Novel Ruthenium Complex-Catalyzed Dimerization of 2,5-Norbornadiene to Pentacyclo[6.6.0.$0^{2,6}$.$0^{3,13}$.$0^{10,14}$]tetradeca-4,11-diene Involving Carbon-Carbon Bond Cleavage

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Supporting Information

X-ray Structure Report

Crystallographic data for **5**

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Experimental

Data Collection

A colorless plate crystal of $C_{14}H_{16}Br_4$ having approximate dimensions of 0.40 x 0.40 x 0.30 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7R diffractometer with graphite monochromated Mo-Kα radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection , obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $20.97 < 2\theta < 26.52°$ corresponded to a primitive orthorhombic cell with dimensions:

a = 14.183(4) $\AA$
b = 16.477(4) $\AA$
c = 6.387(4) $\AA$
V = 1492(1) $\AA^3$

For Z = 4 and F.W. = 503.90, the calculated density is 2.24 g/cm^3. Based on the systematic absences of:

0kl: k+l $\neq$ 2n
hk0: h $\neq$ 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:

Pnma (#62)

The data were collected at a temperature of 18 $\pm$ 1°C using the ω-2θ scan technique to a maximum 2θ value of 60.0°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.31° with a take-off angle of 6.0°. Scans of $(1.10 + 0.30 \tan\theta)°$ were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 2 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 285 mm, and the detector aperture was 6.0 x 6.0 mm (horizontal x vertical).

Data Reduction

A total of 2310 reflections was collected. The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 106.6 cm^{-1}. An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.72 to 1.00. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction was applied (coefficient = 9.14582e-07).

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Some hydrogen atoms were refined isotropically, the rest were included in fixed positions. The final cycle of full-matrix least-squares refinement[3] was based on 729 observed reflections ($I > 3.00\sigma(I)$) and 116 variable parameters and converged (largest parameter shift was 0.13 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.045$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2} = 0.034$$

The standard deviation of an observation of unit weight[4] was 1.88. The weighting scheme was based on counting statistics and included a factor (p = 0.011) to downweight the intense reflections. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.77 and -0.88 $e^-/\AA^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] . Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf" were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SHELXS86: Sheldrick, G.M. (1985). In: "Crystallographic Computing 3" (Eds G.M. Sheldrick, C. Kruger and R. Goddard) Oxford University Press, pp. 175-189.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where w = $\frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

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

(7) 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).

(8) 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).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{14}H_{16}Br_4$
Formula Weight	503.90
Crystal Color, Habit	colorless, plate
Crystal Dimensions	0.40 X 0.40 X 0.30 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	25 (21.0 - 26.5°)
Omega Scan Peak Width at Half-height	0.31°
Lattice Parameters	a = 14.183(4)Å b = 16.477(4) Å c = 6.387(4) Å
	V = 1492(1) Å^3
Space Group	Pnma (#62)
Z value	4
D_{calc}	2.242 g/cm^3
F_{000}	960.00
μ(MoKα)	106.64 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC7R
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factor = 8.66)

Take-off Angle	6.0°
Detector Aperture	6.0 mm horizontal 6.0 mm vertical
Crystal to Detector Distance	285 mm
Temperature	18.0°C
Scan Type	ω-2θ
Scan Rate	16.0°/min (in ω) (up to 2 scans)
Scan Width	$(1.10 + 0.30 \tan \theta)°$
$2\theta_{max}$	60.0°
No. of Reflections Measured	Total: 2310
Corrections	Lorentz-polarization Absorption (trans. factors: 0.7155 - 1.0000) Secondary Extinction (coefficient: 9.14582e-07)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS-86)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$
p-factor	0.0110
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00σ(I))	729
No. Variables	116
Reflection/Parameter Ratio	6.28
Residuals: R; Rw	0.045 ; 0.034
Residuals: R1	0.000
No. of Reflections to calc R1	0

Goodness of Fit Indicator	1.88
Max Shift/Error in Final Cycle	0.13
Maximum peak in Final Diff. Map	0.77 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-0.88 $e^-/\AA^3$

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

atom	x	y	z	B_{eq}	occ
Br(1)	0.11469(8)	0.46828(6)	0.1987(2)	4.47(3)	1.0000
Br(2)	0.34977(9)	0.37322(8)	0.1749(2)	6.35(4)	1.0000
C(1)	0.1747(7)	0.3247(6)	-0.329(2)	2.9(3)	1.0000
C(2)	0.2540(6)	0.2965(6)	-0.176(2)	2.9(3)	1.0000
C(3)	0.2354(8)	0.3428(7)	0.026(2)	2.9(3)	1.0000
C(4)	0.1776(7)	0.4144(6)	-0.037(2)	2.9(3)	1.0000
C(5)	0.1082(7)	0.3812(5)	-0.197(2)	3.6(2)	1.0000
C(6)	0.0310(9)	0.3256(9)	-0.116(3)	6.2(5)	1.0000
C(7)	0.027(1)	0.2500	-0.249(2)	2.8(4)	0.5000
C(8)	0.1170(10)	0.2500	-0.390(2)	3.1(4)	0.5000
H(1)	0.199(5)	0.353(5)	-0.45(1)	1(1)	1.0000
H(2)	0.304(4)	0.310(4)	-0.22(1)	0(1)	1.0000
H(3)	0.209(6)	0.316(5)	0.13(1)	2(1)	1.0000
H(4)	0.219(5)	0.461(4)	-0.09(1)	1(1)	1.0000
H(5)	0.0832	0.4241	-0.2827	4.1742	1.0000
H(6b)	0.00(1)	0.321(8)	0.02(2)	11.3(6)	1.0000
H(6a)	-0.022(6)	0.350(5)	-0.12(1)	1(1)	1.0000
H(7)	-0.023(9)	0.2500	-0.33(2)	4.3(9)	0.5000
H(8)	0.07(1)	0.2500	-0.52(3)	10.7(6)	0.5000

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	0.0631(8)	0.0492(7)	0.0574(8)	0.0143(7)	-0.0039(8)	-0.0133(7)
Br(2)	0.0700(9)	0.0885(10)	0.083(1)	0.0284(8)	-0.0451(9)	-0.0367(9)
C(1)	0.040(6)	0.043(7)	0.028(7)	-0.012(5)	-0.004(6)	0.021(6)
C(2)	0.025(6)	0.045(7)	0.039(7)	-0.006(5)	0.002(6)	0.000(6)
C(3)	0.041(7)	0.037(7)	0.032(8)	0.005(6)	-0.008(6)	-0.002(6)
C(4)	0.035(6)	0.030(6)	0.044(8)	0.008(5)	0.005(6)	0.002(6)
C(5)	0.052(6)	0.037(6)	0.046(7)	0.009(6)	-0.011(7)	-0.004(7)
C(6)	0.030(8)	0.07(1)	0.14(2)	-0.012(7)	0.033(9)	-0.04(1)
C(7)	0.034(9)	0.05(1)	0.03(1)	0.0000	-0.012(7)	0.0000
C(8)	0.033(9)	0.041(9)	0.04(1)	0.0000	-0.010(9)	0.0000

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 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
Br(1)	C(4)	1.961(10)	Br(2)	C(3)	1.95(1)
C(1)	C(2)	1.56(1)	C(1)	C(5)	1.57(1)
C(1)	C(8)	1.53(1)	C(1)	H(1)	0.95(7)
C(2)	C(2)	1.53(2)	C(2)	C(3)	1.52(1)
C(2)	H(2)	0.80(6)	C(3)	C(4)	1.49(1)
C(3)	H(3)	0.87(8)	C(4)	C(5)	1.52(1)
C(4)	H(4)	1.01(6)	C(5)	C(6)	1.52(1)
C(5)	H(5)	0.96	C(6)	C(7)	1.51(2)
C(6)	H(6b)	1.0(1)	C(6)	H(6a)	0.85(7)
C(7)	C(8)	1.56(2)	C(7)	H(7)	0.9(1)
C(8)	H(8)	1.1(2)			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(1)	C(5)	105.9(9)	C(2)	C(1)	C(8)	107.8(9)
C(2)	C(1)	H(1)	112(4)	C(5)	C(1)	C(8)	107.0(8)
C(5)	C(1)	H(1)	110(5)	C(8)	C(1)	H(1)	112(5)
C(1)	C(2)	C(2)	107.3(5)	C(1)	C(2)	C(3)	104.8(9)
C(1)	C(2)	H(2)	108(5)	C(2)	C(2)	C(3)	120.2(6)
C(2)	C(2)	H(2)	105(4)	C(3)	C(2)	H(2)	109(5)
Br(2)	C(3)	C(2)	113.5(7)	Br(2)	C(3)	C(4)	112.7(7)
Br(2)	C(3)	H(3)	96(5)	C(2)	C(3)	C(4)	105.5(8)
C(2)	C(3)	H(3)	117(5)	C(4)	C(3)	H(3)	111(5)
Br(1)	C(4)	C(3)	113.8(7)	Br(1)	C(4)	C(5)	112.7(7)
Br(1)	C(4)	H(4)	99(4)	C(3)	C(4)	C(5)	104.6(8)
C(3)	C(4)	H(4)	111(3)	C(5)	C(4)	H(4)	115(4)
C(1)	C(5)	C(4)	100.7(8)	C(1)	C(5)	C(6)	105.0(8)
C(1)	C(5)	H(5)	110.8	C(4)	C(5)	C(6)	116(1)
C(4)	C(5)	H(5)	110.9	C(6)	C(5)	H(5)	111.8
C(5)	C(6)	C(7)	109(1)	C(5)	C(6)	H(6b)	131(8)
C(5)	C(6)	H(6a)	110(6)	C(7)	C(6)	H(6b)	115(7)
C(7)	C(6)	H(6a)	110(6)	H(6b)	C(6)	H(6a)	71(9)
C(6)	C(7)	C(6)	111(1)	C(6)	C(7)	C(8)	107.3(8)
C(6)	C(7)	H(7)	111(4)	C(6)	C(7)	C(8)	107.3(8)
C(6)	C(7)	H(7)	111(4)	C(8)	C(7)	H(7)	108(9)
C(1)	C(8)	C(1)	107(1)	C(1)	C(8)	C(7)	106.8(8)
C(1)	C(8)	H(8)	122(2)	C(1)	C(8)	C(7)	106.8(8)
C(1)	C(8)	H(8)	122(2)	C(7)	C(8)	H(8)	84(9)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
Br(1)	C(4)	C(3)	Br(2)	-71.5(8)	Br(1)	C(4)	C(3)	C(2)	164.1(6)
Br(1)	C(4)	C(5)	C(1)	-166.4(6)	Br(1)	C(4)	C(5)	C(6)	-53(1)
Br(2)	C(3)	C(2)	C(1)	-145.2(6)	Br(2)	C(3)	C(2)	C(2)	94.1(6)
Br(2)	C(3)	C(4)	C(5)	165.2(7)	C(1)	C(2)	C(2)	C(1)	0.0
C(1)	C(2)	C(2)	C(3)	-119.5(10)	C(1)	C(2)	C(3)	C(4)	-21(1)
C(1)	C(5)	C(4)	C(3)	-42.3(10)	C(1)	C(5)	C(6)	C(7)	-20(1)
C(1)	C(8)	C(1)	C(2)	15(1)	C(1)	C(8)	C(1)	C(5)	129.1(9)
C(1)	C(8)	C(7)	C(6)	2(1)	C(1)	C(8)	C(7)	C(6)	-117(1)
C(2)	C(1)	C(5)	C(4)	28.4(9)	C(2)	C(1)	C(5)	C(6)	-93(1)
C(2)	C(1)	C(8)	C(7)	98(1)	C(2)	C(2)	C(1)	C(5)	-123.9(6)
C(2)	C(2)	C(1)	C(8)	-9.6(10)	C(2)	C(2)	C(3)	C(4)	142.0(6)
C(2)	C(3)	C(4)	C(5)	40(1)	C(3)	C(2)	C(1)	C(5)	-5(1)
C(3)	C(2)	C(1)	C(8)	-119.3(10)	C(3)	C(2)	C(2)	C(3)	0.0
C(3)	C(4)	C(5)	C(6)	70(1)	C(4)	C(5)	C(1)	C(8)	143.2(9)
C(4)	C(5)	C(6)	C(7)	-130(1)	C(5)	C(1)	C(8)	C(7)	-14(1)
C(5)	C(6)	C(7)	C(6)	128(1)	C(5)	C(6)	C(7)	C(8)	11(1)
C(6)	C(5)	C(1)	C(8)	21(1)					

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
Br(1)	H(4)	2.97(7)	56504	Br(1)	H(1)	3.19(7)	55601
Br(1)	H(6a)	3.31(8)	56505	Br(1)	H(5)	3.36	56505
Br(1)	H(5)	3.42	55601	Br(2)	H(6b)	3.0(1)	8
Br(2)	H(1)	3.25(8)	55601	Br(2)	H(4)	3.28(7)	56504
Br(2)	H(6a)	3.39(10)	8	Br(2)	H(5)	3.48	56504
Br(2)	H(7)	3.5(1)	55402	C(1)	H(3)	3.50(8)	55401
C(2)	H(7)	3.3(1)	55402	C(2)	H(6a)	3.56(8)	55408
C(3)	H(1)	3.41(8)	55601	C(6)	H(2)	3.39(6)	45408
C(7)	H(2)	3.32(6)	45402	C(7)	H(2)	3.32(6)	45408
C(8)	H(3)	3.52(8)	55401	C(8)	H(3)	3.52(8)	55407
H(1)	H(3)	2.8(1)	55401	H(1)	H(4)	3.4(1)	56404
H(2)	H(7)	2.7(1)	55402	H(2)	H(6a)	2.75(10)	55408
H(2)	H(6b)	3.4(2)	55408	H(3)	H(8)	3.2(2)	55601
H(4)	H(4)	3.56(6)	56504	H(4)	H(4)	3.56(6)	56404
H(6b)	H(8)	3.3(2)	55601				

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus ±4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

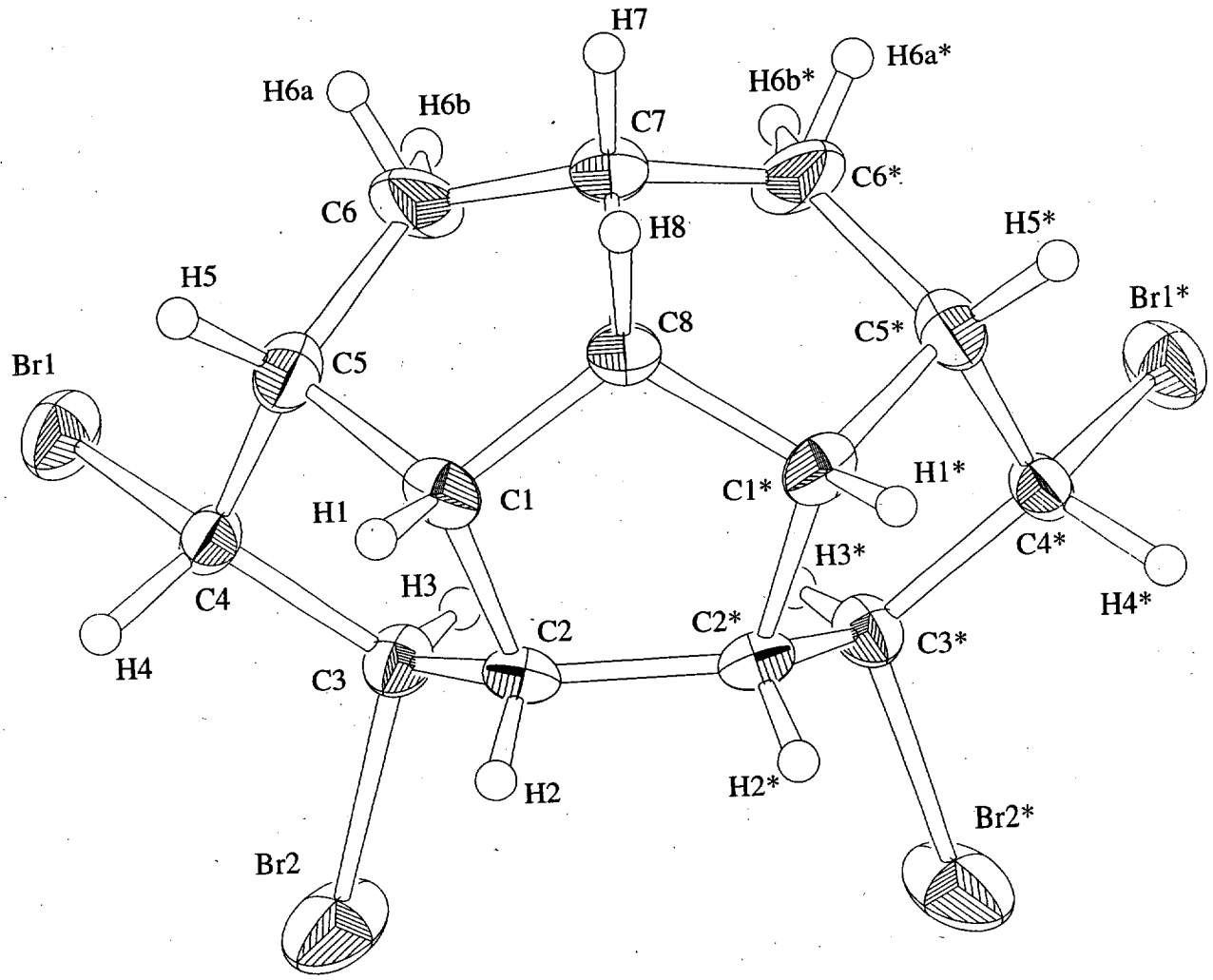
The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure are given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

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



X-ray Structure Report

Crystallographic data for **6**

Tue Oct 6 1998

Experimental

Data Collection

A colorless needle crystal of $C_{15}H_{16}F_3O_3SAg$ having approximate dimensions of 0.10 x 0.10 x 0.40 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7R diffractometer with graphite monochromated Mo-Kα radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection , obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $28.24 < 2\theta < 29.80°$ corresponded to a primitive orthorhombic cell with dimensions:

a = 19.708(4) $\AA$

b = 7.742(4) $\AA$

c = 9.858(4) $\AA$

V = 1504.2(9) $\AA^3$

For Z = 4 and F.W. = 441.21, the calculated density is 1.95 g/cm^3. Based on the systematic absences of:

0kl: $l \neq 2n$

h0l: $h \neq 2n$

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

$Pca2_1$ (#29)

The data were collected at a temperature of 23 $\pm$ 1°C using the ω-2θ scan technique to a maximum 2θ value of 55.0°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.22° with a take-off angle of 6.0°. Scans of $(0.79 + 0.30 \tan \theta)°$ were made at a speed of 8.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 3 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm and the crystal to detector distance was 235 mm, The computer-controlled slits were set to 3.0 mm (horizontal) and 3.0 mm (vertical).

Data Reduction

A total of 2015 reflections was collected. The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 15.2 cm^{-1}. Azimuthal scans of several reflections indicated no need for an absorption correction. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction was applied (coefficient = 6.78120e-07).

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included, but their positions were not refined; isotropic B values were refined The final cycle of full-matrix least-squares refinement[3] was based on 1499 observed reflections ($I > 2.00\sigma(I)$) and 225 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.029$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2} = 0.030$$

The standard deviation of an observation of unit weight[4] was 1.28. The weighting scheme was based on counting statistics and included a factor (p = 0.020) to downweight the intense reflections. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.34 and -0.31 $e^-/\AA^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] . Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf" were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SHELXS86: Sheldrick, G.M. (1985). In: "Crystallographic Computing 3" (Eds G.M. Sheldrick, C. Kruger and R. Goddard) Oxford University Press, pp. 175-189.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where w = $\frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

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

(7) 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).

(8) 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).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{15}H_{16}F_3O_3SAg$
Formula Weight	441.21
Crystal Color, Habit	colorless, needle
Crystal Dimensions	0.10 X 0.10 X 0.40 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	25 (28.2 - 29.8°)
Omega Scan Peak Width at Half-height	0.22°
Lattice Parameters	a = 19.708(4)Å b = 7.742(4) Å c = 9.858(4) Å V = 1504.2(9) Å^3
Space Group	$Pca2_1$ (#29)
Z value	4
D_{calc}	1.948 g/cm^3
F_{000}	880.00
μ(MoKα)	15.19 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC7R
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factor = 8.60)

Take-off Angle	6.0°
Detector Aperture	3.0 mm horizontal 3.0 mm vertical
Crystal to Detector Distance	235 mm
Voltage, Current	50kV, 220mA
Temperature	23.0°C
Scan Type	ω-2θ
Scan Rate	8.0°/min (in ω) (up to 3 scans)
Scan Width	$(0.79 + 0.30 \tan\theta)°$
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 2015
Corrections	Lorentz-polarization Secondary Extinction (coefficient: 6.78120e-07)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS-86)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$
p-factor	0.0200
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	1499
No. Variables	225
Reflection/Parameter Ratio	6.66
Residuals: R; Rw	0.029 ; 0.030
Residuals: R1	0.029
No. of Reflections to calc R1	1499

Goodness of Fit Indicator	1.28
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	0.34 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.31 $e^-/Å^3$

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

atom	x	y	z	B_{eq}
Ag(1)	0.31515(2)	0.05808(5)	0.926(2)	3.174(9)
S(1)	0.16412(8)	0.1000(2)	1.069(2)	2.88(3)
F(1)	0.0516(2)	-0.0241(6)	1.169(2)	5.2(1)
F(2)	0.0550(2)	-0.0224(6)	0.953(2)	6.1(2)
F(3)	0.1114(2)	-0.2097(5)	1.064(2)	6.0(1)
O(1)	0.2961(2)	0.0372(6)	0.681(2)	3.8(1)
O(2)	0.1965(2)	0.0730(7)	0.941(2)	4.5(1)
O(3)	0.1336(3)	0.2642(7)	1.090(2)	5.1(1)
C(1)	0.3522(3)	0.5700(8)	1.261(2)	2.9(1)
C(2)	0.3208(3)	0.4053(7)	1.193(2)	2.7(1)
C(3)	0.2958(3)	0.4596(7)	1.050(2)	2.7(1)
C(4)	0.3455(3)	0.3704(7)	0.956(2)	2.7(1)
C(5)	0.3910(3)	0.2733(8)	1.023(2)	2.8(1)
C(6)	0.3807(3)	0.2767(8)	1.175(2)	3.0(1)
C(7)	0.4402(4)	0.3471(8)	1.257(2)	3.6(2)
C(8)	0.4302(3)	0.5430(7)	1.263(2)	3.1(1)
C(9)	0.4569(3)	0.6418(8)	1.138(2)	3.1(1)
C(10)	0.4056(3)	0.7876(7)	1.112(2)	2.7(1)
C(11)	0.3913(3)	0.8113(8)	0.964(2)	3.3(2)
C(12)	0.3314(3)	0.7412(7)	0.931(2)	3.3(1)
C(13)	0.2958(3)	0.6621(7)	1.047(2)	2.8(1)
C(14)	0.3369(3)	0.7244(7)	1.170(2)	2.5(1)
C(15)	0.0924(3)	-0.0466(9)	1.062(2)	3.6(2)
H(1)	0.3343	0.5889	1.3493	6.3(7)

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

atom	x	y	z	B_{eq}
H(2)	0.2844	0.3566	1.2457	2.2(9)
H(3)	0.2502	0.4168	1.0318	1.6(9)
H(4)	0.3613	0.4348	0.8762	5.3(7)
H(5)	0.4363	0.2728	0.9877	10.1(5)
H(6)	0.3693	0.1631	1.2067	3.0(9)
H(7A)	0.4435	0.2970	1.3424	5.2(7)
H(7B)	0.4832	0.3203	1.2079	3.5(9)
H(8)	0.4503	0.5909	1.3427	1.1(9)
H(9A)	0.5018	0.6872	1.1510	7.7(6)
H(9B)	0.4594	0.5661	1.0586	6.0(7)
H(10)	0.4198	0.8935	1.1528	1.5(9)
H(11)	0.4218	0.8702	0.8982	3.4(9)
H(12)	0.3125	0.7414	0.8395	5.9(7)
H(13)	0.2499	0.7069	1.0499	2.3(9)
H(14)	0.3142	0.8142	1.2172	4.0(9)

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	0.0451(2)	0.0277(2)	0.0478(2)	0.0014(2)	-0.0045(3)	-0.0005(4)
S(1)	0.0313(7)	0.0374(8)	0.0407(8)	-0.0024(6)	0.0021(7)	0.0039(8)
F(1)	0.048(2)	0.070(3)	0.081(3)	-0.011(2)	0.023(2)	0.012(3)
F(2)	0.067(3)	0.085(3)	0.080(6)	-0.028(2)	-0.037(3)	0.022(3)
F(3)	0.072(3)	0.033(2)	0.125(5)	-0.006(2)	0.000(3)	-0.015(3)
O(1)	0.044(2)	0.061(3)	0.040(2)	-0.002(2)	0.006(2)	-0.001(3)
O(2)	0.045(2)	0.090(3)	0.036(4)	-0.007(2)	0.001(3)	0.009(4)
O(3)	0.049(3)	0.037(3)	0.110(5)	0.002(2)	0.000(4)	0.002(3)
C(1)	0.041(4)	0.035(3)	0.036(3)	0.002(3)	-0.001(3)	-0.005(3)
C(2)	0.040(3)	0.022(3)	0.042(3)	0.002(2)	0.009(3)	0.007(3)
C(3)	0.031(3)	0.019(3)	0.053(4)	-0.004(2)	-0.012(3)	0.004(3)
C(4)	0.042(3)	0.019(3)	0.043(5)	-0.007(2)	-0.001(3)	-0.004(3)
C(5)	0.043(4)	0.021(3)	0.043(4)	-0.004(3)	0.004(3)	-0.004(3)
C(6)	0.044(4)	0.018(3)	0.051(4)	0.002(2)	-0.001(3)	0.005(3)
C(7)	0.058(4)	0.035(4)	0.042(4)	0.010(3)	-0.013(3)	0.007(3)
C(8)	0.042(4)	0.030(3)	0.045(4)	0.006(3)	-0.011(3)	-0.005(3)
C(9)	0.031(3)	0.028(3)	0.060(4)	0.000(2)	-0.005(3)	-0.003(3)
C(10)	0.035(3)	0.018(3)	0.051(4)	-0.002(2)	-0.002(3)	0.000(3)
C(11)	0.043(3)	0.023(3)	0.058(5)	0.008(3)	0.006(3)	0.007(3)
C(12)	0.061(4)	0.022(3)	0.043(3)	0.003(2)	-0.016(5)	0.010(5)
C(13)	0.031(3)	0.024(3)	0.051(4)	0.002(2)	-0.004(3)	0.001(3)
C(14)	0.030(3)	0.023(3)	0.043(4)	0.005(2)	0.004(3)	-0.005(3)
C(15)	0.042(4)	0.044(4)	0.051(4)	-0.006(3)	0.000(3)	0.012(4)

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 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
Ag(1)	O(1)	2.448(5)	Ag(1)	O(2)	2.346(4)
Ag(1)	C(4)	2.509(5)	Ag(1)	C(5)	2.435(6)
Ag(1)	C(11)	2.459(6)	Ag(1)	C(12)	2.474(5)
S(1)	O(1)	1.436(5)	S(1)	O(2)	1.433(9)
S(1)	O(3)	1.421(5)	S(1)	C(15)	1.814(7)
F(1)	C(15)	1.337(9)	F(2)	C(15)	1.322(9)
F(3)	C(15)	1.318(8)	C(1)	C(2)	1.564(8)
C(1)	C(8)	1.552(9)	C(1)	C(14)	1.519(9)
C(1)	H(1)	0.95(2)	C(2)	C(3)	1.556(9)
C(2)	C(6)	1.554(8)	C(2)	H(2)	0.96(1)
C(3)	C(4)	1.513(9)	C(3)	C(13)	1.568(8)
C(3)	H(3)	0.975(6)	C(4)	C(5)	1.341(9)
C(4)	H(4)	0.98(2)	C(5)	C(6)	1.52(1)
C(5)	H(5)	0.958(9)	C(6)	C(7)	1.527(9)
C(6)	H(6)	0.960(8)	C(7)	C(8)	1.530(9)
C(7)	H(7A)	0.93(2)	C(7)	H(7B)	1.00(1)
C(8)	C(9)	1.534(9)	C(8)	H(8)	0.96(2)
C(9)	C(10)	1.538(8)	C(9)	H(9A)	0.961(6)
C(9)	H(9B)	0.98(2)	C(10)	C(11)	1.494(9)
C(10)	C(14)	1.551(8)	C(10)	H(10)	0.956(9)
C(11)	C(12)	1.339(8)	C(11)	H(11)	1.00(1)
C(12)	C(13)	1.47(1)	C(12)	H(12)	0.98(2)
C(13)	C(14)	1.539(9)	C(13)	H(13)	0.969(6)
C(14)	H(14)	0.95(1)			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	Ag(1)	O(2)	85.0(2)	O(1)	Ag(1)	C(4)	102.7(2)
O(1)	Ag(1)	C(5)	121.9(2)	O(1)	Ag(1)	C(11)	101.3(2)
O(1)	Ag(1)	C(12)	88.7(3)	O(2)	Ag(1)	C(4)	100.5(2)
O(2)	Ag(1)	C(5)	123.6(2)	O(2)	Ag(1)	C(11)	129.5(2)
O(2)	Ag(1)	C(12)	100.1(2)	C(4)	Ag(1)	C(5)	31.4(2)
C(4)	Ag(1)	C(11)	125.8(2)	C(4)	Ag(1)	C(12)	157.2(2)
C(5)	Ag(1)	C(11)	95.5(2)	C(5)	Ag(1)	C(12)	126.1(2)
C(11)	Ag(1)	C(12)	31.5(2)	O(1)	S(1)	O(2)	112.6(3)
O(1)	S(1)	O(3)	114.9(4)	O(1)	S(1)	C(15)	104.0(3)
O(2)	S(1)	O(3)	116.6(4)	O(2)	S(1)	C(15)	102.9(3)
O(3)	S(1)	C(15)	103.6(3)	Ag(1)	O(1)	S(1)	130.5(3)
Ag(1)	O(2)	S(1)	120.5(4)	C(2)	C(1)	C(8)	106.7(5)
C(2)	C(1)	C(14)	108.4(5)	C(2)	C(1)	H(1)	111.5(6)
C(8)	C(1)	C(14)	108.1(5)	C(8)	C(1)	H(1)	112.1(7)
C(14)	C(1)	H(1)	110.0(6)	C(1)	C(2)	C(3)	106.8(4)
C(1)	C(2)	C(6)	105.7(5)	C(1)	C(2)	H(2)	112.9(9)
C(3)	C(2)	C(6)	108.0(5)	C(3)	C(2)	H(2)	110(1)
C(6)	C(2)	H(2)	112.2(5)	C(2)	C(3)	C(4)	103.1(4)
C(2)	C(3)	C(13)	106.7(5)	C(2)	C(3)	H(3)	111(1)
C(4)	C(3)	C(13)	116.4(5)	C(4)	C(3)	H(3)	109.2(9)
C(13)	C(3)	H(3)	109.7(5)	Ag(1)	C(4)	C(3)	111.1(4)
Ag(1)	C(4)	C(5)	71.2(3)	Ag(1)	C(4)	H(4)	117.8(7)
C(3)	C(4)	C(5)	113.0(6)	C(3)	C(4)	H(4)	117.6(6)
C(5)	C(4)	H(4)	117.7(6)	Ag(1)	C(5)	C(4)	77.3(4)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
Ag(1)	C(5)	C(6)	108.6(4)	Ag(1)	C(5)	H(5)	115.3(8)
C(4)	C(5)	C(6)	112.6(6)	C(4)	C(5)	H(5)	116(1)
C(6)	C(5)	H(5)	118(1)	C(2)	C(6)	C(5)	103.2(6)
C(2)	C(6)	C(7)	107.0(6)	C(2)	C(6)	H(6)	111.8(6)
C(5)	C(6)	C(7)	115.4(6)	C(5)	C(6)	H(6)	109(1)
C(7)	C(6)	H(6)	109.5(10)	C(6)	C(7)	C(8)	105.8(5)
C(6)	C(7)	H(7A)	112.6(7)	C(6)	C(7)	H(7B)	108.5(9)
C(8)	C(7)	H(7A)	113.3(8)	C(8)	C(7)	H(7B)	109.3(6)
H(7A)	C(7)	H(7B)	107.3(8)	C(1)	C(8)	C(7)	105.1(5)
C(1)	C(8)	C(9)	105.2(5)	C(1)	C(8)	H(8)	111.5(7)
C(7)	C(8)	C(9)	115.1(6)	C(7)	C(8)	H(8)	110.9(6)
C(9)	C(8)	H(8)	108.8(8)	C(8)	C(9)	C(10)	106.1(5)
C(8)	C(9)	H(9A)	113(1)	C(8)	C(9)	H(9B)	111.0(8)
C(10)	C(9)	H(9A)	111.0(6)	C(10)	C(9)	H(9B)	109.6(7)
H(9A)	C(9)	H(9B)	105(1)	C(9)	C(10)	C(11)	112.4(5)
C(9)	C(10)	C(14)	106.2(5)	C(9)	C(10)	H(10)	111.4(7)
C(11)	C(10)	C(14)	103.7(5)	C(11)	C(10)	H(10)	111(1)
C(14)	C(10)	H(10)	111.8(8)	Ag(1)	C(11)	C(10)	111.2(4)
Ag(1)	C(11)	C(12)	74.9(3)	Ag(1)	C(11)	H(11)	84.9(4)
C(10)	C(11)	C(12)	110.7(7)	C(10)	C(11)	H(11)	125.3(10)
C(12)	C(11)	H(11)	124(1)	Ag(1)	C(12)	C(11)	73.6(3)
Ag(1)	C(12)	C(13)	111.7(5)	Ag(1)	C(12)	H(12)	85.9(3)
C(11)	C(12)	C(13)	113.7(8)	C(11)	C(12)	H(12)	124.0(9)
C(13)	C(12)	H(12)	122.3(7)	C(3)	C(13)	C(12)	115.5(6)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(3)	C(13)	C(14)	107.4(5)	C(3)	C(13)	H(13)	110.9(5)
C(12)	C(13)	C(14)	103.3(5)	C(12)	C(13)	H(13)	108(1)
C(14)	C(13)	H(13)	110(1)	C(1)	C(14)	C(10)	107.0(5)
C(1)	C(14)	C(13)	108.7(5)	C(1)	C(14)	H(14)	112(1)
C(10)	C(14)	C(13)	105.3(5)	C(10)	C(14)	H(14)	111.2(6)
C(13)	C(14)	H(14)	111.4(9)	S(1)	C(15)	F(1)	111.0(5)
S(1)	C(15)	F(2)	112.1(5)	S(1)	C(15)	F(3)	112.2(5)
F(1)	C(15)	F(2)	106.8(6)	F(1)	C(15)	F(3)	106.7(6)
F(2)	C(15)	F(3)	107.6(6)				

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
Ag(1)	O(1)	S(1)	O(2)	-175.0(3)	Ag(1)	O(1)	S(1)	O(3)	-38.2(5)
Ag(1)	O(1)	S(1)	C(15)	74.4(4)	Ag(1)	O(2)	S(1)	O(1)	-27.3(4)
Ag(1)	O(2)	S(1)	O(3)	108.7(4)	Ag(1)	O(2)	S(1)	C(15)	-138.7(3)
Ag(1)	C(4)	C(3)	C(2)	80.1(4)	Ag(1)	C(4)	C(3)	C(13)	-163.5(4)
Ag(1)	C(4)	C(5)	C(6)	-105.1(5)	Ag(1)	C(5)	C(4)	C(3)	105.6(4)
Ag(1)	C(5)	C(6)	C(2)	-86.6(5)	Ag(1)	C(5)	C(6)	C(7)	157.0(4)
S(1)	O(2)	Ag(1)	O(1)	-176.0(4)	S(1)	O(2)	Ag(1)	C(4)	-74.0(4)
S(1)	O(2)	Ag(1)	C(5)	-50.7(5)	S(1)	O(2)	Ag(1)	C(11)	83.5(4)
S(1)	O(2)	Ag(1)	C(12)	96.3(4)	F(1)	C(15)	S(1)	O(1)	69.5(5)
F(1)	C(15)	S(1)	O(2)	-172.9(5)	F(1)	C(15)	S(1)	O(3)	-51.0(6)
F(2)	C(15)	S(1)	O(1)	-171.1(5)	F(2)	C(15)	S(1)	O(2)	-53.5(6)
F(2)	C(15)	S(1)	O(3)	68.4(7)	F(3)	C(15)	S(1)	O(1)	-49.8(6)
F(3)	C(15)	S(1)	O(2)	67.8(6)	F(3)	C(15)	S(1)	O(3)	-170.3(6)
O(1)	Ag(1)	C(4)	C(3)	119.9(4)	O(1)	Ag(1)	C(4)	C(5)	-132.0(4)
O(1)	Ag(1)	C(5)	C(4)	58.6(4)	O(1)	Ag(1)	C(5)	C(6)	168.5(4)
O(2)	Ag(1)	C(4)	C(3)	32.7(5)	O(2)	Ag(1)	C(4)	C(5)	140.8(4)
O(2)	Ag(1)	C(5)	C(4)	-48.2(5)	O(2)	Ag(1)	C(5)	C(6)	61.7(5)
C(1)	C(2)	C(3)	C(4)	109.3(5)	C(1)	C(2)	C(3)	C(13)	-13.8(6)
C(1)	C(2)	C(6)	C(5)	-109.9(6)	C(1)	C(2)	C(6)	C(7)	12.4(7)
C(1)	C(8)	C(7)	C(6)	31.7(7)	C(1)	C(8)	C(9)	C(10)	27.2(6)
C(1)	C(14)	C(10)	C(9)	14.5(7)	C(1)	C(14)	C(10)	C(11)	133.1(5)
C(1)	C(14)	C(13)	C(3)	-9.0(6)	C(1)	C(14)	C(13)	C(12)	-131.6(5)
C(2)	C(1)	C(8)	C(7)	-23.9(7)	C(2)	C(1)	C(8)	C(9)	98.0(6)
C(2)	C(1)	C(14)	C(10)	-112.9(5)	C(2)	C(1)	C(14)	C(13)	0.4(7)

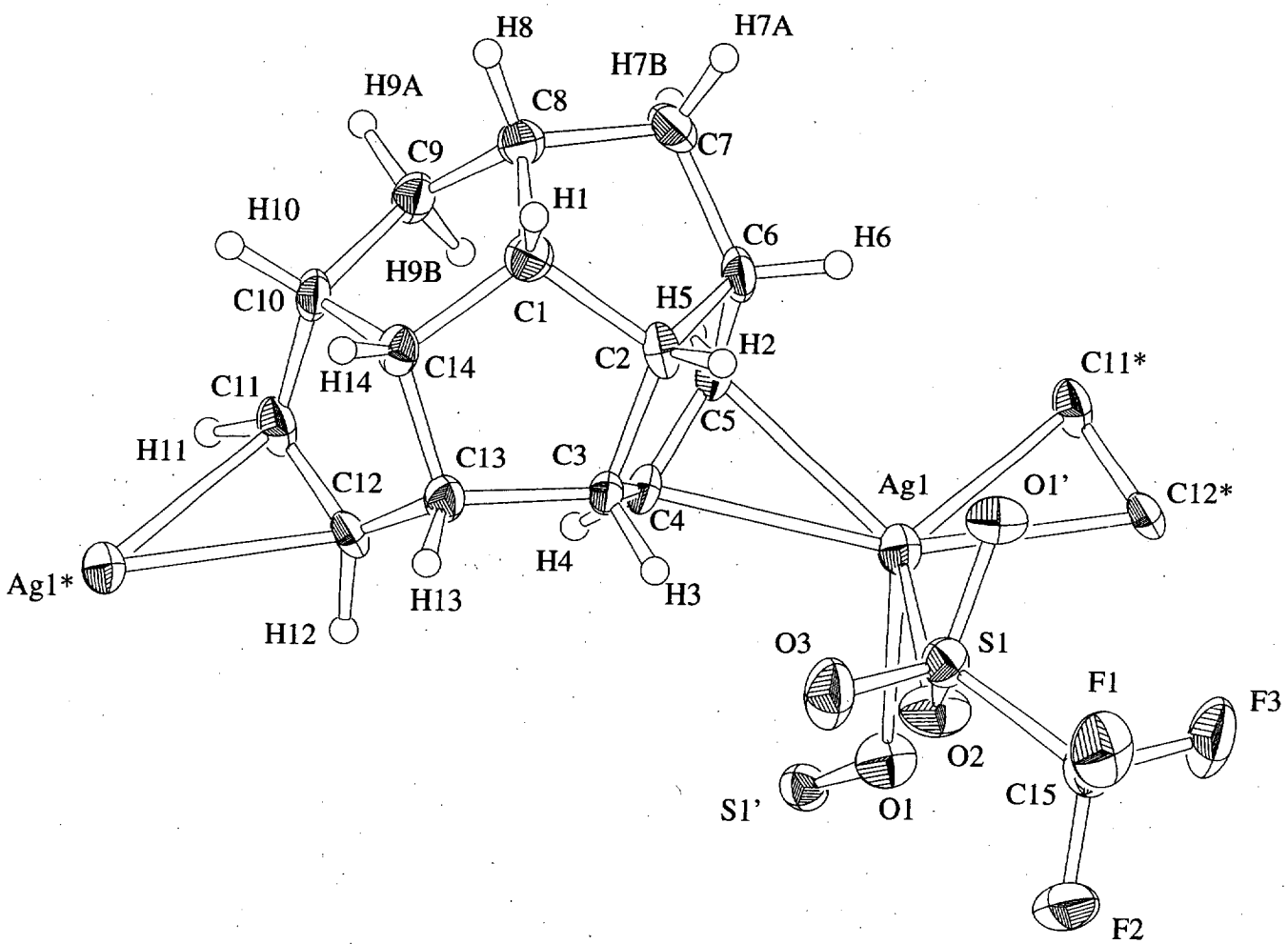
Table 5. Torsion Angles(°) (continued)

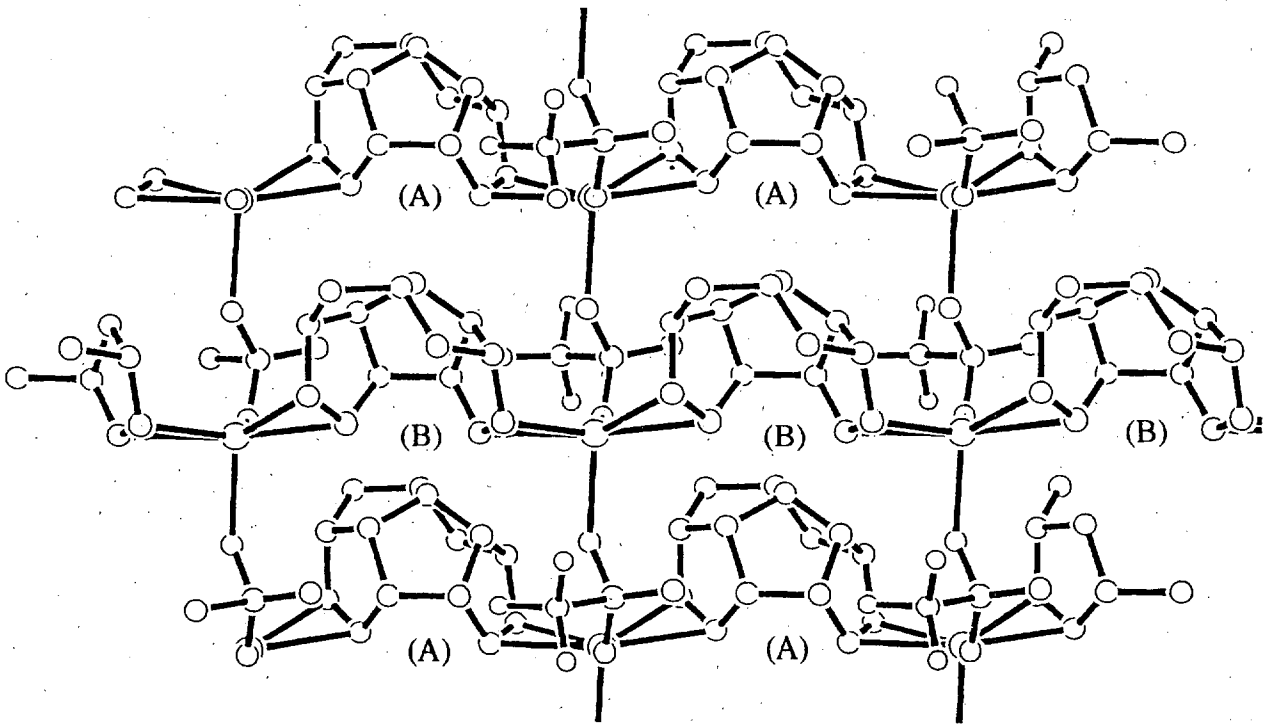
atom	atom	atom	atom	angle
C(2)	C(3)	C(4)	C(5)	2.3(6)
C(2)	C(3)	C(13)	C(14)	14.1(6)
C(2)	C(6)	C(7)	C(8)	-27.5(7)
C(3)	C(2)	C(1)	C(14)	8.4(6)
C(3)	C(2)	C(6)	C(7)	126.4(6)
C(3)	C(4)	Ag(1)	C(11)	-125.9(4)
C(3)	C(4)	C(5)	C(6)	0.4(7)
C(3)	C(13)	C(14)	C(10)	105.4(5)
C(4)	C(3)	C(2)	C(6)	-4.0(6)
C(4)	C(3)	C(13)	C(14)	-100.2(6)
C(4)	C(5)	Ag(1)	C(12)	173.5(5)
C(5)	C(4)	Ag(1)	C(11)	-17.8(5)
C(5)	C(4)	C(3)	C(13)	118.7(6)
C(6)	C(2)	C(1)	C(8)	7.1(7)
C(6)	C(2)	C(3)	C(13)	-127.1(5)
C(6)	C(5)	Ag(1)	C(12)	-76.6(6)
C(7)	C(8)	C(1)	C(14)	-140.2(5)
C(8)	C(1)	C(14)	C(10)	2.3(7)
C(8)	C(9)	C(10)	C(11)	-138.7(5)
C(9)	C(8)	C(1)	C(14)	-18.3(7)
C(9)	C(10)	C(14)	C(13)	-101.0(5)
C(10)	C(11)	C(12)	C(13)	0.5(7)
C(11)	Ag(1)	C(12)	C(13)	109.5(8)
C(11)	C(12)	C(13)	C(14)	10.9(7)

atom	atom	atom	atom	angle
C(2)	C(3)	C(13)	C(12)	128.8(6)
C(2)	C(6)	C(5)	C(4)	-2.9(7)
C(3)	C(2)	C(1)	C(8)	-107.7(5)
C(3)	C(2)	C(6)	C(5)	4.2(6)
C(3)	C(4)	Ag(1)	C(5)	-108.1(6)
C(3)	C(4)	Ag(1)	C(12)	-121.7(8)
C(3)	C(13)	C(12)	C(11)	-106.0(6)
C(4)	Ag(1)	C(5)	C(6)	109.9(6)
C(4)	C(3)	C(13)	C(12)	14.4(8)
C(4)	C(5)	Ag(1)	C(11)	165.6(4)
C(4)	C(5)	C(6)	C(7)	-119.3(6)
C(5)	C(4)	Ag(1)	C(12)	-13.6(10)
C(5)	C(6)	C(7)	C(8)	86.7(7)
C(6)	C(2)	C(1)	C(14)	123.3(6)
C(6)	C(5)	Ag(1)	C(11)	-84.6(4)
C(6)	C(7)	C(8)	C(9)	-83.5(7)
C(7)	C(8)	C(9)	C(10)	142.4(6)
C(8)	C(1)	C(14)	C(13)	115.6(5)
C(8)	C(9)	C(10)	C(14)	-26.0(6)
C(9)	C(10)	C(11)	C(12)	102.6(6)
C(10)	C(11)	Ag(1)	C(12)	-106.7(7)
C(10)	C(14)	C(13)	C(12)	-17.2(6)
C(11)	C(10)	C(14)	C(13)	17.5(6)
C(12)	C(11)	C(10)	C(14)	-11.6(7)

Table 5. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle





X-ray Structure Report

Crystallographic data for **7**

Tue Oct 6 1998

Experimental

Data Collection

A yellow prismatic crystal of $C_{20}H_{26}O_8Ru$ having approximate dimensions of 0.10 x 0.10 x 0.05 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC7R diffractometer with graphite monochromated Mo-Kα radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection , obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $29.42 < 2\theta < 29.95°$ corresponded to a primitive monoclinic cell with dimensions:

a = 7.816(5) $\AA$

b = 25.190(5) $\AA$ $\quad \beta = 109.01(4)°$

c = 11.050(6) $\AA$

V = 2056(1) $\AA^3$

For Z = 4 and F.W. = 495.49, the calculated density is 1.60 g/cm^3. The systematic absences of:

h0l: $l \neq 2n$

0k0: $k \neq 2n$

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of 23 $\pm$ 1°C using the ω scan technique to a maximum 2θ value of 55.0°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.22° with a take-off angle of 6.0°. Scans of $(0.52 + 0.30 \tan \theta)°$ were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 1 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 0.5 mm and the crystal to detector distance was 235 mm, The computer-controlled slits were set to 3.0 mm (horizontal) and 3.0 mm (vertical).

Data Reduction

Of the 5088 reflections which were collected, 4852 were unique ($R_{int} = 0.026$). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 8.1 cm^{-1}. Azimuthal scans of several reflections indicated no need for an absorption correction. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included, but their positions were not refined; isotropic B values were refined The final cycle of full-matrix least-squares refinement[3] was based on 2892 observed reflections ($I > 3.00\sigma(I)$) and 288 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.040$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2} = 0.041$$

The standard deviation of an observation of unit weight[4] was 0.66. The weighting scheme was based on counting statistics and included a factor (p = 0.002) to downweight the intense reflections. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.40 and -0.40 $e^-/\mathring{A}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] . Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf" were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SHELXS86: Sheldrick, G.M. (1985). In: "Crystallographic Computing 3" (Eds G.M. Sheldrick, C. Kruger and R. Goddard) Oxford University Press, pp. 175-189.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where w = $\frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables