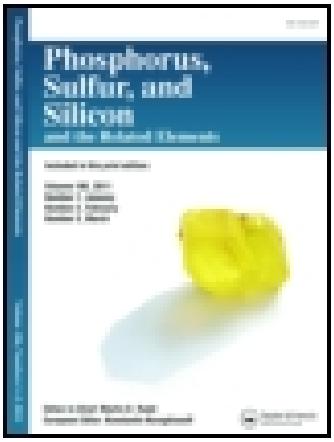

Phosphorus, Sulfur, and Silicon and the Related Elements

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Optically active dithiophosphoric acid, its ammonium salt, and S-esters on the basis of (1R)-endo-(+)-fenchyl alcohol

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OPTICALLY ACTIVE DITHIOPHOSPHORIC ACID, ITS AMMONIUM SALT, AND S-ESTERS ON THE BASIS OF (1*R*)-*ENDO*-(+)-FENCHYL ALCOHOL

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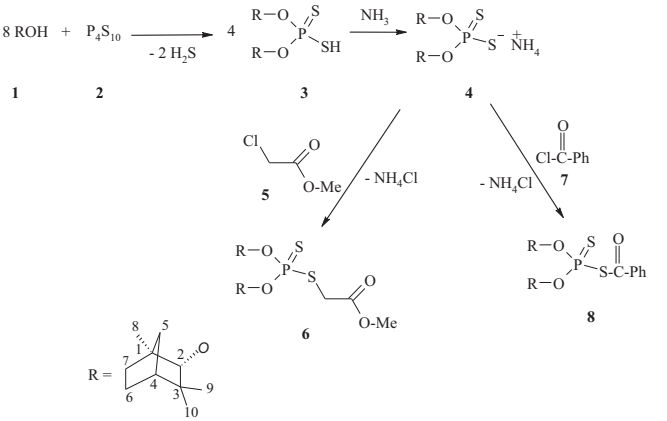
GRAPHICAL ABSTRACT



O is a place of attachment of the oxygen atom

Abstract *Optically active dithiophosphoric acid was prepared by the reaction of tetraphosphorus decasulfide with (1R)-endo-(+)-fenchyl alcohol. The ammonium salt of dithiophosphoric acid prepared reacts with methyl chloroacetate and benzoyl chloride to give dithiophosphate S-esters.*

Keywords Tetraphosphorus decasulfide; (1*R*)-*endo*-(+)-fenchyl alcohol; dithiophosphoric acid; dithiophosphate S-esters; methyl chloroacetate; benzoyl chloride

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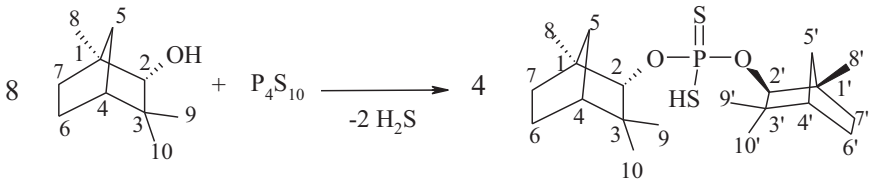
INTRODUCTION

The terpenoids and their derivatives such as geranyl pyrophosphate, geranylgeranic acid amides, methyl ether of farnesylacetic acid have been reported to possess metabolic regulation activity such as antiulcering, gastritis- and wound-healing, lowering blood pressure, antithrombic, and antiplatelet aggregation activities.[1–3] Phosphorylated terpenoid derivatives are expected to be a prospective class of nontoxic bioregulators for creating new drugs.[4–9] Organothiophosphorus compounds contained pharmacophoric functionalities in *O*-substituents at the tetracoordinated phosphorus atom seem to possess appreciated biological activity. Thus, biologically active dithiophosphate-containing pharmacophoric functionalities in the *O*-organyl groups, e.g., nucleoside, oligonucleoside, and peptide dithiophosphates were reported.[10–15] The *tris*(*O,O′*-diborneyl and *O,O′*-dimenthyl dithiophosphato-*S,S′*)chromium(III) complexes have been reported however without identification of corresponding dithiophosphoric acids.[16] We have also obtained optically active dithiophosphoric and dithiophosphonic acids by the reactions of (1*R*,2*S*,5*R*)-(–)-menthol and (1*S*,2*R*,5*S*)-(+)-menthol in the reactions with tetraphosphorus decasulfide and 2,4-diaryl 1,3,2,4-dithiadiphosphetane-2,4-disulfides.[17,18] On the other hand, traditional pesticides on the basis of dithiophosphoric acids are known to have pharmacophoric functionalities in the *S*-organyl substituents.[19–26] These dithiophosphate pesticides were usually obtained by the reactions of *O,O*-dialkyl dithiophosphoric acids or their salts with chloroanhydrides of carboxylic acids, esters, and amides of α-chlorocarboxylic acids, imides, diethyl maleate, functionally substituted olefins, unsaturated terpenes, etc.[19–26] Consequently, we also decided to involve optically active dithiophosphoric acids prepared on the basis of enantiopure monoterpene alcohols in the interactions with chloroanhydrides of carboxylic acids, α-chlorocarboxylic acid esters, and chlorosubstituted epoxides. We have already reported our preliminary results of study of reactions of tetraphosphorus decasulfide with chiral terpenols and ammonium dithiophosphates with methyl chloroacetate, benzoyl chloride, and epichlorohydrin as an abstract of conference report[27] without experimental details and structure identification. So in this article, optically active dithiophosphoric acid, its ammonium salt, and S-esters on the basis of (1*R*)-*endo*-(+)-fenchyl alcohol **1** are presented.

RESULTS AND DISCUSSION

In spite of that the reaction of tetraphosphorus decasulfide with alcohols is a common method for preparing dithiophosphoric acids,[28] the experimental conditions of this reaction with participation of monoterpene alcohols remained unclear. Thus, Ohta et al. carried out the reaction of tetraphosphorus decasulfide with *D*-borneol and *L*-menthol in toluene with heating under reflux for 2 h.[16] In contrast to this, we have found more mild conditions for the formation of optically active dithiophosphoric acids. Thus, (1*R*,2*S*,5*R*)-(–)-menthol and (1*S*,2*R*,5*S*)-(+)-menthol react with tetraphosphorus decasulfide at 50°C for 2 h.[18] That is why we have searched optimal conditions of the reaction of tetraphosphorus decasulfide **2** with (1*R*)-*endo*-(+)-fenchyl alcohol **1**. Indeed, the reaction of **1** with **2** occurs in benzene at 50°C for 2 h resulting in the formation of *O,O*-di[(1*R*)-*endo*-(+)-1,3,3-trimethylbicyclo[2.2.1]hept-2-yl) dithiophosphoric acid **3** in 98% yield (Scheme 1).

Dithioacid **3** possesses optical activity ($[\alpha]^{22}_{D}$ +30.0°, *c* 1.0, C_6H_6). The ^{31}P NMR spectrum of **3** in benzene solution shows a signal at $\delta_P = 87.6$ ppm. This resonance is typical to dithiophosphoric acids.[29] We have observed shifting of signal of methine proton

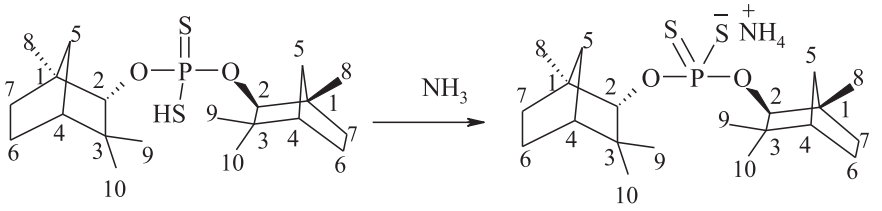


1 **2** **3**

Scheme 1

toward low field (the fragment P-OC^{2}H, $\delta = 3.98$ ppm, $^3J_{PH} = 15.9$ Hz) in the ^{1}H NMR spectrum of **3** relative to the chemical shift of the similar proton of alcohol **1** (the fragment C-OC^{2}H, $\delta = 3.37$ ppm). Three intensive singlets at $\delta = 0.61$, 0.76, and 0.84 ppm are assigned to the protons of six methyl groups. Weak bands at 2584, 2552, and 2470 cm^{-1} in the IR spectrum of **3** are due to the S-H stretching vibrations.

Dithioacid **3** was transformed into the corresponding crystalline ammonium salt **4** in benzene solution at 50°C for 1 h as depicted in Scheme 2. The optical rotation value $[\alpha]^{22}_D$ of **4** is +16.0° (*c* 1.0, C_6H_6). A signal at $\delta_P = 112.1$ ppm was observed in the ^{31}P NMR spectrum of **4** in benzene solution. It should be noted that the ^{31}P resonances of ammonium dithiophosphates are usually situated in this range.[28] Strong H_4N^+ absorption bands are appeared at 3316 and 3160 cm^{-1} in the IR spectrum of **4**.

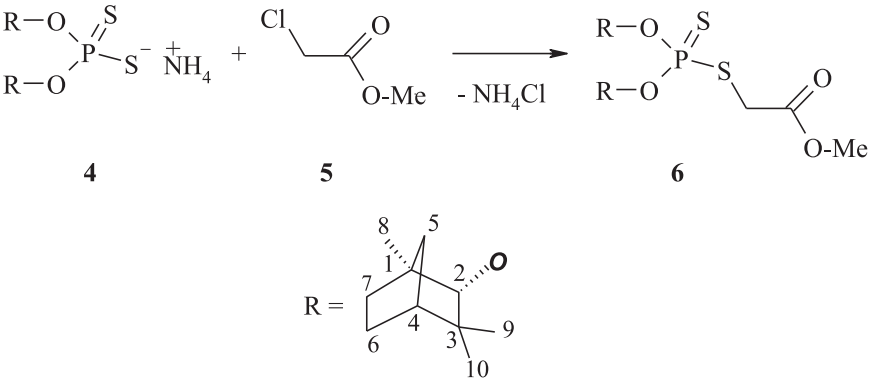


3 **4**

Scheme 2

We deemed it to be necessary to prepare new dithiophosphate S-esters by using replacement reactions of salt **4** with esters of α-chlorocarboxylic acids. We have shown that the reaction of **4** with methyl chloroacetate **5** in benzene at 20°C for 1 h yielded *O,O*-di[(1*R*)-*endo*-(+)-1,3,3-trimethylbicyclo[2.2.1]hept-2-yl) *S*-(methoxycarbonylmethyl)dithiophosphate **6** (Scheme 3).

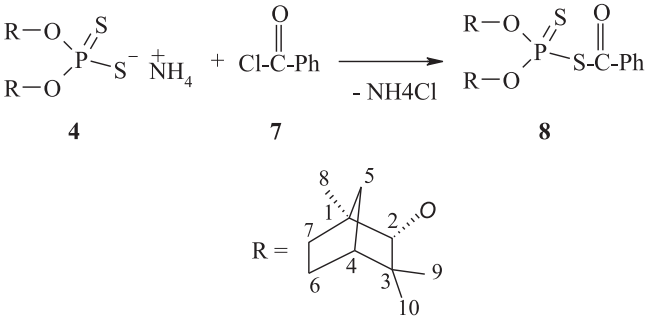
Optically active ester **6** ($[\alpha]^{22}_D$ +14.8°, *c* 1.0, C_6H_6) is an oily liquid. The ^{31}P NMR spectrum of **6** reveals a signal at $\delta_P = 99.7$ ppm (C_6H_6). This resonance is shifted toward low field in comparison with the ^{31}P NMR data of dithioacid **3** ($\delta_P = 87.6$ ppm). Band at 1744 cm^{-1} is assigned to the O-C = O stretching vibrations. Compound **6** was formed as the mixture of diastereoisomers. Thus, the methylene protons of the fragment PSC$\underline{H}_2$ appear as two doublets at $\delta = 3.67$ and 3.79 ppm ($^3J_{PH} = 16.1$ and 15.9 Hz, respectively). An intensive singlet observed at $\delta = 3.75$ ppm has been assigned to the methyl protons of the OC$\underline{H}_3$ group.



O is a place of attachment of the oxygen atom

Scheme 3

This replacement reaction may be extended to other chlorosubstituted pharmacophoric compounds containing the C–Cl bond such as chloroanhydrides of carboxylic acids. Thus, benzoyl chloride **7** is known to react with ammonium salt of alkylene dithiophosphates to yield 2-benzoyl alkylene dithiophosphates.[21] The formation of products of similar structure could be expected in the case of salt **4**. In fact, the reaction of **4** with **7** at 20°C for 1 h has brought about the formation of optically active *O,O*-di[(1*R*)-*endo*-(+)-1,3,3-trimethylbicyclo[2.2.1]hept-2-yl) *S*-phenylcarbonyldithiophosphate **8** ($[\alpha]^{22}_D$ +37.3°, *c* 1.0, C_6H_6) in 83% yield (Scheme 4).



O is a place of attachment of the oxygen atom

Scheme 4

The ^{31}P signal of **8** (δ_P = 84.0 ppm) is situated in the practically same region as that of the corresponding free dithioacid (δ_P = 87.6 ppm) and of 2-benzoyl alkylene dithiophosphates (δ_P 79–85 ppm).[21] The ^{1}H NMR spectrum of **8** shows characteristic resonances due to the presence of phenyl group. Thus, multiplet in the region δ = 7.46–7.94 ppm is assigned to phenyl protons. The strong (Ph)C=O absorption band of **8** is appeared at 1693 cm^{-1}. The electron impact mass spectrum of **8** exhibits the peak *m/e* 506 due to its molecular $[M]^+$ (calculated molecular mass of **8** is 506.7).

It should be emphasized that optical activities of dithiophosphoric acid on the basis of enantiomeric pure (1*R*)-*endo*-(+)-fenchyl alcohol, its ammonium salt, *S*-(methoxycarbonylmethyl), and *S*-phenylcarbonyl esters have been retained.

EXPERIMENTAL

Moisture was carefully excluded throughout the experimental manipulations. The solvents were dried prior to use. The ^{31}P NMR spectra were taken on a Bruker Avance 400 (161.98 MHz) spectrometer (Bruker BioSpin Gmbh Rheinstetten, Germany) in C_6H_6 with 85% H_3PO_4 as an external reference. The ^{1}H NMR spectra were recorded at ambient temperature with a Bruker Avance 400 (400 MHz) (Bruker Biospin Gmbh) and a Bruker Avance 600 (600 MHz) instruments in $CDCl_3$. Chemical shifts δ are presented in ppm relative to residual resonance of solvent (^{1}H: 7.26 ppm), coupling constants J are given in Hz. FTIR spectra were obtained in KBr pellet or in film with a Bruker Vector 22 (400–4000 cm^{-1}) (Bruker Optik Gmbh, Ettlingen, Germany) and expressed in cm^{-1}, δ = the deformation vibration. Mass spectra (EI, 70 eV) were determined on a DFS Thermo Electron Corporation chromato-mass-spectrometer. Optical rotations were measured with a Perkin Elmer 341 polarimeter (λ 589 nm, $l = 55$ mm; Waltham, Mass. USA).

Reaction of Tetraphosphorus Decasulfide With (1*R*)-*endo*-(+)-Fenchyl Alcohol

Sulfide **2** (0.7 g, 1.6 mmol) was added portionwise under dry argon under stirring at 20°C to the solution of alcohol **1** (2.0 g, 13.0 mmol) in anhydrous benzene (20 mL), and the stirring was continued for 2 h at 50°C. The mixture was filtered. The filtrate was evaporated under vacuum (0.5 mm Hg) at 40°C for 1 h and then under vacuum (0.02 mm Hg) at 40°C for 1 h and gave 2.55 g (98%) of **3**. n_D^{20} 1.5062. Anal. found C 59.32; H 8.45; P 7.39; S 16.10. $C_{20}H_{35}O_2PS_2$. Calcd. C 59.67; H 8.76; P 7.69; S 15.93%. IR: film, ν (cm^{-1}) = 2954, 2870 (CH_3 as, s; CH_2 as, s; CH); 2584, 2552 (S-H, free); 2470 (S-H, bonded); 1462 δ (CH_3 as); 1377, 1367 δ [$(CH_3)_2C$ gem. s]; 1062 [(P)O-C]; 989 (OC-C); 800 (PO_2 as, s); 676 (P=S); 585 (P-S). ^{1}H NMR: $\delta = 0.61$ s (6H, $C^8\underline{H}_3$); 0.76 and 0.84 two s [12H, $(C^{9,10}\underline{H}_3)_2C$]; 0.90 m (8H, $C^6\underline{H}_2$-$C^7\underline{H}_2$); 1.18 m (2H, $C^4\underline{H}$); 1.43 m (4H, $C^5\underline{H}_2$); 3.98 d (2H, P-O$C^2\underline{H}$, $^3J_{PH} = 15.9$ Hz). ^{31}P NMR: $\delta_P = 87.6$.

Ammonium *O,O*-Di[(1*R*)-*endo*-(+)-1,3,3-trimethylbicyclo[2.2.1]hept-2-yl) Dithiophosphate

Excess of anhydrous gaseous ammonium was passed through the solution of **3** (3.0 g, 7.5 mmol) in anhydrous benzene (20 mL) for 1 h. The mixture was stored at ~20°C for ~12 h and evacuated under vacuum (0.5 mm Hg) at 40°C for 1 h and then under vacuum (0.02 mm Hg) at 40°C for 1 h to yield 1.5 g (48%) of salt **4**. mp 120–121 °C. Anal. found C 57.56; H 9.33; N 3.52; P 7.66; S 15.15. $C_{20}H_{38}NO_2PS_2$. Calcd. C 57.24; H 9.13; N 3.34; P 7.38; S 15.28. IR: suspension in vaseline oil, ν (cm^{-1}) = 3316, 3160 (H_4N^+); 2922, 2853 (CH_3 as, s; CH_2 as, s; CH); 1455 δ (CH_3 as), δ (H_4N^+); 1377 δ (CH_3 s); 1063, 1045 [(P)O-C]; 1008, 994, 974 (OC-C); 816 (PO_2 as, s); 664 (P=S); 586 (P-S). ^{31}P NMR: $\delta_P = 112.1$.

O,O-Di[(1*R*)-*endo*-(+)-1,3,3-trimethylbicyclo[2.2.1]hept-2-yl) *S*-(Methoxycarbonylmethyl)dithiophosphate

Methyl chloroacetate **5** (0.13 g, 1.2 mmol) was added dropwise under a dry argon atmosphere with stirring at 20°C to the solution of salt **4** (0.5 g, 1.2 mmol) in anhydrous benzene (10 mL), and the stirring was continued for 1 h at 20°C. The mixture was evaporated under vacuum (0.5 mm Hg) at 40°C for 1 h and then under vacuum (0.02 mm Hg) at 40°C for 1 h and gave 0.3 g (53%) of **6**. Anal. found C 58.03; H 8.12; P 6.33; S 13.55. $C_{23}H_{39}O_4PS_2$. Calcd. C 58.20; H 8.28; P 6.53; S 13.51. IR: film, ν (cm^{-1}) = 2954, 2872 (CH_3 as, s; CH_2 as, s; CH); 1744 (O-C=O); 1460 δ (CH_3 as); 1377 δ (CH_3 s); 1065 [(P)O-C]; 975 (OC-C); 799 (PO_2 as, s); 690 (P=S); 566 (P-S). ^{1}H NMR: δ = 0.87 s (6H, $C^8\underline{H}_3$); 1.00 and 1.09 two s [12H, $(C^{9,10}\underline{H}_3)_2C$]; 1.45 m (4H, $C^5\underline{H}_2$); 1.64 m (2H, C^4H); 1.69 m (8H, $C^6\underline{H}_2$,$C^7\underline{H}_2$); 3.75 s (3H, $O\underline{CH}_3$); 3.67 (2H, $PS\underline{CH}_2$, $^3J_{PH}$ = 16.1 Hz) and 3.79 (2H, $PS\underline{CH}_2$, $^3J_{PH}$ = 15.9 Hz); 4.15 d (2H, $POC^2\underline{H}$, $^3J_{PH}$ = 16.4 Hz) and 4.21 d (2H, $POC^2\underline{H}$, $^3J_{PH}$ = 13.0 Hz). ^{31}P NMR: δ_P = 99.7.

O,O-Di[(1*R*)-*endo*-(+)-1,3,3-trimethylbicyclo[2.2.1]hept-2-yl) *S*-Phenylcarbonyldithiophosphate

Benzoyl chloride **7** (0.17 g, 1.2 mmol) was added dropwise under dry argon under stirring at 20°C to the solution of salt **4** (0.5 g, 1.2 mmol) in anhydrous benzene (10 mL), and the stirring was continued for 1 h at 20°C. The mixture was evaporated under vacuum (0.5 mm Hg) at 40°C for 1 h and then under vacuum (0.02 mm Hg) at 40°C for 1 h and gave 0.5 g (83%) of **8**. Anal. found C 63.94; H 7.67; P 6.38; S 12.38. $C_{27}H_{39}O_3PS_2$. Calcd. C 64.00; H 7.76; P 6.11; S 12.66. IR: film, ν (cm^{-1}) = 3055 (= C-H, arom.); 2955, 2872 (CH_3 as, s; CH_2 as, s; CH); 1693 [(Ph)C=O]; 1582, 1462 (C = C, arom.); 1450 δ (CH_3 as); 1387 δ (CH_3 s); 1080 [(P)O-C]; 990 (OC-C); 772 (PO_2 as, s); 673 (P=S); 515 (P-S). ^{1}H NMR: δ = 0.88 s (6H, $C^8\underline{H}_3$) and 0.97 s (6H, $C^8\underline{H}_3$); 1.04 and 1.11 two s [12H, $(C^{9,10}\underline{H}_3)_2C$] and 1.06 and 1.13 two s [12H, $(C^{9,10}\underline{H}_3)_2C$]; 1.46 m (4H, $C^5\underline{H}_2$); 1.59 m (2H, $C^4\underline{H}$); 1.70 m (8H, $C^6\underline{H}_2C^7\underline{H}_2$); 4.39 d (1H, $POC^2\underline{H}$, $^3J_{PH}$ = 15.0 Hz) and 4.47 d (1H, $POC^2\underline{H}$, $^3J_{PH}$ = 14.7); 7.46–7.63 m [5H, $C_6\underline{H}_5C(O)$]. MS (EI), *m/e* (I_{rev}, %): 506 (5). ^{31}P NMR: δ_P = 84.0.

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