such as water and amine molecules (Antolini, Battaglia, Bonamartini Corradi, Marcotrigiano, Menabue, Pellacani & Saladini, 1982; Antolini, Menabue, Saladini, Sola, Battaglia & Bonamartini Corradi, 1984). The coordination of peptidic O is not usual and is observed in few instances for square-pyramidal copper(II) complexes with peptides (Dehand, Jordanov, Keck, Mosset, Bonnet & Galy, 1979; Freeman, Healy & Scudder, 1977; Freeman, Robinson & Schoone, 1964; Franks & van der Helm, 1971).

In the ligand, the peptide oxygen–metal coordination causes a significant lengthening of the C(3)—O(3) distance with respect to other Cu^{II} *N*-acetyl amino acidates, but the peptide group remains strictly planar. The remaining distances and angles within the *N*-acetyl-DL-valinate moiety are consistent with those found in previously cited Cu^{II} complexes.

The presence of hydrogen bonding involving the uncoordinated carboxylic O and amide N

N—H(2)	N···O(2^{i})	H(2)···O(2^{i})	N—H(2)···O(2^{i})
0·99 Å	2·936 (4) Å	1·975 Å	162°

[(i) $x+1$, y, z] contributes to crystal packing connecting adjacent chains.

The authors are grateful to the Ministero della Pubblica Istruzione of Italy for financial support, the Centro di Calcolo Elettronico dell'Università di Modena for computing support, the Centro Interdipartimentale Grandi Strumenti dell'Università di Modena which supplied the diffractometer and the Dipartimento di Chimica dell'Università di Modena.

References

Antolini, L., Battaglia, L. P., Bonamartini Corradi, A., Marcotrigiano, G., Menabue, L., Pellacani, G. C. & Saladini, M. (1982). *Inorg. Chem.* **21**, 1391–1395, and references cited therein.

Antolini, L., Menabue, L., Saladini, M., Sola, M., Battaglia, L. P. & Bonamartini Corradi, A. (1984). *Inorg. Chim. Acta*, **93**, 61–66.

Battaglia, L. P., Bonamartini Corradi, A., Marcotrigiano, G., Menabue, L. & Pellacani, G. C. (1981). *Inorg. Chem.* **20**, 1075–1080.

Dehand, J., Jordanov, J., Keck, F., Mosset, A., Bonnet, J. J. & Galy, J. (1979). *Inorg. Chem.* **18**, 1543–1549.

Franks, W. A. & van der Helm, D. (1971). *Acta Cryst.* B**27**, 1299–1310.

Freeman, H. C., Healy, M. J. & Scudder, M. L. (1977). *J. Biol. Chem.* **252**, 8840–8847.

Freeman, H. C., Robinson, G. & Schoone, J. C. (1964). *Acta Cryst.* **17**, 719–730.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99, 101. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Marcotrigiano, G., Menabue, L. & Pellacani, G. C. (1980). *Inorg. Chim. Acta*, **46**, 107–112.

Marcotrigiano, G., Pellacani, G. C., Battaglia, L. P. & Bonamartini Corradi, A. (1976). *Cryst. Struct. Commun.* **5**, 923–926.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1988). C**44**, 2089–2091

Chloro(dimethyl sulfide)gold(I)

By Peter G. Jones* and Jürgen Lautner

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, 3400 *Göttingen,*
Federal Republic of Germany

(*Received* 16 *May* 1988; *accepted* 27 *July* 1988)

Abstract. [AuCl(C_2H_6S)], $M_r = 294{\cdot}55$, monoclinic, $P2_1/c$, $a = 6{\cdot}031\ (1)$, $b = 14{\cdot}825\ (3)$, $c = 6{\cdot}417\ (3)$ Å, $\beta = 95{\cdot}67\ (2)°$, $V = 571{\cdot}0$ Å^3, $Z = 4$, $D_x = 3{\cdot}43$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 26{\cdot}4$ mm^{-1}, $F(000) = 520$, $T = 293$ K. The structure was refined to $R = 0{\cdot}028$ for 913 observed reflections. The coordination geometry at gold is linear, with Au—S 2·271 (2), Au—Cl 2·288 (2) Å, S—Au—Cl 176·9 (1)°. The molecules are linked into chains by short Au···Au contacts of 3·226 (1) Å, with an Au···Au···Au angle of 168·1 (1)°.

Introduction. Chloro(dimethyl sulfide)gold(I) is an important synthetic intermediate in gold chemistry because the Me_2S ligand is easily replaced. Our attempts to obtain single crystals of Me_2SAuCl were unsuccessful; colourless needles were obtained from a variety of solvent mixtures but were always twinned. The crystal used in this investigation arose from an attempted reaction of Me_2SAuCl with an organic amine. Colourless prisms and tablets were obtained and mounted in glass capillaries.

Experimental. 1657 profile-fitted intensities were measured from $\omega/2\theta$ scans on a Stoe–Siemens four-circle diffractometer using monochromated Mo $K\alpha$ radiation ($2\theta_{\max}$50°, quadrant $\pm h+k+l$ and most of the

---

*Current address: Institut für Anorganische und Analytische Chemie der Technischen Universität, Hagenring 30, 3300 Braunschweig, Federal Republic of Germany.

0108-2701/88/122089-03$03.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

	x	y	z	U_{eq}*
Au	6923·9 (5)	2387·0 (3)	2275·8 (5)	36 (1)
S	4640 (3)	3589 (2)	1561 (3)	42 (1)
Cl	9201 (4)	1157 (2)	2805 (3)	53 (1)
C(1)	6241 (15)	4558 (7)	2512 (13)	48 (3)
C(2)	2575 (15)	3563 (8)	3396 (16)	58 (4)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and angles* (°)

Au—S	2·271 (2)	Au—Cl	2·288 (2)
Au···Au^i	3·226 (1)	S—C(1)	1·804 (10)
S—C(2)	1·797 (10)		

Symmetry operator: (i) x, $0·5-y$, $0·5+z$.

S—Au—Cl	176·9 (1)	Au—S—C(1)	105·3 (3)
Au—S—C(2)	107·3 (4)	C(1)—S—C(2)	100·2 (5)

quadrant $\pm h-k+l$). An absorption correction based on ψ scans was applied, with transmission factors ranging from 0·41 to 1·00 (crystal size 0·3 × 0·08 × 0·04 mm). Three repeatedly monitored check reflections increased gradually in intensity by 20%, and appropriate scaling was applied; this increase in intensity was attributed to a reduction of extinction effects on irradiation (see below). Merging equivalents gave 996 unique reflections (R_{int} 0·036, index ranges after merging h −7 to 7, k 0 to 17, l 0 to 7). Cell constants were refined from 52 2θ values in the range 20–25°.

The structure was solved by the heavy-atom method and refined anisotropically on F to R 0·028, wR 0·035 for 913 reflections with $F > 4\sigma(F)$. H atoms were included using a riding model and assuming C—H = 0·96 Å and $U(\mathrm{H}) = 0·08$ $Å^2$. The weighting scheme was $w^{-1} = \sigma^2(F) + 0·00035F^2$. An extinction correction of the form $F_{corr} = F_c/(1 + xF^2/\sin 2\theta)^{0·25}$ was applied, where x refined to $1·5\ (1) \times 10^{-5}$. The assumption that extinction effects were involved is consistent with the increase of check-reflection intensity and with the extremely narrow reflection profiles.

Details of final refinement cycles: 47 parameters, S 1·4, max. Δ/σ 0·006, max. $\Delta\rho$ 2 e $Å^{-3}$ near Au, min. $\Delta\rho$ −1·5 e $Å^{-3}$. Final atomic coordinates are presented in Table 1, with derived bond lengths and angles in Table 2.*

Discussion. The structure consists to a first approximation of discrete Me_2SAuCl molecules (Fig. 1). The expected linear coordination geometry at gold is observed [S—Au—Cl 176·9 (1)°]. Au—Cl bond lengths are not very sensitive to the nature of the other ligand (Jones, 1981, 1983, 1986), and generally lie in the range 2·27–2·29 Å for a variety of P and S ligands *trans* to Cl. The value observed here, 2·288 (2) Å, is consistent with this observation and with the individual bond lengths in, for example, $(PhCH_2)_2SAuCl$ [2·290 (9) Å; Strähle, Hiller & Conzelmann, 1984]. Surprisingly few structures of compounds with the S—Au—Cl moiety have been published; our S—Au bond length of 2·271 (2) Å agrees well with the 2·275 (4) Å in $(PhCH_2)_2SAuCl$ (Strähle *et al.*, 1984) and 2·258 (11), 2·260 (12) Å in $CH_2[S(Ph)AuCl]_2$ (Drew & Riedl, 1973). $Ph_3PSAuCl$ (Hussain & Schlemper, 1987) appears to be anomalous, with a very long Au—Cl bond of 2·555 (1) Å and a rather short Au—S bond of 2·256 (1) Å.

The extended structure (Fig. 2) is marked by short gold–gold contacts of 3·226 (1) Å, a common feature of gold(I) complexes. These contacts link the molecules into chains, in which the Au···Au···Au angle is 168·1 (1)°. Very similar packing is observed in Cl_3PAuCl (Arai, 1962); Au···Au is 3·13 Å, Au···Au···Au 162°, the space group is also $P2_1/c$ and the cell constants are very similar. Furthermore, the same symmetry operator generates the second Au atom of the Au···Au contact. However, the compounds are not

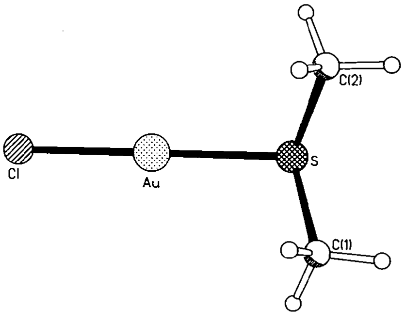

Fig. 1. The molecule of the title compound in the crystal. Radii are arbitrary.

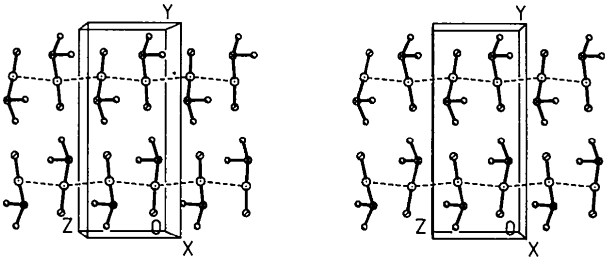

Fig. 2. Stereographic packing diagram of the title compound. H atoms are omitted for clarity.

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51269 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

isostructural, being shifted appreciably in the x and z directions with respect to each other.

We thank the Fonds der Chemischen Industrie and the Deutsche Forschungsgemeinschaft for financial assistance, and Degussa (Hanau) for a generous gift of gold metal. Calculations were performed with the program system *SHELX*76 (which incorporates atomic scattering factors), locally modified by its author Professor G. M. Sheldrick.

References

Arai, G. J. (1962). *Recl Trav. Chim. Pays-Bas*, **81**, 307–312.
Drew, M. G. B. & Riedl, M. J. (1973). *J. Chem. Soc. Dalton Trans.* pp. 52–55.
Hussain, M. S. & Schlemper, E. O. (1987). *Acta Cryst.* C**43**, 450–453.
Jones, P. G. (1981). *Gold Bull.* **14**, 102–117.
Jones, P. G. (1983). *Gold Bull.* **16**, 114–124.
Jones, P. G. (1986). *Gold Bull.* **19**, 46–57.
Strähle, J., Hiller, W. & Conzelmann, W. (1984). *Z. Naturforsch. Teil B*, **39**, 538–541.

Acta Cryst. (1988). C**44**, 2091–2093

Redetermination of the Structure of Cyano(triphenylphosphine)gold(I)

By Peter G. Jones* and Jürgen Lautner

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, 3400 *Göttingen,*
Federal Republic of Germany

(*Received* 23 *May* 1988; *accepted* 4 *July* 1988)

Abstract. $[Au(C_6H_5)_3P(CN)]$, $M_r = 485{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 10{\cdot}388\,(2)$, $b = 12{\cdot}420\,(2)$, $c = 13{\cdot}057\,(2)$ Å, $V = 1684{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}91$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 8{\cdot}8$ mm^{-1}, $F(000) = 920$, $T = 293$ K. The structure was refined to $R = 0{\cdot}037$ for 4202 unique observed reflections. The absolute structure was determined with an η refinement. The coordination geometry at gold is linear, with Au—P 2·278 (2), Au—C 2·003 (7) Å, P—Au—C 177·0 (2)°. Considerable distortions of the structure (involving the light atoms) were observed until systematic errors (extinction, absolute structure) were minimized.

Introduction. Many linear gold(I) complexes of general formula Ph_3PAuX crystallize isostructurally in space group $P2_12_12_1$ (Barron, Engelhardt, Healy, Oddy & White, 1987; Jones, 1981, 1983, 1984*a*, 1986; Ahrland, Dreisch, Norén & Oskarsson, 1987, and references therein). Presumably the bulky PPh_3 groups determine the packing. An early structure from this series was that of the cyano derivative Ph_3PAuCN (Bellon, Manassero & Sansoni, 1969). The structure was determined from photographic intensity measurements, which limited its precision; the bond lengths at gold were Au—P 2·27 (1), Au—C 1·85 (4) Å, and the bond angle P—Au—C was 169 (2)°. The Au—C bond thus appeared unusually short, and despite its high e.s.d. is often quoted as an example of a short Au—C bond. We decided to redetermine the structure to obtain a more precise value.

Experimental. The title compound was prepared by the method of Nichols & Charleston (1969) and recrystallized as colourless prisms by diffusion of petroleum ether into a solution in dichloromethane.

A crystal 0·6 × 0·2 × 0·1 mm was mounted in a glass capillary on a Stoe–Siemens four-circle diffractometer. Using monochromated Mo $K\alpha$ radiation, 5950 profile-fitted intensities (Clegg, 1981) were measured ($2\theta_{\max}$ 60°, octants $+h+k+l$ and $-h-k-l$). Merging equivalents gave 4874 reflections ($R_{\rm int}$ 0·016, index ranges after merging and reorienting h −14 to 14, k 0 to 17, l 0 to 18). An absorption correction based on φ scans was applied, with transmission factors 0·73–0·96; to avoid problems with Friedel opposites, only reflections with one zero index were used for this correction. Cell constants were refined from 2θ values of 60 reflections in the range 20–25°.

The coordinates of Bellon *et al.* (1969) were used as a starting model. Refinement proceeded to a final $R = 0{\cdot}037$, $wR = 0{\cdot}036$ [full matrix on F, all non-H atoms anisotropic, H-atoms included using a riding model, 4202 reflections $>4\sigma(F)$]. The absolute structure (Jones, 1984*b*) was determined by an η refinement (Rogers, 1981); η refined to −1·08 (3), whereupon the structure was inverted to give the final model. Six low-angle reflections with $F_o \ll F_c$ were omitted; additionally, an extinction correction of the form $F_{\rm corr} = F_c(1 + xF^2/\sin 2\theta)^{-0{\cdot}25}$ was applied, where x refined to $8\,(1) \times 10^{-7}$. The weighting scheme was $w^{-1} = \sigma^2(F) + 0{\cdot}00025F^2$.

---

[*] Current address: Institut für Anorganische und Analytische Chemie der Technischen Universität, Hagenring 30, 3300 Braunschweig, Federal Republic of Germany.

0108-2701/88/122091-03$03.00