Crystal structure of bis[(*N*-2,6-di-*i*-propylphenyl)-5-*t*-butyl-pyrrol-2-methylenimine-$\kappa^2 N,N'$]nickel(II), $Ni(C_{21}H_{29}N_2)_2$

Yong Xiao[I], Jianjun Yi[II], Baichun Zhu[II], Pengjuan Zhao[I] and Haijun Hao[*,I]

[I] Faculty of Science, Beijing Univeristy of Chemical Technology, P.O. Box 144, Beijing 100029, P. R. of China
[II] Key Lab for Synthetic Resin, Petrochemical Research Institute, PetroChina, Beijing 100195, P. R. China

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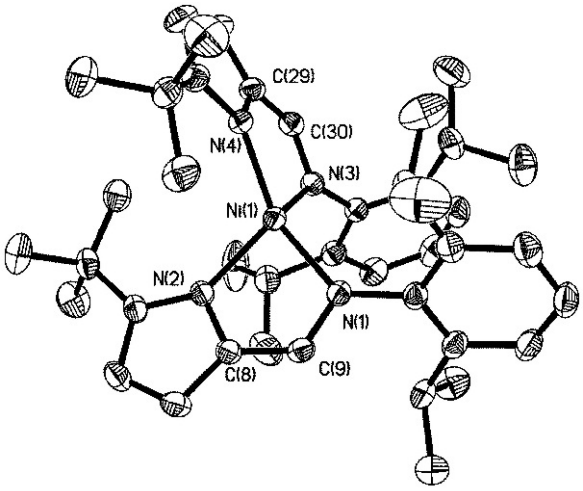

Abstract

$C_{42}H_{58}N_4Ni$, monoclinic, $P2_1/n$ (no. 14), a = 10.4521(6) Å, b = 19.979(1) Å, c = 19.368(1) Å, β = 102.325(1)°, V = 3951.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.044, $wR_{ref}(F^2)$ = 0.119, T = 298 K.

Source of material

The compound 2-(2,6-*i*-$Pr_2C_6H_3N$=CH)-5-*t*-Bu-C_4H_2NNa, made from the corresponding pyrrole and NaH, was mixed with *trans*-$Ni(PPh_3)_2PhCl$ in a molar ratio of 1:1 in a 100 ml Schlenk flask. Then 30 ml of toluene was added into the flask, the resulting suspension was stirred at room temperature for 36 h. After filtration and removal of all the volatiles from the filtrate, the residue was dissolved in 30 ml of hexane. The green hexane solution was stored at 0 °C overnight, affording dark green crystals suitable for X-ray single crystal diffraction analysis (m.p. >220 °C, decomp.). Elemental analysis — found: C, 74,77 %; H, 8.88 %; N, 8.20 %; calculated for $C_{42}H_{58}N_4Ni$: C, 74.44 %; H, 8.63 %; N, 8.27 %.

Discussion

The reaction of 2-(2,6-*i*-$Pr_2C_6H_3N$=CH)-5-*t*-Bu-C_4H_2NNa [1] with *trans*-$Ni(PPh_3)_2PhCl$ was expected to afford [2-(2,6-*i*-$Pr_2C_6H_3N$=CH)-5-*t*-Bu-C_4H_2N-$\kappa^2 N,N$]\NiPh(PPh_3). However, homoleptic [2-(2,6-*i*-$Pr_2C_6H_3N$=CH)-5-*t*-Bu-C_4H_2N-$\kappa^2 N,N$]$_2$Ni formed after work-up, which displayed a distorted tetrahedral environment of the Ni center with four coordinating N atoms at vertices. The severe deviations of four coordinating N atoms from their supposed square planar arrangement is attributed to the bulkiness of the ligand, evidenced by a previously reported [2-(2,4,6-$Me_3C_6H_2N$=CH)C_4H_3N-$\kappa^2 N,N$]$_2$Ni [2], in which the steric bulkiness of the ancillary ligand is much smaller than the ligand in the title compound.

* Correspondence author (e-mail: hjhao@mail.buct.edu.cn)

Table 1. Data collection and handling.

Crystal:	black prism, size 0.2 × 0.2 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.23 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX-CCD, ω
$2\theta_{max}$:	52.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	24338, 8073
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6142
$N(param)_{refined}$:	645
Programs:	SHELXS-97, SHELXL-97, SHELXTL [3]

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

Atom	Site	x	y	z	U_{iso}
H(41A)	4*e*	1.0671	0.6790	0.6314	0.174
H(41B)	4*e*	0.9252	0.6837	0.6455	0.174
H(41C)	4*e*	1.0410	0.6589	0.7054	0.174
H(1)	4*e*	0.824(3)	0.769(1)	0.882(1)	0.071(8)
H(2)	4*e*	0.879(3)	0.744(2)	0.813(2)	0.087(9)
H(3)	4*e*	0.826(3)	0.695(2)	0.866(1)	0.075(8)
H(4)	4*e*	0.943(3)	0.753(2)	1.006(1)	0.073(9)
H(5)	4*e*	0.938(3)	0.675(2)	0.992(2)	0.082(9)
H(6)	4*e*	1.083(3)	0.708(2)	1.022(2)	0.09(1)
H(7)	4*e*	1.163(3)	0.663(1)	0.926(2)	0.076(9)
H(8)	4*e*	1.031(3)	0.632(2)	0.889(2)	0.10(1)
H(9)	4*e*	1.096(3)	0.677(2)	0.845(2)	0.12(1)
H(10)	4*e*	1.258(3)	0.770(1)	0.993(1)	0.071(8)
H(11)	4*e*	1.342(3)	0.880(1)	0.968(1)	0.066(7)
H(12)	4*e*	1.236(2)	0.982(1)	0.869(1)	0.040(5)
H(13)	4*e*	1.409(3)	1.015(2)	0.798(2)	0.10(1)
H(14)	4*e*	1.455(3)	0.965(2)	0.749(1)	0.080(9)
H(15)	4*e*	1.424(3)	1.039(1)	0.723(1)	0.074(8)
H(16)	4*e*	1.304(3)	0.919(2)	0.637(2)	0.10(1)
H(17)	4*e*	1.276(3)	0.990(2)	0.616(2)	0.09(1)
H(18)	4*e*	1.157(4)	0.941(2)	0.619(2)	0.11(1)
H(19)	4*e*	1.241(2)	0.943(1)	0.740(1)	0.046(6)
H(20)	4*e*	1.217(3)	1.093(1)	0.657(1)	0.063(7)
H(21)	4*e*	1.078(2)	1.180(1)	0.663(1)	0.061(7)
H(22)	4*e*	0.919(2)	1.168(1)	0.730(1)	0.055(6)
H(23)	4*e*	0.890(3)	1.040(2)	0.842(2)	0.078(9)
H(24)	4*e*	0.988(4)	1.143(2)	0.888(2)	0.12(1)
H(25)	4*e*	0.837(5)	1.170(2)	0.839(3)	0.17(2)
H(26)	4*e*	0.854(4)	1.125(2)	0.901(2)	0.11(1)
H(27)	4*e*	0.717(3)	1.119(2)	0.760(2)	0.10(1)
H(28)	4*e*	0.673(3)	1.069(2)	0.803(2)	0.10(1)
H(29)	4*e*	0.726(5)	1.036(3)	0.745(3)	0.21(3)
H(30)	4*e*	0.902(3)	0.894(2)	0.960(2)	0.09(1)
H(31)	4*e*	0.871(3)	0.960(1)	0.932(2)	0.069(8)
H(32)	4*e*	0.868(3)	0.953(2)	1.008(2)	0.094(9)
H(33)	4*e*	0.643(3)	1.002(2)	0.912(2)	0.08(1)
H(34)	4*e*	0.525(4)	0.962(2)	0.927(2)	0.13(2)
H(35)	4*e*	0.628(3)	0.989(2)	0.984(2)	0.09(1)
H(36)	4*e*	0.704(4)	0.888(2)	1.043(2)	0.11(1)
H(37)	4*e*	0.590(3)	0.842(2)	0.989(2)	0.09(1)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(38)	4*e*	0.732(4)	0.823(2)	1.001(2)	0.11(1)
H(39)	4*e*	0.485(3)	0.837(1)	0.866(1)	0.075(8)
H(40)	4*e*	0.485(3)	0.802(1)	0.744(1)	0.067(7)
H(41)	4*e*	0.682(2)	0.821(1)	0.665(1)	0.060(7)
H(42)	4*e*	0.584(3)	0.916(2)	0.589(2)	0.09(1)
H(43)	4*e*	0.620(4)	0.957(2)	0.523(2)	0.14(2)
H(44)	4*e*	0.582(4)	0.995(2)	0.584(2)	0.12(1)
H(45)	4*e*	0.796(3)	1.037(2)	0.529(2)	0.11(1)
H(46)	4*e*	0.751(3)	1.070(2)	0.591(2)	0.10(1)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(47)	4*e*	0.914(4)	1.043(2)	0.601(2)	0.13(1)
H(48)	4*e*	0.777(2)	0.974(1)	0.655(1)	0.064(7)
H(49)	4*e*	0.874(3)	0.940(1)	0.495(1)	0.072(8)
H(50)	4*e*	1.009(3)	0.854(1)	0.475(1)	0.076(8)
H(51)	4*e*	1.090(3)	0.774(1)	0.562(1)	0.077(8)
H(52)	4*e*	1.003(3)	0.766(1)	0.729(1)	0.073(8)
H(53)	4*e*	1.221(5)	0.813(3)	0.721(3)	0.16(2)
H(54)	4*e*	1.216(4)	0.739(2)	0.756(2)	0.12(1)
H(55)	4*e*	1.210(4)	0.754(2)	0.678(2)	0.15(2)

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}
C(1)	4*e*	0.8745(3)	0.7342(1)	0.8656(2)	0.072(2)	0.042(1)	0.081(2)	–0.005(1)	0.014(1)	0.010(1)
C(2)	4*e*	0.9871(4)	0.7157(2)	0.9911(2)	0.110(3)	0.069(2)	0.067(2)	–0.010(2)	0.031(2)	0.015(2)
C(3)	4*e*	1.0806(4)	0.6690(2)	0.8927(2)	0.094(3)	0.053(2)	0.090(2)	0.019(2)	0.018(2)	–0.002(2)
C(4)	4*e*	1.0075(2)	0.7288(1)	0.9158(1)	0.066(2)	0.041(1)	0.048(1)	0.009(1)	0.017(1)	0.0090(9)
C(5)	4*e*	1.0919(2)	0.7907(1)	0.9168(1)	0.051(1)	0.045(1)	0.040(1)	0.0102(9)	0.0139(9)	0.0068(9)
C(6)	4*e*	1.2157(2)	0.7991(1)	0.9615(1)	0.055(1)	0.064(2)	0.055(1)	0.015(1)	0.004(1)	0.018(1)
C(7)	4*e*	1.2632(2)	0.8606(1)	0.9473(1)	0.039(1)	0.076(2)	0.053(1)	0.003(1)	0.003(1)	0.007(1)
C(8)	4*e*	1.1679(2)	0.8892(1)	0.8939(1)	0.037(1)	0.051(1)	0.038(1)	0.0023(8)	0.0074(8)	0.0029(9)
C(9)	4*e*	1.1633(2)	0.9500(1)	0.8572(1)	0.036(1)	0.048(1)	0.042(1)	–0.0067(9)	0.0083(8)	–0.0029(9)
C(10)	4*e*	1.0640(2)	1.0249(1)	0.7670(1)	0.046(1)	0.038(1)	0.041(1)	–0.0071(8)	0.0040(9)	0.0013(8)
C(11)	4*e*	1.3964(3)	1.0004(2)	0.7492(2)	0.055(2)	0.078(2)	0.080(2)	–0.001(2)	0.011(1)	0.005(2)
C(12)	4*e*	1.2467(4)	0.9543(2)	0.6415(2)	0.079(2)	0.092(2)	0.070(2)	–0.005(2)	0.027(2)	–0.012(2)
C(13)	4*e*	1.2572(2)	0.9767(1)	0.7178(1)	0.058(1)	0.050(1)	0.057(1)	–0.009(1)	0.023(1)	0.006(1)
C(14)	4*e*	1.1570(2)	1.0302(1)	0.7239(1)	0.050(1)	0.044(1)	0.046(1)	–0.0125(9)	0.0077(9)	0.0007(9)
C(15)	4*e*	1.1562(3)	1.0894(1)	0.6851(1)	0.063(2)	0.056(2)	0.061(2)	–0.017(1)	0.016(1)	0.008(1)
C(16)	4*e*	1.0703(3)	1.1404(1)	0.6877(1)	0.076(2)	0.045(1)	0.063(2)	–0.014(1)	0.005(1)	0.013(1)
C(17)	4*e*	0.9796(3)	1.1341(1)	0.7292(1)	0.071(2)	0.044(1)	0.061(1)	0.002(1)	0.003(1)	0.005(1)
C(18)	4*e*	0.9746(2)	1.0768(1)	0.7697(1)	0.056(1)	0.046(1)	0.049(1)	–0.000(1)	0.006(1)	0.003(1)
C(19)	4*e*	0.8748(3)	1.0739(2)	0.8165(2)	0.084(2)	0.061(2)	0.069(2)	0.026(1)	0.030(1)	0.019(1)
C(20)	4*e*	0.8966(6)	1.1299(3)	0.8703(2)	0.104(3)	0.192(5)	0.073(2)	0.008(3)	0.020(2)	–0.046(3)
C(21)	4*e*	0.7370(4)	1.0717(3)	0.7749(2)	0.076(2)	0.123(4)	0.110(3)	–0.020(2)	0.043(2)	–0.028(3)
C(22)	4*e*	0.8474(3)	0.9320(2)	0.9602(2)	0.072(2)	0.096(2)	0.049(2)	–0.009(2)	0.022(1)	–0.011(2)
C(23)	4*e*	0.6175(4)	0.9731(2)	0.9418(2)	0.100(3)	0.088(2)	0.072(2)	0.025(2)	0.025(2)	–0.021(2)
C(24)	4*e*	0.6783(4)	0.8625(2)	0.9987(2)	0.092(3)	0.116(3)	0.066(2)	–0.002(2)	0.033(2)	0.024(2)
C(25)	4*e*	0.7048(2)	0.9116(1)	0.9419(1)	0.058(1)	0.067(1)	0.049(1)	0.007(1)	0.025(1)	0.002(1)
C(26)	4*e*	0.6661(2)	0.8786(1)	0.8697(1)	0.048(1)	0.043(1)	0.058(1)	0.0064(9)	0.023(1)	0.004(1)
C(27)	4*e*	0.5497(2)	0.8440(1)	0.8430(2)	0.050(1)	0.061(2)	0.085(2)	–0.007(1)	0.035(1)	–0.002(1)
C(28)	4*e*	0.5511(2)	0.8246(1)	0.7750(1)	0.042(1)	0.057(1)	0.075(2)	–0.009(1)	0.015(1)	–0.010(1)
C(29)	4*e*	0.6700(2)	0.8473(1)	0.7615(1)	0.036(1)	0.041(1)	0.051(1)	–0.0018(8)	0.0073(9)	–0.0007(9)
C(30)	4*e*	0.7300(2)	0.8403(1)	0.7033(1)	0.041(1)	0.039(1)	0.044(1)	–0.0016(8)	0.0027(9)	–0.0035(9)
C(31)	4*e*	0.8994(2)	0.8606(1)	0.6439(1)	0.043(1)	0.046(1)	0.0334(9)	–0.0051(9)	0.0067(8)	–0.0022(8)
C(32)	4*e*	0.6255(3)	0.9558(2)	0.5725(3)	0.069(2)	0.068(2)	0.128(3)	0.012(2)	–0.007(2)	0.002(2)
C(33)	4*e*	0.8155(4)	1.0347(2)	0.5800(2)	0.118(3)	0.054(2)	0.069(2)	–0.006(2)	0.007(2)	0.003(1)
C(34)	4*e*	0.7703(3)	0.9681(1)	0.6049(1)	0.072(2)	0.049(1)	0.044(1)	0.005(1)	–0.000(1)	0.002(1)
C(35)	4*e*	0.8572(2)	0.9102(1)	0.5926(1)	0.054(1)	0.051(1)	0.039(1)	–0.007(1)	0.0032(9)	–0.0010(9)
C(36)	4*e*	0.9013(3)	0.9055(1)	0.5291(1)	0.077(2)	0.067(2)	0.036(1)	–0.009(1)	0.008(1)	0.007(1)
C(37)	4*e*	0.9827(3)	0.8555(2)	0.5178(1)	0.087(2)	0.079(2)	0.039(1)	–0.014(2)	0.026(1)	–0.007(1)
C(38)	4*e*	1.0281(3)	0.8090(1)	0.5696(1)	0.067(2)	0.064(2)	0.057(1)	–0.002(1)	0.026(1)	–0.008(1)
C(39)	4*e*	0.9883(2)	0.8107(1)	0.6342(1)	0.050(1)	0.053(1)	0.044(1)	–0.002(1)	0.0134(9)	–0.0018(9)
C(40)	4*e*	1.0428(3)	0.7598(1)	0.6913(1)	0.063(2)	0.056(1)	0.058(1)	0.012(1)	0.025(1)	0.004(1)
C(41)	4*e*	1.0167(5)	0.6889(2)	0.6661(2)	0.187(4)	0.062(2)	0.099(2)	–0.030(2)	0.030(3)	0.009(2)
C(42)	4*e*	1.1855(4)	0.7722(3)	0.7223(3)	0.087(3)	0.107(3)	0.149(5)	0.001(2)	–0.017(3)	0.052(3)
N(1)	4*e*	1.0600(2)	0.96559(8)	0.80881(8)	0.0399(9)	0.0383(9)	0.0401(8)	–0.0040(7)	0.0086(7)	0.0025(7)
N(2)	4*e*	1.0622(2)	0.84580(8)	0.87516(8)	0.0391(9)	0.0420(9)	0.0365(8)	0.0054(7)	0.0092(7)	0.0041(7)
N(3)	4*e*	0.8495(2)	0.86145(8)	0.70783(8)	0.0400(9)	0.0412(9)	0.0363(8)	–0.0007(7)	0.0066(7)	–0.0009(7)
N(4)	4*e*	0.7404(2)	0.88081(8)	0.81992(8)	0.0368(9)	0.0400(9)	0.0416(9)	0.0000(7)	0.0112(7)	0.0020(7)
Ni(1)	4*e*	0.92150(2)	0.89449(1)	0.80763(1)	0.0354(2)	0.0450(2)	0.0347(2)	–0.0047(1)	0.0061(1)	0.0041(1)

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References

1. Hao, H.; Bhandari, S.; Ding, Y.; Roesky, H. W.; Magull, J.; Schmidt, H.-G.; Noltemeyer, M.; Cui, C.: Pyrrolylaldiminato complexes of Zn, Mg and Al. Eur. J. Inorg. Chem. (2002) 1060-1065.
2. Anderson, C. E.; Batsanov, A. S.; Dyer, P. W.; Fawcett, J.; Howard, J. A. K.: Chelating *N*-pyrrolylphosphino-*N*′-arylaldimine ligands: synthesis, ligand behaviour and applications in catalysis. Dalton Trans. (2006) 5362-5378.
3. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.