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Structure of Potassium 1,1-Dicyanoethylene-2,2-dithiolate Monohydrate

BY HANS-ULRICH HUMMEL

Institut für Anorganische Chemie der Universität Erlangen–Nürnberg, Egerlandstrasse 1, *D*-8520 *Erlangen, Federal Republic of Germany*

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Abstract. $K_2[C_4N_2S_2].H_2O$, $M_r = 236{\cdot}4$, orthorhombic, *Pnma*, $a = 7{\cdot}215\ (4)$, $b = 13{\cdot}162\ (5)$, $c = 9{\cdot}449\ (4)$ Å, $V = 897{\cdot}3$ Å^3, $Z = 4$, $D_m = 1{\cdot}73\ (1)$, $D_x = 1{\cdot}75\ (1)$ Mg m^{-3}, $\lambda(\text{Ag } K\alpha) = 0{\cdot}5587$ Å, $\mu = 0{\cdot}667$ mm^{-1}, $F(000) = 464$, $T = 298$ K, final $R = 0{\cdot}036$ for 587 observed independent reflections. K has a monocapped trigonal-antiprismatic coordination: three S, three N and one O atom. The S and N atoms are involved in the coordination of three different K atoms. One O is coordinated with two K atoms. The 1,1-dicyanoethylene-2,2-dithiolate ligand (dedt) has approximate C_{2v} symmetry with central C—C 1·405 Å. The water molecule is involved in O—H···S hydrogen bonds with O···S 3·395 and 3·508 Å.

Introduction. The geometry of a molecule as observed in the solid state is a compromise between inter- and intramolecular forces. In the compounds of the series $M_2(S_2C_4N_2).nH_2O$ (M = Li, Na, K, Rb, Cs) the metal–ligand interactions are expected to be small. Structure determinations of these compounds offer the possibility of comparing bond distortions in the dedt ligand with respect to different packing arrangements. A K salt of dedt was first synthesized by Söderbäck (1963). The polydentate ligand was thought to be a planar delocalized system. This has been confirmed by the structure analysis of the anion $[Cu_8(\text{dedt})_6]^{4-}$ by McCandlish, Bissell, Coucouvanis, Fackler & Knox (1968).

Experimental. Stoichiometric amounts of $KCH(CN)_2$ and CS_2 in ethanol; evaporation and cooling at about 285 K. Crystal 0·08 × 0·08 × 0·34 mm. D_m determined pycnometrically. Precession photographs. Orthorhombic, Laue class *mmm*, systematic extinctions $0kl$, $k + l = 2n + 1$, $hk0$, $h = 2n + 1$, space group *Pnma*. PW 1100 diffractometer, ω scan, scan width $(1{\cdot}00 + 0{\cdot}20\tan\theta)^\circ$. Cell dimensions based on 25 high indexed diffractometer-measured intensities. 2068 symmetry-dependent reflections ($-8 \le h \le 8$, $-16 \le k \le 16$, $-11 \le l \le 11$; $3^\circ \le \theta \le 20^\circ$) averaged to give 861 symmetry-independent reflections, $R_{\text{int}} = 0{\cdot}028$. Three standard reflections, no significant deviations from their initial intensities. Corrections for Lorentz, polarization and absorption effects; transmission factors between 0·952 and 0·940 (Sheldrick, 1976). Structure solved by direct methods (*MULTAN*, Germain, Main & Woolfson, 1971). 587 observed reflections $[I > 2\sigma(I)]$ in full-matrix least-squares refinement ($\sin\theta_{\max}/\lambda$ 0·537 Å^{-1}); 56 parameters; H atoms as parts of rigid H_2O molecules with H···H 1·560 (5) and O···H 0·960 (5) Å, and common isotropic temperature factor; $\sum w||F_o| - |F_c||^2$ minimized; $w = 0{\cdot}1[\sigma^2(F_o) + 7 \times 10^{-4}F_o^2]^{-1}$; *SHELX*76 system (Sheldrick, 1976); $(\Delta/\sigma)_{\max} = 0{\cdot}011$, $(\Delta/\sigma)_{\min} = 0{\cdot}000$, $(\Delta/\sigma)_{\text{ave}} = 0{\cdot}0002$; $\Delta\rho = -0{\cdot}28$ to $0{\cdot}24$ e Å^{-3}; correction for isotropic extinction, $F^* = F(1 + 1{\cdot}2 \times 10^{-8}F^2/\sin\theta)$. Final refinement converged to $R = 0{\cdot}036$, $wR = 0{\cdot}037$. Scattering factors, f' and f'' from *International Tables for X-ray Crystallography* (1974).

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Table 1. *Final atomic coordinates and equivalent isotropic temperature factors of the non-hydrogen atoms*

$$U_{\rm eq}=\tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j.$$

	Site symmetry	*x*	*y*	*z*	$U_{\rm eq}$(Å^2)
K	1	0·2810 (1)	0·0591 (1)	0·1068 (1)	0·0521 (9)
S	1	0·3599 (1)	0·1366 (1)	0·4389 (1)	0·0437 (10)
C(1)	*m*	0·3790 (6)	0·2500	0·5266 (5)	0·0307 (21)
C(2)	*m*	0·4083 (6)	0·2500	0·6737 (5)	0·0302 (21)
C(3)	1	0·4194 (4)	0·3422 (2)	0·7511 (3)	0·0380 (27)
N	1	0·4283 (5)	0·4154 (2)	0·8158 (3)	0·0383 (27)
O	*m*	0·4267 (4)	0·2500	0·1202 (3)	0·0558 (20)

Table 2. *Selected distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

(*a*) The coordination polyhedron of the K$^+$ ion			
K—O	2·727 (2)		
K—N	2·917 (3), 2·967 (3), 3·124 (3)		
K—S	3·191 (2), 3·234 (1), 3·348 (1)		
(*b*) The dedt ion			
S—S	2·985 (2) (bite)	S—C(1)—S	121·3 (3)
S—C(1)	1·713 (3) (2×)	S—C(1)—C(2)	119·4 (1)
C(1)—C(2)	1·405 (7)	C(1)—C(2)—C(3)	121·2 (2)
C(2)—C(3)	1·420 (4) (2×)	C(3)—C(2)—C(3)	117·5 (4)
C(3)—N	1·143 (4) (2×)	C(2)—C(3)—N	178·7 (3)
(*c*) The hydrogen bonds			
S—O	3·395 (3), 3·508 (3)		
S—H(1)	2·675 (2×)		
S—H(2)	2·574 (2×)		

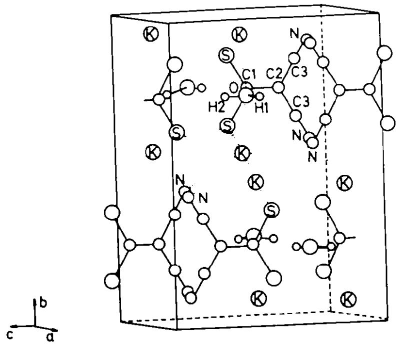

Fig. 1. The structure of $K_2[S_2C_4N_2].H_2O$.

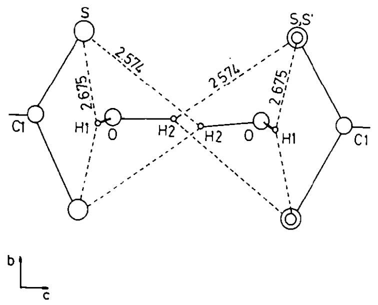

Fig. 2. Coordination of the H_2O molecule. (Distances are in Å.)

Discussion. Final atomic parameters are given in Table 1,* and selected distances and angles in Table 2. The structure is depicted in Fig. 1. K is coordinated by three S, three N and one O atom forming a monocapped trigonal antiprism with average K—S distance 3·26 Å. Each S and N coordinate two more K$^+$ ions while each O atom is directed to one more K. The resulting framework can be described by the formalism $^3_\infty[KS_{3/3}N_{3/3}O_{1/2}]$. Two S···O distances are in the range given by Mereiter, Preisinger & Guth (1979) with H atoms forming two sets of two equidistant OH···S hydrogen bonds (Fig. 2). The observed conformation of the dedt ion corresponds to approximate C_{2v} symmetry with the central C(1) and C(2) atoms situated on a mirror plane. The maximum deviation from the least-squares plane for the whole molecule is 0·027 Å [C(2)]. The S—C(1) bond length indicates some double-bond character (Hlawatschek, Dräger & Gattow, 1982). Bond distances for C(1)—C(2) and C(2)—C(3), respectively, clearly show a delocalized π system. The bond length for C(2)—C(3) is close to 1·42 Å which is the value for a C_{sp^2}—C_{sp} bond with a bond order of $\frac{4}{3}$ (Bent, 1961). Compared to the C—N triple-bond length in the highly symmetrical $C(CN)_3^-$ ion [1·17 (1) Å], the C—N triple bond for dedt is somewhat shorter (Witt & Britton, 1971). This could be caused by the lowering of the contribution of the cyanide groups to the π system in dedt.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, intermolecular distances and mean-plane data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42358 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.