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TRIS(N,N-DIETHYLDITHIOCARBAMATO)GOLD(III), $C_{15}H_{30}AuN_3S_6$

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Preliminary information. This complex (abbreviated as $Au(dtc)_3$) was first synthesized by Åkerström (1959). During the course of studies on the redox properties of dithiocarbamate complexes of transition metals, it was synthesized at the Inorganic Chemistry Department of this University. Magnetic susceptibility measurements revealed the complex to be diamagnetic ($\chi_m = -80 \times 10^{-6}$ (cgsu)), ruling out an octahedral coordination geometry of the gold atom. The X-ray diffraction study was undertaken to establish the coordination geometry of the gold atom.

Crystal data. Monoclinic, $a = 7.512(2)$, $b = 15.479(3)$, $c = 20.762(3)$ Å, $\beta = 90.46(10)^\circ$. (From zero-layer Weissenberg photographs with Pt calibration, $\lambda(CuK\alpha) = 1.5418$, $\lambda(CuK\alpha_1) = 1.5405$, $\lambda(CuK\alpha_2) = 1.5443$, $a(Pt) = 3.9231$ Å). Space group $P2_1/c$. $D_m = 1.79(3)$ g cm $^{-3}$, $D_c = 1.77$ g cm $^{-3}$ for $Z = 4$.

Intensity data, structure determination and refinement.

2489 independent reflections with $\sin \theta / \lambda \leq 0.46$ Å $^{-1}$ were measured on a Nonius automatic three-circle single-crystal diffractometer ($\theta - 2\theta$ scan), employing Ni-filtered Cu radiation ($\lambda = 1.5418$ Å, linear absorption coefficient $\mu = 161.3$ cm $^{-1}$). Conventional data reduction and absorption correction were applied to the measured intensities. For the latter

correction (calculated according to the Busing and Levy(1957) scheme) 10 x 8 x 16 volume elements and 6 accurately located boundary planes were taken into account. (Minimum and maximum absorption factors in the intensities 1.75 and 4.42 respectively.) Of the complete data set of 2489 reflections, 608 reflections with $I \leq 3\sigma(I)$, ($\sigma(I)$ based on counting statistics), were considered to be insignificantly different from the background and not used in the structure refinement. The structure was solved from a three-dimensional sharpened Patterson map (sharpening function: $(1/6 + \sin^2\theta/\lambda^2) \cdot 1/\bar{f}^2 \cdot \exp(A \cdot \sin^2\theta/\lambda^2)$, with $A = 4.0$ and $\bar{f} = \sum f_j / \sum Z_j$; Jacobson, Wunderlich and Lipscomb(1961)). The structure was refined by means of a full-matrix least-squares refinement program, minimizing the function $\sum w[|F_o| - K|F_c|]^2$. The weight w for each reflection was calculated according to the formula $w = [\sigma_c^2 + (0.04 F_o)^2]^{-1}$, where σ_c^2 is based on counting statistics. Refined parameters: atomic coordinates and anisotropic temperature factors for all non-hydrogen atoms. The hydrogen atoms were placed in the maxima (0.2 to $0.5 \text{ e.}\text{\AA}^{-3}$) of a difference map, at fixed distances of 1.09 \AA from the parent carbon atoms and not refined. The atomic scattering factors used were those of Au^+ , S, N, C and H as given in the International Tables for X-ray Crystallography(1962). Parameter shifts in the last refinement cycle were less than 0.25 e.s.d. Final conventional R-value: 0.046 .

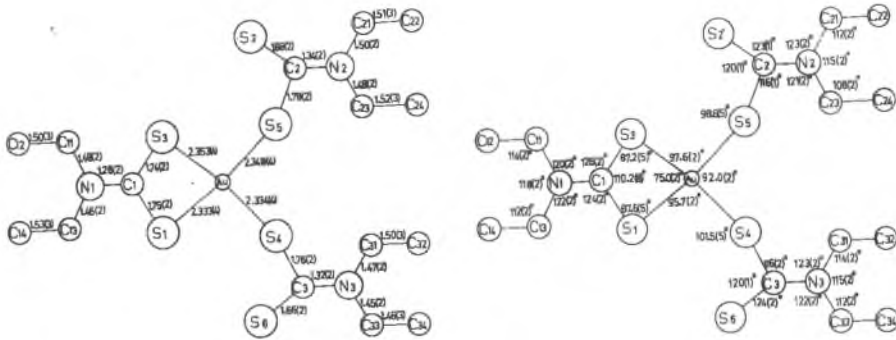
Fractional atomic coordinates of non-hydrogen atoms (with e.s.d.)

Atom	x/a	y/b	z/c	Atom	x/a	y/b	z/c
Au	0.91951(9)	0.08645(4)	0.21035(3)	C(13)	0.5689(21)	0.3638(9)	0.2203(8)
S(1)	0.8633(5)	0.2312(3)	0.2342(2)	C(14)	0.5205(25)	0.3726(10)	0.2915(8)
S(2)	1.1584(6)	0.0641(2)	0.0906(2)	C(2)	1.0926(18)	-0.0356(9)	0.1070(7)
S(3)	0.6325(5)	0.1134(2)	0.1687(2)	C(21)	1.0851(21)	-0.1950(9)	0.0883(8)
S(4)	1.1921(6)	0.0798(3)	0.2648(2)	C(22)	0.9191(25)	-0.2218(11)	0.0524(9)
S(5)	0.9488(6)	-0.0558(3)	0.1727(2)	C(23)	1.2458(19)	-0.0950(8)	0.0131(7)
S(6)	0.9140(6)	0.0211(3)	0.3556(2)	C(24)	1.4403(23)	-0.1080(10)	0.0309(8)
N(1)	0.5351(17)	0.2767(7)	0.1957(5)	C(3)	1.1258(21)	0.0453(9)	0.3417(7)
N(2)	1.1387(16)	-0.1044(7)	0.0719(5)	C(31)	1.4395(21)	0.0609(10)	0.3724(8)
N(3)	1.2542(18)	0.0368(8)	0.3852(6)	C(32)	1.4816(22)	0.1532(10)	0.3887(9)
C(1)	0.6527(21)	0.2170(9)	0.1996(6)	C(33)	1.2197(23)	0.0099(11)	0.4506(8)
C(11)	0.3550(20)	0.2577(10)	0.1698(8)	C(34)	1.2327(26)	-0.0833(13)	0.4581(9)
C(12)	0.3222(23)	0.2940(11)	0.1040(8)				

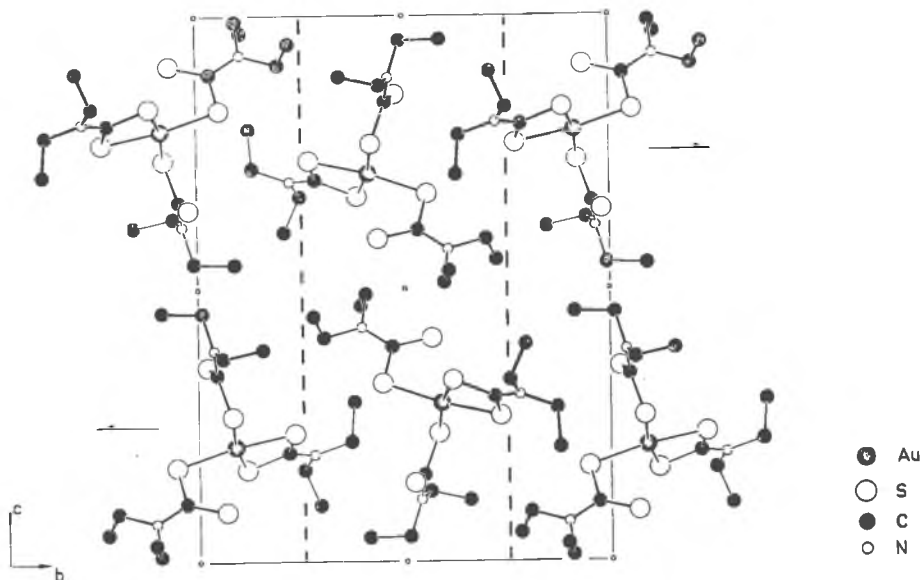
Fractional coordinates of hydrogen atoms (not refined, see text).

Atom	x/a	y/b	z/c	Atom	x/a	y/b	z/c
H(111)	0.25	0.28	0.20	H(231)	1.20	-0.14	-0.02
H(112)	0.33	0.19	0.17	H(232)	1.23	-0.03	-0.01
H(121)	0.40	0.27	0.07	H(241)	1.46	-0.17	0.05
H(122)	0.18	0.27	0.09	H(242)	1.49	-0.06	0.07
H(123)	0.32	0.36	0.10	H(243)	1.52	-0.10	-0.01
H(131)	0.49	0.41	0.19	H(311)	1.47	0.05	0.32
H(132)	0.71	0.38	0.21	H(312)	1.53	0.02	0.40
H(141)	0.54	0.43	0.30	H(321)	1.44	0.17	0.44
H(142)	0.61	0.34	0.32	H(322)	1.41	0.20	0.36
H(143)	0.38	0.35	0.30	H(323)	1.61	0.17	0.38
H(211)	1.19	-0.24	0.08	H(331)	1.32	0.04	0.48
H(212)	1.06	-0.20	0.14	H(332)	1.09	0.03	0.47
H(221)	0.80	-0.18	0.06	H(341)	1.19	-0.11	0.51
H(222)	0.95	-0.21	0.00	H(342)	1.35	-0.11	0.45
H(223)	0.88	-0.29	0.07	H(343)	1.15	-0.12	0.42

The first two numbers of the hydrogen atom numbering refer to the numbers of the parent carbon atoms.



Dimensions of $\text{Au}(\text{dte})_3$, with estimated standard deviations



Projection of the structure along a.

Comments. One of the dithiocarbamate groups in $\text{Au}(\text{dte})_3$ is functioning as a bidentate ligand, whereas the other two are functioning as monodentate ligands. The coordination geometry of the gold atom is best described as square-planar. The atoms Au, S(1), S(3), S(4) and S(5) have distances to their weighted mean plane of 0.0003(6), 0.085(4), -0.087(4), -0.090(4) and 0.081(4) Å respectively. The sulfur atoms S(2) and S(6) (not coordinated to the gold atom) are situated at distances of 2.789(4) and -2.885(5) Å from this plane.

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