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On the Coordination Properties of New Bicyclophosphite-Carbohydrates

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Summary. The coordination behavior of newly developed bicyclophosphite ligands toward Mo(0), Rh(I), and Pt(II) was studied.

Reactions at different ratios of metal to ligand were done. It was found that the ligands act as monodentate ones and form with hexacarbonylmolybdenum(0) complexes with the general formula $Mo(CO)_5L$ by replacing only one carbonyl ligand. Coordination experiments toward acetylacetonatodicarbonylrhodium(I) resulted in the formation of undefined compounds. Toward dichlorocyclooctadienylplatinum(II) the monomeric phosphorus ligands act as monodentate ligands forming complexes of the general structure *cis*-$PtCl_2L_2$. All the synthesized platinum-complexes possess *cis*-configuration (proven by ^{31}P NMR). The corresponding coordination compounds were isolated and characterized by elemental analyses and ^{1}H, ^{13}C, and ^{31}P NMR.

Keywords. Transition metal compounds; NMR spectroscopy; Phosphorus compounds; Coordination behavior.

Introduction

Carbohydrates which possess many functionalities are highly suitable for derivatization by classical synthetic methods. Their natural occurrence and defined chirality induced widespread use in enantioselective catalysis research [1–4]. Especially when equipped with a weak donor atom excellent reactivities and selectivities were obtained. The introduction of phosphorus groups into the carbohydrate system was extensively studied. Phosphorus ligands are successfully used in many catalytic processes. Their high affinity towards transition metals, like Rh, Pd, Pt, Cu, Mo, Cr, Ni, *etc.*, results in the formation of highly catalytically active species [5–8].

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Sugar moieties containing two phosphorus(III) atoms are well known as useful ligands in different enantioselective reactions [9–12]. Diphosphorus compounds usually form chelate structures if the P-atoms are in appropriate distance to each other. The establishment of the latter structure type is a crucial step to obtain high enantioselectives in catalytic processes [13]. Nevertheless, there are several examples of natural polyol-derivatives known in which the P-atoms are not appropriately arranged to act as bidentate ligand – phosphites of mannite, of dihydromannite, and of carbohydrates [14–16]. All these systems are rather rigid what is on the one hand advantageous for their catalytic potential on the other hand the rigid backbone of such structures prevents the chelate formation with transition metals. The limitation of such P(III)-compounds is that the phosphorus atom can be hydrolyzed (even the complete loss of the donor group is possible) or oxidized (formation of P(V) residues) [11, 17, 18].

In contrast, bicyclophosphites with carbohydrate backbone are compounds overcoming the disadvantageous behavior of their close relatives – they are easy to handle and surprisingly rather stable [19, 20]. Hence, they possess some obvious properties outclassing their non-cyclic analogues. Additionally, they represent a class of ligands with interesting coordination properties. Examples for the coordination of bicyclophosphites to transition metal centers are known with, *e.g.*, Cu(I), Ag(I), Cr(0), Mo(0), Pt(II), and Rh(I). It should be mentioned that in the case of Pt(II)-complexes with alkylidene-protected glucofuranose-ligands *cis-trans*-isomerization was observed when storing the complex with the general formula $PtCl_2L_2$ for 48 h at room temperature in *DMSO* [21].

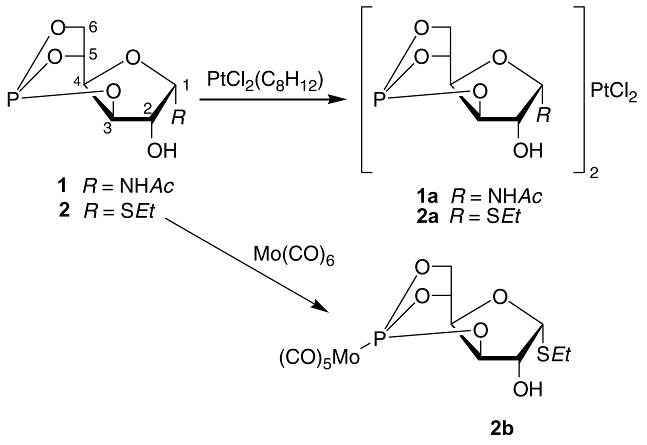
Recently the synthesis of a new type of ligand which contains two bicyclophosphite moieties and a rather flexible linker was reported [22–24]. Herein the syntheses of coordination compounds with the latter carbohydrates and with monomeric analogues as well as investigations on the coordination properties of those new bicyclophosphites toward Pt(II), Rh(I), and Mo(0) are presented.

Results and Discussions

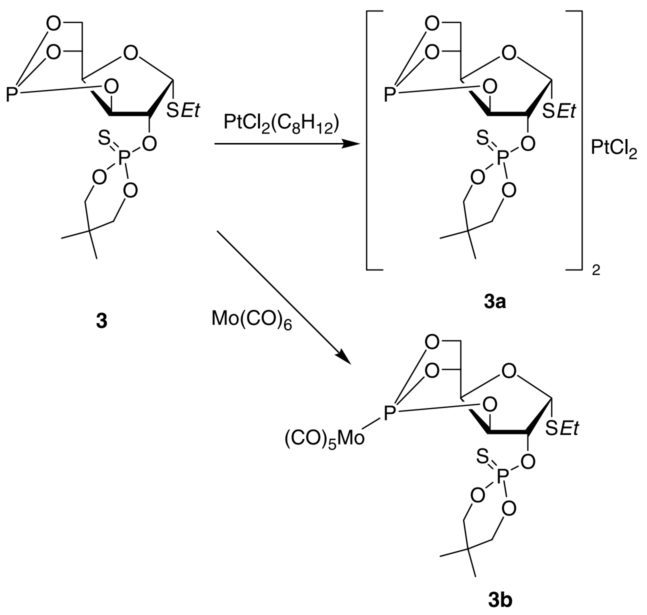
In order to study the coordination behavior of several bicyclophosphites, transition metals with different properties, oxidation state (0, +I, +II), and coordination geometry (octahedral, 2× square-planar) were chosen: Mo(0), Rh(I), and Pt(II), respectively. Additionally all these transition metals are applied in a wide range of catalytic processes (for reviews see Refs. [4, 25, 26]).

Coordination Behavior Toward Dichlorocyclooctadienylplatinum(II)

The reactions of the bicyclophosphites **1** to **5** with dichlorocyclooctadienylplatinum(II) were performed in CH_2Cl_2 by dropwise addition of the platinum complex to the corresponding phosphites (see Schemes 1–3). The reactions were monitored by means of TLC and ^{31}P NMR – the disappearance of the bicyclophosphite spot was monitored as well as the decrease of the signal assignable to the uncoordinated phosphorus atom at 118 ppm, respectively. Exploiting the low solubility of complexes **1a**, **2a**, **4a**, and **5a** in common organic solvents, *e.g.*, chloroform, dichloromethane, benzene, 1,4-dioxane, *etc.*, resulted in isolation of



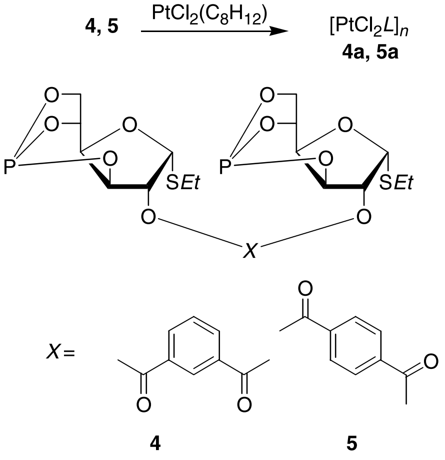
Scheme 1



Scheme 2

the complexes with good yield and in high purity by filtration. In contrast, complex **3a** was precipitated by the addition of *n*-hexane.

The formation of the platinum complexes was indicated by the appearance of the corresponding signals in ^{31}P NMR between 85 and 88 ppm (for NMR data see Tables 1 and 2). The coupling constants for all synthesized coordination compounds were about 5500 Hz which is, according to the literature data, typical for *cis*-platinum(II)-complexes [11, 27]. Due to the low solubility in solvents like $CDCl_3$ or C_6D_6, ^{1}H NMR spectra for complexes **1a** and **2a** were obtained in



Scheme 3

DMSO-d_6. The ^{1}H NMR spectra for **3a** and **4a** and a ^{13}C NMR spectrum for **3a** were recorded in $CDCl_3$.

In the ^{1}H NMR spectra of complexes **1a** and **2a** peak broadening and low-field shifting of all hydrogen atoms, which are located closely to the phosphorus(III) atom, were observed. For compound **3a** the corresponding ^{1}H NMR spectrum displayed a similar set of signals as found for the uncoordinated ligand. In the ^{13}C NMR spectrum a set of broad singlets was found, only with the exception of the C4-atom signal which exhibits doublet structure. In the case of compounds **4** and **5** the signals in the ^{1}H NMR spectra were very broad and unassignable.

In contrast to **1a**, compounds **2a** to **5a** are containing additional to the P-donor groups thioethyl-rests being also suitable to form stable coordination compounds with transition metal acceptors. However, in all experiments no coordination to the latter functionalities was observed. Even at higher platinum ratios, in ^{1}H NMR spectra no platinum satellites for the neighbored protons could be detected.

For compound **2a**, ^{31}P NMR spectra were recorded in different solvent systems being characterized by different coordination properties. In CH_2Cl_2 the signal of the bicyclophosphite moiety coordinated to the platinum center was found at 85 ppm with a coupling constant of 5597 Hz, whereas in *DMF* and pyridine the signal shifted high-field over time (24 h). In *DMF* the signal was observed at 50 ppm with the coupling constant being 5638 Hz, whereas in pyridine the signal was even detected at 36 ppm with a coupling constant of 5476 Hz. This behavior indicates that the complex remains in *cis*-configuration (much higher coupling constants than *trans*-complexes [11, 27]) but chloro ligands might be exchanged to *DMF* or pyridine, respectively.

Ligand **3** contains two P-centers in the molecule – the bicyclophosphite and the 5,5-dimethyl-2-sulfide-1,3,2-dioxaphosphorinane group. It was shown by ^{31}P NMR that only the P(III) atom was involved in the coordination to the Pt-center. The ^{31}P NMR signal for this phosphorus moiety was shifted from 118 to 88 ppm accompanied by the formation of the typical coupling for platinum complexes in

Table 1. ^{1}H NMR signals for the synthesized complexes

	H1 ($^3J_{1,2}$; $^4J_{1,P'}$)	H2 ($^3J_{2,P'}$)	H3 ($^3J_{3,4}$)	H4 ($^3J_{4,5}$; $^4J_{4,P}$)	H5 ($^3J_{5,6'}$; $^3J_{5,P}$)	H6 ($^2J_{6,6'}$)	H6′ ($^3J_{6',P}$)
1a in *DMSO*-d_6	5.92	4.05	4.80	4.63	5.19	4.38	4.27
	(2.6)		(3.0)	(*)	(*; 8.1)	(9.0)	(*)
2a in *DMSO*-d_6	5.67	4.44	4.93	4.63	5.32	4.90	4.47
	(3.8)		(3.4)	(3.0; 3.0)	(5.1; 10.2)	(9.0)	(2.1)
3a in $CDCl_3$	5.82	5.05	5.34	4.49	5.14	4.53	3.95
	(3.4; 2.1)	(10.2)	(3.4)	(*)	(*)	(11.1)	(2.1)
4a in $CDCl_3$	6.00	5.73	5.12	5.61	5.21	4.59	4.50
	(4.7)		(*)	(2.1)	(*; 9.8)	(9.5)	(*)
2b in $CDCl_3$	5.59	4.36	4.70	4.25	5.03	4.40	4.20
	(3.4)		(3.4)	(2.6; 2.6)	(5.5; 15.3)	(9.4)	(1.7)
3b in $CDCl_3$	5.71	5.04	5.20	3.81	4.12	3.16	3.16
	(3.8; 2.1)	(10.2)	(3.8)	(2.6; 2.6)	(*; 12.8)	(*)	(*)
4b in $CDCl_3$	5.69	5.67	4.27	3.57	4.06	3.13	3.13
	(3.8)		(3.4)	(2.6; 3.0)	(*; 17.4)	(*)	(*)

	Other hydrogen atoms	^{31}P ($J_{Pt,P}$) in CH_2Cl_2
1a in *DMSO*-d_6	$N\underline{H}COCH_3$ 7.84 ($^3J_{NH,H1}$ 9.4), $NHCOC\underline{H}_3$ 1.92	86.0 (5585.4)
2a in *DMSO*-d_6	$SC\underline{H}_2CH_3$ 2.73, $SCH_2C\underline{H}_3$ 1.30 ($^2J_{H,H}$ 7.3)	85.3 (5597.3)
3a in $CDCl_3$	$SC\underline{H}_2CH_3$ 2.82, $SCH_2C\underline{H}_3$ 1.38, $C\underline{H}_3$ 1.28, $C\underline{H}_3$ 0.90, H_e 3.86 (2H), H_a 4.32 (2H, $^2J_{Ha,He}$ 11.1, $^3J_{Ha,P'}$ 3.8)	87.9 (5654.5) 58.6
4a in $CDCl_3$	$SC\underline{H}_2CH_3$ 2.77, $SCH_2C\underline{H}_3$ 1.34 ($^2J_{H,H}$ 7.5), H(Ar) 8.29–8.15 (4H)	88.3 (5668.0)
2b in $CDCl_3$	$SC\underline{H}_2CH_3$ 2.84, $SCH_2C\underline{H}_3$ 1.43 ($^2J_{H,H}$ 7.7), $O\underline{H}$ 3.2	162.3
3b in $CDCl_3$	$SC\underline{H}_2CH_3$ 2.51, $SCH_2C\underline{H}_3$ 1.19 ($^2J_{H,H}$ 7.7), $C\underline{H}_3$ 0.86, $C\underline{H}_3$ 0.42, H_e 3.55 (2H), H_a 4.24 (2H, $^2J_{Ha,He}$ 10.7, $^3J_{Ha,P'}$ 6.0, $^3J_{He,P'}$ 20.0)	161.7; 58.4
4b in $CDCl_3$	$SC\underline{H}_2CH_3$ 2.31, $SCH_2C\underline{H}_3$ 1.00 ($^2J_{H,H}$ 7.3), H(Ar) 8.98 (1H), 8.15 (2H), 6.96 (1H)	162.1

* No coupling constant determination possible

cis-configuration. The signal at 59 ppm assignable to the uncoordinated 5,5-dimethyl-2-sulfide-1,3,2-dioxaphosphorinane group of the molecule was not influenced by the addition of dichlorocyclooctadienylplatinum(II) even when raising the ratio to 1:5 (ligand:platinum).

Compounds **4** and **5** are containing two phosphorus moieties suitable for coordination and therefore possess potential to form chelate complexes. The reaction

Table 2. ^{13}C NMR signals for the synthesized complexes

	C1 ($^3J_{C1,P'}$)	C2 ($^3J_{C2,P}$)	C3	C4 ($^3J_{C4,P}$)	C5 ($^2J_{C5,P}$)	C6 ($^2J_{C6,P}$)	Other carbon atoms
3a in $CDCl_3$	88.19 (7.5)	79.84	76.35	77.95 (7.8)	75.84	69.01	$S\underline{C}H_2CH_3$ 29.56, $SCH_2\underline{C}H_3$ 15.37, C(7, 8) 81.71, C(9) 32.11 ($^3J_{C9,P'}$ 7.0), C(10, 11) 21.83, 20.56
2b in $CDCl_3$	90.16	78.46 (7.8)	77.80	77.03	73.98 (5.3)	68.06 (8.4)	$S\underline{C}H_2CH_3$ 25.70, $SCH_2\underline{C}H_3$ 15.38, C(O) 203.48, 202.87
4b in $CDCl_3$	88.12	79.48 (7.5)	76.99	76.35	73.71 (5.1)	68.09 (9.6)	$S\underline{C}H_2CH_3$ 25.90, $SCH_2\underline{C}H_3$ 14.98, C(O) 202.94, 202.67, C(O) 170.27, 163.76, C(Ar) 134.72, 131.46, 129.88, 129.37, 128.99

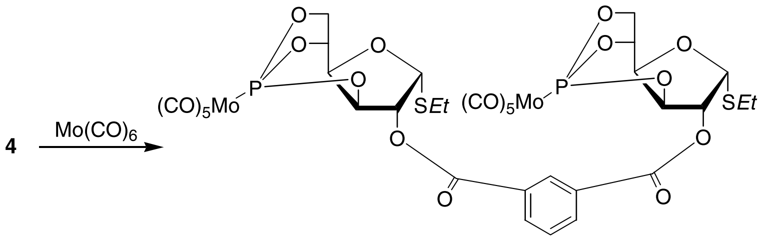
with dichlorocyclooctadienylplatinum(II) did not yield identifiable products – it is assumed that oligomeric structures are formed. Additionally, it can be reported that in MALDI-TOF-MS a mixture of monomeric, dimeric, trimeric, tetrameric, up to oligomeric structures was observed. In accordance to the MS results and the assumption that oligomerization occurs, the signals in the ^{1}H NMR spectrum appeared clearly broadened. Also the addition of a large excess of dichlorocyclooctadienylplatinum(II) did not result in the selective formation of defined dinuclear platinum complexes. All these findings strongly indicate the oligomeric structure of the formed complexes.

Coordination Behavior Toward Acetylacetonatodicarbonylrhodium(I)

The reactions of all the investigated ligands with acetylacetonatodicarbonylrhodium(I) did not result in characterizable compounds. It is assumed that the ligand was oxidized or polymerization effects occurred. The oxidation theory is supported by the fact that in ^{31}P NMR, when dissolving the obtained precipitate in *DMSO* or *DMF*, signals in the P(V) area were detected.

Coordination Behavior Toward Hexacarbonylmolybdenum(0)

The mononuclear molybdenum complexes **2b** and **3b** were obtained from equimolar mixtures of hexacarbonylmolybdenum(0) and the corresponding phosphites in 1,4-dioxane at 90°C (see Schemes 1 and 3, respectively). The dinuclear complex **4b** was synthesized by treating the ligand with the molybdenum complex at molar ratio 1:2 under the same reaction conditions as for the mononuclear analogues (see Scheme 4). The reactions were monitored by means of TLC (slightly change of the



4b

Scheme 4

R_f-value) and ^{31}P NMR (increasing signal assignable to the coordinated phosphorus atom at about 162 ppm). The products were precipitated by addition of *n*-hexane.

The ^{1}H NMR spectra of the resulting complexes **2b** and **3b** contained similar sets of signals with respect to the spectra of the ligands **2** and **3**, correspondingly. Only the coupling constant $J_{H5,P}$ was sensitive to coordination – it was increased from 10 to 13–17 Hz.

In ^{13}C NMR spectra of **2b** and **4b** an increase of the coupling constants was found being for $J_{C2,P}$, $J_{C5,P}$, and $J_{C6,P}$ +2.8, +1.0, and +2.7 Hz, respectively. The synthesis of chelate structures was unsuccessful: variation of the ligand-to-complex ratio, increasing the reaction temperature, as well as UV-irradiation led in a first step to the formation of mononuclear complexes and in a second one to decomposition.

In contrast to the platinum complexes, the reaction of hexacarbonylmolybdenum(0) with ligand **4** at different metal-to-ligand ratios resulted neither in chelate formation nor in oligo- or polymerization. Consequently, it can be concluded that the complex bearing five carbonyl-ligands is energetically the most stable form.

Conclusion

Several new complexes between stable P(III)-ligands and transition metals at different oxidation states as well as with different coordination geometry and properties were synthesized and characterized.

In the case of the platinum complexes, it was found that with bidentate ligands oligomers are formed – even high Pt(II)-excess did not result in chelation. When coordinating monodentate ligands to dichlorocyclooctadienylplatinum(II), well defined and characterizable compounds were obtained. These complexes are *cis*-configured and undergo ligand exchange reactions – dissolution in solvents possessing donor groups results in an exchange of chloro-ligands by solvent molecules.

The reaction of the bicyclophosphite-carbohydrates with acetylacetonatodicarbonylrhodium(I) led to not clearly identifiable products.

The hexacarbonylmolybdenum(0) allows the exchange of exactly one carbonyl ligand by a phosphorus-equipped sugar. Addition of an excess of the ligand as well as variation of the reaction parameters did not affect the type of the formed complex.

Experimental

All reactions were carried out in dry solvents and under argon atmosphere. The bicyclophosphite ligands were prepared following methods reported earlier [20, 22, 24]. The NMR spectra were recorded on a Bruker WM-200 (^{1}H, with TMS as an internal standard), a Bruker AC-200 P (^{13}C, at 50.3 MHz, with TMS as an internal standard), and a Bruker W-80 SY (^{31}P, at 32.4 MHz, with 85% H_3PO_4 as external standard). Chemical shifts are depicted in ppm and coupling constants in Hz (for NMR data of the coordination compounds see Tables 1 and 2). Melting points were determined in open capillaries and are uncorrected. The elemental analyses data were in good agreement with the calculated values. Silica gel plates Siluphol UV-254 and a solvent mixture of benzene and 1,4-dioxane (1:5) were used for thin layer chromatography.

(SP-4-2)-Bis(N-acetyl-3,5,6-bicyclophosphite-α-D-glucofuranosylamine-κP) dichloroplatinum(II) (**1a**, $C_{16}H_{24}Cl_2N_2O_{12}P_2Pt$)

A solution of 0.19 g of Pt(*COD*)Cl_2 (0.5 mmol) in 3 cm^3 of CH_2Cl_2 was added dropwise at room temperature to a solution of 0.25 g of *N*-acetyl-3,5,6-bicyclophosphite-α-D-glucofuranosylamine (**1**, 1 mmol) in 10 cm^3 of CH_2Cl_2. The resulting white solid was filtered off, washed with cold *n*-hexane (2 × 5 cm^3) and dried in vacuum. Yield 0.33 g (86%), mp 240°C (decomp).

(SP-4-2)-Bis(3,5,6-bicyclophosphite-ethyl-1-thio-α-D-glucofuranoside-κP) dichloroplatinum(II) (**2a**, $C_{16}H_{26}Cl_2O_{10}P_2PtS_2$)

Following the same procedure as described for compound **1a**, complex **2a** was obtained from 3,5,6-bicyclophosphite-ethyl-1-thio-α-D-glucofuranoside (**2**). Yield 0.32 g (84%), mp 223°C (decomp).

(SP-4-2)-Bis[3,5,6-bicyclophosphite-ethyl-2-(5,5-dimethyl-2-sulfide-1,3,2-dioxaphosphorinane-2-yl)-1-thio-α-D-glucofuranoside-κP]dichloroplatinum(II) (**3a**, $C_{26}H_{44}Cl_2O_{14}P_4PtS_4$)

A solution of 0.19 g of Pt(*COD*)Cl_2 (0.5 mmol) in 3 cm^3 of CH_2Cl_2 was added dropwise at room temperature to a solution of 0.42 g of 3,5,6-bicyclophosphite-ethyl-2-(5,5-dimethyl-2-sulfide-1,3,2-dioxaphosphorinane-2-yl)-1-thio-α-D-glucofuranoside (**3**, 1 mmol) in 10 cm^3 of CH_2Cl_2. The reaction mixture was stirred for 4 h and 10 cm^3 of *n*-hexane were added. The obtained white solid was filtered off, washed with cold *n*-hexane (2 × 5 cm^3), and dried in vacuum. Yield 0.43 g (78%), mp 192–194°C (decomp).

(SP-4-2)-{μ-[2,2′-Bis(3,5,6-bicyclophosphite-ethyl-1-thio-α-D-glucofuranoside)(1,3-benzenedicarboxylate)-κ^{2}P,P′]}dichloroplatinum(II) (**4a**, oligomer)

Following the same procedure as described for compound **1a**, complex **4a** was obtained from 2,2′-bis(3,5,6-bicyclophosphite-ethyl-1-thio-α-D-glucofuranoside)(1,3-benzenedicarboxylate) (**4**). Yield 0.80 g (88%), mp 208–210°C (decomp).

(SP-4-2)-{μ-[2,2′-Bis(3,5,6-bicyclophosphite-ethyl-1-thio-α-D-glucofuranoside)(1,4-benzenedicarboxylate)-κ^{2}P,P′]}dichloroplatinum(II) (**5a**, oligomer)

Following the same procedure as described for compound **1a**, complex **5a** was obtained from 2,2′-bis(3,5,6-bicyclophosphite-ethyl-1-thio-α-D-glucofuranoside)(1,4-benzenedicarboxylate) (**5**). Yield 0.60 g (66%), mp 227–229°C (decomp).

(OC-6-22)-(3,5,6-Bicyclophosphite-ethyl-1-thio-α-D-glucofuranoside-κP) pentacarbonylmolybdenum(0) (**2b**, $C_{13}H_{13}MoO_{10}PS$)

A solution of 0.25 g of 3,5,6-bicyclophosphite-ethyl-1-thio-*α-D*-glucofuranoside (**2**, 1 mmol) and of 0.27 g of hexacarbonylmolybdenum(0) (1 mmol) in 2 cm^3 of 1,4-dioxane was stirred for 4 h at 100°C. The reaction mixture was cooled down to room temperature and 5 cm^3 of *n*-hexane were added. The white precipitate was filtered off, washed with *n*-hexane ($3 \times 5\,cm^3$), and dried in vacuum. Yield 0.39 g (80%), mp 156–157°C, $R_f = 0.85$.

(OC-6-22)-[3,5,6-Bicyclophosphite-ethyl-2-(5,5-dimethyl-2-sulfide-1,3,2-dioxaphosphorinane-2-yl)-1-thio-α-D-glucofuranoside-κP] pentacarbonylmolybdenum(0) (**3b**, $C_{19}H_{22}MoO_{12}P_2S_2$)

Complex **3b** was synthesized from 0.42 g of 3,5,6-bicyclophosphite-ethyl-2-(5,5-dimethyl-2-sulfide-1,3,2-dioxaphosphorinane-2-yl)-1-thio-*α-D*-glucofuranoside (**3**, 1 mmol) and 0.54 g of hexacarbonylmolybdenum(0) (2 mmol) following the same procedure as described for compound **2b**. Yield 0.40 g (60%), mp 72–74°C, $R_f = 0.89$.

(OC-6-22)-μ-[2,2′-Bis(3,5,6-bicyclophosphite-ethyl-1-thio-α-D-glucofuranoside) (1,3-benzenedicarboxylate)-κP,P′]-bis[pentacarbonylmolybdenum(0)] (**4b**, $C_{34}H_{28}Mo_2O_{22}P_2S_2$)

Complex **4b** was synthesized from 0.63 g of 2,2′-bis(3,5,6-bicyclophosphite-ethyl-1-thio-*α-D*-glucofuranoside)(1,3-benzenedicarboxylate) **4** (1 mmol) and 0.54 g of hexacarbonylmolybdenum(0) (2 mmol) following the same procedure as described for compound **2b**. Yield 0.86 g (78%), mp 115–117°C, $R_f = 0.83$.

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