

Short Communications

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Acta Cryst. (1971). B27, 242

Disemicarbazide hydrobromide. By P. BOLDRINI, *Département des Sciences, Université du Québec à Trois-Rivières, Trois-Rivières, P. Q., Canada.*

(Received 16 September 1970)

Crystallographic data for the newly prepared disemicarbazide hydrobromide are compared with those of disemicarbazide hydrochloride.

During a study (Boldrini, 1970) on compounds with semicarbazide, $H_2NCONHNH_2$ (from now on SEM), a new salt of this base with HBr was prepared. Since two moles of SEM were found combined with one of HBr, the salt is a disemicarbazide hydrohalide. SEM_2HBr separated as white, irregular, asymmetric crystals by slowly evaporating equimolar solutions of the components in absolute ethanol.

An X-ray investigation showed that the compound is monoclinic, with systematic extinctions $h+k$ odd, for the hkl reflexions, in addition to h and l odd for the $h0l$ reflexions. The choice of a space group favours $Cc-9$ (*International Tables for X-ray Crystallography*, 1955) on morphological grounds and because the molecule should not have a twofold axis to occupy position 4(e) of $C2/c-15$. Crystallographic constants for SEM_2HBr were measured by precession methods with $Mo K\alpha$ radiation and $PbCl_2$ as calibrating substance; they are reported in Table 1, with standard deviations in parenthesis. The specific gravity was deduced by flotation in $CHBr_3-CCl_4$ -benzene mixtures.

Table 1. *Crystallographic data for disemicarbazide hydrobromide (SEM_2HBr) and disemicarbazide hydrochloride (SEM_2HCl)**

Standard deviations are parenthesized.

	SEM_2HBr	SEM_2HCl
Molecular formula	$C_2H_{11}N_6O_2Br$	$C_2H_{11}N_6O_2Cl$
Molecular weight	231.057	186.602
a	12.942 (12) Å	12.855 (2) Å
b	9.792 (4)	9.713 (2)
c	6.602 (4)	6.506 (2)
β	92.78 (20)°	93.10 (9)°
Volume	835.43 Å ³	811.39 Å ³
D_m (30°C)	1.81 g.cm ⁻³	1.52 g.cm ⁻³
D_c	1.83	1.53
Space groups	{ $C2/c-15$ $Cc-9$	{ $C2/c-15$ $Cc-9$
Z	4	4
Z_{calc}	3.94	3.97

* $Mo K\alpha$ was used ($\lambda=0.71069$ Å) and $PbCl_2$ as calibrating standard with cell constants: $a=7.61892$, $b=9.04443$, $c=4.53345$ Å.

For the sake of comparison, crystallographic data were obtained for SEM_2HCl too, in the same way as for SEM_2HBr , but the disemicarbazide hydrochloride was reprepared (Haller & La Forge, 1937) from ethanol at

90%. SEM_2HCl gave crystals of two distinct habits, both very rich in external faces and looking like those reported for two different substances by Phillips (1963) in his Figs. 188 and 189. While, on one hand, the tabular crystals of SEM_2HCl were already known (Haller & La Forge, 1937), the others appeared equal to the ones mentioned above for SEM_2HBr . Both types of SEM_2HCl crystals were therefore investigated and no difference was found between them, except for a marked diffuse scattering presented by 20 of the strongest reflections of the tabular crystals.

By comparing the cell constants in Table 1, by superimposing and studying in details the diffraction patterns of SEM_2HBr and SEM_2HCl , it can be concluded that the two salts are isostructural, and by analogy with similar compounds of diglycine (Hahn & Buerger, 1957) or thiosemicarbazide (Andreotti, Domiano, Gasparri, Nardelli & Sgarabotto, 1970), it can be inferred that the three-dimensional crystal structure of these disemicarbazide hydrohalides should be held together by a complex network of hydrogen bonds. As far as the position of the proton of the halogenidric acids is concerned, a full structural investigation is needed.

SEM_2HBr and SEM_2HCl are very soluble in water, slightly soluble in absolute ethanol and ethylene glycol, and are also sparingly soluble in methanol and aqueous ammonia. Finally, while the melting point of SEM_2HCl is confirmed to be at 132°C (Haller & La Forge, 1937), the one of SEM_2HBr was found at 110°C, but the substance decomposes appreciably.

Thanks are due to the National Research Council of Canada for financial assistance and to D. Grondin and M. Labbé for the cooperation.

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