$+ 0.73, -0.57$ e Å^{-3}. The atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a MicroVAX II computer. Atomic parameters are listed in Table 1,* and selected bond distances and angles in Table 2. A view of the molecule, with the atom-labeling scheme, is shown in Fig. 1.

Related literature. The structure of the analogous chromate bridged complex [(C$_9$H$_{21}$N$_3$)Fe(CrO$_4$)$_3$-Fe(C$_9$H$_{21}$N$_3$)] has been reported (Chaudhuri *et al.*, 1988), as well as μ-phosphato [(C$_9$H$_{21}$N$_3$)Fe-(O)(C$_6$H$_5$OPO$_3$)$_2$Fe(C$_9$H$_{21}$N$_3$)] (Drueke, Wieghardt, Nuber, Weiss, Fleischhauer, Gehring & Haase, 1989) and μ-carbonato species (Drueke, Wieghardt, Nuber & Weiss, 1989). A complex containing three-[(C$_9$H$_{21}$N$_3$)Fe] moieties bridging a [Mo$_4$O$_{14}$(μ-

OMe)$_3$] core has also been described (Chaudhuri, Wieghardt, Nuber & Weiss, 1987).

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53998 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Diaqua-1,6-bis(benzimidazol-2-yl)-2,5-dithiahexanecobalt(II) Iodide Monohydrate at 233 K

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Abstract. [Co(C$_{18}$H$_{18}$N$_4$S$_2$)(H$_2$O)$_2$]I$_2$.H$_2$O, $M_r = 721.29$, monoclinic, $C2/c$, $a = 18.804$ (4), $b = 11.101$ (5), $c = 12.326$ (5) Å, $\beta = 109.86$ (3)°, $V = 2420$ Å^3, $Z = 4$, $D_x = 1.980$ g cm^{-3}, λ(Mo $K\alpha$) = 0.70930 Å, $\mu = 34.246$ cm^{-1}, $F(000) = 1396$, $T = 233$ K, $R = 0.043$ for 1913 observed reflections. The Co atom of the complex is surrounded by two N atoms from the benzimidazole groups in a *trans* arrangement [Co—N = 2.122 (5) Å] and two sulfur atoms of the thioether groups with a *cis* geometry [Co—S = 2.521 (2) Å]. The O atoms of the two symmetrically related water molecules [Co—O = 2.087 (6) Å] complete the coordination around the metal ion to form a distorted octahedron. The packing in the crystal results in hydrogen-bond interactions between the water molecules, and between the N—H groups of the imidazoles and the I atoms.

Experimental. Colorless prisms of the title compound were obtained by slowly evaporating solvent from a solution of [Co(BBDH)(OH$_2$)$_2$]I$_2$ [BBDH = 1,6-bis-(benzimidazol-2-yl)-2,5-dithiahexane] in ethanol. Crystal size 0.08 × 0.08 × 0.16 mm, Enraf–Nonius CAD-4 four-circle single-crystal automated diffrac-

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	$U_{eq}(\text{Å}^2)$
I	0·13794 (3)	−0·15884 (5)	0·48008 (4)	0·0341 (2)
Co	0·500	0·1809 (1)	0·750	0·0216 (5)
S	0·4431 (1)	0·0166 (2)	0·8356 (2)	0·0344 (9)
O(1)	0·5463 (3)	0·3187 (5)	0·6798 (4)	0·038 (2)
N(1)	0·3962 (3)	0·1560 (6)	0·6127 (5)	0·025 (2)
N(2)	0·2869 (3)	0·0663 (6)	0·5202 (6)	0·029 (3)
C(1)	0·3649 (4)	0·2082 (7)	0·5029 (5)	0·025 (3)
C(2)	0·3899 (4)	0·3016 (7)	0·4498 (6)	0·027 (3)
C(3)	0·3456 (5)	0·3310 (8)	0·3385 (6)	0·037 (4)
C(4)	0·2778 (5)	0·2747 (9)	0·2815 (7)	0·038 (4)
C(5)	0·2506 (4)	0·1822 (8)	0·3341 (7)	0·038 (4)
C(6)	0·2956 (4)	0·1521 (7)	0·4449 (6)	0·030 (3)
C(7)	0·3479 (4)	0·0734 (7)	0·6184 (7)	0·030 (3)
C(8)	0·3534 (4)	−0·0016 (8)	0·7206 (8)	0·042 (4)
C(9)	0·5048 (5)	−0·1093 (8)	0·6908 (9)	0·053 (5)
Solvent				
O(2)	0·500	0·4723 (7)	0·250	0·041 (4)

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

Co—S	2·521 (2)	C(1)—C(2)	1·40 (1)
Co—O(1)	2·087 (6)	C(1)—C(6)	1·402 (9)
Co—N(1)	2·122 (5)	C(2)—C(3)	1·380 (9)
S—C(8)	1·809 (7)	C(3)—C(4)	1·38 (1)
S—C(9)	1·80 (2)	C(4)—C(5)	1·41 (1)
N(1)—C(1)	1·404 (8)	C(5)—C(6)	1·39 (2)
N(1)—C(7)	1·31 (1)	C(7)—C(8)	1·48 (1)
N(2)—C(6)	1·38 (2)	C(9)—C(9^i)	1·53 (2)
N(2)—C(7)	1·358 (8)		
I—H(11)	2·69 (6)	O(1)—H(12^{ii})	1·149 (6)
I—H(20)	2·73 (6)	O(2^{iv})—H(12)	1·64 (6)
O(1)—H(12^{ii})	0·887 (6)	N(2)—H(20)	0·93 (6)
I—O(1^{iii})	3·461	O(1)—O(2^v)	2·720 (4)
I—N(2)	3·659 (3)		
S—Co—S^i	87·29 (8)	N(1)—C(1)—C(6)	108·8 (7)
S—Co—O(1)	179·2 (2)	C(2)—C(1)—C(6)	119·7 (6)
S—Co—O(1^i)	93·5 (2)	C(1)—C(2)—C(3)	117·3 (7)
S—Co—N(1)	88·7 (2)	C(2)—C(3)—C(4)	122·7 (8)
S—Co—N(1^i)	80·4 (2)	C(3)—C(4)—C(5)	121·1 (7)
O(1)—Co—O(1^i)	85·7 (2)	C(4)—C(5)—C(6)	116·0 (7)
O(1)—Co—N(1)	99·5 (2)	N(2)—C(6)—C(1)	105·4 (6)
O(1)—Co—N(1^i)	91·6 (2)	N(2)—C(6)—C(5)	131·3 (7)
N(1)—Co—N(1^i)	165·0 (3)	C(1)—C(6)—C(5)	123·2 (8)
C(8)—S—C(9)	101·0 (4)	N(1)—C(7)—N(2)	112·6 (7)
C(1)—N(1)—C(7)	105·7 (5)	N(1)—C(7)—C(8)	126·1 (6)
C(6)—N(2)—C(7)	107·7 (7)	N(2)—C(7)—C(8)	121·2 (7)
N(1)—C(1)—C(2)	131·5 (6)	S—C(8)—C(7)	112·4 (6)
		S—C(9)—C(9^i)	114·9 (6)
I—H(11)—O(1^{iii})	146 (5)	O(1^{iii})—H(12)—O(2^v)	155 (5)
I—H(20)—N(2)	177 (6)		

Symmetry code: (i) $1 - x$, y, $1·5 - z$; (ii) $0·5 + x$, $0·5 + y$, z; (iii) $x - 0·5$, $y - 0·5$, z; (iv) $x - 0·5$, $0·5 - y$, $1 - z$; (v) $1 - x$, $1 - y$, $1 - z$.

tometer, Mo $K\alpha$ radiation, graphite mono-chromator, ω/θ scan. Accurate cell dimensions from least-squares procedure on 25 reflections ($8·5 < \theta < 15·5°$). Empirical absorption correction applied (*DIFABS*; Walker & Stuart, 1983), max./min. correction factors 1·233 and 0·720. Data collected to

$(\sin\theta)/\lambda$ of 0·68 Å^{-1}. Total of 3465 reflections ($3 < \theta < 29°$) measured in the range $0 < h < 25$, $0 < k < 15$ and $-16 < l < 16$ of which 3273 were unique, $R_{int} = 0·035$ and 1913 with $I > 3\sigma(I)$. The intensities of two reflections ($\bar{3}\bar{3}4$, 060) measured at 3600 s exposure-time intervals did not show significant variation.

The structure was solved from interpretation of Patterson map, full-matrix least-squares refinement based on F applied to all non-H atoms. A difference Fourier map revealed all the H-atom positions [except those of the water molecules and H(4)] which were included in the final refinement with com-mon fixed thermal parameters ($B = 4·0$ Å^2). The remaining H atoms, located geometrically, were included in the structure factor calculations. $R = 0·043$, $wR = 0·046$, where $w = 1/\sigma^2(F)$, $S = 4·99$ for 162 variables, secondary-extinction factor $7·44$ (1) $\times 10^{-8}$ (Zachariasen, 1963). $\Delta/\sigma < 0·01$, largest peak in final ΔF map 0·839 e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections from *Inter-national Tables for X-ray Crystallography* (1974, Vol. IV). Programs used were *SHELXS86* (Sheldrick, 1986), Enraf–Nonius *SDP/VAX* (Frenz, 1986) and *SCHAKAL* (Keller 1988). All calculations were performed on a DEC MicroVAX 3500.

Final atomic coordinates for non-H atoms are given in Table 1, bond lengths and angles are listed
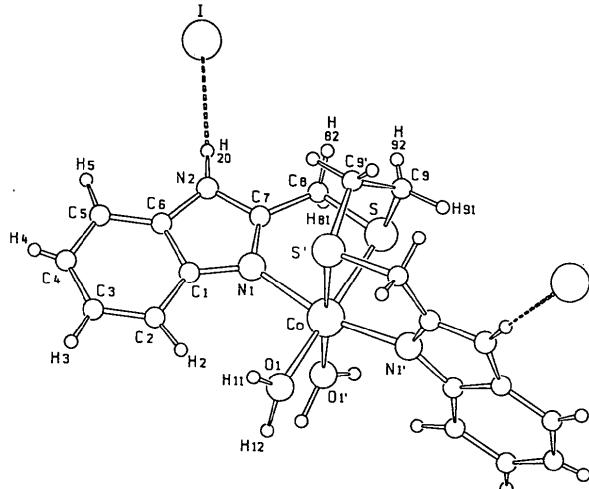
Fig. 1. The molecule of [Co(BBDH)(OH$_2$)$_2$]I$_2$ with the numbering scheme.
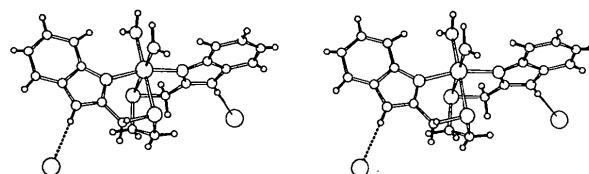
Fig. 2. Stereoview of [Co(BBDH)(OH$_2$)$_2$]I$_2$.

in Table 2.* The molecular structure and the atom-numbering scheme are shown in Fig. 1, Fig. 2 shows a stereoscopic view.

Related literature. The structural parameters of this molecule can be compared with those found in the isostructural complex [Ni(BBDH)(OH₂)₂]-(ClO₄)₂.H₂O (Castiñeiras, Carballo, Hiller & Strähle, 1990), as well as those of the same ligand [Cu-(BBDH)Cl]Cl.2C₂H₅OH (Birker, Helder, Henkel, Krebs & Reedijk, 1982), [Ni(BBDH)Br₂] (Smits, Janssen, Beurskens, Van Rijn & Reedijk, 1987), [Co(BBDH)(OH₂)(ONO₂)](NO₃).H₂O (Castiñeiras, Carballo, Bermejo, Gayoso, Molleda, Hiller & Strähle, 1988), [Co(BBDH)(OH₂)₂]Br₂.2H₂O (Hiller, Strähle, Castiñeiras, Carballo, Bermejo & Gayoso, 1990), and [Ni(BBDH)(OH₂)₂]Br₂.2H₂O (Castiñeiras, Hiller, Strähle, Carballo, Bermejo & Gayoso, 1990).

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, torsion angles, mean-planes data and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53978 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of
trans-Diiodobis(triphenylphosphine)palladium(II)–Trichloromethane (1/1)

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Abstract. [Pd(I)₂{P(C₆H₅)₃}₂].CHCl₃, $M_r = 1004.2$, monoclinic, $C2/c$, $a = 12.630$ (1), $b = 15.237$ (2), $c = 19.893$ (3) Å, $\beta = 91.67$ (1)°, $V = 3827$ (1) Å³, $Z = 4$, $D_m = 1.72$ (2), $D_x = 1.74$ Mg m⁻³, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 2.39$ mm⁻¹, $F(000) = 1944$, $T = 300$ K, $R = 0.040$ for 2470 unique reflections. The Pdᴵᴵ atom lies on a crystallographic twofold axis and the monomeric complex is *trans* square planar with no additional ligands in the octahedral positions.

Experimental. The palladium complex obtained in the course of catalytic reaction was provided by Professor Jin Douman, Henan Academy of Sciences, People's Republic of China. Red tabular crystals were grown from trichloromethane solution. D_m measured by flotation in aqueous KI solution. X-ray intensities were measured on a Rigaku AFC-5 four-

circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation, θ–2θ scan, scan speed 6° min⁻¹ in θ, crystal size $0.10 \times 0.13 \times 0.30$ mm, $-16 \le h \le 16$, $0 \le k \le 19$, $-25 \le l \le 0$ ($4 \le 2\theta \le 55°$), 4506 reflections measured, 2548 reflections observed with $|F_o| > 3\sigma(|F_o|)$, 2470 unique ($R_{int} = 0.017$); lattice constants based on 24 2θ values ($20 < 2\theta < 30°$). Ratio of five standard reflections $0.99 < \Sigma(|F_o|/|F_o|_{initial})/5 < 1.01$. Absorption correction (analytical, relative transmission factors 0.72–0.79). Systematic absences (*hkl*, $h + k$ odd; *h0l*, l odd) indicated the space group to be Cc or $C2/c$. Assuming the centrosymmetric space group $C2/c$, the structure could be solved successfully by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); coordinates of all the non-H atoms refined by block-diagonal least squares with anisotropic thermal parameters using the *UNICS*III system (Sakurai & Kobayashi, 1979); all the H atoms

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