Table 2. *Bond lengths* (Å) *and angles* (°)

Mo—Cl(1)	2.376 (1)	Mo—Cl(2)	2.331 (1)
Mo—Cl(3)	2.404 (1)	Mo—Cl(4)	2.368 (1)
Mo—O	1.640 (2)	Mo—Cl(3^{i})	2.928 (1)
		C(1)—C(2)	1.521 (4)
C(1)—N(1)	1.294 (3)	C(1)—N(1′)	1.296 (3)
C(2)—F(2)	1.317 (3)	C(2)—F(2′)	1.322 (3)
C(2)—F(2″)	1.320 (4)		
Cl(1)—Mo—Cl(2)	87.1 (1)	Cl(1)—Mo—Cl(3)	89.0 (1)
Cl(2)—Mo—Cl(3)	156.8 (1)	Cl(1)—Mo—Cl(4)	163.6 (1)
Cl(2)—Mo—Cl(4)	88.5 (1)	Cl(3)—Mo—Cl(4)	88.8 (1)
Cl(1)—Mo—O	97.5 (1)	Cl(2)—Mo—O	103.2 (1)
Cl(3)—Mo—O	100.0 (1)	Cl(4)—Mo—O	98.9 (1)
Cl(1)—Mo—Cl(3^{i})	81.5 (1)	Cl(2)—Mo—Cl(3^{i})	80.6 (1)
Cl(3)—Mo—Cl(3^{i})	76.2 (1)	Cl(4)—Mo—Cl(3^{i})	82.2 (1)
O—Mo—Cl(3^{i})	176.0 (1)	Mo—Cl(3)—Mo^{i}	103.8 (1)
C(2)—C(1)—N(1)	118.2 (2)	C(2)—C(1)—N(1′)	116.3 (2)
N(1)—C(1)—N(1′)	125.5 (3)	C(1)—C(2)—F(2)	112.0 (2)
C(1)—C(2)—F(2′)	110.5 (2)	F(2)—C(2)—F(2′)	108.4 (2)
C(1)—C(2)—F(2″)	109.5 (2)	F(2)—C(2)—F(2″)	108.0 (2)
F(2′)—C(2)—F(2″)	108.4 (2)		

Symmetry code: (i) $-x, 1-y, -z$.

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows a plot with the atom numbering.

Related literature. For structures containing the monomeric $[MoOCl_4]^-$ anion, see Garner, Hill, Mabbs, McFadden & McPhail (1977), Knopp, Lörcher & Strähle (1977) and Weller, Müller, Weiher & Dehnicke (1980); for a structure containing the dimeric anion, see Klinzing, El-Kholi, Müller, Dehnicke & Findeisen (1989).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55230 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE1001]

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Tri-μ-chloro-bis(1,4,7-trithiacyclononane)dinickel(II) Tetrafluoroborate Acetonitrile Solvate

By Alexander J. Blake,* Malcolm A. Halcrow and Martin Schröder

Department of Chemistry, University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

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Abstract. $[Ni_2(C_6H_{12}S_3)_2Cl_3][BF_4].C_2H_3N$, $M_r = 712.28$, monoclinic, $P2_1/c$, $a = 12.379\ (5)$, $b = 13.790\ (6)$, $c = 16.031\ (6)$ Å, $\beta = 99.73\ (4)°$, $V = 2697$ Å^3, $Z = 4$, $D_x = 1.754$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 2.184$ mm^{-1}, $F(000) = 1448$, $T = 150.0\ (1)$ K, $R = 0.0562$ for 2173 unique observed reflections. Each Ni atom is approximately octahedrally coordinated to all three S atoms of one ligand molecule and to the three Cl atoms. Unusually, the Ni^{II} centres are triply chloride bridged, giving a cation which consists of two face-sharing octahedra in which the Ni···Ni distance is 2.9211 (20) Å.

Experimental. Compound prepared by reaction of $NiCl_2$ with 1,4,7-trithiacyclononane and $NaBF_4$ in CH_3NO_2; crystals obtained from CH_3CN/Et_2O. To prevent crystal degradation resulting from the loss of solvent, a pale-green lath with dimensions 0.08 × 0.21 × 0.70 mm was transferred from its cold mother liquor into a drop of mineral oil and then onto a Stoe Stadi-4 four-circle diffractometer equipped with an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986) operating at 150 K. Graphite-monochromated Mo $K\alpha$ radiation, cell parameters from 2θ values of 23 reflections measured at $\pm\,\omega$ ($20 < 2\theta < 22°$). For data collection, ω–2θ scans with ω-scan width $(1.32 + 0.35\tan\theta)°$, $2\theta_{\max} = 45°$, h $-13 \rightarrow 13$, k $0 \rightarrow 14$, l $0 \rightarrow 17$, three standard reflections, no significant crystal decay or movement, no absorption correction, 4448 reflections collected, 3084 unique ($R_{\rm int} = 0.05$), giving 2173 with $F > 4\sigma(F)$ for structure solution (from a Patterson synthesis

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* To whom correspondence should be addressed.

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

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}/U_{iso}(Å^2)
Ni(1)	0.01696 (11)	0.21717 (10)	0.12985 (9)	0.0211 (8)
Ni(2)	−0.16722 (11)	0.30581 (11)	0.01784 (9)	0.0213 (8)
Cl(1)	0.02603 (23)	0.34992 (22)	0.03296 (18)	0.0276 (15)
Cl(2)	−0.10620 (23)	0.13990 (21)	0.01595 (18)	0.0250 (15)
Cl(3)	−0.14003 (23)	0.29803 (23)	0.16926 (17)	0.0303 (16)
S(1)	−0.00622 (24)	0.08746 (23)	0.21974 (19)	0.0278 (16)
C(2)	0.0874 (9)	0.1183 (9)	0.3167 (7)	0.034 (3)
C(3)	0.1006 (9)	0.2258 (10)	0.3291 (7)	0.037 (3)
S(4)	0.13801 (24)	0.29363 (24)	0.24074 (18)	0.0281 (16)
C(5)	0.2721 (9)	0.2415 (8)	0.2324 (7)	0.028 (3)
C(6)	0.2798 (9)	0.2099 (9)	0.1425 (7)	0.033 (3)
S(7)	0.16798 (24)	0.13631 (23)	0.08929 (18)	0.0267 (16)
C(8)	0.1804 (10)	0.0267 (9)	0.1538 (8)	0.035 (3)
C(9)	0.0688 (9)	−0.0065 (9)	0.1720 (8)	0.031 (3)
S(11)	−0.34873 (23)	0.24937 (22)	0.00260 (19)	0.0265 (16)
C(12)	−0.3723 (10)	0.2184 (9)	−0.1073 (7)	0.035 (3)
C(13)	−0.3263 (9)	0.2864 (9)	−0.1675 (7)	0.034 (3)
S(14)	−0.18569 (23)	0.32309 (23)	−0.12998 (18)	0.0261 (16)
C(15)	−0.1940 (9)	0.4543 (9)	−0.1374 (7)	0.035 (3)
C(16)	−0.2655 (10)	0.5018 (9)	−0.0830 (7)	0.038 (3)
S(17)	−0.23015 (25)	0.46627 (23)	0.02592 (19)	0.0297 (17)
C(18)	−0.3617 (10)	0.4402 (9)	0.0533 (8)	0.036 (3)
C(19)	−0.4275 (9)	0.3613 (9)	0.0040 (8)	0.036 (3)
B	−0.4801 (11)	0.4860 (12)	0.2570 (10)	0.039 (4)
F(1)	−0.4367 (6)	0.5092 (6)	0.3422 (4)	0.049 (3)
F(2)	−0.4606 (6)	0.5603 (5)	0.2050 (5)	0.051 (3)
F(3)†	−0.4000 (11)	0.4149 (10)	0.2417 (8)	0.048 (3)
F(4)†	−0.5915 (10)	0.4805 (9)	0.2576 (8)	0.041 (3)
F(5)‡	−0.5745 (16)	0.4366 (15)	0.2440 (13)	0.053 (3)
F(6)‡	−0.4550 (15)	0.3959 (13)	0.2285 (11)	0.041 (3)
C(1*S*)	0.3402 (13)	0.4773 (11)	0.4449 (10)	0.068 (4)
C(2*S*)	0.3523 (10)	0.3750 (12)	0.4419 (8)	0.047 (3)
N(1*S*)	0.3592 (9)	0.2908 (10)	0.4453 (8)	0.060 (3)

† Site occupancy 0.6.
‡ Site occupancy 0.4.

Table 2. *Bond lengths* (Å), *angles* (°) *and torsion angles* (°)

Ni(1)—Ni(2)	2.9211 (20)	C(3)—S(4)	1.821 (13)
Ni(1)—Cl(1)	2.416 (3)	S(4)—C(5)	1.834 (12)
Ni(1)—Cl(2)	2.419 (3)	C(5)—C(6)	1.524 (17)
Ni(1)—Cl(3)	2.415 (3)	C(6)—S(7)	1.810 (12)
Ni(1)—S(1)	2.345 (3)	S(7)—C(8)	1.822 (13)
Ni(1)—S(4)	2.370 (3)	C(8)—C(9)	1.529 (17)
Ni(1)—S(7)	2.361 (3)	S(11)—C(12)	1.788 (12)
Ni(2)—Cl(1)	2.440 (3)	S(11)—C(19)	1.828 (12)
Ni(2)—Cl(2)	2.411 (3)	C(12)—C(13)	1.524 (17)
Ni(2)—Cl(3)	2.396 (3)	C(13)—S(14)	1.815 (12)
Ni(2)—S(11)	2.351 (3)	S(14)—C(15)	1.815 (12)
Ni(2)—S(14)	2.354 (3)	C(15)—C(16)	1.495 (17)
Ni(2)—S(17)	2.357 (3)	C(16)—S(17)	1.795 (13)
S(1)—C(2)	1.826 (12)	S(17)—C(18)	1.793 (13)
S(1)—C(9)	1.836 (12)	C(18)—C(19)	1.502 (17)
C(2)—C(3)	1.501 (17)		

Cl(1)—Ni(1)—Cl(2)	86.58 (11)	Ni(1)—Cl(3)—Ni(2)	74.76 (10)
Cl(1)—Ni(1)—Cl(3)	86.86 (11)	Ni(1)—S(1)—C(2)	102.7 (4)
Cl(1)—Ni(1)—S(1)	175.61 (12)	Ni(1)—S(1)—C(9)	99.2 (4)
Cl(1)—Ni(1)—S(4)	93.04 (11)	C(2)—S(1)—C(9)	103.1 (6)
Cl(1)—Ni(1)—S(7)	93.20 (11)	S(1)—C(2)—C(3)	112.4 (8)
Cl(2)—Ni(1)—Cl(3)	88.19 (11)	C(2)—C(3)—S(4)	116.2 (9)
Cl(2)—Ni(1)—S(1)	90.05 (11)	Ni(1)—S(4)—C(3)	98.2 (4)
Cl(2)—Ni(1)—S(4)	179.60 (12)	Ni(1)—S(4)—C(5)	103.7 (4)
Cl(2)—Ni(1)—S(7)	90.66 (11)	C(3)—S(4)—C(5)	102.0 (5)
Cl(3)—Ni(1)—S(1)	90.23 (11)	S(4)—C(5)—C(6)	112.5 (8)
Cl(3)—Ni(1)—S(4)	91.90 (11)	C(5)—C(6)—S(7)	116.1 (8)
Cl(3)—Ni(1)—S(7)	178.84 (12)	Ni(1)—S(7)—C(6)	100.6 (4)
S(1)—Ni(1)—S(4)	90.34 (12)	Ni(1)—S(7)—C(8)	103.3 (4)
S(1)—Ni(1)—S(7)	89.65 (12)	C(6)—S(7)—C(8)	102.3 (6)
S(4)—Ni(1)—S(7)	89.25 (11)	S(7)—C(8)—C(9)	111.4 (8)
Cl(1)—Ni(2)—Cl(2)	86.21 (11)	S(1)—C(9)—C(8)	113.9 (8)
Cl(1)—Ni(2)—Cl(3)	86.73 (11)	Ni(2)—S(11)—C(12)	100.1 (4)
Cl(1)—Ni(2)—S(11)	175.09 (12)	Ni(2)—S(11)—C(19)	102.8 (4)
Cl(1)—Ni(2)—S(14)	90.09 (11)	C(12)—S(11)—C(19)	102.4 (6)
Cl(1)—Ni(2)—S(17)	95.05 (11)	S(11)—C(12)—C(13)	117.5 (9)
Cl(2)—Ni(2)—Cl(3)	88.83 (11)	C(12)—C(13)—S(14)	113.7 (8)
Cl(2)—Ni(2)—S(11)	88.88 (11)	Ni(2)—S(14)—C(13)	103.3 (4)
Cl(2)—Ni(2)—S(14)	93.48 (11)	Ni(2)—S(14)—C(15)	99.3 (4)
Cl(2)—Ni(2)—S(17)	177.17 (12)	C(13)—S(14)—C(15)	102.5 (6)
Cl(3)—Ni(2)—S(11)	93.36 (11)	S(14)—C(15)—C(16)	115.5 (9)
Cl(3)—Ni(2)—S(14)	175.94 (12)	C(15)—C(16)—S(17)	112.2 (9)
Cl(3)—Ni(2)—S(17)	88.71 (11)	Ni(2)—S(17)—C(16)	103.4 (4)
S(11)—Ni(2)—S(14)	90.02 (11)	Ni(2)—S(17)—C(18)	98.5 (4)
S(11)—Ni(2)—S(17)	89.86 (12)	C(16)—S(17)—C(18)	102.2 (6)
S(14)—Ni(2)—S(17)	89.05 (12)	S(17)—C(18)—C(19)	116.8 (9)
Ni(1)—Cl(1)—Ni(2)	73.96 (10)	S(11)—C(19)—C(18)	112.0 (8)
Ni(1)—Cl(2)—Ni(2)	74.42 (10)		

C(9)—S(1)—C(2)—C(3)	133.1 (9)	C(19)—S(11)—C(12)—C(13)	66.0 (10)
C(2)—S(1)—C(9)—C(8)	−58.7 (10)	C(12)—S(11)—C(19)—C(18)	−131.1 (9)
S(1)—C(2)—C(3)—S(4)	−52.7 (11)	S(11)—C(12)—C(13)—S(14)	45.8 (12)
C(2)—C(3)—S(4)—C(5)	−62.2 (10)	C(12)—C(13)—S(14)—C(15)	−127.5 (9)
C(3)—S(4)—C(5)—C(6)	129.5 (9)	C(13)—S(14)—C(15)—C(16)	62.6 (10)
S(4)—C(5)—C(6)—S(7)	−49.0 (11)	S(14)—C(15)—C(16)—S(17)	53.2 (11)
C(5)—C(6)—S(7)—C(8)	−64.2 (10)	C(15)—C(16)—S(17)—C(18)	−133.8 (9)
C(6)—S(7)—C(8)—C(9)	135.0 (9)	C(16)—S(17)—C(18)—C(19)	60.4 (10)
S(7)—C(8)—C(9)—S(1)	−54.3 (10)	S(17)—C(18)—C(19)—S(11)	51.9 (11)

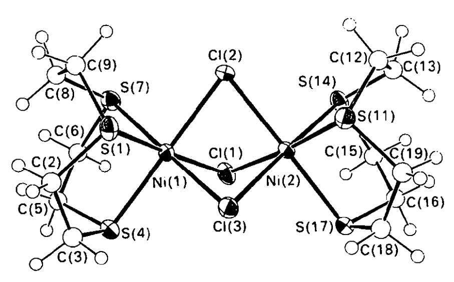

Fig. 1. A general view of the triply chloro-bridged dimer showing the atom-numbering scheme; thermal ellipsoids are drawn at the 30% probability level, excepting those of the H atoms which, for clarity, are drawn with artificial radii of 0.10 Å.

followed by iterative cycles of least-squares refinement and difference Fourier synthesis) and refinement [full-matrix least squares on F using *SHELX*76 (Sheldrick, 1976)]. Disorder in the BF_4^- anion was modelled by allowing partial occupancies of alternative positions for some F atoms. Anisotropic thermal parameters for all ordered non-H atoms, isotropic for disordered F, H atoms in fixed, calculated positions or as part of a rigid CH_3 group, no secondary-extinction parameter. At final convergence, $R = 0.0562$, $wR = 0.0640$, $S = 1.059$ for 282 parameters, $(\Delta/\sigma)_{max}$ in final cycle 0.06, maximum and minimum residuals in final ΔF synthesis 0.84, -1.11 e Å^{-3} respectively. The weighting scheme $w = [\sigma^2(F) + 0.000664F^2]^{-1}$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976) except for the Ni atoms for which values from Cromer & Mann (1968) were used. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, and selected bond lengths, valence angles and torsion angles in Table 2.* The atom-numbering scheme for the molecule is shown in Fig. 1, which was generated using *ORTEP*II (Mallinson & Muir, 1985). Molecular-geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

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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 55226 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0100]

Related literature. Although common for other metals (*e.g.* Ru, Os), triple chloride bridges are rare for Ni which prefers to form doubly bridged species such as $Ni_2Cl_8^{4-}$ (Ross & Stucky, 1970) and $[Ni_2(en)_4Cl_2]^{2+}$ where en = 1,2-diaminoethane (Carlin, Joung, O'Connor & Sinn, 1979). The only other structurally characterized example of a halo-bridged Ni^{II}/Ni^{II} dimer involving thioether ligands is the doubly chloride bridged $[Ni_2\{N(CH_2CH_2S^iPr)_3\}_2Cl_2]^{2+}$ (Carrié, Holm, Muetterties & Stavropoulos, 1991).

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Structure of Ethyl 8,9-Dimethoxy-3-(4-methoxyphenyl)-10b-methyl-1-phenyl-4,5,6,10b-tetrahydro-1,2,4-triazolo[3,4-*a*]isoquinoline-5-carboxylate

By François Theobald*
14 *Impasse Marguerite*, 78000 *Versailles*, *France*

Noël Rodier
Laboratoire de Chimie Minérale, *Faculté de Pharmacie*, 5 *rue J. B. Clément*,
92296 *Châtenay-Malabry CEDEX*, *France*

and Khadija Moustaid and Bernard Laude
Laboratoire de Chimie Organique, *Université de Franche-Comté*, 16 *route de Gray*, *La Bouloie*,
25030 *Besançon CEDEX*, *France*

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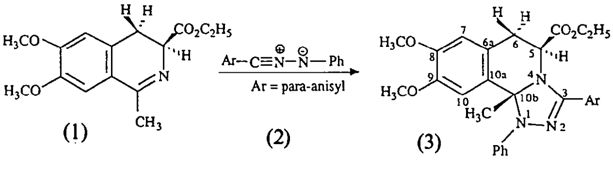
Abstract. $C_{29}H_{31}N_3O_5$, $M_r = 501.6$, orthorhombic, $Pna2_1$, $a = 23.867$ (2), $b = 9.492$ (1), $c = 11.6150$ (6) Å, $V = 2631.2$ (6) Å^3, $Z = 4$, $D_m = 1.27$, $D_x = 1.266$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 0.67$ mm^{-1}, $F(000) = 1064$, $T = 295$ (1) K, $R = 0.033$ for 1933 independent observed reflections. The title compound has the methyl group at C(10b) and the ester moiety at C(5) in a *cis* disposition; so the name may be specified as ethyl 3-*p*-anisyl-8,9-dimethoxy-*exo*-10b-methyl-1-phenyl-4,5,6,10b-tetrahydro-1,2,4-triazolo[3,4-*a*]isoquinoline-*exo*-5-carboxylate. In the cycloaddition synthesis, the approach of the 1,3-dipole occurs to the less-hindered diastereotopic side of the dipolarophile. Moreover, the conformation of the hexagonal N heterocycle is of the 1,3-diplanar type; the methyl group at C(10b) lies in a pseudo-equatorial disposition, whereas the ester group at C(5) lies in a pseudo-axial one.

Experimental. The 1,3-dipolar cycloaddition of C-*p*-anisyl-*N*-phenylnitrilimine (2) with ethyl 6,7-dimethoxy-1-methyl-3,4-dihydroisoquinoline-3-carboxylate (1) yields a single cycloadduct (3), regio- and stereospecifically.



Data were collected using a crystal with an approximate parallelepipedic shape, 0.19 × 0.30 × 0.34 mm. Density was measured by flotation. Intensi-

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* To whom correspondence should be addressed.

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