

Discussion. This investigation was undertaken as a part of a project to clarify the structure of Al_2X_7^- ions in the solid and liquid state. A linear Al–X–Al bridge gave better correspondence between observed and calculated Raman frequencies for the melt (Rytter *et al.*, 1973). However, a bent bridge was found for solid $\text{Pd}_2(\text{C}_6\text{H}_5)_2(\text{Al}_2\text{Cl}_7)_2$ and $\text{Te}_4(\text{Al}_2\text{Cl}_7)_2$ (Couch, Lokken & Corbett, 1972), the former with an eclipsed and the latter with a staggered conformation, indicating that environmental effects are important.

The anion in the present structure, shown in Fig. 1, is very similar to the anion in the $\text{Te}_4(\text{Al}_2\text{Cl}_7)_2$ structure. It is noteworthy that the Al–Br–Al angle (109.3°) is very close to the tetrahedral angle and that the bridging Al–Br distances are about 5% larger than the terminal ones. The Al_2Br_7^- ion is staggered with almost C_s symmetry, the main discrepancy being the angle of 14.8° between the planes through the atoms Br(1), Al(1), Br(4) and Br(4), Al(2), Br(7). A stereographic view of the structure is given in Fig. 2.

The potassium ion is surrounded by 9 bromine ions in the range 3.3 to 4.0 Å in an irregular way.

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Mescaline Hydrobromide

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Abstract. Mescaline hydrobromide (3,4,5-trimethoxyphenethylamine hydrobromide, $\text{C}_{11}\text{H}_{18}\text{NO}_3\text{Br}$), triclinic space group $P\bar{1}$, $a = 7.4274$ (12), $b = 9.1782$ (16), $c = 11.8979$ (9) Å, $\alpha = 121.180$ (17), $\beta = 104.194$ (18), $\gamma = 92.689$ (13)° ($20 \pm 1^\circ\text{C}$), $D_m = 1.48$ g cm $^{-3}$, $D_x = 1.48$ g cm $^{-3}$ ($Z = 2$). The hydrobromide salt was prepared by the method of Heffter [*Ber. deutsch. chem. Ges.* (1898). **31**, 1193–1199] from mescaline sulfate dihydrate

supplied by the L. Light Co. Crystals were grown from aqueous n-butanol.

Introduction. Cell parameters were determined by least-squares refinement from 22 reflections measured on a four-circle Syntex $P\bar{1}$ diffractometer using graphite monochromated $\text{Mo K}\alpha$ radiation (0.710688 Å) (Ernst, 1973). A small acicular crystal (elongated along

Table 1. Atomic parameters in fractional coordinates and thermal parameters with *e.s.d.*'s ($\times 10^4$)

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Br $^-$	–2340 (2)	–2810 (2)	8801 (1)	211 (3)	168 (2)	70 (1)	16 (2)	44 (2)	65 (2)
C(1)	3188 (14)	339 (14)	7018 (12)	88 (20)	136 (21)	80 (15)	35 (16)	23 (14)	75 (15)
C(2)	2874 (15)	–523 (13)	5605 (12)	114 (21)	86 (17)	57 (13)	26 (15)	25 (14)	34 (13)
C(3)	2348 (15)	285 (15)	4933 (11)	120 (22)	136 (21)	39 (13)	16 (17)	34 (13)	42 (14)
C(4)	2143 (15)	2081 (15)	5687 (12)	109 (22)	156 (22)	76 (15)	50 (17)	33 (15)	95 (16)
C(5)	2359 (14)	2873 (12)	7061 (11)	122 (21)	45 (15)	61 (13)	22 (14)	48 (14)	19 (12)
C(6)	2921 (14)	2054 (14)	7777 (12)	104 (21)	123 (20)	60 (14)	24 (16)	35 (14)	58 (14)
C(7)	3766 (17)	–544 (16)	7787 (14)	154 (25)	157 (23)	89 (17)	52 (20)	18 (16)	88 (17)
C(8)	2058 (19)	–1780 (17)	7625 (14)	213 (31)	156 (24)	84 (17)	53 (22)	42 (18)	73 (17)
C(9)	2250 (19)	–2190 (15)	2750 (14)	218 (31)	104 (21)	74 (16)	46 (21)	60 (18)	4 (15)
C(10)	3175 (21)	3958 (18)	5095 (16)	253 (36)	176 (27)	145 (23)	–3 (24)	76 (23)	129 (22)
C(11)	2380 (21)	5482 (17)	9153 (13)	290 (38)	149 (24)	45 (15)	78 (24)	44 (19)	36 (16)
O(3)	2030 (11)	–415 (10)	3544 (8)	152 (18)	138 (15)	44 (9)	32 (13)	38 (10)	27 (10)
O(4)	1585 (11)	2833 (10)	4969 (8)	176 (18)	138 (15)	55 (10)	20 (13)	39 (11)	69 (11)
O(5)	2034 (11)	4526 (10)	7673 (8)	169 (18)	109 (14)	50 (9)	36 (13)	34 (10)	44 (10)
N	553 (14)	–803 (13)	8072 (11)	163 (22)	142 (19)	73 (13)	31 (16)	52 (14)	60 (13)

the *c* axis) was cleaved from a large crystal. This was used to obtain two-dimensionally integrated Weissenberg photographs of layers *hk0* through *hk10* using nickel-filtered copper *K* α radiation. The crystal was *ca.* 0.14 mm in cross-section and no absorption corrections were applied [$\mu(\text{Cu})=47.4 \text{ cm}^{-1}$]. Film packs of Kodak No-Screen film were used to record the data. The intensities were measured with a Nonius Mark I microdensitometer. In all, 2184 reflections (73% of the data in the copper sphere of reflection) were measured, of which 1499 were significantly more intense than the background. The structure was solved by the heavy atom method. Refinement of positional parameters and anisotropic temperature factors (unit weights assigned to observed reflections) with hydrogen atom positions and isotropic temperature factors included (but not refined) gave a final residual ($R = \sum ||kF_o| - |F_c|| / \sum |kF_o|$, where *k* is the scale factor) of 0.081 (all reflections). The reported positions of the hydrogen atoms (Table 2) were obtained from a ΔF map prepared when $R = 0.09$. The calculations were performed with the X-RAY

system of crystallographic programs (Stewart, Kundell & Baldwin, 1970) and the illustrations were prepared with the ORTEP plotting program (Johnson, 1970). The final parameters are given in Tables 1 and 2 and the bond lengths and angles in Figs. 1 and 2. For those

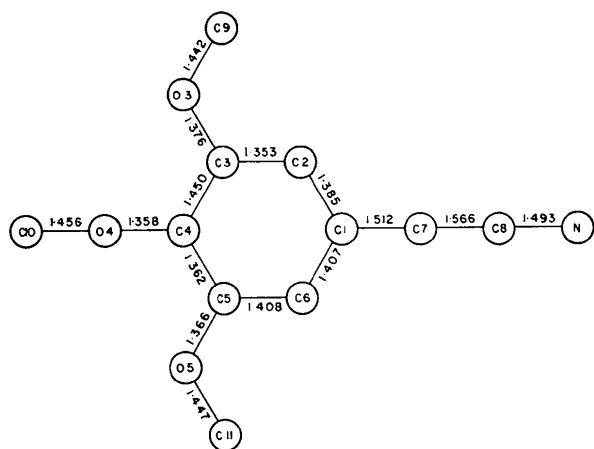


Fig. 1. Bond lengths of mescaline hydrobromide.

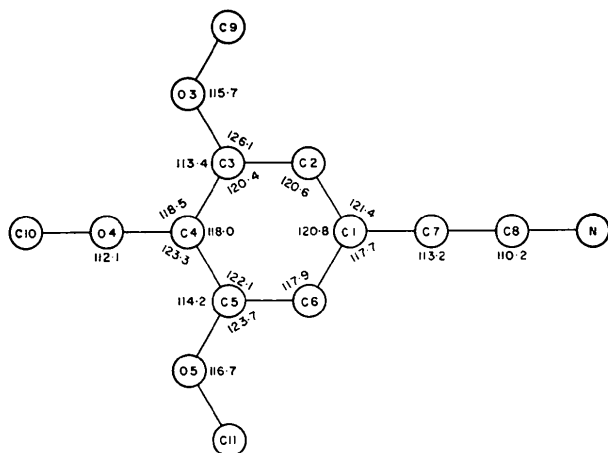


Fig. 2. Bond angles of mescaline hydrobromide.

Table 2. Hydrogen atom parameters in fractional coordinates ($\times 10^3$)

All hydrogen atoms were assigned isotropic temperature factors of 3.0 \AA^2 .

	<i>x</i>	<i>y</i>	<i>z</i>
H(2)	304	814	504
H(6)	316	279	887
H(1,7)	399	37	881
H(2,7)	491	873	735
H(1,8)	176	730	655
H(2,8)	217	755	815
H(1,9)	158	686	290
H(2,9)	229	769	169
H(3,9)	316	690	267
H(1,10)	286	435	452
H(2,10)	364	427	603
H(3,10)	423	302	452
H(1,11)	173	456	926
H(2,11)	380	551	942
H(3,11)	199	670	971
H(1,N)	14	990	774
H(2,N)	9	21	923
H(3,N)	-55	863	818

Table 3. Least-squares planes showing magnitudes of deviations in \AA

	Plane number				
	1	2	3	4	5
C(1)	0.016*				
C(2)	0.007*				
C(3)	0.015*	0.0*			
C(4)	0.029*		0.0*		
C(5)	0.021*			0.0*	
C(6)	0.002*				
C(7)	0.018				0.0*
C(8)					0.0*
C(9)		0.0*			
C(10)			0.0*		
C(11)				0.0*	
O(3)	0.012	0.0*			
O(4)	0.001		0.0*		
O(5)	0.053			0.0*	
N					0.0*
A†	6.988	7.019	-3.241	6.930	1.182
B	2.186	2.051	3.847	2.579	-3.020
C	-2.722	-2.641	6.547	-3.286	10.581
D	-2.561	-2.455	-7.676	-2.635	-5.828

Angles between planes

	2	3	4	5
1	0.9°	81.5	3.1	76.5
2		81.1	3.8	76.2
3			81.2	56.3
4				79.2

* Denotes atom included in least-squares fit.

† *A*, *B*, *C* and *D* are coefficients in the equation of a plane: $Aa + Bb + Cc + D = 0$.

bonds not involving hydrogen atoms, the e.s.d.'s in bond lengths and angles are approximately 0.02 Å and 0.7°, respectively.*

* The structure factors have been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30090. Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. Mescaline is used extensively as a prototype hallucinogen and psychotomimetic agent. This structure analysis was undertaken to provide information about the solid state conformation of the mescaline molecule. As can be seen from Figs. 3 and 4, the ethylammonium side chain is bent toward the ring. The observed N-C(1) distance of 2.98 Å may be compared to the N-C van der Waals distance of about 3.15 Å and

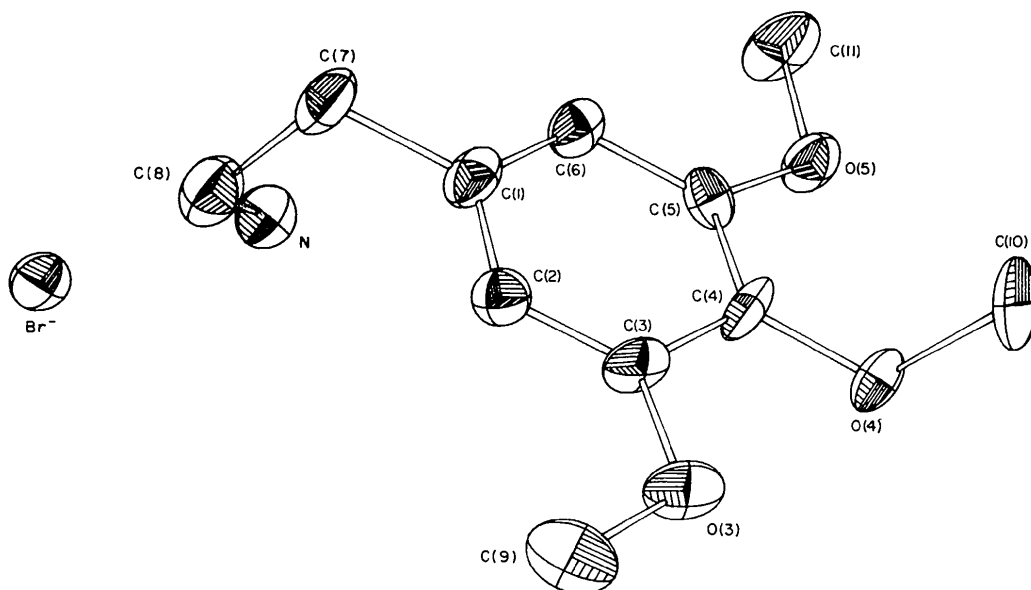


Fig. 3. Mescaline hydrobromide (50% probability ellipsoids) viewed along the direction of the *a* axis.

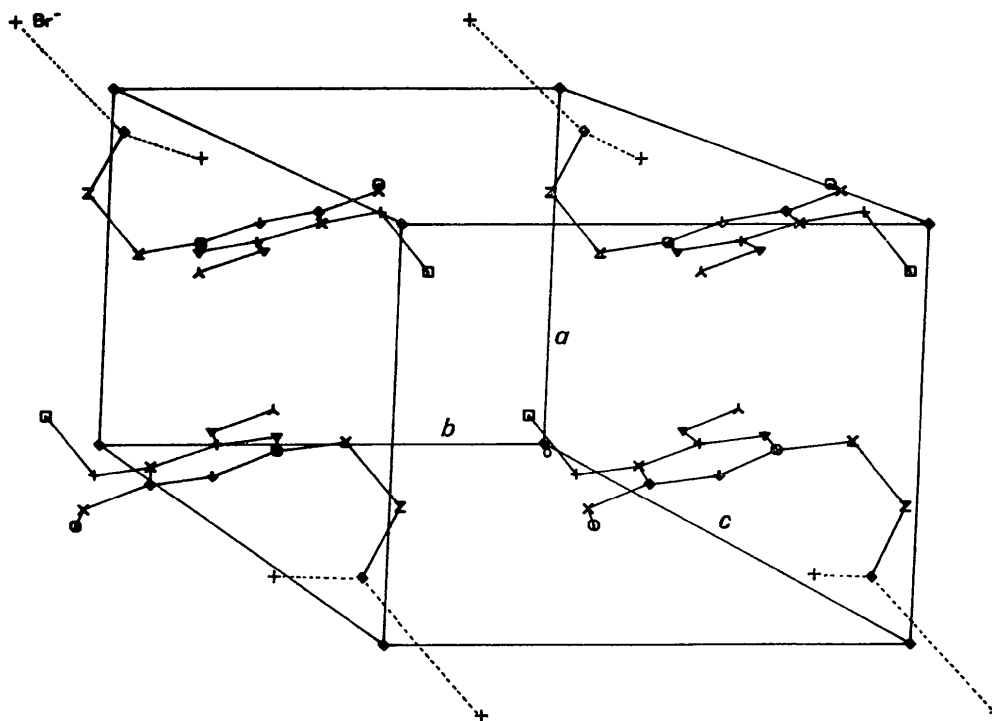


Fig. 4. Arrangement of molecules in the unit cell of mescaline hydrobromide.

to the 2.69 Å distance, which represents the closest approach allowed by rotation about the bonds.

This behavior contrasts with that of the phenethylamines and the phenylisopropylamines previously studied, in which the amine side chain tends to be maximally extended (Bergin, 1971, and references cited therein). Some evidence is thus provided for an attraction (possibly electrostatic) between the NH_3^+ group and the aromatic ring. The three methoxy groups adopt a configuration similar to that observed in reserpine (Karle & Karle, 1968); *i.e.*, the two outer methoxy groups are approximately coplanar with the benzene ring and the center group is nearly perpendicular to the plane of the ring (Table 3). The N-Br distances of 3.311 and 3.314 Å are normal for amine hydrobromides (Wunderlich, 1969).

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SHORT COMMUNICATIONS

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A new crystal structure study of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$. By B. RIBAR and V. DIVJAKOVIĆ, *Faculty of Science, Physics Department, Novi Sad, Yugoslavia*, and R. HERAK and B. PRELESNIK, *Boris Kidrič Institute, Belgrade, Yugoslavia*

(Received 16 October 1972; accepted 19 February 1973)

The unit cell has $a=6.277(7)$, $b=9.157(9)$, $c=14.484(10)$ Å, $\beta=98.6(2)^\circ$. The structure contains $\text{Ca}_2(\text{H}_2\text{O})_8(\text{NO}_3)_4$ dimers, joined by hydrogen bonds.

Introduction

The X-ray structure analysis of calcium nitrate tetrahydrate was recently carried out by Leclaire & Monier (1970). The structure was solved in the space group $P2_1/c$ on the basis of photographic data for 680 reflexions, giving a final R value of 0.051. As part of a series of studies of the crystal structures of hydrated nitrates of bivalent metals, we have undertaken an independent X-ray crystal structure analysis of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$. From the data for 1194 independent non-zero reflexions in the space group $P2_1/n$, the atomic parameters were derived and a final R index of 0.088 was achieved. Abrahams & Keve (1971) consider that duplicate measurements of the material being studied considerably increase the chances that the crystallographic results reported are indeed typical of that material. Hence, it may be useful to present our results as well, although the data from both determinations are in good agreement.

The colourless, transparent and hygroscopic crystals of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ were prepared by the slow evaporation of the CaO solution in HNO_3 in the presence of P_2O_5 . The lattice parameters were determined from Weissenberg $h0l$ and $hk0$ photographs, calibrated with a Debyeagram of

germanium. The standard deviations were estimated from several film measurements. The crystal data obtained are presented in Table 1.

Table 1. *Crystal data for $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$*

Crystal system:	Monoclinic	
a	6.277 (7) Å	6.268 (6) Å*
b	9.157 (9)	9.116 (9)
c	14.484 (10)	14.830 (10)
β	98.6 (2)°	106.5 (3)°
Z	4	4
D_{meas}	1.88 g cm ⁻³	1.88 g cm ⁻³
D_{calc}	1.90	1.90
Space group	$P2_1/n$	$P2_1/c$

* The data of Leclaire & Monier (1970).

Three-dimensional intensity data ($0kl \rightarrow 3kl$ and $h0l \rightarrow h5l$) were collected from two spherical crystals sealed in Lindemann capillaries; integrated multiple films and the equi-inclination Weissenberg technique were used. The