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Benzyl cobaloximes with thiodioximes: Synthesis, structure and reactivity

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ABSTRACT

Five benzyl cobaloximes with different thiodioximes, BnCo[d(SR)gH]$_2$Py, have been synthesized and four of these complexes have been characterized by X-ray. The reactivity of these complexes towards molecular oxygen has been studied. The puckering of the Co(dioxime)$_2$ unit, caused by dioxime side chain, the SR group, significantly influences the Co–C bond reactivity. Structural features in one of the oxygen inserted cobaloximes have been studied to confirm if puckering of dioxime is the guiding factor. The reactivity is also affected, to some extent, by the C–H…π interaction between the benzyl and the dioxime moiety.

1. Introduction

Co–C bond cleavage is generally accepted as a key step in the mechanism of action of many enzymes which require a B_{12} coenzyme [1–3]. The modifications of the side chains on the periphery of the corrin ring lead to considerable variation in coenzyme activity [1,4,5]. Since the changes in the side chain are not such to alter significantly the electronic properties of the corrin ring system, it is unlikely that the variation in activity results from the inductive effect. A reasonable hypothesis advanced to explain this observation is that specific tight interactions between the enzyme and coenzyme bring about distortion of the corrin ring system, and thus facilitate the Co–C bond cleavage. The study of Cobaloximes[1] has provided an experimental test of this idea [6–15].

In our on-going efforts to find factors that influence the Co–C bond reactivity in cobaloximes, we have found that the equatorial dioxime moiety (gH, chgH, dpgH, dmestgH, dSPhgH) (*cis*-influence) plays a significant role, for example, in Diels–Alder reaction, alkyl-alkenyl cross coupling reactions and in the oxygen insertion reactions [16–28]. We have also found that the *cis* influence and the rate of oxygen insertion into the Co–C bond in cobaloximes are related and follow the same order dmestgH >> dpgH > chgH > dSPhgH ≥ dmgH > gH [16]. More complexes, however, need to be studied to generalize this relationship. A question was raised during our recent study with dSPhgH [bis (thiophenyl glyoxime)] complexes whether the variation in SR will alter the reactivity of the Co–C bond [16]? Keeping this in view we have synthesized and characterized five benzyl cobaloximes (**3a**–**3e**) with thio dioximes in which the SR group on the dioxime has been varied (Scheme 1). All complexes except **3e** are new. The X-ray structures of **3a**, **3c**, **3d** and **3e** are reported for the first time. Comparative studies of X-ray structural parameters of **3a**, **3c**, **3d** and **3e** and the rate of oxygen insertion in **3a**–**3e** have been discussed. We have also studied the structural features of an oxygen inserted cobaloxime **4** (Chart 1) to confirm if puckering of dioxime is the guiding factor for the highest reactivity of **3c**.

2. Experimental section

2.1. Materials and physical measurements

$CoCl_2.6H_2O$ (SD fine, India), glyoxime (Caution! it is highly flammable and explosive when dry) (Alfa Aesar), benzyl chloride, ethanethiol, 2,4-dimethylbenzene-thiol, 3,5-dimethylbenzene-thiol, 2,6-dimethylbenzene-thiol, thiophenol (Aldrich chemical company) were purchased and used as received. Silica gel (100–200 mesh) and distilled solvents were used in all reactions and chromatographic

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[1] Cobaloximes have the general formula RCo(L)$_2$B, where R is an organic group σ-bonded to cobalt. B is an axial base trans to the organic group, and L is a monoanionic dioxime ligand e.g. glyoxime (gH), dimethylglyoxime (dmgH), 1,2-cyclohexanedione dioxime (chgH), diphenylglyoxime (dpgH), dimesitylglyoxime (dmestgH), and dithiophenylglyoxime (dSPhgH).

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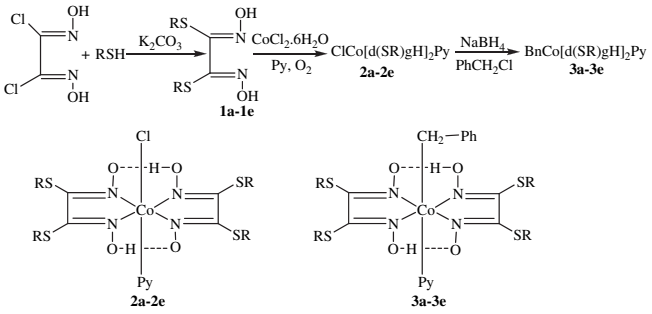

Scheme 1.

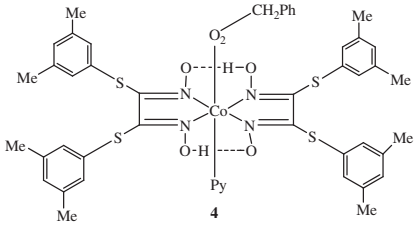

Chart 1.

separations. A julabo UC-20 low temperature refrigerated circulator was used to maintain the desired temperature. ^{1}H and ^{13}C Spectra were recorded on a JEOL JNM LAMBDA 400 FT NMR Spectrometer (400 MHz for ^{1}H and 100 MHz for ^{13}C) in $CDCl_3$ solution with TMS as internal standard. NMR data are reported in ppm. Elemental analysis was carried out at IIT Kanpur.

2.2. X-ray crystal structure determination and refinements

Single-crystal X-ray data were collected using graphite-monochromated Mo-$K\alpha$ radiation (λ = 0.71073 Å) on "Bruker SMART APEX CCD" diffractometer at 100 K for **3a**, **3c** and **3d**; at 298 K for **3e** and 293 K for **4**. The linear absorption coefficients, scattering factors for the atoms and the anomalous dispersion corrections were taken from the *International Tables for X-ray Crystallography* [29]. The data integration and reduction were processed with SAINT [30] software. An empirical absorption correction was applied to the collected reflections with SADABS [31] using XPREP [32]. All the structures were solved by the direct method using SIR-97 [33] and were refined on F^2 by the full-matrix least-squares technique using the SHELXL-97 [34] program package. All non-hydrogen atoms were refined anisotropically in all the structure. The hydrogen atoms of the OH group of oxime were located on difference Fourier maps and were constrained to those difference Fourier map positions. The hydrogen atom positions or thermal parameters were not refined but were included in the structure factor calculations. The pertinent crystal data and refinement parameters for compound **3a**, **3c**, **3d**, **3e** and **4** are compiled in Table 1.

2.3. Synthesis

Synthesis of **3a-3e** is presented in Scheme 1. The dioximes (**1a-1d**) were prepared from dichloroglyoxime [16] and the corresponding thiol in the presence of K_2CO_3 following the reported procedure [16]. However, the synthesis of **1e** required the addition of KOH (2 pellets for 0.636 mmol dichloroglyoxime) also. The complexes (**3a-3e**) were synthesized from the corresponding chlorocobaloximes (**2a-2e**) following a general procedure outlined earlier [16]. Yield = 68–75%. The oxygen insertion rate studies were carried out following the procedures outlined in our earlier paper [28] and the reported rate constants value is an average of the 2–3 trials in each case.

Table 1
Crystal data and structure refinement details for compounds **3a**, **3c**, **3d**, **3e**.

Parameters	3a	3c	3d	3e	4
Empirical formula	$C_{24}H_{34}CoN_5O_4S_4$	$C_{48}H_{50}CoN_5O_4S_4$	$C_{49}H_{52}Cl_2CoN_5O_4S_4$	$C_{40}H_{34}CoN_5O_4S_4$	$C_{48}H_{50}CoN_5O_6S_4$
Formula weight	643.77	948.14	1033.07	835.89	980.14
Temp (K)	100(2)	100(2)	100(2)	298(2)	293(2)
Crystal system	monoclinic	monoclinic	triclinic	monoclinic	triclinic
Space group	*P 21/n*	*P 21/n*	*P-1*	*P 21/c*	*P1*
Unit cell dimensions					
a (Å)	8.892(5)	15.928(5)	11.383(5)	18.494(5)	8.7836(11)
b (Å)	17.269(5)	17.112(5)	17.471(5)	13.156(5)	11.9350(15)
c (Å)	19.062(5)	33.105(5)	25.134(5)	16.549(5)	12.0157(15)
α(deg)	90.000(5)	90.000(5)	81.446(5)	90.000(5)	68.202(2)
β (deg)	100.163(5)	95.268(5)	84.347(5)	114.433(5)	89.140(2)
γ (deg)	90.000(5)	90.000(5)	80.999(5)	90.000(5)	83.050(2)
V (Å^3)	2881(2)	8985(4)	4867(3)	3666(2)	1160.4(3)
Z	4	8	4	4	1
ρ (calc), mg/m^3	1.484	1.402	1.410	1.515	1.403
μ (Mo-$K\alpha$) (mm^{-1})	0.925	0.619	0.683	0.747	0.604
F (000)	1344	3968	1984	1728	512
Crystal size (mm^3)	0.35 × 0.30 × 0.25	0.40 × 0.35 × 0.26	0.40 × 0.35 × 0.26	0.30 × 0.25 × 0.20	0.42 × 0.36 × 0.28
Index ranges	$-11 \le h \le 11$, $-22 \le k \le 21$, $-25 \le l \le 17$	$-19 \le h \le 19$, $-20 \le k \le 20$, $-40 \le l \le 29$	$-13 \le h \le 13$, $-21 \le k \le 10$, $-30 \le l \le 29$	$-24 \le h \le 24$, $-17 \le k \le 16$, $-21 \le l \le 14$	$-10 \le h \le 9$, $-14 \le k \le 11$, $-14 \le l \le 13$
No. of rflns collected	18,808	46,253	26,651	23,706	62,14
No. of indep rflns	7088	16,705	17,823	8960	5061
GOOF on F^2	1.116	0.995	0.991	1.153	1.062
Final *R* indices ($I > 2\sigma(I)$)	*R*1 = 0.0582, wR2 = 0.1500	*R*1 = 0.0727, wR2 = 0.1669	*R*1 = 0.0814, wR2 = 0.2287	*R*1 = 0.0443, wR2 = 0.0904	*R*1 = 0.0498, wR2 = 0.1297
R indices (all data)	*R*1 = 0.0889, wR2 = 0.2282	*R*1 = 0.1333, wR2 = 0.2131	*R*1 = 0.1182, wR2 = 0.2598	*R*1 = 0.0621, wR2 = 0.1301	*R*1 = 0.0527, wR2 = 0.1340
Data/restraints/param	7088/0/343	16,705/0/1117	17,823/0/1171	8960/0/487	5061/42/585

Table 2
^{1}H NMR data (ppm) for **3a**–**3e** and **4**.

Comp no	Py_α	Py_β	Py_γ	Aromatic Proton	CH_2	Other Protons	O–H…O
3a	8.53	7.32	7.71	7.03–7.18	3.12	3.16(m), 2.94(m), 1.04(t)	18.33
3b	8.21	[a]	7.91	6.30–7.30	3.16	2.16	17.88
3c	8.32	[a]	7.80	6.47–7.35	3.10	2.01	18.29
3d	8.01	[a]	7.77	6.89–7.25	3.01	2.07	17.69
3e	8.13	[a]	7.81	6.76–7.22,	3.13		18.11,
4	8.19	[a]	7.77	6.66–7.22	4.36	2.05	18.29

[a] merge with aromatic protons.

3. Results and discussion

3.1. Spectroscopy

Free thiodioximes (**1a-1e**) are partially soluble in $CDCl_3$ and a drop of DMSO-d^6 is necessary to dissolve it. The ^{1}H NMR spectra of complexes **3a-3e** are easily assigned based on the chemical shifts, relative intensities and the assignments are consistent with the previously described complexes. $^{13}CPy_\alpha$ and C═N resonances are confirmed by DEPT as they appear close together. The ^{1}H NMR spectral data for **3a-3e** is given in Table 2.

As the aim is to study the effect of substituent on sulphur, the NMR chemical shifts in **3a-3e** have been compared; the upfield shift of Py_α is highest in **3d** (due to strong C–H….π interaction 3.654 Å) and lowest in **3a** (SEt group lacks C–H….π interaction due to the absence of aromatic ring current) (Table S1). O–H…O follows the same trend. Similarly, CH_2 resonance is most upfield shifted in **3d** but the values are similar in **3a-3c** and **3e** (Table S2). It seems that the electronic *cis*-influence does not change significantly with the change of substituent on S in these complexes.

The $\delta^{13}C_{C=N}$ is sensitive to any change in axial or equatorial environment in cobaloximes and generally shifts upfield on coordination to cobalt. This upfield shift [$\Delta\delta$ ($^{13}C_{C=N}$)] is more in **3a** compared to **3e** which may mean that the charge density on C═N is more in the former (Table S3).

Insertion of O_2 into the Co–C bond affects the chemical shift of CH_2 and Py_α in **4**; for example, CH_2 is directly attached to O_2 and hence shifts downfield by 1.24 ppm whereas Py_α is upfield shifted due to very strong C–H….π interaction (3.362 Å).

3.2. Structural studies

Good quality single crystals were obtained by slow evaporation of solvent (DCM/MeOH for **3a**, **3c**, **3e** and DCM/MeOH/CH_3CN for **3d**). Selected bond lengths and bond angles are given in Table 3 and the molecular structures are shown in Figs. 1–4. The geometrical deformations of Co(dioxime)$_2$ unit is roughly represented by the displacement of cobalt atom out of the plane of four nitrogen in the dioxime (d) and by the bending angle between the two dioxime units (α). Positive value of *d* and α indicate displacement towards Py and bending towards R. Distortion in Co(dioxime)$_2$ moiety is generally related, to some degree, to the bulk of the axial ligands; α and *d* have high values when one of the two axial ligands is bulky (Table S4 and Table S5) and the displacement is towards and the bending is away from the bulkier ligand. When both the axial ligands are sufficiently bulky, α and *d* values are significantly smaller, with a small tilt of dioxime units.

However, not much is known about the distortion with respect to the change in the dioxime moiety. This is because of the paucity of crystal data on cobaloximes with different dioximes. The data in alkyl cobaloximes shows that the distortion is highest in the mesitylglyoxime complexes (Table S6). A very significant observation has been made in the present study that the strategic variation of SR group on the dioxime induces changes in *d* and α value from positive to negative. The puckering of the dioxime plane is highest in **3c** and lowest in **3a**. It is difficult to say with certainty, until we have data on large number of compounds, whether the orientation of the SR group has any role in this deviation. For example, the

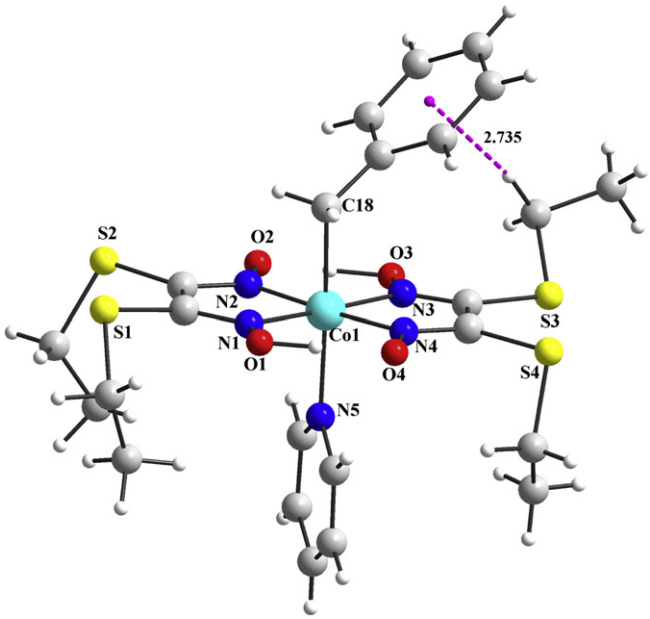

Fig. 1. Molecular structure of **3a**.

Table 3
Selected Bond Lengths (Å), Bond Angles (deg) and Structural Data for **3a**, **3c**, **3d** and **3e**.

	3a	3c	3d	3e
Co–C	2.044(4)	2.060(5) 2.073(5)	2.061(6) 2.064(6)	2.071(3)
Co–N	2.056(3)	2.047(4) 2.045(4)	2.049(5) 2.056(5)	2.062(3)
C–Co–N	174.50(16)	175.81(2) 174.46(19)	175.1(2) 176.8(2)	177.10(12)
d (Å)	+0.037 (6)	+0.074 (7) +0.062 (7)	−0.028 (9) −0.041 (9)	−0.019 (5)
α (deg)	+1.695(125)	+10.747 (120) +7.845 (170)	−5.942 (243) −6.466 (361)	−6.964 (86)
τ (deg)	75.420 (122)	86.447 (123) 89.703 (114)	83.393 (157) 83.803 (196)	89.544 (83)

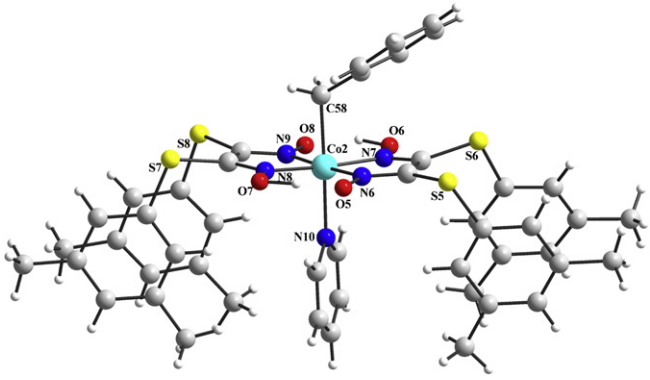

Fig. 2. Molecular structure of **3c**.

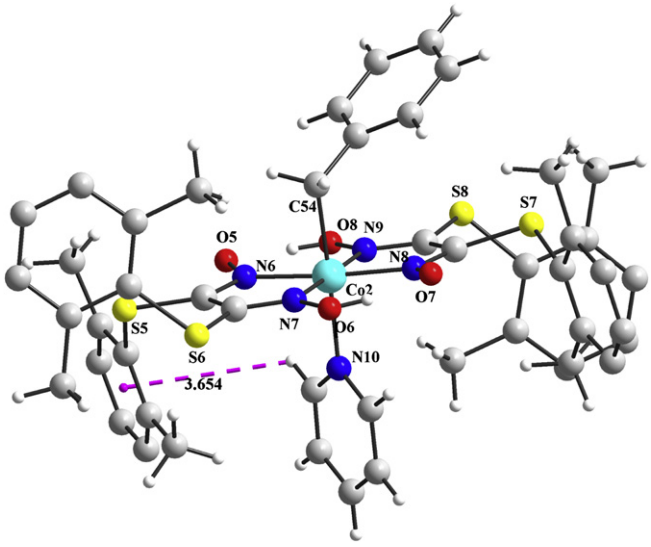

Fig. 3. Molecular structure of **3d**.

different orientation of SR groups (with respect to the dioxime plane); the down–down; down–down conformation in **3e** and down–down; up-down in **3a** has affected the α value in these compounds. Whereas, **3c** is much more puckered (+10.747 and +0.074 Å) than **3e** even though both have the same conformation of the SR groups. It is worth mentioning that this is the highest α value observed among all the reported benzyl cobaloxime structures so far.

Bending angle is a measure of the steric *cis*-influence and it increases as the steric bulk of the dioxime increases. Does this affect the Co–C bond reactivity? Nothing has been reported in the literature on this issue.

As we move from **3e** to **3d** there is almost no change in α and *d* value but a huge change occurs in **3c**. This may mean that the strategic placement of methyl groups on the SPh increases the steric bulk of the dioxime. We will see if the puckering of the dioxime has any correlation with the Co–C bond reactivity.

A slow evaporation of solvent from the solution of **4** ($CHCl_3$/MeOH) results in the formation of brown crystal. The diamond diagram is shown in Fig. 5 and the selected bond lengths and bond angles are presented in Table 4. Co–N (1.991 Å), Co–O (1.891 Å), O–O (1.424 Å) bond distances and Co-O-O-C dihedral angle (114.4)

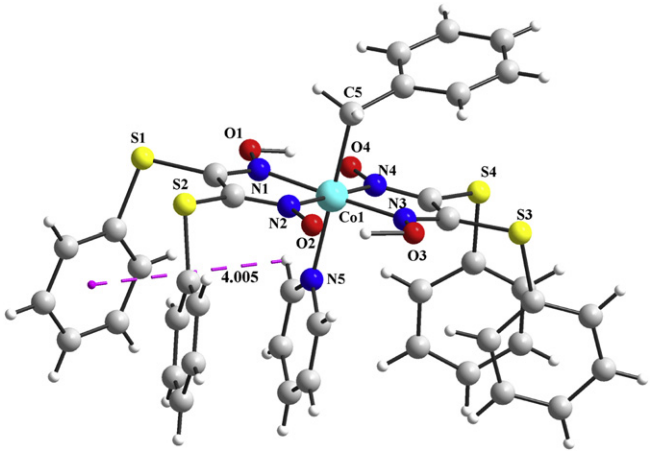

Fig. 4. Molecular structure of **3e**.

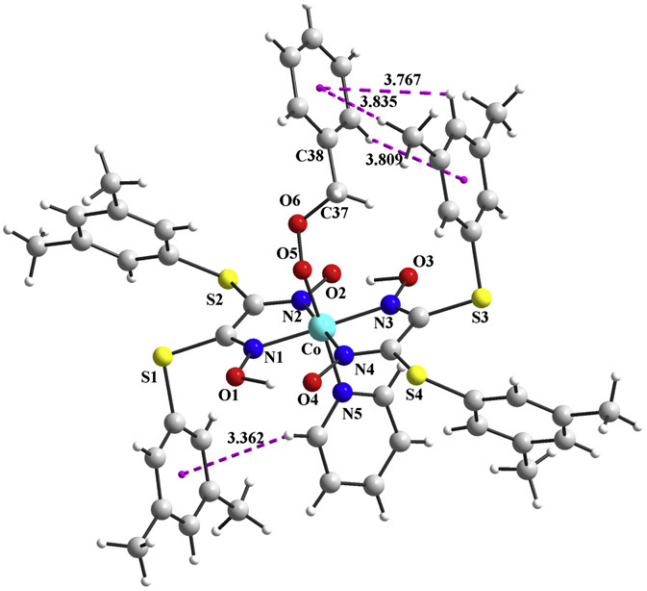

Fig. 5. Molecular structure of **4**.

do not differ significantly from the previously reported values of oxygen inserted cobaloximes [28], [35,36]. The important observation is that the strain in **3c** decreases after oxygen insertion since α and *d* values reduce to +1.105° and +0.007 Å respectively in **4**. Orientation of the benzyl group in **4** is markedly different from the previously reported oxygen inserted cobaloxime complexes; for example benzyl group in **4** lies vertically up and perpendicular to the dioxime plane, unlike the previous structures. A similar orientation of the benzyl group was found in the SO_2 inserted cobaloxime, $ArCH_2SO_2Co(dioxime)_2B$, reported earlier from our group [37]. C–H…π interactions between benzyl centroid and phenyl proton (3.767 Å), upward phenyl centroid and benzyl proton (3.809 Å) and benzyl centroid and thiophenyl methyl proton (3.835 Å) are the guiding factors of such orientation.

3.3. Molecular oxygen insertion

We have, in the recent past, studied the reactivity of the Co–C bond by measuring the rate of oxygen insertion in organocobaloximes [28]. Since the cleavage of the Co–C bond is the key step in this reaction and the effect of *cis*-influence is felt most on the Co–CH_2 bond, these two processes, *cis*-influence and rate of insertion, are related to each other and follow the same order [16]. Benzyl cobaloximes, in general, have inherently weak Co–C bond and are ideal systems for such study. The reaction follows a pseudo

Table 4
Selected Bond Lengths (Å), Bond Angles (deg) and Structural Data for **4**.

Co-O_{ax}	1.891(5)
Co-N_{ax}	1.991(5)
O–O	1.424(6)
Co–O–O	116.0(3)
O–O–C	103.8(4)
Co-O-O-C	−114.4(5)
O–C–C	108.4(5)
O-O-C-C	−165.6(5)
N_{py}-Co-O_{ax}	173.5(3)
d(Å)	+0.007(9)
α(deg)	+1.105(159)
τ (deg)	68.779(147)

Table 5
Pseudo-first order rate constant (kobs) for oxygen insertion in **3a**–**3e**.

	3a	3b	3c	3d	3e
k_{obs} (s^{-1})	4.03×10^{-3}	1.67×10^{-3}	1.12×10^{-2}	1.96×10^{-3}	1.29×10^{-3}

first order kinetics. A comparison of the rate of oxygen insertion in **3a-3e** has been made and presented in Table 5. The data shows that the puckering of the Co(dioxime)$_2$ moiety significantly affects the rate of oxygen insertion and the rate correlates well with α. For example, **3c** has the highest puckering and the highest rate of insertion. The rate is slightly faster in **3a** than **3b**, **3d** and **3e**. This is attributed to the very strong C–H…π interaction (2.735 Å) between SEt and the benzyl group.

4. Conclusion

In conclusion, this is the first study in cobaloximes which correlates the Co–C bond reactivity with the puckering in the dioxime. The puckering is caused by the strategic variation of the dioxime side chain. The strain in the precursor cobaloxime gets released after the oxygen insertion and this has been confirmed by the X-ray structure data in the oxygen inserted product. In addition, the weak interactions between the benzyl and the dioxime moiety also affect the rate of oxygen insertion but only to some extent.

Acknowledgement

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Appendix A. Supplementary material

CCDC 775938, 775939, 775936, 775937 and 788655 contains the supplementary crystallographic data for **3a**, **3c**, **3d**, **3e** and **4** respectively. Copies of the data can be obtained from The Cambridge Crystallographic Data Centre via deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk/. Supplementary data associated with this article can be found, in the online version.

Appendix. Supplementary material

Supplementary data related to this article can be found online, at doi:10.1016/j.jorganchem.2010.10.066.

References

[1] T. Toraya, E. Krodel, A.S. Mildvan, R.H. Abeles, Biochemistry 18 (1979) 417–426 (and references cited therein).
[2] R.H. Abeles, D. Dolphin, Acc. Chem. Res. 9 (1976) 114–120.
[3] J. Halpern, Ann. N. Y. Acad. Sci. 239 (1974) 2–21.
[4] J.S. Krouwer, B. Holmquist, R.S. Kipnes, B.M. Babior, Biochim. Biophys. Acta 612 (1980) 153–159.
[5] D.L. Anton, P.K. Tsai, H.P.C. Hogenkamp, J. Biol.Chem. 255 (1980) 4507–4510.
[6] N. Bresciani-Pahor, M. Forcolin, L.G. Marzilli, L. Randaccio, M.F. Summers, P.J. Toscano, Coord. Chem. Rev. 63 (1985) 1–125 (and references cited therein).
[7] L. Randaccio, N. Bresciani-Pahor, E. Zangrando, L.G. Marzilli, Chem. Soc. Rev. 18 (1989) 225–250.
[8] L. Randaccio, Comments Inorg. Chem. 21 (1999) 327–376.
[9] B.D. Gupta, R. Yamuna, V. Singh, U. Tiwari, Organometallics 22 (2003) 226–232.
[10] B.D. Gupta, K. Qanungo, T. Barcley, W. Cordes, J. Organomet, Chem 560 (1998) 155–161.
[11] B.D. Gupta, K. Qanungo, R. Yamuna, A. Pandey, U. Tiwari, V. Vijaikanth, V. Singh, T. Barcley, W. Cordes, J. Organomet. Chem. 608 (2000) 106–114.
[12] B.D. Gupta, S. Roy, Inorg. Chim. Acta 146 (1988) 209–221.
[13] B.D. Gupta, D. Mandal, Organometallics 25 (2006) 3305–3307.
[14] B.D. Gupta, R. Yamuna, D. Mandal, Organometallics 25 (2006) 706–714.
[15] V. Vijaikanth, B.D. Gupta, D. Mandal, S. Sekhar, Organometallics 24 (2005) 4454–4460.
[16] G. Dutta, K. Kumar, B.D. Gupta, Organometallics 28 (2009) 3485–3491.
[17] D. Mandal, B.D. Gupta, Organometallics 24 (2005) 1501–1510 (and references therein).
[18] B.D. Gupta, K. Qanungo, J. Organomet. Chem. 543 (1997) 125–134.
[19] B.D. Gupta, K. Qanungo, J. Organomet. Chem. 557 (1998) 243–249.
[20] B.D. Gupta, V. Singh, R. Yamuna, T. Barcley, W. Cordes, Organometallics 22 (2003) 2670–2678.
[21] B.D. Gupta, U. Tiwari, T. Barcley, W. Cordes, J. Organomet. Chem. 629 (2001) 83–92.
[22] B.D. Gupta, V. Vijaikanth, V. Singh, J. Organomet. Chem. 570 (1998) 1–7.
[23] B.D. Gupta, M. Roy, I. Das, J. Organomet. Chem. 397 (1990) 219–230.
[24] G. Dutta, M. Laskar, B.D. Gupta, Organometallics 27 (2008) 3338–3345.
[25] B.D. Gupta, S. Roy, Inorg. Chim. Acta 108 (1985) 261–264.
[26] M.W. Wright, T.L. Smalley, M.E. Welker, A.L. Rheingold, J. Am. Chem. Soc. 116 (1994) 6777–6791.
[27] M.W. Wright, M.E. Welker, J. Org. Chem. 61 (1996) 133–141.
[28] M. Bhuyan, M. Laskar, D. Mandal, B.D. Gupta, Organometallics 26 (2007) 3559–3567.
[29] International Tables for X-ray Crystallography, Vol. IV, Kynoch Press, Birmingham, England, 1974.
[30] SMART & SAINT Software Reference manuals, Version 6.45. Bruker Analytical X-ray Systems, Inc., Madison, WI, 2003.
[31] G.M. Sheldrick, SADABS, software for empirical absorption correction, Ver. 2.05. University of Göttingen, Göttingen, Germany, 2002.
[32] XPREP, 5.1 ed. Siemens Industrial Automation Inc., Madison, WI, 1995.
[33] G.M. Sheldrick, SHELXL97, Program for Crystal Structure Refinement. University of Göttingen, Göttingen, Germany, 2008.
[34] A. Altomare, M.C. Burla, M. Camalli, G.L. Cascarano, C. Giacovazzo, A. Guagliardi, A.G.G. Moliterni, G. Polidori, R. Spagna, J. Appl. Crystallogr 32 (1999) 115–119.
[35] C. Giannotti, C. Fontaine, A. Chiaroni, C. Riche, J. Organomet. Chem. 113 (1976) 57–65.
[36] N.W. Alcock, B.T. Golding, S. Mwesigye-Kibende, J. Chem. Soc. Dalton Trans. (1985) 1997–2000.
[37] P. Chadha, K. Mahata, B.D. Gupta, Organometallics 25 (2006) 92–98.