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Spectroscopic and thermal study of monoalkyl [α-(4-benzeneazoanilino)-N-benzyl]phosphonates and their palladium(II) complexes

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Abstract

Spectroscopic (IR, UV/VIS) and thermal (TG, DTA) studies of the monoethyl (HL1) and monobutyl (HL2) esters of [α-(4-benzeneazoanilino)-N-benzyl]phosphonic acid and their cyclopalladated binuclear complexes, $[PdL(\mu\text{-}Cl)]_2$, as well as mononuclear chelate complexes, PdL_2, have been carried out to determine structural and decomposition properties of these biologically interesting compounds. Thermal decomposition of the complexes takes place through a multistep process. In the chloro-bridged cyclopalladated complexes ligand deesterification coupled with dehalogenation is followed by a complex pyrolysis, while decomposition of the mononuclear complexes includes deesterification and other ligand degradation processes that leads to metallic palladium and P_2O_5. The X-ray and infrared spectroscopic study confirmed this mixture as the final residue in both types of complexes. No stable intermediate products were found during the thermal decomposition due to its complexity and to the overlap of the degradation processes. The spectral and thermal properties of the complexes were compared with those obtained for the complexes of dialkyl [α-(4-benzeneazoanilino)-N-benzyl]phosphonates and discussed with respect to their structure-stability relationship. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Palladium(II) complex; Aminophosphonate complex; Thermal decomposition; Spectroscopy; DTA

1. Introduction

Interest in metallocyclic complexes of palladium(II) with nitrogen donor ligands has received increasing attention in recent years because of their large-scale application in organic synthesis and catalysis [1–3]. In addition, these compounds have been studied with regard to their biological and toxicological properties [4,5]. It was shown that some cyclopalladated complexes posses remarkable antitumor activity [4–7]. The specific interest in this field is focused on the synthesis of new complexes with little structural resemblance to cisplatin or carboplatin, with the goal of forming different and perhaps irreparable DNA lesions in tumor cells.

Continuing our research programme on preparation of palladium(II) complexes with dialkyl and monoalkyl esters of various aminophosphonic acids which might be of interest as anticancer agents [7–11], we recently reported the synthesis of metallocyclic complexes of palladium(II) with monoalkyl esters of [α-(4-benzeneazoanilino)-N-benzyl]phosphonic acid as well as their spectroscopic analysis (NMR, ESI-MS) and antitumor activity [12]. In this report, we describe

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decomposition behavior of these metal complexes as well as of the free organophosphorus ligands investigated by thermogravimetry (TG) and differential thermal analysis (DTA) along with the IR and UV/VIS spectroscopic studies. The results obtained were compared with those previously reported for the palladium(II) complexes of dialkyl [α-(4-benzeneazoanilino)-N-benzyl]phosphonates [7,13].

2. Experimental

Monoethyl and monobutyl esters of [α-(4-benzeneazoanilino)-N-benzyl]phosphonic acid (HL1 and HL2), prepared as previously described [14], were purified by recrystallization from absolute ethanol prior to use. Their cyclopalladated binuclear [C,N] complexes **1** and **3**, $[PdL(\mu\text{-}Cl)]_2$, as well as [N,O] chelate mononuclear complexes **2** and **4**, PdL_2 ($L = L1$ and L2), were prepared according to the published methods [12].

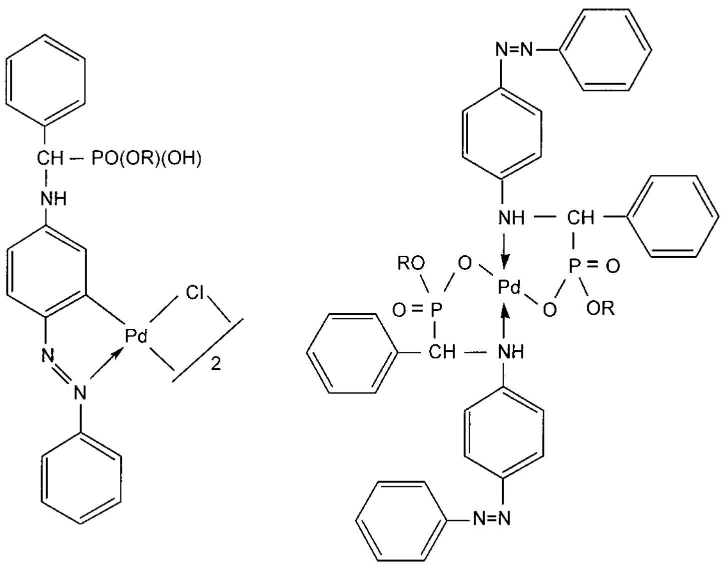
The thermogravimetric analyses (TG) were carried out on a Cahn RG electromicrobalance in an air atmosphere at a heating rate of $4°C\ min^{-1}$ up to 850°C. Differential thermal analyses (DTA) were performed with a Netzsch differential thermal analyzer applying a heating rate of $5°C\ min^{-1}$ in static air atmosphere. The reference substance was pure alumina.

Infrared spectra were recorded on a Perkin-Elmer 580B spectrophotometer using KBr pellets (250–4000 cm^{-1}) and Nujol mulls in polyethylene (200–400 cm^{-1}). The UV–VIS spectra were obtained in chloroform solution with a Perkin-Elmer 124 spectrophotometer in the range 250–600 nm.

The X-ray powder diffraction patterns were taken with a Philips counter diffractometer (monochromatised CuK_α radiation).

3. Results and discussion

Investigations of the interaction of monoalkyl [α-(4-benzeneazoanilino)-N-benzyl]phosphonates towards palladium(II) ion have shown that these ligands act in a bidentate manner forming two types of metallocyclic complexes, those with [C,N] and [N,O] five-membered chelate rings (Fig. 1). In the first case are



1; R=Et
3; R=Bu

2; R=Et
4; R=Bu

Fig. 1. Palladium(II) complexes formed with the monoalkyl [α-(4-benzeneazoanilino)-N-benzyl]phosphonates.

formed the cyclopalladated binuclear complexes with the metal–metal chloro bridge $[PdL(\mu\text{-}Cl)]_2$ (**1** and **3**), in which the deprotonated ligand undergoes palladation at the azo nitrogen and the *ortho*-carbon of the aromatic ring distant from the coordinated azo nitrogen atom. Coordination through the aniline nitrogen and the phosphonic acid oxygen occurs in the basic media where phosphonate acid group is deprotonated, giving mononuclear [N,O] chelate complexes PdL_2 (**2** and **4**). The IR and UV/VIS spectroscopic studies along with TG and DTA analyses presented for the free monoalkyl benzeneazoanilinophosphonate ligands and their palladium complexes confirm the structural information obtained by multinuclear magnetic resonance and mass spectroscopic studies [12].

3.1. Infrared spectra

The selected IR data of the free phosphonate ligands and their palladium complexes are summarized in Table 1. In the spectra of the free ligands, the absorption of the antisymmetric and symmetric NH-stretching vibrations occurs as a medium to strong band at 3360 and 3380 cm^{-1}, respectively [15]. In their palladium complexes, this band is reduced in intensity and appears as a broad band with maximum approximately at the same frequency region. More significant changes in the spectra between two types of complexes may be noticed between 1500 and 1600 cm^{-1} where are found the NH-deformation modes along with the benzene ring stretching vibrations. A very strong band at 1576 cm^{-1} and a week one at about 1510 cm^{-1} are characteristic for the cyclopalladated complexes **1** and **3**, while the chelate complexes **2** and **4** in this frequency region exhibit two very intense bands near 1600 and 1585 cm^{-1} and one of a medium intensity near 1510 cm^{-1}. There are marked differences also between 1400 and 1480 cm^{-1} where are expected to occur the N=N stretching vibrations, but the complexity of this spectral part renders the assignment of this vibration difficult. It is worth noting that complexes **1** and **3** show an almost identical absorption pattern between 1600 and 1400 cm^{-1} to that of the corresponding cyclopalladated complexes of dialkyl esters of benzenazoanilinophosphonates, indicating the same palladium–ligand bonding in these complexes [7]. The absorption of $\nu(C_{arom}\text{–}NH)$, which appears in free ligands as two bands at about 1362 and 1330 cm^{-1}, remains almost at the same position in the cyclopalladated complexes. Only the higher frequency band appears as a shoulder. In the complexes in which the anilino group is involved in palladium bonding, this absorption exhibits the red frequency shift along with the band broadening.

The absorption bands associated with the acidic phosphonic monoester group PO(OR)(OH) are very complex. As a result of coupling of some vibrational modes the proposed assignments must be regarded as approximate description of the vibrations. The free ligands and their cyclopalladated complexes show bands attributed to the P–OH and PO–H absorptions. In HL1 and HL2 there is no band at about 3500 cm^{-1} normally ascribed to the OH-stretching because of the very strong intermolecular hydrogen bonding between the phosphonic acid groups P(O)OH. It was found that these monoesters are dimers in chloroform solution, and are associated, most probably dimeric, also in the solid state [15]. In their spectra are present three typical broad shallow bands in the 1650–2700 cm^{-1} region which are characteristic for the hydrogen bonded P(O)OH group [16,17]. In going from the free ligands to their cyclopalladated complexes this type of hydrogen bonding is less favorable due to the steric factors, and therefore these bands exhibit a marked intensity decrease and are displayed at higher frequencies. The PO–H stretching vibration both in free monoesters and their cyclopalladated complexes is superimposed on the PO–C stretching, giving broad and complex bands between 990 and 1030 cm^{-1}. The position and complexity of the P=O stretching absorption is arising from the hydrogen bonding existing in these compounds. Thus, HL1 and HL2 give broad and complex bands with maximum at about 1231 and 1213 cm^{-1}, respectively, while in the complexes **1** and **3** two bands at 1237 and 1198 cm^{-1} could be ascribed to this vibration. As expected, there is no evidence of the bands associated with P–O–H vibrations in the spectra of the mononuclear complexes **2** and **4** in which the phosphonate ligands act in the anionic form. This is supported by the presence of the $\nu(PO_2^-)$ absorptions between 1200 and 1230 cm^{-1} for the antisymmetric and between 1025 and 1070 cm^{-1} for the symmetric mode of this vibration. In the latter frequency range the $\nu(PO\text{–}C)$ absorption is superimposed upon that of the symmetric PO_2^- vibration [18].

Table 1
Selected infrared spectroscopic data (cm^{-1})[a]

Compound	ν(NH)	ν[P(O)OH][b]	ν(C=O) δ(NH)	$\nu(C_{arom}–N)$	ν(P=O) $\nu_{as}(PO_2^-)$	ν(CO=P)	ν(PO–C), ν_{sym} (PO_2^-), ν(P–OH)	ν(C–C)	ν(P–C)	ν(Pd–Cl)
HL1	3360 m-s	2600 w,br 2300, 2170 w,br 1680 w, br	1598 vs 1551 m 1512 s 1491 m	1362 s 1330 m-s	1231 s, br 1218 sh	1165 m-s 1146 s 1137 s	1028 vs 996 s	957 s	760 m-s	-
1 $[Pd(L1)Cl]_2$	3378 w-m,br	2620 vw,br 2330 vw,br 1700 vw,br	1576 vs 1554 sh 1511 w 1491 w	1360 sh 1330 s	1237 w 1198 w-m	1165 w-m 1130 m-s	1031 s 997 m	957 m	760 m	307 w 255 vw
2 $Pd(L1)_2$	3380 w-m,br	–	1601 vs 1583 vs 1514 w-m 1495 w	1350 sh 1320 m-s,br	1224 m 1207 m	1165 m 1147 m 1132 sh	1070 m 1046 s	955 m-s	762 m	–
HL2	3380 m	2580 w,br 2250, 2120 w,br 1680 m, br	1600 vs 1553 w-m 1505 s 1490 sh	1364 m-s 1332 m	1227 sh 1213 m-s,br	1158 m-s 1135 s	1022 sh 1007 vs 995 sh	975 s	758 m	–
3 $[Pd(L2)Cl]_2$	3380 w-m,br	2600 vw,br 2300 vw,br 1700 vw,br	1576 vs 1555 sh 1507 w 1490 w	1364 sh 1332 s	1235 w 1198 m	1158 w-m 1135 m-s	1023 m-s 997 m-s	955 m,br	758 m	306 w 255 vw
4 $Pd(L2)_2$	3380 m,br	-	1598 vs 1589 vs 1509 m 1490 sh	1345 sh 1325 sh 1315 s,br	1205 s,br	1160 sh 1135 s	1063 s 1025 s	975 s	760 m	-

[a] Abbreviations: vs, very strong; s, strong; m, medium; w, weak; vw, very weak; br, broad; sh, shoulder.

[b] Bands typical for hydrogen bonded P(O)OH group.

Table 2
UV–VIS data

Compound	λ_{max} (nm) (log ε/dm^3 mol^{-1} cm^{-1})[a]
HL1	382 (4.57)
1	295 (4.29), 330 (4.22), 390 (4.23); 492 (4.45)
2	297 (4.29), 376 (4.54), 492 (4.32)
HL2	382 (4.56)
3	294 (4.45), 330 (4.38), 389 (4.40), 492 (4.64)
4	297 (4.24), 375 (4.49), 492 (4.23)

[a] In chloroform (260–600 nm).

The ligand bands attributed to the ν(CO–P), ν(C–C) and ν(P–C) vibrations appeared between 1130 and 1165 cm^{-1}, between 955 and 975 cm^{-1} and near 760 cm^{-1}, respectively, in the spectra of all complexes remain almost at the same position as in the free ligands.

In the far-IR region the chloro-bridged cyclopalladated complexes **1** and **3** show two ν(Pd–Cl) bands at 307 and 255 cm^{-1}. The first one is ascribed to the vibration of the Pd–Cl bond *trans* to the nitrogen atom, while the second one to that *trans* to the σ-bonded carbon, as a greater is *trans* influence of a σ-bonded carbon compared to that of a nitrogen atom [19].

3.2. UV–VIS spectra

The electronic absorption spectral data of the free ligands and their palladium complexes obtained in chloroform in the 260–600 nm region are summarized in Table 2, and the spectra of the monoethyl ester and its complexes are presented in Fig. 2. Free ligands are characterized by the broad and intense band at about 380 nm due to the conjugation through the azo group between the π and n electrons from the aromatic systems disposed on both sides of the azo group [20]. Upon complexation there are spectral differences depending on which type of complex is formed, the [C,N] or the [N,O] metallocyclic complex. The both types of palladium complexes are diamagnetic compounds suggesting a square-planar geometry, for which three spin-allowed d–d transitions are expected. They correspond to transitions from the three lower lying d levels to the empty $d_{x^2-y^2}$ orbitals [21]. The ground state is $^1A_{2g}$ and the excited states related to these transitions are $^1A_{2g}$ $^1B_{1g}$ ^1E_g in order of increasing energy. Overlapping of the strong absorptions arising from the charge-transfer or intraligand transitions prevents the observation of the expected bands. The dark colour of the complexes indicates strong charge-transfer absorptions originating in the ultraviolet and trailing off well into the visible region. The

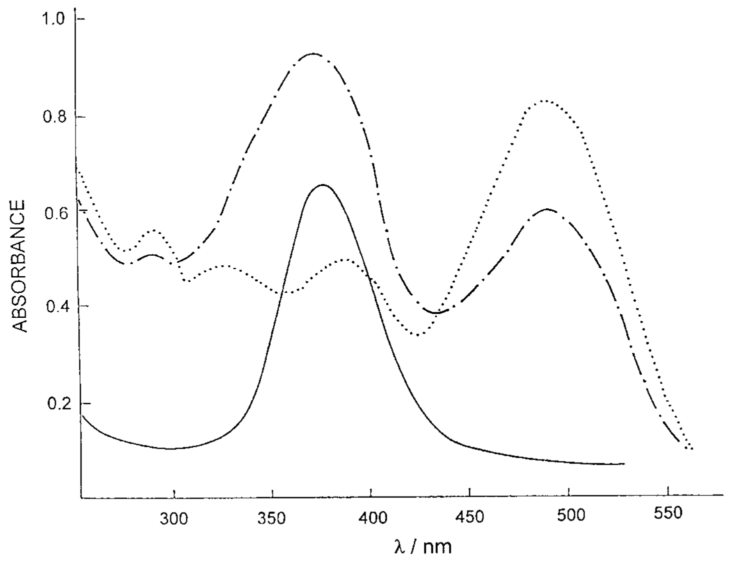


Fig. 2. UV–VIS spectra of HL1 (—) and its complexes **1** (-·-·-) and **2** (……).

most prominent absorption in both types of complexes in the visible region is at 492 nm and could be ascribed to the metal-to-ligand charge-transfer transitions. Its intensity is higher in the cyclopalladated complexes presumably due to contribution of the π–π* transition localized on the orthometallated benzeneazo ligand [22,23]. The less intense absorption at 390 nm in the cyclopalladated complexes **1** and **3**, as well as the most intense one in the [N,O] metallocyclic complexes **2** and **4** at about 375 nm, most probably contains contributions from the charge transfer and different intraligand π–π* and n–p* transitions. The second transition of the orthometallated azobenzene contributes to the absorption at 330 nm in complexes **1** and **3**, while for the band present in both types of complexes around 295 nm, the great contributions from the different d–d transitions could be proposed.

3.3. Thermal studies

Thermal data for the free ligands and their palladium complexes obtained from the TG and DTA curves presented in Figs. 3 and 4 are summarized in Table 3. Although in all compounds no stable intermediate products could be found during the thermal decomposition owing to its complexity and to the overlap of the degradation processes, thermal analysis subsequently followed by infrared spectroscopic study provides some information related to the mode of their decomposition. IR spectra were recorded every 50°C and are compared with the corresponding spectra at room temperature. It was shown that the decomposition of the ligands started with their deesterification by loosing the alkyl ester group presumably as ethanol (HL1) or butanol (HL2) molecule. It is worth noting that the alcohol loss is often observed also in the fragmentation pattern in mass spectrometry of monoalkyl esters [10,24]. In the infrared spectra this is indicated by the loss of the absorption bands arising from various modes of the Pd–O–Et(Bu) and P–O–H groups around 1140 and 1025 cm^{-1}, respectively, as well as from the alkyl C–C vibrations between 950 and 980 cm^{-1}. In the DTA curves this process is visible as one (HL1) or two (HL2) exothermic peaks around 200°C. The pyrolytic decomposition of the ligands continues with a progressive weight loss and is overlapped by sublimation of P_2O_5, which is determined as a final residue by X-ray powder diffraction and infrared analyses. Both ligands show in their DTA curves one endothermic effect at 190 and 170°C, respectively, which is not accompanied by the weight loss on the TG curves and corresponds approximately to the melting points of these compounds [14].

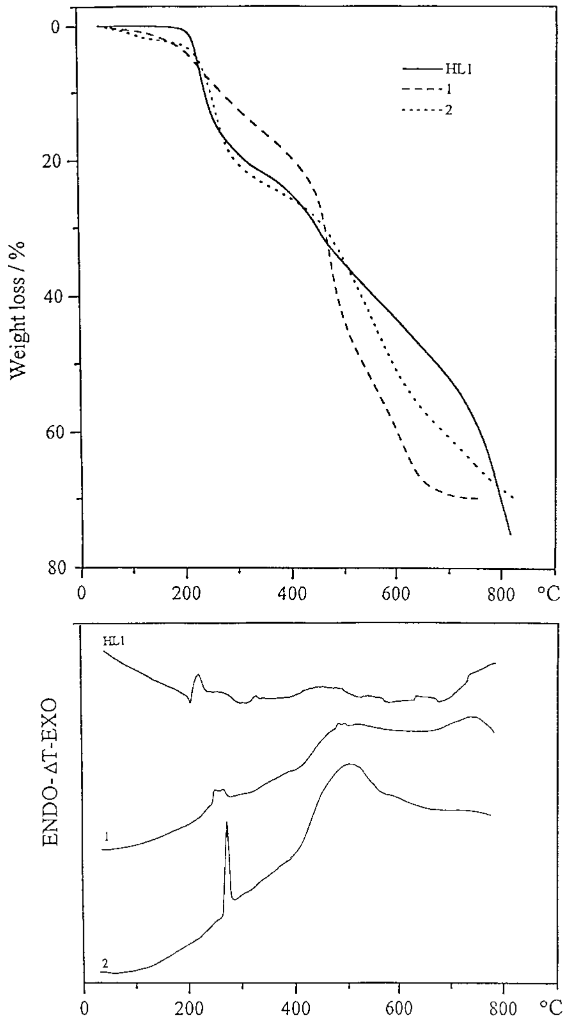


Fig. 3. TG–DTA curves for HL1 and its complexes **1** and **2**.

Investigation of the decomposition behavior of the cyclopalladated complexes **1** and **3** has shown that their first thermal decomposition step, which covered approximately the range up to 350°C, could be attributed to deesterification of the ligand overlapped with dehalogenation of the complex. In the DTA curves these processes are visible as few exothermic peaks. The weight loss of about 16% for complex **1** and 20%

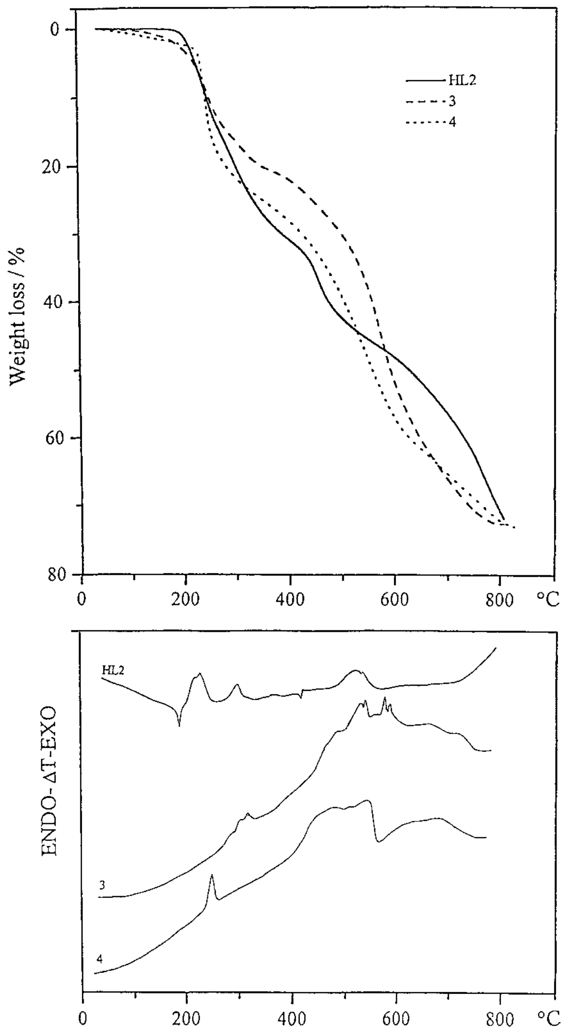


Fig. 4. TG–DTA curves for HL2 and its complexes **3** and **4**.

for complex **3** corresponds well with the theoretical weight loss of both chlorides and two alcohol molecules. The subsequent infrared spectral study has shown an intensity decrease of the Pd–Cl stretching vibration around 300 cm^{-1}, while decreasing of the phosphonate ester bands is accompanied by increasing of the P=O stretching band around 1235 cm^{-1}.

The chelate complexes **2** and **4** in the same temperature region exhibit a sharp exothermic effect at 265 and 245°C, respectively, which corresponds to ligand deesterification. The decomposition of the complexes continues with a progressive weight loss giving a mixture of Pd and P_2O_5, which was identified as the final pyrolytic residue in all palladium complexes **1–4**, similarly as in a number of palladium complexes of various aminophosphonic acid derivatives [13,25,26]. Differences obtained between the calculated and found residue values could be ascribed to partial sublimation of P_2O_5 which takes place above 300°C [27].

From the initial decomposition temperatures, which correspond to the beginning of the deesterification process, it could be seen that decomposition of the free ligands started at higher temperatures (20–70°C) than that of their palladium complexes. The great stability of the free monoalkyl anilinobenzylphosphonates could be connected with their association to dimers arisen from the strong intermolecular hydrogen bonding between the phosphonic acid groups, which was confirmed by their molecular weight determination and by the NMR studies [12,15]. Monobutyl ester is less stable than ethyl ester most probably due to the steric factors, while there are no significant differences between palladium complexes of ethyl and butyl ester in both types of the metallocyclic complexes. When comparing results of the free monoalkyl esters HL1 and HL2 and their cyclopalladated complexes with those obtained for the corresponding dialkyl phosphonates and their palallated complexes, which are included in Table 3 for comparison, it could be seen that both, the diesters and their complexes are more stable compounds. This behaviour is in accordance with the assumption that the phosphonic acid group facilitates deesterification in the monoalkyl derivatives.

It is interesting to point out that in the case of palladium(II) dihalide adducts of diethyl and monoethyl esters of 2-quinolylmethylphosphonic acid, PdL_2X_2 (X=Cl, Br), complexes of monoester are more stable compounds [26], as well as are dihalide adducts of diethyl benzeneazoanilinophosphonates with respect to their butyl analogues [13]. As it was shown that decomposition of these complexes started with their dehalogenation, it may be presumed that the steric hindrance effects that increased from monoester to diester and from ethyl to butyl derivatives are of greater influence for the dehalogenation than for the deesterification process. In addition, it was shown that palladium complexes with terminal halides are less stable than cyclopalladated complexes with bridging halides.

Table 3
TG and DTA data

Compound	Temperature range (°C), TG [a]	Dehalogenation and pyrolysis at T (°C), DTA[b]
HL1	184–816	190 (endo), 205 (exo), 320 (exo), 450 (exo)
	(196–829)	[140 (endo), 265 (exo), 300 (endo), 350 (exo), 390 (exo), 500 (exo)]
1	114–760	225 (exo), 240 (exo), 260 (exo), 475 (exo), 485 (exo), 735 (exo)
	(208–820)	[240 (exo), 480 (exo), 535 (exo), 670 (endo), 745 (exo)]
2	134–829	265 (exo), 500 (exo)
HL2	172–806	170 (endo), 200 (exo), 210 (exo), 290 (exo), 405 (endo), 515 (exo)
	(190–843)	[130 (endo), 265 (exo), 400 (endo), 485 (exo), 745 (exo)]
3	117–806	285 (exo), 295 (exo), 310 (exo), 480 (exo), 520 (exo), 530 (exo), 565 (exo), 575 (exo), 655 (exo), 710 (exo)
	(208–802)	[230 (exo), 500 (exo), 645 (exo), 735 (exo), 765 (exo)]
4	130–825	245 (exo), 480 (exo), 510 (exo), 545 (exo), 565 (endo), 680 (exo)

[a] Data for the dialkyl phosphonate ligands and their cyclopalladated complexes taken from Ref. 13. are given in parenthesis for comparison.

[b] Most of the DTA peaks above 300°C are diffuse.

Acknowledgements

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