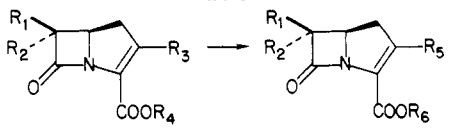
Table I



	substrate					product				
	R_1	R_2	R_3	R_4		R_1	R_2	R_5	R_6	yield, %
2a	H	Et	SCH_2CH_2NHAc	Bh	**3a**	H	Et	SCH_2CH_2NHAc	Na	56.3
2b	Me	Me	SCH_2CH_2NHAc	Bh	**3b**	Me	Me	SCH_2CH_2NHAc	Na	56.3
2c	H	Et	$SCH_2CH_2NHCO_2PMB$	PMB	**3c**	H	Et	$SCH_2CH_2NH_2$	H	53.6
2d	Et	H	$SCH_2CH_2NHCO_2PMB$	PMB	**3d**	Et	H	$SCH_2CH_2NH_2$	H	54.0
2e	H	Et	CH_2CO_2Et	PMB	**3e**	H	Et	CH_2CO_2Et	Na	64.0
2f	H	Et	CH_2CO_2Bh	PMB	**3f**	H	Et	CH_2CO_2Na	Na	82.0
2g	H	$Me_2C(OH)$	SCH_2CH_2NHAc	PMB	**3g**	H	$Me_2C(OH)$	SCH_2CH_2NHAc	Na	47.6
2h	H	$Me_2C(OH)$	$SCH_2CH_2NHCO_2PMB$	PMB	**3h**	H	$Me_2C(OH)$	$SCH_2CH_2NH_2$	H	56.7
2i	H	$MeOCH_2CH(OH)$[a]	SCH_2CH_2NHAc	PMB	**3i**	H	$MeOCH_2CH(OH)$[a]	SCH_2CH_2NHAc	Na	60.0
2j	H	$MeOCH_2CH(OH)$[a]	$SCH_2CH_2NHCO_2PMB$	PMB	**3j**	H	$MeOCH_2CH(OH)$[a]	$SCH_2CH_2NH_2$	H	36.9
2k	H	$MeOCH_2CO(OH)$[b]	SCH_2CH_2NHAc	PMB	**3k**	H	$MeOCH_2CH(OH)$[b]	SCH_2CH_2NHAc	Na	29.0
2l	H	$MeOCH_2CH(OH)$[b]	$SCH_2CH_2NHCO_2PMB$	PMB	**3l**	H	$MeOCH_2CH(OH)$[b]	$SCH_2CH_2NH_2$	H	24.0

[a] 8*S* isomer. [b] 8*R* isomer: see ref 6.

a mixture of anisole (0.8 mL) and CH_2Cl_2 (0.2 mL) was cooled to −50 °C and treated with aluminum trichloride (33.3 mg, 0.25 mmol). The reaction mixture was stirred for 30 min at −50 °C, quenched with aqueous 5% sodium bicarbonate solution (3 mL) at the same temperature, and partitioned between ethyl acetate and water. The resulting inorganic precipitates were removed by filtration and the filtrate was separated. The aqueous phase was passed through an HP-20 (30 mL) column, with elution with deionized water followed by freeze–drying, and gave 18.0 mg (56.3%) of **3a** as a pale yellow powder: mp 135–142 °C dec; identical in all respects (UV, IR, ^1H NMR, HPLC) except for biological activity (half of (+)-PS-5) with natural PS-5.

Compound **3b** (56.3%): pale yellow powder; mp 150 °C dec; IR (KBr) 1753, 1655, 1596, 1553; UV λ_{max} (H_2O) 300 nm (ϵ 8600). Anal. Calcd for $C_{13}H_{17}N_2O_4SNa\cdot H_2O$: C, 46.14; H, 5.67; N, 8.28. Found: C, 46.31; H, 6.00; N, 8.07. Compound **3c** (53.6%): mp 140 °C dec; IR (KBr) 1766, 1570 cm^{-1}; UV λ_{max} (H_2O) 297 nm (ϵ 6800). Anal. Calcd for $C_{11}H_{16}N_2O_3S\cdot 0.7H_2O$: C, 49.12; H, 6.53; N, 10.42. Found: C, 49.01; H, 6.43; N, 10.18. **3d** (54.0%): mp 140 °C dec; IR (KBr) 1773, 1595 cm^{-1}; UV λ_{max} (H_2O) 294 nm (ϵ 6800). Anal. Calcd for $C_{11}H_{16}N_2O_3S$: C, 51.53; H, 6.30; N, 10.93. Found: C, 51.11; H, 6.08; N, 10.92. **3e** (64.0%): mp 120 °C dec; IR (KBr) 1760, 1737, 1605 cm^{-1}; ^1H NMR (D_2O) δ 0.93 (s, 3 H), 1.20 (t, J = 7 Hz, 3 H), 1.73 (m, 2 H), 2.60–3.10 (m, 3 H), 3.60–4.26 (m, 3 H), 4.13 (q, J = 7 Hz, 2 H); UV λ_{max} (H_2O) 273 nm (ϵ 3200). **3f** (82.0%): mp 200 °C dec; IR (KBr) 1753, 1587 cm^{-1}; UV λ_{max} 270 nm (ϵ 2700). 6-Epicarpetimycin derivative **3g** (47.6%): colorless powder; IR (KBr) 3400, 1750, 1640, 1586, 1552, 1394 cm^{-1}; UV λ_{max} (H_2O) 302 nm (ϵ 8800); ^1H NMR (D_2O–Me_4Si as an external reference) δ 1.76 (s, 3 H), 1.81 (s, 3 H), 2.44 (s, 3 H), 3.30–3.90 (m, 6 H), 3.87 (d, J = 2.7 Hz, 1 H), 4.64 (td, J = 9, 2.7 Hz, 1 H). Anal. Calcd for $C_{14}H_{19}N_2O_5SNa\cdot 1.4H_2O$: C, 44.77; H, 5.85; N, 7.46. Found: C, 44.81; H, 5.64; N, 7.79. Compound **3h** (56.7%): pale yellow powder; IR (KBr) 3385, 1755, 1580, 1387 cm^{-1}; UV λ_{max} (H_2O) 298 nm (ϵ 6800); ^1H NMR [D_2O–Me_4Si (external)] δ 1.76 (s, 3 H), 1.81 (s, 3 H), 3.20–3.80 (m, 6 H), 3.91 (d, J = 2.8 Hz, 1 H), 4.67 (td, J = 9, 2.8 Hz, 1 H). Anal. Calcd for $C_{12}H_{18}N_2O_4S\cdot 1.5H_2O$: C, 45.99; H, 6.75; N, 8.94. Found: C, 46.13; H, 6.51; N, 8.85.

9-Methoxythienamycin derivative **3i** (60%): pale yellow powder; IR (KBr) 1750, 1655, 1590 cm^{-1}; UV λ_{max} (H_2O) 301 nm (ϵ 4800); ^1H NMR (D_2O) δ 1.98 (s, 3 H), 2.88, 2.98 (AB qd, J = 14, 7 Hz, 2 H), 3.10, 3.24 (AB qd, J = 17, 10, 8 Hz, 2 H), 3.39 (s, 3 H), 3.30–3.64 (m, 5 H), 4.10–4.32 (m, 2 H). Compound **3j** (36%): pale yellow powder; IR (Nujol) 1753, 1575 cm^{-1}; UV λ_{max} (H_2O) 299 nm (ϵ 6100); ^1H NMR (D_2O) δ 2.90–3.32 (m, 6 H), 3.40 (s, 3 H), 3.52, 3.60 (AB qd, J = 10, 6, 4, Hz, 2 H), 3.54 (m, 1 H), 4.16–4.34 (m, 2 H). Compound **3k** (29%): pale yellow powder, mp 130 °C dec; IR (KBr) 1755, 1660, 1610 cm^{-1}; UV λ_{max} (H_2O) 301 nm (ϵ 3900); ^1H NMR (D_2O) δ 1.98 (s, 3 H), 2.88, 2.97, (AB qd, J = 14, 8, 6 Hz, 2 H), 3.11, 3.25 (AB qd, J = 17, 9 Hz, 2 H), 3.30–3.70 (m, 5 H), 3.40 (s, 3 H), 4.10–4.32 (m, 2 H). Compound **3l** (24%): pale yellow powder; IR (KBr) 1755, 1583 cm^{-1}; UV λ_{max} (H_2O) 299 nm (ϵ 4600); ^1H NMR (D_2O) δ 2.86–3.65 (m, 9 H), 3.40 (s, 3 H), 4.10–4.30 (m, 2 H).

Registry No. (±)-**2a**, 93451-21-9; (±)-**2b**, 93349-51-0; (±)-**2c**, 93349-52-1; (±)-**2d**, 93349-53-2; (±)-**2e**, 93451-22-0; (±)-**2f**, 93349-54-3; **2g**, 93349-55-4; **2h**, 93349-56-5; **2i**, 93349-57-6; **2j**, 93349-58-7; **2k**, 93451-23-1; **2l**, 93451-24-2; (±)-**3a**, 77058-07-2; (±)-**3b**, 93451-25-3; (±)-**3c**, 93451-26-4; (±)-**3d**, 93451-27-5; (±)-**3e**, 93451-28-6; (±)-**3f**, 93451-29-7; **3g**, 82744-14-7; **3h**, 92936-51-1; **3i**, 93349-59-8; **3j**, 93349-60-1; **3b**, 93451-30-0; **3e**, 93451-31-1; $AlCl_3$, 7446-70-0.

Vicarious Substitution of Hydrogen with Carbanions of Dithioacetals of Aldehydes[1]

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In previous papers[2,3] we have shown that carbanions having leaving groups X at the carbanion centers react with aromatic nitro compounds, replacing the hydrogen ortho or para to the nitro group with the carbanion moiety. The reaction, termed vicarious nucleophilic substitution of hydrogen (VNS), proceeds via the formation of σ complexes followed by base-induced β-elimination of HX.[4]

We have shown that PhS, MeS, and $Me_2NC(S)S$ substituents are efficient leaving groups in the reaction; thus nitriles and esters containing these substituents in the α-position are suitable starting materials for this process.[3] These substituents not only are good leaving groups but also stabilize carbanions efficiently. $(PhS)_3CH$ has been recently shown to react with some nitroarenes in the presence of base to form dithioacetals of *p*-nitrobenzaldehyde derivatives.[5]

The valuable properties of PhS or other similar groups—namely, stabilization of carbanions, ability to depart in the β-elimination process, and resistance toward direct nucleophilic substitution (S_N2) can also be exploited for direct introduction of α-(RS)alkyl substituents into nitroaromatic rings via VNS with dithioacetals of aldehydes. These possibilities might not have been anticipated

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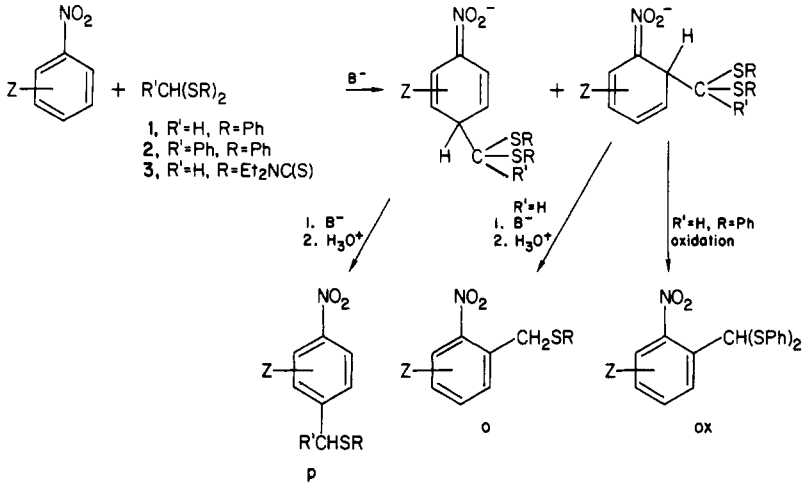
Table I



dithioacethal	Z	yields of isolated compd, %		
		p	o	ox
1[a]	H	14	27	4[d]
1[a]	4-Ph		27	14
1[a]	4-PhS		28	10
2[a]	H	52		
2[b]	H	53		
2[a]	2-PhS	31		
3[c]	H	27	23	
3[c]	4-Cl		55	
3[c]	4-Ph		25	

[a] *t*-BuOK/Me_2SO, room temperature. [b] *t*-BuOK/DMF, 0 °C. [c] *t*-BuOK/DMF, −10 °C. [d] Yield estimated from NMR spectra of crude reaction product. This compound was identified by comparison with an authentic sample.[9]

in view of a report that, in an *n*-BuLi/THF system, some dithioacetal carbanions react with nitroarenes mainly via an electron-transfer pathway, giving products of oxidative dimerization of the carbanions.[6] We sought to perform VNS using diphenyl dithioacetals of formaldehyde (1) and benzaldehyde (2) since they are sufficiently acidic (1, $pK_a(Me_2SO) = 30.8$; 2, $pK_a(Me_2SO) = 23.0$)[7] to form carbanions in a *t*-BuOK/Me_2SO system (*t*-BuOH $pK_a(Me_2SO = 32.2)$,[8] which was shown to be convenient for other VNS reaction. Reactions were carried out at room temperature for 30 min, with a great excess of base. Longer reaction time or use of a stoichometric amount of base caused lower yields of VNS products. Similar results were obtained in the *t*-BuOK/DMF system (2 h, 0 °C). Results of these reactions are presented in Table I. The carbanion from 1 reacts with nitrobenzene with the formation of two isomeric products of VNS ortho and para to the nitro group. The tertiary carbanion from 2 reacts exclusively at the para position. When this position is occupied, e.g., in *p*-nitrobiphenyl, the VNS does not proceed, possibly due to steric hindrance of the position ortho to the nitro group. For the same reason the carbanion from 2 does not react even with *m*-chloronitrobenzene although a chlorine atom is a smaller substituent than the nitro group.

In the reactions of the carbanion from 1, minor quantities of products from oxidation of the σ complexes (only ortho) were formed. Similar behavior of other carbanions have already been observed.[3b] In searching for dithioacetals that could enter VNS without formation of these byproducts, we found that dithioacetal 3 is suitable. Perhaps due to the higher nucleofugality of the $Et_2NC(S)S$ group than PhS, fast elimination precludes oxidation. The reactions of 3 were carried out in the *t*-BuOK/DMF system, 3 h at −10 °C, because these conditions gave better yields than the *t*-BuOK/Me_2SO system.

The results presented in this paper demonstrate that dithioacetal carbanions can react with nitroarenes according to the VNS principle. This reaction can serve as a method for introduction of α-(RS)alkyl substituent into the ring of aromatic nitro compounds.

Experimental Section

Potassium *tert*-Butoxide/Me_2SO System. General Procedure. A solution of 0.005 mol of dithioacetal and 0.005 mol of nitro compound in 10 mL of Me_2SO was added during 5 min to a stirring solution of 3.5 g of potassium *tert*-butoxide in 10 mL of Me_2SO which had been cooled to room temperature. Stirring was continued for an additional 25 min at room temperature. Then the reaction mixture was poured into an ice–HCl slurry. Extraction with CH_2Cl_2, drying over $MgSO_4$, and evaporation gave a residue that was chromatographed on silica gel with hexane–chloroform mixture as eluent or on aluminum oxide with hexane–ethyl acetate mixture as eluent.

2-Nitrobenzyl phenyl sulfide: mp 60–61 °C (EtOH) (lit.[10] mp 64 °C): NMR ($CDCl_3$) δ 4.48 (s, 2 H), 7.2–7.6 (m, 8 H), 8.05 (m, 1 H).

(1) Part 123 in the series Reactions of Organic Anions. Part 122: Mąkosza, M.; Kwast, A.; Jończyk, A.; Słomka, E., submitted for publication in *J. Org. Chem.*

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4-Nitrobenzyl phenyl sulfide: mp 74.5–76.5 °C (EtOH) (lit.[11] mp 76–77 °C); NMR ($CDCl_3$) δ 4.21 (s, 2 H), 7.34 (s, 5 H), 7.45 (d, 2 H), 8.23 (d, 2 H).

2-Nitro-5-phenylbenzyl phenyl sulfide: oil; NMR ($CDCl_3$) δ 4.55 (s, 2 H), 7.2–7.7 (m, 12 H), 8.18 (d, 1 H). Anal. Calcd for $C_{19}H_{15}NO_2S$: C, 71.01; H, 4.70; N, 4.36. Found: C, 71.15; H, 4.68; N, 4.13.

(2-Nitro-5-phenylphenyl)bis(phenylthio)methane: oil; NMR ($CDCl_3$) δ 6.68 (s, 1 H), 7.2–7.7 (m, 16 H), 8.03 (d, 1 H), 8.18 (s, 1 H). Anal. Calcd for $C_{25}H_{19}NO_2S_2$: C, 69.90; H, 4.46; N, 3.26. Found: C, 69.31; H, 4.37; N, 3.13.

(2-Nitro-5-(phenylthio)benzyl) phenyl sulfide: oil; NMR ($CDCl_3$) δ 4.40 (s, 2 H), 7.03 (s, 1 H), 7.18 (d, 1 H), 7.33 (s, 5 H), 7.50 (s, 5 H), 8.01 (d, 1 H). Anal. Calcd for $C_{25}H_{19}N_2O_2S_2$: C, 64.56; H, 4.28; N, 3.96. Found: C, 64.77; H, 4.21; N, 3.75.

(2-Nitro-5-(phenylthio)phenyl)bis(phenylthio)methane: mp 135–136.5 °C (EtOH); NMR ($CDCl_3$) δ 6.63 (s, 1 H), 7.08 (d, 1 H), 7.35 (d, 10 H), 7.53 (s, 5 H), 7.69 (s, 1 H), 7.84 (d, 1 H). Anal. Calcd for $C_{25}H_{19}NO_2S_3$: C, 65.05; H, 4.15; N, 3.03. Found: C, 64.81; H, 3.95; N, 3.50.

(4-Nitrophenyl)(phenylthio)phenylmethane: oil; NMR ($CDCl_3$) δ 5.46 (s, 1 H), 7.1–7.3 (m, 10 H), 7.41 (d, 2 H), 7.98 (d, 2 H). Anal. Calcd for $C_{19}H_{15}NO_2S$: C, 71.01; H, 4.70; N, 4.36. Found: C, 70.83; H, 4.51; N, 4.34.

(4-Nitro-3-(phenylthio)phenyl)(phenylthio)phenylmethane: mp 86–87.5 °C (EtOH); NMR ($CDCl_3$) δ 5.19 (s, 1 H), 6.83 (s, 1 H), 7.1–7.4 (m, 16 H), 8.06 (d, 1 H). Anal. Calcd for $C_{25}H_{19}N_2O_2S_2$: C, 69.90; H, 4.46; N, 3.26. Found: C, 69.77; H, 4.31; N, 3.05.

Potassium *tert*-Butoxide/DMF System. General Procedure. A solution of 1.55 g (0.005 M) of **3** and 0.005 M nitrocompound in 10 mL of DMF was slowly added to stirred solution of 3.5 g of potassium *tert*-butoxide in 10 mL of DMF, which had been cooled to −10 °C. Stirring was continued for an additional 3 h at −10 °C. Workup and isolation of products proceed as in previous procedure.

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2-Nitrobenzyl diethyldithiocarbamate: mp 28–29 °C (EtOH); NMR ($CDCl_3$) δ 1.28 (t, 6 H), 3.93 (m, 4 H), 5.08 (s, 2 H), 7.48–7.78 (m, 2 H), 7.98 8.19 (m, 2 H). Anal. Calcd for $C_{12}H_{16}N_2O_2S_2$: C, 50.68; H, 5.67; N, 9.85. Found: C, 50.07; H, 5.67; N, 9.82.

4-Nitrobenzyl diethyldithiocarbamate: mp 76–77 °C (EtOH) (lit.[12] 75–76 °C); NMR ($CDCl_3$) δ 1.30 (t, 6 H), 3.93 (m, 4 H), 4.75 (s, 2 H), 7.65 (d, 2 H), 8.25 (d, 2 H).

5-Chloro-2-nitrobenzyl diethyldithiocarbamate: mp 74–75.5 °C (EtOH); NMR ($CDCl_3$) δ 1.28 (t, 6 H), 3.98 (m, 4 H), 5.05 (s, 2 H), 7.48 (d, 1 H), 7.96 (s, 1 H), 8.08 (d, 1 H). Anal. Calcd for $C_{12}H_{15}N_2ClO_2S_2$: C, 45.20; H, 4.74; N, 8.79. Found: C, 45.28; H, 4.78; N, 8.86.

2-Nitro-5-phenylbenzyl diethyldithiocarbamate: mp 87–88 °C (EtOH); NMR ($CDCl_3$) δ 1.23 (t, 6 H), 3.93 (m, 4 H), 5.13 (s, 2 H), 7.4–7.8 (m, 6 H), 8.18 (d, 1 H), 8.23 (s, 1 H). Anal. Calcd for $C_{18}H_{20}N_2O_2S_2$: C, 59.97; H, 5.59; N, 7.78. Found: C, 59.77; H, 5.47; N, 7.65.

Registry No. **1**, 3561-67-9; **2**, 7695-69-4; **3**, 22656-77-5; nitrobenzene, 98-95-3; 4-phenyl-1-nitrobenzene, 92-93-3; 4-(phenylthio)-1-nitrobenzene, 952-97-6; 2-(phenylthio)-1-nitrobenzene, 4171-83-9; 4-chloro-1-nitrobenzene, 100-00-5; 2-nitrobenzyl phenyl sulfide, 91718-67-1; 4-nitrobenzyl phenyl sulfide, 7703-38-0; 2-nitro-5-phenylbenzyl phenyl sulfide, 93304-88-2; (2-nitro-5-phenylphenyl)bis(phenylthio)methane, 93304-89-3; 2-nitro-5-(phenylthio)benzyl phenyl sulfide, 93304-90-6; (2-nitro-5-(phenylthio)phenyl)bis(phenylthio)methane, 93304-91-7; (4-nitrophenyl)(phenylthio)phenylmethane, 93304-92-8; (4-nitro-3-(phenylthio)phenyl)(phenylthio)phenylmethane, 93304-93-9; 2-nitrobenzyl diethyldithiocarbamate, 93304-94-0; 4-nitrobenzyl diethyldithiocarbamate, 28371-57-5; 5-chloro-2-nitrobenzyl diethyldithiocarbamate, 93304-95-1; 2-nitro-5-phenylbenzyl diethyldithiocarbamate, 93304-96-2; (2-nitrophenyl)bis(phenylthio)methane, 93304-97-3.

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Communications

Ligand-Assisted Catalysis: New Active and Selective Nickel Modified Homogeneous Catalysts for Linear Dimerization of Butadiene

Summary: New aminophosphinite-modified Ni(0) catalysts have been found to be the most active ever described for linear dimerization of butadiene, affording almost exclusively 1,3,6- and 2,4,6-octatrienes, according to the reaction conditions.

Sir: Linear dimerization of butadiene into mixtures of octatrienes has been effected on Ni^0 catalysts, using nickel carbonyl phosphine or phosphite complexes in the presence of alcohols[1,2] or morpholine (morpholine/Ni = 50),[3] in which case the products are almost exclusively (*Z,E*)- and (*E,E*)-1,3,6-octatrienes. More recent results by Pittman have shown that upon reduction of $NiBr_2(PPh_3)_2$ by sodium borohydride in a mixture of THF and ethanol, the catalyst gave selectively the (*E,E*)-1,3,6-octatriene, with yields up to 95%.[4] We now report unprecedented results for linear dimerization of butadiene which occur at ambient temperature with nickel catalysts, and where a mixture of (*E,E*)- and (*Z,E*)-1,3,6-octatrienes is obtained selectively, before the occurrence of an isomerization process which gives essentially the (*E,E,E*)-2,4,6-octatriene. As a consequence of our recent studies in the field of synthesis of new aminophosphine–phosphinite ligands for asymmetric catalytic reactions,[5] we have synthesized a series of aminophosphinite compounds, which combine both characteristics desired for linear dimerization of butadiene on nickel systems: (i) a ligand containing at least one (–P–O–) moiety; (ii) a pendant N–H group at the other side of the chain, favoring a stereochemical approach of the proton in close proximity to the metal. These ligands

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