ORGANOMETALLICS
Organometallics, 1998, 17(10), 2067-2075, DOI:10.1021/om9800086

Table 1. Crystal data and structure refinement for 9

Identification code	cad12
Empirical formula	$C_{10}H_6Mn_2O_8S_2$
Formula weight	428.15
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 6.0508(4) Å alpha = 90° b = 14.2411(10) Å beta = 107.374(2)° c = 9.5560(6) Å gamma = 90°
Volume, Z	785.87(9) $Å^3$, 2
Density (calculated)	1.809 Mg/m^3
Absorption coefficient	1.904 mm^{-1}
F(000)	424
Crystal size	0.10 x 0.23 x 0.36 mm
θ range for data collection	2.65 to 23.32°
Limiting indices	$-6 \le h \le 5$, $-15 \le k \le 15$, $-10 \le l \le 10$
Reflections collected	3053
Independent reflections	1084 (R_{int} = 0.0391)
Absorption correction	Empirical
Max. and min. transmission	0.862, 0.377
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1084 / 0 / 100
Goodness-of-fit on F^2	1.042
Final R indices [$I>2\sigma(I)$]	R1 = 0.0492, wR2 = 0.1276
R indices (all data)	R1 = 0.0601, wR2 = 0.1352
Largest diff. peak and hole	0.900 and -0.642 $eÅ^{-3}$

***** Notes for writing up ~~cadi2~~ 9 *****

The crystal was a small, clear orange irregular chip. X-ray data collection was carried out using a Siemens P4 single-crystal diffractometer equipped with a CCD area detector and controlled by SMART version 4 (1) software. Data reductionwas carried out by SAINT version 4 (1) and included profile analysis; this was followed by absorption correction by use of the program SADABS (2).

The structure was determined by direct methods and refined on F squared by use of programs in the SHELXTL PC version 5 (3) package, which were also used for all figures. All three hydrogen atoms appeared in a difference map, and were introduced in ideal positions, riding on the carbon atom to which they are bonded; each was refined with isotropic temperature factor 20% greater than that of the ridden atom. All other atoms were refined with anisotropic thermal parameters.

The molecule sits on a center of inversion, so only one half is independent. Most of the molecule is nearly planar, while two carbonyl groups on each manganese are approximately normal to this plane, and the methyl groups on the sulfur atoms are tipped out of the plane. The bonds from manganese to carbonyl carbon are shorter (average 1.816 A) along the direction of the sulfur - manganese bonds, compared to the average 1.857 for bonds to the carbons of the out-of-plane carbonyl groups.

Acknowledgement

The X-ray equipment was purchased with assistance from an instrument grant from the National Science Foundation (CHE-8206423) and a grant from the National Institutes of Health (RR-06462).

References

(1) Siemens Industrial Automation Incorporated
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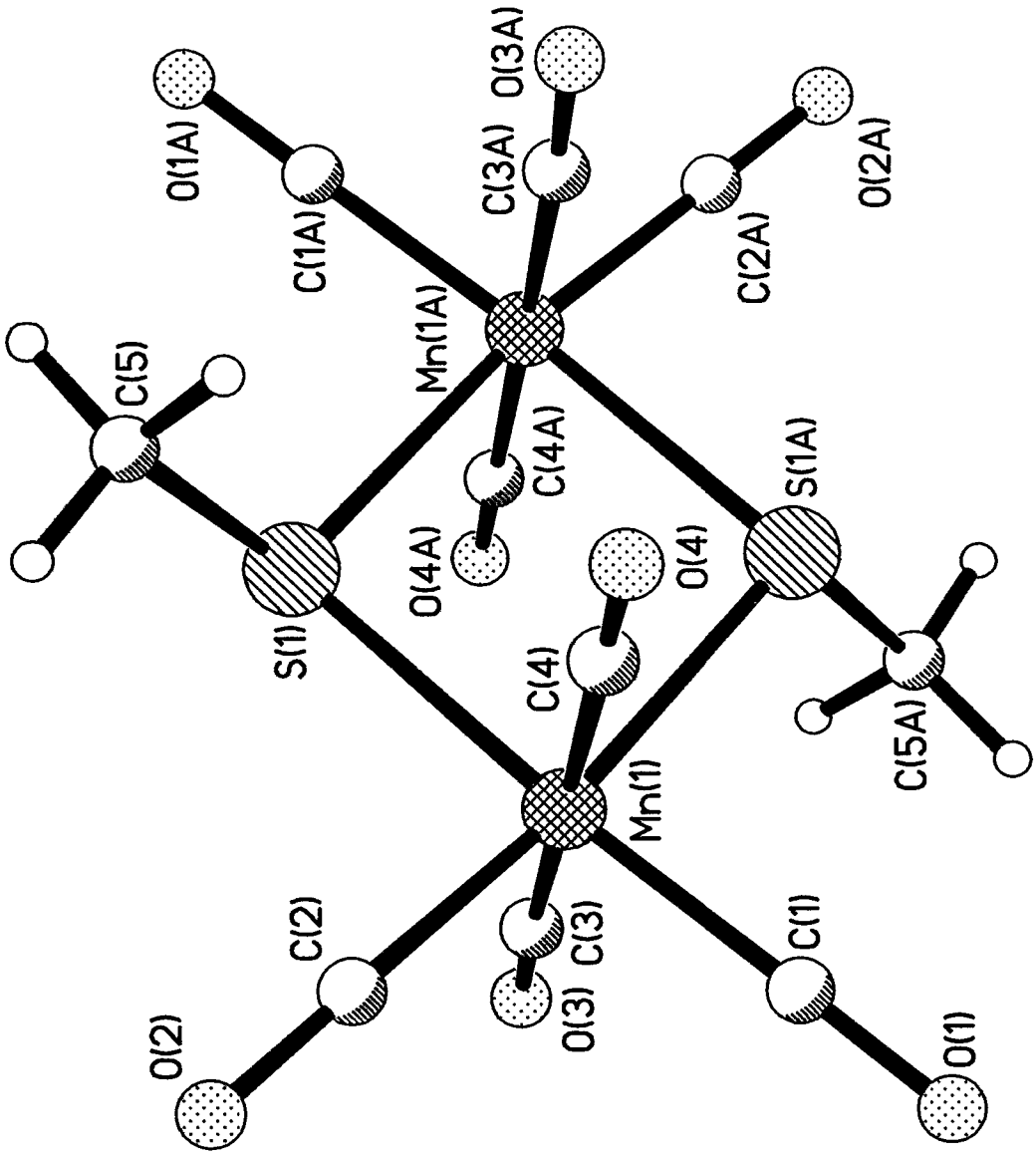
(2) G. M. Sheldrick (1996) SADABS, Empirical absorption (and other) corrections. Univ. of Goettingen, Germany.

(3) Siemens Analytical X-Ray Instruments, Inc.
(same address as first reference)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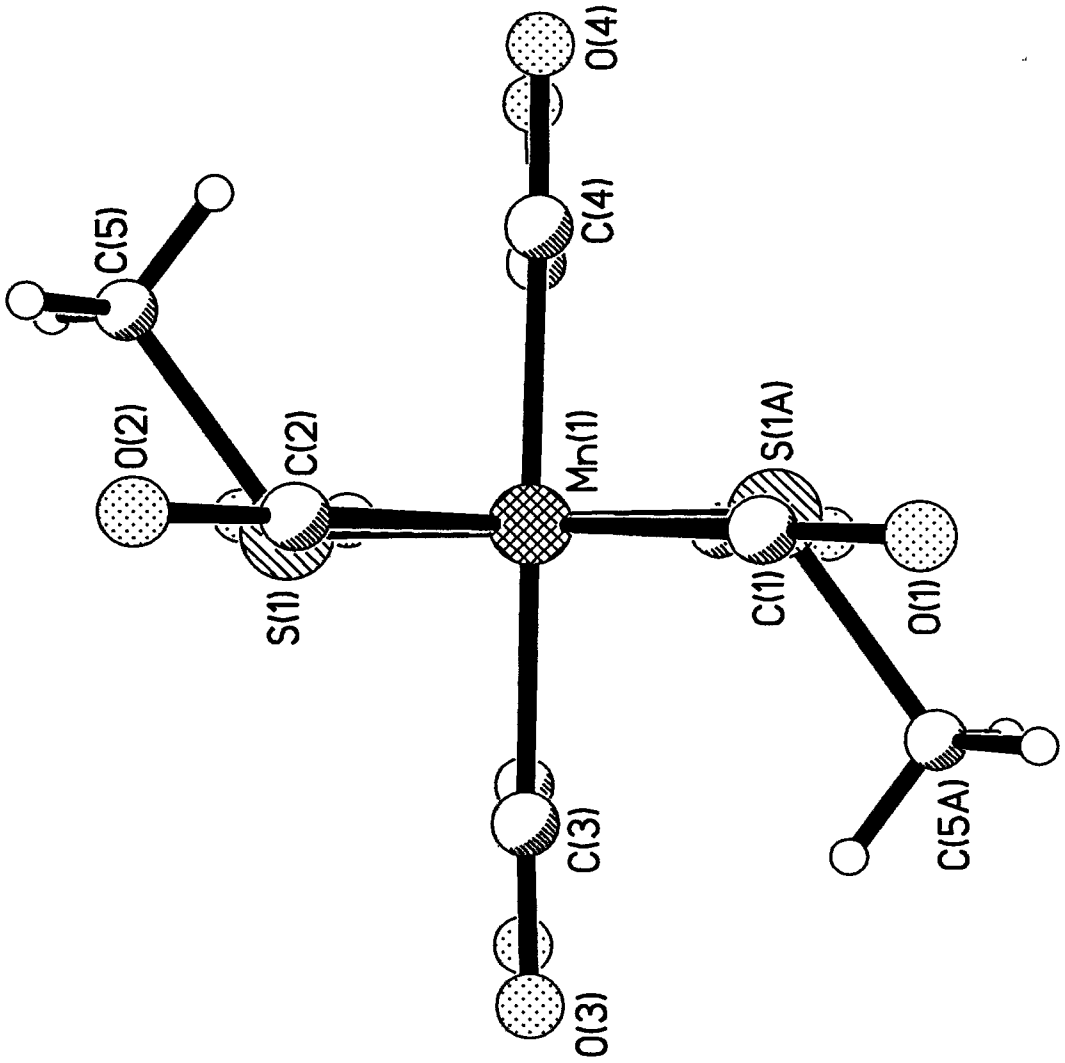
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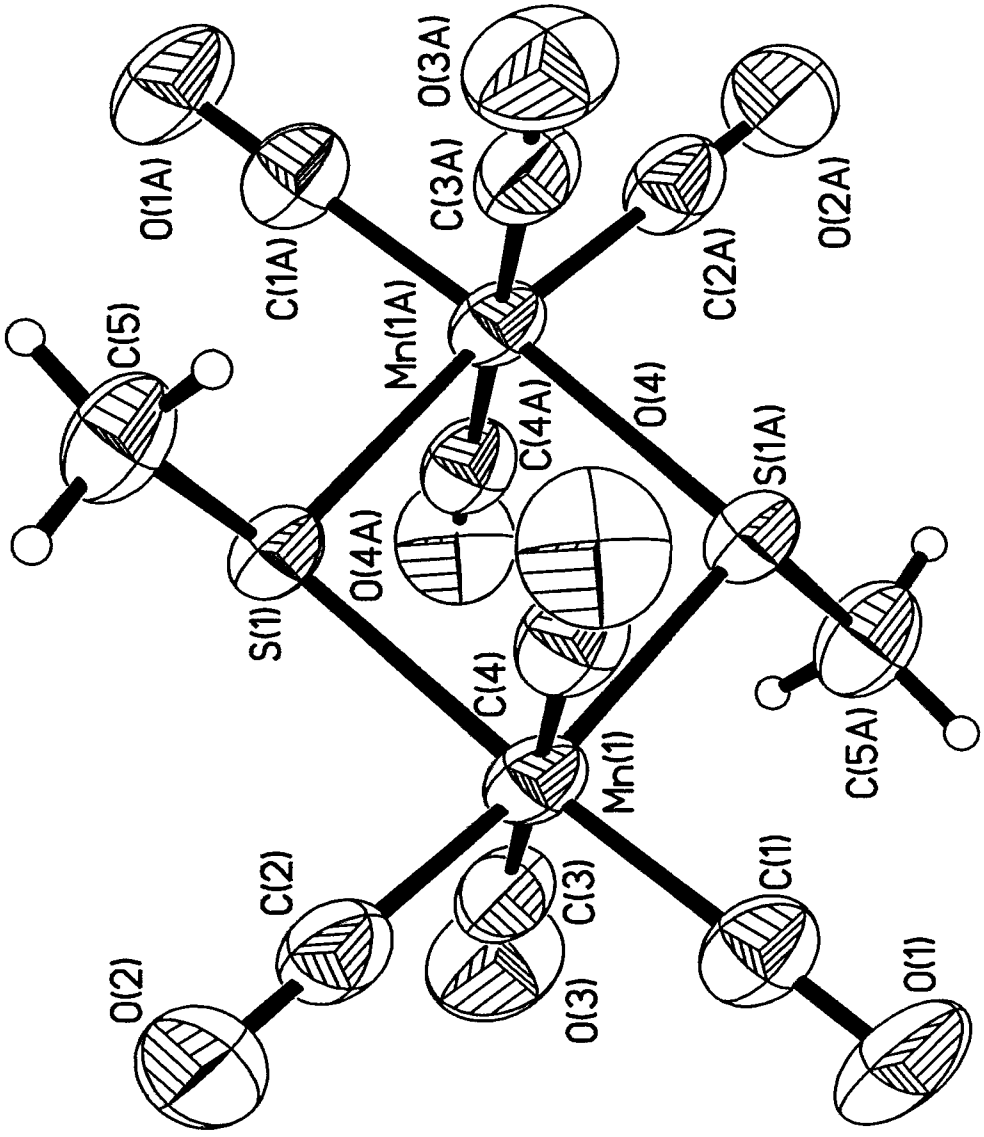
O(3)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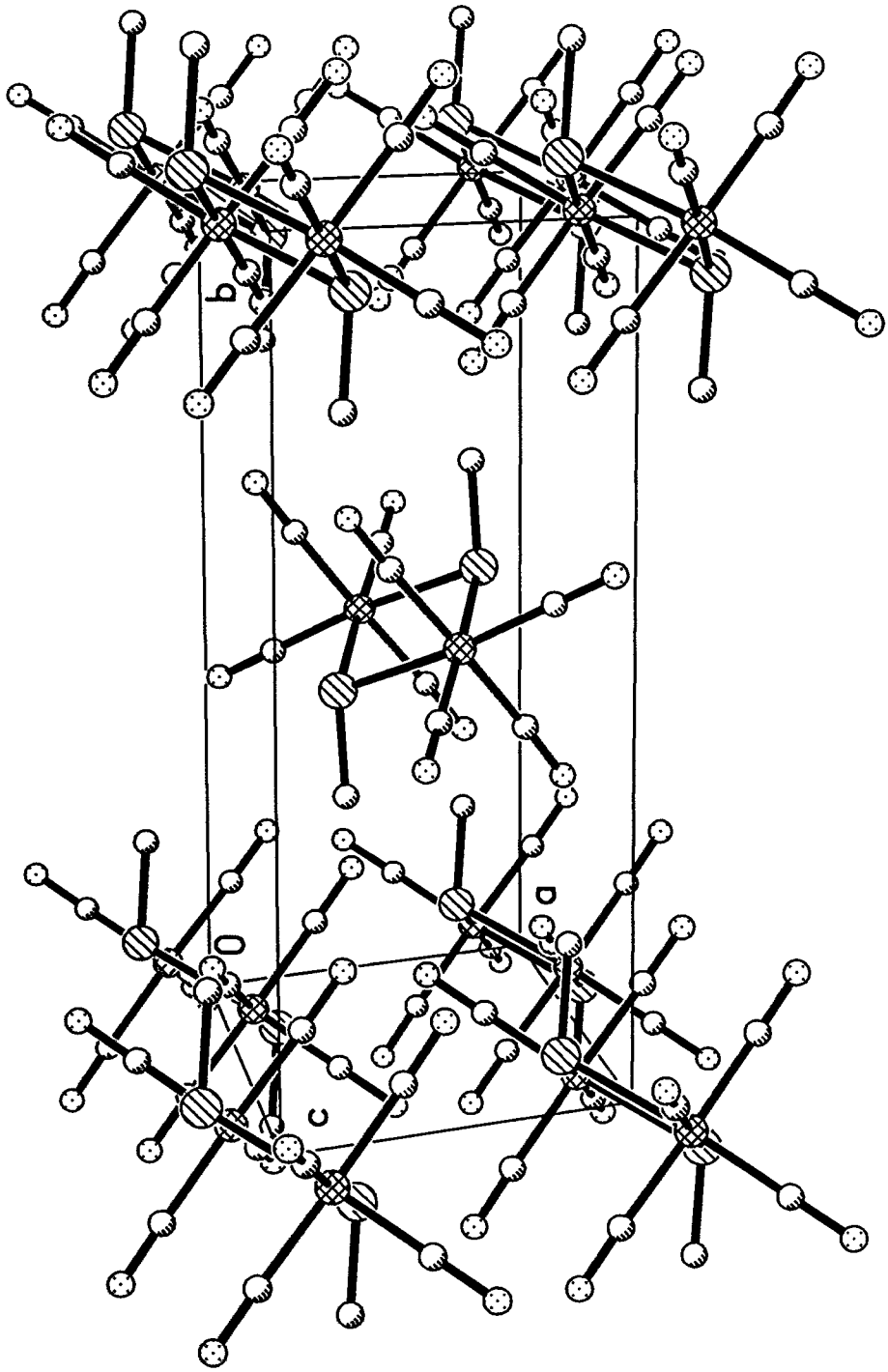
b
D
O
c

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Mn(1)	947(1)	24(1)	1961(1)	40(1)
C(1)	-66(10)	645(3)	3319(6)	53(1)
O(1)	-681(8)	1038(3)	4172(5)	75(1)
C(2)	3267(10)	-578(4)	3307(6)	54(1)
O(2)	4733(7)	-956(3)	4160(5)	76(1)
C(3)	2896(10)	1020(4)	1885(6)	52(1)
O(3)	4038(7)	1645(3)	1871(5)	82(1)
C(4)	-1063(10)	-961(4)	1950(6)	49(1)
O(4)	-2250(7)	-1573(3)	1976(5)	75(1)
S(1)	2091(2)	-660(1)	8(1)	40(1)
C(5)	1867(10)	-1932(3)	46(6)	61(2)

Table 3. Bond lengths [Å] and angles [°] for 9

Mn(1)-C(2)	1.812(6)	Mn(1)-C(1)	1.821(5)
Mn(1)-C(4)	1.854(6)	Mn(1)-C(3)	1.860(6)
Mn(1)-S(1)#1	2.3825(14)	Mn(1)-S(1)	2.3842(14)
C(1)-O(1)	1.139(6)	C(2)-O(2)	1.144(6)
C(3)-O(3)	1.130(6)	C(4)-O(4)	1.134(6)
S(1)-C(5)	1.817(4)	S(1)-Mn(1)#1	2.3825(14)
C(2)-Mn(1)-C(1)	94.5(2)	C(2)-Mn(1)-C(4)	90.8(2)
C(1)-Mn(1)-C(4)	90.7(2)	C(2)-Mn(1)-C(3)	91.3(2)
C(1)-Mn(1)-C(3)	90.6(2)	C(4)-Mn(1)-C(3)	177.4(2)
C(2)-Mn(1)-S(1)#1	172.9(2)	C(1)-Mn(1)-S(1)#1	91.9(2)
C(4)-Mn(1)-S(1)#1	86.2(2)	C(3)-Mn(1)-S(1)#1	91.6(2)
C(2)-Mn(1)-S(1)	91.0(2)	C(1)-Mn(1)-S(1)	174.0(2)
C(4)-Mn(1)-S(1)	91.7(2)	C(3)-Mn(1)-S(1)	86.8(2)
S(1)#1-Mn(1)-S(1)	82.70(5)	O(1)-C(1)-Mn(1)	179.3(5)
O(2)-C(2)-Mn(1)	179.8(4)	O(3)-C(3)-Mn(1)	177.2(5)
O(4)-C(4)-Mn(1)	178.1(5)	C(5)-S(1)-Mn(1)#1	110.5(2)
C(5)-S(1)-Mn(1)	110.6(2)	Mn(1)#1-S(1)-Mn(1)	97.30(5)

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y,-z

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for 4.9

The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Mn(1)	55(1)	40(1)	29(1)	0(1)	16(1)	1(1)
C(1)	74(4)	47(3)	42(3)	4(3)	23(3)	5(3)
O(1)	113(3)	77(3)	49(3)	-11(2)	44(3)	11(2)
C(2)	73(4)	53(3)	36(3)	0(3)	19(3)	11(3)
O(2)	84(3)	76(3)	58(3)	4(2)	5(2)	22(2)
C(3)	56(3)	52(3)	52(4)	-12(3)	22(3)	-1(3)
O(3)	83(3)	76(3)	94(3)	-21(3)	39(3)	-33(3)
C(4)	63(3)	49(3)	40(3)	4(2)	24(3)	5(3)
O(4)	80(3)	63(3)	89(3)	16(2)	37(2)	-10(2)
S(1)	53(1)	34(1)	36(1)	-1(1)	19(1)	2(1)
C(5)	84(4)	37(3)	68(4)	-1(3)	33(3)	7(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 2.9

	x	y	z	U(eq)
H(5A)	330(10)	-2105(3)	37(6)	73
H(5B)	2964(10)	-2171(3)	919(6)	73
H(5C)	2189(10)	-2192(3)	-800(6)	73

Planes for ~~cadiz~~ 9

Least-squares plane number 1 (XO = orthogonal, x = Crystal coordinates)
Plane through manganese and sulfur

0.5641 XO + 0.8018 YO + -0.1970 ZO = 0.0000
3.614 x + 11.419 y + -1.883 z = 0.0000

	Deviation	Weight	
+	0.0000	1.0000	MN1
	0.0875	1.0000	C1
	0.1542	1.0000	O1
	-0.1024	1.0000	C2
	-0.1653	1.0000	O2
	1.8562	1.0000	C3
	2.9855	1.0000	O3
	-1.8486	1.0000	C4
	-2.9812	1.0000	O4
+	0.0000	1.0000	S1
	-1.5400	1.0000	C5
	-2.2915	1.0000	H5A
	-1.5803	1.0000	H5B
	-1.5619	1.0000	H5C
+	0.0000	1.0000	MN1A
	-0.0875	1.0000	C1A
	-0.1543	1.0000	O1A
	0.1024	1.0000	C2A
	0.1653	1.0000	O2A
	-1.8561	1.0000	C3A
	-2.9854	1.0000	O3A
	1.8486	1.0000	C4A
	2.9812	1.0000	O4A
+	0.0000	1.0000	S1A
	1.5400	1.0000	C5A
	2.2915	1.0000	H5AA
	1.5802	1.0000	H5BA
	1.5619	1.0000	H5CA

Mean deviation from plane = 0.0000 Angstroms

Least-squares plane number 2 (XO = orthogonal, x = Crystal coordinates)
Plane through manganes and "orthogonal" carbonyls

0.7345 XO + -0.6439 YO + -0.2141 ZO = 0.0000
4.629 x + -9.170 y + -2.046 z = 0.0000

	Deviation	Weight	
+	0.0151	1.0000	MN1
	-1.3011	1.0000	C1
	-2.1208	1.0000	O1
	1.3662	1.0000	C2
	2.2165	1.0000	O2
+	0.0200	1.0000	C3

+	-0.0220	1.0000	O3
+	-0.0097	1.0000	C4
+	-0.0036	1.0000	O4
	1.5718	1.0000	S1
	2.6266	1.0000	C5
	2.0753	1.0000	H5A
	3.1744	1.0000	H5B
	3.1872	1.0000	H5C
+	-0.0151	1.0000	MN1A
	1.3011	1.0000	C1A
	2.1209	1.0000	O1A
	-1.3661	1.0000	C2A
	-2.2165	1.0000	O2A
+	-0.0200	1.0000	C3A
+	0.0220	1.0000	O3A
+	0.0097	1.0000	C4A
+	0.0036	1.0000	O4A
	-1.5718	1.0000	S1A
	-2.6266	1.0000	C5A
	-2.0753	1.0000	H5AA
	-3.1744	1.0000	H5BA
	-3.1872	1.0000	H5CA

Mean deviation from plane = 0.0141 Angstroms

Angles to previous planes:

1: 93.4

Selected torsion angles FOR ~~CMD12~~ 9

C2 - Mn1 - C1 - O1	68.26	(46.15)
C4 - Mn1 - C1 - O1	159.13	(46.14)
C3 - Mn1 - C1 - O1	-23.08	(46.15)
S1_$1 - Mn1 - C1 - O1	-114.68	(46.13)
S1 - Mn1 - C1 - O1	-87.67	(46.37)
C1 - Mn1 - C2 - O2	-20.62	(99.99)
C4 - Mn1 - C2 - O2	-111.38	(99.99)
C3 - Mn1 - C2 - O2	70.06	(99.99)
S1_$1 - Mn1 - C2 - O2	-176.01	(99.99)
S1 - Mn1 - C2 - O2	156.92	(99.99)
C2 - Mn1 - C3 - O3	-103.94	(11.56)
C1 - Mn1 - C3 - O3	-9.41	(11.55)
C4 - Mn1 - C3 - O3	110.36	(11.52)
S1_$1 - Mn1 - C3 - O3	82.53	(11.55)
S1 - Mn1 - C3 - O3	165.13	(11.56)
C2 - Mn1 - C4 - O4	12.81	(14.17)
C1 - Mn1 - C4 - O4	-81.71	(14.18)
C3 - Mn1 - C4 - O4	158.52	(12.60)
S1_$1 - Mn1 - C4 - O4	-173.60	(14.17)
S1 - Mn1 - C4 - O4	103.83	(14.16)
C2 - Mn1 - S1 - C5	61.60	(0.26)
C1 - Mn1 - S1 - C5	-142.39	(1.66)
C4 - Mn1 - S1 - C5	-29.24	(0.26)
C3 - Mn1 - S1 - C5	152.84	(0.27)
S1_$1 - Mn1 - S1 - C5	-115.16	(0.21)
C2 - Mn1 - S1 - Mn1_$1	176.76	(0.17)
C1 - Mn1 - S1 - Mn1_$1	-27.23	(1.66)
C4 - Mn1 - S1 - Mn1_$1	85.92	(0.17)
C3 - Mn1 - S1 - Mn1_$1	-92.00	(0.18)
S1_$1 - Mn1 - S1 - Mn1_$1	0.00	

Operator for generating equivalent atoms:
$1 -x, -y, -z

Table 1. Crystal data and structure refinement for 12b

Identification code	cad16
Empirical formula	$C_{16}H_8Mn_2O_7S$
Formula weight	454.16
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 8.2343(2) Å alpha = 90° b = 22.1349(8) Å beta = 99.5070(10)° c = 9.7080(4) Å gamma = 90°
Volume, Z	1745.13(10) $Å^3$, 4
Density (calculated)	1.729 Mg/m^3
Absorption coefficient	1.602 mm^{-1}
F(000)	904
Crystal size	0.03 x 0.14 x 0.22 mm
θ range for data collection	1.84 to 26.76°
Limiting indices	$-8 \le h \le 10$, $-22 \le k \le 27$, $-11 \le l \le 12$
Reflections collected	9871
Independent reflections	3545 (R_{int} = 0.0934)
Absorption correction	Empirical (SADABS)
Max. and min. transmission	0.962 and 0.493
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3543 / 0 / 236
Goodness-of-fit on F^2	0.941
Final R indices [$I>2\sigma(I)$]	R1 = 0.0782, wR2 = 0.1774
R indices (all data)	R1 = 0.1532, wR2 = 0.2161
Largest diff. peak and hole	1.154 and -1.045 $eÅ^{-3}$

***** Notes for writing up 12b ~~cadi6~~ *****

The crystal was a very thin, clear red plate. X-ray data collection was carried out using a Siemens P4 single-crystal diffractometer equipped with a CCD area detector and controlled by SMART version 4 (1) software. Data reduction was carried out by SAINT version 4 (1) and included profile analysis; this was followed by absorption correction by use of the program SADABS (2).

The structure was determined by direct methods and refined on F squared by use of programs in the SHELXTL PC version 5 (3) package, which were also used for all figures. Seven of eight expected hydrogen atoms appeared in difference maps, and were introduced in ideal positions, riding on the carbon atom to which they are bonded; each was refined with isotropic temperature factor 20% to 50% greater than that of the ridden atom. All other atoms were refined with anisotropic thermal parameters. The final R values are somewhat high because of the low intensities from such a samll crystal.

The molecule is very similar to the earlier one of the same molecule without the methyl group on C(2), but the space groups are different: monoclinic for the current structure, triclinic for the earlier one.

Acknowledgement

The X-ray equipment was purchased with assistance from an instrument grant from the National Science Foundation (CHE-8206423) and a grant from the National Institutes of Health (RR-06462).

References

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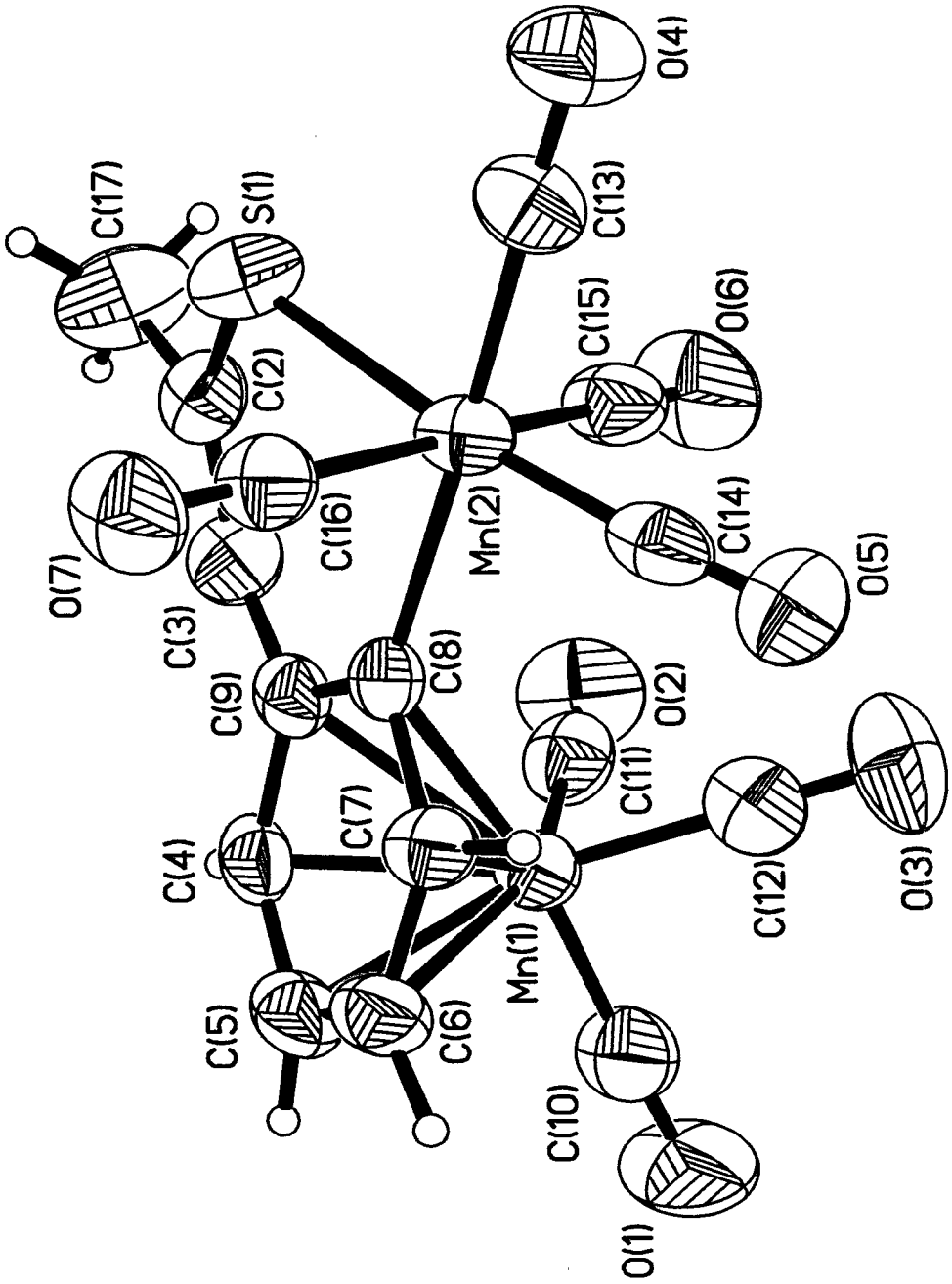
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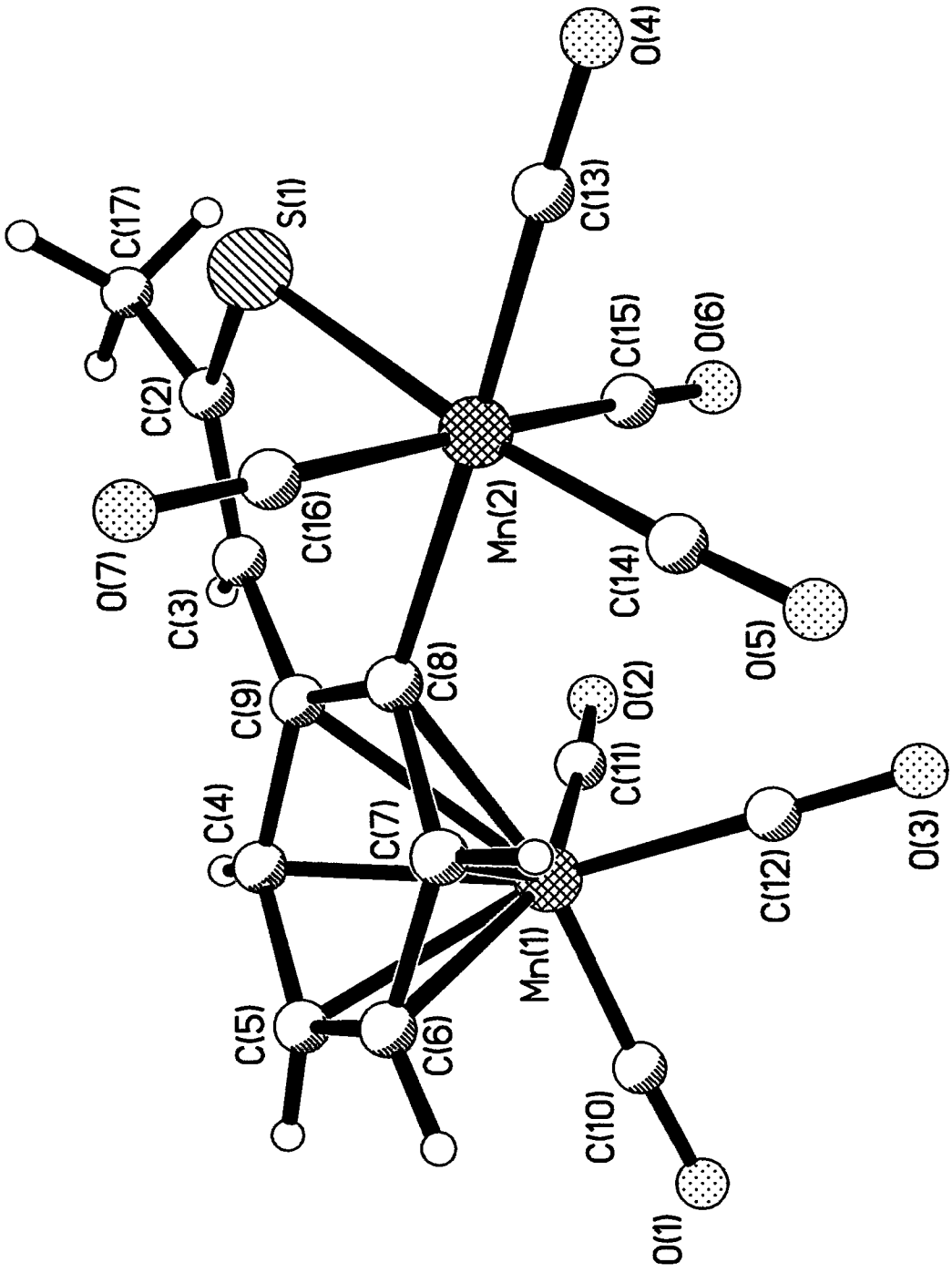
0.88 Å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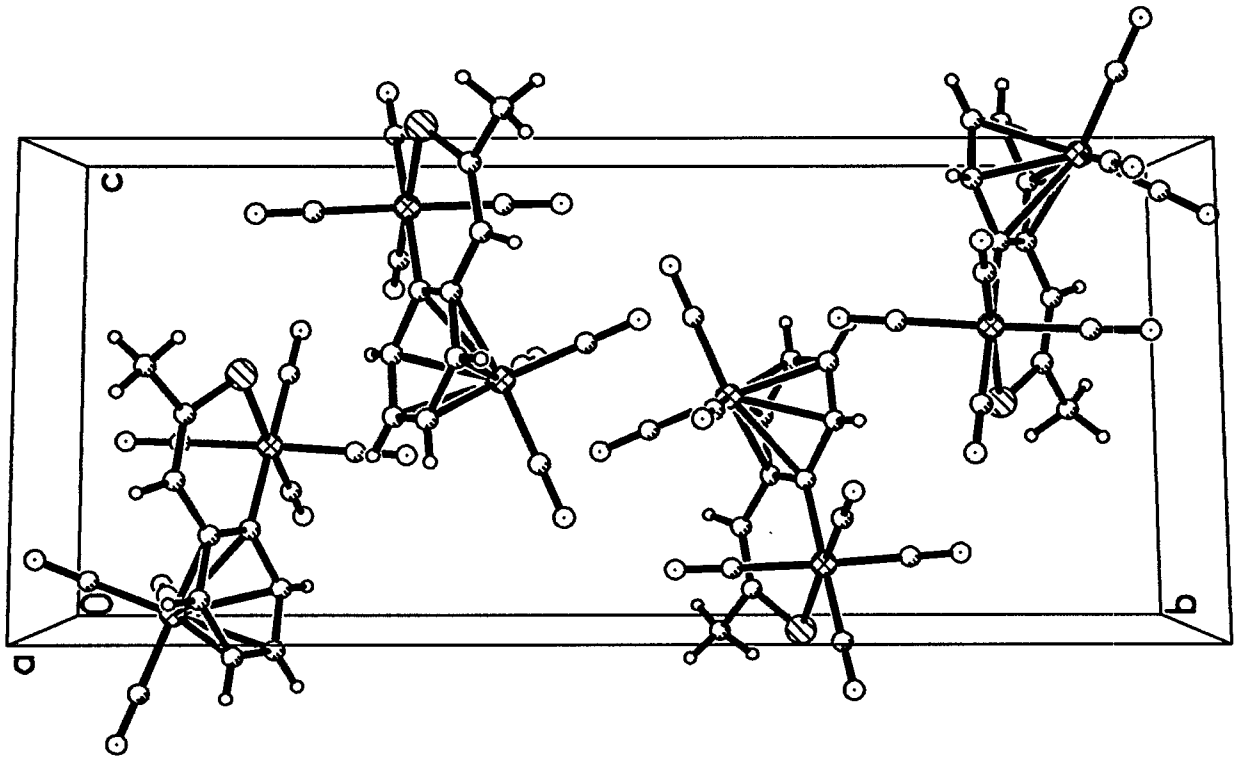
c
a
b

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 12b. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Mn(1)	2610(2)	999(1)	179(1)	44(1)
Mn(2)	1956(1)	1825(1)	3753(1)	41(1)
S(1)	4453(3)	1657(1)	5293(2)	56(1)
C(2)	5744(10)	1223(4)	4472(8)	50(2)
C(3)	5620(10)	1131(4)	3093(8)	54(2)
C(4)	5140(10)	1338(4)	610(8)	49(2)
C(5)	4285(12)	1602(4)	-616(9)	60(2)
C(6)	2862(11)	1913(4)	-570(8)	55(2)
C(7)	2239(10)	1947(3)	695(7)	43(2)
C(8)	3053(9)	1691(3)	1984(7)	39(2)
C(9)	4569(9)	1396(3)	1922(8)	42(2)
C(10)	2156(13)	680(4)	-1539(10)	71(3)
O(1)	1809(12)	474(4)	-2639(7)	118(3)
C(11)	3294(11)	273(4)	911(9)	58(2)
O(2)	3782(10)	-173(3)	1381(7)	87(2)
C(12)	532(13)	841(4)	456(8)	54(2)
O(3)	-762(9)	729(3)	638(8)	83(2)
C(13)	1056(10)	1958(4)	5326(9)	51(2)
O(4)	460(8)	2041(3)	6287(6)	72(2)
C(14)	-77(12)	1947(3)	2718(8)	46(2)
O(5)	-1369(8)	2019(3)	2128(6)	64(2)
C(15)	1663(11)	993(4)	3827(8)	56(2)
O(6)	1493(9)	491(3)	3846(7)	88(2)
C(16)	2630(10)	2617(4)	3632(8)	46(2)
O(7)	3132(8)	3092(3)	3567(6)	66(2)
C(17)	7143(14)	940(5)	5467(10)	87(3)

Table 3. Bond lengths [Å] and angles [°] for 12b

Mn(1)-C(10)	1.793(9)	Mn(1)-C(11)	1.809(10)
Mn(1)-C(12)	1.810(10)	Mn(1)-C(5)	2.151(9)
Mn(1)-C(6)	2.169(8)	Mn(1)-C(4)	2.188(7)
Mn(1)-C(7)	2.190(7)	Mn(1)-C(9)	2.310(7)
Mn(1)-C(8)	2.311(7)	Mn(2)-C(14)	1.824(10)
Mn(2)-C(13)	1.827(8)	Mn(2)-C(16)	1.849(8)
Mn(2)-C(15)	1.860(9)	Mn(2)-C(8)	2.090(7)
Mn(2)-S(1)	2.363(2)	S(1)-C(2)	1.723(9)
C(2)-C(3)	1.341(10)	C(2)-C(17)	1.511(12)
C(3)-C(9)	1.434(10)	C(4)-C(5)	1.406(12)
C(4)-C(9)	1.434(10)	C(5)-C(6)	1.366(12)
C(6)-C(7)	1.409(11)	C(7)-C(8)	1.435(10)
C(8)-C(9)	1.420(11)	C(10)-O(1)	1.153(10)
C(11)-O(2)	1.135(10)	C(12)-O(3)	1.136(10)
C(13)-O(4)	1.139(9)	C(14)-O(5)	1.133(9)
C(15)-O(6)	1.120(9)	C(16)-O(7)	1.136(9)
C(10)-Mn(1)-C(11)	91.0(4)	C(10)-Mn(1)-C(12)	90.3(4)
C(11)-Mn(1)-C(12)	90.6(4)	C(10)-Mn(1)-C(5)	87.6(4)
C(11)-Mn(1)-C(5)	121.0(4)	C(12)-Mn(1)-C(5)	148.4(4)
C(10)-Mn(1)-C(6)	94.2(4)	C(11)-Mn(1)-C(6)	156.7(4)
C(12)-Mn(1)-C(6)	112.1(4)	C(5)-Mn(1)-C(6)	36.8(3)
C(10)-Mn(1)-C(4)	111.0(4)	C(11)-Mn(1)-C(4)	90.3(4)
C(12)-Mn(1)-C(4)	158.6(3)	C(5)-Mn(1)-C(4)	37.8(3)
C(6)-Mn(1)-C(4)	66.7(3)	C(10)-Mn(1)-C(7)	125.1(4)
C(11)-Mn(1)-C(7)	143.8(3)	C(12)-Mn(1)-C(7)	88.9(3)
C(5)-Mn(1)-C(7)	67.1(3)	C(6)-Mn(1)-C(7)	37.7(3)
C(4)-Mn(1)-C(7)	78.0(3)	C(10)-Mn(1)-C(9)	147.6(4)
C(11)-Mn(1)-C(9)	85.1(3)	C(12)-Mn(1)-C(9)	121.8(3)
C(5)-Mn(1)-C(9)	67.4(3)	C(6)-Mn(1)-C(9)	78.3(3)
C(4)-Mn(1)-C(9)	37.1(3)	C(7)-Mn(1)-C(9)	64.9(3)
C(10)-Mn(1)-C(8)	161.7(4)	C(11)-Mn(1)-C(8)	106.9(3)
C(12)-Mn(1)-C(8)	92.9(3)	C(5)-Mn(1)-C(8)	80.3(3)
C(6)-Mn(1)-C(8)	68.0(3)	C(4)-Mn(1)-C(8)	66.5(3)
C(7)-Mn(1)-C(8)	37.1(2)	C(9)-Mn(1)-C(8)	35.8(3)
C(14)-Mn(2)-C(13)	88.5(3)	C(14)-Mn(2)-C(16)	94.7(3)
C(13)-Mn(2)-C(16)	93.9(4)	C(14)-Mn(2)-C(15)	93.2(4)
C(13)-Mn(2)-C(15)	93.2(4)	C(16)-Mn(2)-C(15)	169.6(4)
C(14)-Mn(2)-C(8)	92.9(3)	C(13)-Mn(2)-C(8)	178.0(3)
C(16)-Mn(2)-C(8)	84.6(3)	C(15)-Mn(2)-C(8)	88.1(3)
C(14)-Mn(2)-S(1)	174.2(2)	C(13)-Mn(2)-S(1)	85.9(3)
C(16)-Mn(2)-S(1)	87.2(2)	C(15)-Mn(2)-S(1)	85.7(3)
C(8)-Mn(2)-S(1)	92.8(2)	C(2)-S(1)-Mn(2)	109.5(3)
C(3)-C(2)-C(17)	120.0(8)	C(3)-C(2)-S(1)	126.5(6)
C(17)-C(2)-S(1)	113.5(6)	C(2)-C(3)-C(9)	131.7(8)
C(5)-C(4)-C(9)	121.6(8)	C(5)-C(4)-Mn(1)	69.7(5)
C(9)-C(4)-Mn(1)	76.1(4)	C(6)-C(5)-C(4)	119.6(8)
C(6)-C(5)-Mn(1)	72.3(5)	C(4)-C(5)-Mn(1)	72.5(5)
C(5)-C(6)-C(7)	119.6(8)	C(5)-C(6)-Mn(1)	70.8(5)
C(7)-C(6)-Mn(1)	71.9(4)	C(6)-C(7)-C(8)	123.6(7)
C(6)-C(7)-Mn(1)	70.4(4)	C(8)-C(7)-Mn(1)	76.0(4)
C(9)-C(8)-C(7)	115.6(7)	C(9)-C(8)-Mn(2)	127.1(5)
C(7)-C(8)-Mn(2)	117.2(6)	C(9)-C(8)-Mn(1)	72.1(4)
C(7)-C(8)-Mn(1)	66.9(4)	Mn(2)-C(8)-Mn(1)	133.0(3)

C(8)-C(9)-C(4)	119.9(7)	C(8)-C(9)-C(3)	125.0(7)
C(4)-C(9)-C(3)	115.1(7)	C(8)-C(9)-Mn(1)	72.1(4)
C(4)-C(9)-Mn(1)	66.9(4)	C(3)-C(9)-Mn(1)	133.0(5)
O(1)-C(10)-Mn(1)	177.7(10)	O(2)-C(11)-Mn(1)	177.4(8)
O(3)-C(12)-Mn(1)	178.5(8)	O(4)-C(13)-Mn(2)	178.4(8)
O(5)-C(14)-Mn(2)	176.9(7)	O(6)-C(15)-Mn(2)	178.7(8)
O(7)-C(16)-Mn(2)	176.2(8)		

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for 12b

The anisotropic displacement factor exponent takes the form:

$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Mn(1)	59(1)	36(1)	40(1)	-7(1)	11(1)	-8(1)
Mn(2)	57(1)	30(1)	36(1)	0(1)	12(1)	3(1)
S(1)	71(2)	53(1)	41(1)	-5(1)	2(1)	12(1)
C(2)	54(5)	36(4)	56(5)	2(4)	1(4)	0(4)
C(3)	62(6)	47(5)	55(5)	-10(4)	13(4)	3(4)
C(4)	46(5)	51(5)	53(5)	-14(4)	18(4)	-17(4)
C(5)	74(7)	65(6)	47(5)	-12(4)	28(4)	-22(5)
C(6)	78(7)	45(5)	42(5)	7(4)	12(4)	-12(4)
C(7)	61(5)	28(4)	38(4)	-5(3)	10(4)	-1(3)
C(8)	45(4)	31(4)	40(4)	-3(3)	6(3)	-9(3)
C(9)	49(5)	32(4)	46(4)	-3(3)	12(3)	-9(4)
C(10)	99(8)	54(6)	61(6)	-5(5)	14(5)	-25(5)
O(1)	196(9)	107(7)	52(4)	-29(4)	23(5)	-64(6)
C(11)	57(6)	57(6)	59(5)	-14(5)	9(4)	-1(5)
O(2)	126(6)	40(4)	96(5)	1(4)	17(4)	16(4)
C(12)	70(6)	36(5)	59(5)	-11(4)	20(5)	-2(4)
O(3)	71(5)	63(5)	122(6)	-12(4)	35(4)	-21(4)
C(13)	55(5)	46(5)	55(5)	1(4)	21(4)	9(4)
O(4)	80(5)	83(5)	56(4)	-5(3)	24(3)	10(4)
C(14)	74(6)	31(4)	39(4)	-4(3)	25(4)	-10(4)
O(5)	56(4)	70(4)	64(4)	-1(3)	1(3)	0(3)
C(15)	82(7)	44(5)	42(4)	-1(4)	13(4)	-11(4)
O(6)	136(7)	35(4)	99(5)	6(4)	33(5)	-21(4)
C(16)	56(5)	37(5)	46(4)	-2(4)	9(4)	-1(4)
O(7)	88(5)	37(4)	74(4)	2(3)	21(3)	-9(3)
C(17)	102(9)	84(8)	70(7)	-2(6)	-3(6)	33(6)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 12b

	x	y	z	U(eq)
H(3A)	6348(10)	844(4)	2851(8)	65
H(4A)	6098(10)	1122(4)	570(8)	59
H(5A)	4686(12)	1565(4)	-1453(9)	72
H(6A)	2308(11)	2100(4)	-1368(8)	66
H(7A)	1245(10)	2147(3)	695(7)	51
H(17A)	6709(16)	654(24)	6053(52)	131
H(17B)	7735(56)	1249(6)	6035(53)	131
H(17C)	7874(51)	738(29)	4943(10)	131

Selected torsion angles for ~~cad16~~ Pb

C14	-	Mn2	-	S1	-	C2	142.40 (2.40)
C13	-	Mn2	-	S1	-	C2	157.26 (0.41)
C16	-	Mn2	-	S1	-	C2	-108.64 (0.39)
C15	-	Mn2	-	S1	-	C2	63.77 (0.40)
C8	-	Mn2	-	S1	-	C2	-24.15 (0.37)
Mn2	-	S1	-	C2	-	C3	17.83 (0.87)
Mn2	-	S1	-	C2	-	C17	-163.30 (0.64)
C17	-	C2	-	C3	-	C9	-171.28 (0.91)
S1	-	C2	-	C3	-	C9	7.52 (1.44)
C10	-	Mn1	-	C4	-	C5	55.39 (0.64)
C11	-	Mn1	-	C4	-	C5	146.61 (0.55)
C12	-	Mn1	-	C4	-	C5	-121.16 (0.98)
C6	-	Mn1	-	C4	-	C5	-29.99 (0.50)
C7	-	Mn1	-	C4	-	C5	-67.89 (0.52)
C9	-	Mn1	-	C4	-	C5	-131.84 (0.74)
C8	-	Mn1	-	C4	-	C5	-105.02 (0.56)
C10	-	Mn1	-	C4	-	C9	-172.77 (0.52)
C11	-	Mn1	-	C4	-	C9	-81.54 (0.51)
C12	-	Mn1	-	C4	-	C9	10.68 (1.19)
C5	-	Mn1	-	C4	-	C9	131.84 (0.74)
C6	-	Mn1	-	C4	-	C9	101.85 (0.52)
C7	-	Mn1	-	C4	-	C9	63.95 (0.46)
C8	-	Mn1	-	C4	-	C9	26.82 (0.42)
C9	-	C4	-	C5	-	C6	-1.09 (1.22)
Mn1	-	C4	-	C5	-	C6	56.99 (0.74)
C9	-	C4	-	C5	-	Mn1	-58.07 (0.66)
C10	-	Mn1	-	C5	-	C6	100.20 (0.58)
C11	-	Mn1	-	C5	-	C6	-170.00 (0.50)
C12	-	Mn1	-	C5	-	C6	13.47 (0.89)
C4	-	Mn1	-	C5	-	C6	-130.05 (0.74)
C7	-	Mn1	-	C5	-	C6	-29.81 (0.46)
C9	-	Mn1	-	C5	-	C6	-100.96 (0.54)
C8	-	Mn1	-	C5	-	C6	-66.05 (0.49)
C10	-	Mn1	-	C5	-	C4	-129.75 (0.59)
C11	-	Mn1	-	C5	-	C4	-39.95 (0.61)
C12	-	Mn1	-	C5	-	C4	143.52 (0.63)
C6	-	Mn1	-	C5	-	C4	130.05 (0.74)
C7	-	Mn1	-	C5	-	C4	100.24 (0.51)
C9	-	Mn1	-	C5	-	C4	29.09 (0.45)
C8	-	Mn1	-	C5	-	C4	64.00 (0.49)
C4	-	C5	-	C6	-	C7	-2.19 (1.24)
Mn1	-	C5	-	C6	-	C7	54.91 (0.67)
C4	-	C5	-	C6	-	Mn1	-57.10 (0.72)
C10	-	Mn1	-	C6	-	C5	-80.40 (0.59)
C11	-	Mn1	-	C6	-	C5	22.11 (1.10)
C12	-	Mn1	-	C6	-	C5	-172.43 (0.51)
C4	-	Mn1	-	C6	-	C5	30.72 (0.47)
C7	-	Mn1	-	C6	-	C5	131.54 (0.73)
C9	-	Mn1	-	C6	-	C5	67.74 (0.50)
C8	-	Mn1	-	C6	-	C5	103.65 (0.54)
C10	-	Mn1	-	C6	-	C7	148.06 (0.55)
C11	-	Mn1	-	C6	-	C7	-109.43 (0.93)
C12	-	Mn1	-	C6	-	C7	56.03 (0.55)
C5	-	Mn1	-	C6	-	C7	-131.54 (0.73)
C4	-	Mn1	-	C6	-	C7	-100.82 (0.49)
C9	-	Mn1	-	C6	-	C7	-63.80 (0.47)
C8	-	Mn1	-	C6	-	C7	-27.89 (0.44)
C5	-	C6	-	C7	-	C8	2.59 (1.17)
Mn1	-	C6	-	C7	-	C8	56.99 (0.64)
C5	-	C6	-	C7	-	Mn1	-54.40 (0.71)
C10	-	Mn1	-	C7	-	C6	-40.18 (0.68)
C11	-	Mn1	-	C7	-	C6	140.84 (0.65)
C12	-	Mn1	-	C7	-	C6	-129.75 (0.54)
C5	-	Mn1	-	C7	-	C6	29.17 (0.48)
C4	-	Mn1	-	C7	-	C6	67.23 (0.49)
C9	-	Mn1	-	C7	-	C6	103.95 (0.53)
C8	-	Mn1	-	C7	-	C6	133.98 (0.71)
C10	-	Mn1	-	C7	-	C8	-174.17 (0.53)
C11	-	Mn1	-	C7	-	C8	6.86 (0.80)
C12	-	Mn1	-	C7	-	C8	96.26 (0.50)
C5	-	Mn1	-	C7	-	C8	-104.81 (0.52)
C6	-	Mn1	-	C7	-	C8	-133.98 (0.71)
C4	-	Mn1	-	C7	-	C8	-66.75 (0.46)
C9	-	Mn1	-	C7	-	C8	-30.03 (0.42)
C6	-	C7	-	C8	-	C9	0.39 (1.01)
Mn1	-	C7	-	C8	-	C9	54.86 (0.56)
C6	-	C7	-	C8	-	Mn2	177.64 (0.56)
Mn1	-	C7	-	C8	-	Mn2	-127.90 (0.41)
C6	-	C7	-	C8	-	Mn1	-54.47 (0.63)
C14	-	Mn2	-	C8	-	C9	-159.84 (0.64)
C13	-	Mn2	-	C8	-	C9	64.78 (9.53)
C16	-	Mn2	-	C8	-	C9	105.69 (0.65)
C15	-	Mn2	-	C8	-	C9	-66.78 (0.66)
S1	-	Mn2	-	C8	-	C9	18.81 (0.62)
C14	-	Mn2	-	C8	-	C7	23.27 (0.57)
C13	-	Mn2	-	C8	-	C7	-112.11 (9.28)
C16	-	Mn2	-	C8	-	C7	-71.19 (0.56)
C15	-	Mn2	-	C8	-	C7	116.34 (0.59)
S1	-	Mn2	-	C8	-	C7	-158.08 (0.50)
C14	-	Mn2	-	C8	-	Mn1	-59.65 (0.50)
C13	-	Mn2	-	C8	-	Mn1	164.98 (9.11)
C16	-	Mn2	-	C8	-	Mn1	-154.11 (0.52)
C15	-	Mn2	-	C8	-	Mn1	33.42 (0.51)

Planes for ~~cad16~~ 12b

Least-squares plane number 1 (XO = orthogonal, x = Crystal coordinates)
The benzene ring

0.5095 XO + 0.8514 YO + 0.1250 ZO = 4.6512
3.967 x + 18.845 y + 1.213 z = 4.6512

	Deviation	Weight	
	-1.7107	1.0000	MN1
	0.0190	1.0000	MN2
	0.8799	1.0000	S1
	0.4748	1.0000	C2
	0.0858	1.0000	C3
	-0.1959	1.0000	H3A
+	-0.0157	1.0000	C4
	-0.0492	1.0000	H4A
+	-0.0068	1.0000	C5
	-0.0188	1.0000	H5A
+	0.0195	1.0000	C6
	0.0564	1.0000	H6A
+	-0.0096	1.0000	C7
	-0.0278	1.0000	H7A
+	-0.0125	1.0000	C8
+	0.0251	1.0000	C9
	-2.7013	1.0000	C10
	-3.3610	1.0000	O1
	-2.7187	1.0000	C11
	-3.3099	1.0000	O2
	-2.8002	1.0000	C12
	-3.5031	1.0000	O3
	0.1038	1.0000	C13
	0.1405	1.0000	O4
	-0.6829	1.0000	C14
	-1.1305	1.0000	O5
	-1.6563	1.0000	C15
	-2.6671	1.0000	O6
	1.7648	1.0000	C16
	2.8520	1.0000	O7
	0.6181	1.0000	C17
	-0.0230	1.0000	H17A
	1.5044	1.0000	H17B
	0.4636	1.0000	H17C

Mean deviation from plane = 0.0149 Angstroms

Least-squares plane number 2 (XO = orthogonal, x = Crystal coordinates)
The C-C=C-S part from the thiophene ring

0.6248 XO + 0.7708 YO + -0.1242 ZO = 4.5329
5.243 x + 17.063 y + -1.206 z = 4.5329

	Deviation	Weight	
	-1.4807	1.0000	MN1
	-0.8466	1.0000	MN2
+	-0.0097	1.0000	S1
+	0.0260	1.0000	C2
+	-0.0289	1.0000	C3
	-0.1081	1.0000	H3A
	0.3720	1.0000	C4
	0.5093	1.0000	H4A
	0.5216	1.0000	C5
	0.7698	1.0000	H5A
	0.2998	1.0000	C6
	0.4257	1.0000	H6A
	-0.1209	1.0000	C7
	-0.3015	1.0000	H7A
	-0.2862	1.0000	C8
+	0.0125	1.0000	C9
	-2.0567	1.0000	C10
	-2.4579	1.0000	O1
	-2.4493	1.0000	C11
	-3.0123	1.0000	O2
	-2.8744	1.0000	C12
	-3.7664	1.0000	O3
	-1.2807	1.0000	C13
	-1.5673	1.0000	O4
	-1.5790	1.0000	C14
	-2.0616	1.0000	O5
	-2.4286	1.0000	C15
	-3.3762	1.0000	O6
	0.8735	1.0000	C16
	1.9556	1.0000	O7
	0.1575	1.0000	C17
	-0.6299	1.0000	H17A
	0.9266	1.0000	H17B
	0.2589	1.0000	H17C

Mean deviation from plane = 0.0193 Angstroms

Angles to previous planes:

1: 16.5

Table 1. Crystal data and structure refinement for 16

Identification code	xz01a
Empirical formula	$C_{24}H_{25}MnO_4RuS$
Formula weight	565.51
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 9.0923(2) Å alpha = 90.0000(10)°
	b = 13.3934(3) Å beta = 95.3480(10)°
	c = 19.3603(3) Å gamma = 90.0000(10)°
Volume, Z	2347.37(8) $Å^3$, 4
Density (calculated)	1.600 Mg/m^3
Absorption coefficient	1.297 mm^{-1}
F(000)	1144
Crystal size	0.21 x 0.21 x 0.18 mm
θ range for data collection	1.85 to 23.28°
Limiting indices	$-10 \le h \le 9$, $-14 \le k \le 13$, $-21 \le l \le 15$
Reflections collected	8943
Independent reflections	3348 (R_{int} = 0.0364)
Absorption correction	Semi-empirical
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3348 / 0 / 286
Goodness-of-fit on F^2	1.033
Final R indices [$I>2\sigma(I)$]	R1 = 0.0322, wR2 = 0.0802
R indices (all data)	R1 = 0.0393, wR2 = 0.0843
Largest diff. peak and hole	0.343 and -0.546 $eÅ^{-3}$

***** Notes for writing up ~~xz01a~~ *****

The crystal was a clear yellow block. X-ray data collection was carried out using a Siemens P4 single-crystal diffractometer equipped with a CCD area detector and controlled by SMART version 4 (1) software. Data reduction was carried out by SAINT version 4 (1) and SADABS (2), and included profile analysis, correction for intensity changes during data collection, and an empirical absorption correction.

The structure had been determined earlier on a smaller crystal by direct methods. When a larger crystal became available, better quality data were collected, and the structure was further refined on F squared by use of programs in the SHELXTL PC version 5 (3) package, which were also used for all figures. Nineteen of the 25 hydrogen atoms appeared in a difference map, and each was introduced in an ideal position, riding on the atom to which it is bonded; each was refined with isotropic temperature factor, 20% greater than that of the ridden atom for for those on the fused ring part, 50% greater for the methyl hydrogen atoms. All other atoms were refined with anisotropic thermal parameters.

The hexamethylbenzene portion appears to be both tilted (13.0 deg. from the plane of the aromatic part of the plane on the other side of the ruthenium atom) and distorted (C(14) and especially C(20) pushed away from the ruthenium atom) by repulsion between carbonyl oxygen O(3) and methyl carbon C(20). The distance O(3)-C(20) is 3.25 A, somewhat less than the methyl-oxygen van der Waals distance of about 3.4 A. (The next closest carbon to O(3) is C(19) in the adjacent methyl group, at a distance of 3.55 A.)

Acknowledgement

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References

(1) Siemens Industrial Automation Incorporated
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6300 Enterprise Lane
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(2) G. M. Sheldrick (1996) SADABS, Empirical absorption (and other) corrections. Univ. of Goettingen, Germany.

(3) Siemens Analytical X-Ray Instruments, Inc.
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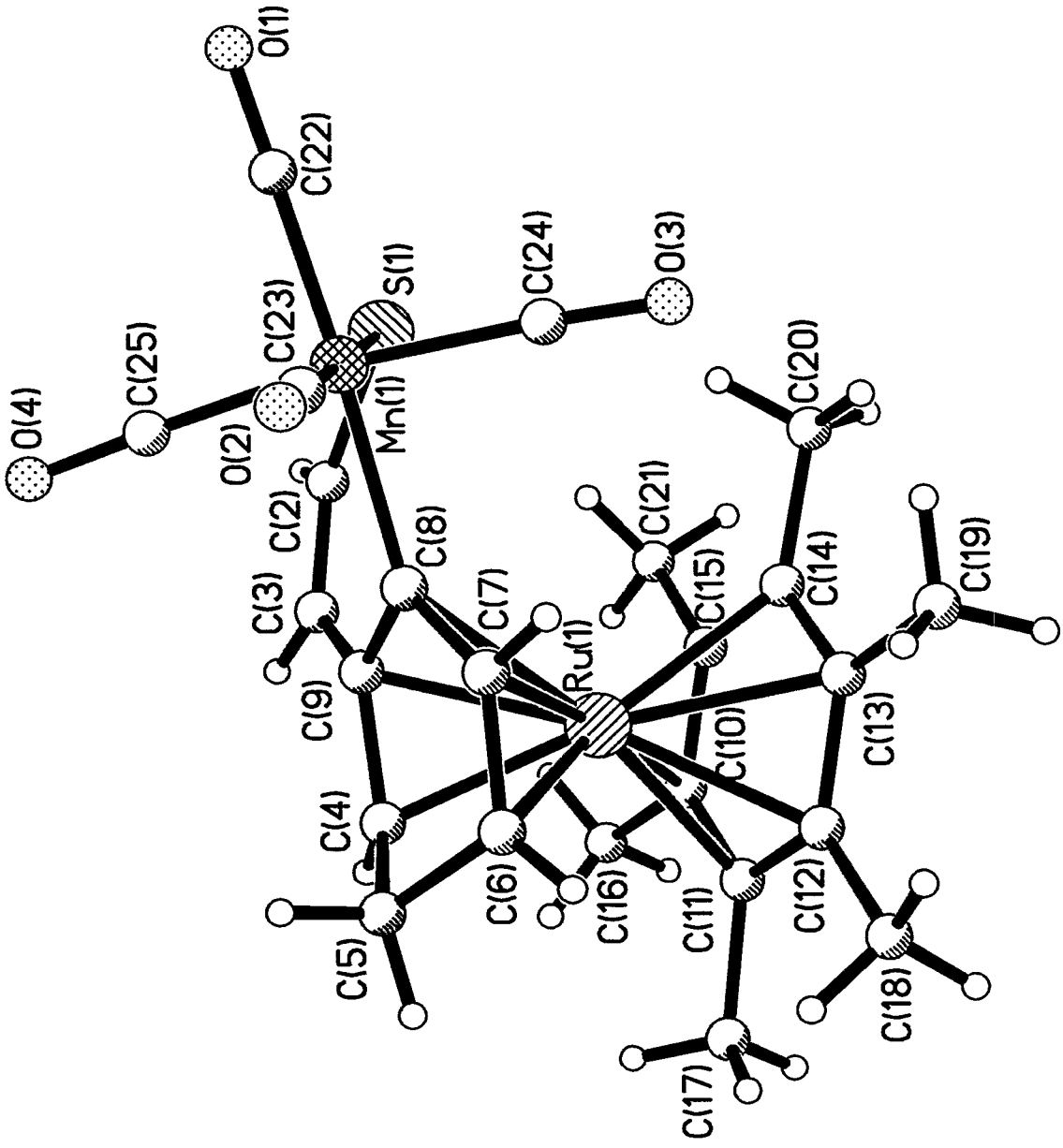
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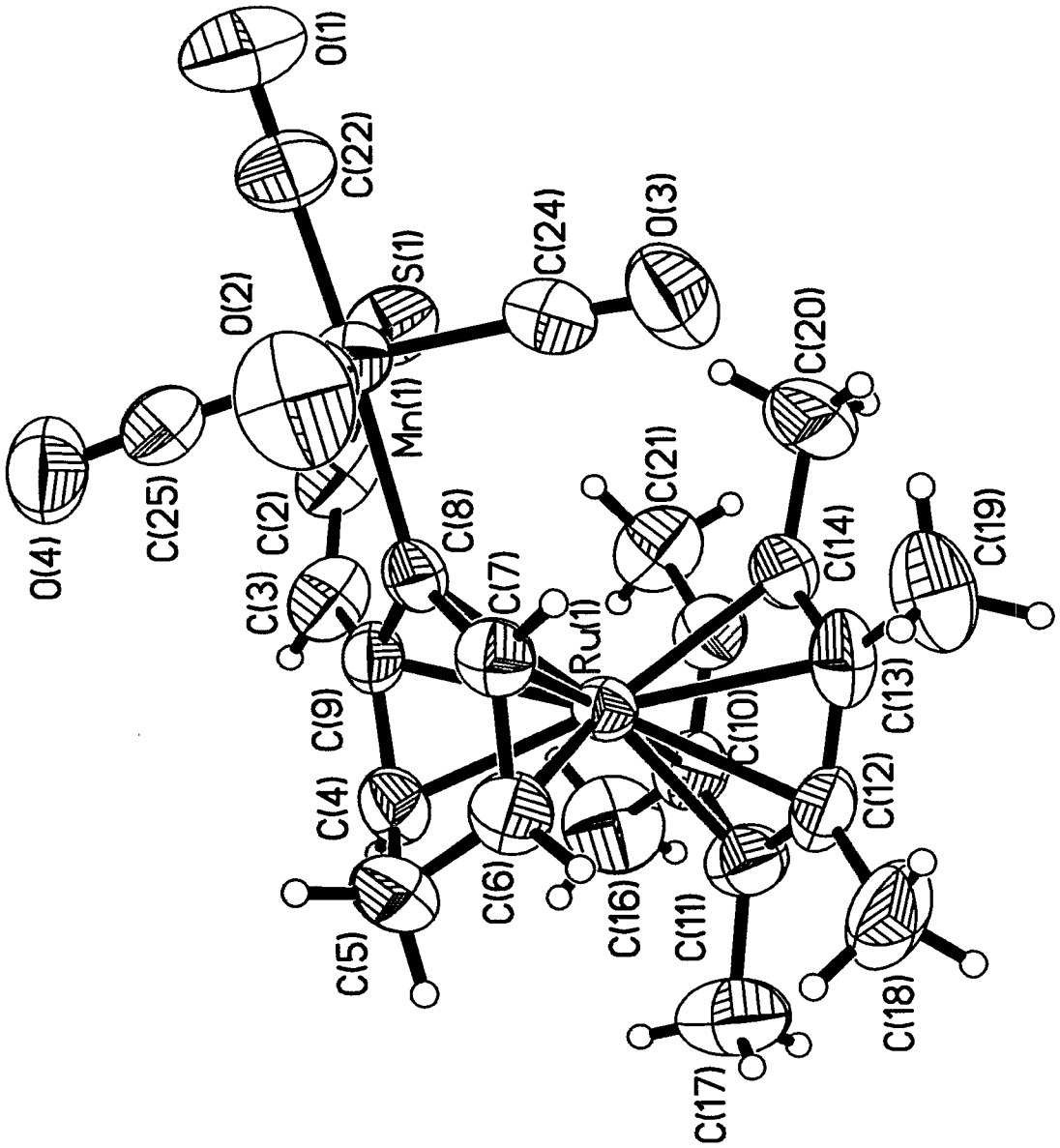
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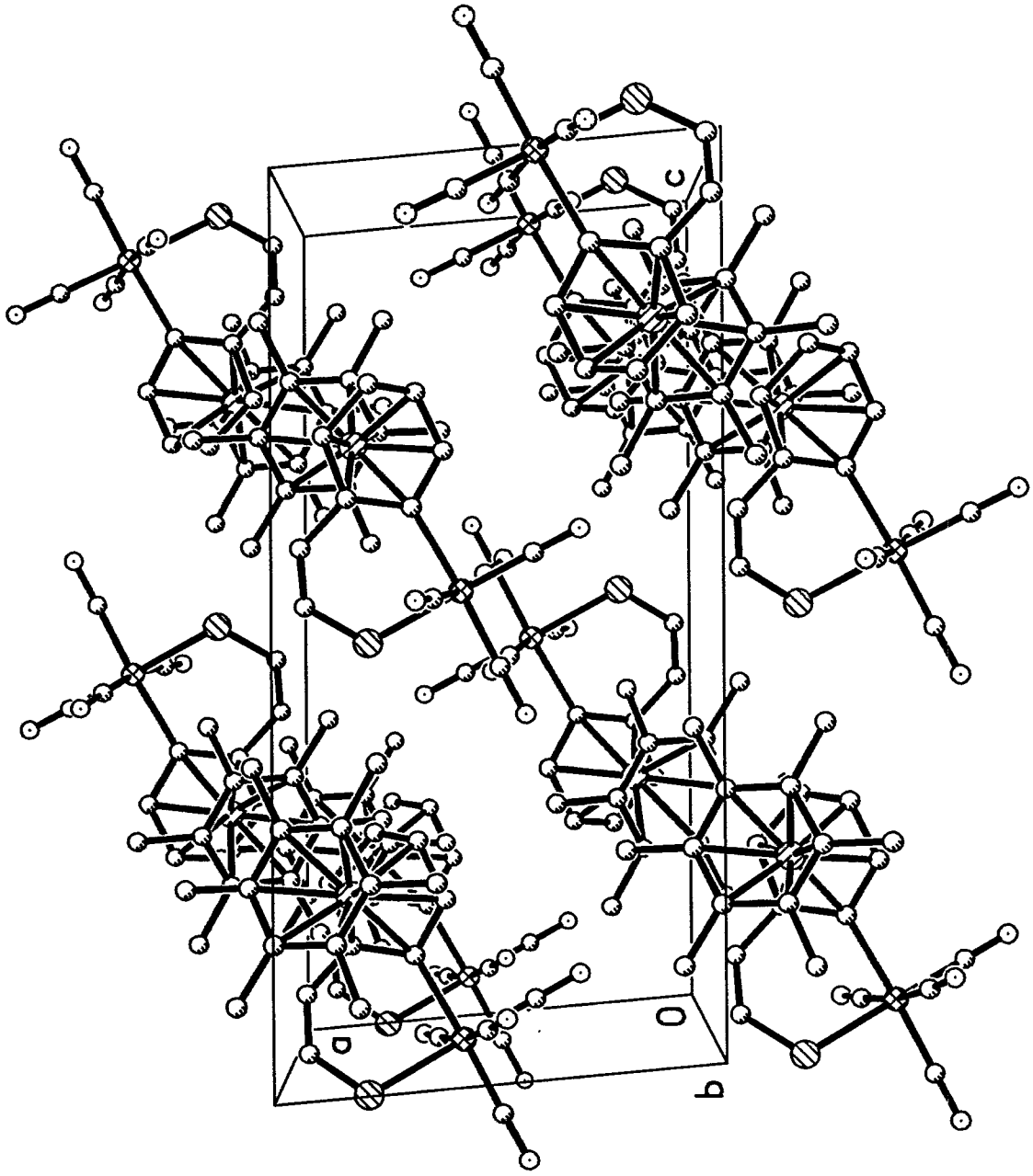
c
a
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b

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 16 U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Ru(1)	1631(1)	3328(1)	2932(1)	40(1)
Mn(1)	4157(1)	2361(1)	4696(1)	47(1)
S(1)	1982(1)	2883(1)	5174(1)	65(1)
C(2)	566(4)	2254(3)	4711(2)	65(1)
C(3)	492(4)	1870(3)	4069(2)	61(1)
C(4)	1031(5)	1763(3)	2850(2)	58(1)
C(5)	2083(6)	1500(3)	2328(3)	75(1)
C(6)	3119(5)	2348(3)	2429(2)	59(1)
C(7)	3749(4)	2571(3)	3105(2)	48(1)
C(8)	3025(4)	2325(2)	3708(2)	40(1)
C(9)	1549(4)	1955(3)	3558(2)	45(1)
C(10)	-628(4)	3976(3)	2714(2)	59(1)
C(11)	109(5)	4025(3)	2105(2)	61(1)
C(12)	1495(5)	4496(3)	2120(2)	61(1)
C(13)	2149(5)	4945(3)	2750(3)	62(1)
C(14)	1382(4)	4914(3)	3358(2)	54(1)
C(15)	-28(4)	4429(3)	3333(2)	53(1)
C(16)	-2114(5)	3434(4)	2702(3)	93(2)
C(17)	-581(6)	3590(5)	1419(3)	97(2)
C(18)	2287(7)	4527(4)	1467(2)	93(2)
C(19)	3622(5)	5473(4)	2772(3)	91(2)
C(20)	1930(6)	5442(4)	4022(2)	81(1)
C(21)	-877(5)	4413(4)	3965(2)	72(1)
C(22)	5045(4)	2334(4)	5567(2)	64(1)
O(1)	5637(4)	2311(3)	6122(2)	96(1)
C(23)	5821(4)	1952(3)	4344(2)	56(1)
O(2)	6871(4)	1734(3)	4123(2)	98(1)
C(24)	4708(4)	3652(4)	4515(2)	57(1)
O(3)	5146(4)	4403(3)	4358(2)	88(1)
C(25)	3537(4)	1058(4)	4752(2)	58(1)
O(4)	3171(4)	241(3)	4762(2)	87(1)

Table 3. Bond lengths [Å] and angles [°] for Mn, 16

Ru(1)-C(4)	2.168(4)	Ru(1)-C(7)	2.174(3)
Ru(1)-C(6)	2.179(4)	Ru(1)-C(9)	2.207(4)
Ru(1)-C(12)	2.214(4)	Ru(1)-C(11)	2.222(4)
Ru(1)-C(10)	2.233(4)	Ru(1)-C(13)	2.252(4)
Ru(1)-C(15)	2.296(4)	Ru(1)-C(14)	2.298(4)
Ru(1)-C(8)	2.305(3)	Mn(1)-C(23)	1.800(4)
Mn(1)-C(22)	1.801(5)	Mn(1)-C(25)	1.840(5)
Mn(1)-C(24)	1.842(5)	Mn(1)-C(8)	2.085(4)
Mn(1)-S(1)	2.3667(12)	S(1)-C(2)	1.720(5)
C(2)-C(3)	1.340(6)	C(3)-C(9)	1.447(6)
C(4)-C(9)	1.431(5)	C(4)-C(5)	1.497(6)
C(5)-C(6)	1.477(6)	C(6)-C(7)	1.411(5)
C(7)-C(8)	1.431(5)	C(8)-C(9)	1.434(5)
C(10)-C(15)	1.408(6)	C(10)-C(11)	1.411(6)
C(10)-C(16)	1.532(6)	C(11)-C(12)	1.407(6)
C(11)-C(17)	1.532(6)	C(12)-C(13)	1.438(6)
C(12)-C(18)	1.513(6)	C(13)-C(14)	1.424(6)
C(13)-C(19)	1.512(6)	C(14)-C(15)	1.433(5)
C(14)-C(20)	1.510(5)	C(15)-C(21)	1.506(5)
C(22)-O(1)	1.156(5)	C(23)-O(2)	1.121(5)
C(24)-O(3)	1.134(5)	C(25)-O(4)	1.144(5)
C(4)-Ru(1)-C(7)	76.9(2)	C(4)-Ru(1)-C(6)	63.1(2)
C(7)-Ru(1)-C(6)	37.82(14)	C(4)-Ru(1)-C(9)	38.18(14)
C(7)-Ru(1)-C(9)	66.53(14)	C(6)-Ru(1)-C(9)	78.4(2)
C(4)-Ru(1)-C(12)	129.4(2)	C(7)-Ru(1)-C(12)	115.4(2)
C(6)-Ru(1)-C(12)	96.0(2)	C(9)-Ru(1)-C(12)	167.6(2)
C(4)-Ru(1)-C(11)	102.6(2)	C(7)-Ru(1)-C(11)	142.2(2)
C(6)-Ru(1)-C(11)	107.6(2)	C(9)-Ru(1)-C(11)	134.1(2)
C(12)-Ru(1)-C(11)	37.0(2)	C(4)-Ru(1)-C(10)	98.1(2)
C(7)-Ru(1)-C(10)	174.81(14)	C(6)-Ru(1)-C(10)	138.4(2)
C(9)-Ru(1)-C(10)	110.50(14)	C(12)-Ru(1)-C(10)	66.5(2)
C(11)-Ru(1)-C(10)	36.9(2)	C(4)-Ru(1)-C(13)	166.8(2)
C(7)-Ru(1)-C(13)	106.1(2)	C(6)-Ru(1)-C(13)	111.2(2)
C(9)-Ru(1)-C(13)	154.9(2)	C(12)-Ru(1)-C(13)	37.6(2)
C(11)-Ru(1)-C(13)	66.9(2)	C(10)-Ru(1)-C(13)	78.4(2)
C(4)-Ru(1)-C(15)	118.5(2)	C(7)-Ru(1)-C(15)	147.96(14)
C(6)-Ru(1)-C(15)	173.20(14)	C(9)-Ru(1)-C(15)	107.02(14)
C(12)-Ru(1)-C(15)	77.9(2)	C(11)-Ru(1)-C(15)	65.75(14)
C(10)-Ru(1)-C(15)	36.19(14)	C(13)-Ru(1)-C(15)	65.7(2)
C(4)-Ru(1)-C(14)	152.6(2)	C(7)-Ru(1)-C(14)	119.43(14)
C(6)-Ru(1)-C(14)	143.4(2)	C(9)-Ru(1)-C(14)	124.3(2)
C(12)-Ru(1)-C(14)	66.4(2)	C(11)-Ru(1)-C(14)	78.1(2)
C(10)-Ru(1)-C(14)	65.7(2)	C(13)-Ru(1)-C(14)	36.5(2)
C(15)-Ru(1)-C(14)	36.36(14)	C(4)-Ru(1)-C(8)	66.66(14)
C(7)-Ru(1)-C(8)	37.11(12)	C(6)-Ru(1)-C(8)	67.29(14)
C(9)-Ru(1)-C(8)	37.00(12)	C(12)-Ru(1)-C(8)	149.9(2)
C(11)-Ru(1)-C(8)	169.22(14)	C(10)-Ru(1)-C(8)	142.19(14)
C(13)-Ru(1)-C(8)	123.5(2)	C(15)-Ru(1)-C(8)	119.52(13)
C(14)-Ru(1)-C(8)	111.80(13)	C(23)-Mn(1)-C(22)	91.6(2)
C(23)-Mn(1)-C(25)	90.4(2)	C(22)-Mn(1)-C(25)	92.2(2)
C(23)-Mn(1)-C(24)	87.7(2)	C(22)-Mn(1)-C(24)	95.2(2)
C(25)-Mn(1)-C(24)	172.4(2)	C(23)-Mn(1)-C(8)	90.4(2)
C(22)-Mn(1)-C(8)	176.2(2)	C(25)-Mn(1)-C(8)	84.5(2)

C(24)-Mn(1)-C(8)	88.1(2)	C(23)-Mn(1)-S(1)	179.05(14)
C(22)-Mn(1)-S(1)	87.54(14)	C(25)-Mn(1)-S(1)	89.16(13)
C(24)-Mn(1)-S(1)	92.79(13)	C(8)-Mn(1)-S(1)	90.39(10)
C(2)-S(1)-Mn(1)	105.3(2)	C(3)-C(2)-S(1)	129.9(3)
C(2)-C(3)-C(9)	128.7(4)	C(9)-C(4)-C(5)	121.0(4)
C(9)-C(4)-Ru(1)	72.4(2)	C(5)-C(4)-Ru(1)	96.0(3)
C(6)-C(5)-C(4)	99.8(3)	C(7)-C(6)-C(5)	118.9(4)
C(7)-C(6)-Ru(1)	70.9(2)	C(5)-C(6)-Ru(1)	96.1(3)
C(6)-C(7)-C(8)	122.1(3)	C(6)-C(7)-Ru(1)	71.3(2)
C(8)-C(7)-Ru(1)	76.4(2)	C(7)-C(8)-C(9)	114.0(3)
C(7)-C(8)-Mn(1)	121.2(2)	C(9)-C(8)-Mn(1)	124.4(3)
C(7)-C(8)-Ru(1)	66.5(2)	C(9)-C(8)-Ru(1)	67.8(2)
Mn(1)-C(8)-Ru(1)	141.1(2)	C(4)-C(9)-C(8)	118.3(3)
C(4)-C(9)-C(3)	117.4(3)	C(8)-C(9)-C(3)	123.9(4)
C(4)-C(9)-Ru(1)	69.4(2)	C(8)-C(9)-Ru(1)	75.2(2)
C(3)-C(9)-Ru(1)	120.0(3)	C(15)-C(10)-C(11)	121.0(4)
C(15)-C(10)-C(16)	119.3(4)	C(11)-C(10)-C(16)	119.7(4)
C(15)-C(10)-Ru(1)	74.4(2)	C(11)-C(10)-Ru(1)	71.1(2)
C(16)-C(10)-Ru(1)	127.8(3)	C(12)-C(11)-C(10)	119.9(4)
C(12)-C(11)-C(17)	118.8(4)	C(10)-C(11)-C(17)	121.3(4)
C(12)-C(11)-Ru(1)	71.2(2)	C(10)-C(11)-Ru(1)	72.0(2)
C(17)-C(11)-Ru(1)	130.0(3)	C(11)-C(12)-C(13)	120.3(4)
C(11)-C(12)-C(18)	119.4(4)	C(13)-C(12)-C(18)	120.4(4)
C(11)-C(12)-Ru(1)	71.8(2)	C(13)-C(12)-Ru(1)	72.7(2)
C(18)-C(12)-Ru(1)	127.9(3)	C(14)-C(13)-C(12)	119.5(4)
C(14)-C(13)-C(19)	119.5(4)	C(12)-C(13)-C(19)	121.0(4)
C(14)-C(13)-Ru(1)	73.5(2)	C(12)-C(13)-Ru(1)	69.8(2)
C(19)-C(13)-Ru(1)	130.1(3)	C(13)-C(14)-C(15)	119.4(4)
C(13)-C(14)-C(20)	122.7(4)	C(15)-C(14)-C(20)	117.7(4)
C(13)-C(14)-Ru(1)	70.0(2)	C(15)-C(14)-Ru(1)	71.8(2)
C(20)-C(14)-Ru(1)	134.7(3)	C(10)-C(15)-C(14)	119.9(4)
C(10)-C(15)-C(21)	119.9(4)	C(14)-C(15)-C(21)	120.2(4)
C(10)-C(15)-Ru(1)	69.5(2)	C(14)-C(15)-Ru(1)	71.9(2)
C(21)-C(15)-Ru(1)	131.9(3)	O(1)-C(22)-Mn(1)	178.8(4)
O(2)-C(23)-Mn(1)	177.4(4)	O(3)-C(24)-Mn(1)	172.7(4)
O(4)-C(25)-Mn(1)	177.3(4)		

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for 4a /b

The anisotropic displacement factor exponent takes the form:
$-2\pi^2$ [$(ha^*)^2U_{11}$ + ... + $2hka^*b^*U_{12}$]

	U11	U22	U33	U23	U13	U12
Ru(1)	45(1)	38(1)	39(1)	-2(1)	7(1)	1(1)
Mn(1)	39(1)	54(1)	49(1)	-2(1)	3(1)	3(1)
S(1)	58(1)	86(1)	54(1)	-1(1)	15(1)	12(1)
C(2)	42(2)	89(3)	66(3)	24(3)	19(2)	4(2)
C(3)	40(2)	75(3)	69(3)	18(2)	6(2)	-8(2)
C(4)	63(2)	43(2)	65(3)	0(2)	-4(2)	-11(2)
C(5)	105(4)	54(3)	63(3)	-16(2)	-5(3)	8(2)
C(6)	78(3)	52(3)	50(3)	-6(2)	24(2)	13(2)
C(7)	47(2)	47(2)	51(2)	-4(2)	13(2)	7(2)
C(8)	42(2)	32(2)	48(2)	-3(2)	9(2)	4(2)
C(9)	48(2)	37(2)	51(2)	7(2)	-1(2)	-4(2)
C(10)	51(2)	58(3)	65(3)	4(2)	2(2)	11(2)
C(11)	66(3)	64(3)	51(3)	-2(2)	0(2)	10(2)
C(12)	89(3)	46(2)	51(3)	14(2)	20(2)	13(2)
C(13)	70(3)	39(2)	81(3)	10(2)	24(2)	7(2)
C(14)	69(3)	36(2)	56(3)	-5(2)	3(2)	9(2)
C(15)	56(2)	52(2)	53(2)	0(2)	11(2)	17(2)
C(16)	55(3)	109(4)	115(5)	0(3)	1(3)	-3(3)
C(17)	102(4)	120(5)	67(3)	-16(3)	-9(3)	10(3)
C(18)	139(5)	79(3)	70(3)	23(3)	50(3)	8(3)
C(19)	83(3)	66(3)	128(5)	8(3)	27(3)	-18(3)
C(20)	104(4)	63(3)	75(3)	-23(3)	4(3)	6(3)
C(21)	71(3)	84(3)	65(3)	1(2)	25(2)	23(2)
C(22)	48(2)	85(3)	59(3)	1(2)	0(2)	2(2)
O(1)	80(2)	140(3)	64(2)	4(2)	-11(2)	-3(2)
C(23)	40(2)	57(2)	71(3)	0(2)	6(2)	4(2)
O(2)	64(2)	95(3)	139(4)	-2(2)	34(2)	15(2)
C(24)	50(2)	62(3)	58(3)	-13(2)	1(2)	2(2)
O(3)	98(3)	62(2)	105(3)	-8(2)	10(2)	-21(2)
C(25)	53(2)	65(3)	54(3)	8(2)	-2(2)	7(2)
O(4)	91(2)	62(2)	106(3)	20(2)	-1(2)	-4(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 4a/16

	x	y	z	U(eq)
H(2A)	-279(4)	2167(3)	4941(2)	78
H(3A)	-351(4)	1499(3)	3935(2)	73
H(4A)	-14(5)	1594(3)	2743(2)	69
H(5A)	2571(6)	867(3)	2434(3)	90
H(5B)	1597(6)	1482(3)	1861(3)	90
H(6A)	3599(5)	2599(3)	2030(2)	71
H(7A)	4652(4)	2973(3)	3160(2)	57
H(16A)	-2890(7)	3872(10)	2518(18)	140
H(16B)	-2285(19)	3239(25)	3165(4)	140
H(16C)	-2098(15)	2851(16)	2414(16)	140
H(17A)	-1331(31)	4034(15)	1218(11)	146
H(17B)	-1013(39)	2952(14)	1502(4)	146
H(17C)	169(11)	3509(29)	1105(8)	146
H(18A)	1805(27)	4998(22)	1147(8)	140
H(18B)	2263(38)	3877(8)	1258(11)	140
H(18C)	3294(13)	4727(28)	1582(4)	140
H(19A)	3510(11)	6083(15)	2513(17)	137
H(19B)	4323(13)	5051(12)	2572(18)	137
H(19C)	3969(21)	5620(26)	3245(3)	137
H(20A)	1270(21)	5979(16)	4106(9)	121
H(20B)	2900(16)	5705(22)	3980(7)	121
H(20C)	1968(36)	4979(7)	4401(4)	121
H(21A)	-1060(29)	5085(4)	4106(9)	109
H(21B)	-311(15)	4068(20)	4335(5)	109
H(21C)	-1801(15)	4075(19)	3857(5)	109