Discussion. This investigation was undertaken as a part of a project to clarify the structure of Al₂X₇ 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 Pd₂(C₆H₅)(Al₂Cl₇)₂ and Te₄(Al₂Cl₇)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 Te₄(Al₂Cl₇)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 Al₂Br₇ ion is staggered with almost C₃ 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. The authors wish to thank Den Norske Bryggeriindustri Fond for financial support, and are also indebted to lic. techn. J. Hjortás for recording the diffractometer data.

References


Mescaline Hydrobromide

BY S. R. ERNST AND F. W. CAGLE JR

Department of Chemistry, University of Utah, Salt Lake City, Utah 84112, U.S.A.

(Received 8 January 1973; accepted 19 February 1973)

Abstract. Mescaline hydrobromide (3,4,5-trimethoxyphenethylamine hydrobromide, C₁₁H₁₈NO₂Br), triclinic space group P₂₁, a=7.4274 (12), b=9.1782 (16), c=11.8979 (9) Å, α=121.180 (17), β=104.194 (18), γ=92.689 (13) ° (20°±1°C), Dm=1.48 g cm⁻³, Dx=1.48 g cm⁻³ (Z=2). The hydrobromide salt was prepared by the method of Heffter [Bet. dtsch. 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 P₁₁₁₁ diffractometer using graphite monochromated Mo Kα 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 (× 10⁴)

<table>
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<th>Br⁻</th>
<th>x</th>
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the $c$ axis) was cleaved from a large crystal. This was used to obtain two-dimensionally integrated Weissenberg photographs of layers $hko$ through $hk0$ using nickel-filtered copper $K\alpha$ radiation. The crystal was ca. 0.14 mm in cross-section and no absorption corrections were applied [$\mu(Cu)=47.4\text{ cm}^{-1}$]. 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 ($R=\sum |kF_{o}|-|F_{r}|/\sum |kF_{o}|$, where $k$ is the scale factor) of 0.081 (all reflections). The reported positions of the hydrogen atoms (Table 2) were obtained from a $\Delta F$ 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

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

All hydrogen atoms were assigned isotropic temperature factors of 3.0 Å$^2$.

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**Table 3. Least-squares planes showing magnitudes of deviations in Å**

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$A$ $\setminus$ 6.988 7.019 -3.241 6.930 1.318 | 2
$B$ $\setminus$ -2.186 2.051 3.847 2.579 -3.020 | 3
$D$ $\setminus$ -2.561 -2.455 -7.676 -2.635 -5.828 | 5

**Angles between planes**

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<tr>
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</table>

* Denotes atom included in least-squares fit.

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

Fig. 1. Bond lengths of mescaline hydrobromide.

Fig. 2. Bond angles of mescaline hydrobromide.
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).

The equipment used in this work was provided by the University of Utah Research Fund and by the National Science Foundation Science Development Grant GU 3866. One of us (S.R.E.) would like to thank the National Science Foundation for a Graduate Traineeship, 1966–1969.

References


SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.


A new crystal structure study of Ca(NO$_3$)$_2$.4H$_2$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 Ca$_2$(H$_2$O)$_8$(NO$_3$)$_3$ 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 Ca(NO$_3$)$_2$.4H$_2$O. From the data for 1194 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 Ca(NO$_3$)$_2$.4H$_2$O were prepared by the slow evaporation of the CaO solution in HNO$_3$ in the presence of P$_2$O$_5$. The lattice parameters were determined from Weissenberg $h0l$ and $hk0$ photographs, calibrated with a Debyeogram of germanium. The standard deviations were estimated from several film measurements. The crystal data obtained are presented in Table 1.

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<td>$b = 9.157 (9)$</td>
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<td>$c = 14.484 (10)$</td>
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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 equiinclination Weissenberg technique were used. The