Sample: co256/KS-IV-222

X-ray Structure Report

for

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UBC Chemistry

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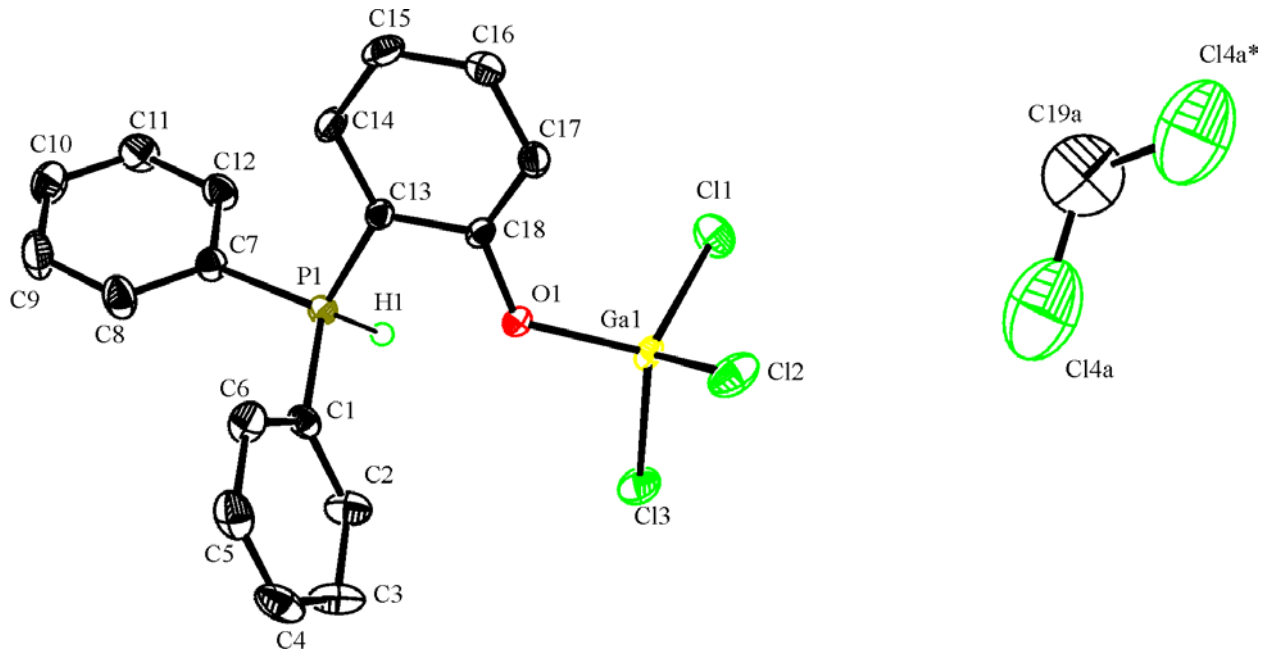
C15
C16
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Cl1
C7
P1
H1
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Ga1
C9
C8
Cl2
C6
C1
C2
C5
C3
C4
Cl3
Cl4a*
C19a
Cl4a

Experimental

Data Collection

A colourless block crystal of $C_{18}H_{15}OCl_3PGa.1/2[CH_2Cl_2]$ having approximate dimensions of 0.25 x 0.20 x 0.05 mm was mounted on a glass fiber. All measurements were made on a Rigaku/ADSC diffractometer with graphite monochromated Mo-Kα radiation.

The data were collected at a temperature of -100.0 $\pm$ 0.1^{o}C to a maximum 2θ value of 57.4^{o}. Data were collected in a series of ϕ and ω scans in 0.50^{o} oscillations with 58.0 second exposures. The crystal-to-detector distance was 39.17 mm.

Data Reduction

Of the 9559 reflections that were collected, 4272 were unique (R_{int} = 0.037); equivalent reflections were merged. Data were collected and processed the d*TREK[1] software package. The linear absorption coefficient, μ, for Mo-Kα radiation is 18.97 cm^{-1}. Data were corrected for absorption effects using a multi-scan technique (d*TREK), with normalized minimum and maximum transmission coefficients of 0.752 and 1.000, respectively. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2]. The material crystallizes with one half-molecule of CH_2Cl_2, disordered in three orientations about an inversion centre. Populations were refined such that the sum of the three Cl fragments in the asymmetric unit equaled 1 (within experimental error). Restraints were employed to maintain reasonable C-Cl bond distances. All non-hydrogen atoms except those of the disordered solvent molecule were refined anisotropically. Hydrogen H1 was located in a difference map and refined isotropically, all other hydrogen atoms were included in calculated positions but not refined. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 4272 reflections and 252 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.041$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2 \,) / \, \Sigma \, w(Fo^2)^2]^{1/2} = 0.086$$

The standard deviation of an observation of unit weight[4] was 1.06. The weighting scheme was based on counting statistics. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.64 and –0.59 $e^-/Å^3$, 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 Hubbell[8]. All refinements were performed using SHELXL-97[9].

References

(1) d*TREK. Area Detector Software. Version 4.13. Molecular Structure Corporation (1996-1998).

(2) SIR97 - Altomare A., Burla M.C., Camalli M., Cascarano G.L., Giacovazzo C. , Guagliardi A., Moliterni A.G.G., Polidori G.,Spagna R. (1999) J. Appl. Cryst. 32, 115-119.

(3) Least Squares function minimized:

$$\Sigma w(F_o^2-F_c^2)^2$$

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

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

where: N_o = number of observations
N_v = number of variables

(5) 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) Sheldrick, G. M. SHELXL-97. Programs for Crystal Structure Analysis (Release 97-2). University of Göttingen, Germany (1997).