	O1	3.580(4)	S1	O5	3.365(4)
S1	C5	3.335(5)	S1	C6	3.351(5)
S2	O6	3.329(4)	O1	O2	2.902(6)
O1	O3	3.307(7)	O1	O5	2.589(6)
O1	C4	3.353(9)	O1	C7	3.214(8)
O1	C8	3.543(9)	O1	C10	3.548(8)
O2	O3	3.587(7)	O2	C6	3.104(7)
O3	O5	3.366(7)	O3	O6	3.441(5)
O3	C1	3.080(10)	O3	C8	3.416(7)
O3	C9	3.472(11)	O4	O5	2.941(7)
O4	C3	3.455(8)	O4	C6	3.576(7)
O4	C8	3.253(6)	O4	C9	3.328(10)
O5	O6	3.310(6)	O5	C1	3.225(8)
O5	C3	3.480(7)	O5	C7	3.173(7)
O6	C1	3.460(8)	O6	C2	3.441(7)
O6	C4	3.440(7)	O6	C9	3.109(14)
O6	C10	3.425(11)	C1	C4	3.452(11)
C1	C5	3.171(10)	C2	C4	3.381(10)
C2	C5	3.498(8)	C3	C7	3.545(8)
C3	C8	3.423(9)	C4	C9	2.868(12)
C6	C9	3.300(14)			

Table 12. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S1	H8	2.735	S1	H10B	3.103
S1	H10C	3.103	S2	H7	3.009
O1	H2	2.875	O1	H2A	3.197
O1	H3	3.070	O1	H5	3.414
O1	H10B	3.538	O1	H10C	2.803
O2	H1A	3.229	O2	H1B	2.614
O2	H3	2.396	O3	H1A	2.886
O3	H2A	2.550	O3	H9A	3.505
O3	H9C	3.128	O4	H5	2.768
O4	H8	3.501	O4	H9A	2.861
O4	H9C	3.574	O5	H1A	2.834
O5	H8	2.476	O6	H3	2.484
O6	H8	3.189	O6	H9B	3.502
O6	H9C	2.916	O6	H10C	2.752
C1	H2	2.366	C1	H3	3.191
C3	H1A	2.634	C3	H1B	3.259
C3	H2	2.887	C3	H9C	3.247
C4	H1A	3.049	C4	H3	3.013
C4	H5	2.544	C4	H8	3.094
C4	H9A	2.723	C4	H9C	2.951
C5	H1A	2.992	C5	H3	3.113
C5	H7	3.235	C5	H9A	2.777
C5	H9B	3.440	C5	H9C	2.883
C6	H1A	2.602	C6	H1B	3.137
C6	H2	3.389	C6	H2A	3.253
C6	H5	3.124	C6	H7	3.048
C6	H8	3.206	C6	H9C	3.167
C6	H10C	3.338	C7	H5	3.591
C7	H9A	3.226	C7	H9B	2.634
C7	H9C	2.737	C7	H10C	3.418
C8	H5	2.621	C9	H7	2.610
H1A	H2	3.241	H1A	H2A	2.327
H1A	H3	3.599	H1A	H5	3.465
H1B	H2	2.255	H1B	H2A	2.649
H2	H2A	2.310	H2	H3	3.044
H2A	H3	2.639	H3	H9C	2.993
H5	H8	2.358	H7	H8	2.724

Table 12. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H7	H9A	3.440	H7	H9B	2.408
H7	H9C	2.718	H8	H9A	2.247
H8	H9B	2.435	H8	H9C	2.968

Table 13. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	O2[1]	2.962(7)	O1	O5[2]	3.419(7)
O1	O7[2]	3.433(9)	O2	O1[2]	2.962(7)
O2	O4[3]	3.525(8)	O2	O5[2]	2.987(9)
O2	O7[4]	2.776(8)	O3	O4[3]	3.113(6)
O3	O7[3]	3.069(7)	O4	O2[5]	3.525(8)
O4	O3[5]	3.113(6)	O4	O7	3.205(7)
O4	O7[3]	3.343(7)	O4	C2[5]	3.176(10)
O4	C3[5]	3.219(7)	O5	O1[1]	3.419(7)
O5	O2[1]	2.987(9)	O5	O7	2.663(7)
O5	C1[1]	3.200(9)	O6	C8[6]	3.440(8)
O7	O1[1]	3.433(9)	O7	O2[7]	2.776(8)
O7	O3[5]	3.069(7)	O7	O4	3.205(7)
O7	O4[5]	3.343(7)	O7	O5	2.663(7)
O7	C5	3.362(7)	O7	C7[8]	3.384(8)
C1	O5[2]	3.200(9)	C2	O4[3]	3.176(10)
C3	O4[3]	3.219(7)	C5	O7	3.362(7)
C7	O7[6]	3.384(8)	C8	O6[8]	3.440(8)

Symmetry Operators:

(1) -X+1,Y+1/2,-Z+1
(2) -X+1,Y+1/2-1,-Z+1
(3) -X+1,Y+1/2-1,-Z
(4) X,Y-1,Z
(5) -X+1,Y+1/2,-Z
(6) -X+2,Y+1/2-1,-Z+1
(7) X,Y+1,Z
(8) -X+2,Y+1/2,-Z+1

Table 14. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S1	H1B[1]	3.110	S1	H2[1]	3.283
S1	H2A[1]	3.516	S1	H3[2]	3.156
S1	H9C[2]	3.516	S1	H10A[3]	3.090
S2	H1A[4]	3.094	S2	H2A[4]	3.382
S2	H7B[5]	3.455	S2	H10A[3]	3.435
O1	H2[1]	2.493	O1	H5[6]	3.180
O1	H7A[6]	2.991	O2	H1B[6]	3.586
O2	H5[6]	3.469	O2	H7A[7]	1.961
O2	H7B[7]	3.227	O2	H7[5]	3.536
O2	H9B[5]	3.012	O2	H10B[6]	3.252
O3	H7A[8]	3.351	O3	H7B[8]	2.231
O3	H10B[9]	3.334	O4	H2A[10]	2.602
O4	H3[10]	3.051	O4	H7A[8]	3.127
O4	H7B	2.803	O4	H7B[8]	2.811
O4	H10B[9]	3.389	O5	H1B[1]	2.399
O5	H2[1]	2.280	O5	H7A	3.148
O5	H7B	3.103	O5	H10C[1]	3.512
O6	H8[5]	2.643	O6	H9A[5]	3.477
O6	H9B[5]	3.255	O7	H1B[1]	2.939
O7	H2[11]	3.179	O7	H5	1.837
O7	H7[2]	2.427	O7	H8	2.983
O7	H9B[2]	3.587	O7	H10B[1]	2.982
O7	H10C[1]	3.439	C1	H2[1]	3.268
C1	H5[6]	3.113	C1	H7A[6]	3.437
C1	H9B[12]	3.248	C1	H9C[12]	3.466
C2	H7A[7]	3.020	C3	H7A[7]	3.585
C3	H7B[8]	3.482	C4	H7B	3.469
C4	H7B[8]	2.918	C4	H10B[9]	3.522
C5	H1B[1]	3.528	C5	H2[1]	3.296
C5	H7B	3.583	C6	H2[1]	3.334
C7	H3[2]	3.391	C7	H7A[5]	3.586
C7	H8[5]	3.276	C9	H1A[13]	3.394
C9	H1B[13]	3.557	C9	H10B[9]	3.489
C9	H10C[2]	3.521	C10	H7A[6]	3.335
C10	H7B[6]	3.599	C10	H9A[5]	3.473
C10	H9C[14]	3.323	H1A	S2[15]	3.094
H1A	C9[12]	3.394	H1A	H2[1]	3.199

Table 14. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H1A	H9B^{12}	2.791	H1A	H9C^{12}	3.093
H1B	S1^{6}	3.110	H1B	O2^{1}	3.586
H1B	O5^{6}	2.399	H1B	O7^{6}	2.939
H1B	C5^{6}	3.528	H1B	C9^{12}	3.557
H1B	H2^{1}	3.423	H1B	H5^{6}	2.262
H1B	H7A^{6}	2.899	H1B	H7^{12}	3.599
H1B	H8^{6}	3.187	H1B	H9B^{12}	2.983
H1B	H9C^{12}	3.021	H2	S1^{6}	3.283
H2	O1^{6}	2.493	H2	O5^{6}	2.280
H2	O7^{7}	3.179	H2	C1^{6}	3.268
H2	C5^{6}	3.296	H2	C6^{6}	3.334
H2	H1A^{6}	3.199	H2	H1B^{6}	3.423
H2	H5^{6}	2.808	H2	H7A^{7}	2.367
H2	H9B^{5}	3.134	H2	H10B^{6}	3.344
H2A	S1^{6}	3.516	H2A	S2^{15}	3.382
H2A	O4^{8}	2.602	H2A	H7A^{7}	3.074
H2A	H7B^{8}	3.540	H2A	H9A^{8}	3.432
H2A	H10A^{15}	3.418	H2A	H10B^{6}	3.116
H3	S1^{5}	3.156	H3	O4^{8}	3.051
H3	C7^{5}	3.391	H3	H7A^{7}	3.142
H3	H7^{5}	3.135	H3	H8^{5}	3.202
H3	H9B^{5}	3.392	H5	O1^{1}	3.180
H5	O2^{1}	3.469	H5	O7	1.837
H5	C1^{1}	3.113	H5	H1B^{1}	2.262
H5	H2^{1}	2.808	H5	H7A	2.348
H5	H7B	2.313	H5	H7^{2}	3.485
H5	H10B^{1}	3.600	H5	H10C^{1}	3.246
H7A	O1^{1}	2.991	H7A	O2^{11}	1.961
H7A	O3^{10}	3.351	H7A	O4^{10}	3.127
H7A	O5	3.148	H7A	C1^{1}	3.437
H7A	C2^{11}	3.020	H7A	C3^{11}	3.585
H7A	C7^{2}	3.586	H7A	C10^{1}	3.335
H7A	H1B^{1}	2.899	H7A	H2^{11}	2.367
H7A	H2A^{11}	3.074	H7A	H3^{11}	3.142
H7A	H5	2.348	H7A	H7^{2}	2.653
H7A	H9B^{2}	3.313	H7A	H10B^{1}	2.634
H7A	H10C^{1}	3.192	H7B	S2^{2}	3.455

Table 14. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H7B	O2[11]	3.227	H7B	O3[10]	2.231
H7B	O4	2.803	H7B	O4[10]	2.811
H7B	O5	3.103	H7B	C3[10]	3.482
H7B	C4	3.469	H7B	C4[10]	2.918
H7B	C5	3.583	H7B	C10[1]	3.599
H7B	H2A[10]	3.540	H7B	H5	2.313
H7B	H7[2]	2.809	H7B	H8	3.364
H7B	H10B[1]	2.902	H7	O2[2]	3.536
H7	O7[5]	2.427	H7	H1B[13]	3.599
H7	H3[2]	3.135	H7	H5[5]	3.485
H7	H7A[5]	2.653	H7	H7B[5]	2.809
H7	H8[5]	2.770	H8	O6[2]	2.643
H8	O7	2.983	H8	C7[2]	3.276
H8	H1B[1]	3.187	H8	H3[2]	3.202
H8	H7B	3.364	H8	H7[2]	2.770
H8	H9C[2]	3.503	H9A	O6[2]	3.477
H9A	C10[2]	3.473	H9A	H2A[10]	3.432
H9A	H10A[2]	3.337	H9A	H10B[9]	3.131
H9A	H10C[2]	3.030	H9B	O2[2]	3.012
H9B	O6[2]	3.255	H9B	O7[5]	3.587
H9B	C1[13]	3.248	H9B	H1A[13]	2.791
H9B	H1B[13]	2.983	H9B	H2[2]	3.134
H9B	H3[2]	3.392	H9B	H7A[5]	3.313
H9B	H10C[2]	3.190	H9C	S1[5]	3.516
H9C	C1[13]	3.466	H9C	C10[9]	3.323
H9C	H1A[13]	3.093	H9C	H1B[13]	3.021
H9C	H8[5]	3.503	H9C	H10A[9]	2.938
H9C	H10B[9]	3.054	H10A	S1[16]	3.090
H10A	S2[16]	3.435	H10A	H2A[4]	3.418
H10A	H9A[5]	3.337	H10A	H9C[14]	2.938
H10B	O2[1]	3.252	H10B	O3[14]	3.334
H10B	O4[14]	3.389	H10B	O7[6]	2.982
H10B	C4[14]	3.522	H10B	C9[14]	3.489
H10B	H2[1]	3.344	H10B	H2A[1]	3.116
H10B	H5[6]	3.600	H10B	H7A[6]	2.634
H10B	H7B[6]	2.902	H10B	H9A[14]	3.131
H10B	H9C[14]	3.054	H10C	O5[6]	3.512

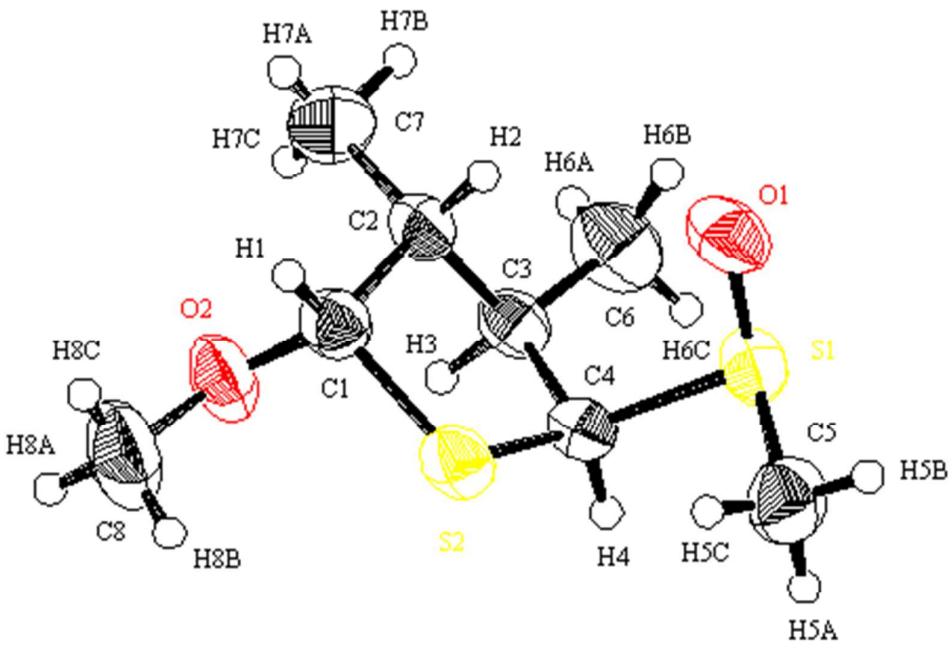
Table 14. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H10C	O7[6]	3.439	H10C	C9[5]	3.521
H10C	H5[6]	3.246	H10C	H7A[6]	3.192
H10C	H9A[5]	3.030	H10C	H9B[5]	3.190

Symmetry Operators:

(1) -X+1,Y+1/2,-Z+1
(2) -X+2,Y+1/2,-Z+1
(3) -X+2,Y+1/2,-Z+2
(4) X+1,Y,Z+1
(5) -X+2,Y+1/2-1,-Z+1
(6) -X+1,Y+1/2-1,-Z+1
(7) X,Y-1,Z
(8) -X+1,Y+1/2-1,-Z
(9) X,Y,Z-1
(10) -X+1,Y+1/2,-Z
(11) X,Y+1,Z
(12) X-1,Y,Z
(13) X+1,Y,Z
(14) X,Y,Z+1
(15) X-1,Y,Z-1
(16) -X+2,Y+1/2-1,-Z+2

X-ray experimental details of 3.



ORTEP drawing of compound **3**

Experimental

Data Collection

A colorless block crystal of $C_8H_{16}O_2S_2$ having approximate dimensions of 0.300 x 0.250 x 0.150 mm was mounted on a glass fiber. All measurements were made on a Rigaku R-AXIS RAPID diffractometer using graphite monochromated Cu-Kα radiation.

The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a C-centered monoclinic cell with dimensions:

a = 16.4838(7) Å

b = 7.4579(3) Å β = 102.915(7)^o

c = 9.0767(4) Å

V = 1087.61(9) Å^3

For Z = 4 and F.W. = 208.33, the calculated density is 1.272 g/cm^3. Based on the reflection conditions of:

hkl: h+k = 2n
h0l: l = 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

Cc (#9)

The data were collected at a temperature of 23 $\pm$ 1^{o}C to a maximum 2θ value of 136.2^o. A total of 45 oscillation images were collected. A sweep of data was done using ω scans from 80.0 to 260.0^o in 20.00^o step, at χ=54.0^o and ϕ = 0.0^o. The exposure rate was 60.0 [sec./^{o}]. A second sweep was performed using ω scans from 80.0 to 260.0^o in 20.00^o step, at χ=54.0^o and ϕ = 90.0^o. The exposure rate was 60.0 [sec./^{o}]. Another sweep was

performed using ω scans from 80.0 to 260.0^{o} in 20.00^{o} step, at χ=54.0^{o} and ϕ = 180.0^{o}. The exposure rate was 60.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^{o} in 20.00^{o} step, at χ=54.0^{o} and ϕ = 270.0^{o}. The exposure rate was 60.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^{o} in 20.00^{o} step, at χ=0.0^{o} and ϕ = 0.0^{o}. The exposure rate was 60.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 5844 reflections were collected, where 1793 were unique (R_{int} = 0.0580); equivalent reflections were merged.

The linear absorption coefficient, μ, for Cu-Kα radiation is 41.464 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.320 to 0.537. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction[1] was applied (coefficient = 0.001070).

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^{2} was based on 1793 observed reflections and 150 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0343$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1171$$

The goodness of fit[4] was 1.15. Unit weights were used. The maximum and

minimum peaks on the final difference Fourier map corresponded to 0.22 and -0.23 $e^-/Å^3$, respectively. The final Flack parameter [5] was 0.064(17),

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [7]. Anomalous dispersion effects were included in Fcalc[8]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[9]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[10]. All calculations were performed using the CrystalStructure[11] crystallographic software package except for refinement, which was performed using SHELXL Version 2016/6[12].

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_8H_{16}O_2S_2$
Formula Weight	208.33
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.300 X 0.250 X 0.150 mm
Crystal System	monoclinic
Lattice Type	C-centered
Lattice Parameters	a = 16.4838(7) Å b = 7.4579(3) Å c = 9.0767(4) Å β = 102.915(7) ^{o} V = 1087.61(9) Å^3
Space Group	Cc (#9)
Z value	4
D_{calc}	1.272 g/cm^3
F_{000}	448.00
μ(CuKα)	41.464 cm^{-1}

B. Intensity Measurements

Diffractometer	R-AXIS RAPID
Radiation	CuKα (λ = 1.54187 Å) graphite monochromated
Voltage, Current	40kV, 100mA
Temperature	23.0^{o}C
Detector Aperture	460.0 x 256.0 mm
Data Images	45 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	60.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=90.0)	80.0 - 260.0^{o}
Exposure Rate	60.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=180.0)	80.0 - 260.0^{o}
Exposure Rate	60.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=270.0)	80.0 - 260.0^{o}
Exposure Rate	60.0 sec./^{o}
ω oscillation Range (χ=0.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	60.0 sec./^{o}

Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	136.1°
No. of Reflections Measured	Total: 5844 Unique: 1793 (R_{int} = 0.0580) Parsons quotients (Flack x parameter): 707
Corrections	Lorentz-polarization Absorption (trans. factors: 0.320 - 0.537) Secondary Extinction (coefficient: 1.07000e-003)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXT Version 2014/5)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w (Fo^2 - Fc^2)^2
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + (0.0788 · P)^2 + 0.0000 · P] where P = (Max(Fo^2,0) + 2Fc^2)/3
$2\theta_{max}$ cutoff	136.2^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	1793
No. Variables	150
Reflection/Parameter Ratio	11.95
Residuals: R1 (I>2.00σ(I))	0.0343
Residuals: R (All reflections)	0.0392
Residuals: wR2 (All reflections)	0.1171
Goodness of Fit Indicator	1.151
Flack parameter (Parsons' quotients = 707)	0.064(17)
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.22 e^-/Å^3
Minimum peak in Final Diff. Map	-0.23 e^-/Å^3

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
S1	0.67074(7)	0.39905(18)	0.55576(11)	4.03(3)
S2	0.50010(8)	0.57578(13)	0.47843(14)	4.00(3)
O1	0.6598(3)	0.3488(6)	0.3930(5)	5.02(8)
O2	0.3551(3)	0.3997(5)	0.4493(6)	5.08(9)
C1	0.4250(3)	0.4066(6)	0.3861(7)	3.95(10)
C2	0.4714(3)	0.2280(6)	0.4144(7)	3.75(9)
C3	0.5212(3)	0.2352(6)	0.5783(6)	3.51(9)
C4	0.5663(3)	0.4168(6)	0.5970(6)	3.38(8)
C5	0.6974(4)	0.6309(9)	0.5677(8)	5.35(13)
C6	0.5761(6)	0.0726(8)	0.6272(11)	5.59(15)
C7	0.4134(5)	0.0684(8)	0.3746(14)	5.85(16)
C8	0.3006(5)	0.5481(11)	0.4120(13)	7.3(2)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1	0.40202	0.44202	0.26937	3.251
H2	0.51049	0.23150	0.34659	3.838
H3	0.47552	0.24546	0.63384	3.604
H4	0.57902	0.45940	0.69280	4.299
H5A	0.70511	0.67023	0.67056	6.418
H5B	0.74793	0.64851	0.53378	6.418
H5C	0.65335	0.69882	0.50504	6.418
H6A	0.54656	-0.02860	0.63771	4.829
H6B	0.60803	0.03753	0.55713	7.952
H6C	0.61496	0.08266	0.73091	7.830
H7A	0.38834	0.07164	0.27912	7.906
H7B	0.45070	-0.04126	0.38865	4.473
H7C	0.38522	0.05010	0.43771	6.294
H8A	0.25429	0.53414	0.45916	8.767
H8B	0.32986	0.65660	0.44728	8.767
H8C	0.28061	0.55397	0.30433	8.767

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S1	0.0430(6)	0.0594(7)	0.0513(8)	0.0071(5)	0.0119(5)	0.0043(6)
S2	0.0534(7)	0.0324(5)	0.0677(10)	0.0058(5)	0.0170(6)	0.0047(5)
O1	0.068(2)	0.073(2)	0.056(2)	0.014(2)	0.0274(19)	-0.0031(19)
O2	0.045(2)	0.051(2)	0.099(4)	0.0119(16)	0.020(2)	0.003(2)
C1	0.051(3)	0.043(3)	0.057(4)	0.0045(19)	0.013(3)	0.000(2)
C2	0.043(2)	0.041(2)	0.058(3)	0.0060(19)	0.013(2)	-0.005(2)
C3	0.050(2)	0.036(2)	0.050(3)	0.0068(19)	0.016(2)	0.0052(19)
C4	0.047(3)	0.042(2)	0.042(3)	0.0043(18)	0.014(2)	-0.0012(19)
C5	0.058(3)	0.079(4)	0.070(5)	-0.014(3)	0.023(3)	-0.002(3)
C6	0.083(5)	0.047(3)	0.084(6)	0.014(3)	0.022(4)	0.017(3)
C7	0.073(4)	0.045(3)	0.101(7)	-0.002(3)	0.012(5)	-0.015(3)
C8	0.055(4)	0.076(4)	0.145(8)	0.021(3)	0.017(4)	0.012(5)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
S1	O1	1.495(4)	S1	C4	1.846(6)
S1	C5	1.781(7)	S2	C1	1.834(5)
S2	C4	1.798(5)	O2	C1	1.399(8)
O2	C8	1.418(9)	C1	C2	1.529(7)
C2	C3	1.531(7)	C2	C7	1.519(9)
C3	C4	1.537(7)	C3	C6	1.519(9)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1	1.075	C2	H2	0.985
C3	H3	0.998	C4	H4	0.905
C5	H5A	0.960	C5	H5B	0.960
C5	H5C	0.960	C6	H6A	0.915
C6	H6B	0.948	C6	H6C	1.016
C7	H7A	0.873	C7	H7B	1.014
C7	H7C	0.825	C8	H8A	0.960
C8	H8B	0.960	C8	H8C	0.960

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O1	S1	C4	107.9(2)	O1	S1	C5	106.1(3)
C4	S1	C5	98.4(3)	C1	S2	C4	94.4(2)
C1	O2	C8	114.2(6)	S2	C1	O2	111.8(4)
S2	C1	C2	105.1(3)	O2	C1	C2	109.1(4)
C1	C2	C3	105.8(4)	C1	C2	C7	112.2(5)
C3	C2	C7	115.5(6)	C2	C3	C4	106.2(4)
C2	C3	C6	114.3(5)	C4	C3	C6	115.4(5)
S1	C4	S2	113.2(3)	S1	C4	C3	111.4(4)
S2	C4	C3	107.5(3)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
S2	C1	H1	109.5	O2	C1	H1	105.8
C2	C1	H1	115.6	C1	C2	H2	104.2
C3	C2	H2	108.9	C7	C2	H2	109.7
C2	C3	H3	101.1	C4	C3	H3	106.1
C6	C3	H3	112.4	S1	C4	H4	101.2
S2	C4	H4	108.8	C3	C4	H4	114.7
S1	C5	H5A	109.5	S1	C5	H5B	109.5
S1	C5	H5C	109.5	H5A	C5	H5B	109.5
H5A	C5	H5C	109.5	H5B	C5	H5C	109.5
C3	C6	H6A	113.1	C3	C6	H6B	114.1
C3	C6	H6C	115.0	H6A	C6	H6B	103.3
H6A	C6	H6C	101.8	H6B	C6	H6C	108.2
C2	C7	H7A	110.3	C2	C7	H7B	105.7
C2	C7	H7C	112.8	H7A	C7	H7B	106.6
H7A	C7	H7C	118.6	H7B	C7	H7C	101.5
O2	C8	H8A	109.5	O2	C8	H8B	109.5
O2	C8	H8C	109.5	H8A	C8	H8B	109.5
H8A	C8	H8C	109.5	H8B	C8	H8C	109.5

Table 8. Torsion Angles(°)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O1	S1	C4	S2	-57.5(3)	O1	S1	C4	C3	63.9(4)
C5	S1	C4	S2	52.5(3)	C5	S1	C4	C3	173.8(3)
C1	S2	C4	S1	115.3(3)	C1	S2	C4	C3	-8.2(4)
C4	S2	C1	O2	99.1(4)	C4	S2	C1	C2	-19.1(4)
C8	O2	C1	S2	72.6(6)	C8	O2	C1	C2	-171.5(5)
S2	C1	C2	C3	41.2(5)	S2	C1	C2	C7	167.9(3)
O2	C1	C2	C3	-78.9(5)	O2	C1	C2	C7	47.8(6)
C1	C2	C3	C4	-48.4(5)	C1	C2	C3	C6	-176.8(4)
C7	C2	C3	C4	-173.1(5)	C7	C2	C3	C6	58.5(7)
C2	C3	C4	S1	-91.2(4)	C2	C3	C4	S2	33.4(5)
C6	C3	C4	S1	36.5(7)	C6	C3	C4	S2	161.1(5)

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
S1	C2	3.490(5)	S1	C6	3.038(8)
S2	O1	3.363(5)	S2	C5	3.197(7)
S2	C8	3.215(8)	O1	C2	3.280(7)
O1	C3	3.239(8)	O1	C6	3.461(10)
O2	C3	2.990(6)	O2	C4	3.443(6)
O2	C7	2.789(9)	C6	C7	3.115(13)

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S1	H2	3.147	S1	H6B	2.889
S1	H6C	3.097	S2	H2	2.854
S2	H3	2.912	S2	H5A	3.510
S2	H5C	2.647	S2	H8B	2.822
O1	H2	2.557	O1	H4	3.388
O1	H5A	3.436	O1	H5B	2.812
O1	H5C	2.812	O1	H6B	2.984
O2	H2	3.174	O2	H3	2.566
O2	H7A	3.009	O2	H7C	2.661
C1	H3	2.522	C1	H4	3.345
C1	H7A	2.699	C1	H7B	3.366
C1	H7C	2.803	C1	H8A	3.178
C1	H8B	2.576	C1	H8C	2.576
C2	H4	3.241	C2	H6A	2.861
C2	H6B	2.732	C2	H6C	3.462
C3	H1	3.412	C3	H7A	3.316
C3	H7B	2.769	C3	H7C	2.698
C4	H1	3.552	C4	H2	2.644
C4	H5A	2.927	C4	H5C	2.777
C4	H6A	3.366	C4	H6B	2.952
C4	H6C	2.810	C5	H4	2.779
C6	H2	2.798	C6	H4	2.944
C6	H7B	2.772	C6	H7C	3.236
C7	H1	2.938	C7	H3	2.693
C7	H6A	2.950	C7	H6B	3.273
C8	H1	2.463	H1	H2	2.365
H1	H3	3.573	H1	H7A	2.774
H1	H7C	3.338	H1	H8A	3.353
H1	H8B	2.728	H1	H8C	2.255
H2	H3	2.795	H2	H4	3.529
H2	H6A	3.224	H2	H6B	2.637
H2	H7A	2.302	H2	H7B	2.329
H2	H7C	2.746	H3	H4	2.310
H3	H6A	2.352	H3	H6B	2.888
H3	H6C	2.575	H3	H7A	3.470
H3	H7B	3.047	H3	H7C	2.515
H4	H5A	2.650	H4	H5C	2.920

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H4	H6B	3.451	H4	H6C	2.876
H6A	H7B	2.453	H6A	H7C	2.925
H6B	H7B	2.763	H6B	H7C	3.591

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	C4[1]	3.286(6)	O1	C5[1]	3.153(9)
O1	C8[2]	3.204(9)	C4	O1[3]	3.286(6)
C5	O1[3]	3.153(9)	C8	O1[4]	3.204(9)

Symmetry Operators:

(1) X,-Y+1,Z+1/2-1
(2) X+1/2,Y+1/2-1,Z
(3) X,-Y+1,Z+1/2
(4) X+1/2-1,Y+1/2,Z

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S1	H4[1]	3.468	S1	H8A[2]	3.258
S1	H8B[2]	3.503	S2	H1[3]	3.393
S2	H3[1]	3.340	S2	H4[1]	3.161
S2	H6A[4]	3.300	S2	H7B[4]	3.031
O1	H4[1]	2.458	O1	H5A[1]	2.308
O1	H5C[1]	3.517	O1	H6A[5]	3.553
O1	H6C[5]	3.547	O1	H8A[2]	2.806
O1	H8B[2]	3.088	O1	H8C[2]	3.188
O2	H1[3]	3.070	O2	H5B[6]	2.797
C1	H4[1]	3.537	C2	H6A[5]	3.383
C3	H7B[7]	3.583	C4	H1[3]	3.576
C5	H4[1]	3.587	C5	H6B[4]	3.363
C5	H8A[8]	3.363	C5	H8C[9]	3.272
C6	H2[7]	3.353	C6	H5C[10]	3.354
C6	H7B[7]	3.486	C6	H8C[11]	3.523
C7	H3[5]	3.508	C7	H5B[6]	3.412
C7	H6A[5]	3.412	C7	H8B[10]	3.489
C8	H1[3]	3.301	C8	H5A[12]	3.186
C8	H5B[6]	3.359	C8	H6C[13]	3.281
H1	S2[1]	3.393	H1	O2[1]	3.070
H1	C4[1]	3.576	H1	C8[1]	3.301
H1	H3[1]	3.013	H1	H4[1]	3.231
H1	H8A[1]	3.290	H1	H8B[1]	2.991
H2	C6[5]	3.353	H2	H4[1]	3.040
H2	H6A[5]	2.597	H2	H6C[5]	3.220
H3	S2[3]	3.340	H3	C7[7]	3.508
H3	H1[3]	3.013	H3	H7A[7]	3.201
H3	H7B[7]	2.874	H4	S1[3]	3.468
H4	S2[3]	3.161	H4	O1[3]	2.458
H4	C1[3]	3.537	H4	C5[3]	3.587
H4	H1[3]	3.231	H4	H2[3]	3.040
H4	H5C[3]	3.062	H5A	O1[3]	2.308
H5A	C8[9]	3.186	H5A	H6A[4]	3.408
H5A	H6B[4]	3.223	H5A	H6C[4]	3.512
H5A	H7A[11]	3.467	H5A	H8A[8]	3.521
H5A	H8A[9]	3.381	H5A	H8B[9]	3.146
H5A	H8C[9]	2.565	H5B	O2[8]	2.797

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H5B	C7[8]	3.412	H5B	C8[8]	3.359
H5B	H7A[11]	3.272	H5B	H7C[8]	2.702
H5B	H8A[8]	2.962	H5B	H8C[9]	3.264
H5C	O1[3]	3.517	H5C	C6[4]	3.354
H5C	H4[1]	3.062	H5C	H6A[4]	3.103
H5C	H6B[4]	2.706	H5C	H6C[1]	2.923
H5C	H8A[8]	3.083	H5C	H8C[9]	3.554
H6A	S2[10]	3.300	H6A	O1[7]	3.553
H6A	C2[7]	3.383	H6A	C7[7]	3.412
H6A	H2[7]	2.597	H6A	H5A[10]	3.408
H6A	H5C[10]	3.103	H6A	H7A[7]	3.170
H6A	H7B[7]	3.093	H6B	C5[10]	3.363
H6B	H5A[10]	3.223	H6B	H5C[10]	2.706
H6B	H6C[5]	3.120	H6B	H8A[2]	2.750
H6B	H8C[11]	3.277	H6C	O1[7]	3.547
H6C	C8[11]	3.281	H6C	H2[7]	3.220
H6C	H5A[10]	3.512	H6C	H5C[3]	2.923
H6C	H6B[7]	3.120	H6C	H7B[7]	3.348
H6C	H8A[11]	2.863	H6C	H8C[11]	2.850
H7A	H3[5]	3.201	H7A	H5A[13]	3.467
H7A	H5B[13]	3.272	H7A	H6A[5]	3.170
H7A	H7C[5]	3.219	H7A	H8B[1]	3.579
H7B	S2[10]	3.031	H7B	C3[5]	3.583
H7B	C6[5]	3.486	H7B	H3[5]	2.874
H7B	H6A[5]	3.093	H7B	H6C[5]	3.348
H7B	H8B[10]	3.131	H7C	H5B[6]	2.702
H7C	H7A[7]	3.219	H7C	H8B[10]	3.080
H8A	S1[14]	3.258	H8A	O1[14]	2.806
H8A	C5[6]	3.363	H8A	H1[3]	3.290
H8A	H5A[6]	3.521	H8A	H5A[12]	3.381
H8A	H5B[6]	2.962	H8A	H5C[6]	3.083
H8A	H6B[14]	2.750	H8A	H6C[13]	2.863
H8A	H8C[3]	3.135	H8B	S1[14]	3.503
H8B	O1[14]	3.088	H8B	C7[4]	3.489
H8B	H1[3]	2.991	H8B	H5A[12]	3.146
H8B	H7A[3]	3.579	H8B	H7B[4]	3.131
H8B	H7C[4]	3.080	H8C	O1[14]	3.188

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H8C	C5[12]	3.272	H8C	C6[13]	3.523
H8C	H5A[12]	2.565	H8C	H5B[12]	3.264
H8C	H5C[12]	3.554	H8C	H6B[13]	3.277
H8C	H6C[13]	2.850	H8C	H8A[1]	3.135

Symmetry Operators:

(1) X,-Y+1,Z+1/2-1
(2) X+1/2,Y+1/2-1,Z
(3) X,-Y+1,Z+1/2
(4) X,Y+1,Z
(5) X,-Y,Z+1/2-1
(6) X+1/2-1,Y+1/2-1,Z
(7) X,-Y,Z+1/2
(8) X+1/2,Y+1/2,Z
(9) X+1/2,-Y+1/2+1,Z+1/2
(10) X,Y-1,Z
(11) X+1/2,-Y+1/2,Z+1/2
(12) X+1/2-1,-Y+1/2+1,Z+1/2-1
(13) X+1/2-1,-Y+1/2,Z+1/2-1
(14) X+1/2-1,Y+1/2,Z

References

(1) Larson, A.C. (1970), Crystallographic Computing, 291-294. F.R. Ahmed, ed. Munksgaard, Copenhagen (equation 22, with V replaced by the cell volume).

(2) SHELXT Version 2014/5: Sheldrick, G. M. (2014). Acta Cryst. A70, C1437.

(3) Least Squares function minimized: (SHELXL Version 2016/6)

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Goodness of fit is defined as:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations

N_v = number of variables

(5) Parsons, S. and Flack, H. (2004), Acta Cryst. A60, s61.

(6) Flack, H.D. and Bernardinelli (2000), J. Appl. Cryst. 33, 114-1148.

(7) International Tables for Crystallography, Vol.C (1992). Ed. A.J.C. Wilson, Kluwer Academic Publishers, Dordrecht, Netherlands, Table 6.1.1.4, pp. 572.

(8) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(9) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(10) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(11) CrystalStructure 4.2.5: Crystal Structure Analysis Package, Rigaku Corporation (2000-2017). Tokyo 196-8666, Japan.

(12) SHELXL Version 2016/6: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.