SUPPLEMENTARY INFORMATION

Field-induced slow relaxation of magnetization in the *S* = 3/2 octahedral complexes *trans*-$[Co\{(OPPh_2)(EPPh_2)N\}_2(dmf)_2]$, E = S, Se: Effects of Co−Se *vs* Co−S coordination†

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IR spectroscopy

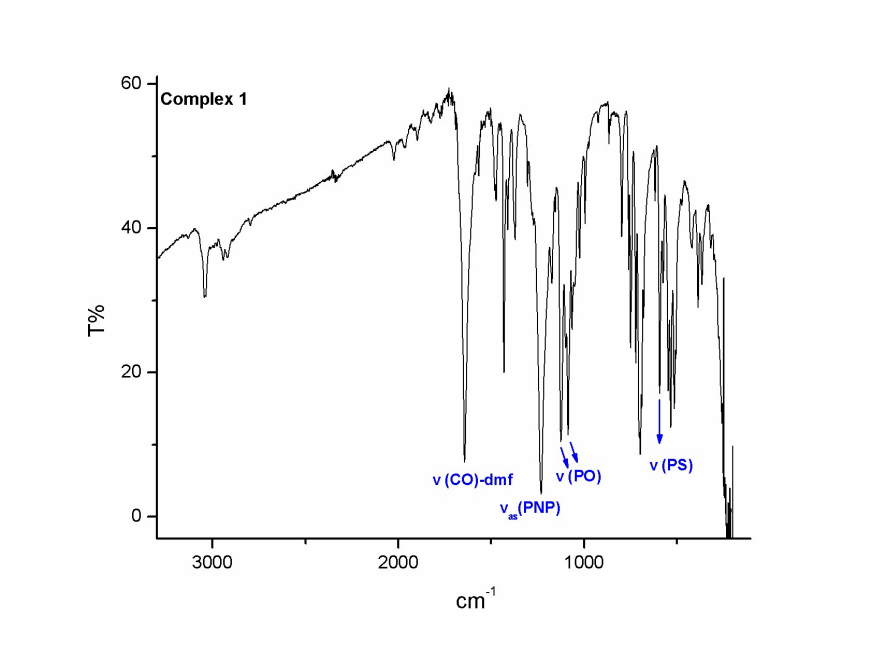


Figure S1. IR spectrum of complex **1** (KBr pellet).

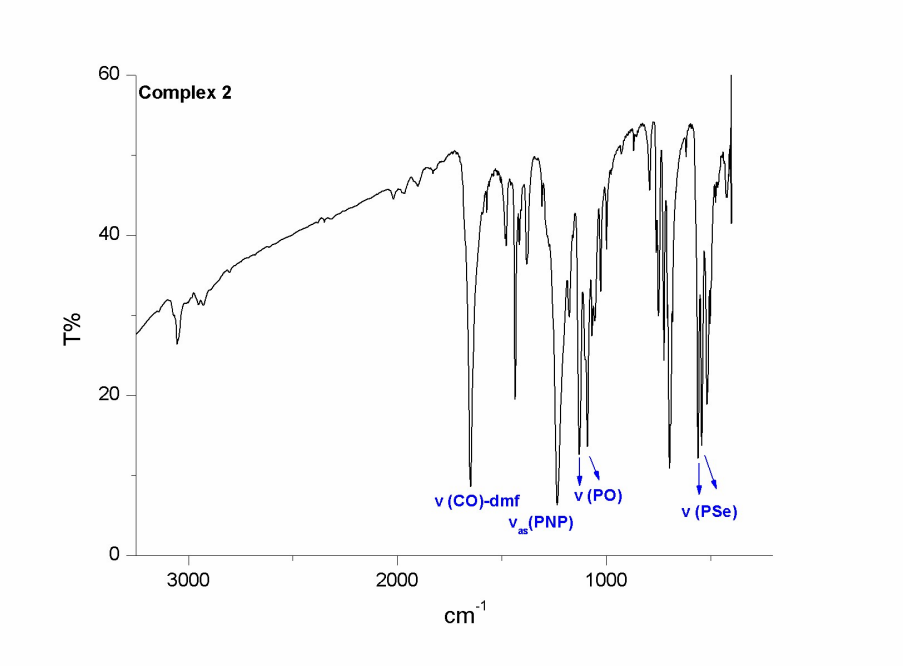


Figure S2. IR spectrum of complex **2** (KBr pellet).

IR spectroscopic characterization of complexes 1 and 2. The IR spectra of complexes **1** and **2** are shown in Figures S1 and S2, respectively. The strong bands observed for complexes **1** and **2** at 1232 cm^{-1} and 1086-1129 cm^{-1} are assigned to the ν_{as}(P-N-P) and ν(P-O) stretching vibrations, respectively, while the intense bands at 593 cm^{-1} for complex **1** and 560, 542 cm^{-1} for complex **2** correspond to the ν(P-S) and ν(P-Se) vibrations, respectively. The $(OPPh_2)(SPPh_2)NH$ ligand exhibits a ν_{as}(P-N-P) band at 934 cm^{-1}, ν(P-O) bands at 1202 and 1187 cm^{-1} and a ν(P-S) band at 626 and 613 cm^{-1}.[1] In the case of the $(OPPh_2)(SePPh_2)NH$ ligand, strong bands appear at 938, 1182-1200 and 541 cm^{-1} which have been assigned to ν_{as}(P-N-P), ν(P-O) and ν(P-Se) vibrations, respectively.[1] This behavior confirms the increase in the P–N bond order and the decrease in the P–O and P–S bonds order upon deprotonation and coordination of the ligand to Co(II), due to the delocalization of π-electronic density.[2, 3] On the other hand, complex **2** shows two ν(P-Se) bands, one of the same frequency with the free $(OPPh_2)(SePPh_2)NH$ ligand (542 cm^{-1}) and one shifted to higher energy by 19 cm^{-1}. It should be noted that this unexpected response (*i.e.* splitting and higher frequency shifting) of the ν(P-Se) vibration bands has also been observed for $K[(OPPh_2)(SePPh_2)N]$ (561 cm^{-1}), as well as for the *cis*-$[Pd\{(OPPh_2)(SePPh_2)N\}_2]$ complex (566 and 554 cm^{-1}). Complexes **1** and **2** also exhibit an intense band at 1644 and 1648 cm^{-1}, respectively, which is assigned to the ν(C−O) stretch of coordinated DMF. The ν(C−O) band is shifted to lower energy by ~ 30 cm^{-1}, compared to free dmf (1675 cm^{-1}), consistent with coordination of DMF to Co(II), as verified by X-ray crystallography. Similar IR bands (1643 cm^{-1}) have also been observed for *trans*-$[Ni\{(OPPh_2)(EPPh_2)N\}_2(dmf)_2]$, E = (O, S, Se), complexes.[4]

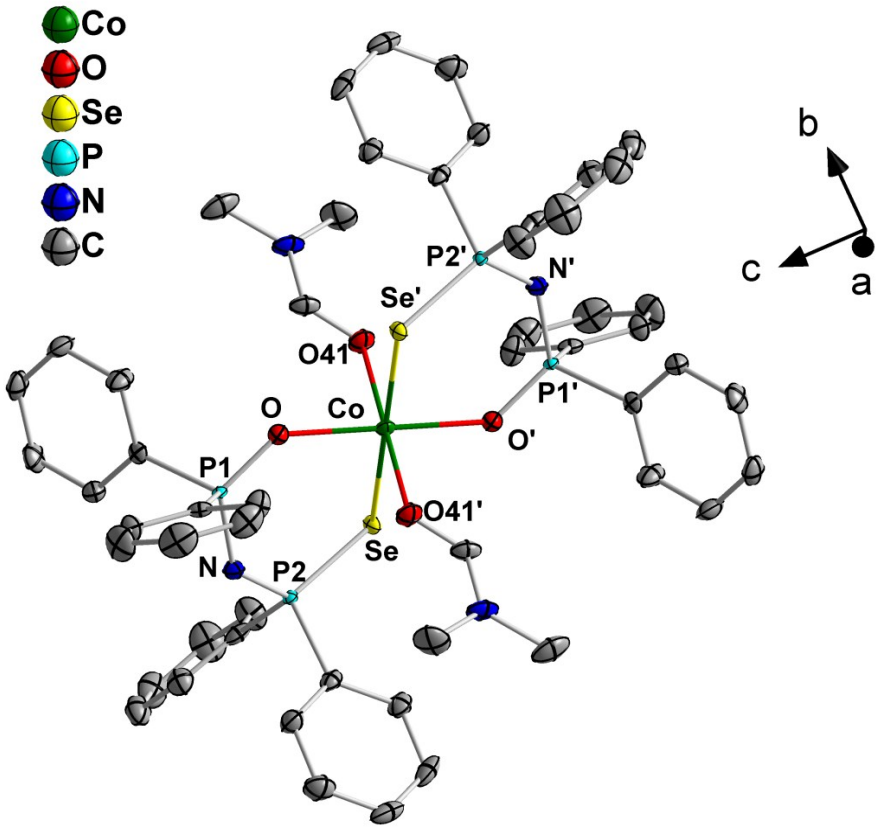


Figure S3. Ortep plot of complex **2.** Thermal ellipsoids are presented at a 50% probability level.

PXRD patterns of complexes 1 and 2.

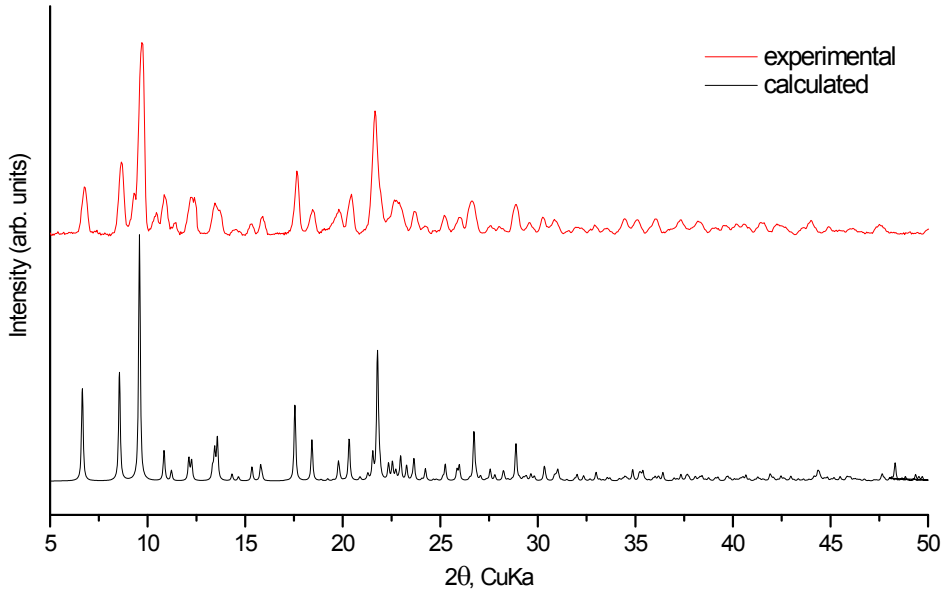


Figure S4. PXRD pattern of complex **1**.

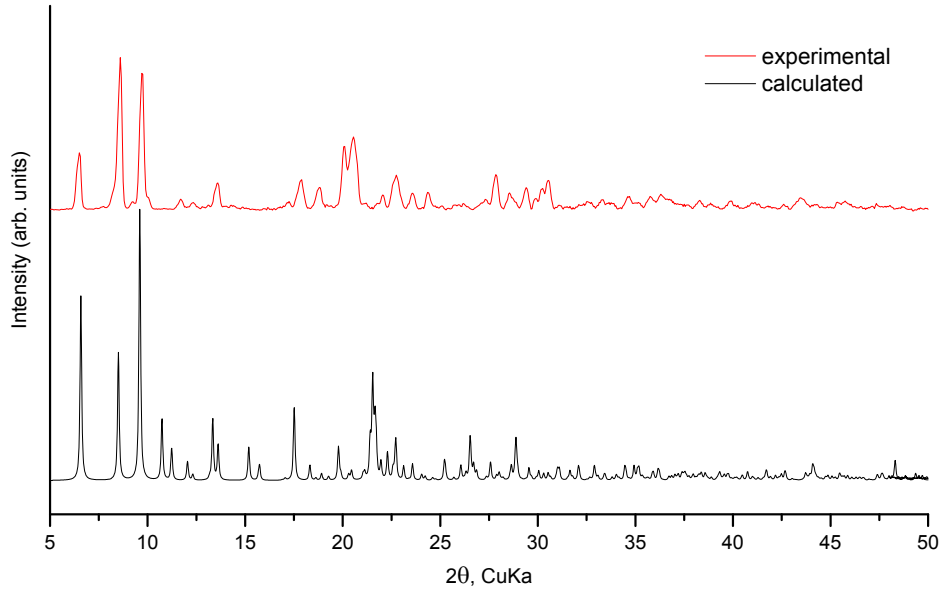


Figure S5. PXRD pattern of complex **2**.

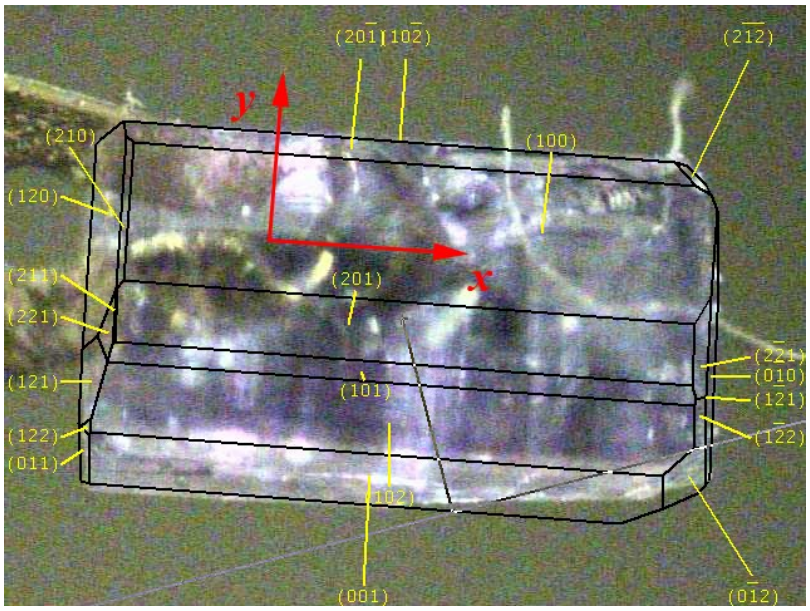


Figure S6. Orientation of a crystal of complex **1**. The XYZ coordinates are defined as follows: The intersecting line between face (100) and (001) as X axis, the normal direction of face (100) as Z axis and the Y axis is defined according to the right hand rule.

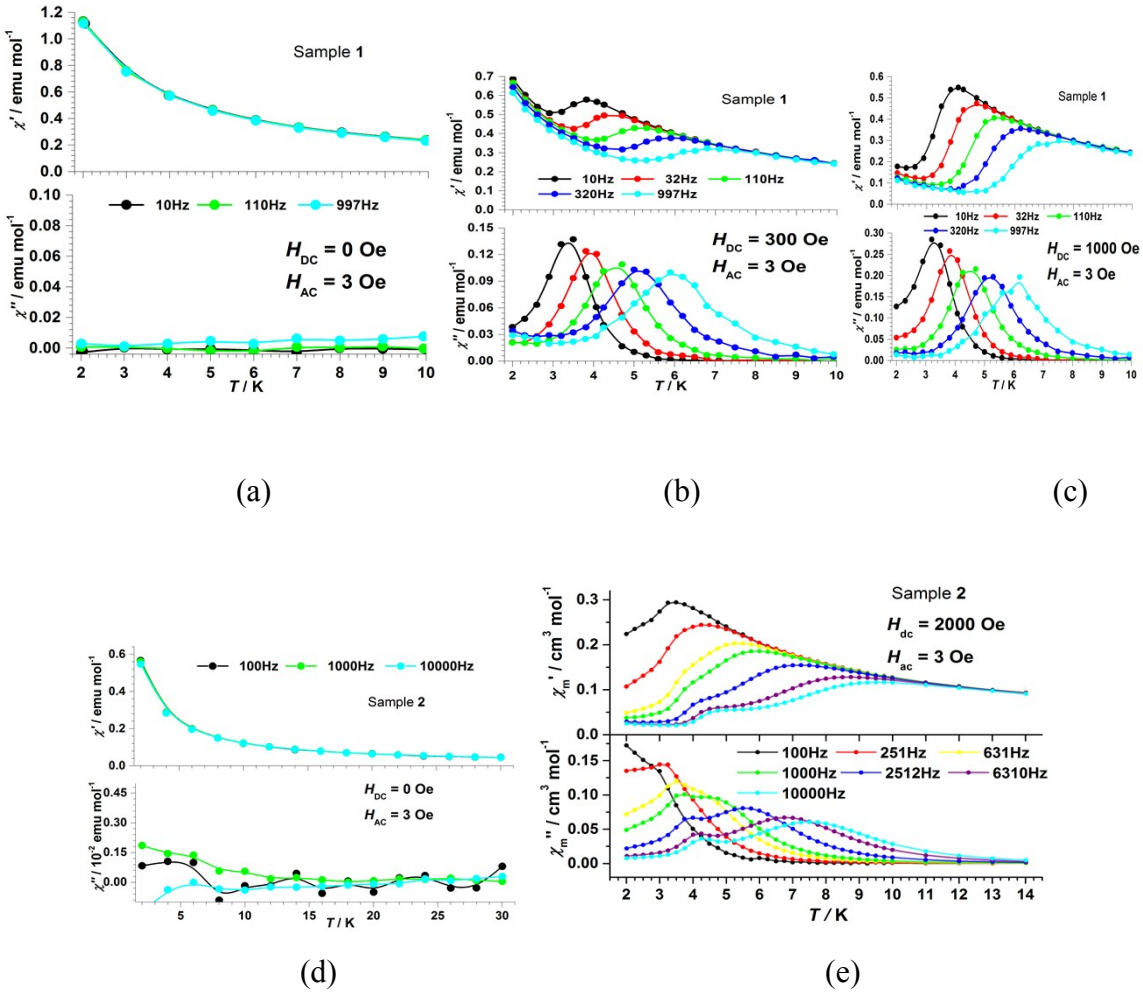
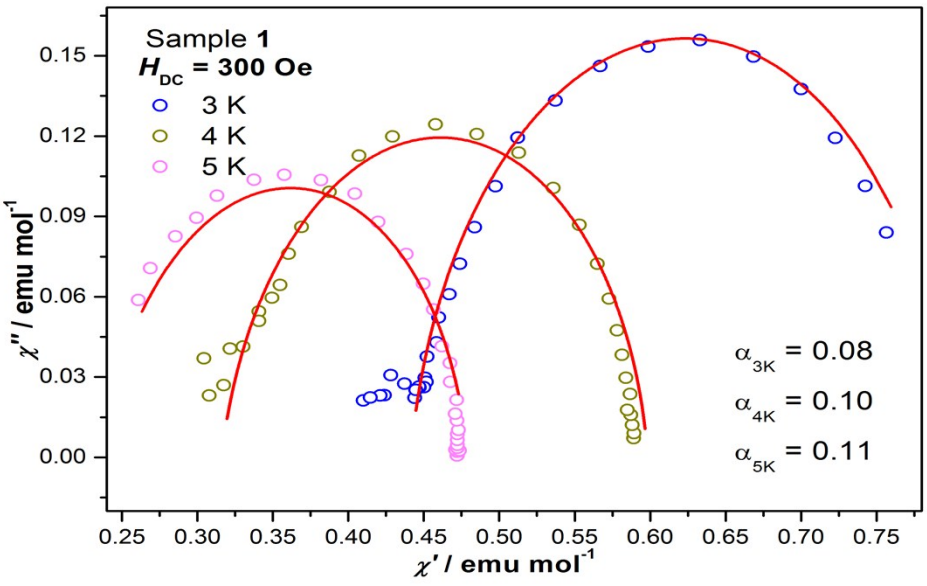
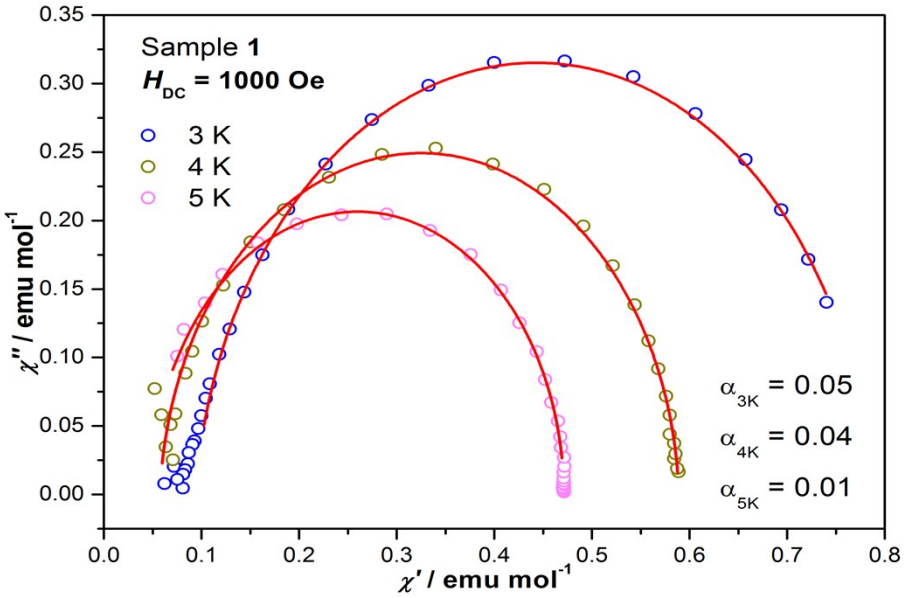


Figure S7. Temperature dependence of the in-phase (upper) and out-of-phase (below) AC susceptibility under 0 Oe (a), 300 Oe (b) and 1000 Oe (c) for complex **1** between 10 and 1000 Hz. The analogous data for complex **2** under 0 Oe (d) and 2000 Oe (e), between 100 and 10000 Hz.



(a)



(b)

Figure S8. Cole-Cole plots for complex **1** under 300 Oe (a) and 1000 Oe (b) from 3 to 5 K. Red solid lines represent the fitting results using a generalized Debye model.

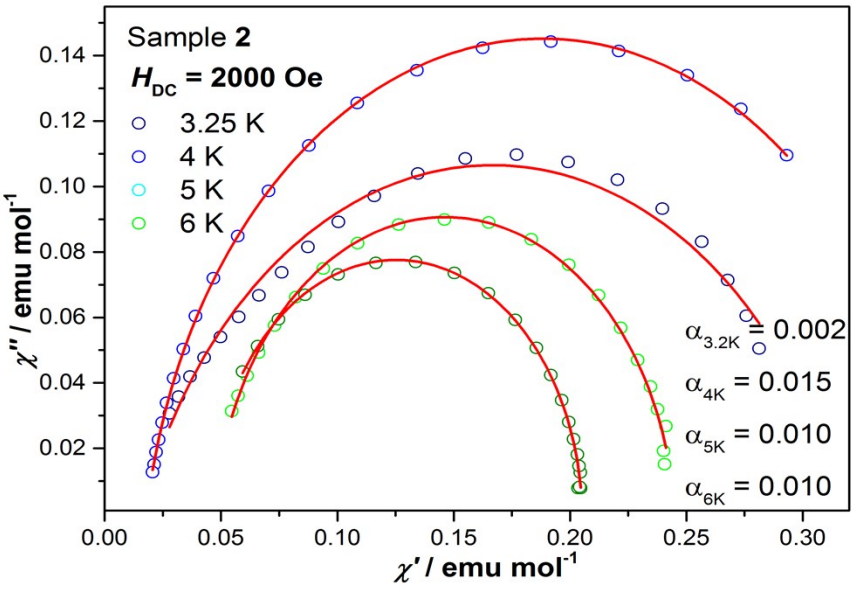


Figure S9. Cole-Cole plots for complex **2** under 2000 Oe from 3.25 to 6 K. Red solid lines represent the fitting results using a generalized Debye model..

Table S1. Octahedral $S = 3/2$ Co(II) complexes showing field-induced slow relaxation of magnetization.

Complex	Coordination sphere	U_{eff} (cm^{-1})	D or Δ_{ax} (cm^{-1})	E or Δ_{rh} (cm^{-1})
Complex **1** (this work)	CoO_4S_2	23.6	Δ_{ax} = -487.4	Δ_{rh} = 0.15
Complex **2** (this work)	CoO_4Se_2	18.1	Δ_{ax} = -448.4	Δ_{rh} = -0.43
$[Co(dmphen)_2(NCS)_2]$[5] dmphen = 2,9-dimethyl-1,10-phenanthroline	CoN_6	17.0	D = 98 Δ_{ax} = 493	E = 8.4 Δ_{rh} = 22.5
$(HNEt_3)[Co^{II}Co^{III}_3L_6]$[6] H_2L = R-4-bromo-2-((2-hydroxy-1-phenylethylimino)methyl)phenol	CoO_6	76	D = -115	E = 2.8
$[Co(\mu\text{-}L')(\mu\text{-}OAc)Y(NO_3)_2]$[7] H_2L' = N,N',N''-trimethyl-N,N''-bis(2-hydroxy-3-methoxy-5-methylbenzyl)-diethylenetriamine	CoO_3N_3	15.7	D = 47 Δ_{ax} = 645.5	Δ_{rh} = -55.8
$[Co^{II}Co^{III}(L''H_2)_2Cl(H_2O)](H_2O)_3$[8] L''$H_4$ = 2-[[(2-hydroxy-3-methoxyphenyl)methylene]amino]-2-(hydroxymethyl)-1,3-propanediol	CoO_5Cl	7.9	D = -7.4 Δ_{ax} = 339	
$[Co^{II}Co^{III}(L''H_2)_2Br(H_2O)](H_2O)_3$[8] L''$H_4$ = 2-[[(2-hydroxy-3-methoxyphenyl)methylene]amino]-2-(hydroxymethyl)-1,3-propanediol	CoO_5Br	14.5	D = -9.7 Δ_{ax} = 231	
$[Co(abpt)_2(tcm)_2]$[9] abpt = 4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole tcm = tricyanomethanide anion	CoN_6	60.3	D = 48	E = 13
$[Co(acac)_2(H_2O)_2]$[10] acac = acetylacetonate	CoO_6	14-17	D = 57	E = 17
$[(TBA)_9HCo(SiW_9O_{31})(SiO_9O_{32})]^{2+}$[11] TBA = tetra-n-butylammonium	CoO_6	13.5	D = 95.2	E = 7.2
$[Co(dca)_2(atz)_2]_n$[12]	CoN_6	5.1	Δ_{ax} = -509	

dca = dicyanamide atz = 2-amino-1,3,5-triazine				
$[Co(oda)(aterpy)]$[13] oda^{2-} = oxodiacetate aterpy = 4'-azido-2,2':6',2''-terpyridine	CoO_3N_3	2.9	D = -7.44	
$[Co(btm)_2(SCN)_2]_n$[14] btm = bis(1H-1,2,4-triazol-1-yl)methane	CoN_6	31.6	D = 56.6	E = 3.7
$[Co(ppad)_2]_n$[15] ppad = N^3-(3-pyridoyl)-3-pyridinecarboxamidrazone	CoO_4N_2	11.4	D = 76 Δ_{ax} = -482	E = +6.5
$[Co(bpm)_2(N_3)_2]$[16] bpm = bis(pyrazol-1-yl)methane	CoN_6	23.1		
$[Co(bpm)_2(H_2O)_2]^{2+}$[16] bpm = bis(pyrazol-1-yl)methane	CoN_4O_2	23.1		
$[Co(9Accm)_2(py)_2]$[17] 9Accm = curcuminoid ligand	CoO_4N_2		D = 74	E = 1.2
$[Co(9Accm)_2(bpy)]$[17] 9Accm = curcuminoid ligand	CoO_4N_2		D = 24	E = -1.9
$[Co(AcO)_2(py)_2(H_2O)_2]$[18] py = pyridine	CoO_4N_2	25.0		
$[Co(dca)_2(bim)_4]$[19] dca = dicyanamide bim = 1-benzylimidazole	CoN_6	5.5-7.7	D = |69.6| Δ_{ax} = -416	
$[Co(dca)_2(bim)_2]_n$[19] dca = dicyanamide bim = 1-benzylimidazole	CoN_6	4.5-9.2	D = |74.3| Δ_{ax} = -403	
$[Co(dca)_2(bmim)_2]_n$[19] dca = dicyanamide bmim = 1-benzyl-2-methylimidazole	CoN_6	11.5-15.4	D = |75.8| Δ_{ax} = -606	

$[Co(bpy)_2(ClAn)]$[20] bpy = 2,2'-bipyridine $ClAn^{2-}$ = chloranilate	CoN_4O_2	16.6	$D = 64.5$	$E = 10.1$
$[Co(SDZ)_2bpy]$[21] SDZ = sulfadiazine bpy = 2,2'-bipyridine	CoN_6	35.2	$D = \|81.1\|$	$E = 2$
$[Co(SDZ)_2(6MQ)_2]$[21] SDZ = sulfadiazine 6MQ = 6-methoxyquinoline	CoN_6	7.0	$D = \|78.9\|$	
$[Co(3,5\text{-dnb})_2(py)_2(H_2O)_2]$[22] 3,5-Hdnb = 3,5-dinitrobenzoic acid py = pyridine	CoO_4N_2	~ 20	$\Delta_{ax} = 644$ $\Delta_{ax} = 423$	
$Et_4N[Co(hfac)_3]$[23] hfac = hexafluoroacetylacetonate	CoO_6	13.6	$D = 117.8$ Δ_{ax}=428.3	$\Delta_{rh} = 90.3$
$\{[Co(3,3'\text{-Hbpt})_2(SCN)_2].2H_2O\}_n$[24] 3,3′-Hbpt = 1H-3-(3-pyridyl)-5-(3′-pyridyl)-1,2,4-triazole	CoN_6	22.6	$D = 70.1$	$E = 0.7$
$[Co(T_p^*)_2]$[25] KT_p^* = Potassium hydridotris(3,5-dimethylpyrazole)borate	CoN_6	26.8		
$[Co^{II}Co^{III}(LH_2)_2(CH_3COO)(H_2O)](H_2O)_3$[26] LH_4 = 2-[[(2-hydroxy-3-methoxyphenyl)-methylene]amino]-2-(hydroxymethyl)-1,3-propanediol	CoO_6	16.9	$\Delta_{ax} = -711$	$\Delta_{rh} = 44$
$[Co(dppm^{O,O})_3][CoBr_4]$[27] dppm = bis(diphenylphosphanoxido) methane	CoO_6 $CoBr_4$		$D = 147$	
$[Co(dppm^{O,O})_3][CoCl_4]$[28] $dppm^{O,O}$ = bis-(diphenylphosphan)methane	CoO_6 $CoCl_4$	106	$D = 77$	
$[Co(dppm^{O,O})_3][CoBr_4]$[28]	CoO_6		$D = 122$	

$dppm^{O,O}$ = bis-(diphenylphosphan)methane	$CoBr_4$			
$[Co(dppm^{O,O})_3][CoI_4]$[28] $dppm^{O,O}$ = bis-(diphenylphosphan)methane	CoO_6 CoI_4	47.9	$D = 99$	
$[Co(hfac)_2(H_2O)_2]$[29] hfac = hexafluoroacetylacetonate	CoO_6	8.1	Δ_{ax}=-499.7	Δ_{rh}=136.3
$[Co(pydm)_2](dnbz)_2]$[30] pydm = 2,6 – pyridinedimethanol dnbz = dinitrobenzoato	CoO_4N_2	27.3	$D = 44$	
$[Co(bzpy)_4Cl_2]$[31] bzpy = 4-benzylpyridine	CoN_4Cl_2		$D = 106$	
$[Co(bzpy)_4(NCS)_2]$[30] bzpy = 4-benzylpyridine	CoN_6		$D = 90.5$	
$[Co(L^1)_2]^{2+}$[32] L^1 = pyridine-2,6-bis(oxazoline) (pybox type)	CoN_6	10.35	$D = 61.1$	$E = -5.5$
$[Co(L^2)_2]^{2+}$[32] L^2 = pyridine-2,6-bis(oxazoline) (pybox type)	CoN_6	19.1	$D = 68.1$	$E = -5.7$
$[Co(L^3)_2]^{2+}$[32] L^3 = pyridine-2,6-bis(oxazoline) (pybox type)	CoN_6	15.8	$D = 56.4$	$E = -10.5$
$[Co(L^4)_2]^{2+}$[32] L^4 = pyridine-2,6-bis(oxazoline) (pybox type)	CoN_6	21.5	$D = -66.4$	$E = 10.2$
$[Co(L^5)_2]^{2+}$[32] L^5 = pyridine-2,6-bis(oxazoline) (pybox type)	CoN_6	14.3	$D = 62.9$	$E = -2.4$
$[Co(L^6)_2]^{2+}$[32] L^6 = pyridine-2,6-bis(oxazoline) (pybox type)	CoN_6	5.6	$D = 74.9$	$E = -2.7$
$[Co(hfa)_2(pic)_2]$[33] hfa = 1,1,1,5,5,5-hexafluoro-2,4-pentanedione pic = 4-methylpyridine	CoO_4N_2	24.7	$D = 24.17$	$E = 6.9$
$[Co(dcnm)(H_2O)(phen)_2](dcnm)$[34]	CoN_4O_2	1.4	$D = 79$	$E = 0.83$

dcnm = dicyanonitrosomethanide				
$[Co(H_2pimdc)_2(phen)]_n$[35] H_2pimdc = 2-propyl-imidazole-4,5-dicarboxylate	CoN_4O_2	23.1	D = 3.5	E = 1.0
$[Co(abpt)_2(H_2O)_2](N[C(CN)_2]_2)_2$[36] abpt = 4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole	CoN_4O_2	66.4	D = 34.9	
$[Co(abpt)_2(H_2O)_2](NO_2C(CN)_2)_2$[36] abpt = 4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole	CoN_4O_2	72.2	D = 31.0	
$[Co(abpt)_2(CH_3OH)_2](C(CN)[C(CN)_2]_2)_2$[36] abpt = 4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole	CoN_4O_2	13.1	D = 37.8	
$[Co(abpt)_2(NO_2NCN)_2]$[36] abpt = 4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole	CoN_6	44.5	D = 41.4	
$[Co(abpt)_2(NCSe)_2]$[36] abpt = 4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole	CoN_6	51.4	D = 37.7	
$[Co(abpt)_2[ONC(CN)_2]_2]$[36] abpt = 4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole	CoN_4O_2	47.3	Δ_{ax} = -1345	
$[Co(bppCOOH)_2](ClO_4)_2$[37] bpp-COOMe = methyl 2,6-di(pyrazol-1-yl)pyridine-4-carboxylate	CoN_6	11	D = 51.6	
$[Co(pyrtpy)_2](BPh_4)_2$[38] pyrtpy =4'-pyrrolidine-2,2':6',2''-terpyridine	CoN_6	6.8	D = -53.6	E = 5.53
$[Co(bpp\text{-}COOMe)_2](ClO_4)_2$[39] bpp-COOMe = methyl 2,6-di(pyrazol-1-yl)pyridine-4-carboxylate	CoN_6	30.3	D = -57.5 Δ_{ax} = -1850	E = |15.7| Δ_{rh} = 130
$[CoL_2(H_2O)_2]$[40] HL = quinoline-2-carboxylic acid	CoO_4N_2	38.2	D = 62.1 Δ_{ax} = 106	E = |3.17|
$[CoL_2(CH_3CH_2O)_2]$[40] HL = quinoline-2-carboxylic acid	CoO_4N_2	30.3	D = 72.08 Δ_{ax} = 150	E = |7.37|
$[Co(pydca)(dmpy)]_2$[41] pydca = pyridine-2,6-dicarboxylato	CoO_4N_2		D/hc = 55 Δ_{ax} = -846	E/hc = 19

dmpy = 2,6-dimethanolpyridine)				
$[Co(teaH_3)](piv)]$[42] $teaH_3$ = triethanolamine piv = trimethylacetate	CoO_4N_2		*D*/hc = 23.1	
$[Co(L)_2](BF_4)_2 \cdot 2MeNO_2$[43] L = 2,6-di(pyrazol-1-yl)-4-(bromomethyl)pyridine	CoN_6	14.6		
$[Co(L)_4(NO_3)_2]$[44] L = 3-phenylpyrazole	CoN_4O_2	10.3	*D* = 71.4	*E* = |13|
$[Co(L)_4(NO_3)_2]$[44] L = 4-methylpyridine	CoN_4O_2	5.1	*D* = 71.5	*E* = |9.8|
$[Co(imidazole)_6](BPh_4)_2$[45]	CoN_6	15		
$[Co(imidazole)_6](NO_3)_2$[45]	CoN_6	4.4		
$[Co(2\text{-}Himap)_2]$[46] 2-Himap = o-[(1H-imidazol-2-yl)methylideneamino]phenol	CoN_4O_2	9.7	*D* = 36.7	*E* = 2.0
$[Co(AcPyOx)_3BC_6H_5]ClO_4$[47] AcPyOx = 2-Acetylpyridineoxime	CoN_6	70.5	*D* = -86	
$[[Co(L1)(2,2'\text{-bipy})] \cdot 0.5DMF]_n$[48] H_2L1 = 2,2′-[benzene-1,4-diylbis(methanediylsulfanediyl)]dibenzoic acid	CoO_4N_2	9.7	*D* = -56.2	*E* = |16.4|
$[[Co(bimb)(H_2O)_4] \cdot (L2) \cdot 2DMF]_n$[48] H_2L2 = 2,2'-(1,4-phenylenebis(methylene))bis(sulfanediyl)dinicotinic acid bimb = 1,4-bis(benzoimidazo-1-ly)benzene	CoO_4N_2	6.1	*D* = 57.5	*E* = |13.0|
$[Co(HATN)(hfca)_2] \cdot CH_2Cl_2$[49] HATN = 5,6,11,12,17,18-hexaazatrinaphthylene	CoO_4N_2	11.9	*D* = -60	
$[Co_2(3\text{-}fba)_4(4,4'\text{-bpy})_3(CH_2OH)_2]$[50] 3-Hfba = 3-fluorobenzoic acid 4,4′-bpy = 4,4′- bipyridine	CoO_4N_2	15.8		

$[Co(abtp)_2(N_3)_2]$[51] abpt = 4-amino-3,5-bis(pyridin-2-yl)-1,2,4-triazole	CoN_6	54.7	D = -24.1	E = 7.5
$[Co(bim)_4(tcm)_2]$[52] bim = 1-benzylimidazole tcm^- = tricyanomethanide ion	CoN_6	28	D = 46.1 Δ_{ax} = 667	
$[Co(bmim)_4(tcm)_2]$[52] bmim= 1-benzyl-2-methylbenzimidazole tcm^- = tricyanomethanide ion	CoN_6	20	D = 80.1 Δ_{ax} = -631	
$[Co(2,6\text{-}dfba)_2(bpp)_2(H_2O)_2]_n$[53] 2,6-Hdfba = 2,6-difluorobenzoic acid bpp = 1,3-bis(4-pyridyl)propane	CoO_4N_2	31.5	D = 53.2 Δ_{ax} = 119	E = |7.7|
$[Co(2,6\text{-}dfba)_2(bpe)_2(H_2O)_2]_n$[53] 2,6-Hdfba = 2,6-difluorobenzoic acid bpe = 1,2-bis(4-pyridyl)ethylene	CoO_4N_2	40.3	D = 65.7 Δ_{ax} = -502	E = |4.5|

Table S2. Crystallographic data for complexes **1** and **2**.

	compound **1**	compound **2**
Formula	$C_{54}H_{54}CoN_4O_4P_4S_2$	$C_{54}H_{54}CoN_4O_4P_4Se_2$
Fw	1069.94	1163.74
Space group	P-1	P-1
a (Å)	9.2942 (1)	9.2622(2)
b (Å)	10.3734 (1)	10.4256(2)
c (Å)	13.3197 (2)	13.4612(3)
α (º)	89.528 (1)	89.4230(10)
β (º)	85.318 (1)	85.6970(10)
γ (º)	84.698 (1)	85.3890(10)
V (Å^3)	1274.42 (3)	1292.00(5)
Z	1	1
T (ºC)	-113	-113
Radiation	Cu *K*α	Cu *K*α
ρ_{calcd} (g cm^{-3})	1.394	1.496
μ (mm^{-1})	4.995	5.783
Reflections with $I>2\sigma(I)$	3608	3920
R_1[a] $[I>2\sigma(I)]$	0.0442	0.0317
wR_2[a] $[I>2\sigma(I)]$	0.1119	0.0734

[a] $R_1 = \Sigma(||F_o|-|F_c||)/\Sigma(|F_o|)$ and $wR_2 = [\Sigma[w(F_o^2-F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{1/2}$, $w=1/[\sigma^2(F_o^2)+(\alpha P)^2+bP]$ and $P = [\max(F_o^2,0)+2F_c^2]/3$. α=0.0571, b=1.2764 for **1**; α=0.0304, b=2.1175 for **2**.

Further experimental crystallographic details for **1**: $2\theta_{max}$ = 130°; reflections collected/unique/used, 15108/4038 [R_{int} = 0.0472]/4038; 397 parameters refined; $(\Delta/\sigma)_{max}$ = 0.005; $(\Delta\rho)_{max}/(\Delta\rho)_{min}$ = 0.513/-0.461 e/Å^3; *R*1/w*R*2 (for all data), 0.0496/0.1190.

Further experimental crystallographic details for **2**: $2\theta_{max}$ = 130 °; reflections collected/unique/used, 26137/ 4124 [R_{int} = 0.0310]/4124; 417 parameters refined; $(\Delta/\sigma)_{max}$ = 0.006; $(\Delta\rho)_{max}/(\Delta\rho)_{min}$ = 0.589/-0.454 e/Å^3; *R*1/w*R*2 (for all data), 0.0335/0.0751.

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