Crystal structure of hexamethanolo-iron diiodide, $Fe(HOCH_3)_6I_2$

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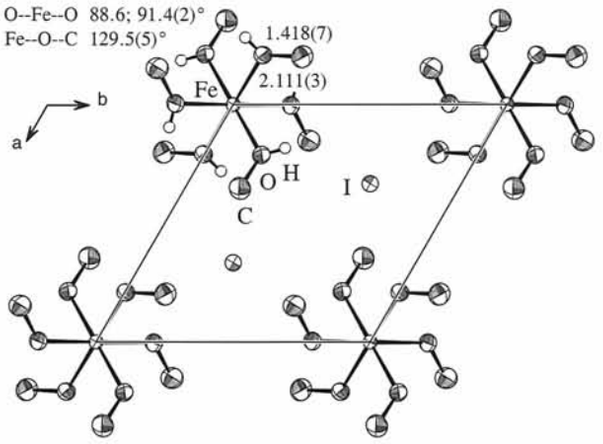

Abstract

$C_6H_{24}FeI_2O_6$, trigonal, $P\bar{3}$ (No. 147), $a = 8.2947(8)$ Å, $c = 7.0884(6)$ Å, $V = 422.4$ Å^3, $Z = 1$, $R_{gt}(F) = 0.023$, $wR_{ref}(F^2) = 0.065$, $T = 193$ K.

Source of material

Iron diiodide (0.31 g), prepared from iron and iodine [1], was dissolved in 2.0 mL of anhydrous methanol. After addition of 10 mL of anhydrous diethylether the title compound crystallized in the course of two hours. After decantation the crystals were dried in a stream of dry argon.

Experimental details

$Fe(HOCH_3)_6I_2$ is isotypic to $Ca(HOCH_3)_6Br_2$ [2]. As noted for the latter, merohedral twinning occurs. However, contrary to [2], we do not find (001) as a twinning plane, but twins of twins with the twinning planes (110) and (120). The domains of the (120)-twinning are very small, as evidenced by the refinement results: assuming macroscopic twinning along (120) (superposition of F^2 values) resulted in $R_{gt}(F) = 0.035$; superposition of F values yielded $R_{gt}(F) = 0.023$. However, for another crystal the F^2 superposition was slightly better. The four components of the reported crystal had volume fractions of 0.61(1), 0.13 (110)-twin, 0.22 (120) and 0.04 (210). If all four components had the same volume fractions, their superposition would feign the space group $P6/mmm$, which is a supergroup of $P\bar{3}$.

Discussion

The methyl groups and iodide ions are arranged like in a hexagonal closest-packing. One eighth of the octahedral voids are occupied by Fe^{2+} ions together with the O atoms linking them to the methyl groups. Another description relates the structure to the CdI_2 type, in which $Fe(OCH_3)_6^{2+}$ ions take the positions of the cadmium ions.

Table 1. Data collection and handling.

Crystal:	colourless hexagonal prism, size 0.13 × 0.24 × 0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	45.61 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS 2; $\Delta\varphi = 1°$, $\omega = 0° - 180°$ at $\varphi = 30°$, $\omega = 0° - 98°$ at $\varphi = 120°$
$2\theta_{max}$:	57.26°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5978, 730
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 726
$N(param)_{refined}$:	37
Program:	SHELX-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	6*g*	0.19(1)	0.28(1)	0.20(1)	0.04(2)
H(2)	6*g*	0.4335	0.3229	0.3337	0.16
H(3)	6*g*	0.4297	0.1717	0.1905	0.16
H(4)	6*g*	0.2967	0.1055	0.3717	0.16

References

1. Brauer, G.: Handbuch der präparativen anorganischen Chemie, 3. Aufl., p. 1644. F. Enke, Stuttgart 1981.
2. Halut-Desportes, S.; Philoche-Levisaller, M.: Structures comparés des solvates de formule $MBr_2 \cdot 6CH_3OH$. Acta Crystallogr. **B34** (1978) 432-435.
3. Sheldrick, G. M.: SHELX-97. A program package for crystal structure solution and refinement, University of Göttingen, Germany 1997.

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe	1*a*	0	0	0	0.0196(3)	U_{11}	0.0294(6)	$U_{11}/2$	0	0
I	2*d*	1/3	2/3	0.23363(7)	0.0259(2)	U_{11}	0.0450(2)	$U_{11}/2$	0	0
O	6*g*	0.2127(5)	0.2083(5)	0.1675(6)	0.033(2)	0.024(2)	0.050(2)	0.016(1)	–0.017(2)	–0.010(2)
C	6*g*	0.3541(8)	0.202(1)	0.2741(8)	0.043(4)	0.045(3)	0.056(4)	0.022(3)	–0.029(3)	–0.009(3)

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