

Table 2. Intramolecular bond lengths (Å), bond angles (°) and torsion angles (°)

C(1)—O(1)	1.381 (2)	C(6a)—C(6b)	1.510 (2)
C(1)—C(2)	1.405 (2)	C(6a)—C(7)	1.531 (2)
C(1)—C(11b)	1.394 (2)	C(6b)—C(11b)	1.420 (2)
O(1)—C(1')	1.412 (3)	C(7)—C(7a)	1.510 (2)
C(2)—O(2)	1.368 (2)	C(7a)—C(8)	1.397 (2)
C(2)—C(3)	1.375 (2)	C(7a)—C(11a)	1.391 (2)
O(2)—C(2')	1.415 (3)	C(8)—C(9)	1.385 (2)
C(3)—C(3a)	1.396 (2)	C(9)—O(9)	1.368 (2)
C(3a)—C(4)	1.513 (2)	C(9)—C(10)	1.396 (2)
C(3a)—C(6b)	1.388 (2)	O(9)—C(9')	1.422 (3)
C(4)—O(4)	1.428 (2)	C(10)—O(10)	1.376 (2)
C(4)—C(5)	1.506 (2)	C(10)—C(11)	1.378 (2)
C(5)—N(6)	1.468 (2)	O(10)—C(10')	1.425 (2)
N(6)—C(6')	1.467 (2)	C(11)—C(11a)	1.414 (2)
N(6)—C(6a)	1.479 (2)	C(11a)—C(11b)	1.483 (2)

C(2)—C(1)—C(11b)	121.1 (1)	C(6a)—C(6b)—C(11b)	117.7 (1)
O(1)—C(1)—C(11b)	121.0 (1)	C(3a)—C(6b)—C(11b)	120.0 (1)
O(1)—C(1)—C(2)	117.8 (1)	C(6a)—C(7)—C(7a)	108.5 (1)
C(1)—O(1)—C(1')	112.6 (1)	C(7)—C(7a)—C(11a)	118.6 (1)
C(1)—C(2)—C(3)	119.6 (2)	C(7)—C(7a)—C(8)	121.5 (2)
C(1)—C(2)—O(2)	114.3 (1)	C(8)—C(7a)—C(11a)	119.9 (2)
O(2)—C(2)—C(3)	126.0 (2)	C(7a)—C(8)—C(9)	120.9 (2)
C(2)—O(2)—C(2')	117.2 (2)	C(8)—C(9)—C(10)	119.4 (1)
C(2)—C(3)—C(3a)	120.3 (2)	C(8)—C(9)—O(9)	124.7 (1)
C(3)—C(3a)—C(6b)	120.4 (2)	O(9)—C(9)—C(10)	115.9 (1)
C(3)—C(3a)—C(4)	118.7 (1)	C(9)—O(9)—C(9')	117.3 (2)
C(4)—C(3a)—C(6b)	120.8 (1)	C(9)—C(10)—C(11)	120.2 (1)
C(3a)—C(4)—C(5)	111.4 (1)	C(9)—C(10)—O(10)	115.0 (1)
C(3a)—C(4)—O(4)	110.5 (1)	O(10)—C(10)—C(11)	124.8 (1)
O(4)—C(4)—C(5)	108.7 (2)	C(10)—O(10)—C(10')	117.0 (1)
C(4)—C(5)—N(6)	112.5 (1)	C(10)—C(11)—C(11a)	120.6 (1)
C(5)—N(6)—C(6a)	110.5 (1)	C(7a)—C(11a)—C(11)	118.9 (1)
C(5)—N(6)—C(6')	108.2 (1)	C(11)—C(11a)—C(11b)	123.0 (1)
C(6')—N(6)—C(6a)	110.4 (1)	C(7a)—C(11a)—C(11b)	118.0 (1)
N(6)—C(6a)—C(7)	110.5 (1)	C(6b)—C(11b)—C(11a)	118.7 (1)
N(6)—C(6a)—C(6b)	111.7 (1)	C(1)—C(11b)—C(11a)	123.1 (1)
C(6b)—C(6a)—C(7)	108.3 (1)	C(1)—C(11b)—C(6b)	118.2 (1)
C(3a)—C(6b)—C(6a)	122.3 (1)		

Torsion angles (°) for rings B and C

C(4)—C(3a)—C(6b)—C(6a)	0.3 (2)
C(6b)—C(3a)—C(4)—C(5)	13.3 (2)
C(3a)—C(4)—C(5)—N(6)	-45.8 (2)
C(4)—C(5)—N(6)—C(6a)	65.7 (2)
C(5)—N(6)—C(6a)—C(6b)	-48.9 (2)
N(6)—C(6a)—C(6b)—C(3a)	17.3 (2)
N(6)—C(6a)—C(7)—C(7a)	-177.1 (1)
C(6a)—C(6b)—C(11b)—C(11a)	-3.2 (2)
C(6a)—C(7)—C(7a)—C(11a)	-41.5 (2)
C(7)—C(7a)—C(11a)—C(11b)	-1.5 (2)
C(7a)—C(11a)—C(11b)—C(6b)	25.7 (2)

*Acta Cryst.* (1984). C40, 178–181

## Structures of *trans*-6,7-Dibromo-8-oxabicyclo[3.2.1]octan-3-one, C<sub>7</sub>H<sub>8</sub>Br<sub>2</sub>O<sub>2</sub>, and 6-Bromo-8-oxabicyclo[3.2.1]oct-6-en-3-one, C<sub>7</sub>H<sub>7</sub>BrO<sub>2</sub>

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(Received 7 July 1983; accepted 15 September 1983)

**Abstract.** C<sub>7</sub>H<sub>8</sub>Br<sub>2</sub>O<sub>2</sub> (1):  $M_r = 283.96$ ,  $C2/c$ ,  $T = 140$  K,  $a = 16.244$  (10),  $b = 12.500$  (8),  $c = 12.134$  (7) Å,  $\beta = 137.24$  (3)°,  $Z = 8$ ,  $V = 1673$  (2) Å<sup>3</sup>,  $D_x(140$  K) = 2.25 g cm<sup>-3</sup>,  $Mo K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu$

= 95.5 cm<sup>-1</sup>,  $R = 0.051$ , 1245 unique observed reflections,  $F(000) = 1088$ , recrystallized from dichloromethane/diethyl ether. C<sub>7</sub>H<sub>7</sub>BrO<sub>2</sub> (2):  $M_r = 203.04$ ,  $P2_1/c$ ,  $T = 140$  K,  $a = 7.900$  (2),  $b =$

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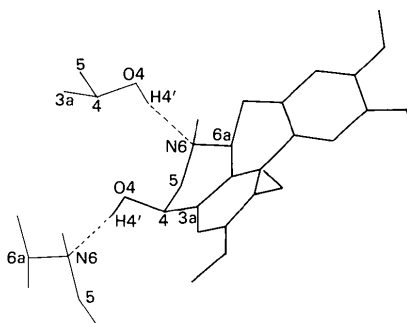


Fig. 2. A scheme of the molecule with the hydrogen bond.

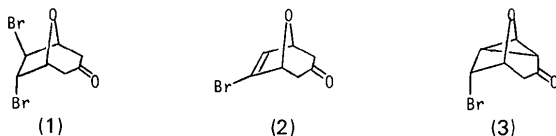
We thank Professor Castedo (Departamento de Productos Naturales, Santiago de Compostela, Spain) for the preparation and crystallization of cataline, and the Centro de Proceso de Datos, MEC, Madrid, Spain, for providing facilities for the use of the 1108 Univac computer.

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6.473 (2),  $c = 13.980$  (4) Å,  $\beta = 101.55$  (2)°,  $Z = 4$ ,  $V = 700.4$  (3) Å<sup>3</sup>,  $D_m(298\text{ K}) \approx 1.9\text{ g cm}^{-3}$ ,  $D_x(140\text{ K}) = 1.93\text{ g cm}^{-3}$ , Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 57.4\text{ cm}^{-1}$ ,  $R = 0.048$ , 1230 unique observed reflections,  $F(000) = 400$ , recrystallized from diethyl ether. Both structures display chair conformations for the cyclohexane-like pyran portions of the bicyclic systems. For comparison purposes, NMR measurements have been made and their relevance to solution structures discussed.

**Introduction.** In the course of a synthetic study aimed towards the preparation of derivatives of  $\gamma$ -tropolone, we prepared and investigated the synthetic utility of the title compounds (1) and (2). These molecules both proved to be extraordinarily resistant to what would ordinarily be considered a straightforward elimination process to open the oxygen bridge. Indeed, compound (1) undergoes an internal displacement under elimination conditions leading to highly strained tricyclic system (3) in preference to the desired simple elimination reaction (Sampath & Schore, 1983). In order to understand the peculiar chemistry of this ring system we found it desirable to determine the crystal and molecular structures of (1) and (2) and, for comparison purposes, to study the solution spectroscopy of both compounds to see if conformational effects were involved. This report presents the structures of both compounds in this context.



**Experimental.** Compound (1) obtained as nearly colorless plates, grown by slow cooling of a saturated dichloromethane/diethyl ether solution, dimensions  $0.2 \times 0.3 \times 0.5$  mm; Syntex  $P2_1$  diffractometer.  $T = 140\text{ K}$ , Mo  $K\alpha$  radiation, graphite monochromator. Unit-cell dimensions from a least-squares fit of ten reflections having  $8^\circ < 2\theta < 14^\circ$ ; space group either  $C2/c$  or  $Cc$  based on conditions  $h0l$ ,  $l = 2n$  and  $hkl$ ,  $h + k = 2n$ , and confirmed as the former by successful solution and refinement; absorption correction factors 4.2 to 4.9, absorption correction applied;\*  $\omega$  scan ( $60^\circ\text{ mm}^{-1}$ ) (Hope & Nichols, 1981),  $1.0^\circ$  range,  $1.0^\circ$  offset for background,  $2\theta_{\max} = 50^\circ$ ,  $h, k, l$  ranges 0 to 14, 0 to 14,  $-10$  to 10, respectively; two check reflections, monitored every 200 reflections, displayed a random fluctuation of  $< 1\%$ ; structure solution by direct methods (program *SIGNS*) to locate the Br atoms from  $E$  map, C and O atoms by Fourier

\* The method obtains an empirical absorption tensor from an expression relating  $F_o$  and  $F_c$  (H. Hope & B. Moezzi, unpublished results).

synthesis, 1245 unique reflections, 924 observed [ $I > 2\sigma(I)$ ] used in the solution and refinement (based on  $F$ ); full-matrix least-squares refinement, anisotropic thermal parameters for two Br and two O atoms only, H atoms included at calculated positions with a common, adjusted thermal parameter of  $U = 0.019\text{ Å}^2$ ;  $R = 0.051$ ,  $R_w = 0.053$ ,  $w = 1/[\sigma^2(F_o) + 0.0022 F_o^2]$ ,  $S = 0.999$ ,  $(\Delta/\sigma)_{\max} = 0.001$  for  $U_{12}$  of O(1),  $(\Delta/\sigma)_{\text{ave}} < 0.001$ , maximum and minimum  $\Delta\rho$  excursions  $1.1$  and  $-1.0\text{ e Å}^{-3}$ , respectively; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); computer programs from the *SHELXTL* (version 3) package (Sheldrick, 1978).

Compound (2) obtained as pale-yellow plates, grown by slow evaporation of a diethyl ether solution, dimensions  $0.25 \times 0.25 \times 0.80$  mm. Unit-cell dimensions from a least-squares fit of 18 reflections having  $36^\circ < 2\theta < 49^\circ$ ; space group  $P2_1/c$  based on conditions  $h0l$ ,  $l = 2n$  and  $0k0$ ,  $k = 2n$ ; absorption correction factors 3.1 to 4.9, absorption correction applied;\*  $\omega$  scan ( $60^\circ\text{ min}^{-1}$ ),  $1.0^\circ$  range,  $1.0^\circ$  offset for background,  $2\theta_{\max} = 50^\circ$ ,  $h, k, l$  ranges 0 to 10, 0 to 8,  $-17$  to 17, respectively; two check reflections, monitored every 100 reflections, displayed a random fluctuation of  $< 2\%$ ; structure solution by Patterson technique to locate the Br atom, followed by Fourier synthesis, 1230 unique reflections, 957 observed [ $I > 2\sigma(I)$ ] used in the solution and refinement (based on  $F$ ); full-matrix least-squares refinement, anisotropic thermal parameters for nonhydrogen atoms, H atoms included in the refinement with isotropic  $U = 0.025\text{ Å}^2$ ;  $R = 0.048$ ,  $R_w = 0.054$ ,  $w = 1/[\sigma^2(F_o) + 0.035 F_o^2]$ ,  $S = 0.975$ ,  $(\Delta/\sigma)_{\max} = 0.061$  for  $x$  of H(7B),  $(\Delta/\sigma)_{\text{ave}} = 0.008$ , final difference Fourier map displays  $\Delta\rho$  of  $1.81$  and  $1.48\text{ e Å}^{-3}$ ,  $1.18$  and  $1.16\text{ Å}$  from Br atom, respectively. Equipment, procedures, and computer programs as for compound (1).

**Discussion.** The final atomic fractional coordinates and equivalent isotropic thermal parameters for compound (1) are given in Table 1, and for compound (2) in Table 3. Bond angles and bond distances are listed in Table 2 (for 1) and Table 4† (for 2). The structure of (1) (Fig. 1) consists of discrete molecules containing the bicyclo[3.2.1]octane ring system with a bridging O atom. The carbon-oxygen bridge bonds C(3)-O(1) and C(7)-O(1) are  $1.432$  (12) and  $1.446$  (11) Å, respectively. The C(3)-O(1)-C(7) angle is  $103.3$  (7)°. The *endo* Br atom has C(1)-Br(1) of  $1.945$  (9) while for the *exo*, C(2)-Br(2) is  $1.981$  (15) Å. The strain in

\* See previous footnote.

† Lists of structure factors, anisotropic thermal parameters, and hydrogen coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38868 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the ring system is evident from the wide range of C—C bond lengths [1.475 (12) to 1.556 (20) Å] and bond angles about 'tetrahedral' carbon [99.9 (10) to 115.9 (8)°]. The carbonyl group is normal with C(5)—O(2) 1.212 (10) Å. Compound (2) (Fig. 2) is overall quite similar. For the oxygen bridge, C(3)—O(2) and C(6)—O(2) are 1.436 (7) and 1.460 (7) Å, respectively, with a C(3)—O(2)—C(6) angle of 103.7 (4)°. The vinyl C—Br bond, C(4)—Br, is 1.882 (5) Å and the double bond C(4)—C(5) is 1.324 (9) Å. The carbonyl C(1)—O(1) is 1.211 (7) Å. One previous X-ray structure determination on an 8-oxabicyclo[3.2.1]octanone derivative has been reported (Hamor, Hamor, Jenkins, Stephens & Tatlow, 1977); although more highly substituted, the molecule was found to be quite similar in structure to (1).

Most significant for our purposes is the conformation of the pyran portion of each molecule. Only three ring atoms have any degree of mobility in these systems: carbons 4, 5, and 6 in (1), and 7, 1, and 2 in (2). In both molecules the carbonyl group is found to be oriented *endo*, giving rise to a chair-like conformation for the six-membered pyran ring. This results in nearly ideal geometries for *trans*-elimination involving α-C—H bonds and bridge C—O bonds in each molecule: pertinent dihedral angles in (1) are H(4-*endo*)—C(4)—C(3)—O(1), 179°, and H(6-*endo*)—C(6)—C(7)—O(1), 173°, while in (2) H(2-*endo*)—C(2)—C(3)—O(2) is 175° and H(7-*endo*)—C(7)—C(6)—O(2) is 173°.

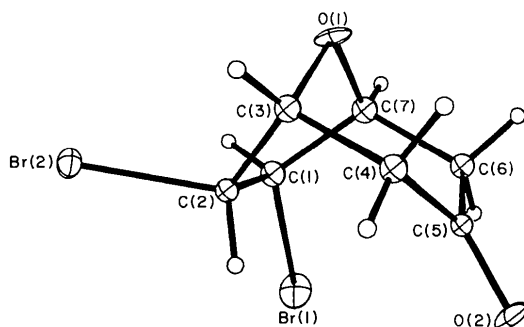


Fig. 1. *trans*-6,7-Dibromo-8-oxabicyclo[3.2.1]octan-3-one (1).

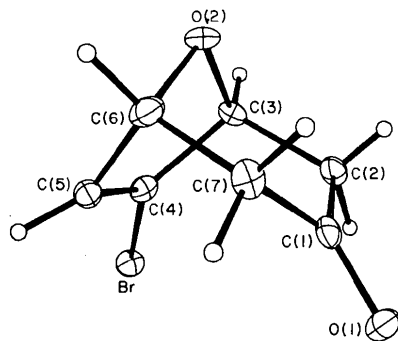


Fig. 2. 6-Bromo-8-oxabicyclo[3.2.1]oct-6-en-3-one (2).

Table 1. Atom coordinates ( $\times 10^4$ ) and temperature factors ( $\text{\AA}^2 \times 10^3$ ) for (1)

	x	y	z	U or $U_{eq}$
Br(1)	3814 (1)	2518 (1)	192 (1)	27 (1)*
Br(2)	1119 (1)	4806 (1)	-2520 (1)	17 (1)*
O(1)	1847 (6)	3498 (5)	-4388 (8)	17 (5)*
O(2)	5452 (6)	4110 (6)	-699 (8)	19 (5)*
C(1)	2524 (9)	3042 (8)	-2026 (12)	16 (2)
C(2)	2452 (8)	4246 (8)	-2141 (11)	12 (2)
C(3)	2251 (8)	4485 (8)	-3518 (11)	13 (2)
C(4)	3465 (8)	4782 (8)	-2903 (12)	13 (2)
C(5)	4380 (8)	3931 (7)	-1873 (11)	9 (2)
C(6)	3892 (8)	2801 (8)	-2374 (12)	13 (2)
C(7)	2633 (9)	2712 (8)	-3111 (12)	16 (2)

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

Table 2. Bond lengths (Å) and angles (°) for (1)

Br(1)—C(1)	1.945 (9)	Br(2)—C(2)	1.981 (15)
O(1)—C(3)	1.432 (12)	O(1)—C(7)	1.446 (11)
O(2)—C(5)	1.212 (10)	C(1)—C(2)	1.508 (14)
C(1)—C(7)	1.509 (25)	C(2)—C(3)	1.479 (22)
C(3)—C(4)	1.556 (20)	C(4)—C(5)	1.475 (12)
C(5)—C(6)	1.512 (13)	C(6)—C(7)	1.520 (20)
C(3)—O(1)—C(7)	103.3 (7)	Br(1)—C(1)—C(2)	113.3 (6)
Br(1)—C(1)—C(7)	114.7 (8)	C(2)—C(1)—C(7)	104.1 (12)
Br(2)—C(2)—C(1)	111.8 (10)	Br(2)—C(2)—C(3)	111.7 (6)
C(1)—C(2)—C(3)	104.1 (11)	O(1)—C(3)—C(2)	105.1 (9)
O(1)—C(3)—C(4)	107.0 (12)	C(2)—C(3)—C(4)	110.8 (8)
C(3)—C(4)—C(5)	111.6 (10)	O(2)—C(5)—C(4)	123.2 (8)
O(2)—C(5)—C(6)	121.4 (8)	C(4)—C(5)—C(6)	115.4 (7)
C(5)—C(6)—C(7)	112.7 (10)	O(1)—C(7)—C(1)	99.9 (10)
O(1)—C(7)—C(6)	109.2 (12)	C(1)—C(7)—C(6)	115.9 (8)

Table 3. Atom coordinates ( $\times 10^4$ ) and temperature factors ( $\text{\AA}^2 \times 10^3$ ) for (2)

	x	y	z	$U_{eq}$ *
Br	7650 (1)	-4939 (1)	5507 (1)	20 (1)
O(1)	10436 (5)	-1349 (7)	3296 (3)	22 (1)
O(2)	5344 (5)	-1224 (6)	3259 (3)	19 (1)
C(1)	8891 (7)	-1293 (9)	3256 (4)	17 (2)
C(2)	7803 (7)	-3245 (10)	3061 (4)	18 (2)
C(3)	6234 (7)	-3122 (8)	3559 (4)	14 (2)
C(4)	6870 (7)	-2749 (9)	4647 (4)	16 (2)
C(5)	6946 (7)	-739 (11)	4828 (4)	18 (2)
C(6)	6406 (8)	342 (9)	3847 (5)	20 (2)
C(7)	7978 (8)	702 (10)	3396 (4)	18 (2)

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

Table 4. Bond lengths (Å) and angles (°) for (2)

Br—C(4)	1.882 (5)	O(1)—C(1)	1.211 (7)
O(2)—C(3)	1.436 (7)	O(2)—C(6)	1.460 (7)
C(1)—C(2)	1.522 (8)	C(1)—C(7)	1.511 (9)
C(2)—C(3)	1.541 (8)	C(3)—C(4)	1.522 (7)
C(4)—C(5)	1.324 (9)	C(5)—C(6)	1.522 (9)
C(6)—C(7)	1.519 (10)		
C(3)—O(2)—C(6)	103.7 (4)	O(1)—C(1)—C(2)	120.8 (5)
O(1)—C(1)—C(7)	121.5 (5)	C(2)—C(1)—C(7)	117.7 (5)
C(1)—C(2)—C(3)	110.7 (5)	O(2)—C(3)—C(2)	107.7 (4)
O(2)—C(3)—C(4)	101.5 (4)	C(2)—C(3)—C(4)	109.0 (4)
Br—C(4)—C(3)	121.3 (4)	Br—C(4)—C(5)	128.4 (4)
C(3)—C(4)—C(5)	109.9 (5)	C(4)—C(5)—C(6)	106.6 (5)
O(2)—C(6)—C(5)	102.3 (5)	O(2)—C(6)—C(7)	107.4 (5)
C(5)—C(6)—C(7)	109.8 (5)	C(1)—C(7)—C(6)	112.0 (5)

Despite these near-perfect *trans*-periplanar relationships in the solid state, the desired eliminations have not been found to occur readily in these molecules. In order to obtain some information concerning the conformations of these systems in solution, the 360 MHz NMR spectra were measured. For either compound, a solution structure like that found in the crystal should ideally exhibit NMR couplings between a bridgehead hydrogen and the two neighboring  $\alpha$ -protons of nearly identical magnitudes (Karplus, 1959, 1963). The presence of electronegative substituents modifies these expectations somewhat (Williams & Bhacca, 1964); nonetheless, the spectra of the majority of 4-pyranone derivatives are completely consistent with chair conformations in solution (Hirsch & Havinga, 1976). In contrast, in both (1) and (2) it is found that the *exo*  $\alpha$ -protons couple strongly to the bridgehead hydrogen ( $J = 4.9\text{--}5.3$  Hz) while the *endo* protons exhibit virtually no coupling at all [ $J \sim 0$  Hz for (1), 0.8 Hz for (2)]. Similar spectroscopic results have also been obtained for certain other derivatives of this ring system and have been interpreted as resulting from a flattening of the pyran portion of the molecule (Cookson, Nye & Subrahmanyam, 1967; Vinter & Hoffmann, 1974). Such a conformational change in solution would have the effect of reducing the H( $\alpha$ -*endo*)-C-C-O dihedral angle well below the  $180^\circ$  value required for facile elimination, thus providing at least a partial explanation for the resistance shown by these molecules to this particular kind of reactivity. The uncertain effects of the additional functionality in (1) and (2) on their spectra cause us to question the validity of this interpretation in our systems, however. We see no obvious structural reason for either of these compounds to possess

different conformations in solution *vs* the solid state. Moreover, both compounds also strongly resist normally facile elimination under acidic conditions. Thus other factors unrelated to the geometry for the simple *trans*-elimination mechanism must apparently be involved in the problem. These most likely include reversibility of the desired ring-opening process.

We thank the National Institutes of Health (Grant No. PHS-GM26294) and both the Committee on Research and the Cancer Research Coordinating Committee of the University of California for financial support. NES thanks the Camille and Henry Dreyfus Foundation for a Teacher-Scholar award.

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## Structure of Telluric Acid–Glycine (1:2) Monohydrate, $\text{Te}(\text{OH})_6 \cdot 2\text{C}_2\text{H}_5\text{NO}_2 \cdot \text{H}_2\text{O}$ , at 120 K and Determination of Hydrogen Positions

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(Received 27 January 1983; accepted 2 September 1983)

**Abstract.**  $M_r = 406$ , monoclinic,  $P2_1/n$ ,  $a = 7.924$  (3),  $b = 12.154$  (5),  $c = 7.188$  (3) Å,  $\beta = 110.67$  (8)°,  $V = 648$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x$  (at 120 K) =  $2.08$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 2.45$  mm<sup>-1</sup>,  $F(000) = 392$ . Final  $R = 2.0\%$  for 1942 independent observed reflections. The structure consists of slightly distorted  $\text{Te}(\text{OH})_6$  octahedra with the Te atom placed at the center of symmetry. The Te–OH distances range from

1.905 (2) to 1.929 (2) Å [average: 1.920 (2) Å]. Glycine species are found to be in the zwitterionic form. In addition, through the hydrogen bonds such as Te–OH...O–C, each glycine group is linked to either one or two corners of the  $\text{Te}(\text{OH})_6$  octahedra.

**Introduction.** The title compound was studied as part of a systematic investigation in our laboratory of adduct