

This article was downloaded by:

On: 28 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713618290>

Synthesis Properties and Characterization of Di Cyclopentadienyl Vanadium Bis Dialkyl and Alkylene Dithiophosphate Complexes

Rehab Abu El Komboz^a; A. A. S. El. Khaldy^a; O. S. Nasman^a; R. M. Baraka^a

^a Chemistry Department, Al-Azhar University, Gaza, Palestine

Online publication date: 27 October 2010

To cite this Article Komboz, Rehab Abu El , Khaldy, A. A. S. El. , Nasman, O. S. and Baraka, R. M.(2002) 'Synthesis Properties and Characterization of Di Cyclopentadienyl Vanadium Bis Dialkyl and Alkylene Dithiophosphate Complexes', *Phosphorus, Sulfur, and Silicon and the Related Elements*, 177: 3, 567 — 575

To link to this Article: DOI: 10.1080/10426500210264

URL: <http://dx.doi.org/10.1080/10426500210264>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.



SYNTHESIS PROPERTIES AND CHARACTERIZATION OF DI CYCLOPENTADIENYL VANADIUM BIS DIALKYL AND ALKYLENE DITHIOPHOSPHATE COMPLEXES

Rehab Abu El Komboz, A. A. S. El. Khaldy, O. S. Nasman,
and R. M. Baraka
PO Box 1277, Chemistry Department, Al-Azhar University
Gaza, Palestine

(Received August 20, 2001)

The reaction of dichloro bis cyclopentadienyl vanadium with O,O dialkyl and alkylene dithiophosphoric acids proceed in 1:2 molar ratio in refluxing benzene to yield di cyclopentadienyl vanadium bis O,O dialkyl and alkylene dithiophosphates, $[\text{Cp}_2\text{V}(\text{S}_2\text{P}(\text{OR})_2)_2]$, where $\text{R} = \text{Et}, \text{Pr}^i, \text{Pr}^n, \text{Bu}^i, \text{Ph}$ and $[\text{Cp}_2\text{V}(\text{S}_2\text{POG}\ddot{\text{O}})_2]$ where $\text{G} = \text{CMe}_2\text{CMe}_2, \text{CH}_2\text{CEt}_2\text{CH}_2, \text{CH}_2\text{CMe}_2\text{CH}_2$. These complexes are semi solids or solids soluble in common organic solvents. Elemental analysis, molecular weight determination, magnetic susceptibility, UV-vis spectrophotometer, IR, ^1H , ^{13}C , and ^{31}P NMR spectra indicate a hexa coordinated octahedral structure.

INTRODUCTION

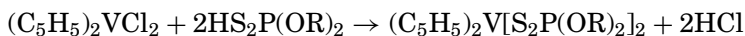
The cyclopentadienyl group, C_5H_5 , form complexes with all the d-block transition elements. It can be used as a stabilizing ligand for whole series of complexes Cp ML_n ($n = 2, 3$, or 4); these are referred to as two, three, or four-legged piano stool, the Cp group being regarded as the seat, and the other ligands as the legs. So, it is important to make complexes containing cyclopentadienyl groups and, transition metals with sulfur ligands because of diverse bonding, possibilities associated with their reactions and applications in homogeneous catalysis,¹ oil additives, pesticides,² and using as chelating reagents^{3–5} for the solvent extraction (soft base). Compared to the few literatures on vanadium

Address correspondence to A. A. S. El. Khaldy, PO Box 1277, Chemistry Department, Al-Azhar University, Gaza, Palestine. E-mail: AdnanKhaldy@hotmail.com

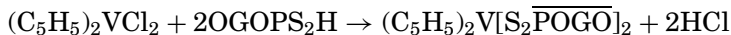
complexes of dialkyl dithiophosphoric acids,^{6,7} no work has been done on the cyclic derivatives of alkylene dithiophosphoric acids and even in cyclopentadienyl derivatives. In view of the exciting chemistry of dithio ligands with transition metal^{8,9} and nontransition metals^{10–13} it was thought of interest to synthesize a number of cyclopentadienyl vanadium bis dialkyl and alkylene dithiophosphate derivatives⁶ and characterize them by various physicochemical techniques.

RESULT AND DISCUSSION

Di-cyclopentadienyl vanadium bis dialkyl and alkylene dithiophosphate derivatives have been synthesized by the reaction of dialkyl and alkylene dithiophosphoric acids with dichloro bis cyclopentadienyl vanadium. All these reactions are quite facile and completed by refluxing the reactants in benzene for ~5–6 h.



where R = Et, Prⁱ, Prⁿ, Buⁱ, and Ph



where G = $-\text{CMe}_2\text{CMe}_2-$, $-\text{CH}_2\text{C}(\text{Et})_2\text{CH}_2-$ and $-\text{CH}_2\text{CMe}_2\text{CH}_2-$

The above complexes (I–V), acyclic ligands are green semisolids. The complexes of the cyclic ligands are greenish black semisolids except the complex (VI), which is a greenish black solid. The yields obtained are in 81–92%. These derivatives are freely soluble in common organic solvents like benzene, chloroform, and dichloromethane. The solubility in general, however, is markedly decreased on storage of the products for a few weeks. These complexes appear to be moisture-sensitive. Molecular weights (Table I), were determined by cryoscopically in benzene, the monomeric nature of these products. Attempts to crystallize the solid compounds from benzene/n-hexane mixture were unsuccessful.

IR Spectra

IR spectra of these new derivatives have been recorded in the region 4000–200 cm^{-1} and tentative assignments have been made on the basis of earlier reports.^{14,15} The band shown by the parent acids in the region 2600–2300 cm^{-1} , due to the $\nu\text{S-H}$ stretching vibrations, are absent for vanadium (IV) dithiophosphate derivatives, indicating the formation of V–S bond which appeared as new bands in the regions 370–310 cm^{-1} . The bands present in the region 1217–1066 cm^{-1} and 1097–993 cm^{-1}

TABLE I Synthesis and Physical Properties of Di Cyclopentadienyl Vanadium Bis Dialkyl and Alkylene Dithiophosphates

Comp no.	Reactants, (g) (mmol)		Molar ratio	Products, g (mmol)	Physical state	m.p °C	Yield %	Analyses% found/(calc.)		Molecular weight found/(calc.)
	Cp_2VCl_2	Dithiophosphoric acids						Vanadium	Sulfur	
1	0.16 (0.64)	$(\text{OEt})_2\text{PS}_2\text{H}$ 0.24 (1.3)	1:2	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OEt})_2]_2$ 0.53 (0.65)	Green semisolid		81	8.8 (9.2)	11.3 (11.6)	542.2 (550.94)
2	0.16 (0.64)	$(\text{OPr}^i)_2\text{PS}_2\text{H}$ 0.27 (1.2)	1:2	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OPr}^i)_2]_2$ 0.51 (0.62)	Green semisolid		82	8.21 (8.4)	10.43 (10.5)	598.74 (606.94)
3	0.28 (1.1)	$(\text{OPr}^n)_2\text{PS}_2\text{H}$ 0.47 (2.2)	1:2	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OPr}^n)_2]_2$ 0.97 (1.1)	Green semisolid		88	7.95 (8.4)	10.1 (10.5)	597.13 (606.94)
4	0.29 (1.2)	$(\text{OBu}^i)_2\text{PS}_2\text{H}$ 0.56 (2.3)	1:2	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OBu}^i)_2]_2$ 0.99 (1.15)	Green semisolid		85	7.31 (7.7)	9.61 (9.7)	654.44 (662.94)
5	0.24 (0.95)	$(\text{OPh})_2\text{PS}_2\text{H}$ 0.54 (1.9)	1:2	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OPh})_2]_2$ 0.88 (0.95)	Green semisolid		92	6.62 (6.9)	8.55 (8.6)	735.04 (742.94)
6	0.25 (0.99)	$\text{HS}_2\text{POCMe}_2\text{CMe}_2\text{O}$ 0.42 (1.97)	1:2	$\text{Cp}_2\text{V}[\text{S}_2\text{POCMe}_2\text{CMe}_2\text{O}]_2$ 0.90 (0.99)	Greenish black solid	179	91	7.9 (8.4)	10.25 (10.6)	597.54 (602.94)
7	0.47 (1.9)	$\text{HS}_2\text{POCH}_2\text{CEt}_2\text{CH}_2\text{O}$ 0.30 (1.5)	1:2	$\text{Cp}_2\text{V}[\text{S}_2\text{POCH}_2\text{CEt}_2\text{CH}_2\text{O}]_2$ 0.78 (0.95)	Greenish black semisolid		82	7.85 (8.1)	9.74 (10.1)	627.04 (630.94)
8	0.22 (0.87)	$\text{HS}_2\text{POCH}_2\text{CMe}_2\text{CH}_2\text{O}$ 0.39 (1.7)	1:2	$\text{Cp}_2\text{V}[\text{S}_2\text{POCH}_2\text{CMe}_2\text{CH}_2\text{O}]_2$ 0.71 (0.85)	Greenish black semisolid		84	8.94 (8.9)	10.68 (11.1)	570.84 (574.94)

TABLE II IR Spectral Data (cm^{-1}) of Di Cyclopentadienyl Vanadium Bis Dialkyl and Alkylene Dithiophosphates

Comp no.	$\nu(\text{P})\text{--O--C})$	$\nu(\text{P--O--C})$	Ring vibration	$\nu(\text{P}=\text{S})$	$\nu(\text{P--S})$	$\nu(\text{M--S})$
1	1155.9 s	1020.4 s		630.5 s	540 m	370.9 m
2	1173.1 s	1097.4 s		642.3 s	570 m	340.0 m
3	1215.1 s	993.3 s		659.1 s	585 s	354.0 m
4	1217.0 s	1045.3 s		666.2 s	575 m	320.0 m
5	1180.3 s	1050.2 s		624.9 s	574 m	335.3 m
6	1137.9 s	1010.6 s	960.5 m	669.3 s	569 s	310.0 m
7	1066.6 s	1018.3 s	997.1 m	670.4 s	530 m	342.0 m
8	1070 s	1012.4 s	950.2	650.3 s	528 m	331.4 m

s = sharp; m = medium.

have been assigned to $\nu[(\text{P})\text{--O--C}]$ and $\nu[\text{P--O--C}]$ stretching vibrations respectively. A band in the region $971\text{--}950\text{ cm}^{-1}$ is most probably due to dioxaphospholane and dioxaphorinane rings.^{16,17} A sharp bands present in the region $670\text{--}624\text{ cm}^{-1}$ are due to $\nu(\text{P}=\text{S})$, which show shifting of $27\text{--}14\text{ cm}^{-1}$ toward lower wave numbers. This shifting is probably due to comparatively strong bidentate chelation in vanadium complexes. The band of medium intensities in the region $575\text{--}528\text{ cm}^{-1}$ may be attributed to $\nu(\text{P--S})$ asymmetric and symmetric vibrations. A sharp band present in the region $756\text{--}825\text{ cm}^{-1}$ is due to $\nu\text{ Cp--V}$ asymmetric or symmetric vibrations, which is attributed to trans position of C_5H_5 .¹⁸ Details regarding the individual peaks have been included in Table II.

¹H NMR Spectra

The ¹H NMR spectra of di cyclopentadienyl vanadium bis dialkyl and alkylene dithiophosphates recorded in CDCl_3 , show the expected characteristic resonance due to alkoxy and glycoxy groups (Table III). The cyclopentadienyl protons attached to vanadium appear as singlet in the range $2.4\text{--}1.55\text{ ppm}$. The singlet peak observed at $3.1\text{--}3.5\text{ ppm}$ in the parent dithiophosphoric acids have been assigned for SH proton,¹⁹ and are absent in the spectra of vanadium dithiophosphate derivatives, indicating the deprotonation of SH group and formation of V--S bonds.^{20,21}

¹³C NMR Spectra

¹³C NMR spectra of these complexes recorded in CDCl_3 (Table IV) show very small chemical shifts to those obtained for the parent dithiophosphoric acids and indicate no substantial change in structural features.

TABLE III ^1H and ^{31}P NMR Spectral Data δ ppm of Di Cyclopentadienyl Vanadium Bis Dialkyl and Alkylene Dithiophosphates

Comp no.	Compound	^1H chemical shift in δ ppm in CDCl_3	^{31}P chemical shift in δ ppm in CDCl_3 (parent acids)
1	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OEt})_2]_2$	1.15–1.60, t, ($J = 6$ Hz), 12H (Me) 4.23–4.45, m, 8H (CH_2O) 2.01–2.23, s, 10H (C_5H_5)	86.03 (86.3)
2	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OPr}^i)_2]_2$	1.16–1.70, s, 24H (Me) 2.25–2.16, s, 10H (C_5H_5) 4.88–5.10, m, 2H (CHO)	82.3 (85.5)
3	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OPr}^n)_2]_2$	0.85, t, ($J = 7$ Hz), 12H (Me) 1.7, s, 10H (C_5H_5) 2.01, m, 8H (CH_2) 4.3, m, 8H (CH_2O)	85.87 (86.3)
4	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OBu}^i)_2]_2$	1.6, d, ($J = 6$ Hz), 24H (Me) 1.73, s, 10H (C_5H_5) 2.3, m, 4H (CH) 4.4, m, 8H (CH_2O)	86.18 (85.7)
5	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OPh})_2]_2$	7.2, m, 10H (PhO) 2.4, s, 10H (C_5H_5)	78.6 (78.2)
6	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OCMe}_2\text{CMe}_2\text{O})_2]_2$	1.95, m, 24H (Me) 2.02, m, 10(C_5H_5)	101.12 (93.3)
7	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OCH}_2\text{CET}_2\text{CH}_2\text{O})_2]_2$	0.90, t, ($J = 7$ Hz), 12H (Me) 1.97, s, 10H (C_5H_5) 1.8, m, 8H (CH_2) 4.32, qd, 8H (CH_2O)	82.98 (78.7)
8	$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OCH}_2\text{CMe}_2\text{CH}_2\text{O})_2]_2$	1.1, s, 12H (Me) 1.55, s, 10H (C_5H_5) 4.45, qd, 8H (CH_2O)	81.02 (77.3)

TABLE IV ^{13}C NMR Spectral Data of Some Representative Derivatives of Di Cyclopentadienyl Vanadium Bis Dialkyl and Alkylene Dithiophosphates (cm^{-1})

Comp no.	CH_3	CH_2	^{13}C chemical shift, in ppm		
			C	OC	C_5H_5
1	15.7 s	64.90, d,			129.4
2	10.5 s	($J = 6.1$) Hz	71.16, d, ($J = 7.3$) Hz		129.3
3	12.1 s	25.57, d,	70.6, d, ($J = 6.1$) Hz		128.7
6	25.2, d, ($J = 6.1$) Hz	($J = 6.1$) Hz	23.6, d, ($J = 6.1$) Hz		127.5
7	7.26, d, ($J = 6.1$) Hz	23.1, d, ($J = 6.1$) Hz	23.6, d, ($J = 7.3$) Hz	37.8 s	123 s
8	27.58 s		28.91, d, ($J = 6.1$) Hz	39.24 s	85.88 s

d = doublet; t = triplet; m = multiplet.

³¹P NMR Spectra

³¹P NMR spectra of di cyclopentadienyl vanadium bis dialkyl and alkylene dithiophosphates (Table III) show that compounds give a single peak when freshly prepared. The ³¹P shifts for dialkyl dithiophosphate complexes are almost in the same range as those for the parent acids and gave very small shift (2.3–0.27 ppm).

The phosphorus atom in cyclic derivatives is more shielded (7.82–3.72 ppm). The phosphorus chemical shifts of various metal dithiophosphate compounds have been reported in recent years and an attempt has also been made by Glidewell²¹ who suggested that phosphorus deshielding increase in the order monodentate attachment < bidentate attachment < anion complexes. Our attempts to correlate the same in metal dialkyl and alkylene dithiophosphates in this article with Glidewell have been successful. The dithio ligand behaves as bidentate.

MAGNETIC MOMENTS

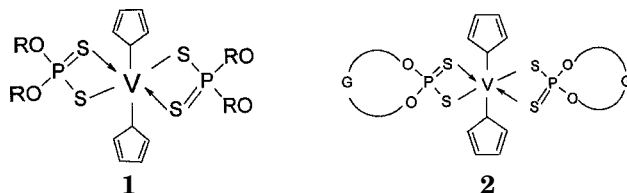
The complex $\text{Cp}_2\text{V}[\text{S}_2\overline{\text{POCMe}_2\text{CMe}_2\text{O}}]_2$ is paramagnetic with magnetic moment value 1.05 b.m., which is lower than the theoretically calculated value 1.73 b.m., for one unpaired electron. The value correspond to one electron anticipated for octahedral geometry of vanadium (IV) complexes.²²

ELECTRONIC SPECTRA

The electronic spectra of these derivatives have been recorded in CHCl_3 solution as shown in Table V. All the di cyclopentadienyl bis dialkyl and alkylene dithiophosphates absorb strongly in the region 512–378 nm, corresponding to $\pi \rightarrow \pi^*$ transition. Other rather weak absorption's

TABLE V UV Spectral Data for Some Representative Derivatives of Di Cyclopentadienyl Vanadium Bis Dialkyl and Alkylene Dithiophosphate (cm^{-1})

Compound	Abs	λ_{max}
$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OPr}^n)_2]_2$	0.0005	512
	3.52	215
$\text{Cp}_2\text{V}[\text{S}_2\text{P}(\text{OBu}^i)_2]_2$	3.6	249
	5	230.5
$\text{Cp}_2\text{V}[\text{S}_2\overline{\text{POCMe}_2\text{CMe}_2\text{O}}]_2$	5	233.5
	0.63	378
$\text{Cp}_2\text{V}[\text{S}_2\overline{\text{POCH}_2\text{CEt}_2\text{CH}_2\text{O}}]_2$	3.43	246



FIGURES 1 and 2 Octahedral geometry for di cyclopentadienyl vanadium bis dialkyl and alkylene dithiophosphates. R = Me, Et, Prⁱ, Prⁿ, Buⁱ, and Ph and Cp₂V[S₂POGO]₂ where G = CMe₂CMe₂, CH₂CEt₂CH₂ and CH₂CMe₂CH₂.

occur at 249–215 nm, which may be due to $n \rightarrow \pi^*$ transition. The dialkyl dithiophosphoric acids also absorb in these regions.

STRUCTURAL ELUCIDATION

Our observations based on spectral studies, such as IR, NMR (¹H, ¹³C, and ³¹P), magnetic susceptibility studies, molecular weight determinations, and UV spectrophotometer indicate the bidentate behavior of ligand and monomeric nature of these complexes. According to this, an *trans*-octahedral geometry's (Figures 1 and 2) can be suggested for vanadium (IV) dialkyl and alkylene dithiophosphate complexes because in IR spectra one stretching have been obtain in the range 850–756, but *cis*-octahedral have two stretching vibrations.¹⁸

EXPERIMENTAL

Stringent precautions were taken to exclude moisture from the experimental set up. Solvents (benzene, chloroform, and n-hexane) were dried by standard methods. Alcohols and glycols were purified by distillation before use. Cp₂VCl₂ (Aldrich) was used as such. The dialkyl dithiophosphoric acids were prepared using literature method.¹⁸ Vanadium was estimated gravimetrically as metal penta oxide, and sulfur was estimated as barium sulfate²⁰ (Messenger's method). Molecular weights were determined cryoscopically in benzene.

Magnetic susceptibility measurements were carried out using a Faraday balance MSB-1 Ltd. IR spectra were recorded as KBr discs or Nujol mulls on Shimadzu FTIR-8201 PC Spectrophotometer. NMR spectra were recorded in CDCl₃ using a Jeol JNM-L A 300-NMR spectrophotometer with TMS as an internal reference for ¹H, ¹³C. ³¹P NMR spectra were recorded in CDCl₃ using 85% H₃PO₄ as external reference on the same instrument.

Reaction of Dichloro Bis Cyclopentadienyl Vanadium (IV) with Diethyl Dithiophosphoric Acids

Diethyl dithiophosphoric acids (0.24 g, 1.3 mmol) in benzene (20 ml) was added dropwise with constant stirring to dichloro bis cyclopentadienyl vanadium(IV) (0.16 g, 0.64 mmol) in (20 ml) benzene. The mixture was refluxed for ~6 h until liberation of hydrogen chloride gas ceased. Solvent was removed under reduced pressure, the desired product was purified by washing several times with n-hexane, and dried under vacuum.

Reaction of Dichloro Bis Cyclopentadienyl Vanadium with Neopentylene Dithiophosphoric Acids

To benzene (20 ml) solution of neopentylene dithiophosphoric acids (0.47 g, 0.19 mmol) was added dropwise with constant stirring an equivalent amount of dichloro bis cyclopentadienyl vanadium (0.22 g, 0.87 mmol) in (20 ml) benzene. The mixture was refluxed for ~6 h until liberation of hydrogen chloride gas ceased. The benzene solution dried under reduced pressure, the desired products was purified by washing several times with n-hexane and dried under vacuum.

REFERENCES

- [1] V. K. Jain, *Transition Met. Chem.*, **18**, 314 (1993).
- [2] H. Koub, *Agric. Biol. Chem.*, **29**, 43 (1965).
- [3] A. I. Busev, M. I. Ivanyutin, and T. R. Kom, *Anal. Khim. Akad. Nauk SSSR*, **11**, 172 (1960); *Chem. Abstr.*, **55**, 24381 (1961).
- [4] L. S. Levin, Y. U. M. Yukhin, I. A. Bykhovskaya, I. A. Vorsina, and V. V. Sergeeva, *Ser. Khim. Nauk*, **5**, 83 (1973); *Chem. Abstr.*, **80**, 7593 (1974).
- [5] L. S. Levin, V. V. Sergeeva, and I. A. Vorsina, *Ser. Khim. Nauk*, **1075**, 2 (1975); *Chem. Abstr.*, **83**, 6625 (1975).
- [6] C. Furlani, A. A. G. Tomlinson, P. Porta, and A. Sgamelloni, *J. Chem. Soc.*, **A**, 2929 (1970).
- [7] R. Mercier, J. Douglade, and B. Viard, *Acta Cryst.*, **B37**, 949 (1981).
- [8] A. A. S. El. Khaldy and J. R. Durig, *Phosphorus, Sulfur, and Silicon*, **67**, 1–7 (1998).
- [9] A. A. S. El. Khaldy and J. R. Durig, *Phosphorus, Sulfur, and Silicon*, **165**, 197–204 (2000).
- [10] H. P. S. Chauhan, G. Srivastava, and R. C. Mehrotra, *Phosphorus and Sulfur*, **17**, 161 (1983).
- [11] R. C. Mehrotra, G. Srivastava, and B. P. S. Chauhan, *Coord. Chem. Rev.*, **55**, 207 (1984).
- [12] R. J. Rao, G. Srivastava, R. C. Mehrotra, B. S. Saraswat, and J. Mason, *Polyhedron*, **3**, 485 (1984).
- [13] R. J. Rao, G. Srivastava, and R. C. Mehrotra, *Phosphorus and Sulfur*, **25**, 183 (1985).
- [14] R. J. Rao, G. Srivastava, and R. C. Mehrotra, *J. Organomet. Chem.*, **258**, 155 (1983).

- [15] H. P. S. Chauhan, C. P. Bhasin, G. Srivastava, and R. C. Mehrotra, *Phosphorus and Sulfur*, **15**, 99 (1983).
- [16] R. A. Y. Jones and A. R. Katritzky, *J. Chem. Soc.*, **4376** (1960).
- [17] J. Cason, W. N. Baxter, and W. DeAcetis, *J. Org. Chem.*, **24**, 247 (1959).
- [18] K. Nakamoto, *Infrared Spectra of Inorganic and Coordination Compounds* (Wiley, New York, 1970), 2nd ed., p. 215.
- [19] R. K. Gupata, A. K. Rai, R. C. Mehrotra, V. K. Jain, B. F. Hoskins, and E. R. T. Tibkin, *Inorg. Chem.*, **24**, 9280 (1985).
- [20] H. P. S. Chauhan, C. P. Bhasin, G. Srivastava, and R. C. Mehrotra, *Phosphorus and Sulfur*, **15**, 49 (1983).
- [21] C. Glidewell, *Inorg. Chim. Acta*, **25**, 159 (1987).
- [22] S. F. A. Kettle, *Physical Inorganic Chemistry* (Spektrum Academic, New York, 1998), p. 194.