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# Bis(1,4,7-trithiacyclononane-*S*,*S*′,*S*″)nickel(II) dibromide tetrahydrate

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**Key indicators**

Single-crystal X-ray study
$T$ = 150 K
Mean $\sigma$(C–C) = 0.007 Å
$R$ factor = 0.038
$wR$ factor = 0.091
Data-to-parameter ratio = 17.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The Ni$^{II}$ centre in the cation of the title compound, $[Ni(C_6H_{12}S_3)_2]Br_2\cdot 4H_2O$, occupies a crystallographic inversion centre and is octahedrally coordinated by six S-donors from two [9]ane$S_3$ ligands. Ni—S distances range from 2.3749 (16) to 2.4077 (15) Å and S—Ni—S angles where both thia donors belong to the same ligand lie in a narrow range between 88.09 (5) and 88.67 (6)°. The water molecules participate in extensive hydrogen bonding with each other and with the Br$^-$ anions to form double chains with eight- and 12-membered hydrogen-bonded rings running along the crystallographic *a* direction.

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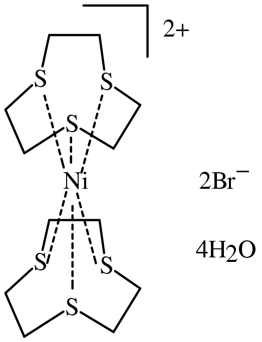
## Comment

In the title compound, (I), the Ni$^{II}$ centre occupies a crystallographic inversion centre and is octahedrally coordinated by six S-donors from two [9]ane$S_3$ ligands (Fig. 1). The Ni—S distances range from 2.3749 (16) to 2.4077 (15) Å, while *cis*-S—Ni—S angles lie in a narrow range between 88.09 (5) and 88.67 (6)°. Fig. 2 shows a view along the *b* axis of part of one double chain of paired water molecules and Br$^-$ ions running along the *a* direction. Each double chain has alternating smaller and larger hydrogen bonded rings denoted by the graph-set symbols $R_4^2(8)$ and $R_6^4(12)$.



(I)

## Experimental

The title compound was prepared by reaction of $NiBr_2\cdot 3H_2O$ with 1,4,7-trithiacyclononane in MeOH. The resulting precipitate was washed with $Et_2O$, dissolved in MeOH and $Et_2O$ vapour was then diffused into the solution to afford a purple microcrystalline solid. A crystal grew spontaneously by evaporation of the solvent while the solution was being examined on the microscope slide. This crystal was mounted in a film of RS3000 perfluoropolyether oil (Hoechst) and

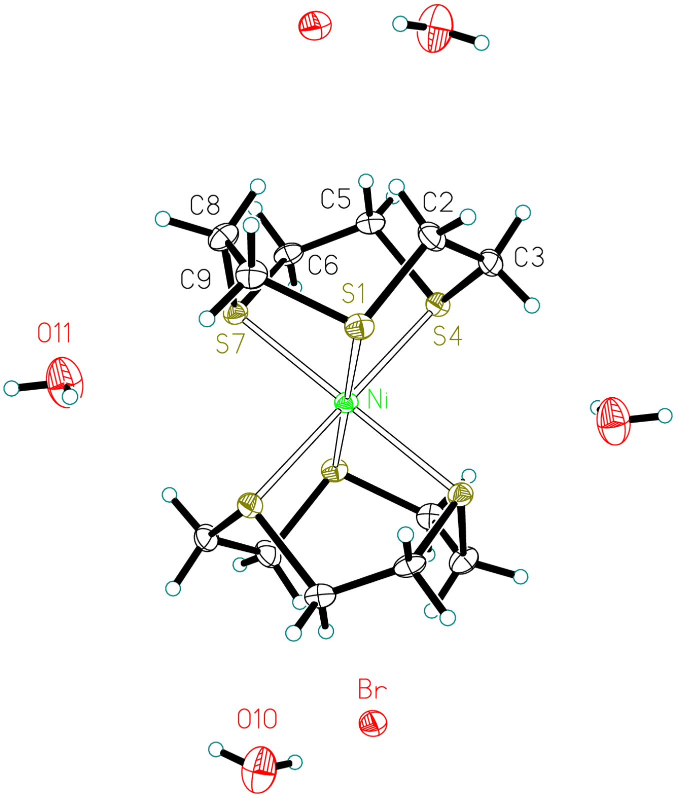


**Figure 1**
A view of two asymmetric units of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

transferred into the cold stream of an Oxford Cryosystems open-flow cryostat (Cosier & Glazer, 1986) operating at 150 (2) K.

*Crystal data*

$[Ni(C_6H_{12}S_3)_2]Br_2·4H_2O$
$M_r = 651.27$
Triclinic, $P\overline{1}$
$a = 7.654$ (3) Å
$b = 8.972$ (4) Å
$c = 9.132$ (5) Å
$\alpha = 93.34$ (4)°
$\beta = 102.63$ (4)°
$\gamma = 107.79$ (4)°
$V = 577.4$ (5) Å$^3$
$Z = 1$
$D_x = 1.873$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 16 reflections
$\theta = 12$–14°
$\mu = 4.86$ mm$^{-1}$
$T = 150$ (2) K
Tablet, purple
0.23 × 0.12 × 0.08 mm

*Data collection*

Stoe Stadi-4 four-circle diffractometer
$\omega/\theta$ scans
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1997)
$T_{min} = 0.551$, $T_{max} = 0.725$
2771 measured reflections
2252 independent reflections
1985 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 26.2$°
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -10 \rightarrow 11$
3 standard reflections
frequency: 60 min
intensity decay: random variation: ±2.6%

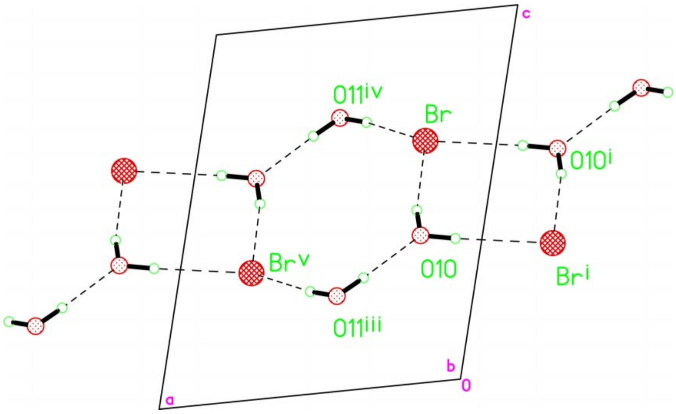


**Figure 2**
A view along the *b* axis of part of one double chain of water molecules and Br$^-$ ions running along the *a* direction. The double chain has alternating smaller and larger hydrogen-bonded rings denoted by the graph-set symbols $R_4^2(8)$ and $R_6^4(12)$. [Symmetry codes: (i) $-x$, $1 - y$, $1 - z$; (iii) $-x$, $1 - y$, $-z$; (iv) $1 + x$, $y$, $1 + z$; (v) $1 - x$, $1 - y$, $1 - z$.]

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.092$
$S = 1.09$
2252 reflections
131 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 3.122P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.68$ e Å$^{-3}$
$\Delta\rho_{min} = -0.88$ e Å$^{-3}$

The structure was solved by direct methods (Sheldrick, 1997) and developed through iterative cycles of least-squares refinement and difference Fourier syntheses. Water H atoms were found from a $\Delta F$ synthesis, while others were placed geometrically; these were then refined as a rigid $H_2O$ group [O—H 0.85 (1) Å and H···H 1.34 (2) Å], or using a riding model with C—H distances of 0.99 Å.

Data collection: *STADI*-4 (Stoe & Cie, 1997); cell refinement: *STADI*-4; data reduction: *X-RED* (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2001).

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