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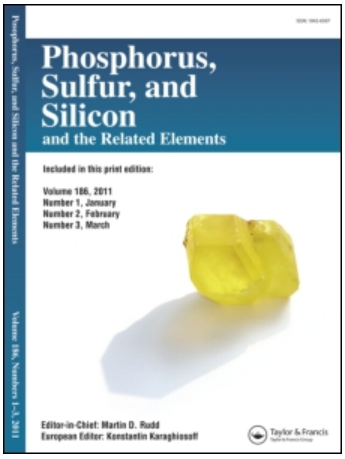

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NOVEL REACTION OF ARYLAZO ARYL AND ARYLAZOXY ARYL SULFONES WITH NORBORNENE CATALYZED BY A PALLADIUM(0) COMPLEX

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NOVEL REACTION OF ARYLAZO ARYL AND ARYLAZOXY ARYL SULFONES WITH NORBORNENE CATALYZED BY A PALLADIUM(0) COMPLEX

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The reactions of arylazo aryl sulfone and arylazoxy aryl sulfones with norbornene in the presence of a tetrakis(triphenylphosphine)palladium(0) were found to give *cis,exo*-1,2,3,4,4a,9b-hexahydro-1,4-methanodibenzothiophene 5,5-dioxides in good yield. The reaction mechanism involving aryl(arylsulfonyl)palladium(II) species as a common intermediate in both reactions is proposed. The result of an X-ray single crystal structure analysis of the novel reaction product is also described.

Key words: Arylazo aryl sulfone; arylazoxy aryl sulfone; arylsulfonylation; X-ray diffraction.

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INTRODUCTION

Recently, much attention has been paid to the palladium catalyzed reactions in organic syntheses.[1] Arylation of alkenes with aryl halides or aryl mercurious compounds catalyzed by a palladium complex is well known as the Heck reaction.[2] Previously, we reported a novel arylation of olefins or α,β-unsaturated esters and nitriles with arylazo aryl sulfones[3] (**1**) and arylazoxy aryl sulfones[4] (**2**) catalyzed by a palladium(0) complex and proposed a reaction mechanism involving oxidative addition of **1** (or **2**) to the palladium(0) catalyst and the subsequent extrusion of nitrogen (or nitrous oxide) and sulfur dioxide to give a diarypalladium(II) intermediate. In the course of our systematic studies on the palladium(0) catalyzed reactions of arylazo aryl sulfones (**1**) and arylazoxy aryl sulfones (**2**) with olefins, we found a hitherto unknown tandem arylsulfonylation and cyclization of **1** and **2** with norbornene.

RESULTS AND DISCUSSION

When the reaction of phenylazo phenyl sulfone (**1a**) with norbornene was carried out in the presence of a catalytic amount of tetrakis(triphenylphosphine)palladium(0) in benzene in a degassed sealed tube at 80°C, *cis,exo*-1,2,3,4,4a,9b-hexahydro-1,4-methanodibenzothiophene 5,5-dioxide (**3a**) and biphenyl (**4a**) were obtained in 45 and 33% yield, respectively. The structure of **3a** was determined by

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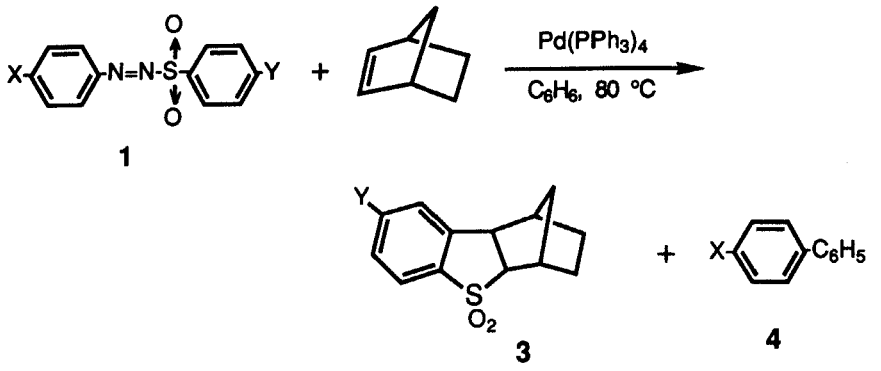

TABLE I

Reactions of **1** and **2** with norbornene catalyzed by a palladium(0) complex

	X and Y in **1** or **2**		Products, Yield/%	
	X	Y		
1a	H	H	**3a** 45	**4a** 23
1b	H	CH_3	**3b** 50	**4a** 28
1c	CH_3	H	**3a** 52	**4b** 30
1d	CH_3	CH_3	**3b** 53	**4b** 35
2a	H	H	**3a** 65	**4a** 43
2b	H	CH_3	**3b** 70	**4a** 45
2c	CH_3	H	**3a** 62	**4b** 40
2d	CH_3	CH_3	**3b** 80	**4b** 43
2e	Cl	Cl	**3c** 71	**4c** 46

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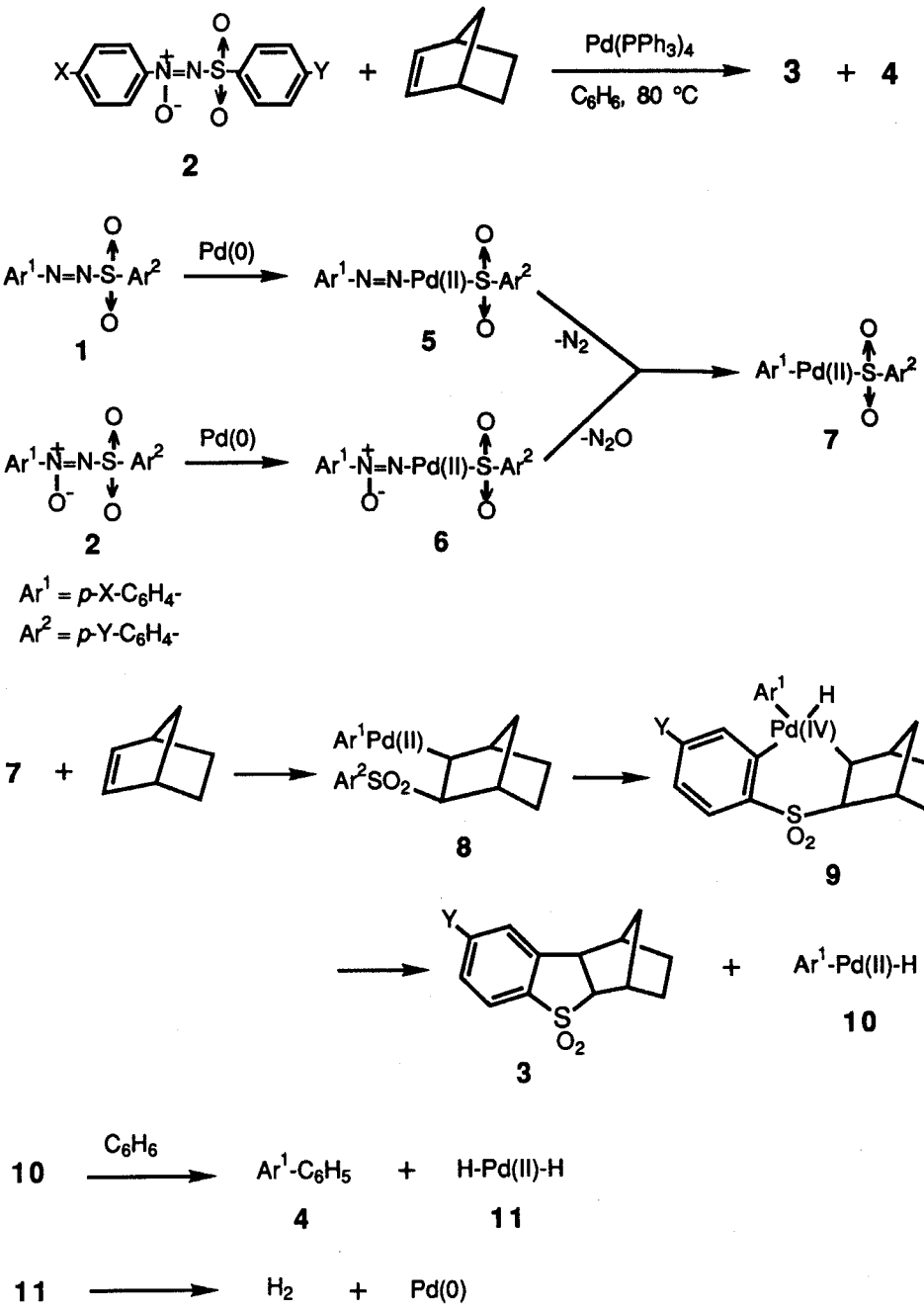
the characteristic ^1H NMR spectra; i.e. the AB quartet signals of the two endo protons bonded to 4a and 9b carbons were appeared at 3.27 and 3.54 ppm (J = 8.0 Hz) and four (not five) aromatic protons were observed at 7.36–7.70 ppm. Similarly, the reactions of phenylazo *p*-tolyl sulfone (**1b**), *p*-tolylazo phenyl sulfone (**1c**), and *p*-tolylazo *p*-tolyl sulfone (**1d**), with norbornene were carried out in the presence of a palladium(0) catalyst to give *cis,exo*-1,2,3,4,4a,9b-hexahydro-1,4-methanodibenzothiophene 5,5-dioxide derivative (**3**) and biaryl (**4**) in good yields. The results are summarized in Table I.

It is of interest that the palladium(0) catalyzed reactions of **1** with norbornene give selectively arylsulfonylated compounds and that no arylated compound is obtained in contrast to the reaction of **1** with acyclic and cyclic alkenes.[3]

The reaction of phenylazoxy phenyl sulfone (**2a**) with norbornene was also carried out in the presence of a palladium(0) catalyst in benzene in a degassed sealed tube at 80°C to give **3a** (65%) and biphenyl (43%). Similarly, the reactions of arylazoxy aryl sulfone (**2b–e**) with norbornene catalyzed by the palladium(0) complex were carried out to give the corresponding *cis,exo*-1,2,3,4,4a,9b-hexahydro-1,4-methanodibenzothiophene 5,5-dioxide derivative (**3**) and biaryl in good yield. The results are shown in Table I.



The fact that the same products were obtained in either reactions of **1** and **2** with norbornene in the presence of the palladium(0) catalyst suggests the formation of a common reactive intermediate. The reactions are accounted for by the oxidative additions of **1** and **2** to the palladium(0) catalyst giving the arylazo(arylsulfonyl)palladium(II) (**5**) and arylazoxy(arylsulfonyl)palladium(II) (**6**), respectively.

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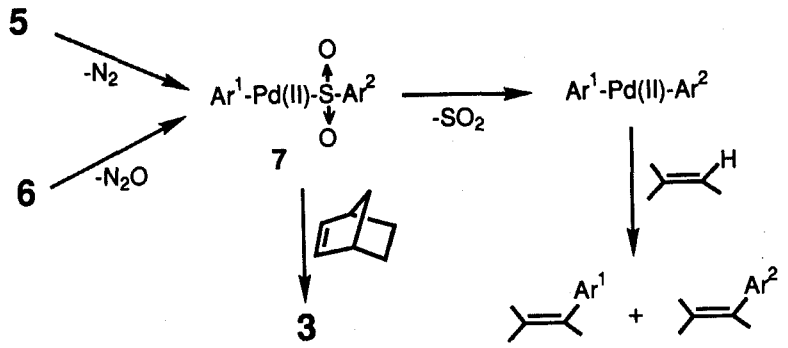

Nitrogen or nitrous oxide preferentially eliminates from **5** or **6** to give a common intermediate, aryl(arylsulfonyl)palladium(II) (**7**). Addition of **7** to norbornene affords the syn-adduct **8** which undergoes subsequent intramolecular ortho-palladation to give the palladium(IV) complex **9**. The reductive elimination of **3** from **9** forms arylhydrogenpalladium(II) (**10**). The arylhydrogenpalladium(II) (**10**) eliminated reacts with the solvent benzene to afford a biaryl and dihydrogenpalladium(II) (**11**) which extrudes hydrogen and palladium(0), although no hydrogen could be detected. Thus, the palladium(0) catalyst is regenerated and which participates in the subsequent catalytic cycle. The formation of biaryl by the reaction of **10** with solvent benzene was supported by the further experimental results; i.e., the palladium(0) catalyzed reaction of **1a** (or **2a**) with norbornene in toluene gave a mixture of ortho-, meta-, and para-methylbiphenyls as well as **3a**.

Previously, we proposed the formation of diarylpalladium(II) species by the oxidative addition of **1** (or **2**) to the palladium(0) catalyst giving the adduct **5** (or

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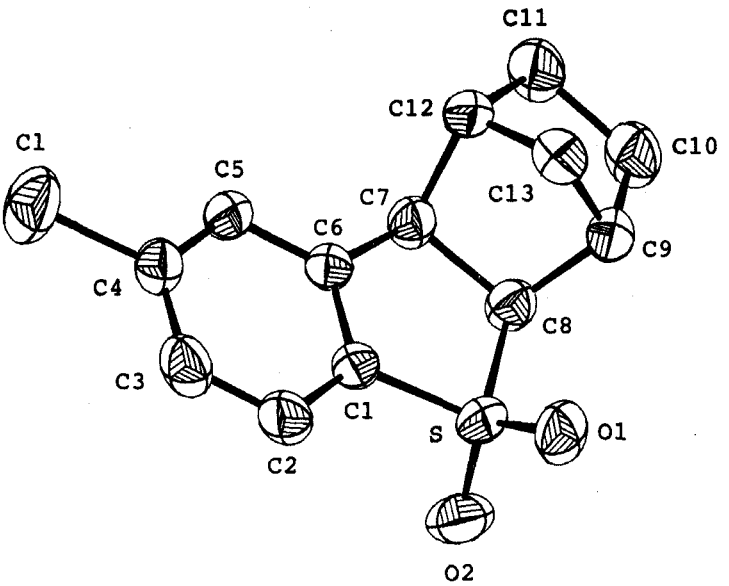

FIGURE 1 ORTEP drawing of compound **3c**, showing the numbering scheme of the atoms.

TABLE II

Positional parameters and equivalent isotropic thermal parameters with e.s.d. in parentheses

ATOM	X	Y	Z	B (eq)
S	0.12523(5)	0.22503(4)	0.34347(4)	3.78 (1)
Cl	0.87644(6)	0.24054(5)	0.76450(4)	6.76 (2)
O(1)	-0.0346 (1)	0.0720 (1)	0.2530 (1)	4.59 (3)
O(2)	0.0657 (1)	0.3408 (1)	0.4227 (1)	5.61 (4)
C(1)	0.3200 (2)	0.2113 (1)	0.4643 (1)	3.18 (4)
C(2)	0.3005 (2)	0.1538 (2)	0.5735 (1)	4.32 (5)
C(3)	0.4728 (2)	0.1609 (2)	0.6643 (2)	4.68 (5)
C(4)	0.6598 (2)	0.2259 (2)	0.6458 (1)	3.86 (4)
C(5)	0.6786 (2)	0.2824 (2)	0.5370 (1)	3.66 (4)
C(6)	0.5043 (2)	0.2742 (1)	0.4446 (1)	3.23 (4)
C(7)	0.5047 (2)	0.3387 (2)	0.3247 (1)	3.53 (4)
C(8)	0.2831 (2)	0.3139 (2)	0.2498 (1)	3.72 (4)
C(9)	0.2313 (2)	0.2087 (2)	0.0928 (1)	4.07 (5)
C(10)	0.3571 (2)	0.3130 (2)	0.0254 (2)	5.40 (6)
C(11)	0.5769 (2)	0.3356 (2)	0.0965 (2)	5.74 (6)
C(12)	0.5532 (2)	0.2458 (2)	0.1987 (1)	4.57 (5)
C(13)	0.3453 (2)	0.1058 (2)	0.1095 (2)	4.38 (5)

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6) and the subsequent extrusion of nitrogen (or nitrous oxide) and sulfur dioxide in the reaction of **1** (or **2**) with alkenes, however, it has not been clarified that which one is eliminated in advance between nitrogen (or nitrous oxide) and sulfur dioxide.[3,4] The present reaction of **1** (or **2**) with norbornene revealed that nitrogen (or nitrous oxide) is preferentially extruded from **5** (or **6**). It is presumed that **7** reacts with norbornene, a reactive olefin, before which eliminates sulfur dioxide giving diarylpalladium(II) in contrast to the reactions of **7** with usual alkenes reported previously.[3,4]

Recently, Catellani and Chiusoli have reported an interesting reaction of bromobenzene with norbornene catalyzed by a palladium(0) complex in the presence of potassium *t*-butoxide to give double arylation and cyclization product, hexahydromethanotriphenylene.[5] However, to our knowledge, the present palladium(0) catalyzed tandem arylsulfonylation and cyclization is a hitherto unknown reaction.

X-Ray Single Crystal Structure Analysis of **3c**. Since the structure of **3** was chiefly assigned by ^{1}H and ^{13}C NMR, the X-ray diffraction of compound **3c** was determined. Figure 1 shows an ORTEP drawing of compound **3c**. The numbering given in Figure 1 is arbitrary and is not consistent with that of the IUPAC nomenclature. Positional parameters and equivalent isotropic thermal parameters are summarized in Table II. Bond length and bond angles are listed in Table III. These results clearly show that the five-membered ring involving sulfur atom of **3c** lie in *exo*-direction for the norbornane skeleton, and thus the *exo*-structure of **3c** assigned by NMR is confirmed.

TABLE III

Bond distances (Å) and bond angles (degrees) with e.s.d. in parentheses

(a) Bond Distances

ATOM	ATOM	DISTANCE	ATOM	ATOM	DISTANCE
S	O(1)	1.4315 (8)	C(5)	C(6)	1.388 (2)
S	O(2)	1.443 (1)	C(6)	C(7)	1.516 (2)
S	C(1)	1.752 (1)	C(7)	C(8)	1.557 (2)
S	C(8)	1.803 (2)	C(7)	C(12)	1.535 (2)
Cl	C(4)	1.725 (1)	C(8)	C(9)	1.524 (2)
C(1)	C(2)	1.385 (2)	C(9)	C(10)	1.540 (2)
C(1)	C(6)	1.373 (2)	C(9)	C(13)	1.524 (2)
C(2)	C(3)	1.373 (2)	C(10)	C(11)	1.543 (2)
C(3)	C(4)	1.388 (2)	C(11)	C(12)	1.530 (3)
C(4)	C(5)	1.374 (2)	C(12)	C(13)	1.533 (2)

(b) Bond Angles

ATOM	ATOM	ATOM	ANGLE	ATOM	ATOM	ATOM	ANGLE
O(1)	S	O(2)	116.78(6)	C(5)	C(6)	C(7)	123.8 (1)
O(1)	S	C(1)	111.43(6)	C(6)	C(7)	C(12)	113.8 (1)
O(1)	S	C(8)	113.53(6)	C(6)	C(7)	C(8)	108.5 (1)
O(2)	S	C(1)	109.16(6)	C(8)	C(7)	C(12)	102.46(9)
O(2)	S	C(8)	108.16(7)	C(9)	C(8)	C(7)	103.8 (1)
C(1)	S	C(8)	95.70(6)	C(9)	C(8)	S	114.43(8)
C(2)	C(1)	S	126.0 (1)	C(7)	C(8)	S	107.5 (1)
C(6)	C(1)	S	111.7 (1)	C(8)	C(9)	C(10)	106.6 (1)
C(2)	C(1)	C(6)	122.2 (1)	C(8)	C(9)	C(13)	102.1 (1)
C(1)	C(2)	C(3)	118.2 (1)	C(10)	C(9)	C(13)	101.4 (1)
C(2)	C(3)	C(4)	119.8 (2)	C(9)	C(10)	C(11)	103.2 (2)
C(3)	C(4)	Cl	119.4 (1)	C(10)	C(11)	C(12)	103.4 (1)
C(5)	C(4)	Cl	118.7 (1)	C(7)	C(12)	C(11)	108.3 (1)
C(3)	C(4)	C(5)	121.9 (1)	C(7)	C(12)	C(13)	101.9 (1)
C(4)	C(5)	C(6)	118.2 (1)	C(11)	C(12)	C(13)	101.0 (1)
C(1)	C(6)	C(5)	119.6 (1)	C(9)	C(13)	C(12)	94.8 (1)
C(1)	C(6)	C(7)	116.6 (1)				

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EXPERIMENTAL

Melting points were determined on a Yamato Model MP-21 melting point apparatus and are uncorrected. IR spectra were recorded on a Hitachi Model 260-10 spectrophotometer with KBr disks. ^{1}H NMR spectra were recorded on a Varian XL-GEM 200 FT NMR (200 MHz) spectrometer. ^{13}C NMR spectra were measured on a JOEL JNM Fx 90Q FT NMR (22.5 MHz) spectrometer. Mass spectra were determined with a JEOL JMS-DX300 mass spectrometer with a JEOL JMA-5000 Mass Data System at an ionizing voltage of 70 eV. Gas chromatography (GC) was carried out with Hitachi Model 163 and 263-30 gas chromatographs (FID) with a 1 m column packed with 10% SE-30. The gel permeation chromatography was performed with a JAI LC-08 liquid chromatograph with two JAIGEL-1H columns (20 mm × 600 mm) with chloroform as eluent. Phenylazo phenyl sulfone (**1a**) (mp 75–76°C (76–77°C)), phenylazo *p*-tolyl sulfone (**1b**) (mp 90–91°C (90–91°C)), *p*-tolylazo phenyl sulfone (**1c**) (mp 82–83°C),

and *p*-tolylazo *p*-tolyl sulfone (**1d**) (mp 96–97°C (96–97°C)) were prepared by the published method.[6] Phenylazoxy phenyl sulfone (**2a**) (mp 119–121°C (123°C)), phenylazoxy *p*-tolyl sulfone (**2b**) (mp 109–110°C (112–113°C)), phenyl *p*-tolylazoxy sulfone (**2c**) (mp 80–82°C), *p*-tolylazoxy *p*-tolyl sulfone (**2d**) (mp 102–104°C (106°C)), and *p*-chlorophenylazoxy *p*-chlorophenyl sulfone (**2e**) (mp 174.5–175.0°C) were prepared by the published procedures.[7] Tetrakis(triphenylphosphine)palladium(0) was prepared by the method described in the literature.[8] Norbornene of Tokyo Kasei Chemicals was purified by distillation under nitrogen prior to use.

General procedure for the reaction of arylazo aryl sulfone (**1**) *or arylazoxy aryl sulfone* (**2**) *with norbornene.* A solution containing arylazo aryl sulfone (**1**) or arylazoxy aryl sulfone (**2**) (1.0 mmol), norbornene (1.0 mmol), and tetrakis(triphenylphosphine)palladium(0) (0.01 mmol) in benzene (5 ml) was degassed by a freeze-thaw cycle, sealed in an ampoule, and heated at 80°C for 24 h. The reaction mixture was subjected to short column chromatography on Florisil using benzene as an eluent to remove the metal complex. The products were isolated from the reaction mixture by column chromatography on Florisil using hexane:ethyl acetate:chloroform (50:25:1) as the eluent and/or by gel-permeation chromatography, and the structures were determined by IR, NMR, and mass spectroscopy. The structures of biphenyl, *p*-methylbiphenyl, and *p*-chlorobiphenyl were identified by comparison with their IR, ^{1}H NMR, and mass spectral data with those of authentic samples.

cis,exo-1,2,3,4,4a,9b-Hexahydro-1,4-methanodibenzothiophene 5,5-dioxide (**3a**): mp 137–138°C; IR (KBr) 1310 and 1145 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ = 1.10 (1H, d, J = 11.0 Hz), 1.22 (1H, d, J = 11.0 Hz), 1.26–1.78 (4H, m), 2.47 (1H, br s), 2.93 (1H, br s), 3.27 and 3.54 (2H, ABq, J = 8.0 Hz), and 7.36–7.70 (4H, m); ^{13}C NMR ($CDCl_3$) δ = 27.4, 28.1, 33.6, 39.1, 43.0, 50.1, 66.1, 121.2, 126.7, 128.3, 128.8, 133.5, and 139.3; MS *m/z* 234 (M^+), 216, 199, 167, 142, 137, and 129; HRMS: Found 234.0676, Calcd for $C_{13}H_{14}O_2S$: M, 234.0714.

cis,exo-8-Methyl-1,2,3,4,4a,9b-hexahydro-1,4-methanodibenzothiophene 5,5-dioxide (**3b**): mp 116–117°C; IR (KBr) 1320 and 1150 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ = 1.12 (1H, d, J = 11.1 Hz), 1.18 (1H, d, J = 11.1 Hz), 1.27–1.73 (4H, m), 2.39 (3H, s), 2.46 (1H, br s), 2.92 (1H, br s), 3.23 and 3.47 (2H, ABq, J = 7.8 Hz), 7.16 (1H, s), and 7.21 and 7.54 (2H, ABq, J = 8.0 Hz); ^{13}C NMR ($CDCl_3$) δ = 21.7, 27.4, 28.1, 33.6, 39.1, 43.0, 49.9, 66.5, 120.9, 126.9, 129.9, 138.5, 139.7, and 144.5; MS *m/z* 248 (M^+), 231, 213, 201, 181, 156, 151, and 143; HRMS: Found 248.0914, Calcd for $C_{14}H_{16}O_2S$: M, 248.0871.

cis,exo-8-Chloro-1,2,3,4,4a,9b-hexahydro-1,4-methanodibenzothiophene 5,5-dioxide (**3c**): mp 131–132°C; IR (KBr) 1320 and 1160 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ = 1.11 (1H, d, J = 11.0 Hz), 1.20 (1H, d, J = 11.0 Hz), 1.28–1.76 (4H, m), 2.48 (1H, br s), 2.94 (1H, br s), 3.28 and 3.50 (2H, ABq, J = 7.7 Hz), 7.35 and 7.60 (2H, ABq, J = 9.2 Hz), and 7.37 (1H, s); ^{13}C NMR ($CDCl_3$) δ = 27.3, 28.1, 33.8, 39.2, 43.0, 49.7, 66.6, 122.5, 126.9, 129.5, 139.7, 140.4, and 141.4; MS m/z 268 (M^+), 234, 216, and 199; HRMS: Found 268.0356, Calcd for $C_{13}H_{13}ClO_2S$: M, 268.0324.

X-Ray crystal structure analysis of **3c**. Dimensions of the colorless crystal of **3c** used for the measurement were 0.75 × 0.45 × 0.02 mm^3. The intensity data collected for $3° \leqq 2\theta \leqq 55°$ by the ω-2θ scan technique using graphite-monochromated MoK_α radiation (λ = 0.7117 Å). Independent 3641 points were collected and 2936 reflections with $F_0 > 3.0\sigma(F_0)$ were used for the analysis.

Crystallographic data: $C_{13}H_{13}O_2SCl$, F.W. = 245.6, triclinic, $P\bar{1}$, a = 7.380(4), b = 9.679(6), c = 10.174(7) Å, α = 105.96(5), β = 102.15(3), τ = 109.67(6)°, V = 620.0(3) Å^3, Z = 2, $D_{calc.}$ = 1.32 gcm^{-3}, μ(Mo − K_α) = 4.05 cm^{-1}. The structure was solved by the Monte-Carlo direct methods[9] by use of MULTAN-78 program.[10] The full-matrix least squares refinement for non-H atoms was carried out for $\Sigma w(|F_0|^2 - |F_c|^2)^2$, where the weight $w = 1.0/[\sigma^2(|F_0|) + 0.004(|F_0|^2)]$. The final discrepancy factors were R = 0.054 and R_w = 0.070, and $(\Delta/\sigma)_{max}$ in the final refinement cycle was 0.29. The scattering factors were taken from Reference 11. The calculations were carried out on a Mac Science MXC18 SYSTEM, and ORTEP[12] was employed for drawing molecular structure.

REFERENCES

1. J. Tsuji, Organic Synthesis with Palladium Compounds, Springer-Verlag, New York, 1980; R. F. Heck, Palladium Reagents in Organic Syntheses, Academic Press, New York, 1985.
2. R. F. Heck, *Pure Appl. Chem.*, **50,** 691 (1978); R. F. Heck, *Acc. Chem. Res.*, **12,** 146 (1979).
3. N. Kamigata, T. Kondoh, M. Kameyama, T. Satoh and M. Kobayashi, *Chem. Lett.*, **1987,** 345; N. Kamigata, A. Satoh, T. Kondoh, and M. Kameyama, *Bull. Chem. Soc. Jpn.*, **61,** 3575 (1988); N. Kamigata, A. Satoh, M. Yoshida, and M. Kameyama, ibid., **62,** 605 (1989).

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4. N. Kamigata, T. Fukushima, A. Satoh, and M. Kameyama, *Sulfur Lett.*, **7,** 181 (1988); N. Kamigata, T. Fukushima, A. Satoh, and M. Kameyama, *J. Chem. Soc., Perkin Trans.*, 1, **1990,** 549; N. Kamigata, M. Satoh, and T. Fukushima, *Bull. Chem. Soc. Jpn.*, **63,** 2118 (1990).
5. M. Catellani and G. P. Chiusoli, *J. Organomet. Chem.*, **286,** C13 (1985).
6. A. Hantzsch and M. Singer, Ber., **30,** 312 (1897); idem., **31,** 636 (1988); M. Kojima, H. Minato, and M. Kobayashi, *Bull. Chem. Soc. Jpn.*, **45,** 2032 (1972).
7. W. V. Farrar and J. M. Gulland, *J. Chem. Soc.*, **1944,** 368.
8. D. R. Coulson, *Inorg. Synth.*, **13,** 12 (1972).
9. A. Furusaki, *Acta Cryst.*, **A35,** 220 (1979).
10. P. Main, S. E. Hull, L. Lessimger, G. Germain, J. P. Declercq and M. M. Woolfson, MULTAN 78, Univ. of York, England, and Louvain, Belgium.
11. "International Tables for X-Ray Crystallography," Kynock Press, Birmingham (1974), Vol. IV.
12. C. K. Johnson, ORTEP, Report ORNL-3794 (1965), Oak Ridge National Laboratory, Tennessee.

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