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Retraction of articles

This article reports the retraction of 39 articles published in *Acta Crystallographica Section E* between 2004 and 2009.

After thorough investigation (see Harrison *et al.*, 2010), 39 additional articles are retracted as a result of problems with the data sets or incorrect atom assignments. Full details of all the articles are given in Table 1.

Table 1
Details of articles to be retracted, in order of publication.

Title	Reference	Retracted by	DOI	Refcode
trans-Bis{1-[3-(cyclohexylamino)propyliminomethyl]-2-naphtholato}copper(II) dichloride dihydrate	Zhang (2004)	Journal	10.1107/S1600536804028296	BIPDUA
Bis(4-bromo-2-formylphenolato-κ^2O,O')copper(II)	Sun & Gao (2005)	Author	10.1107/S160053680500187X	FEYSUY
Bis(salicylaldehydo)zinc(II)	Xiong & Liu (2005)	Journal	10.1107/S1600536805010913	GAMDUU
Bis(4-bromo-2-formylphenolato-κ^2O,O')zinc(II)	Chen (2006)	Journal	10.1107/S1600536805040432	SAZCUS
Bis(2-formylphenolato-κ^2O,O')nickel(II)	Li & Chen (2006)	Journal	10.1107/S1600536806012931	IDAZAP
Bis(2-formylphenolato)cobalt(II)	Qiu (2006)	Journal	10.1107/S1600536806015704	GEJDUV
Bis(2-formylphenolato-κ^2O,O')manganese(II)	Wang & Fang (2006)	Journal	10.1107/S1600536806021039	IDOVED
Tetraaqua(1,10-phenanthroline-κ^2N,N')copper(II) naphthalene-1,5-disulfonate dihydrate	Liu *et al.* (2006)	Author	10.1107/S1600536806030637	GENYOO
Tetraaqua(1,10-phenanthroline-κ^2N,N')nickel(II) naphthalene-1,5-disulfonate dihydrate	Liu & Fan (2006)	Author	10.1107/S1600536806035410	KERBEP
{6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratolutetium(III)copper(II)	Sui *et al.* (2006)	Journal	10.1107/S160053680604565X	HESPEP
Bis(2-formylphenolato-κ^2O,O')iron(II)	Yang *et al.* (2007)	Author	10.1107/S1600536807021721	PIFCAJ
2,6-Dimethoxybenzohydrazide	Qadeer *et al.* (2007*a*)	Journal	10.1107/S1600536807022593	PIFHES
2-(2,4-Dichlorophenylsulfanyl)acetohydrazide	Qadeer *et al.* (2007*b*)	Journal	10.1107/S1600536807022891	YIFSOW
{6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratoeuropium(III)zinc(II)	Hu *et al.* (2007)	Author	10.1107/S1600536807031121	WIHKEE
{μ-6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratocerium(III)zinc(II)	Sui, Zhang, Hu & Yin (2007)	Author	10.1107/S1600536807032564	WIHREL
{μ-6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratopraseodymium(III)zinc(II)	Chen *et al.* (2007)	Author	10.1107/S1600536807032540	WIHRIP
{μ-6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratopraseodymium(III)nickel(II)	Sui, Li *et al.* (2007)	Author	10.1107/S1600536807032618	UFACUA
{6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato-1$\kappa^4O^1,O^{1'},O^6,O^{6'}$:2$\kappa^4O^1,N,N',O^{1'}$}(methanol-1κO)-μ-nitrato-1:2κ^2O:O′-dinitrato-1κ^4O,O'-cerium(III)zinc(II)	Sui, Fang, Hu & Lin (2007)	Author	10.1107/S1600536807033314	UDUYIC
{6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-methanol-μ-nitrato-dinitratosamarium(III)nickel(II)	Sui, Zhang, Hu & Jiang (2007)	Author	10.1107/S1600536807037130	AFECEU
{6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-methanol-μ-nitrato-dinitratopraseodymium(III)zinc(II)	Sui, Fang & Yuan (2007)	Author	10.1107/S1600536807037488	AFICEY
{6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-methanol-μ-nitrato-dinitratolutetium(III)zinc(II)	Sui, Sui *et al.* (2007)	Author	10.1107/S1600536807037737	AFEFOH
catena-Poly[[chloridonickel(II)]-di-μ-chlorido-[chloridonickel(II)]-μ-4,4′-methylenebis(3,5-dimethylpyrazole)-κ^2N^2:$N^{2'}$]	Huang & Chen (2007)	Author	10.1107/S1600536807039384	VIJYOD
{2,2′-[o-Phenylenebis(nitrilomethylidyne)]diphenolato}zinc(II)	Liu *et al.* (2007*a*)	Author	10.1107/S1600536807040640	DIKYUS
trans-Bis(ethylenediamine-κ^2N,N')bis(nitrato-κO)zinc(II)	Liu, Zeng & Chen (2007)	Author	10.1107/S1600536807042390	XIKYEW
[N,N′-(o-Phenylene)bis(picolinamido)-κ^4N,N',N'',N''']cobalt(II)	Liu & Zeng (2007*a*)	Author	10.1107/S1600536807044571	XILFII
[N,N′-(o-Phenylene)dipicolinamide-κ^4N]nickel(II)	Liu & Zeng (2007*b*)	Author	10.1107/S1600536807048386	WINWEW
{2,2′-[o-Phenylenebis(nitrilomethylidyne)]diphenolato}manganese(II)	Liu *et al.* (2007*b*)	Author	10.1107/S1600536807052993	VIQPIV
N-(2-Amino-3-pyridyl)urea monohydrate	Li *et al.* (2007)	Author	10.1107/S1600536807047526	SIMFEA
N-(2-Fluorophenyl)carbamic acid monohydrate	Yang (2007)	Author	10.1107/S1600536807052464	WINMOW
Aqua(dimethylglyoxime-κ^2N,N')(3,5-dinitro-2-oxidobenzoato-κ^2O^1,O^2)-copper(II)	Liu & Wen (2007)	Author	10.1107/S1600536807054244	HIQCAM
μ-Acetato-tri-μ-ferrocenecarboxylatobis[(N,N-dimethylformamide)-copper(II)]	Liu, Lin *et al.* (2007)	Journal	10.1107/S1600536807059041	HIQQEE

 doi:10.1107/S1600536809054300

Table 1 (continued)

Title	Reference	Retracted by	DOI	Refcode
{μ-6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-μ-nitrato-dinitratoeuropium(III)zinc(II)	Hu *et al.* (2008)	Author	10.1107/S160053680706151X	MIRPAF
Bis(4-chloro-2-formylphenolato)nickel(II)	Li *et al.* (2008)	Author	10.1107/S1600536807056309	RISTET
{μ-6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-μ-nitrato-dinitratoterbium(III)zinc(II)	Chen *et al.* (2008)	Author	10.1107/S1600536808006958	QIXHIP
Bis(2-ethoxy-6-formylphenolato-κ^2O^1,O^6)nickel(II)	Han (2008)	Journal	10.1107/S160053680800809X	QIXLIT
{μ-6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-μ-nitrato-dinitratoholmium(III)zinc(II)	Xiao, Sui *et al.* (2008)	Author	10.1107/S1600536808013743	BIZTUA
{μ-6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratoholmium(III)nickel(II)	Xiao, Fu *et al.* (2008)	Author	10.1107/S1600536808013755	BIZVAI
Hydrogen-bonding patterns in the cocrystal terephthalic acid–4,4′-bipyridine (2/1)	Wang *et al.* (2009)	Journal	10.1107/S160053680903236X	DUCZEH
{6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato-1$\kappa^4O^1,O^{1'},O^6,O^{6'}$:2$\kappa^4O^1,N,N',O^{1'}$}(ethanol-1κO)-μ-nitrato-1:2κ^2O:O′-dinitrato-1κ^4O,O′-samarium(III)zinc(II)	Huang *et al.* (2009)	Journal	10.1107/S1600536809033558	YUCWAV

References

Chen, Q. (2006). *Acta Cryst.* E**62**, m56–m57.
Chen, J.-R., Sui, Y., Luo, Q.-Y. & Jiang, R.-Q. (2007). *Acta Cryst.* E**63**, m2091–m2092.
Chen, J.-R., Sui, Y., Wen, J.-W. & Yin, L.-Y. (2008). *Acta Cryst.* E**64**, m562–m563.
Han, Z.-Q. (2008). *Acta Cryst.* E**64**, m592.
Harrison, W. T. A., Simpson, J. & Weil, M. (2010). *Acta Cryst.* E**66**, e1–e2.
Hu, R.-H., Sui, Y., Chen, L. & He, C.-M. (2008). *Acta Cryst.* E**64**, m8–m9.
Hu, R.-H., Sui, Y., Fang, X.-N. & Chen, H.-M. (2007). *Acta Cryst.* E**63**, m2039–m2040.
Huang, C.-F. & Chen, H.-L. (2007). *Acta Cryst.* E**63**, m2356–m2357.
Huang, Q., Sui, Y.-H. & Zhang, G.-X. (2009). *Acta Cryst.* E**65**, m1161–m1162.
Li, Y.-G. & Chen, H.-J. (2006). *Acta Cryst.* E**62**, m1038–m1039.
Li, N.-G., Tao, R.-M. & Fu, B.-F (2007). *Acta Cryst.* E**63**, o4228.
Li, Z., Zhang, X. & Pu, X. (2008). *Acta Cryst.* E**64**, m215.
Liu, J.-T. & Fan, S.-D. (2006). *Acta Cryst.* E**62**, m2507–m2508.
Liu, J.-T., Fan, S.-D. & Li, D.-Q. (2006). *Acta Cryst.* E**62**, m2165–m2166.
Liu, D., Lin, J., Xu, Y., Huang, C. & Li, X. (2007). *Acta Cryst.* E**63**, m3094.
Liu, Y.-Q. & Wen, H.-R. (2007). *Acta Cryst.* E**63**, m2928.
Liu, Y.-Q. & Zeng, X.-R. (2007*a*). *Acta Cryst.* E**63**, m2547.
Liu, Y.-Q. & Zeng, X.-R. (2007*b*). *Acta Cryst.* E**63**, m2684.
Liu, Y.-Q., Zeng, X.-R. & Chen, W.-T. (2007). *Acta Cryst.* E**63**, m2462.
Liu, Y.-Q., Zeng, X.-R., Luo, Q.-Y. & Xu, Y.-P. (2007*a*). *Acta Cryst.* E**63**, m2396.
Liu, Y.-Q., Zeng, X.-R., Luo, Q.-Y. & Xu, Y.-P. (2007*b*). *Acta Cryst.* E**63**, m2854.
Qadeer, G., Rama, N. H. & Chen, W.-T. (2007*a*). *Acta Cryst.* E**63**, o2892.
Qadeer, G., Rama, N. H. & Chen, W.-T. (2007*b*). *Acta Cryst.* E**63**, o2932.
Qiu, X.-Y. (2006). *Acta Cryst.* E**62**, m1190–m1191.
Sui, Y., Fang, X.-N., Hu, P. & Lin, J. (2007). *Acta Cryst.* E**63**, m2135–m2136.
Sui, Y., Fang, X.-N. & Yuan, M.-W. (2007). *Acta Cryst.* E**63**, m2275–m2276.
Sui, Y., Li, X.-F., Huang, G.-S. & Wang, G.-J. (2007). *Acta Cryst.* E**63**, m2093–m2094.
Sui, Y., Sui, Y.-H., Luo, Q.-Y. & Wang, Y.-D. (2007). *Acta Cryst.* E**63**, m2277–m2278.
Sui, Y., Xiao, Y.-A., Fang, X.-N., Zeng, X.-R. & Li, M.-H. (2006). *Acta Cryst.* E**62**, m3205–m3207.
Sui, Y., Zhang, J.-H., Hu, R.-H. & Jiang, R.-Q. (2007). *Acta Cryst.* E**63**, m2256–m2257.
Sui, Y., Zhang, J.-H., Hu, R.-H. & Yin, L.-Y. (2007). *Acta Cryst.* E**63**, m2089–m2090.
Sun, Y.-X. & Gao, G.-Z. (2005). *Acta Cryst.* E**61**, m354–m355.
Wang, Q. & Fang, Z.-N. (2006). *Acta Cryst.* E**62**, m1492–m1493.
Wang, S., Yang, T., Li, Z. & Yu, X. (2009). *Acta Cryst.* E**65**, o2198.
Xiao, Y.-A., Fu, X.-K., Sui, Y., Wu, Q. & Xiong, S.-H. (2008). *Acta Cryst.* E**64**, m806–m807.
Xiao, Y.-A., Sui, Y., Yi, X.-G., Wu, J.-H. & Zhang, L.-P. (2008). *Acta Cryst.* E**64**, m804–m805.
Xiong, Z.-Y. & Liu, L.-J. (2005). *Acta Cryst.* E**61**, m863–m864.
Yang, X.-M. (2007). *Acta Cryst.* E**63**, o4453.
Yang, Y.-M., Lu, P.-C., Zhu, T.-T. & Liu, C.-H. (2007). *Acta Cryst.* E**63**, m1613.
Zhang, P. (2004). *Acta Cryst.* E**60**, m1808–m1810.

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catena-Poly[[chloridonickel(II)]-di-μ-chlorido-[chloridonickel(II)]-μ-4,4′-methylenebis(3,5-dimethylpyrazole)-$\kappa^2N^2:N^{2'}$]

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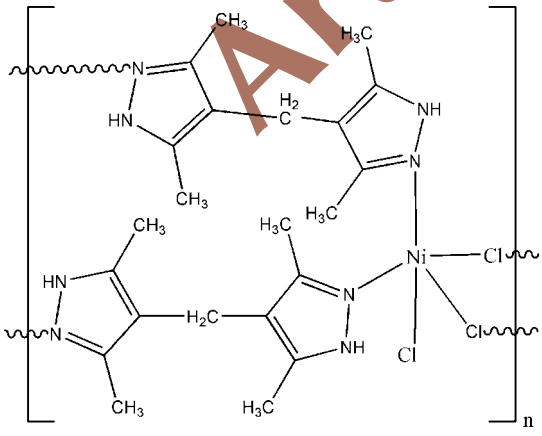
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.012 Å; R factor = 0.065; wR factor = 0.208; data-to-parameter ratio = 13.7.

The title compound, $[NiCl_2(C_{11}H_{16}N_4)]_n$, is a one-dimensional polymer built up from alternating $(NiCl_2)_2$ units and bridging 4,4′-methylenebis(3,5-dimethylpyrazole) ligands. An unusual $NiCl_3N_2$ square-based pyramidal coordination arises for the metal atom. The packing is consolidated by N—H···Cl hydrogen bonds.

Related literature

For related literature, see: Constable & Cargill Thompson (1992); Hennigar *et al.* (1997); Kaes *et al.* (1998); Loi *et al.* (1999) Neels *et al.* (1997); Neeraj *et al.* (1999); Veltan & Rehahn (1996); Yaghi *et al.* (1998).



Experimental

Crystal data

$[NiCl_2(C_{11}H_{16}N_4)]$
M_r = 333.89
Triclinic, $P\overline{1}$
a = 8.759 (3) Å
b = 8.879 (3) Å
c = 9.735 (3) Å
α = 79.269 (6)°
β = 63.584 (5)°
γ = 86.922 (5)°
V = 665.8 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.85 mm^{-1}
T = 298 (2) K
0.28 × 0.22 × 0.15 mm

Data collection

Bruker APEX II CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.626, T_{max} = 0.769
3330 measured reflections
2312 independent reflections
1534 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.208
S = 0.97
2312 reflections
169 parameters
40 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −1.05 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni2—N3	1.992 (6)	Ni2—Cl2	2.311 (2)
Ni2—N1^{i}	2.013 (6)	Ni2—Cl2^{ii}	2.713 (2)
Ni2—Cl1	2.294 (2)		

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y, -z+2$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···Cl1^{iii}	0.96	2.45	3.227 (6)	138
N2—H2···Cl1^{i}	0.96	2.59	3.123 (6)	116
N4—H4···Cl1^{ii}	0.99	2.19	3.167 (7)	169

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y, -z+2$; (iii) $x+1, y+1, z-1$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2501).

References

Bruker (1998). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Constable, E. C. & Cargill Thompson, A. M. W. (1992). *J. Chem. Soc. Dalton Trans.* pp. 3467–3475.
Hennigar, T. L., MacQuarrie, D. C., Losier, P., Gogers, R. D. & Zaworotko, M. J. (1997). *Angew. Chem. Int. Ed. Engl.* **36**, 972–973.
Kaes, C., Hosseini, M. W., Richard, C. E. F., Skelton, B. B. & White, A. (1998). *Angew. Chem. Int. Ed.* **37**, 920–922.
Loi, M., Graf, E., Hosseini, M. W., De Cian, A. & Fischer, J. (1999). *Chem. Commun.* pp. 603–604.
Neels, A., Neels, B. M. & Stoeckli-Evans, H. (1997). *Inorg. Chem.* **36**, 3402–3409.
Neeraj, S., Natarajan, S. & Rao, C. N. R. (1999). *Angew. Chem. Int. Ed.* **38**, 3480–3483.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Veltan, U. & Rehahn, M. (1996). *Chem. Commun.* pp. 2639–2640.
Yaghi, O. M., Li, H., Davis, C., Richardson, D. & Groy, T. L. (1998). *Acc. Chem. Res.* **31**, 474–484.

supplementary materials

Acta Cryst. (2007). E**63**, m2356-m2357 [**doi:10.1107/S1600536807039384**]

catena-Poly[[chloridonickel(II)]-di-μ-chlorido-[chloridonickel(II)]-μ-4,4'-methylenebis(3,5-dimethylpyrazole)-$\kappa^2N^2:N^{2'}$]

C.-F. Huang and H.-L. Chen

Comment

Interest in one dimensional chain structures arises partly because these structures are expected to play a crucial role as precursors in the formation of two- and three-dimensional structures (Neeraj *et al.*, 1999). In the past, the majority of one-dimensional coordination networks were found to be composed of bis-monodentate tectons (Yaghi *et al.*, 1998; Hennigar *et al.*, 1997), while few examples of complexes with bis-bidentate (Veltan & Rehahn, 1996; Kaes *et al.*, 1998), and bis-tridentate tectons (Constable & Cargill Thompson, 1992; Neels *et al.*, 1997; Loi *et al.*, 1999) were published.

In this paper, we report the crystal structure of the title compound, (I), (Fig. 1), containing the bis-bidentate organic tecton 4,4'-methylene-bis(3,5-dimethylpyrazole) and Cl ligands. The Ni atom is coordinated by three Cl^- ions and two N-bonded H_2mbdpz ligands (Table 1). The four nearest atoms result in a *cis*-$NiCl_2N_2$ square planar geometry and a third chloride ion with a much longer Ni—Cl bond distance completes a distorted $NiCl_3N_2$ square pyramid. The alternating $(NiCl_2)_2$ groups and pairs of bridging H_2mbdpz ligands form an infinite one-dimensional chain (Fig. 2). The dihedral angle between the two pyrazole rings within one ligand is 81.8 (3)°. which is slightly smaller than that in the free ligand. The Ni···Ni non-bonding distance between adjacent metal ions in the chain is 3.728 (4) Å. The structure is completed by N—H···Cl hydrogen bonds (Table 2).

Experimental

H_2mbdpz (102 mg, 0.5 mmol) in ethanol (10 ml) was added to a solution of $NiCl_2$ (12.9 mg, 0.1 mmol) in H_2O (10 ml). The mixture was refuxed for 2 h with stirring, yielding a brown precipitate. The solution was then filtered to remove the precipitate, which was subsequently washed with water, methanol and acetone, and finally dried. The solid was dissolved in DMF, producing a clear solution, which was allowed to stand undisturbed at room temperature for a few weeks at which time green blocks of (I) were obtained.

Refinement

The H atoms were refined with a riding model [C—H = 0.93–0.97Å (geometrically placed) and N—H = 0.96–0.98Å (located in a difference map); U_{iso}(H) = 1.2U_{eq} or 1.5 U_{eq}(carrier)]. The methyl groups were allowed to rotate but not to tip. The maximum difference peak is 1.12Å from Cl2.

Figures

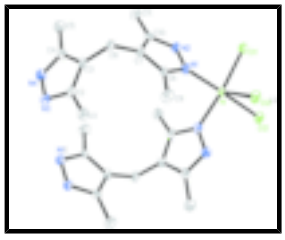


Fig. 1. The structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level. Symmetry codes: (i) *x*, 1 − *y*, 1 + *z* and (ii) 1 − *x*, −*y*, 2 − *z*.

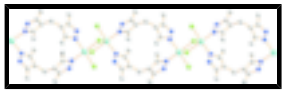


Fig. 2. Part of a polymeric chain in (I), viewed along the *a* axis.

catena-Poly[[chloridonickel(II)]-di-μ-chlorido-[chloridonickel(II)]-μ-\ 4,4'-methylenebis(3,5-dimethylpyrazole)-$\kappa^2N^2:N^{2'}$]

Crystal data

$[NiCl_2(C_{11}H_{16}N_4)]$	$Z = 2$
$M_r = 333.89$	$F_{000} = 344$
Triclinic, $P\overline{1}$	$D_x = 1.665$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
$a = 8.759$ (3) Å	Cell parameters from 3212 reflections
$b = 8.879$ (3) Å	θ = 2.3–25.1°
$c = 9.735$ (3) Å	μ = 1.85 mm^{-1}
α = 79.269 (6)°	$T = 298$ (2) K
β = 63.584 (5)°	Block, green
γ = 86.922 (5)°	0.28 × 0.22 × 0.15 mm
$V = 665.8$ (4) Å^3	

Data collection

Bruker APEX II CCD diffractometer	2312 independent reflections
Radiation source: fine-focus sealed tube	1534 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.033$
Detector resolution: 0 pixels mm^{-1}	$\theta_{max} = 25.1°$
$T = 298(2)$ K	$\theta_{min} = 2.3°$
ω scans	$h = -10\rightarrow9$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -7\rightarrow10$
$T_{min} = 0.626$, $T_{max} = 0.769$	$l = -11\rightarrow11$
3330 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.065$

$wR(F^2) = 0.208$

$S = 0.97$

2312 reflections

169 parameters

40 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1397P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.73$ e Å^{-3}

$\Delta\rho_{min} = -1.05$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni2	0.36689 (12)	0.08445 (11)	0.90152 (10)	0.0275 (4)
Cl1	0.1129 (2)	0.0033 (2)	1.1136 (2)	0.0370 (5)
Cl2	0.4813 (3)	−0.1538 (2)	0.9274 (2)	0.0384 (6)
C8	0.6764 (9)	0.5680 (8)	0.1917 (8)	0.0277 (16)
N2	0.8976 (7)	0.7132 (6)	0.1257 (7)	0.0324 (15)
C10	0.9389 (9)	0.5714 (9)	0.1731 (8)	0.0299 (17)
C3	0.7286 (9)	0.2395 (8)	0.4362 (8)	0.0295 (17)
N4	0.7209 (8)	0.1467 (8)	0.6655 (7)	0.0371 (17)
N3	0.5559 (8)	0.1395 (8)	0.6870 (7)	0.0373 (16)
C4	0.8261 (10)	0.2016 (10)	0.5168 (9)	0.039 (2)
C6	0.7886 (10)	0.3010 (9)	0.2662 (8)	0.0339 (19)
H39A	0.7122	0.2606	0.2331	0.041*
H39B	0.9005	0.2615	0.2101	0.041*
C7	0.8005 (9)	0.4745 (8)	0.2170 (8)	0.0289 (17)
C1	0.4016 (10)	0.1983 (10)	0.5276 (9)	0.042 (2)
H1C	0.3209	0.1224	0.6070	0.063*
H1A	0.4272	0.1766	0.4267	0.063*
H1B	0.3538	0.2979	0.5364	0.063*
C2	0.5595 (10)	0.1951 (8)	0.5472 (9)	0.0306 (17)
C5	1.0141 (10)	0.2126 (12)	0.4657 (10)	0.049 (2)
H45A	1.0450	0.3135	0.4691	0.074*

H45B	1.0742	0.1934	0.3611	0.074*
H45C	1.0438	0.1380	0.5342	0.074*
C9	0.5025 (10)	0.5253 (9)	0.2176 (10)	0.041 (2)
H30A	0.4218	0.5337	0.3219	0.061*
H30B	0.5007	0.4215	0.2028	0.061*
H30C	0.4728	0.5929	0.1446	0.061*
C11	1.1094 (10)	0.5454 (11)	0.1706 (10)	0.045 (2)
H47A	1.1901	0.6217	0.0919	0.068*
H47B	1.1461	0.4453	0.1477	0.068*
H47C	1.1020	0.5524	0.2706	0.068*
N1	0.7387 (7)	0.7146 (6)	0.1358 (7)	0.0348 (16)
H4	0.7577	0.1015	0.7460	0.070 (6)*
H2	0.9769	0.7979	0.0653	0.069 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni2	0.0317 (6)	0.0230 (6)	0.0266 (6)	0.0017 (4)	−0.0143 (4)	0.0015 (4)
Cl1	0.0349 (11)	0.0368 (12)	0.0387 (11)	−0.0040 (9)	−0.0201 (9)	0.0060 (9)
Cl2	0.0499 (13)	0.0277 (11)	0.0434 (12)	0.0046 (9)	−0.0264 (10)	−0.0058 (9)
C8	0.033 (4)	0.022 (4)	0.024 (4)	0.004 (3)	−0.011 (3)	0.000 (3)
N2	0.029 (3)	0.026 (4)	0.039 (4)	−0.001 (3)	−0.016 (3)	0.005 (3)
C10	0.029 (4)	0.033 (4)	0.026 (4)	−0.001 (3)	−0.011 (3)	−0.003 (3)
C3	0.033 (4)	0.020 (4)	0.033 (4)	0.001 (3)	−0.014 (3)	−0.001 (3)
N4	0.032 (4)	0.044 (4)	0.038 (4)	−0.004 (3)	−0.022 (3)	0.003 (3)
N3	0.036 (4)	0.038 (4)	0.035 (4)	0.009 (3)	−0.017 (3)	−0.001 (3)
C4	0.045 (5)	0.039 (5)	0.030 (4)	0.005 (4)	−0.019 (4)	0.003 (4)
C6	0.041 (5)	0.028 (4)	0.031 (4)	0.012 (4)	−0.017 (4)	−0.003 (3)
C7	0.035 (4)	0.023 (4)	0.030 (4)	0.002 (3)	−0.016 (3)	−0.004 (3)
C1	0.048 (5)	0.044 (5)	0.037 (5)	0.005 (4)	−0.023 (4)	−0.003 (4)
C2	0.036 (4)	0.023 (4)	0.036 (4)	0.003 (3)	−0.021 (4)	−0.002 (3)
C5	0.041 (5)	0.064 (7)	0.046 (5)	0.003 (5)	−0.027 (4)	0.000 (5)
C9	0.038 (5)	0.031 (5)	0.065 (6)	−0.007 (4)	−0.032 (4)	−0.008 (4)
C11	0.040 (5)	0.047 (6)	0.046 (5)	0.013 (4)	−0.019 (4)	−0.006 (4)
N1	0.032 (4)	0.031 (4)	0.039 (4)	−0.001 (3)	−0.016 (3)	0.002 (3)

Geometric parameters (Å, °)

Ni2—N3	1.992 (6)	N3—C2	1.346 (9)
Ni2—N1^{i}	2.013 (6)	C4—C5	1.497 (11)
Ni2—Cl1	2.294 (2)	C6—C7	1.520 (10)
Ni2—Cl2	2.311 (2)	C6—H39A	0.9700
Ni2—Cl2^{ii}	2.713 (2)	C6—H39B	0.9700
Cl2—Ni2^{ii}	2.713 (2)	C1—C2	1.476 (10)
C8—N1	1.355 (9)	C1—H1C	0.9600
C8—C7	1.413 (10)	C1—H1A	0.9600
C8—C9	1.488 (10)	C1—H1B	0.9600
N2—C10	1.346 (9)	C5—H45A	0.9600

N2—N1	1.351 (7)	C5—H45B	0.9600
N2—H2	0.9600	C5—H45C	0.9600
C10—C7	1.381 (10)	C9—H30A	0.9600
C10—C11	1.489 (10)	C9—H30B	0.9600
C3—C4	1.387 (11)	C9—H30C	0.9600
C3—C2	1.413 (10)	C11—H47A	0.9600
C3—C6	1.494 (10)	C11—H47B	0.9600
N4—C4	1.334 (9)	C11—H47C	0.9600
N4—N3	1.369 (8)	N1—Ni2^{i}	2.013 (6)
N4—H4	0.9864		
N3—Ni2—N1^{i}	88.6 (2)	C7—C6—H39B	108.1
N3—Ni2—Cl1	164.9 (2)	H39A—C6—H39B	107.3
N1^{i}—Ni2—Cl1	88.90 (16)	C10—C7—C8	105.6 (7)
N3—Ni2—Cl2	89.5 (2)	C10—C7—C6	127.7 (7)
N1^{i}—Ni2—Cl2	174.54 (19)	C8—C7—C6	126.4 (7)
Cl1—Ni2—Cl2	91.59 (8)	C2—C1—H1C	109.5
N3—Ni2—Cl2^{ii}	100.5 (2)	C2—C1—H1A	109.5
N1^{i}—Ni2—Cl2^{ii}	100.84 (19)	H1C—C1—H1A	109.5
Cl1—Ni2—Cl2^{ii}	94.60 (8)	C2—C1—H1B	109.5
Cl2—Ni2—Cl2^{ii}	84.54 (8)	H1C—C1—H1B	109.5
Ni2—Cl2—Ni2^{ii}	95.46 (8)	H1A—C1—H1B	109.5
N1—C8—C7	109.2 (7)	N3—C2—C3	109.7 (6)
N1—C8—C9	121.4 (6)	N3—C2—C1	120.3 (7)
C7—C8—C9	129.4 (7)	C3—C2—C1	129.9 (7)
C10—N2—N1	111.6 (4)	C4—C5—H45A	109.5
C10—N2—H2	125.4	C4—C5—H45B	109.5
N1—N2—H2	120.8	H45A—C5—H45B	109.5
N2—C10—C7	107.3 (6)	C4—C5—H45C	109.5
N2—C10—C11	120.0 (7)	H45A—C5—H45C	109.5
C7—C10—C11	132.7 (8)	H45B—C5—H45C	109.5
C4—C3—C2	105.0 (7)	C8—C9—H30A	109.5
C4—C3—C6	128.1 (7)	C8—C9—H30B	109.5
C2—C3—C6	126.7 (7)	H30A—C9—H30B	109.5
C4—N4—N3	111.0 (6)	C8—C9—H30C	109.5
C4—N4—H4	124.8	H30A—C9—H30C	109.5
N3—N4—H4	123.5	H30B—C9—H30C	109.5
C2—N3—N4	106.1 (6)	C10—C11—H47A	109.5
C2—N3—Ni2	133.1 (5)	C10—C11—H47B	109.5
N4—N3—Ni2	120.0 (5)	H47A—C11—H47B	109.5
N4—C4—C3	108.0 (7)	C10—C11—H47C	109.5
N4—C4—C5	119.9 (7)	H47A—C11—H47C	109.5
C3—C4—C5	132.0 (7)	H47B—C11—H47C	109.5
C3—C6—C7	116.8 (6)	N2—N1—C8	106.2 (5)
C3—C6—H39A	108.1	N2—N1—Ni2^{i}	119.9 (3)
C7—C6—H39A	108.1	C8—N1—Ni2^{i}	133.3 (5)
C3—C6—H39B	108.1		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*, −*z*+2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···Cl1^{iii}	0.96	2.45	3.227 (6)	138
N2—H2···Cl1^{i}	0.96	2.59	3.123 (6)	116
N4—H4···Cl1^{ii}	0.99	2.19	3.167 (7)	169

Symmetry codes: (iii) *x*+1, *y*+1, *z*−1; (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*, −*z*+2.

Fig. 1

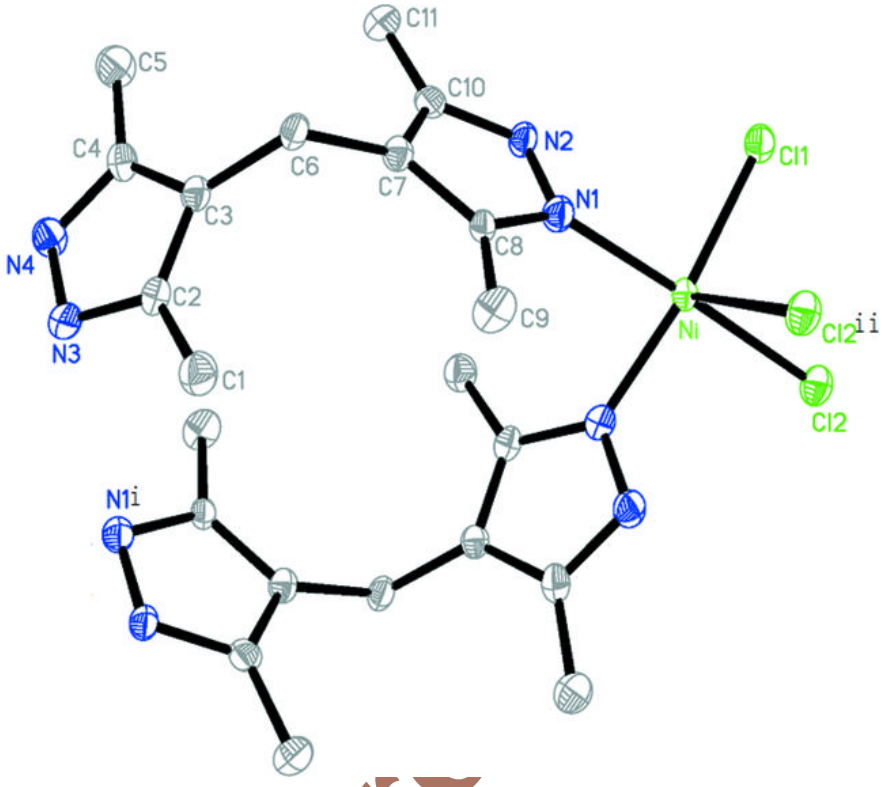


Fig. 2