Supporting Information for

"all-Z-Tetrabenzo[16]- and Pentabenzo[20]annulenes, π-Cavitands Binding to Silver Cation"

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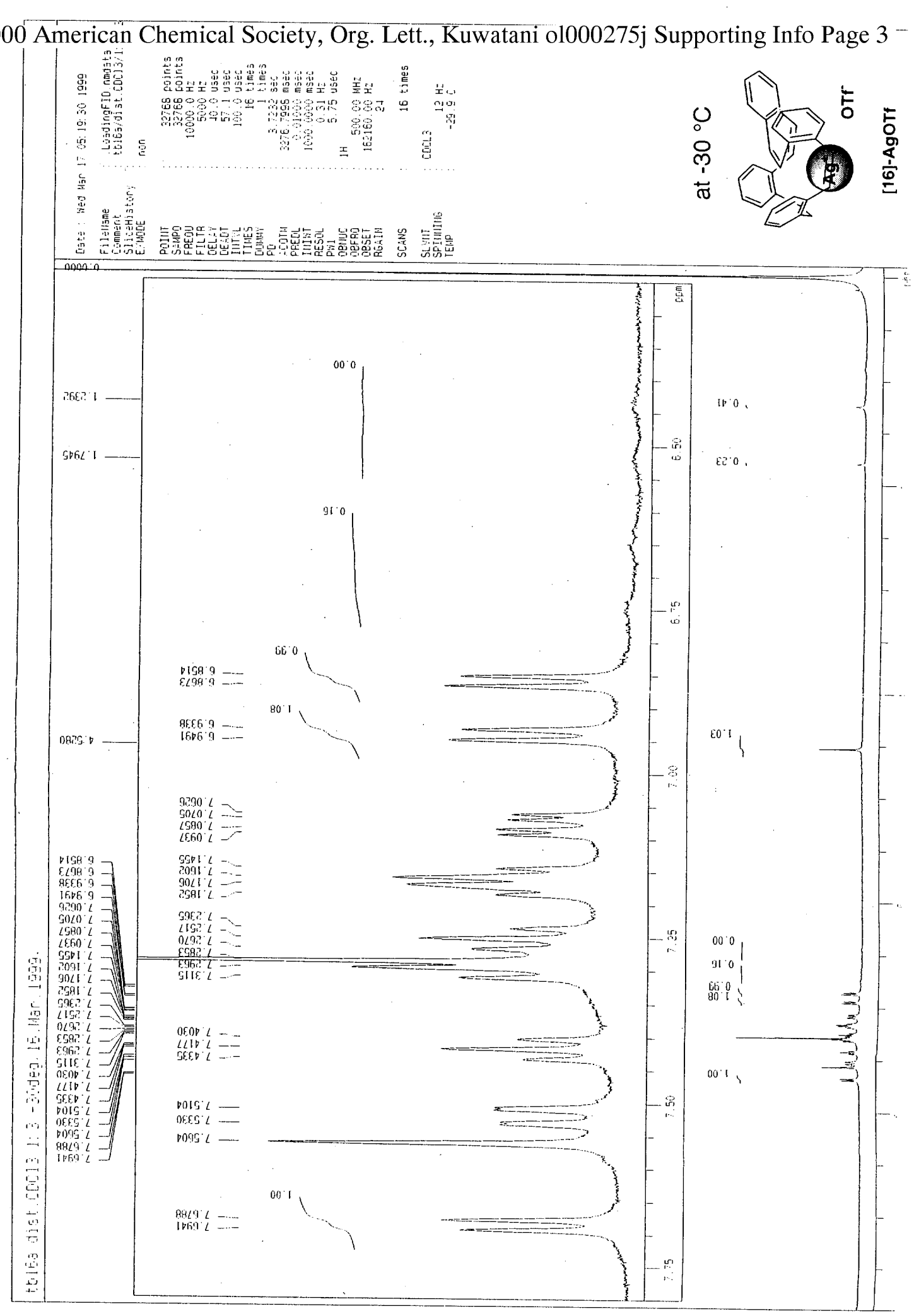
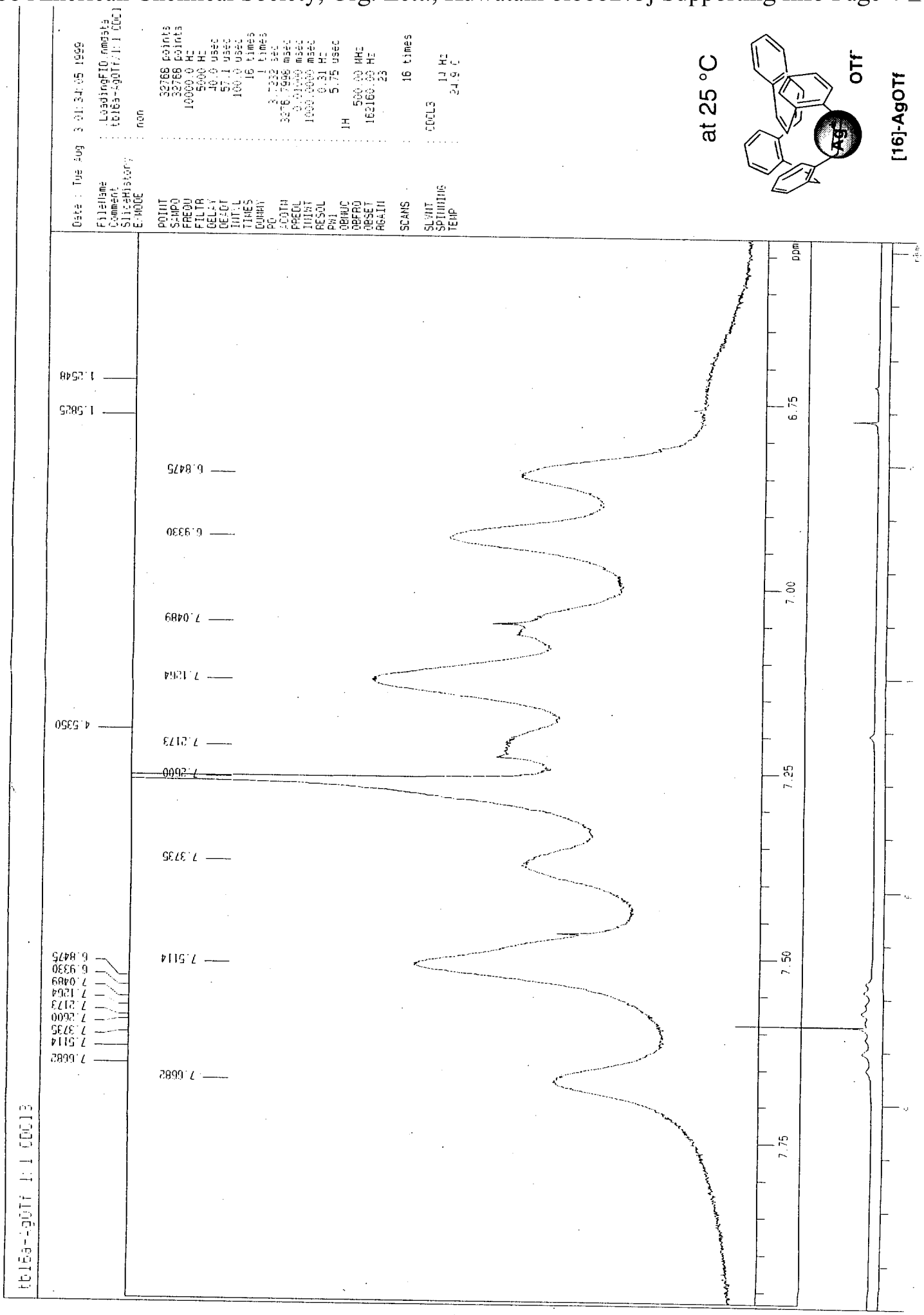
Preparation of *all-Z*-tetrabenzo[16]annulene-AgOTf complex (4)

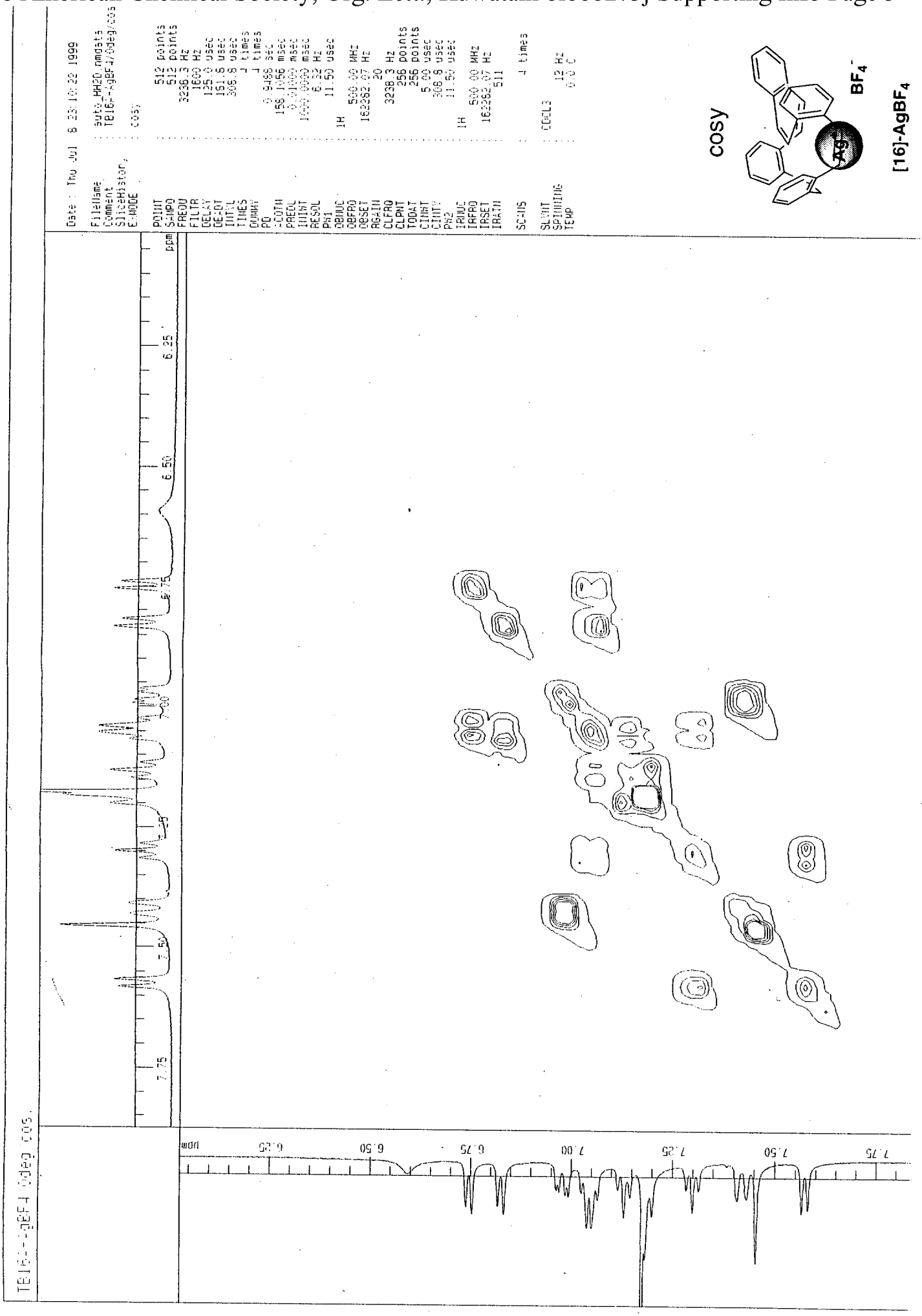
A solution of **2** (11.0 mg, 27 μmol) in THF (5.0 ml) was added AgOTf (13.0 mg, 50 μmol) and allowed to react for 30 min under argon atmosphere. The reaction mixture was concentrated to dryness under reduced pressure and the residual solid was purified by recrystallization from CH_2Cl_2-hexane. The silver complex **4** (13.0 mg, 73%) was obtained as colorless prisms: mp 169-170 °C (decomp.); ^{1}H NMR (500 MHz, $CDCl_3$, -30 °C) δ 7.69 (2H, d, J = 7.5), 7.52 (2H, s), 7.50 (2H, dd, J = 10.5, 1.5), 7.41 (2H, t, J = 7.5), 7.29 (2H, d, J = 7.5), 7.24 (2H, t, J = 7.5), 7.17 (2H, t, J = 7.5), 7.15 (2H, t, J = 7.5), 7.04 (2H, dd, J = 10.5, 3.0), 6.96 (2H, d, J = 7.5), 6.86 (2H, d, J = 7.5), 4.50 (2H, s) ppm.

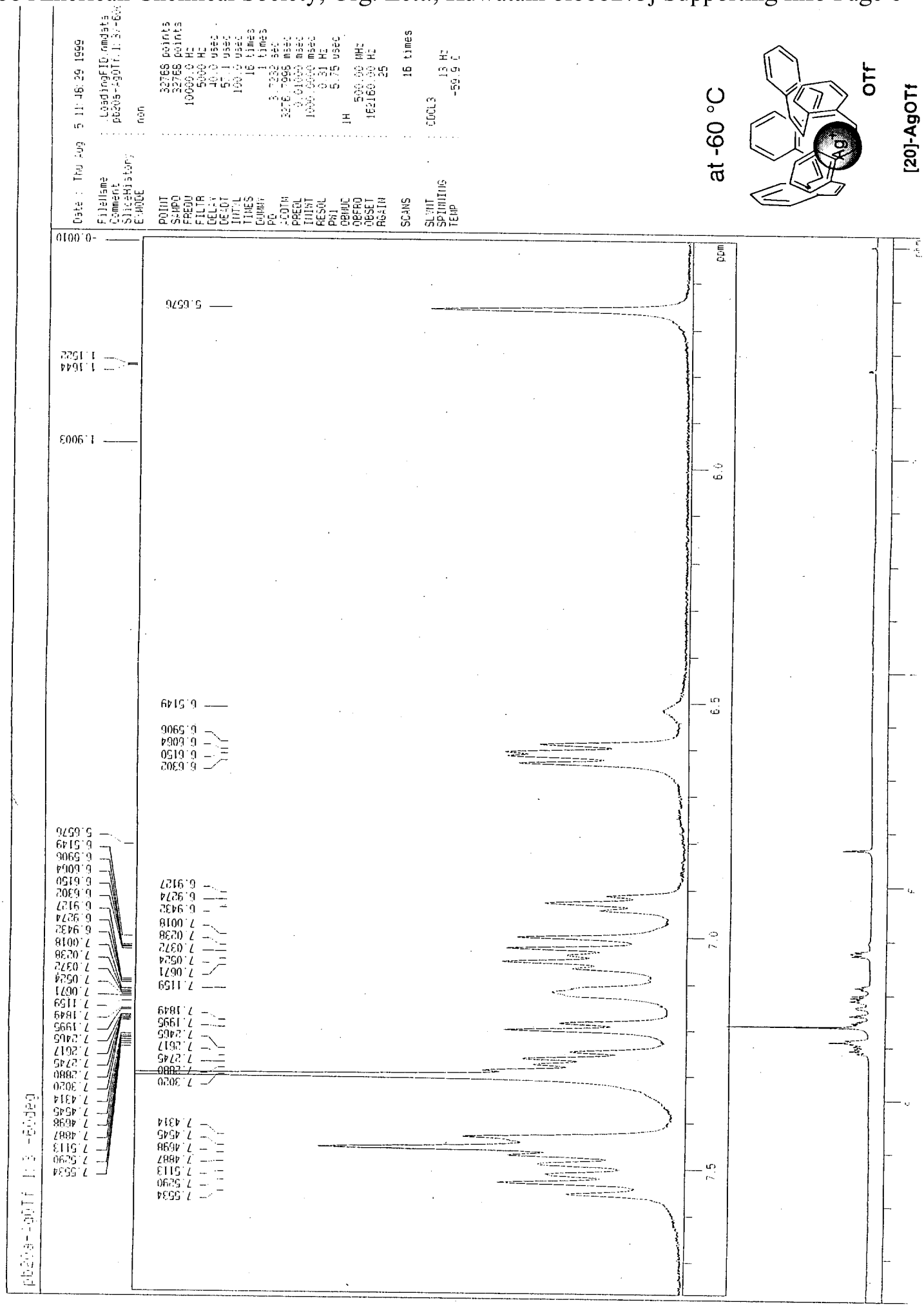
Preparation of *all-Z*-pentabenzo[20]annulene-AgOTf complex (5)

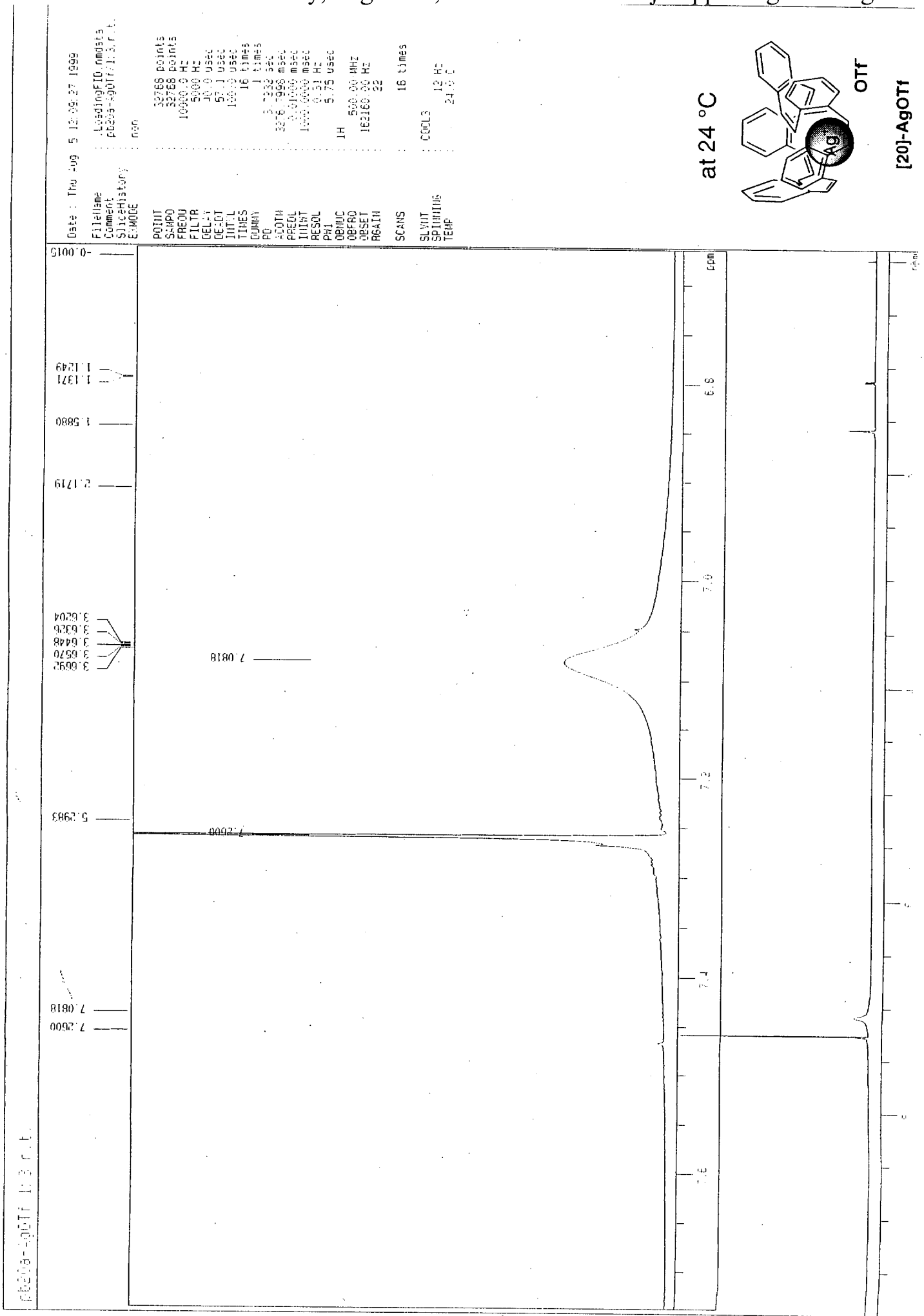
A solution of **3** (10.0 mg, 20 μmol) in THF (5.0 ml) was added AgOTf (7.5 mg, 29 μmol) and allowed to react for 30 min under argon atmosphere. The reaction mixture was concentrated to dryness under reduced pressure and the residual solid was purified by recrystallization from CH_2Cl_2-hexane. The silver complex **5** (9.0 mg, 60%) was obtained as colorless prisms: mp 196-197 °C (decomp.); ^{1}H NMR (500 MHz, $CDCl_3$, -60 °C) δ 7.54 (2H, d, J = 12.0), 7.50 (2H, d, J = 11.0), 7.47-7.43 (6H, m), 7.29-7.25 (4H, m), 7.19 (2H, d, J = 7.5), 7.12 (2H, m), 7.05 (2H, t, J = 7.5), 7.01 (2H, d, J = 12.0), 6.93 (2H, t, J = 7.5), 6.62 (2H, d, J = 7.5), 6.60 (2H, d, J = 7.5), 5.66 (2H, s) ppm.

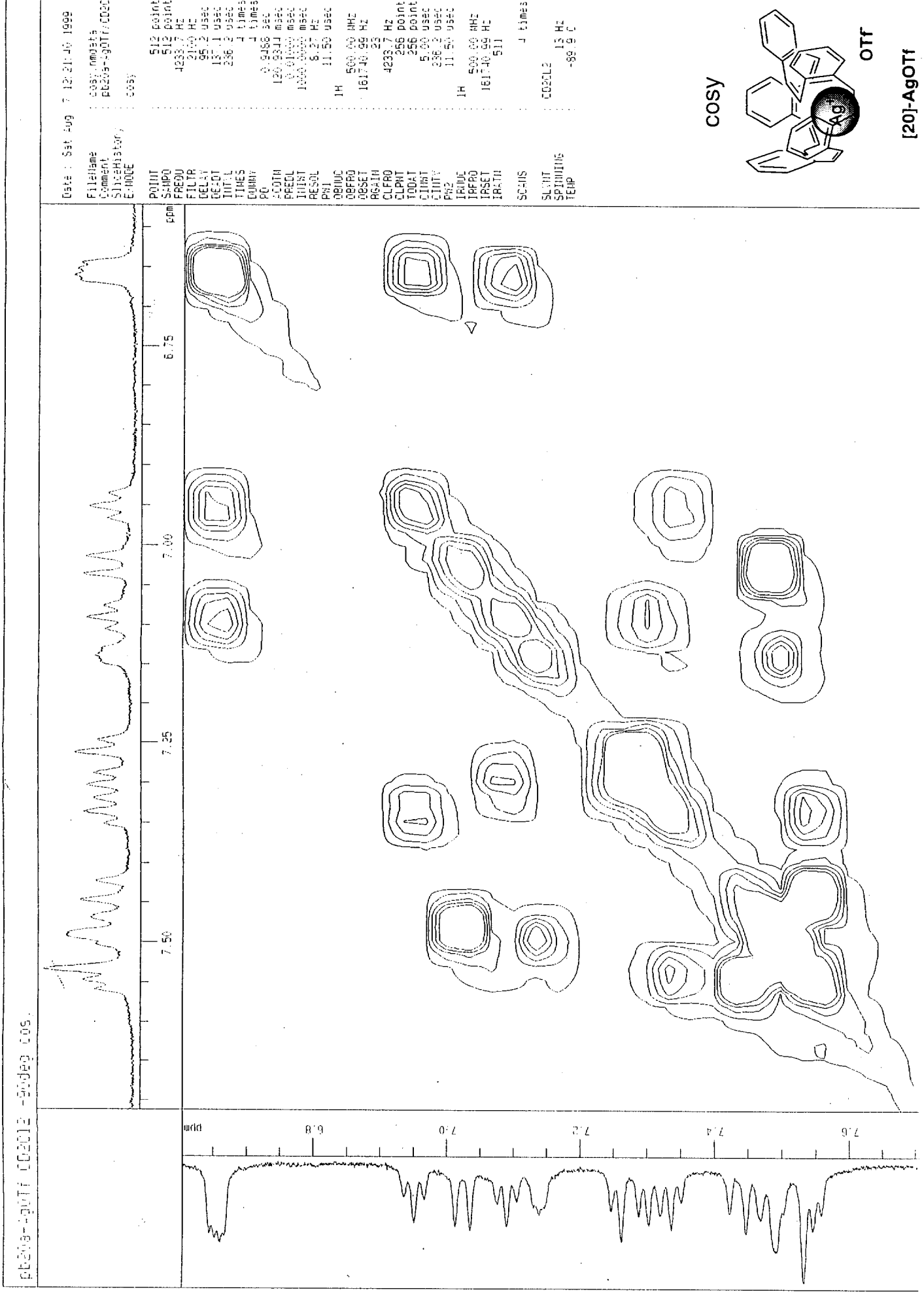




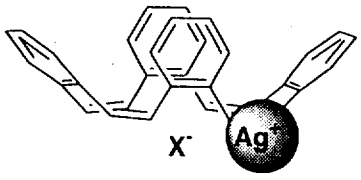




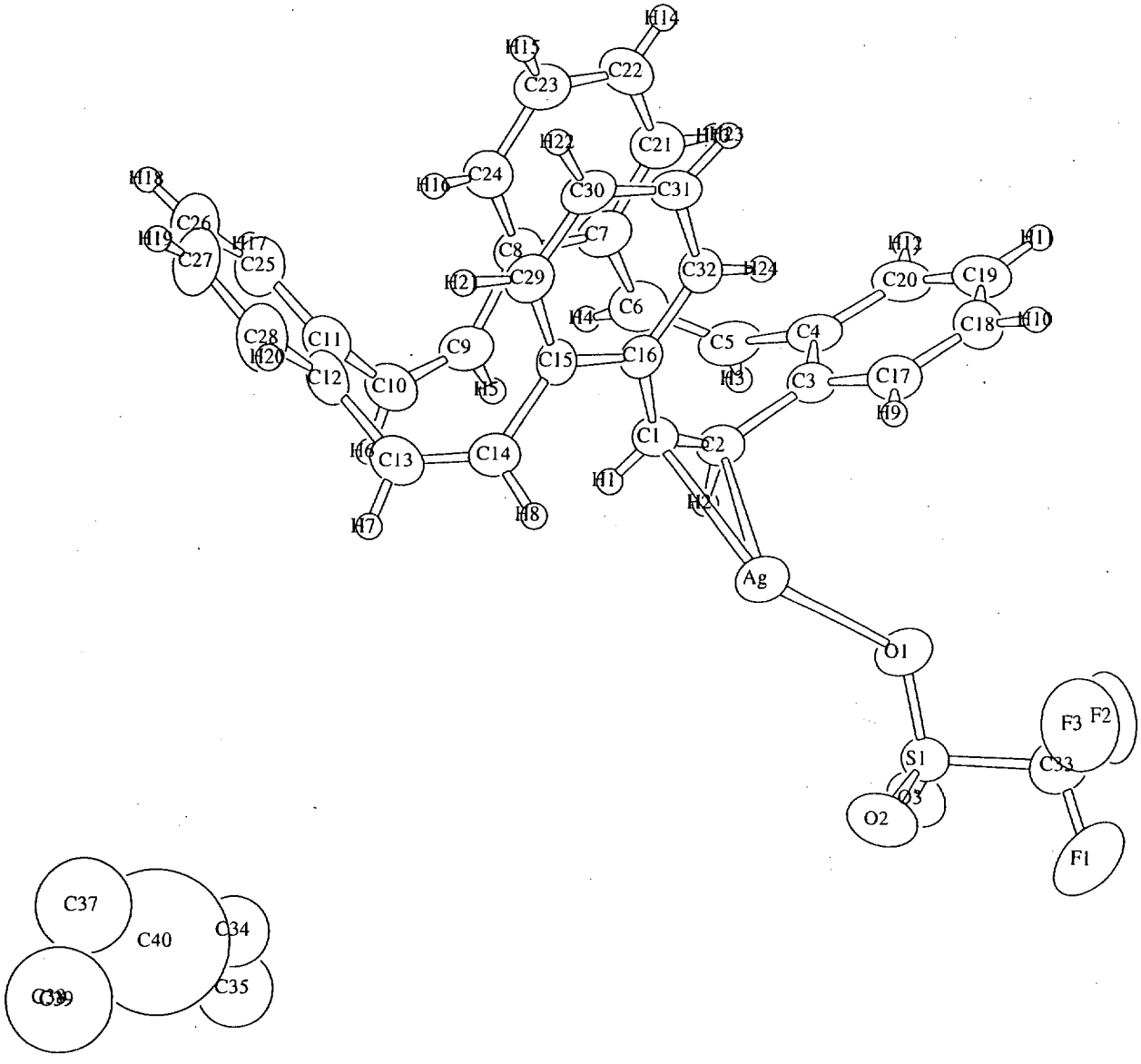




X-ray crystallographic data for **4**



[16]-AgOTf



Experimental

Data Collection

A colorless prismatic crystal of $C_{33}H_{24}O_3F_3SAg$ having approximate dimensions of 0.20 x 0.07 x 0.20 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 24 carefully centered reflections in the range $27.18 < 2\theta < 29.70°$ corresponded to a primitive triclinic cell with dimensions:

a = 11.591(5) $\AA$	α = 99.59(2)°
b = 13.850(3) $\AA$	β = 107.02(2)°
c = 10.408(2) $\AA$	γ = 87.51(3)
V = 1575.2(9) $\AA^3$	

For Z = 2 and F.W. = 665.47, the calculated density is 1.40 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$P\bar{1}$ (#2)

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 0.18° with a take-off angle of 6.0°. Scans of (0.94 + 0.35 tan θ)° were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 5 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 0.3 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 7623 reflections which were collected, 7216 were unique (R_{int} = 0.023). The intensities of three representative reflection were measured after every 150 reflections. Over the course of data collection, the standards decreased by 12.1%. A linear correction factor was applied to the data to account for this phenomenon.

The linear absorption coefficient, μ, for Mo-Kα radiation is 7.5 cm^{-1}. Azimuthal scans of several reflections indicated no need for an absorption correction. A correction for secondary extinction was applied (coefficient = 2.91000e-09).

Structure Solution and Refinement

The structure was solved by heavy-atom Patterson methods[1] and expanded using Fourier techniques[2]. Some non-hydrogen atoms were refined anisotropically, while the rest were refined isotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement[3] was based on 3595 observed reflections ($I > 3.00\sigma(I)$) and 491 variable parameters and converged (largest parameter shift was 3.93 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.049$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2} = 0.049$$

$$R1 = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.049 \quad for \;\; I > 3.0\sigma(I) \;\; data$$

The standard deviation of an observation of unit weight[4] was 1.56. The weighting scheme was based on counting statistics. 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 0.60 and -0.45 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] . Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ 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) PATTY: 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.

(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 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{33}H_{24}O_3F_3SAg$
Formula Weight	665.47
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.20 X 0.07 X 0.20 mm
Crystal System	triclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	24 (27.2 - 29.7°)
Omega Scan Peak Width at Half-height	0.18°
Lattice Parameters	a = 11.591(5) $\AA$ b = 13.850(3) $\AA$ c = 10.408(2) $\AA$ α = 99.59(2)° β = 107.02(2)° γ = 87.51(3)° V = 1575.2(9) $\AA^3$
Space Group	$P\bar{1}$ (#2)
Z value	2
D_{calc}	1.403 g/cm^3
F_{000}	672.00
μ(MoKα)	7.52 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC7R (rotating anode)
Radiation	MoKα (λ = 0.71069 $\AA$) graphite monochromated

Attenuator	Zr foil (factor = 8.53)
Temperature	23.0 °C
Voltage, Current	50 kV, 200 mA
Collimator Size	0.3 mm
Take-off Angle	6.0°
Detector Aperture	3.0 mm horizontal 3.0 mm vertical
Crystal to Detector Distance	235 mm
Scan Type	ω-2θ
Scan Rate	16.0°/min (in ω) (up to 5 scans)
Scan Width	$(0.94 + 0.35 \tan \theta)°$
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 7623 Unique: 7216 (R_{int} = 0.023)
Corrections	Lorentz-polarization Decay (12.14% decline) Secondary Extinction (coefficient: 2.91000e-09)

C. Structure Solution and Refinement

Structure Solution	Patterson Methods (DIRDIF94 PATTY)
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	0.0000
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00σ(I))	3595
No. Variables	491
Reflection/Parameter Ratio	7.32

Residuals: R; Rw	0.049 ; 0.049
Residuals: R1	0.049
No. of Reflections to calc R1	3595
Goodness of Fit Indicator	1.56
Max Shift/Error in Final Cycle	3.934
Maximum peak in Final Diff. Map	0.60 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.45 $e^-/Å^3$

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

atom	x	y	z	B_{eq}
Ag	0.77578(5)	-0.01222(4)	0.53521(6)	3.79(1)
S(1)	1.0327(1)	-0.1422(1)	0.5147(2)	3.40(4)
F(1)	1.1235(6)	-0.3079(4)	0.4402(6)	9.6(2)
F(2)	1.1019(6)	-0.2930(4)	0.6379(6)	9.1(2)
F(3)	0.9492(6)	-0.3190(4)	0.4642(7)	10.2(2)
O(1)	0.9478(4)	-0.1180(4)	0.5921(5)	4.6(1)
O(2)	0.9883(4)	-0.1331(4)	0.3746(5)	5.6(1)
O(3)	1.1525(4)	-0.1036(4)	0.5812(5)	4.5(1)
C(1)	0.6890(6)	0.1107(5)	0.6772(6)	2.8(1)
C(2)	0.7879(6)	0.0757(4)	0.7631(6)	2.9(1)
C(3)	0.7952(5)	0.0063(5)	0.8616(6)	2.8(1)
C(4)	0.8535(5)	0.0384(5)	1.0013(6)	3.3(2)
C(5)	0.9032(6)	0.1387(6)	1.0470(7)	3.8(2)
C(6)	0.8437(7)	0.2222(6)	1.0496(7)	4.0(2)
C(7)	0.7108(6)	0.2332(5)	1.0240(6)	3.4(2)
C(8)	0.6408(6)	0.2907(4)	0.9307(6)	3.3(2)
C(9)	0.6967(7)	0.3323(5)	0.8413(8)	4.0(2)
C(10)	0.6500(7)	0.3802(5)	0.7373(8)	4.4(2)
C(11)	0.5244(7)	0.4164(5)	0.6818(8)	4.5(2)
C(12)	0.4555(7)	0.3801(5)	0.5487(7)	4.2(2)
C(13)	0.5023(7)	0.2999(6)	0.4622(8)	4.5(2)
C(14)	0.5175(6)	0.2079(5)	0.4783(6)	3.5(2)
C(15)	0.4800(6)	0.1612(4)	0.5785(6)	2.8(1)
C(16)	0.5599(5)	0.1048(4)	0.6649(6)	2.7(1)

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

atom	x	y	z	B_{eq}
C(17)	0.7558(6)	-0.0902(5)	0.8192(7)	3.6(2)
C(18)	0.7708(6)	-0.1540(5)	0.9112(7)	3.8(2)
C(19)	0.8259(6)	-0.1230(6)	1.0462(7)	4.2(2)
C(20)	0.8668(6)	-0.0291(6)	1.0904(7)	3.8(2)
C(21)	0.6573(6)	0.1883(5)	1.1028(7)	3.8(2)
C(22)	0.5364(7)	0.2002(6)	1.0914(8)	4.1(2)
C(23)	0.4692(7)	0.2573(6)	1.0026(8)	4.0(2)
C(24)	0.5177(7)	0.3021(5)	0.9213(7)	3.8(2)
C(25)	0.4803(9)	0.4919(6)	0.7570(9)	5.4(2)
C(26)	0.369(1)	0.5315(6)	0.708(1)	6.2(3)
C(27)	0.3009(10)	0.4949(7)	0.578(1)	6.7(3)
C(28)	0.3436(8)	0.4198(6)	0.4978(10)	5.7(2)
C(29)	0.3600(6)	0.1728(5)	0.5835(6)	3.2(2)
C(30)	0.3192(6)	0.1255(5)	0.6708(7)	3.4(2)
C(31)	0.3973(6)	0.0649(5)	0.7500(7)	3.5(2)
C(32)	0.5150(6)	0.0558(5)	0.7470(6)	2.9(1)
C(33)	1.0537(9)	-0.2731(6)	0.5154(9)	5.4(2)
C(34)	0.963(1)	0.4803(8)	0.231(1)	7.5(3)
C(35)	1.027(1)	0.4840(9)	0.210(1)	8.8(3)
C(37)	0.803(2)	0.585(1)	0.113(2)	13.7(5)
C(38)	0.921(2)	0.631(1)	0.097(2)	13.2(5)
C(39)	0.841(2)	0.594(1)	0.007(2)	16.9(7)
C(40)	0.882(4)	0.527(3)	0.135(4)	32(1)
H(1)	0.715(5)	0.152(4)	0.638(5)	1(1)

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

atom	x	y	z	B_{eq}
H(2)	0.871(5)	0.102(4)	0.762(5)	2(1)
H(3)	0.982(6)	0.146(5)	1.075(6)	4(1)
H(4)	0.881(5)	0.284(4)	1.074(6)	3(1)
H(5)	0.775(5)	0.320(4)	0.856(5)	2(1)
H(6)	0.698(5)	0.399(4)	0.690(6)	3(1)
H(7)	0.538(5)	0.320(4)	0.400(6)	3(1)
H(8)	0.555(5)	0.162(4)	0.419(5)	2(1)
H(9)	0.731(6)	-0.110(5)	0.736(7)	5(1)
H(10)	0.738(5)	-0.222(4)	0.879(6)	3(1)
H(11)	0.841(5)	-0.164(4)	1.119(6)	4(1)
H(12)	0.907(6)	-0.008(5)	1.190(7)	4(1)
H(13)	0.715(5)	0.153(4)	1.174(6)	3(1)
H(14)	0.513(6)	0.172(5)	1.147(6)	4(1)
H(15)	0.400(5)	0.264(4)	0.990(6)	2(1)
H(16)	0.473(5)	0.337(4)	0.860(6)	2(1)
H(17)	0.520(6)	0.522(5)	0.835(6)	3(1)
H(18)	0.337(8)	0.583(6)	0.751(8)	7(1)
H(19)	0.231(7)	0.517(5)	0.542(7)	5(1)
H(20)	0.290(7)	0.388(5)	0.407(7)	6(1)
H(21)	0.308(5)	0.220(4)	0.527(6)	3(1)
H(22)	0.240(5)	0.145(4)	0.668(6)	3(1)
H(23)	0.377(6)	0.022(5)	0.810(6)	4(1)
H(24)	0.565(5)	0.012(4)	0.797(5)	2(1)

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag	0.0473(3)	0.0522(3)	0.0468(3)	0.0046(2)	0.0200(3)	0.0035(2)
S(1)	0.0367(10)	0.051(1)	0.046(1)	0.0081(8)	0.0152(8)	0.0179(8)
F(1)	0.147(6)	0.092(4)	0.135(5)	0.044(4)	0.072(5)	0.000(4)
F(2)	0.178(6)	0.077(4)	0.088(4)	0.025(4)	0.020(4)	0.043(3)
F(3)	0.135(6)	0.079(4)	0.163(6)	-0.043(4)	0.038(5)	-0.012(4)
O(1)	0.048(3)	0.073(3)	0.061(3)	0.021(3)	0.027(3)	0.018(3)
O(2)	0.051(3)	0.118(5)	0.050(3)	0.013(3)	0.011(3)	0.041(3)
O(3)	0.044(3)	0.067(3)	0.061(3)	-0.010(2)	0.010(3)	0.018(3)
C(1)	0.040(4)	0.037(4)	0.034(3)	-0.002(3)	0.016(3)	0.002(3)
C(2)	0.037(4)	0.039(4)	0.033(3)	-0.003(3)	0.013(3)	-0.001(3)
C(3)	0.030(3)	0.046(4)	0.032(3)	0.003(3)	0.010(3)	0.007(3)
C(4)	0.027(4)	0.062(5)	0.035(4)	0.005(3)	0.012(3)	0.005(3)
C(5)	0.029(4)	0.069(5)	0.043(4)	-0.004(4)	0.010(3)	-0.003(4)
C(6)	0.053(5)	0.056(5)	0.037(4)	-0.010(4)	0.011(3)	-0.006(4)
C(7)	0.039(4)	0.050(4)	0.033(4)	-0.001(3)	0.008(3)	-0.007(3)
C(8)	0.044(4)	0.039(4)	0.040(4)	-0.001(3)	0.016(3)	-0.002(3)
C(9)	0.053(5)	0.049(4)	0.054(5)	-0.002(4)	0.028(4)	0.000(4)
C(10)	0.071(6)	0.050(5)	0.059(5)	-0.003(4)	0.034(4)	0.013(4)
C(11)	0.069(5)	0.051(5)	0.064(5)	0.007(4)	0.033(4)	0.023(4)
C(12)	0.068(5)	0.040(4)	0.057(5)	0.003(4)	0.023(4)	0.022(4)
C(13)	0.070(6)	0.062(5)	0.049(5)	0.006(4)	0.025(4)	0.021(4)
C(14)	0.044(4)	0.055(4)	0.036(4)	0.006(3)	0.015(3)	0.009(3)
C(15)	0.044(4)	0.029(3)	0.030(3)	0.003(3)	0.010(3)	0.003(3)
C(16)	0.041(4)	0.030(3)	0.033(3)	0.001(3)	0.013(3)	-0.001(3)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(17)	0.036(4)	0.062(5)	0.039(4)	0.008(3)	0.012(3)	0.013(4)
C(18)	0.041(4)	0.054(5)	0.054(5)	0.004(4)	0.015(4)	0.018(4)
C(19)	0.044(4)	0.080(6)	0.044(4)	0.014(4)	0.020(4)	0.023(4)
C(20)	0.037(4)	0.074(5)	0.038(4)	0.006(4)	0.016(3)	0.013(4)
C(21)	0.046(4)	0.057(5)	0.041(4)	0.003(4)	0.015(3)	0.008(3)
C(22)	0.057(5)	0.058(5)	0.050(5)	-0.001(4)	0.024(4)	0.018(4)
C(23)	0.036(4)	0.061(5)	0.058(5)	0.001(4)	0.021(4)	0.009(4)
C(24)	0.048(5)	0.049(4)	0.049(4)	0.003(4)	0.018(4)	0.011(4)
C(25)	0.089(7)	0.048(5)	0.075(7)	0.010(5)	0.035(6)	0.012(5)
C(26)	0.104(8)	0.045(5)	0.103(8)	0.014(5)	0.051(7)	0.019(5)
C(27)	0.084(8)	0.065(6)	0.119(9)	0.040(6)	0.042(7)	0.043(6)
C(28)	0.076(6)	0.058(5)	0.082(7)	0.011(5)	0.016(5)	0.025(5)
C(29)	0.044(4)	0.041(4)	0.032(4)	0.000(3)	0.008(3)	-0.004(3)
C(30)	0.031(4)	0.052(4)	0.043(4)	0.001(3)	0.012(3)	-0.004(3)
C(31)	0.040(4)	0.051(4)	0.048(4)	0.001(3)	0.023(3)	0.007(3)
C(32)	0.034(4)	0.040(4)	0.039(4)	0.006(3)	0.010(3)	0.011(3)
C(33)	0.081(6)	0.063(5)	0.064(6)	0.007(5)	0.030(5)	0.001(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 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
Ag	O(1)	2.411(4)	Ag	O(3)	2.467(4)
Ag	C(1)	2.445(6)	Ag	C(2)	2.447(6)
Ag	C(29)	2.684(6)	Ag	C(30)	2.432(6)
S(1)	F(1)	2.580(5)	S(1)	F(2)	2.606(5)
S(1)	F(3)	2.586(6)	S(1)	O(1)	1.441(5)
S(1)	O(2)	1.422(5)	S(1)	O(3)	1.439(5)
S(1)	C(33)	1.820(8)	F(1)	F(2)	2.120(8)
F(1)	F(3)	2.122(9)	F(1)	O(2)	2.894(8)
F(1)	O(3)	2.947(7)	F(1)	C(33)	1.309(9)
F(2)	F(3)	2.120(8)	F(2)	O(1)	2.967(7)
F(2)	O(3)	2.903(7)	F(2)	C(33)	1.304(9)
F(3)	O(1)	2.880(7)	F(3)	O(2)	2.974(8)
F(3)	C(33)	1.315(10)	O(1)	O(2)	2.418(7)
O(1)	O(3)	2.427(6)	O(1)	C(33)	2.556(9)
O(2)	O(3)	2.407(7)	O(2)	C(33)	2.578(10)
O(3)	C(33)	2.556(10)	C(1)	C(2)	1.358(8)
C(1)	C(3)	2.584(9)	C(1)	C(14)	2.887(9)
C(1)	C(15)	2.459(9)	C(1)	C(16)	1.468(8)
C(1)	C(32)	2.520(9)	C(2)	C(3)	1.501(8)
C(2)	C(4)	2.507(9)	C(2)	C(5)	2.866(9)
C(2)	C(16)	2.579(9)	C(2)	C(17)	2.531(10)
C(3)	C(4)	1.416(8)	C(3)	C(5)	2.503(9)
C(3)	C(17)	1.388(9)	C(3)	C(18)	2.407(9)
C(3)	C(19)	2.777(9)	C(3)	C(20)	2.408(9)

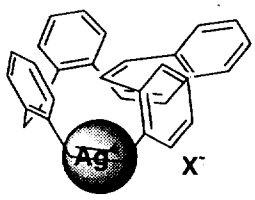
Table 3. Bond Lengths(Å) (continued)

atom	atom	distance	atom	atom	distance
C(4)	C(5)	1.475(10)	C(4)	C(6)	2.51(1)
C(4)	C(17)	2.418(10)	C(4)	C(18)	2.786(10)
C(4)	C(19)	2.41(1)	C(4)	C(20)	1.397(9)
C(5)	C(6)	1.320(10)	C(5)	C(7)	2.506(10)
C(5)	C(20)	2.52(1)	C(6)	C(7)	1.490(9)
C(6)	C(8)	2.541(10)	C(6)	C(9)	2.94(1)
C(6)	C(21)	2.463(10)	C(7)	C(8)	1.406(9)
C(7)	C(9)	2.49(1)	C(7)	C(21)	1.395(9)
C(7)	C(22)	2.409(10)	C(7)	C(23)	2.754(10)
C(7)	C(24)	2.412(10)	C(8)	C(9)	1.476(9)
C(8)	C(10)	2.563(10)	C(8)	C(21)	2.425(9)
C(8)	C(22)	2.801(10)	C(8)	C(23)	2.408(9)
C(8)	C(24)	1.405(9)	C(9)	C(10)	1.33(1)
C(9)	C(11)	2.57(1)	C(9)	C(24)	2.525(10)
C(10)	C(11)	1.50(1)	C(10)	C(12)	2.52(1)
C(10)	C(13)	2.93(1)	C(10)	C(25)	2.48(1)
C(11)	C(12)	1.401(10)	C(11)	C(13)	2.52(1)
C(11)	C(25)	1.37(1)	C(11)	C(26)	2.40(1)
C(11)	C(27)	2.75(1)	C(11)	C(28)	2.39(1)
C(12)	C(13)	1.496(10)	C(12)	C(14)	2.513(10)
C(12)	C(25)	2.40(1)	C(12)	C(26)	2.79(1)
C(12)	C(27)	2.40(1)	C(12)	C(28)	1.38(1)
C(13)	C(14)	1.310(9)	C(13)	C(15)	2.494(9)
C(13)	C(28)	2.49(1)	C(14)	C(15)	1.487(8)

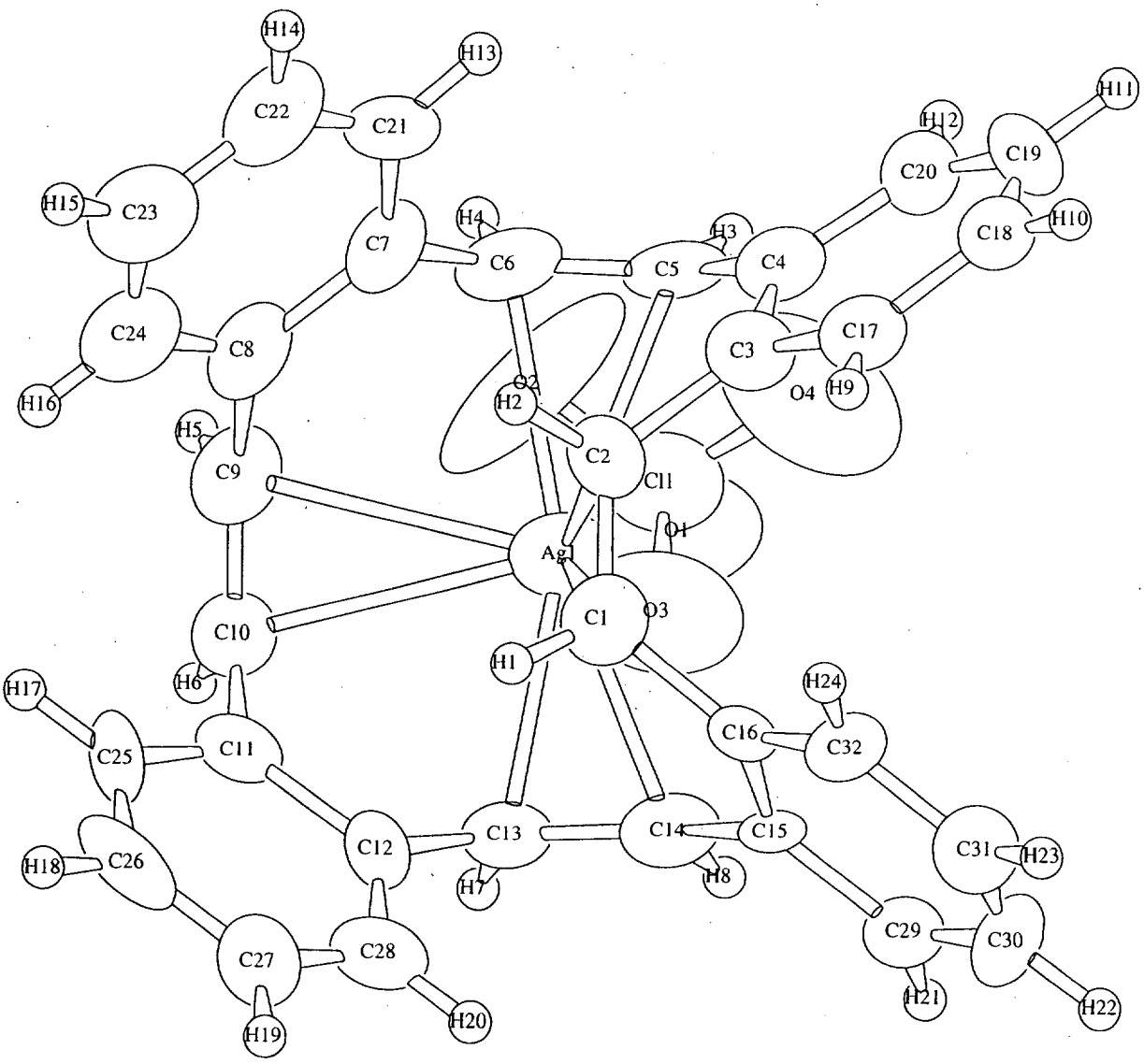
Table 3. Bond Lengths($\AA$) (continued)

atom	atom	distance	atom	atom	distance
C(14)	C(16)	2.521(9)	C(14)	C(29)	2.488(9)
C(15)	C(16)	1.399(8)	C(15)	C(29)	1.408(9)
C(15)	C(30)	2.434(9)	C(15)	C(31)	2.791(9)
C(15)	C(32)	2.399(8)	C(16)	C(29)	2.428(9)
C(16)	C(30)	2.810(9)	C(16)	C(31)	2.424(9)
C(16)	C(32)	1.396(8)	C(17)	C(18)	1.378(9)
C(17)	C(19)	2.378(10)	C(17)	C(20)	2.74(1)
C(18)	C(19)	1.366(10)	C(18)	C(20)	2.36(1)
C(19)	C(20)	1.36(1)	C(21)	C(22)	1.375(10)
C(21)	C(23)	2.36(1)	C(21)	C(24)	2.77(1)
C(22)	C(23)	1.362(10)	C(22)	C(24)	2.40(1)
C(23)	C(24)	1.378(10)	C(25)	C(26)	1.37(1)
C(25)	C(27)	2.36(1)	C(25)	C(28)	2.74(1)
C(26)	C(27)	1.38(1)	C(26)	C(28)	2.41(1)
C(27)	C(28)	1.39(1)	C(29)	C(30)	1.398(9)
C(29)	C(31)	2.407(10)	C(29)	C(32)	2.761(9)
C(30)	C(31)	1.389(9)	C(30)	C(32)	2.392(9)
C(31)	C(32)	1.374(9)	C(34)	C(35)	0.84(1)
C(34)	C(37)	2.47(2)	C(34)	C(38)	2.65(2)
C(34)	C(40)	1.39(4)	C(35)	C(37)	2.89(2)
C(35)	C(38)	2.60(2)	C(35)	C(40)	1.76(4)
C(37)	C(38)	1.61(2)	C(37)	C(39)	1.33(2)
C(37)	C(40)	1.19(4)	C(38)	C(39)	1.17(2)
C(38)	C(40)	1.67(4)	C(39)	C(40)	1.68(4)

X-ray crystallographic data for **6**



[16]-$AgClO_4$



Experimental

Data Collection

A colorless prismatic crystal of $C_{32}H_{24}AgClO_4$ having approximate dimensions of 0.30 x 0.18 x 0.18 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 16 carefully centered reflections in the range $21.95 < 2\theta < 24.36°$ corresponded to amonoclinic cell with dimensions:

a = 31.49(2) $\AA$

b = 10.018(8) $\AA$ $\beta = 127.46(2)°$

c = 20.92(1) $\AA$

V = 5238(5) $\AA^3$

For Z = 8 and F.W. = 615.86, the calculated density is 1.56 g/cm^3. Based on the systematic absences of:

hkl: h+k $\neq$ 2n

h0l: l $\neq$ 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:

C2/c (#15)

The data were collected at a temperature of 23 $\pm$ 1°C using the ω-2θ scan technique to a maximum 2θ value of 50.0°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.36° with a take-off angle of 6.0°. Scans of (0.84 + 0.30 tan θ)° were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 10.0\sigma(I)$) were rescanned (maximum of 5 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 3.5 mm (vertical).

Data Reduction

Of the 4773 reflections which were collected, 4411 were unique ($R_{int} = 0.035$). 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 9.1 cm^{-1}. Azimuthal scans of several reflections indicated no need for an absorption correction. A correction for secondary extinction was applied (coefficient = 8.27800e-08).

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 not refined. The final cycle of full-matrix least-squares refinement[3] was based on 4482 observed reflections ($I > 0.00\sigma(I)$) and 344 variable parameters and converged (largest parameter shift was 0.05 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.275$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2} = 0.092$$

$$R1 = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.065 \quad for \;\; I > 2.0\sigma(I) \;\; data$$

The standard deviation of an observation of unit weight[4] was 1.32. The weighting scheme was based on counting statistics and included a factor (p = 0.010) 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 2.04 and -3.53 $e^-/\mathring{A}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] . Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ 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) SHELXS-97: Sheldrick, G.M. (1997). Program for the Solution of Crystal Structuers. University of Goettingen, Germany.

(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 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{32}H_{24}AgClO_4$
Formula Weight	615.86
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.30 X 0.18 X 0.18 mm
Crystal System	monoclinic
Lattice Type	C-centered
No. of Reflections Used for Unit Cell Determination (2θ range)	16 (22.0 - 24.4°)
Omega Scan Peak Width at Half-height	0.36°
Lattice Parameters	a = 31.49(2) Å b = 10.018(8) Å c = 20.92(1) Å β = 127.46(2)° V = 5238(5) Å^3
Space Group	C2/c (#15)
Z value	8
D_{calc}	1.562 g/cm^3
F_{000}	2496.00
μ(MoKα)	9.06 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC7R (rotating anode)
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factor = 8.53)

Temperature	23.0 °C
Voltage, Current	50 kV, 200 mA
Collimator Size	0.5 mm
Take-off Angle	6.0°
Detector Aperture	3.0 mm horizontal 3.5 mm vertical
Crystal to Detector Distance	235 mm
Scan Type	ω-2θ
Scan Rate	16.0°/min (in ω) (up to 5 scans)
Scan Width	$(0.84 + 0.30 \tan \theta)°$
$2\theta_{max}$	50.0°
No. of Reflections Measured	Total: 4773 Unique: 4411 (R_{int} = 0.035)
Corrections	Lorentz-polarization Secondary Extinction (coefficient: 8.27800e-08)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS-97)
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	0.0100
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>0.00σ(I))	4482
No. Variables	344
Reflection/Parameter Ratio	13.03
Residuals: R; Rw	0.275 ; 0.092
Residuals: R1	0.065

No. of Reflections to calc R1	1441
Goodness of Fit Indicator	1.32
Max Shift/Error in Final Cycle	0.053
Maximum peak in Final Diff. Map	2.04 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-3.53 $e^-/\AA^3$

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

atom	x	y	z	B_{eq}
Ag(1)	0.15420(4)	0.9686(1)	0.24533(7)	5.18(3)
Cl(1)	0.2383(2)	0.9078(4)	0.1971(3)	6.1(1)
O(1)	0.2705(4)	0.949(1)	0.1757(7)	8.8(4)
O(2)	0.2678(5)	0.865(2)	0.2751(8)	15.6(7)
O(3)	0.1999(5)	1.003(1)	0.1808(8)	10.2(4)
O(4)	0.2057(7)	0.799(2)	0.144(1)	15.1(7)
C(1)	0.0485(4)	0.987(1)	0.2250(7)	3.8(3)
C(2)	0.0644(5)	0.861(1)	0.2496(7)	3.5(3)
C(3)	0.0513(5)	0.743(1)	0.1978(8)	4.2(4)
C(4)	0.0884(5)	0.675(1)	0.1935(8)	4.2(4)
C(5)	0.1460(6)	0.716(1)	0.2377(9)	4.8(4)
C(6)	0.1850(5)	0.757(1)	0.3164(9)	5.1(4)
C(7)	0.1795(4)	0.769(2)	0.3804(8)	4.3(4)
C(8)	0.1984(5)	0.887(2)	0.4277(8)	4.8(4)
C(9)	0.2233(4)	0.993(1)	0.4107(8)	4.7(4)
C(10)	0.2086(5)	1.116(1)	0.3868(8)	4.8(4)
C(11)	0.1626(5)	1.197(1)	0.3705(9)	4.1(3)
C(12)	0.1222(5)	1.239(1)	0.2898(8)	3.9(3)
C(13)	0.1283(5)	1.200(1)	0.2279(8)	3.7(3)
C(14)	0.0915(5)	1.152(1)	0.1526(8)	3.9(3)
C(15)	0.0338(5)	1.117(1)	0.1085(7)	2.9(3)
C(16)	0.0147(4)	1.040(1)	0.1419(7)	3.0(3)
C(17)	-0.0009(5)	0.692(1)	0.1515(8)	4.3(3)
C(18)	-0.0137(7)	0.580(1)	0.1039(8)	5.9(4)

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

atom	x	y	z	B_{eq}
C(19)	0.0228(7)	0.518(1)	0.0988(8)	5.9(4)
C(20)	0.0721(6)	0.567(1)	0.1420(8)	4.5(4)
C(21)	0.1597(5)	0.668(1)	0.3998(8)	4.6(4)
C(22)	0.1554(5)	0.681(2)	0.4611(9)	5.2(4)
C(23)	0.1717(6)	0.795(2)	0.5064(9)	6.0(5)
C(24)	0.1941(5)	0.899(2)	0.4898(9)	5.3(4)
C(25)	0.1571(6)	1.230(1)	0.4299(7)	5.1(4)
C(26)	0.1145(7)	1.303(1)	0.412(1)	5.4(5)
C(27)	0.0764(6)	1.343(1)	0.3344(9)	5.1(4)
C(28)	0.0799(5)	1.312(1)	0.2762(9)	4.8(4)
C(29)	-0.0017(5)	1.161(1)	0.0305(7)	3.8(3)
C(30)	-0.0564(5)	1.132(1)	-0.0158(7)	5.0(4)
C(31)	-0.0761(5)	1.062(1)	0.0177(8)	4.4(4)
C(32)	-0.0404(5)	1.016(1)	0.0950(7)	4.0(3)
H(1)	0.0503	1.0423	0.2633	4.6135
H(2)	0.0749	0.8419	0.3018	4.2440
H(3)	0.1612	0.6781	0.2143	5.8037
H(4)	0.2202	0.7347	0.3355	6.0763
H(5)	0.2596	0.9755	0.4333	5.6213
H(6)	0.2364	1.1697	0.3948	5.7260
H(7)	0.1535	1.2555	0.2298	4.4956
H(8)	0.0988	1.1795	0.1167	4.7182
H(9)	-0.0275	0.7339	0.1526	5.1140
H(10)	-0.0489	0.5444	0.0740	7.1039

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

atom	x	y	z	B_{eq}
H(11)	0.0132	0.4426	0.0652	7.0586
H(12)	0.0974	0.5266	0.1375	5.4152
H(13)	0.1486	0.5864	0.3702	5.5031
H(14)	0.1407	0.6102	0.4721	6.2482
H(15)	0.1681	0.8034	0.5481	7.2542
H(16)	0.2064	0.9779	0.5214	6.3920
H(17)	0.1834	1.2012	0.4837	6.0688
H(18)	0.1113	1.3253	0.4526	6.5270
H(19)	0.0470	1.3949	0.3221	6.0664
H(20)	0.0521	1.3397	0.2228	5.7460
H(21)	0.0111	1.2120	0.0074	4.5006
H(22)	-0.0800	1.1602	-0.0702	5.9900
H(23)	-0.1134	1.0455	-0.0120	5.2472
H(24)	-0.0536	0.9650	0.1179	4.8367

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	0.0666(7)	0.0560(7)	0.0745(8)	0.0128(7)	0.0431(6)	0.0105(7)
Cl(1)	0.075(3)	0.074(3)	0.101(4)	0.012(2)	0.064(3)	0.008(3)
O(1)	0.093(8)	0.109(9)	0.17(1)	0.005(7)	0.096(8)	0.034(9)
O(2)	0.15(1)	0.36(2)	0.12(1)	0.15(1)	0.10(1)	0.16(1)
O(3)	0.129(10)	0.11(1)	0.19(1)	0.048(8)	0.12(1)	0.009(9)
O(4)	0.22(2)	0.15(1)	0.28(2)	-0.03(1)	0.19(2)	-0.05(1)
C(1)	0.033(7)	0.06(1)	0.055(9)	0.001(7)	0.027(7)	0.002(7)
C(2)	0.058(9)	0.044(8)	0.047(8)	-0.002(7)	0.040(7)	-0.006(7)
C(3)	0.050(9)	0.046(9)	0.058(9)	-0.001(7)	0.030(8)	0.003(7)
C(4)	0.058(10)	0.046(9)	0.051(9)	0.000(8)	0.031(8)	0.012(7)
C(5)	0.10(1)	0.041(8)	0.06(1)	0.047(9)	0.06(1)	0.024(8)
C(6)	0.07(1)	0.054(9)	0.07(1)	0.026(8)	0.040(10)	0.025(9)
C(7)	0.018(7)	0.07(1)	0.040(9)	0.005(7)	0.002(7)	0.007(8)
C(8)	0.023(7)	0.08(1)	0.044(9)	0.011(7)	0.002(7)	0.008(9)
C(9)	0.024(7)	0.07(1)	0.057(9)	-0.013(7)	0.012(7)	0.005(8)
C(10)	0.055(10)	0.049(10)	0.052(10)	-0.024(8)	0.020(8)	0.003(8)
C(11)	0.034(8)	0.029(8)	0.06(1)	-0.011(6)	0.014(8)	-0.006(7)
C(12)	0.061(9)	0.037(8)	0.037(8)	-0.024(7)	0.023(8)	-0.009(7)
C(13)	0.035(7)	0.034(8)	0.058(10)	-0.008(6)	0.021(8)	0.009(7)
C(14)	0.048(8)	0.044(8)	0.066(10)	-0.008(7)	0.040(8)	0.015(7)
C(15)	0.057(8)	0.018(7)	0.030(8)	0.009(6)	0.022(7)	0.008(6)
C(16)	0.041(7)	0.022(6)	0.037(7)	0.007(6)	0.017(6)	-0.002(6)
C(17)	0.046(8)	0.043(9)	0.052(9)	-0.007(7)	0.019(7)	0.008(7)
C(18)	0.11(1)	0.040(10)	0.040(9)	-0.009(9)	0.028(10)	0.005(8)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(19)	0.11(1)	0.036(10)	0.051(10)	-0.015(9)	0.032(10)	-0.014(8)
C(20)	0.08(1)	0.05(1)	0.042(9)	0.000(8)	0.036(9)	0.002(7)
C(21)	0.043(8)	0.033(8)	0.07(1)	0.000(7)	0.016(8)	0.011(8)
C(22)	0.047(9)	0.10(1)	0.06(1)	0.028(9)	0.035(9)	0.019(10)
C(23)	0.06(1)	0.08(1)	0.08(1)	0.021(10)	0.04(1)	0.01(1)
C(24)	0.038(9)	0.08(1)	0.06(1)	0.031(8)	0.020(8)	0.013(9)
C(25)	0.10(1)	0.056(10)	0.028(8)	-0.017(9)	0.035(9)	-0.017(7)
C(26)	0.09(1)	0.040(9)	0.09(1)	-0.019(8)	0.06(1)	-0.032(9)
C(27)	0.07(1)	0.059(10)	0.05(1)	0.009(8)	0.037(10)	-0.009(9)
C(28)	0.057(10)	0.035(8)	0.07(1)	0.005(7)	0.032(9)	0.000(8)
C(29)	0.076(10)	0.037(8)	0.043(8)	0.004(7)	0.043(8)	0.006(7)
C(30)	0.047(9)	0.07(1)	0.033(8)	0.012(8)	0.003(7)	0.005(7)
C(31)	0.048(8)	0.06(1)	0.054(9)	-0.002(7)	0.028(8)	-0.002(8)
C(32)	0.054(9)	0.042(8)	0.042(8)	0.007(7)	0.022(7)	0.010(7)

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
Ag(1)	O(3)	2.53(1)	Ag(1)	C(1)	3.09(1)
Ag(1)	C(2)	3.08(1)	Ag(1)	C(5)	2.54(1)
Ag(1)	C(6)	2.43(1)	Ag(1)	C(9)	2.76(1)
Ag(1)	C(10)	2.78(1)	Ag(1)	C(13)	2.41(1)
Ag(1)	C(14)	2.52(1)	Cl(1)	O(1)	1.397(9)
Cl(1)	O(2)	1.36(1)	Cl(1)	O(3)	1.409(10)
Cl(1)	O(4)	1.45(2)	C(1)	C(2)	1.34(2)
C(1)	C(16)	1.48(2)	C(2)	C(3)	1.49(2)
C(3)	C(4)	1.40(2)	C(3)	C(17)	1.40(2)
C(4)	C(5)	1.51(2)	C(4)	C(20)	1.39(2)
C(5)	C(6)	1.39(2)	C(6)	C(7)	1.46(2)
C(7)	C(8)	1.42(2)	C(7)	C(21)	1.37(2)
C(8)	C(9)	1.49(2)	C(8)	C(24)	1.39(2)
C(9)	C(10)	1.31(2)	C(10)	C(11)	1.51(2)
C(11)	C(12)	1.43(2)	C(11)	C(25)	1.39(2)
C(12)	C(13)	1.47(2)	C(12)	C(28)	1.39(2)
C(13)	C(14)	1.36(2)	C(14)	C(15)	1.50(2)
C(15)	C(16)	1.40(1)	C(15)	C(29)	1.37(1)
C(16)	C(32)	1.40(1)	C(17)	C(18)	1.39(2)
C(18)	C(19)	1.36(2)	C(19)	C(20)	1.33(2)
C(21)	C(22)	1.38(2)	C(22)	C(23)	1.36(2)
C(23)	C(24)	1.42(2)	C(25)	C(26)	1.36(2)
C(26)	C(27)	1.36(2)	C(27)	C(28)	1.33(2)
C(29)	C(30)	1.40(2)	C(30)	C(31)	1.38(2)

Table 3. Bond Lengths($\AA$) (continued)

atom	atom	distance	atom	atom	distance
C(31)	C(32)	1.37(2)			

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
O(3)	Ag(1)	C(1)	146.5(4)	O(3)	Ag(1)	C(2)	153.6(4)
O(3)	Ag(1)	C(5)	99.9(4)	O(3)	Ag(1)	C(6)	107.6(4)
O(3)	Ag(1)	C(9)	112.8(4)	O(3)	Ag(1)	C(10)	108.3(4)
O(3)	Ag(1)	C(13)	92.2(4)	O(3)	Ag(1)	C(14)	85.6(4)
C(1)	Ag(1)	C(2)	25.0(3)	C(1)	Ag(1)	C(5)	89.4(4)
C(1)	Ag(1)	C(6)	97.5(4)	C(1)	Ag(1)	C(9)	97.4(3)
C(1)	Ag(1)	C(10)	91.7(4)	C(1)	Ag(1)	C(13)	71.7(4)
C(1)	Ag(1)	C(14)	65.1(3)	C(2)	Ag(1)	C(5)	66.0(4)
C(2)	Ag(1)	C(6)	73.1(4)	C(2)	Ag(1)	C(9)	91.8(3)
C(2)	Ag(1)	C(10)	97.9(4)	C(2)	Ag(1)	C(13)	96.2(4)
C(2)	Ag(1)	C(14)	88.5(3)	C(5)	Ag(1)	C(6)	32.4(4)
C(5)	Ag(1)	C(9)	98.0(5)	C(5)	Ag(1)	C(10)	124.8(4)
C(5)	Ag(1)	C(13)	159.6(4)	C(5)	Ag(1)	C(14)	132.4(5)
C(6)	Ag(1)	C(9)	65.9(5)	C(6)	Ag(1)	C(10)	93.1(5)
C(6)	Ag(1)	C(13)	154.8(5)	C(6)	Ag(1)	C(14)	160.1(4)
C(9)	Ag(1)	C(10)	27.3(3)	C(9)	Ag(1)	C(13)	92.4(5)
C(9)	Ag(1)	C(14)	123.5(4)	C(10)	Ag(1)	C(13)	65.3(4)
C(10)	Ag(1)	C(14)	96.8(4)	C(13)	Ag(1)	C(14)	31.8(4)
O(1)	Cl(1)	O(2)	112.2(8)	O(1)	Cl(1)	O(3)	113.8(7)
O(1)	Cl(1)	O(4)	107.0(8)	O(2)	Cl(1)	O(3)	111.1(9)
O(2)	Cl(1)	O(4)	109(1)	O(3)	Cl(1)	O(4)	102.8(8)
Ag(1)	O(3)	Cl(1)	117.5(7)	Ag(1)	C(1)	C(2)	76.7(7)
Ag(1)	C(1)	C(16)	97.3(7)	C(2)	C(1)	C(16)	128(1)
Ag(1)	C(2)	C(1)	78.3(7)	Ag(1)	C(2)	C(3)	96.5(7)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	C(2)	C(3)	126(1)	C(2)	C(3)	C(4)	123(1)
C(2)	C(3)	C(17)	118(1)	C(4)	C(3)	C(17)	117(1)
C(3)	C(4)	C(5)	124(1)	C(3)	C(4)	C(20)	119(1)
C(5)	C(4)	C(20)	115(1)	Ag(1)	C(5)	C(4)	110.5(8)
Ag(1)	C(5)	C(6)	69.4(8)	C(4)	C(5)	C(6)	132(1)
Ag(1)	C(6)	C(5)	78.2(7)	Ag(1)	C(6)	C(7)	105.6(9)
C(5)	C(6)	C(7)	126(1)	C(6)	C(7)	C(8)	118(1)
C(6)	C(7)	C(21)	122(1)	C(8)	C(7)	C(21)	118(1)
C(7)	C(8)	C(9)	119(1)	C(7)	C(8)	C(24)	119(1)
C(9)	C(8)	C(24)	121(1)	Ag(1)	C(9)	C(8)	96.2(8)
Ag(1)	C(9)	C(10)	77.1(9)	C(8)	C(9)	C(10)	130(1)
Ag(1)	C(10)	C(9)	75.5(9)	Ag(1)	C(10)	C(11)	99.2(8)
C(9)	C(10)	C(11)	131(1)	C(10)	C(11)	C(12)	118(1)
C(10)	C(11)	C(25)	123(1)	C(12)	C(11)	C(25)	118(1)
C(11)	C(12)	C(13)	117(1)	C(11)	C(12)	C(28)	116(1)
C(13)	C(12)	C(28)	125(1)	Ag(1)	C(13)	C(12)	109.7(8)
Ag(1)	C(13)	C(14)	78.7(8)	C(12)	C(13)	C(14)	129(1)
Ag(1)	C(14)	C(13)	69.5(7)	Ag(1)	C(14)	C(15)	113.2(7)
C(13)	C(14)	C(15)	131(1)	C(14)	C(15)	C(16)	123(1)
C(14)	C(15)	C(29)	117(1)	C(16)	C(15)	C(29)	118(1)
C(1)	C(16)	C(15)	124(1)	C(1)	C(16)	C(32)	117(1)
C(15)	C(16)	C(32)	118(1)	C(3)	C(17)	C(18)	119(1)
C(17)	C(18)	C(19)	122(1)	C(18)	C(19)	C(20)	118(1)
C(4)	C(20)	C(19)	123(1)	C(7)	C(21)	C(22)	121(1)