*Inorganica Chimica Acta*, *156* (1989) 157–158

# The Crystal Structure of $MgCl_2 \cdot 6C_2H_5OH$

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(Received August 22, 1988)

The interaction between anhydrous $MgCl_2$ and many compounds having electron-donor behaviour (esters, alcohols and amines) has recently been the subject of much research. The reasons can be found both in the practical application of some adducts [1] and in theoretical and structural interest in the coordination produced around the Mg atom.

Crystallographic and spectroscopic techniques, and in particular X-ray diffraction (XRD) and IR measurements, found an important application in, for example, study of the $MgCl_2$–ethyl benzoate system [2, 3].

A crystallographic investigation represents the best approach for the structural definition of the products, even if they are highly unstable. In the course of such a crystallographic investigation, we studied some solvates of $MgCl_2$ with alcohols, such as $MgCl_2 \cdot nC_2H_5OH$, $MgCl_2 \cdot nCH_3OH$ and other alcohols. In particular, we considered the compound with six molecules of ethyl alcohol. This solvate has been previously studied by powder crystallographic techniques [4] and our study demonstrated the complete agreement of our XRD powder pattern with the one reported by the Russian authors.

The preparation of good single crystals of $MgCl_2 \cdot 6C_2H_5OH$ led us to carry out a complete crystallographic investigation, in spite of the instability of the crystal. In this paper we report the results of this XRD single-crystal analysis, which clearly reveal the crystal and molecular structure and the coordination around the Mg atom.

## Experimental

### *Preparation of $MgCl_2 \cdot 6C_2H_5OH$*

The preparation was accomplished by introducing 18.6 g of dried $MgCl_2$ into a flask containing 100 g

TABLE 1. Positional Coordinates and Thermal Anisotropic Parameters $U$ (Å$^2$) of $MgCl_2 \cdot 6C_2H_5OH$

| Atom | $x$ | $y$ | $z$ | $K$ (occupancy) | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Mg | 0.0 | 0.0 | 0.0 | 0.1667 | 0.0141(75) | 0.0200 | 0.0498(32) | 0.0 | 0.0 | −0.0117(119) |
| Cl | −0.3333 | 0.3333 | 0.0901(3) | 0.3333 | 0.0392(125) | 0.0287(160) | 0.0935(23) | 0.0 | 0.0 | 0.0107(227) |
| O1 | −0.0312(6) | 0.1984(6) | 0.1168(4) | 1.0000 | 0.0411(23) | 0.0408(21) | 0.0664(26) | −0.0100(19) | −0.0070(19) | 0.0255(19) |
| C1 | 0.0997(12) | 0.3442(11) | 0.2086(9) | 1.0000 | 0.0589(49) | 0.0419(36) | 0.0864(56) | −0.0126(37) | −0.0115(40) | 0.0237(34) |
| C2 | 0.0190(19) | 0.3285(19) | 0.3427(9) | 1.0000 | 0.1116(89) | 0.1279(91) | 0.0550(54) | −0.0202(59) | −0.0126(58) | 0.0559(76) |
| HO1 | −0.1069(74) | 0.2185(69) | 0.1078(46) | 1.0000 | 0.0334(141) | | | | | |
| H1C1 | 0.2071(122) | 0.3382(107) | 0.2213(72) | 1.0000 | 0.1046(272) | | | | | |
| H2C1 | 0.1209(118) | 0.4438(116) | 0.1785(76) | 1.0000 | 0.0863(306) | | | | | |
| H1C2 | 0.0014(158) | 0.2017(147) | 0.3778(90) | 1.0000 | 0.1665(426) | | | | | |
| H2C2 | −0.1011(142) | 0.2959(141) | 0.3527(86) | 1.0000 | 0.1385(393) | | | | | |
| H3C2 | 0.1204(118) | 0.4266(115) | 0.4044(74) | 1.0000 | 0.1006(257) | | | | | |

0020-1693/89/$3.50

of absolute alcohol [4]. After complete solution of $MgCl_2$, the liquid was heated in a flask with a reflux condenser for 1 h and after filtration the solution was cooled in dry ice. The crystals so obtained were dried in vacuum, yielding the following results. *Anal.* Found: Mg, 6.45; Cl, 19.15; $C_2H_5OH$, 74.4. Calc. for $MgCl_2 \cdot 6C_2H_5OH$: Mg, 6.54; Cl, 19.08; $C_2H_5OH$, 74.38%. Both XRD powder data and the melting point (72 °C) completely agree with the results of Turova *et al.* [4].

Single crystals were grown from a slightly supersaturated solution by recrystallization of the previously obtained powder. The crystals were small, colourless, hexagonal plates and their morphological stability was a problem that was surmounted by immersion in dried degased Vaseline/petrolatum (OBAT-55 Rol Oil).

### *Single Crystal for XRD Analysis*

A diffraction-quality crystal (0.7 × 0.7 × 0.7 mm) was obtained by cutting a larger crystal. Because of the sensitivity of the crystal to oxygen and moisture, the sample was sealed in a quartz capillary and filled with Vaseline/petrolatum. These operations were carried out in a glovebox and in an oxygen- and moisture-free nitrogen atmosphere.

### *Data Collection and Structural Analysis*

The crystals are trigonal, space group $P\bar{3}$ (N.147) with $a = 7.930(1)$ Å (1 Å = 0.1 nm); $c = 9.964(1)$ Å; $V = 542.6$ Å$^3$; $Z = 1$; $d_{calc} = 1.137$ g cm$^{-3}$. These values are in a good agreement with the data reported by Turova *et al.* [4], obtained by powder XRD: $a = 7.95 \pm 0.01$ Å; $c = 9.95 \pm 0.01$ Å; $d_{calc} = 1.13$ g cm$^{-3}$; $d_{obs} = 1.17$ g cm$^{-3}$.

Of the 2041 reflections collected for the sectors $hkl$, $\bar{h}kl$ and $hk\bar{l}$ on a Philips PW 1100 diffractometer, 855 were independent at $R_{int} = 0.1$. Mo K$\alpha$ radiation ($\lambda = 0.71069$ Å) was used and the collection system was the $\theta$–$2\theta$ scan up to $2\theta = 56°$.

The Patterson map indicated the heavier atoms were on the ternary axes. Nevertheless, the corresponding least-squares computation diverged for the trigonal system, whereas, by using the triclinic space group $P1$, the $R$ value ($R = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$, where $F_o$ and $F_c$ are the observed and calculated structure factors) was lowered to 5.7%. By using the triclinic coordinates and least-squares procedures, the trigonal space group $P\bar{3}$ converged to $R = 5.26\%$ for the 475 reflections showing $I \geqslant 3\sigma(I)$.

Table 1 gives the atomic coordinates of the structural unit and their relative errors ($\sigma$).

Figure 1 shows the 001 projection of the complex, including all the chloride ions hydrogen bonded to the coordinated oxygen atoms of the complex.

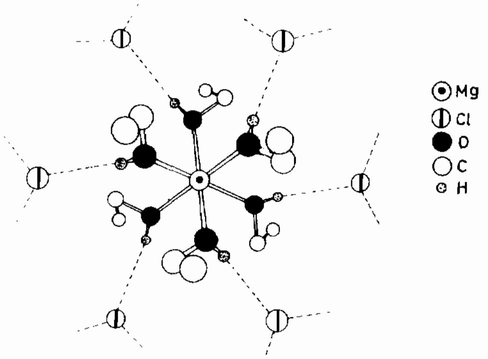

Fig. 1. The 001 projection of $MgCl_2 \cdot 6C_2H_5OH$.

The structure consists of Mg atoms coordinated to six ethanol oxygens at a distance of 2.069(3) Å. The ethanol hydrogen atom forms a hydrogen bond with chlorine at 3.085(2) Å. Each chloride ion contributes to the formation of three hydrogen bonds.

## Supplementary Material

This is available on request from the Cambridge Crystallographic Data Centre.

## References

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