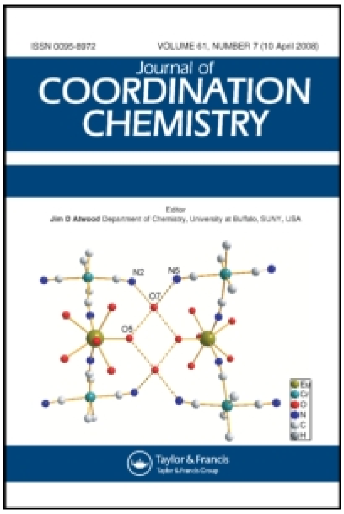

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Richard Pastorek[a]; JiŘÍ KamenÍČek[a]; Marek PavlÍČek[a]; Josef HusÁrek[a]; Zdenk ŠindelÁŘ[a]; Zdirad ŽÁk[b]
[a] Department of Inorganic and Physical Chemistry, Palacký University, Olomouc, Czech Republic [b] Department of Inorganic Chemistry, Masaryk University, Brno, Czech Republic

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DITHIOCARBAMATE COMPLEXES OF NICKEL(II) WITH 1,1′-BIS(DIPHENYLPHOSPHINO) FERROCENE

RICHARD PASTOREK[a], JIŘÍ KAMENÍČEK[a,*], MAREK PAVLÍČEK[a],
JOSEF HUSÁREK[a] ZDENĚK ŠINDELÁŘ[a] and ZDIRAD ŽÁK[b]

[a]*Department of Inorganic and Physical Chemistry, Palacký University, Křížkovského 10, 77147 Olomouc, Czech Republic;* [b]*Department of Inorganic Chemistry, Masaryk University, Kotlářská 2, 602 00 Brno, Czech Republic*

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Ni(II) complexes of composition [Ni(bz^{i}prdtc)(dppf)]X, [Ni(but$_2$dtc)(dppf)]X and [Ni(Rdtc)(dppf)]X [bz = C_7H_7; ^{i}pr = C_3H_7; but = C_4H_9; R = pld = C_4H_8; tz = C_3H_6S; hmi = C_6H_{12}; dtc = S_2CN; dppf = 1,1′-bis(diphenylphosphino)ferrocene $C_{34}H_{28}P_2Fe$; X = ClO_4, I, Br, NCS] were synthesized and characterized X-ray structural analysis of [Ni(hmidtc)(dppf)]ClO_4 confirmed coordination number four for nickel in a distorted, square-planar, NiS_2P_2 arrangement

Keywords: Ni(II)dithiocarbamate; Synthesis; X-ray structure

INTRODUCTION

The replacement of sulfur atoms in the NiS_4 chromophore by phosphorus has been recently studied [1–6]. Using 1,2-bis(diphenylphosphino)ethane (dppe) with the formation of square-planar complexes with an NiS_2P_2 chromophore except for [NiI(but$_2$dtc)(dppe)] [1] which exhibits a pentacoordinated nickel with an NiS_2P_2I chromophore. Other P,P-ligands such as 1,4-bis(diphenylphosphino)butane (dppb), 1,6-bis(diphenylphosphino)hexane (dpph) and 1,1′-bis(diphenylphosphino)ferrocene (dppf) [7–11] give diamagnetic square-planar complexes [Ni(Rdtc)(P,P)]X (Rdtc = pyrrolidine-, thiazolidine-, piperidine-, morpholinedithiocarbamate). dppb and dpph are often bound as bridging ligands (depending on R, X) and diamagnetic binuclear complexes of composition [Ni$_2$(μ-P,P)(Rdtc)$_2$X$_2$] are formed. In the case of dppf, mononuclear [Ni(Rdtc)(dppf)]X compounds were obtained (Rdtc = piperidinedithiocarbamate [10], morpholinedithiocarbamate, [11] as confirmed by X-ray structure analyses of [Ni(pipdtc)(dppf)]ClO_4 and [Ni(morfdtc)(dppf)]$ClO_4 \cdot$ EtOH). The one exception with a bridging dppf ligand was [Ni$_2$(μ-dppf)(morphdtc)$_2$](NCS)$_2$ [11]. In this work we deal

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*Corresponding author.

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with the syntheses of new complexes containing dppf and the influence of the type of dithiocarbamate and X on their structures.

EXPERIMENTAL

Materials

dppf was supplied by Fluka; all remaining reagents were obtained from Lachema and were of p.a. purity.

[Ni(dtc)(dppf)]X (X = ClO_4, I, Br, NCS)

A suspension of finely powdered $[Ni(dtc)_2]$ [12] (1 mmol) in 50 cm^3 of absolute ethanol was mixed with powdered $NiX_2 \cdot nH_2O$ (1 mmol) and dppf (1 mmol) and stirred under reflux for 5–6 h. The hot mixture was filtered and crystals were obtained during three days at room temperature. The product was filtered off, washed with ether and dried at 40°C; in the case **XI** it was possible to obtain single crystals appropriate for X-ray analysis. Yields were about 75–80%.

Physical Measurements

Nickel was determined by chelatometric titration with murexid as indicator. Chlorine and bromine were determined by the Schöniger method [13]. C, H, N, S analyses were performed on an EA 1108 instrument (Fisons). Magnetic susceptibilities were measured by the Faraday method using $Co[Hg(NCS)_4]$ as calibrant on a laboratory-designed instrument based on a Sartorius 4434 MP-8 microbalance. Conductivities were measured with an LF 330 m (WTW GmbH) at 25°C. Diffuse-reflectance electronic absorption spectra (45 000–11 000 cm^{-1}) were recorded on a Specord M 40 instrument and IR spectra (4000–400 cm^{-1}) were on a Specord M 80 (Carl Zeiss, Jena) spectrophotometer using nujol mulls. Thermal analysis was performed on a Exstar 6000, TG/DTA 6200 device (Seiko) with a heating rate of 2.5°C/min.

Crystal Structure Determination

X-ray data collection was made on a Kuma KM-4 diffractometer with graphite-monochromatized Mo$K\alpha$ radiation using an area-sensitive CCD-4 detector. The structure as solved by direct methods using SHELX-86 [14]. No absorption corrections were applied. The structure was refined anisotropically by full-matrix least-squares procedures using SHELXL-97 [15]. Additional calculations were performed using the PARST program [16].

RESULTS AND DISCUSSION

Analytical data for the complexes are given in Table I; important properties are summarized in Table II. In accordance with the assumption of a square-planar arrangement of the NiS_2P_2 chromophore, all compounds are diamagnetic and 1 : 1 electrolytes [17].

TABLE I Analytical data for the complexes

		Found(calcd.)%					
		Ni	*C*	*H*	*N*	*S*	*X*[a]
I	$[Ni(bz^iprdtc)(dppf)]ClO_4$	6.7(6.3)	57.7(57.7)	4.5(4.5)	1.5(1.5)	6.6(6.8)	3.2(3.8)
II	$[Ni(bz^iprdtc)(dppf)]I$	6.0(6.1)	56.4(56.1)	4.8(4.4)	1.2(1.4)	6.4(6.6)	12.8(13.2)
III	$[Ni(bz^iprdtc)(dppf)]NCS$	6.8(6.6)	62.0(61.7)	5.0(4.7)	3.1(3.1)	11.1(10.7)	–
IV	$[Ni(but_2dtc)(dppf)]ClO_4 \cdot H_2O$	6.3(6.3)	55.1(55.2)	5.5(5.2)	1.7(1.5)	7.0(6.9)	3.3(3.8)
V	$[Ni(but_2dtc)(dppf)]I$	6.3(6.2)	54.3(54.7)	5.1(4.9)	1.6(1.5)	7.3(7.0)	13.0(13.4)
VI	$[Ni(plddtc)(dppf)]ClO_4$	7.0(6.8)	54.0(54.5)	4.0(4.2)	1.2(1.6)	7.2(7.4)	3.9(4.1)
VII	$[Ni(plddtc)(dppf)]I$	7.0(6.5)	53.6(53.4)	4.2(4.2)	1.6(1.6)	6.7(7.1)	13.5(14.1)
VIII	$[Ni(plddtc)(dppf)]Br \cdot H_2O$	6.8(6.9)	54.7(54.6)	4.4(4.5)	1.7(1.6)	6.9(7.5)	9.9(9.3)
IX	$[Ni(tzdtc)(dppf)]ClO_4$	6.5(6.7)	51.8(52.2)	3.8(3.7)	1.6(1.6)	10.6(11.0)	3.8(4.0)
X	$[Ni(tzdtc)(dppf)]I$	6.4(6.5)	50.9(50.6)	4.1(3.6)	1.1(1.5)	10.1(10.6)	13.6(14.0)
XI	$[Ni(hmidtc)(dppf)]ClO_4$	6.8(6.6)	55.8(55.5)	4.4(4.5)	1.4(1.6)	7.2(7.2)	3.6(4.0)
XII	$[Ni(hmidtc)(dppf)]I$	6.7(6.4)	53.2(53.9)	4.4(4.4)	1.1(1.5)	6.4(7.0)	14.3(13.9)

[a] X = Cl, Br, I.

TABLE II Selected properties of the complexes

	Color	λ_M[a]	*IR* (cm^{-1})[b]				*Max*[b] ($10^3 cm^{-1}$)	
		($S cm^2 mol^{-1}$)	$\nu(C\overset{\cdots}{-}S)$	$\nu(C\overset{\cdots}{-}N)$	$\nu_3(ClO_4)$	$\nu_4(ClO_4)$		
I	Purple	143	990 m	1530 m	1084 vs	625 m	19.7	31.4
II	Purple	124	990 m	1530 w			20.2	31.4
III	Pink	117	995 w	1527 w			21.0	29.0
IV	Purple	118	990 s	1526 s	1080 vs	622 m	20.0	31.2
V	Dark purple	124	995 m	1520 m			19.8	30.8
VI	Dark red	143	990 m	1534 vs	1086 vs	618 m	19.8	31.0
VII	Violet	112	992 w	1532 m			19.8	31.4
VIII	Dark violet	116	990 m	1526 m			19.6	31.8
IX	Dark violet	128	988 m	1530 w	1085 m	622 m	19.2	31.3
X	Violet	99	992 m	1532 w			19.8	30.8
XI	Dark yellow	141	989 m	1530 m			18.7	29.7
XII	Dark violet	129	990 m	1518 m			19.0	31.6

[a] In acetone solution; $[Ni^{2+}] = 10^{-3}$ mol dm^{-3}; [b] Maxima in nujol.

The ionic nature of the X anion was supported by IR spectra; complexes with $X = ClO_4$ exhibit non-split maxima (ν_3 near 1085 cm^{-1} and ν_4 at 620 cm^{-1}) [18]. For complex **III** (X = NCS) recorded maxima $\nu(C \equiv N)$ at 2092 cm^{-1} and ν(C–S) at 740 cm^{-1} are typical of ionic NCS [19]. Maxima typical for dithiocarbamates [21,22] for $\nu(C\overset{\cdots}{-}N)$ at 1520–1534 cm^{-1} and $\nu(C\overset{\cdots}{-}S)$ at 988–995 cm^{-1} were observed.

Electronic spectra are in good accordance with the assumption of square-planar coordination. Maxima between 18 700 and 21 000 cm^{-1} for all complexes can be attributed to the $^1A_{1g} \rightarrow {}^1B_{1g}$ transition [20]. Maxima at 30 000 cm^{-1} and more can be assigned to intraligand transitions in the S_2CN group [21,22].

Thermal analyses of compounds with ClO_4 were not performed for safety reasons. Decomposition of the remaining complexes starts at 60°C and continues with no thermally stable intermediates; only in the case of $[Ni(plddtc)(dppf)]Br \cdot H_2O$ appears a small endo-effect (80°C) and a plateau in the range 110–230°C, corresponding to water loss ($\Delta M_{calc/found} = 2.1/2.4\%$). Decomposition of complexes is not complete at 1000°C.

The X-ray structure of $[Ni(hmidtc)(dppf)]ClO_4$ (**XI**) has been solved (Fig. 1). Basic crystallographic parameters are listed in Tables III–VI, confirming distorted

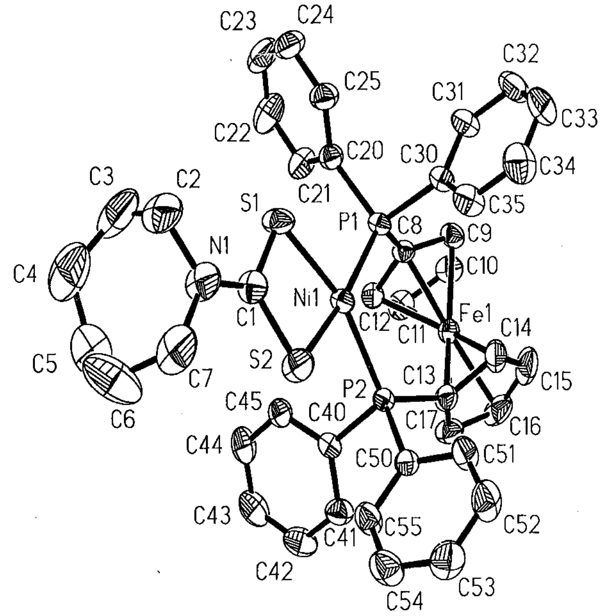

FIGURE 1 Drawing of the [Ni(hmidtc)(dppf)]$^+$ cation with the atom labeling scheme. Thermal ellipsoids are drawn at the 40% probability level; the ClO_4^- group and hydrogen atoms are omitted for clarity.

TABLE III Crystal data and structure refinement details for [Ni(hmidtc)(dppf)]ClO_4

Empirical formula	$C_{41}H_{40}ClFeNNiO_4P_2S_2$
Formula weight	886.81
Temperature	293(2)K
Wavelength	0.71073 Å
Crystal system, space group	triclinic, $P\bar{1}$
Unit cell dimensions	$a = 10.915(2)$ Å
	$b = 11.679(2)$ Å
	$c = 17.505(4)$ Å
	$\alpha = 73.01(3)°$
	$\beta = 81.27(3)°$
	$\gamma = 69.12(3)°$
Volume	1991.1(7) Å^3
z, calculated density	2, 1.479 Mg m^{-3}
Absorption coefficient	1.132 mm^{-1}
$F(000)$	916
Crystal size	0.40 × 0.25 × 0.25 mm
Theta range for data collection	3.57–29.14°
Index ranges	$-13 \leq h \leq 14$
	$-10 \leq k \leq 15$
	$-23 \leq l \leq 23$
Reflections collected/unique	25090/9593 [R(int) = 0.0355]
Completeness to $2\theta = 29.14$	89.5%
Max. and min. transmission	0.7651 and 0.6602
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	9593/0/532
Goodness-of-fit on F^2	0.998
Final R indices ($I > 2\sigma(I)$]	$R1 = 0.0417$, $wR2 = 0.1076$
R indices (all data)	$R1 = 0.0512$, $wR2 = 0.1145$
Largest diff. peak and hole	0.441 and -0.694 e Å^{-3}

TABLE IV Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for **XI**. U(eq) are defined as one-third of the trace of the orthogonalized U_{ij} tensor

Atom	x/a	y/b	z/c	U(eq)
Ni(1)	3292(1)	7867(1)	1933(1)	33(1)
S(1)	5235(1)	7118(1)	1339(1)	54(1)
S(2)	2975(1)	9024(1)	678(1)	54(1)
C(1)	4554(3)	8132(3)	465(2)	48(1)
N(1)	5129(3)	8195(3)	−251(1)	64(1)
C(2)	6454(3)	7329(5)	−380(2)	88(1)
C(3)	6450(5)	6067(5)	−403(3)	117(2)
C(4)	5758(6)	6045(6)	−1049(3)	120(2)
C(5)	4367(6)	6876(6)	−1137(4)	120(2)
C(6)	4214(6)	8233(6)	−1469(4)	144(3)
C(7)	4426(4)	9002(4)	−969(2)	89(1)
Fe(1)	1121(1)	7157(1)	4134(1)	35(1)
C(8)	2978(2)	6144(2)	3883(1)	33(1)
C(9)	2786(2)	6126(2)	4719(1)	42(1)
C(10)	1799(3)	5572(3)	5057(2)	49(1)
C(11)	1378(3)	5236(3)	4451(2)	47(1)
C(12)	2089(2)	5587(2)	3729(1)	37(1)
C(13)	459(2)	8669(2)	3209(1)	39(1)
C(14)	966(3)	9014(2)	3785(2)	47(1)
C(15)	214(3)	8772(3)	4519(2)	62(1)
C(16)	−734(3)	8281(3)	4409(2)	62(1)
C(17)	−609(2)	8218(3)	3605(2)	50(1)
P(1)	4058(1)	6716(1)	3122(1)	31(1)
P(2)	1149(1)	8659(1)	2211(1)	31(1)
C(20)	5435(2)	5253(2)	3121(1)	39(1)
C(21)	5224(3)	4101(3)	3278(2)	55(1)
C(22)	6279(4)	2984(3)	3304(2)	77(1)
C(23)	7536(4)	3016(4)	3170(2)	77(1)
C(24)	7759(3)	4152(4)	2995(2)	68(1)
C(25)	6717(2)	5275(3)	2962(2)	53(1)
C(30)	4708(2)	7616(2)	3541(1)	37(1)
C(31)	5412(3)	7031(3)	4233(2)	50(1)
C(32)	5892(3)	7716(3)	4564(2)	64(1)
C(33)	5678(4)	8987(4)	4201(2)	71(1)
C(34)	4990(4)	9574(3)	3525(2)	71(1)
C(35)	4498(3)	8897(3)	3190(2)	52(1)
C(40)	323(2)	7802(2)	1877(1)	35(1)
C(41)	−1040(2)	8270(3)	1816(2)	49(1)
C(42)	−1662(3)	7580(3)	1592(2)	63(1)
C(43)	−953(3)	6428(3)	1440(2)	63(1)
C(44)	383(3)	5948(3)	1500(2)	55(1)
C(45)	1026(3)	6643(2)	1712(1)	41(1)
C(50)	458(2)	10307(2)	1639(1)	35(1)
C(51)	546(3)	11257(2)	1932(2)	47(1)
C(52)	106(3)	12512(3)	1497(2)	60(1)
C(53)	−428(3)	12833(3)	769(2)	62(1)
C(54)	−520(3)	11895(3)	475(2)	61(1)
C(55)	−84(3)	10637(3)	906(2)	50(1)
Cl(1)	1983(1)	12576(1)	3251(1)	59(1)
O(2)	2892(3)	12519(3)	3757(2)	109(1)
O(1A)	1491(17)	11597(10)	3651(8)	144(6)
O(1B)	1159(15)	11875(15)	3586(7)	143(6)
O(3A)	2413(11)	12566(9)	2521(6)	103(3)
O(3B)	2710(9)	11851(8)	2622(6)	92(3)
O(4A)	889(6)	13750(9)	3274(6)	99(3)
O(4B)	1412(18)	13758(9)	2837(8)	217(9)

square-planar coordination around the central nickel atom. Ni–S and Ni–P bonds are not equal and bond angles differ significantly from 90°C. Deviations of atoms from the NiS_2P_2 plane are Ni(1): 0.013(2), S(1): 0.198(6), S(2): $-0.182(7)$, P(1): $-0.054(4)$, P(2): 0.083(4) Å. Significant shortening of bond lengths in the S_2CN group (see Table V) was found. Common bond distances are [24] C–N 1.47 Å, C–S 1.81 Å. This can be explained by significant π-interaction in these bonds. It was confirmed the perchlorate group is not coordinated (interatomic Ni–Cl distance is 6.20 Å for **XI**), in accordance with other results above. The Ni–Fe distance (4.23 Å for **XI**) is too long for magnetic interaction. Five possible hydrogen bonds for **XI** were found and are listed in Table VI.

We conclude that the structures of [Ni(dtc)(dppf)]X complexes are in good accord with previous measurements (see Table VII) and that the type of dithiocarbamate and anion has no significant influence on the structure.

TABLE V Selected bondlengths [Å] and angles [°] for [Ni(hmidtc) (dppf)]ClO_4

Bond		*Angle*	
Ni(1)–S(1)	2.2102(10)	S(1)–Ni(1)–P(1)	90.90(4)
Ni(1)–P(1)	2.2196(11)	S(1)–Ni(1)–S(2)	78.28(4)
Ni(1)–S(2)	2.2216(11)	P(1)–Ni(1)–S(2)	167.35(3)
Ni(1)–P(2)	2.2222(9)	S(1)–Ni(1)–P(2)	164.25(3)
S(1)–C(1)	1.719(3)	P(1)–Ni(1)–P(2)	102.83(4)
S(2)–C(1)	1.717(3)	S(2)–Ni(1)–P(2)	88.83(4)
C(1)–N(1)	1.308(3)	C(1)–S(1)–Ni(1)	86.42(10)
N(1)–C(7)	1.463(4)	C(1)–S(2)–Ni(1)	86.11(10)
N(1)–C(2)	1.468(4)	N(1)–C(1)–S(2)	125.2(2)
C(8)–P(1)	1.782(2)	N(1)–C(1)–S(1)	125.7(2)
C(13)–P(2)	1.795(2)	S(2)–C(1)–S(1)	109.02(14)
P(1)–C(20)	1.830(3)	C(1)–N(1)–C(7)	121.8(3)
P(1)–C(30)	1.813(2)	C(1)–N(1)–C(2)	121.3(3)
P(2)–C(40)	1.817(2)	C(8)–P(1)–Ni(1)	119.00(8)
P(2)–C(50)	1.830(2)	C(30)–P(1)–C(20)	103.90(11)
		C(13)–P(2)–C(50)	103.83(11)
		C(40)–P(2)–C(50)	104.67(10)

TABLE VI Possible hydrogen bonds for [Ni(hmidtc)(dppf)]ClO_4

Donor-H	*Donor . . . Acceptor*	*H. . .Acceptor*	*Donor-H . . . Acceptor*
C2–H2B 0.876(0.051) 1.080	C2 . . . S1 (0) 3.078 (0.026)	H2B . . . S1 (0) 2.813 (0.007) 2.787	C2–H2B . . . S1 (0) 99.25(0.56) 95.11 (**)
C3–H3A 1.060(0.035) 1.080	C3 . . . S1 (0) 3.516 (0.015)	H3A . . . S1 (0) 2.843 (0.002) 2.833	C3–H3A . . . S1 (0) 121.57(0.40) 121.23 (**)
C5–H5A 1.114(0.035) 1.080	C5 . . . N1 (0) 2.858 (0.057)	H5A . . . N1 (0) 2.698 (0.036) 2.696	C5–H5A . . . N1 (0) 86.64(0.59) 87.37 (**)
C32–H32 0.995(0.034) 1.080	C32 . . . O2 (5) 3.305 (0.030)	H32 . . . O2 (5) 2.497 (0.088) 2.435	C32–H32 . . . O2 (5) 138.11(0.42) 136.77 (**)
C42–H42 0.998(0.043) 1.080	C42 . . . S1 (6) 3.724 (0.047)	H42 . . . S1 (6) 2.925 (0.092) 2.865	C42–H42 . . . S1 (6) 137.59(1.86) 136.48 (**)

(**) Values normalized following G.A. Jeffrey and L. Lewis (1978). *Carbohydr. Res.*, **60**, 179; R. Taylor and O. Kennard (1983). *Acta Cryst.*, **B39**, 133; equivalent positions are (0): x, y, z; (1): $-x+1, -y+2, -z$; (2): $-x, -y+2, -z+1$; (3): $x, +y-1, +z$; (4): $x+1, +y-1, +z$; (5): $-x+1, -y+2, -z+1$; (6): $x-1, +y, +z$.

TABLE VII Comparison of important bondlengths (Å) and angles (°) for some [Ni(dtc)(dppf)]X complexes

Bond/angle	*Complex*[a]			
	A	*B*	*C*	*D*
Ni–S1	2.2260(13)	2.227(2)	2.2060(15)	2.2102(10)
Ni–S2	2.2161(13)	2.210(2)	2.2199(14)	2.2216(11)
Ni–P1	2.2279(13)	2.219(2)	2.1991(15)	2.2196(11)
Ni–P2	2.2164(13)	2.209(2)	2.2180(15)	2.2222(9)
S1–C	1.733(5)	1.715(8)	1.723(5)	1.719(3)
S2–C	1.727(4)	1.690(9)	1.703(5)	1.717(3)
C–N	1.301(8)	1.325(10)	1.315(6)	1.308(3)
P1–Ni–S1	90.32(5)	92.10(9)	91.70(5)	90.90(4)
P1–Ni–P2	99.11(5)	99.85(8)	98.70(2)	102.83(4)
S2–Ni–P2	91.78(5)	89.83(9)	91.07(5)	88.83(4)
S1–Ni–S2	78.75(5)	78.80(8)	78.75(5)	78.28(4)

[a] A: [Ni(pipdtc)(dppf)]ClO_4; [10]; B: [Ni(tzdtc)(dppf)]I · C_2H_5OH; [25]; C: [Ni(morphdtc)(dppf)]$ClO_4 \cdot C_2H_5OH$; [11]; D: [Ni(hmidtc)(dppf)]ClO_4.

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Supplementary Data

Full lists of crystallographic data are available from the authors upon request.

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