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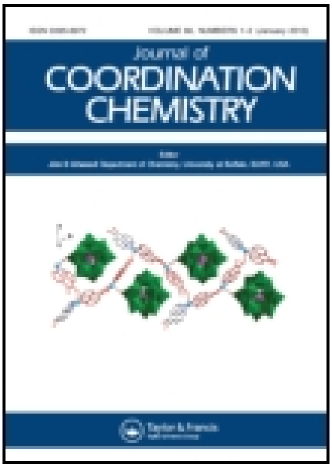

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Cadmium(II) complexes with phosphine tellurides: synthesis and multinuclear (^{31}P, ^{125}Te, and ^{113}Cd) NMR characterization in solution

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Cadmium(II) complexes with phosphine tellurides: synthesis and multinuclear (^{31}P, ^{125}Te, and ^{113}Cd) NMR characterization in solution

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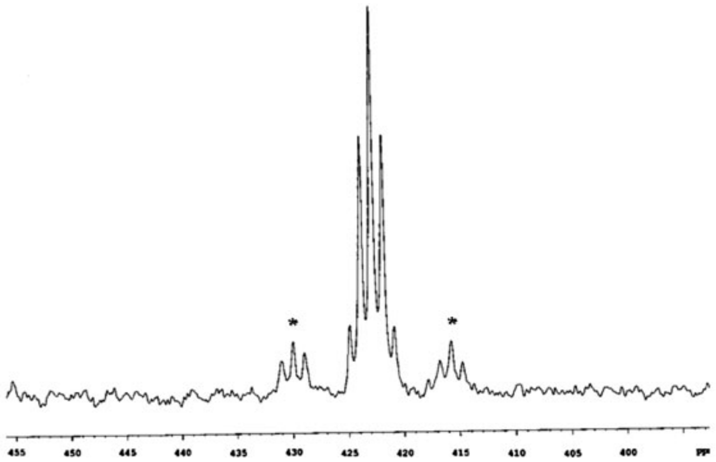

^{113}Cd NMR spectrum of complex **2** in dichloromethane at 188 K (*: tellurium satellites).

New cadmium(II) complexes with phosphine telluride ligands of the type $CdX_2(R_3PTe)_n$ [X = ClO_4^-, $n = 4$: R = *n*-Bu (**1**), Me_2N (**2**), $C_5H_{10}N$ (**3**), C_4H_8N (**4**) or OC_4H_8N (**5**); X = Cl^-, $n = 2$: R = *n*-Bu (**6**), Me_2N (**7**), $C_5H_{10}N$ (**8**), C_4H_8N (**9**) or OC_4H_8N (**10**)] have been synthesized and characterized by elemental analyses, IR and multinuclear (^{31}P, ^{125}Te, and ^{113}Cd) NMR spectroscopy. In particular, the solution structures of these complexes were confirmed by ^{113}Cd NMR at low temperature, which displays a quintuplet for each of the perchlorate complexes and a triplet for each of the chloride complexes due to coupling with four and two equivalent phosphorus atoms, respectively, indicating a four-coordinate tetrahedral geometry for the metal center. These multiplet features were further accompanied by one bond Te–Cd couplings, clearly showing that the ligand is coordinated to the metal through tellurium. The results are discussed and compared with those obtained for closely related phosphine chalcogenide analogs.

Keywords: Phosphine telluride; Cadmium complex; ^{31}P, ^{125}Te, and ^{113}Cd NMR; $^1J_{Cd-Te}$ coupling

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1. Introduction

Phosphine chalcogenides R_3PE ($E = O$, S, Se or Te) are of considerable interest due to their selective complexation properties towards 'hard/soft' metal cations [1–5]. In addition to the extensive studies carried out on their metal complexes, they are one of the most common chalcogenide sources used in colloidal nanocrystal syntheses [6]. Certain metal complexes of this class of compounds are suitable single-source precursors for the production of thin semiconducting films of metal chalcogenides, for example ME ($M = Cd$, Zn or Hg; $E = S$ or Se) [7, 8]. Despite the fact that several studies have been reported on phosphine oxides, sulfides, and to a lesser extent selenides [9–12], the coordination chemistry of phosphine telluride ligands is much less explored and only a few complexes with these ligands have been described [13–16]. This is not unexpected since coordination promotes P=E cleavage and can lead to complete cleavage to form a tertiary phosphine and a chalcogenide that is incorporated in the metal complex with a cleavage rate increasing in the order $S < Se < Te$ [17–20]. Like Cd–Se, compounds containing the Cd–Te bond are of special significance for NMR studies since both ^{125}Te (7.07% natural abundance) and ^{113}Cd (12.3% natural abundance) have spin $I = ½$ and their resonances are readily accessible with modern NMR instrumentation.

In continuation of our interest on the coordination chemistry of monodentate phosphine chalcogenide ligands of type $R_3P(E)$ towards hard [21, 22] and soft [23] metal ions and the influence which might arise from the presence of the chalcogenide and/or the different substituents attached to phosphorus of the ligand [22], we describe herein the synthesis and multinuclear (^{31}P, ^{125}Te, and ^{113}Cd) NMR studies of cadmium complexes with different phosphine tellurides. These are the first examples of cadmium complexes with monodentate phosphine telluride ligands. The new complexes were characterized by elemental analysis, IR, and NMR spectroscopy.

2. Experimental

2.1. *General experimental procedures*

Cadmium salts, such as perchlorate hydrate (Ventron, 99.9%) and chloride anhydrous (Merck, 99%) were used as received. All preparations were carried out under nitrogen in solvents dried by standard techniques [24] and stored over molecular sieves. NMR spectra were recorded on a Bruker Avance-III 300 spectrometer in $CDCl_3$ or CD_2Cl_2 as solvents, ^{31}P at 121 (85% H_3PO_4), ^{1}H at 300 (TMS), ^{113}Cd at 66.5 ($Cd(ClO_4)_2$), and ^{125}Te at 94.7 MHz (Me_2Te). The IR spectra were measured on a YL2000 FT-IR spectrometer. The ligands *n*-Bu_3PTe [25], $(Me_2N)_3PTe$ [26], $Pyrr_3PTe$ ($Pyrr = C_4H_8N$) [27], Pip_3PTe ($Pip = C_5H_{10}N$) [27], and Mor_3PTe ($Mor = OC_4H_8N$) [27] were prepared according to literature methods.

2.2. *Preparation of 1–5*

Caution: cadmium perchlorate and its complexes are potentially explosive and should be handled with extreme care and in small quantities!

In a typical reaction, **1**–**5** were prepared from the hydrated cadmium perchlorate and the ligand in the presence of triethylorthoformate following a method first described by Van Leeuwen *et al.* [28]; the hydrate cadmium perchlorate (1 mM) was dissolved in ethanol and an appropriate amount of triethylorthoformate was added. After 2 h, a filtered solution of R_3PTe (5 mM) in anhydrous dichloromethane (5 mL) was added dropwise and the solution was stirred at room temperature for 4 h. The reaction mixture was then concentrated by distilling off the solvents at reduced pressure. Addition of anhydrous diethyl ether led to the precipitation of the complex, which was collected, washed with diethyl ether, and dried *in vacuo* for several hours.

2.2.1. Cd[*n*-Bu₃PTe]₄(ClO₄)₂ (1). Gray solid, yield = 73%, m.p. = 149 °C. IR: 449 cm^{-1} ($\nu_{P=Te}$). Anal. Calcd for $C_{48}H_{108}CdCl_2O_8P_4Te_4$: C, 35.35; H, 6.67. Found: C, 34.7; H, 6.88%.

2.2.2. Cd[(Me₂ N)₃PTe]₄(ClO₄)₂ (2). Deep green solid, yield = 45%, m.p. = 120 °C. IR: 500 cm^{-1} ($\nu_{P=Te}$). Anal. Calcd for $C_{24}H_{72}CdCl_2N_{12}O_8P_4Te_4$: C, 19.55; H, 4.92; N, 11.40. Found: C, 20.14; H, 5.04; N, 11.67%.

2.2.3. Cd[Pip₃PTe]₄(ClO₄)₂ (3). Gray creamy solid, yield = 50%. IR: 474 cm^{-1} ($\nu_{P=Te}$). Anal. Calcd for $C_{60}H_{120}CdCl_2N_{12}O_8P_4Te_4$: C, 36.86; H, 6.19; N, 8.60. Found: C, 36.78; H, 6.30; N, 8.84%.

2.2.4. Cd[Pyrr₃PTe]₄(ClO₄)₂ (4). Gray creamy solid, yield = 50%. IR: 513 cm^{-1} ($\nu_{P=Te}$) Anal. Calcd for $C_{48}H_{96}CdCl_2N_{12}O_8P_4Te_4$: C, 32.26; H, 5.41; N, 9.41. Found: C, 32.47; H, 5.72; N, 9.48%.

2.2.5. Cd[Mor₃PTe]₄(ClO₄)₂ (5). Deep yellow solid, yield = 80%, m.p. = 114 °C (decomp.) IR: 499 cm^{-1} ($\nu_{P=Te}$). Anal. Calcd for $C_{48}H_{96}CdCl_2N_{12}O_{20}P_4Te_4$: C, 29.13; H, 4.89; N, 8.49. Found: C, 28.51; H, 4.43; N, 7.93%.

2.3. *Preparation of 6–10*

To a stirred solution of cadmium chloride (1 mM) in ethanol (30 mL), a filtered solution of R_3PTe (2.5 mM) in dry dichloromethane (5 mL) was added dropwise. The reaction mixture was stirred for 3 h and the solvent was evaporated *in vacuo*. The solid obtained was washed with diethyl ether and dried *in vacuo*.

2.3.1. CdCl₂[*n*-Bu₃PTe]₂ (6). Pale yellow powder, yield = 80%, m.p. = 88 °C. IR: 454 cm^{-1} ($\nu_{P=Te}$). Anal. Calcd for $C_{24}H_{54}CdCl_2P_2Te_2$: C, 34.19; H, 6.46. Found: C, 33.50; H, 6.73%.

2.3.2. $CdCl_2[(Me_2N)_3PTe]_2$ (7). Black solid, yield = 72%, m.p. = 94 °C. IR: ($\nu_{P=Te}$) 504 cm^{-1} Anal. Calcd for $C_{12}H_{36}CdCl_2N_6P_2Te_2$: C, 18.84; H, 4.74; N, 10.99. Found: C, 18.25; H, 4.43; N, 10.48%.

2.3.3. $CdCl_2[Pip_3PTe]_2$ (8). Gray solid, yield 50%, m.p. = 106 °C. IR: 478 cm^{-1} ($\nu_{P=Te}$). Anal. Calcd for $C_{30}H_{60}CdCl_2N_6P_2Te_2$: C, 35.84; H, 6.02; N, 8.36. Found: C, 34.99; H, 5.80; N, 7.87%.

2.3.4. $CdCl_2[Pyrr_3PTe]_2$ (9). Gray solid, yield 40%. m.p. = 88 °C. IR: 516 cm^{-1} ($\nu_{P=Te}$) Anal. Calcd for $C_{24}H_{48}Cl_2N_6P_2CdTe_2$: C, 31.29; H, 5.25; N, 9.12. Found: C, 30.99; H, 5.36; N, 8.76%.

2.3.5. $CdCl_2[Mor_3PTe]_2$ (10). Pale yellow powder, yield 70% m.p. = 130 °C. IR: 494 cm^{-1} ($\nu_{P=Te}$). Anal. Calcd for $C_{24}H_{48}Cl_2N_6O_6P_2CdTe_2$: C, 28.34; H, 4.76; N, 8.26. Found: C, 29.99; H, 5.36; N, 7.76%.

3. Results and discussion

3.1. *Synthesis*

The cadmium complexes $Cd[n\text{-}Bu_3PTe]_4(ClO_4)_2$ (**1**), $Cd[(Me_2N)_3PTe]_4(ClO_4)_2$ (**2**), $Cd[Pip_3PTe]_4(ClO_4)_2$ (**3**), $Cd[Pyrr_3PTe]_4(ClO_4)_2$ (**4**), $Cd[Mor_3PTe]_4(ClO_4)_2$ (**5**), $CdCl_2(n\text{-}Bu_3PTe)_2$ (**6**), $CdCl_2((Me_2N)_3PTe)_2$ (**7**), $CdCl_2(Pip_3PTe)_2$ (**8**), $CdCl_2(Pyrr_3PTe)_2$ (**9**), and $CdCl_2(Mor_3PTe)_2$ (**10**) were prepared by a method well known in the literature [25]. They are soluble in dichloromethane and chloroform. The characterization of these complexes was based particularly on their NMR data and comparison with those of the free ligands (table 1).

To our knowledge, these are the first monodentate phosphine telluride complexes of cadmium since the only reported cadmium complex of this class of compounds, $Cd[N(TePPr^i_2)_2]_2$, was prepared with a bidentate ligand by Chivers *et al.* [13].

Table 1. NMR data (δ/ppm and *J*/Hz) for **1**–**10** in CD_2Cl_2 at 188 K.

Complex	^{31}P ($\Delta^{31}P$)[a]	^{125}Te ($\Delta^{125}Te$)[b]	^{113}Cd	$^{1}J_{P-Te}$ ($\Delta^{1}J_{P-Te}$)[c]	$^{1}J_{Cd-Te}$	$^{2}J_{Cd-P}$
1	2.09 (15)	−715 (91)	432	1317 (339)	912	52.0
2	44.5 (11)	−630 (200)	422	1674 (438)	946	66.7
3	41.8 (8.1)	−550 (240)	382	1710 (273)	1040	68.2
4	20.4 (11)	−594 (190)	427	1632 (328)	878	60.7
5	43.8 (10)	–	400	–	–	52.2
6	−1.79 (13)	−720 (86)	508	1287 (370)	–	50.2
7	45.7 (10)	−728 (102)	484	1617 (495)	–	57.2
8	40.6 (8.4)	−782 (106)	481	1587 (397)	–	52.9
9	19.4 (8.2)	−665 (119)	490	1580 (381)	–	56.1
10	51.6 (3.1)	–	490	–	–	–

[a] $\Delta^{31}P = |\delta^{31}P_L - \delta^{31}P_{comp}|$.
[b] $\Delta^{125}Te = |\delta^{125}Te_L - \delta^{125}Te_{comp}|$.
[c] $\Delta^{1}J_{P-Te} = |^{1}J_{(P-Te)L} - {}^{1}J_{(P-Te)comp}|$.

Complexes **1**–**10** are air sensitive but stable for a few months when stored under a dry atmosphere, except the morpholine derivatives **5** and **10** which decompose rapidly. Complexes **1** and **8** are the most stable (up to 10 months). All the complexes decompose in solution within a few days liberating elemental tellurium. Due to the air sensitivity of the complexes, attempts to obtain crystals suitable for X-ray analysis were avoided and purification was limited to washing several times with anhydrous ether.

3.2. *Spectroscopic characterization*

The infrared spectrum of each complex shows strong bands at 430–500 cm^{-1}, attributed to $\nu_{P=Te}$ which are shifted by ~13 cm^{-1} towards lower wavenumbers compared to those of corresponding free ligands. The coordination shift is attributed to a lowering of the P=Te bond order in the complex. The small shift compared to that of P=O and P=S absorptions [29] in the corresponding complexes is similar to the selenide analogs [23]. This is reasonable since the vibrations involving the relatively heavy tellurium (or selenium) would be less sensitive to coordination than those with the lighter phosphorus, oxygen or sulfur. Important bands observed at *ca.* 1084 cm^{-1} with a weak stretch at 620 cm^{-1} for the perchlorate derivatives are assigned to $\nu(ClO_4)$.

The NMR spectra of **1**–**10** were recorded in CD_2Cl_2 solutions and the data obtained from these spectra are summarized in table 1.

At room temperature, the ^{31}P-{^{1}H} NMR spectrum of each complex showed one signal, exhibiting $^{1}J(^{125}\text{Te}, ^{31}\text{P})$ satellites with magnitudes smaller than those of the free ligands. This signal is shifted to lower frequency compared with that of the free ligand for the complexes prepared with $(Me_2N)_3PTe$ and Pip_3PTe and to higher frequencies for complexes prepared with *n*-Bu_3PTe. At low temperature, the ^{31}P NMR spectrum of each complex

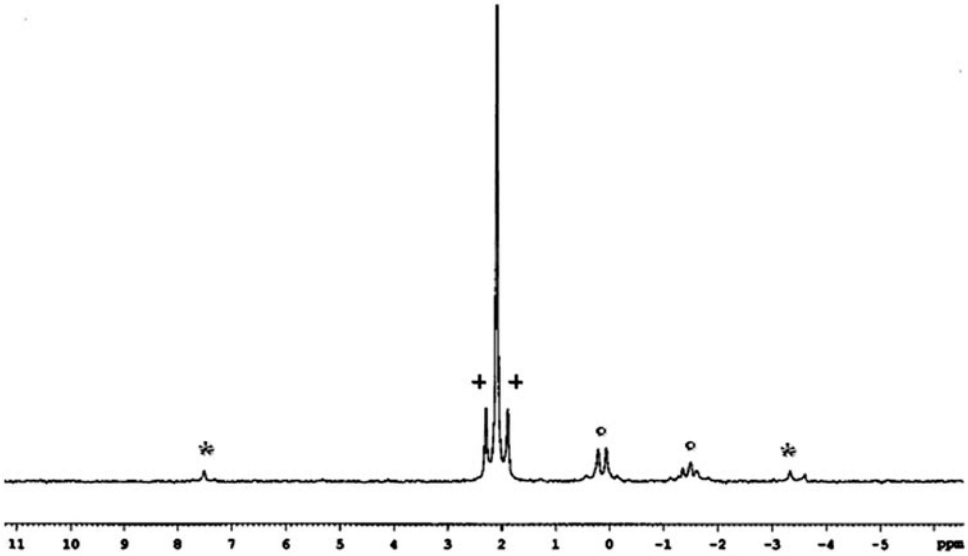

Figure 1. ^{31}P-{^{1}H} NMR spectrum of **1** in dichloromethane at 188 K (*: Te satellites, +: Cd satellites and o: impurity).

showed a central signal which is flanked on one hand by two satellites of equal intensities, (~12.5% each) arising from the two bond cadmium–phosphorus coupling ($^2J^{111/113}$Cd–^{31}P), and on the other hand, by two tellurium satellites ($^1J_{\text{P–Te}}$) (figure 1). For the perchlorate complexes, the magnitude of ^2J($^{111/113}$Cd–^{31}P) coupling follows the order **5** < **1** < **4** < **2** < **3** (table 1).

The ^{125}Te NMR signals of bound ligands are shifted to higher frequency compared with those of the free ligands. These signals were converted at low temperature into a doublet arising from the tellurium–phosphorus coupling; each signal of this doublet is flanked by two satellites due to coupling with cadmium nuclei (see figure 2). The magnitudes of the ^1J (P–Te) coupling are about 20% smaller than those of the free ligands, which are in good agreement with the literature data for related complexes [16]. The difference in the ^{125}Te chemical shift between free and bound ligands is more important than that observed in ^{31}P NMR spectra, confirming coordination to the metal center through tellurium [4, 5].

In order to gain insight into the structure of these complexes in solution, ^{113}Cd NMR spectroscopy was used. At room temperature, the ^{113}Cd NMR spectrum for each complex showed a large signal. The latter was converted at low temperature into a quintet for the perchlorate complexes in which each signal is flanked by two satellites arising from cadmium–tellurium coupling (see figure 3). The quintet is due to the coupling of the cadmium nucleus with four equivalent phosphorus atoms, showing a stoichiometry of $CdL_4(ClO_4)_2$. Further evidence in support of this assignment was obtained from the IR spectra of these complexes, which showed v(ClO_4) as a sharp single stretch at *ca.* 1084 and a weak stretch at 620 cm^{-1}, respectively. For the chloride complexes, the cadmium signal is converted at low temperature into a triplet due to coupling with two equivalent phosphorus atoms, consistent with the formula CdL_2Cl_2 (see figures 4 and 5).

When comparing complexes with the same stoichiometry (i.e. CdL_2X_2 or $CdL_4(ClO_4)_2$), the chemical shifts and coupling constants listed in table 1 suggest that the ligands in the perchlorate complexes **1**–**5** bind more strongly to the cadmium center than in corresponding chloride derivatives **6**–**10**.

The ^1J(^{113}Cd–Te) and ^2J($^{111/113}$Cd–^{31}P) couplings in **1**–**5**, from 878–1040 and 52–68 Hz, are higher than those observed for the related chelate cadmium complex of 786 and 38 Hz, respectively [13]. In addition, the ^2J($^{111/113}$Cd–^{31}P) coupling constant for **2** is considerably

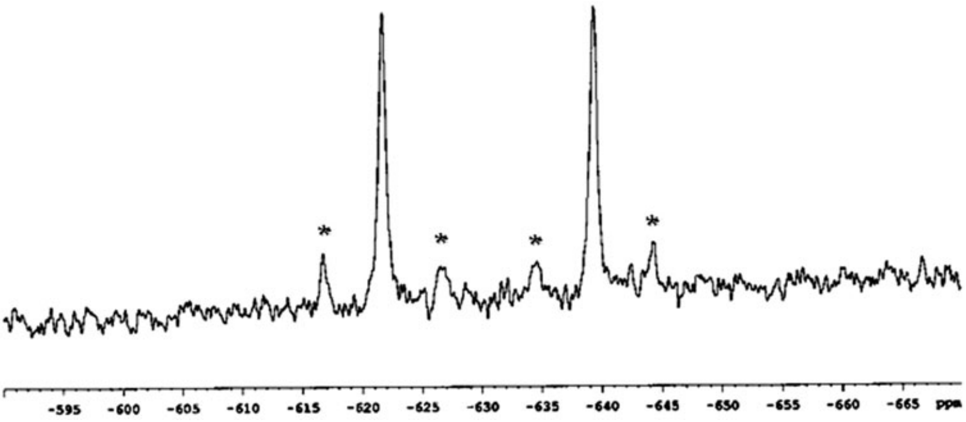

Figure 2. ^{125}Te NMR spectrum of **2** in dichloromethane at 188 K (*: $^{111/113}$Cd satellites).

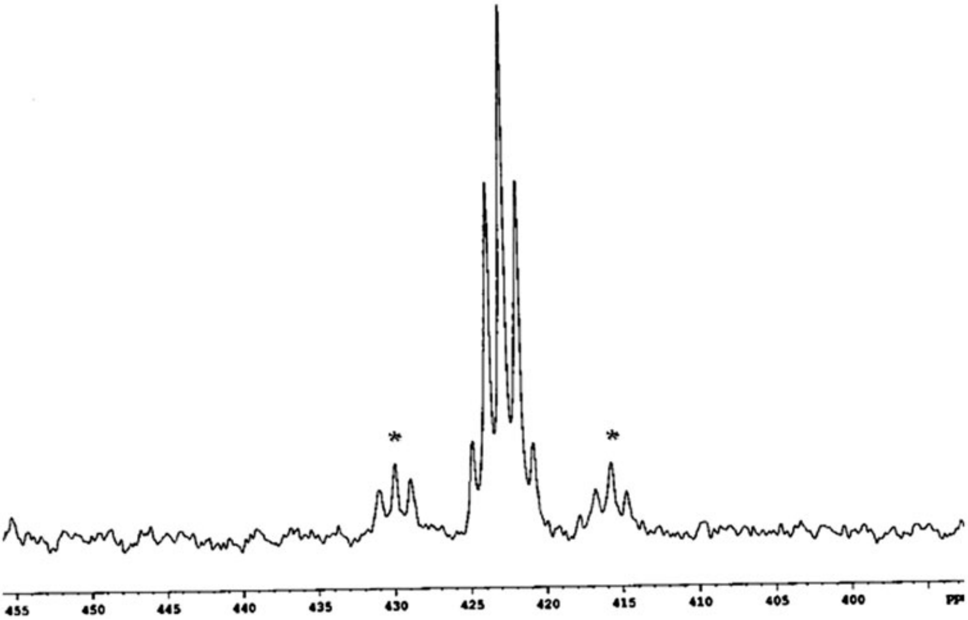

Figure 3. ^{113}Cd NMR spectrum of **2** in dichloromethane at 188 K (*: tellurium satellites).

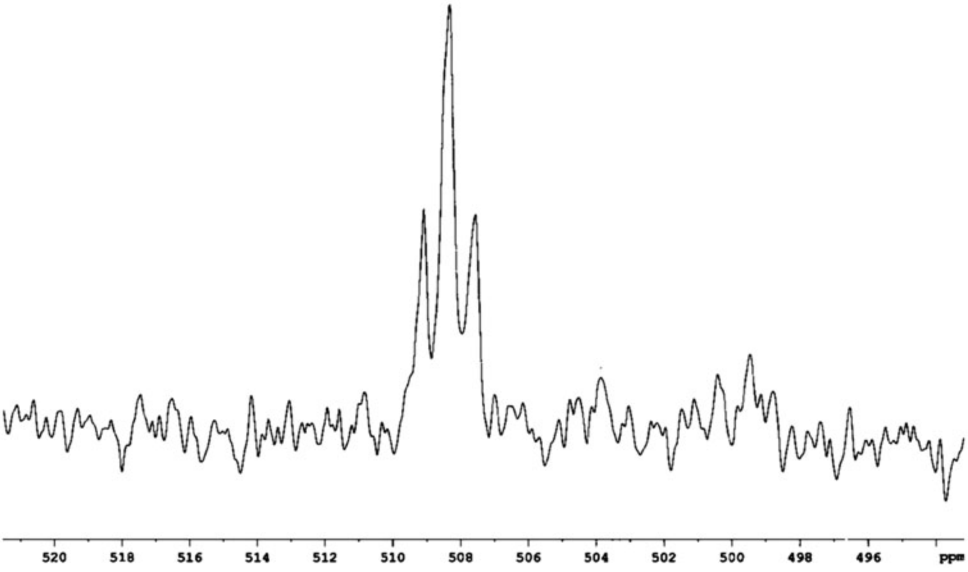

Figure 4. ^{113}Cd NMR spectrum of **6** in dichloromethane at 188 K.

larger than the values measured for the other tris(dimethylamino)phosphine chalcogenide (TDPE) analogs (E=Te: 66 *versus* Se: ~50 [23] and S: 30 Hz [12b]). Such a difference in

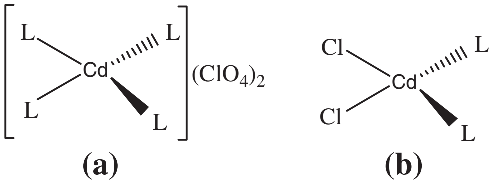

Figure 5. Proposed structures for **1**–**5** (a) and **6**–**10** (b).

the metal–phosphorus coupling constant is probably due to the fact that both Te (or to some extent Se) and Cd are big, whereas there is a considerable size difference in S and Cd. This may account for a more important increase in the valence s electron density at the metal nucleus which would increase the observed $^{2}J(^{113}\text{Cd}–^{31}\text{P})$ value for the TDPTe complex **2** as compared to TDPSe and TDPS species. This is also in agreement with the results obtained for the related complexes which showed that the more bulky the ligand, the more important the $^{2}J(^{113}\text{Cd}–^{31}\text{P})$ value [26, 30].

4. Conclusion

New phosphine telluride complexes of cadmium have been studied in solution by multinuclear (^{31}P, ^{125}Te, and ^{113}Cd) NMR spectroscopy. Our results show that the combination of ^{31}P, ^{125}Te, and ^{113}Cd NMR data in the study of such complexes is very informative of changes related to complex formation as well as in the elucidation of the structure and composition of metal–ligand coordination compounds. These new complexes could also be of interest since they may serve as potential single-source precursors of metal telluride thin films or quantum dots. This is currently under investigation in our laboratory.

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