Related literature. This complex is the second example of a gold(II)–carboxylate complex and is one in a series of dinuclear gold(II) ylide complexes containing pseudo-halogen ligands (Porter, Knachel & Fackler, 1986; Murray, Mazany & Fackler, 1985; Porter & Fackler, 1986).

These studies are supported by the National Science Foundation, grant CHE-8408414, the donors of the Petroleum Research Fund as administered by the American Chemical Society and the Welch Foundation. Preparation of the starting material used in this work by H. H. Murray is gratefully acknowledged. LCP is supported by a fellowship from the Welch Foundation.

References

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

MURRAY, H. H., MAZANY, A. M. & FACKLER, J. P. JR (1985). *Organometallics*, **4**, 154–157.

PORTER, L. C. & FACKLER, J. P. JR (1986). *Acta Cryst.* C**42**, 1128–1131.

PORTER, L. C., KNACHEL, H. C. & FACKLER, J. P. JR (1986). *Acta Cryst.* C**42**, 1125–1128.

SHELDRICK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Gottingen.

Acta Cryst. (1986). C**42**, 1648–1649

Structure of the Propellant Oxalylhydroxamic Acid

BY CHARLOTTE K. LOWE-MA AND DAVID L. DECKER

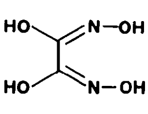
Chemistry Division, Research Department, Naval Weapons Center, China Lake, CA 93555, *USA*

(*Received* 27 *December* 1985; *accepted* 30 *May* 1986)

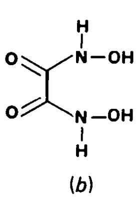
Abstract. $C_2H_4N_2O_4$, $M_r = 120{\cdot}06$, monoclinic, $P2_1/n$, $a = 5{\cdot}210\,(2)$, $b = 3{\cdot}871\,(1)$, $c = 10{\cdot}730\,(3)$ Å, $\beta = 95{\cdot}45\,(3)°$, $V = 215{\cdot}41\,(11)$ Å^3, $Z = 2$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å (graphite monochromator), $\mu = 1{\cdot}69$ cm^{-1}, $D_m = 1{\cdot}85$, $D_x = 1{\cdot}852$ g cm^{-3}, $F(000) = 124$, room temperature (291 K), least-squares refinement, final $R = 0{\cdot}037$ for 563 reflections. The molecule contains a crystallographic center of symmetry. The —C=O bond length of 1·232 (2) Å and the presence of an experimentally determined $>$N—H group indicate that the molecule exists as the keto tautomer rather than as the glyoxime form. There is intermolecular hydrogen bonding between the H of the hydroxy group and the O of the carbonyl group [=O···H—O, 1·725 (2) Å].

Experimental. Crystals of the title compound and the value for D_m were provided by Dr R. L. Willer of Morton Thiokol. Experimental conditions for data collection using a Nicolet $R3$ diffractometer are given in Table 1. Three check reflections (0,0,10, 020, $\bar{1}23$) were monitored every 45 reflections. No decay was observed. Absorption corrections were not applied. Lorentz and polarization corrections, including those for the graphite monochromator, were applied. Systematic absences: $h0l$, $h+l = 2n+1$; $0k0$, $k = 2n+1$. Solution with default parameters for the multi-solution program of *SHELXTL* (Sheldrick, 1983). The C, N, and O atoms were all refined anisotropically; H atoms observed on difference Fourier maps refined isotropically without constraints; minimized $[\sum w(|F_o| - k|F_c|)^2]$ by blocked-cascade least-squares algorithm of *SHELXTL*; $w = 1/[\sigma^2(F) + 0{\cdot}0001F^2]$. 45 parameters varied during the final cycle. Maximum shift to e.s.d. was 0·01 in the last cycle. The final difference Fourier map exhibited peaks and troughs ranging from +0·39 to −0·27 e Å^{-3}. Atomic scattering factors as supplied within *SHELXTL*.

Atomic coordinates and thermal parameters are given in Table 2.* A plot of the molecule with bond lengths and angles is shown in Fig. 1. The title compound is used extensively in gas generator propellant formulations. Because of its wide use, it was of interest to determine whether it exists in the solid state as the glyoxime (*a*) or the di-keto tautomer (*b*).



(*a*)



(*b*)

---

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43127 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/86/111648-02$01.50

Table 1. *Experimental conditions*

Crystal size: 0·12 × 0·16 × 0·54 mm
Number and range of reflections used for lattice parameters: 25 (11 to 39° 2θ, Mo $K\alpha$)
Scan method: $2\theta/\theta$
Scan range: $2\theta(\alpha_1)-1$ to $2\theta(\alpha_2)+1°$
Data collection range: 4 to 60° (2θ), $\lambda = 0{\cdot}71069$ Å
Range of *hkl* values: $\bar{h}k\bar{l}$, $\bar{h}kl$, $hk\bar{l}$, hkl: h −7 to +7, k 0 to +5, l −15 to +15
Scan speed: variable, 3 to 6° (2θ)/min.
Ratio background to scan time: 1·0
Number of measured reflections: 1541
Number of unique reflections: 633
$R_{\rm int}$: 0·018
Number of observed reflections used in refinement: 563 with $|F_o| > 4\sigma(F_o)$
Number of parameters refined: 45
Final R, wR, S: 0·037, 0·039, 2·4

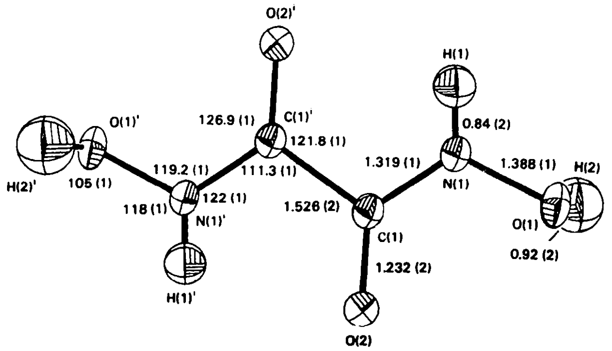


Fig. 1. The title compound plotted with 50% probability thermal ellipsoids. Bond lengths in Å; bond angles in degrees. Atoms with superscripts are related by inversion ($1-x$, $1-y$, $1-z$) to atoms without superscripts.

Table 2. *Atomic coordinates and thermal parameters*

	x	y	z	$U_{\rm eq}/U_{\rm iso}$
C(1)	0·43868 (22)	0·41114 (28)	0·44102 (9)	0·0192 (3)†
N(1)	0·56255 (20)	0·48703 (27)	0·34288 (8)	0·0218 (3)†
O(1)	0·48935 (17)	0·32773 (24)	0·22919 (7)	0·0240 (3)†
O(2)	0·24779 (19)	0·22385 (24)	0·44280 (8)	0·0277 (3)†
H(1)	0·696 (4)	0·610 (4)	0·348 (1)	0·029 (4)‡
H(2)	0·101 (4)	−0·008 (5)	0·318 (2)	0·052 (6)‡

† $U_{\rm eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.
‡ $U_{\rm iso} = \exp[-8\pi^2u(\sin\theta/\lambda)^2]$.

Related literature. The observed hydrogen-atom positions and the C(1)—O(2) bond length [1·232 (2) Å] indicate that the molecule exists primarily as the keto tautomer (*b*). The C—N bond length [1·319 (1) Å] is similar to the C—N bond lengths observed in other molecules containing an $>$N—C=O moiety (Sutton, 1965) and suggests the possibility of some resonance as $>\overset{+}{\rm N}{=}{\rm C}{-}{\rm O}^-$. Torsional angles about N(1) indicate that N(1) is slightly pyramidal but flattened {ω[O(1)—N(1)—C(1)—O(2)] = 4·9, ω[H(1)—N(1)—C(1)—C(1)$^{\rm i}$] = 2·9°}. A distance of 1·725 (2) Å between O(2) of one molecule and H(2) of another molecule indicates the presence of intermolecular hydrogen bonding.

References

Sheldrick, G. M. (1983). *SHELXTL Users Manual*, Revision 4.1. Nicolet XRD Corporation, Madison, Wisconsin.
Sutton, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions*, Spec. Publ. No. 18. London: The Chemical Society.

Acta Cryst. (1986). C**42**, 1649–1651

Redetermination of Pacifenol, a Halogenated Sesquiterpene from the Mediterranean Red Alga *Laurencia majuscula*

By Frank R. Fronczek*
Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803, *USA*

and Salvatore Caccamese
Department of Chemical Sciences, University of Catania, Viale A. Doria 6, 95125 *Catania, Italy*

(*Received* 15 *April* 1986; *accepted* 28 *May* 1986)

Abstract. (2*R*,5*R*,5a*R*,7*S*,8*S*,9a*S*)-2,7-Dibromo-8-chloro-2,5,7,8,9,9a-hexahydro-5,8,10,10-tetramethyl-2,5a-methano-6*H*-1-benzoxepin-5-ol, $C_{15}H_{21}Br_2ClO_2$, $M_r = 428{\cdot}6$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}190$ (2), $b = 19{\cdot}113$ (4), $c = 7{\cdot}004$ (3) Å, $V = 1631{\cdot}8$ (13) Å^3, $Z = 4$, $D_x = 1{\cdot}744$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 50{\cdot}8$ cm^{-1}, $F(000) = 856$, $T = 299$ K, $R = 0{\cdot}049$ for 2515 observations with $I > 0$. The C—OH distance, which was stated to be anomalously long [1·50 (1) Å] in the original, lower resolution determination, is

---

* To whom correspondence should be addressed.

0108-2701/86/111649-03$01.50