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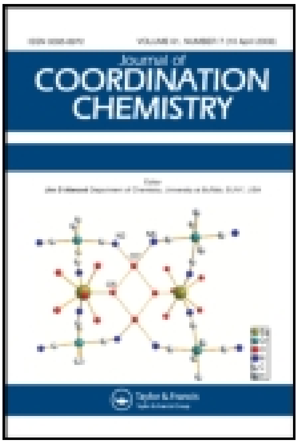

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Synthesis and characterization of nitrogen-containing Lewis base adducts of bis(*O*,*O*′-dialkylmonoselenophosphato) cobalt(II) complexes

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The adducts of bis(*O*,*O*′-dialkylmonoselenophosphato)cobalt(II) complexes, $Co\{O(Se)P(OR)_2\}_2(L)_4$ (where R = n-Pr, i-Pr; $L = C_5H_5N$, NC_5H_4Me-2, NC_5H_4Me-3), were synthesized by *in situ* reactions of $CoCl_2 \cdot 6H_2O$, Lewis base, and $NaO(Se)P(OR)_2$. The single crystal structure of $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$ shows distorted octahedral geometry around cobalt(II) and monoselenophosphates are *trans*. The CoN_4 forms a square plane. These bis(*O*,*O*′-dialkylmonoselenophosphato)cobalt(II) adducts were characterized by elemental analyses, spectroscopic techniques (UV-Vis, infrared, ^1H and ^{31}P), and magnetic moment measurements.

Keywords: Monoselenophosphate; Lewis base adducts; Bis(*O*,*O*′-dialkylmonoselenophosphato) cobalt(II); Monodentate coordination

1. Introduction

Thiophosphate ligands, namely dithiophosphate and monothiophosphate are commercially available and well-characterized [1–3]. Less attention has been paid to the coordination chemistry of monoselenophosphates. Preliminary studies [4] have been carried out on the reactions of di-isopropylphosphoroselenoate with V(IV), Cr(III), Mn(II), Fe(III), Co(II), Co(III), Ni(II), and Pd(II). The nickel and cobalt complexes were unstable due to rapid loss of selenium. Other studies include the synthesis of complexes such as $SiMe_3L$, $GePh_3L$, $SnPh_3L$, $SnMe_3L$, $SnPh_2L_2$, $SnPhL_3$, and AsL_3 ($L = SeOP(OPr^i)_2$) by the reaction of sodium *O*,*O*-di-isopropyl phosphoroselenoate and halides of silicon, germanium, and tin [5]. Monoselenophosphates are mixed chalchogeno ligands and intermediate to diseleno and dioxaphosphate derivatives. *O*,*O*′-dialkyl monoselenophosphoric acids show equilibrium between two forms

$$(RO)_2P(Se)OH \leftrightarrows (RO)_2P(O)SeH.$$

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Dialkyl monoselenophosphates have interesting structural possibilities, such as monodentate (through oxygen/selenium) and bidentate [5]. Attempts to synthesize dialkylmonoselenophosphate complexes with cobalt were unsuccessful. Various methods were adopted for this reaction, such as carrying out the reaction at different temperatures and using various reaction solvents, but all failed. Adducts were synthesized by the *in situ* reaction of $CoCl_2 \cdot 6H_2O$, Lewis bases, and monoselenophosphate. The adduct $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$ represents a rare monoseleno adduct of a transition metal.

Herewith, we report the synthesis and characterization of Lewis base adducts of bis(*O*,*O*′-dialkylmonoselenophosphato)cobalt(II) complexes (**1–6**) and molecular structure of $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$ (**4**). The structure of **4** shows a distorted octahedral geometry in which two oxygens of monoseleno groups form the axial plane and four pyridine nitrogens are in the equatorial positions.

2. Results and discussion

Reactions of cobalt chloride hexahydrate with the sodium salt of *O*,*O*′-dialkyl monoselenophosphates were carried out in various solvents and at varied temperatures, but failed. An indirect method was used to synthesize adducts of bis(*O*,*O*′-dialkylmonoselenophosphato)cobalt(II) (**1–6**). In this *in situ* reaction first we synthesized $Co(L)_4Cl_2$ by the reaction of cobalt chloride hexahydrate and Lewis base and in the second step the sodium salt of *O*,*O*′-dialkyl monoselenophosphate was added. This route readily produced the expected compounds in good yields.

$$CoCl_2 \cdot 6H_2O + 4L \rightarrow Co(L)_4Cl_2$$
$$C(L)_4CL_2 + 2NaO(Se)P(OR)_2 \rightarrow Co\{O(Se)P(OR)_2(L)_4 + NaCl$$

(R = n-Pr, i-Pr; L = pyridine, 2-picoline, 3-picoline).

These reactions were quite facile, but to ensure completion the reaction solution was refluxed for 1 h. Blue solution was obtained and dried under vacuum. Various methods were applied for recrystallization, but finally we obtained crystals from benzene : hexane (1 : 1). During recrystallization selenium appeared at the bottom of the recrystallization pot due to dissociation. In some cases the dissociation occurred in the solid phase. These adducts were characterized by elemental analysis, infrared (IR), UV-Vis, and magnetic susceptibility measurements. The molecular structure of $Co\{O(S)P(O^iPr)_2\}_2(C_5H_5N)_4$ was determined by single-crystal X-ray diffraction.

2.1. *Spectroscopic characterization*

2.1.1. IR spectra. IR band assignments were done by comparison with $NaO(Se)P(OR)_2$ and adducts of bis(*O*,*O*′-dialkylmonothiophosphato)cobalt(II) [3]. The ν(P–O) are found at 1042–1068 cm^{-1}, showing lower wave number shifts by 40–50 cm^{-1}, suggesting coordination of monoselenophosphate through oxygen. The ν(P = Se) at 585–563 cm^{-1} have almost the same frequencies as $NaO(Se)P(OR)_2$, suggesting that P = Se does not participate in coordination. The ν[(P)–O–C] and

ν[P–O–(C)] are observed at 990–976 and 762–750 cm^{-1}, respectively. Medium intensity bands at 452–470 cm^{-1} are assigned to ν(Co–O). Bands at 1606–1572 cm^{-1} may be assigned to ν(C–N) of the Lewis base.

2.1.2. Electronic absorption spectra. In electronic absorption spectra of **1–6** three peaks at 18691–21598, 11520–12787, and 9708–10869 cm^{-1} are assigned for $^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)$, $^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$, and $^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ transitions, respectively. Similar electronic absorption pattern was observed for octahedral adducts of bis(*O*,*O*-dialkylmonothiophosphato)cobalt(II) complexes [3], suggesting octahedral geometry.

2.1.3. ^{1}H NMR spectra. ^{1}H NMR spectra of some representative adducts (**1** and **4**) were determined. These paramagnetic adducts show large downfield shifting for pyridine protons [6, 7], explained on the basis of unpaired delocalization from the metal ion to the σ-orbital of pyridine. The alkyl protons show slight shifting in these complexes.

2.1.4. ^{31}P NMR spectra. ^{31}P NMR spectra yield the chemical shift δ P and the coupling constant $^1J(^{31}P\text{-}^{77}Se)$ arising from ^{77}Se (spin = ½; abundance 7.58%); the data recorded in section 3 enable structure deductions. These cobalt(II) adducts (**1–6**) show a single peak in the range 63.4–58.3 ppm, suggesting O-bonded selenone structure. These cobalt(II) adducts have ^1J (PSe) of 860–872 Hz, also suggesting O-bonded selenone structure.

2.2. *Magnetic moment measurements*

The octahedral geometry of **1–6** is also supported by magnetic susceptibility values. The magnetic susceptibility values of **1–6** are 4.9–5.2 B.M. at 298 K, consistent with high spin octahedral complexes. The magnetic susceptibility values are much higher than the spin only value of 3.8 B.M. due to the orbital moment contribution [8].

The structure of the following type is suggested for these newly synthesized complexes (figure 1).

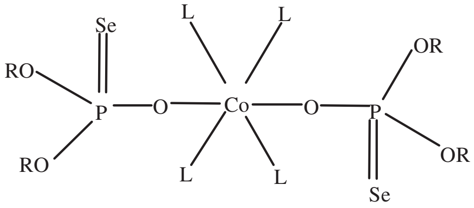

Figure 1. Proposed distorted octahedral geometry for Co{O(Se)P(OR)$_2$(L)$_4$ (where R = n-Pr, i-Pr; L = C_5H_5N, NC_5H_4Me-2, NC_5H_4Me-3).

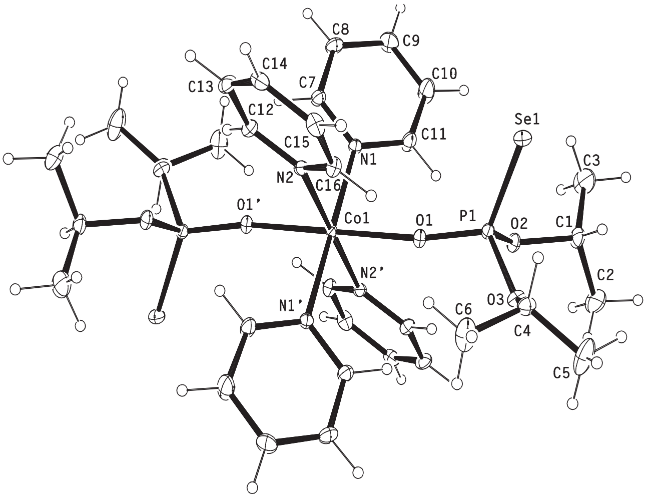

Figure 2. ORTEP plot of $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$.

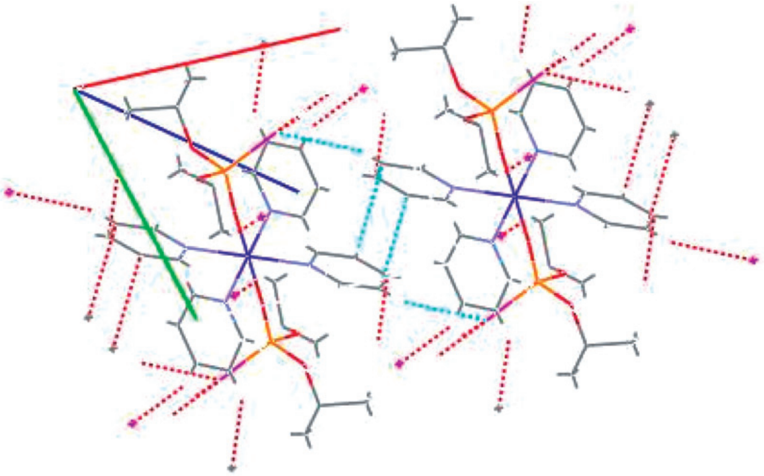

Figure 3. View of the crystal packing for $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$.

2.3. *Crystal structure of $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$*

The molecular structure of $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$ is shown in figure 2. Figure 3 shows perspective views of the crystal packing in the unit cell of $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$. Crystallographic data and refinement parameters are presented in table 1. Bond distances and angles are listed in table 2. $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$ (**4**) was crystallized as triclinic in the space group $P\bar{1}$; **4** has distorted octahedral geometry with CoO_2N_4 chromophore. The monodentate monoselenophosphates are coordinated through oxygen with cobalt(II), probably due to strong donor ability of oxygen and large atomic size of selenium. The CoN_4 chromophore forms the square plane and the O(1)–Co–O(1) plane is perpendicular to

Table 1. Crystal data and structure refinement for $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$.

Empirical formula	$C_{16}H_{22}N_2O_3PSeCo0.5$
Formula weight	429.75
Temperature (K)	150(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions (Å, °)	
a	9.1302(4)
b	10.2168(4)
c	11.9619(5)
α	88.533(2)
β	71.534(2)
γ	69.103(2)
Volume (Å^3), Z	983.94(7), 2
Calculated density ($Mg\,m^{-3}$)	1.457
Absorption coefficient (mm^{-1})	2.411
$F(000)$	441
Crystal size (mm^3)	$0.20 \times 0.08 \times 0.01$
Theta range for data collection (°)	2.53–30.68
Index ranges	$-13 \le h \le 12$; $-14 \le k \le 13$; $-17 \le l \le 16$
Reflections collected	17,454
Independent reflections	5156 [$R_{int} = 0.0253$]
Max. and min. transmission	0.9763 and 0.6442
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	5156/0/218
Goodness-of-fit on F^2	1.029
Final R indices [$F^2 > 4\sigma(F^2)$]	$R_1 = 0.0315$, $wR_2 = 0.0673$
R indices (all data)	$R_1 = 0.0491$, $wR_2 = 0.0729$
Largest difference peak and hole (e Å^{-3})	0.593 and −0.459

Table 2. Bond lengths (Å) and angles (°) for $Co[O(Se)P(O^iPr)_2]_2(C_5H_5N)_4$.

Co(1)–O(1)	2.083(1)	Co(1)–N(1)	2.173(2)
Co(1)–N(2)	2.187(2)	Se(1)–P(1)	2.1105(5)
P(1)–O(1)	1.494(1)	P(1)–O(2)	1.589(2)
P(1)–O(3)	1.601(2)	O(2)–C(1)	1.462(2)
N(1)–Co(1)–N(2)′	87.35(6)	N(1)′–Co(1)–N(2)	87.35(6)
O(1)–Co(1)–N(1)′	88.02(6)	O(1)–Co(1)–N(2)′	89.47(6)
O(1)–Co(1)–N(2)	90.53(6)	O(1)–Co(1)–N(1)	91.98(6)
N(1)–Co(1)–N(2)	92.65(6)	N(1)′–Co(1)–N(2)′	92.65(6)
O(1)–Co(1)–O(1)′	180.00	N(1)–Co(1)–N(1)′	180.00
N(2)′–Co(1)–N(2)	180.00	Co(1)–O(1)–P(1)	163.85(9)
O(1)–P(1)–O(2)	105.05(8)	O(1)–P(1)–Se(1)	117.53(6)
O(1)–P(1)–O(3)	109.35(8)	O(2)–P(1)–Se(1)	112.36(6)

the CoN_4 plane with dihedral angle between these two planes of 89.59°. The two monoselenophosphate ligands are *trans* with O–Co–O bond angle 180°. The Co–O bond distance (2.083(1) Å) is slightly more than $Co\{S(O)P(O^iPr)_2\}_2(C_5H_5N)_4$ (2.0757(8) Å) [3]. The P–Se bond distance is 2.1105(5) Å, which is higher than the P–S bond length $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$ (1.956(4) Å) [3]. The P–Se bond distance suggests the double bond character. The Co–Se distance (4.8429(3) Å) is longer than Cu–(μ_2-Se) bond distances in $[Cu_8(\mu_8\text{-}X)\{Se_2P(OR)_2\}_6](PF_6)$ (X = Cl, R = Et, Pr^n, Pr^i)

(2.365–2.408 Å)] [9], where Cu–Se bond formed, but comparable with the Co–S distance in Co{O(Se)P(O^{i}Pr)$_2$}$_2$(C$_5$H$_5$N)$_4$ (4.397(1) Å) [3]. The average Co–N bond distance (2.18(2) Å) is quite similar to Co{S(O)P(O^{i}Pr)$_2$}$_2$(C$_5$H$_5$N)$_4$ (2.18(9) Å). The cobalt–cobalt bond distances are shorter than expected, due to short interactions between the aromatic rings. The N(1)–Co–N(1) and N(2)–Co–N(2) bond angles are 180°. The N–Co–N bite angles are 87.35(6)°, suggesting distortion in the square plane of Co{O(Se)P(O^{i}Pr)$_2$}$_2$(C$_5$H$_5$N)$_4$ (average 90(3)°). The N(1)–Co–O(1) and N(2)–Co–O(1) angles are 88.02(6) and 89.47(6)°, respectively. The phosphorus is tetrahedral and the angles O(2)–P–O(3), O(1)–P–O(2), O(1)–P–Se(1), and O(3)–P–Se(1) are 99.57(8)°, 105.05(8)°, 117.53(6)°, and 111.34(6)°, respectively.

3. Experimental

Solvents (benzene, hexane, methanol, ethanol, 1-propanol, 2-propanol, diethyl ether, and carbon tetrachloride) were dried by standard method before use. All the reactions were done in anhydrous conditions using pre-dried glassware. Sodium salts of *O,O′*-dialkyl monothiophosphoric acid were prepared by the reported method [10]. Micro elemental analysis (C, H, and N) was carried out on a Vario EL elemental analyzer. Electronic spectra were recorded on a Perkin-Elmer UV-Vis-NIR spectrometer in pyridine, 2-picoline and 3-picoline. IR spectra were recorded on a Perkin-Elmer 983 G spectrometer as Nujol mulls between CsI discs from 4000 to 180 cm^{-1}.

3.1. *Synthesis of adducts of bis(O,O′-dialkylmonoselenophosphato)cobalt(II) complexes (1–6)*

3.1.1. Co{O(Se)P(O^{n}Pr)$_2$}$_2$(C$_5$H$_5$N)$_4$ (1). Typically, reaction of CoCl$_2 \cdot$6H$_2$O (0.185 g, 0.78 mmol) and pyridine (0.246 g, 3.11 mmol) was carried out in methanol (20 mL), followed by the addition of ethanol solution of NaSe(O)P(O^{n}Pr)$_2$ (0.415 g, 1.55 mmol). The resultant reaction mixture was refluxed for 1 h. Blue solution was obtained and solvent was removed by vacuum. Blue; Yield: 0.550 g (88%); Anal. Calcd for C$_{32}$H$_{48}$Se$_2$P$_2$O$_6$N$_4$Co (%): C, 44.5; H, 5.6; N, 6.5. Found (%): C, 44.5; H, 5.6; N, 6.4. UV-Vis spectral data [λ_{max} (cm^{-1}); ε(L mol^{-1} cm^{-1})] in 0.004 mol L^{-1} pyridine solution 18691 (138), 11520 (142), 9708 (73); IR (cm^{-1}) 1598m, 1572m ν(C–N), 976s ν[(P)–O–C], 760s ν[P–O–(C)], 585s ν(P–Se), 520 ν(Co–O); ^{1}H NMR (CDCl$_3$, δ ppm): 0.90–1.10 (t, 12H, CH$_3$), 1.32–1.38 (m, 8H, CH$_2$) 2.82–2.94 (t, 8H, CH$_2$), 22.3 (*o*-Py), 16.6 (*m*-Py), 9.9 (*p*-Py); ^{31}P NMR (CDCl$_3$, δ ppm): 63.8 (s); μ_{eff} 4.9 B.M.

3.1.2. Co{O(Se)P(O^{n}Pr)$_2$}$_2$(NC$_5$H$_4$Me-2)$_4$ (2). Prepared the same as **1**. Blue; Yield: 0.545 g (85%); Anal. Calcd for C$_{36}$H$_{56}$Se$_2$P$_2$O$_6$N$_4$Co (%): C, 47.0; H, 6.1; N, 6.1. Found (%): C, 47.0; H, 6.1; N, 6.0. UV-Vis spectral data [λ_{max} (cm^{-1}); ε(L mol^{-1} cm^{-1})] in 0.005 mol L^{-1} 2-picoline solution 20958 (172), 11734 (102), 10226 (120); IR (cm^{-1}) 1596m, 1586m ν(C–N), 979s ν[(P)–O–C], 753s ν[P–O–(C)], 580s ν(P–Se), 526 ν(Co–O); ^{31}P NMR (CDCl$_3$, δ ppm): 62.6 (s); μ_{eff} 5.0 B.M.

3.1.3. $Co\{O(Se)P(O^nPr)_2\}_2(NC_5H_4Me\text{-}3)_4$ (3). Prepared the same as **1**. Blue; Yield: 0.426 g (87%); Anal. Calcd for $C_{36}H_{56}Se_2P_2O_6N_4Co$ (%): C, 47.0; H, 6.1; N, 6.1. Found (%): C, 46.9; H, 6.1; N, 6.0. UV-Vis spectral data [λ_{max} (cm^{-1}); ε($L\,mol^{-1}\,cm^{-1}$)] in 0.004 mol L^{-1} 3-picoline solution 20882 (300), 11822 (272), 10480 (109); IR (cm^{-1}) 1600m, 1583m ν(C–N), 986s ν[(P)–O–C], 750s ν[P–O–(C)], 576s ν(P–Se), 516 ν(Co–O); ^{31}P NMR ($CDCl_3$, δ ppm): 63.2 (s); μ_{eff} 5.2 B.M.

3.1.4. $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$ (4). Prepared the same as **1**. Blue; Yield: 0.416 g (89%); Anal. Calcd for $C_{32}H_{48}Se_2P_2O_6N_4Co$ (%): C, 44.5; H, 5.6; N, 6.5. Found (%): C, 44.4; H, 5.5; N, 6.4. UV-Vis spectral data [λ_{max} (cm^{-1}); ε($L\,mol^{-1}\,cm^{-1}$)] in 0.005 mol L^{-1} pyridine solution 19530 (192), 11680 (160), 98992 (142); IR (cm^{-1}) 1598m, 1589m ν(C–N), 975s ν[(P)–O–C], 755s ν[P–O–(C)], 568s ν(P–Se), 531 ν(Co–O); ^1H NMR ($CDCl_3$, δ ppm): 1.24 (d, 24H, CH_3), 3.04 (m, 4H, CH), 21.8 (*o*-Py), 16.7 (*m*-Py), 10.2 (*p*-Py); ^{31}P NMR ($CDCl_3$, δ ppm): 58.3 (s); μ_{eff} 4.9 B.M.

3.1.5. $Co\{O(Se)P(O^iPr)_2\}_2(NC_5H_4Me\text{-}2)_4$ (5). Prepared the same as **1**. Blue; Yield: 0.503 g (84%); Anal. Calcd for $C_{36}H_{56}Se_2P_2O_6N_4Co$ (%): C, 47.0; H, 6.1; N, 6.1. Found (%): C, 46.9; H, 6.0; N, 6.0. UV-Vis spectral data [λ_{max} (cm^{-1}); ε($L\,mol^{-1}\,cm^{-1}$)] in 0.006 mol L^{-1} 2-picoline 21598 (146), 12620 (90), 10869 (83); IR (cm^{-1}) 1604m, 1587m ν(C–N), 990s ν[(P)–O–C], 762 ν[P–O–(C)], 572 ν(P–Se), 542 ν(Co–O); ^{31}P NMR ($CDCl_3$, δ ppm): 62.8 (s); μ_{eff} 5.0 B.M.

3.1.6. $Co\{O(Se)P(O^iPr)_2\}_2(NC_5H_4Me\text{-}3)_4$ (6). Prepared the same as **1**. Blue; Yield: 0.436 g (81%); Anal. Calcd for $C_{36}H_{56}Se_2P_2O_6N_4Co$ (%): C, 47.0; H, 6.1; N, 6.1. Found (%): C, 46.4; H, 6.1; N, 6.0. UV-Vis spectral data [λ_{max} (cm^{-1}); ε($L\,mol^{-1}\,cm^{-1}$)] in 0.005 mol L^{-1} 3-picoline solution 20890 (182), 12787 (167), 10826 (108); IR (cm^{-1}) 1604m, 1588m ν(C–N), 980s ν[(P)–O–C], 758 ν[P–O–(C)], 563 ν(P–Se), 534 ν(Co–O); ^{31}P NMR ($CDCl_3$, δ ppm): 62.6 (s); μ_{eff} 5.1 B.M.

3.2. *Crystallography*

A blue plate-like crystal of $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$ (**4**) was mounted on a glass fiber. Data were collected on a Bruker SMART CCD diffractometer. The diffraction data were collected at 150 K using graphite-monochromated Mo-Kα radiation. Cell refinement [11] gave cell constants corresponding to triclinic cells whose dimensions are given in table 1 along with other experimental parameters. An absorption correction was applied [12]. The structure was solved by direct methods [13] and refined using the WinGX version [14] of SHELX-97 [15]. All non-hydrogen atoms were treated anisotropically. Hydrogens were included in idealized positions with isotropic thermal parameters set at 1.2 times that of carbon to which they were attached. Complete list of bond distances and angles are given in table 2 and single-crystal X-ray structure is displayed in figure 2.

4. Concluding comments

We have synthesized and characterized Lewis base adducts of bis(*O*,*O*′-dialkylmonoselenophosphato)cobalt(II) complexes, $Co\{O(Se)P(OR)_2(L)_4$ (where $R = $ n-Pr, i-Pr; $L = C_5H_5N$, NC_5H_4Me-2, NC_5H_4Me-3). The elemental analyses, IR, UV-Vis, and magnetic susceptibility measurement data are consistent with distorted octahedral geometry. The molecular structure of $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$ was determined and has distorted octahedral geometry.

Supplementary material

CCDC 813709 contains the supplementary crystallographic data for $Co\{O(Se)P(O^iPr)_2\}_2(C_5H_5N)_4$. These can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 2EZ, UK (Fax: +44 1223 336033; E-mail: deposit@ccdc.cam.ac.uk).

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