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Reaction of Esters, Ortho Esters, Acetals, Thioacetals and Epoxides with 2,4-Bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane 2,4-disulfide (*Lawesson* Reagent)

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2,4-Bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane 2,4-disulfide, *LR*, reacted with ethyl formate, triethoxymethane, 1,1,1-triethoxyethane and tetraethoxymethane to give **2a** and **3**. 1,1-diethoxymethylbenzene when treated with *LR* produced **2a**, **3**, **7**, **8**, and **9b**. Triethoxymethylbenzene when heated with *LR* gave **2a**, **8** and **11**. The reaction of benzyl formate, tris(benzyloxy)methane and 1,1-dibenzyloxy-*N*,*N*-dimethylmethanamine with *LR* afforded **2b**. Bis(methylthio)methane, tris(ethylthio)methane and 1,1-bis(butylthio)ethane with *LR* gave **9**. 1,1-Bis(2,2-dimethylpropoxy)-*N*,*N*-dimethylmethanamine with *LR* yielded **2c** and **17**. The reaction of 1,2-epoxyethylbenzene with *LR* gave **20**, while 1,2-epoxypropane or 1,2-epoxybutane with *LR* afforded **23a**, **b**.

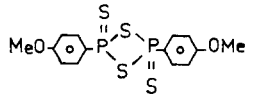
(Keywords: Dithiaphosphetane; Phospha; Thia)

Reaktionen von Estern, Orthoestern, Acetalen, Thioacetalen und Epoxiden mit 2,4-Bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetan-2,4-disulfid (Lawesson Reagens)

Lawesson-Reagens (*LR*) reagiert mit einer Vielzahl von Verbindungen zu entsprechenden Derivaten. Es wurden folgende Reaktionspartner für *LR* eingesetzt: Ethylformiat, Triethoxymethan, 1,1,1-Triethoxyethan, Tetraethoxymethan, 1,1-Diethoxymethylbenzol, Triethoxymethylbenzol, Benzylformiat, Tris(benzyloxy)methan, 1,1-Dibenzyloxy-*N*,*N*-dimethylmethanamin, Bis(methylthio)methan, Tris(ethylthio)methan, 1,1-Bis(butylthio)ethan, 1,1-Bis(2,2-dimethylpropoxy)-*N*,*N*-dimethylmethanamin, 1,2-epoxyethylbenzol, 1,2-epoxypropan und 1,2-Epoxybutan. Die entstandenen Produkte wurden mittels MS und ^{13}C- bzw. ^{1}H-Spektroskopie charakterisiert.

Introduction

The reaction of triethoxymethane with phosphorus pentasulfide was reported by *Brannock*[1] and *Athawale* et al.[2] to give ethyl formate, ethyl thionoformate and triethyl dithiophosphate. Without any spectroscopic proofs*Schrader*[3] cliamed that 2,4-bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane 2,4-disulfide reacted with 1,2-epoxypropane at 35 °C yielding 4-methoxyphenyl-1,4,5-oxathiaphospholidine, a result which could not be reproduced. As *Lawesson* reagent, *LR*, has been shown to be a very efficient thiation reagent for different substrates[4–16], its reaction with esters, ortho esters, acetals, thioacetals and epoxides has been studied and the results are reported in this paper and outlined in Scheme 1.



Lawesson Reagent (LR)

Results and Discussion

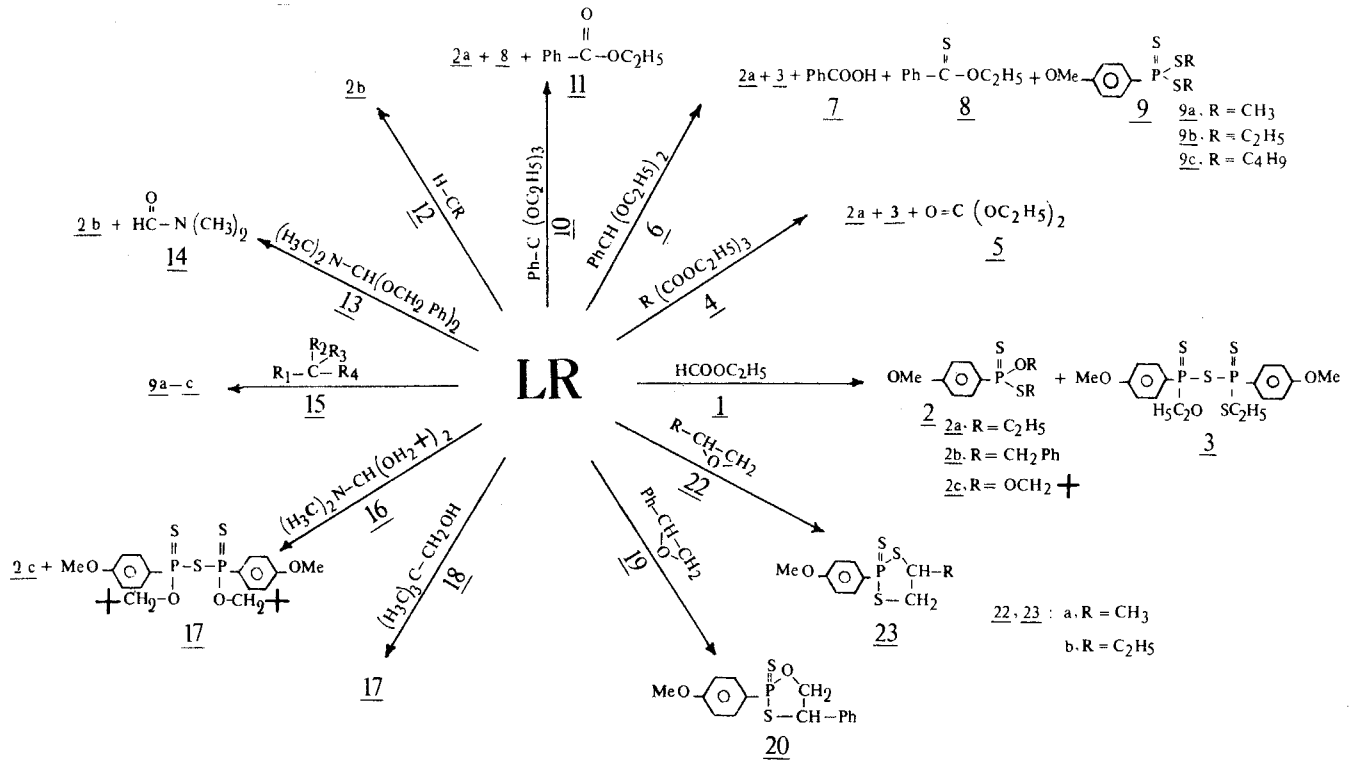
2,4-Bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane-2,4 disulfide (*Lawesson* reagent, *LR*) reacted with ethyl formate (**1**) in anhydrous benzene at 80 °C to give diethyl 4-methoxyphenylphosphonodithioate (**2a**) and *O*-ethyl *S*-ethyl di(4-methoxyphenyl)thiodiphosphonate (**3**) (m.p. and mixed m.p. 112 °C)[8, 17].

Similarly, *LR* reacted with triethoxymethane (**4a**), 1,1,1-triethoxyethane (**4b**), and tetraethoxymethane (**4c**) giving **2a** and **3**. In one case only (**4c**, $R = OC_2H_5$) diethyl carbonate (**5**) was isolated (GLC).

The structural proof of **2a** is based on spectral data (NMR, MS) and elemental analysis.

Its ^{1}H-NMR spectrum contains a multiplet at 1.0–1.5 ppm (6 H, 2 CH_3), a multiplet at 2.3–3.0 ppm (2 H, S—CH_2), a multiplet at 3.7–4.0 ppm (2 H, O—CH_2), and a singlet at 3.85 ppm (3 H, OCH_3). In the ^{13}C-NMR spectrum there are signals at 15.4 ppm ($^3J_{CP}$ = 8.5 Hz, O—CH_2—**C**H_3), 15.0 ppm ($^3J_{CP}$ = 6.4 Hz, S—CH_2—**C**H_3), 63.9 ppm ($^2J_{CP}$ = 6.3 Hz, OCH_2), 28.2 ppm ($^2J_{CP}$ = 3.6 Hz, S—CH_2), and 55.3 ppm (O—CH_3), in accordance with literature[18]. In the ^{31}P-NMR spectrum there is one singlet at 95.5 ppm also in accordance with literature data for the following structure[13, 19]:

Scheme 1



4
4a , R = H
4b , R = CH_3
4c , R = OC_2H_5

12
12a , R = $OOCH_2Ph$
12b , R = $(OCH_2Ph)_3$

15
15a , $R_1 = R_2 = H$; $R_3 = R_4 = SCH_3$
15b , $R_1 = H$; $R_2 = R_3 = R_4 = SC_2H_5$
15c , $R_1 = H$; $R_2 = CH_3$; $R_3 = R_4 = S\text{-}C_4H_9$

The reaction of equimolecular amounts of 1,1-diethoxymethylbenzene (**6**) with *LR* in anhydrous xylene at 140 °C gave the following products: **2a**, **3**, benzoic acid (**7**), ethyl thionobenzoate (**8**)[20], and diethyl 4-methoxyphenylphosphonotrithioate (**9b**)[8,13,17]. Equimolecular amounts of triethoxymethylbenzene (**10**) with *LR* in anhydrous toluene at 110 °C gave **2a**, **8**, and ethyl benzoate (**11**). In the reaction of **10** (1 mol) with excess of *LR* (2 mol) in anhydrous xylene at 140 °C **11** was not isolated while the yield of **8** increased (due to thiation of **11**) but the yield of **2a** did not change.

The reaction of benzyl formate (**12a**) or tris(benzyloxy)-methane (**12b**) with *LR* in anhydrous xylene at 110 °C or 140 °C yielded dibenzyl 4-methoxyphenylphosphonodithioate (**2b**). Compound **2b**—besides dimethyl formamide (**14**) (GLC)—could also be obtained when 1,1-di(benzyloxy)-*N*,*N*-dimethylmethanamine (**13**) reacted with *LR* in anhydrous benzene at 25 °C. The structural proof of **2b** is based on spectral data (NMR, MS) and elemental analysis.

In the ^{1}H-NMR the SCH_2 protons (2 H) appeared as a multiplet at 3.8–4.0 ppm and the OCH_2 protons (2 H) as a multiplet at 4.7–5.2 ppm. In the ^{13}C-NMR spectrum there is one signal at 37.7 ppm ($^2J_{CP}$ = 3.2 Hz, SCH_2), and one signal at 66.6 ppm ($^2J_{CP}$ = 6.1 Hz, OCH_2). In the ^{31}P-NMR spectrum there is one singlet at 96.0 ppm.

The reaction of bis(methylthio)methane (**15a**), tris(ethylthio)methane (**15b**), and 1,1-bis(butylthio)ethane (**15c**) with *LR* in anhydrous xylene at 110 °C or 140 °C gave dimethyl 4-methoxyphenylphosphonotrithioate (**9a**), diethyl 4-methoxyphenylphosphonotrithioate (**9b**), and dibutyl 4-methoxyphenylphosphonotrithioate (**9c**), respectively. The structure of compounds **9a**[13] and **9b**[8,13,17] was known and also confirmed by ^{1}H-, ^{13}C-, ^{31}P-NMR and MS and elemental analysis beside TLC with authentic samples. The structural proof of **9c** is based on spectral data (NMR, MS) and elemental analysis.

In the ^{1}H-NMR spectrum the CH_3 and CH_2 group protons (14 H) appeared as a multiplet at 0.7–1,8 ppm while the SCH_2 protons (4 H) appeared as a multiplet in the region 2.3–3.2 ppm. In the ^{13}C-NMR spectrum the SCH_2 carbon appeared at 33.4 ppm ($^2J_{CP}$ = 3.7 Hz). In the ^{31}P-NMR spectrum there is a singlet at 77.4 ppm which is in accordance with literature data for the following structure[13,21,22]:

1,1-Bis(2,2-dimethylpropoxy)-*N*,*N*-dimethylmethanamine (**16**) with *LR* in anhydrous toluene at 110 °C yielded di(2,2-dimethylpropoxy)-4-methoxyphenylphosphonodithioate (**2c**) and *O*,*O*-

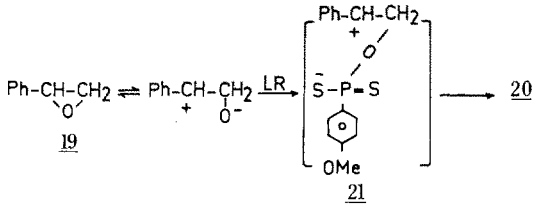
di(2,2-dimethylpropoxy) di(4-methoxyphenyl)thiodiphosphonate (**17**). The structural elucidation of **2c** and **17** is based on spectroscopic data (NMR, MS) and elemental analyses.

In the ^{1}H-NMR spectra of **2c** and **17** the OCH_2 protons (2 H) appeared as a multiplet at 3.4–4.2 and 3.9–4.2 ppm, respectively. In **2c** there is a multiplet at 2.4–2.8 ppm (2 H, SCH_2 protons). In the ^{13}C-NMR spectrum of **2c** there is one signal at 47.6 ppm ($^2J_{CP}$ = 4.1 Hz, SCH_2), one signal at 74.1 ppm ($^2J_{CP}$ = 8.1 Hz, OCH_2) and in **17** a signal at 76.5 ppm ($^2J_{CP}$ = 7.3 Hz, OCH_2). The ^{31}P-NMR spectrum of **2c** showed a singlet at 96.0 ppm, while that for **17** showed three singlet at 84.8, 76.4, and 76.4 ppm which may be due to the asymmetric centers in the molecule. Moreover, the structure of **17** is additionally confirmed by its preparation from 2,2-dimethyl-1-propanol (**18**) and *LR* in anhydrous xylene at 140 °C.

By reacting 1,2-epoxyethylbenzene (**19**) with *LR* in anhydrous benzene at 50 °C, a new phosphorus heterocycle, 1,4,5-oxathiaphospholidine (**20**), was obtained. The structure of **20** is proved by ^{1}H-, ^{13}C-, and ^{31}P-NMR and mass spectroscopy and elemental analysis.

In the ^{1}H-NMR spectrum there is a singlet at 3.85 ppm (3 H, OCH_3) and a multiplet at 4.3–5.4 ppm (3 H, OCH_2 and SCH protons). In the ^{13}C-NMR spectrum there is one signal at 75.9 ppm ($^2J_{CP}$ = 4.1 Hz, OCH_2) and one signal at 55.7 ppm ($^2J_{CP}$ = 1.5 Hz, SCH). In the ^{31}P-NMR spectrum there are two signals at 109.2 and 107.5 ppm (due to the asymmetric center).

As to the mechanism for the formation of **20**, it is suggested that a nucleophilic attack by **19** on *LR* gives the intermediate **21** followed by ring closure to give **20**.



On the other hand, when 1,2-epoxypropane (**22a**) and 1,2-epoxybutane (**22b**) were reacted with *LR* in anhydrous xylene at 35 °C or 140 °C, two new phosphorus heterocycles, 1,4,5-dithiaphospholidines **23a** and **23b**, were isolated.

The structural proof of **23** is based on spectroscopic data and elemental analyses. (For ^{1}H-NMR and MS see Exp. Part.) In the ^{13}C-NMR spectrum of **23a** the SCH_2 appeared as a multiplet at 48.3 ppm and that for **23b** as a multiplet at

46.0 ppm, while the SCH carbon for **23a** and **23b** as a multiplet at 56.3 and 61.0 ppm, respectively. In the ^{31}P-NMR spectrum of **23a** there are two singlets at 92.7 and 92.5 ppm and that for **23b** two singlets at 91.4 and 91.3 ppm (due to the asymmetric centers).

Concerning the mechanism for the formation of **23** it is suggested that an exchange of oxygen by sulfur first occured at the starting epoxides induced by *LR* followed by further reaction of the resulting R—$\overline{\text{CH}}$—$\overline{\text{CH}_2}$—S with *LR* following the same pathway as in the formation of **20** from **19**, producing the final product **23**.

Experimental Part

^{1}H-, ^{13}C- and ^{31}P-NMR and mass spectra were obtained as described earlier (cf. Ref. [13,15,16]). Microanalysis were carried out by NOVO Microanalytical Laboratory, NOVO Industri A/S, NOVO Allé, DK-2880 Bagsvaerd, Denmark. Silica Gel 60 (Merck) was used for column chromatography. M.p.'s are uncorrected.

Starting Materials: Lawesson Reagent (*LR*) (now available from Fluka AGH, CH-9470 Buchs SG) may be prepared as described earlier (cf. Ref. [13–16]). All others chemicals were commercialy obtained.

General Procedure for the Reaction of **1**, **4**, **6**, **10**, **12**, **13**, **15**, **16**, **18**, **19**, *and* **22** *with LR*

0.01 mol of the starting compound and 2.02 g (0.005 mol) of *LR* were mixed in 10 ml of the anhydrous solvent with stirring until no more of the starting materials could be detected (TCL). The solvent was evaporated under reduced pressure and the residue was applied to silica gel column using ether/petrol ether as eluant. The reaction conditions (solvent, temperature, reaction time) and the physical data are given in Table 1.

2a: MS: *m/e* 276 (M^+, 91%), 247 (M^+-C_2H_5), 231 (M^+-OC_2H_5), 215 (M^+-SC_2H_5), 202 [O*Me*C_6H_4P(S)S]. ^{1}H-NMR ($CDCl_3$): δ 1.0–1.5 (6H, 2 CH_3), 2.3–3.0 (2H, m, SCH_2), 3.7–4.0 (2H, m, OCH_2), 6.9 (2H, dd, $^4J_{PH}$ 5 Hz, J_{HH} 9 Hz), 7.9 (2H, dd, $^3J_{PH}$ 15 Hz, J_{HH} 9 Hz). Anal. calcd. for $C_{11}H_{17}O_2PS_2$: C 47.8, H 6.2, P 11.2, S 23.1. Found: C 48.0, H 6.3, P 11.1, S 23.1.

2b: MS: *m/e* 400 (M^+, 100%), 391 (M^+-*Ph*CH_2, 100%), 293 (M^+-OCH_2*Ph*, 10), 277 (M^+-SCH_2*Ph*, 25), 203 [O*Me*C_6H_4P(S)·SH, 50], 187 [O*Me*C_6H_4P(S)OH, 35], 171 [O*Me*C_6H_4P(O)OH, 20], 155 [O*Me*C_6H_4P(O)H, 15], 139 (O*Me*C_6H_4PH, 50). ^{1}H-NMR ($CDCl_3$): δ 3.7 (3H, s, OCH_3), 3.8–4.0 (2H, m, SCH_2), 4.7–5.2 (2H, m, OCH_2), 6.8 (2H, dd, $^4J_{PH}$ 5 Hz, J_{HH} 9 Hz), 7.1 (5H, s, *Ph*), 7.3 (5H, s, *Ph*), 7.8 (2H, dd, $^3J_{PH}$ 15 Hz, J_{HH} 9 Hz). Anal. calcd. for $C_{21}H_{21}O_2PS_2$: C 63.0, H 5.3, P 7.8, S 16.0. Found: C 63.1, H 5.5, P 7.6, S 15.5.

2c: MS: *m/e* 366 (M^+, 100%), 289 (M^+-CH_2—*t-Bu*, 20), 273 (M^+-OCH_2—*t-Bu*, 50), 257 (M^+-SCH_2—*t-Bu*, 100), 241 (M^+-$OSCH_2$—*t-Bu*, 75), 225 (M^+-S_2CH_2—*t-Bu*, 43). ^{1}H-NMR ($CDCl_3$): δ 0.9 (9H, s, 3 CH_3), 1.0 (9H, s, 3 CH_3), 2.4–2.8 (2H, m, SCH_2), 3.4–4.2 (5H, m, OCH_2 and OCH_3), 6.9 (2H, dd, $^4J_{PH}$ 5 Hz, J_{HH} 9 Hz), 7.8 (2H, dd, $^3J_{PH}$ 15 Hz, J_{HH} 9 Hz). Anal. calcd. for $C_{17}H_{29}O_2PS_2$: C 56.7, H 8.1, P 8.6, S 17.8. Found: C 56.7, H 7.9, P 8.8, S 17.6.

Table 1. *Experimental and physical data of the products*

Start comp.	Prod.	Solvent	Reaction time (h)	Reaction temp (°C)	Yield (%)	M.P. (°C)	Precise MS Found (calcd.)
1	**2a**	*X*	10	140	5	oil	276.0429 (276.0442)
	3				7	112	refs. [8,17]
4a	**2a**	*T*	2	100	86		
	3				10		
4b	**2a**	*T*	4	110	93		
	3				10		
4c	**2a**	*B*		80	82		
	3				70		
	5				GLC		
6	**2a**	*X*	10	140	32		
	3				20		
	7				20	122	
	8				50	oil	ref. [20]
	9b				25		refs. [8,13,17]
10	**2a**	*T*	12	110	84		
	8				36		
	11				75	oil	
10	**2a**	*X*	10	140	84		
	8				70		
12a	**2b**	*X*	10	140	40	oil	399.9987 (400.0720)
12b	**2b**	*T*	8	110	55		
13	**2b**	*B*	2	25	55		
	14				GLC		
15a	**9a**	*X*	3	140	72	oil	ref. [13]
15b	**9b**	*T*	10	110	20		
15c	**9c**	*X*	1	140	84	oil	348.0610 (348.0805)
16	**2c**	*T*	9	110	56	oil	360.1332 (360.1345)
	17				11	oil	546.1244 (546.1243)
18	**17**	*X*	10	140	35		
19	**20**	*B*	2	50	25	160	322.0250 (322.0251)
22a	**23a**	*X*	12	140	50	oil	275.9874 (275.9864)
22b	**23b**	*X*	10	140	44	oil	290.0036 (290.0023)

B = Benzene; *T* = Toluene; *X* = Xylene.

9c: MS: *m/e* 348 (M^+, 100), 291 (M^+-C_4H_9, 85), 259 (M^+-SC_4H_9, 73), 203 (63), 202 [O*Me*C_6H_4P(S)S, 94], 187 (40). ^{1}H-NMR ($CDCl_3$): δ 0.7–1.8 (14 H, m, CH_2 and CH_3 protons), 2.3–3.2 (4 H, m, 2 SCH_2), 3.8 (3 H, s, OCH_3), 6.9 (2 H, dd, $^4J_{PH}$ 5 Hz, J_{HH} 9 Hz), 7.9 (2 H, dd, $^3J_{PH}$ 15 Hz, J_{HH} 9 Hz). Anal. calcd. for $C_{15}H_{25}OPS_3$: C 51.7, H 7.2, P 8.9, S 27.6 Found: C 51,8, H 7.2, P 8.8, S 27.4.

17: MS: *m/e* 546 (M^+, 100), 529 (M^+-OH, 82), 513 (M^+-SH, 10), 475 (M^+-CH_2—*t-Bu*, 8), 459 (M^+-$POCH_2$—*t-Bu*, 20), 372 (M^+-2 OCH_2—*t-Bu*, 257 [O*Me*C_6H_4P(S)OCH_2—*t-Bu*, 100), 187 (100), 155 (40). ^{1}H-NMR ($CDCl_3$): δ 0.9 (9 H, s, 3 CH_3), 3.8 (3 H, s, OCH_3), 3.9–4.2 (2 H, m, OCH_2), 6.9 (2 H, dd, $^4J_{PH}$ 5 Hz, J_{HH} 9 Hz), 7.8 (2 H, dd, $^3J_{PH}$ 15 Hz, J_{HH} 9 Hz). Anal. calcd. for $C_{24}H_{36}O_4P_2S_3$: C 52.8, H 6.6, P 11.4, S 17.6. Found: C 52.6, H 6.5, P 11.6, S 17.5.

Table 2. *NMR chemical shifts (ppm) and* P—C *coupling constants (Hz) of the products.* Solvent: $CDCl_3$; ^{13}C chemical shifts relative to *TMS*; ^{31}P chemical shifts relative to 85% H_3PO_4

Prod.	CH_3 (ppm)	J_{P-C} (Hz)	SCH_2 (ppm)	J_{P-C} (Hz)	OCH_2 (ppm)	J_{P-C} (Hz)	SCH (ppm)	J_{P-C} (Hz)	^{31}P (ppm)
2a	15.4 15.0	8.5 6.4	28.2	3.6	63.9	6.3	—		95.5
2b	—		37.7	3.2	66.6	6.1	—		96.0
2c	28.5 26.2	— —	47.6	4.1	74.1	8.1	—		96.0
9c	13.4	—	33.4	3.7	—		—		77.4
17	29.0	—	—		76.5	7.3	—		84.8, 76.4, 76.4
20	—		—		75.9	4.1	55.7	1.5	109.2, 107.5
23a	17.6	(m)	48.3	(m)	—		56.3	(m)	92.7, 92.5
23b	14.5	(m)	46.0	(m)	—		61.0	(m)	91.4, 91.3

20: MS: *m/e* 322 (M, 100), 306 (M-16, 9), 218 (M-*Ph*CH=CH_2, 45), 203 (37), 104 (*Ph*CH=CH_2, 100). ^{1}H-NMR ($CDCl_3$): δ 3.8 (3 H, s, OCH_3), 4.3–5.4 (3 H, m, OCH_2 and SCH), 6.9 (2 H, dd, $^4J_{PH}$ 5 Hz, J_{HH} 9 Hz), 8.0 (2 H, dd, $^3J_{PH}$ 15 Hz, J_{HH} 9 Hz). Anal. calcd. for $C_{15}H_{15}O_2PS_2$: C 55.9, H 4.7, P 9.6, S 19.9. Found: C 56.0, H 4.5, P 9.5, S 20.1.

23a: MS: *m/e* 276 (M^+, 100), 261 (M^+-CH_3, 15), 243 (M^+-SH, 20), 234 (M^+-CH_3—CH=CH_2, 51). ^{1}H-NMR ($CDCl_3$): δ 1.5 (3 H, d, CH_3, 2.8–3.6 (2 H, m, SCH_2), 3.8 (3 H, s, OCH_3), 4.0–4.4 (1 H, m, SCH), 6.9 (2 H, dd, $^4J_{PH}$ 5 Hz, J_{HH} 9 Hz), 7.9 (2 H, dd, $^3J_{PH}$ 15 Hz, J_{HH} 9 Hz). Anal. calcd. for $C_{10}H_{13}OPS_3$: C 43.5, H 4.7, P 11.2, S 34.8. Found: C 43.3, H 4.8, P 11.1, S 34.6.

23b: MS: *m/e* 290 (M^+, 100), 271 (M^+-C_2H_5, 10), 257 (M^+-SH, 20), 234 (M^+-C_2H_5—CH=CH_2, 49). ^{1}H-NMR ($CDCl_3$): δ 0.9 (3 H, t, CH_3), 1.6–2.1 (2 H, m, CH_2—CH_3) 2.9–3.6 (2 H, m, SCH_2), 3.8 (3 H, s, OCH_3), 3.9–4.3 (1 H, m, SCH), 6.8 (2 H, dd, $^4J_{PH}$ 5 Hz, J_{HH} 9 Hz), 7.9 (2 H, dd, $^3J_{PH}$ 15 Hz, J_{HH} 9 Hz). Anal. calcd. for $C_{11}H_{15}OPS_3$: C 45.5, H 5.2, P 10.7, S 33.1. Found: C 45.4, H 5.2, P 10.5, S 32.8.

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