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Diels–Alder Reactions of Anthracenes with *C*-Sulfonyldithioformates

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ABSTRACT: C-*Sulfonyldithioformates (***2***) (R^1 = $ArSO_2$, R^2 = ArS) readily add to anthracene and 9-methylanthracene (***1***) in a Diels–Alder fashion with formation of 9,10-dihydro-10,9-(epithiomethano)-anthracenes (***3***) which in turn may suffer thermally induced elimination of arenesulfinic acid to yield the 9-anthracenedithiocarboxylic esters (***4***). The reactions with the unsymmetrical diene 9-methylanthracene take place in a highly stereoselective fashion.* © 2003 Wiley Periodicals, Inc. Heteroatom Chem 14:170–174, 2003; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.10119

INTRODUCTION

Anthracene and anthracene derivatives **1** can be used as diene components of Diels–Alder reactions. Adducts between **1** and thiocarbonyl compounds **2**, i.e. 9,10-dihydro-10,9-(epithiomethano)anthracenes (**3**), play an important role in the trapping of elusive thiocarbonyl compounds such as thioaldehydes and thioketenes and can in turn in a retro-Diels–Alder reaction regenerate the thiocarbonyl compound **2** [1].

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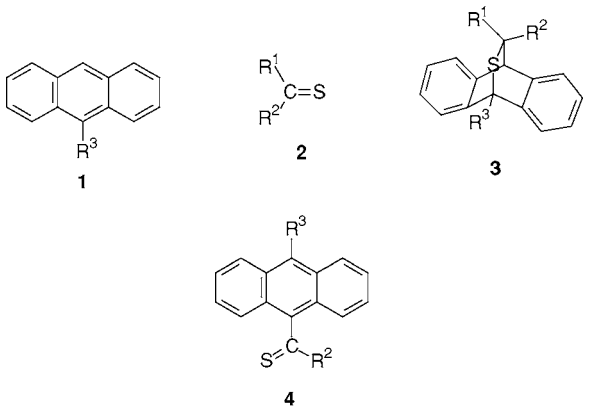
RESULTS AND DISCUSSION

In 1976 Allgeier and Winkler [1b] reported the facile Diels–Alder addition of thiophosgene **2** (R^1 = R^2 = Cl) to anthracene **1** (R^3 = H) and to 9-alkylanthracenes **1** (R^3 = alkyl), the latter taking place in a regiospecific fashion, most likely determined by steric factors (cf. Scheme 1).

Another neat Diels–Alder reaction was observed by Maletzko and Sundermeyer [1j] in their study of the addition of trichlorothioacetyl chloride to **1** (R = H). However, when an analogous reaction was attempted with trichloromethyl chlorodithioformate **2** (R^1 = Cl, R^2 = CCl_3S) the Friedel–Crafts substitution product **4** (R^2 = CCl_3S, R^3 = H) was obtained rather than the Diels–Alder adduct **3** (R^1 = Cl, R^2 = CCl_3S, R^3 = H) [2]. Conceivably, **3** is the primary product of the reaction and subsequently forms **4** by ring-opening fragmentation (cf. Scheme 2 for the conversion of **3b** to **4c** as an example).

Following earlier studies of Diels–Alder reactions of *C*-sulfonyldithioformates **2** (R^1 = $ArSO_2$, R^2 = ArS) with aliphatic 1,3-dienes by ourselves [3] and others [4] (according to their detailed kinetic data Sauer et al. found **2** (R^1 = $ArSO_2$, R^2 = ArS) to be "superdienophiles" [4b]) we have now examined the behavior of *C*-sulfonyldithioformates **2** (R^1 = $ArSO_2$, R^2 = ArS) towards anthracene **1** (R^3 = H) and 9-methylanthracene **1** (R^3 = Me). 4-Chlorophenyl *C*-(phenylsulfonyl)dithioformate **2**



SCHEME 1

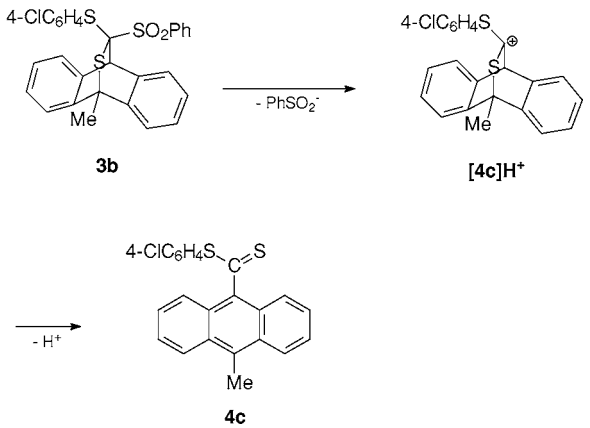
(R^1 = $PhSO_2$, R^2 = 4-ClC_6H_4S) as well as phenyl *C*-(4-methylphenylsulfonyl)dithioformate **2** (R^1 = 4-$MeC_6H_4SO_2$, R^2 = PhS) form the corresponding Diels–Alder adducts **3**, while pentachlorophenyl *C*-(phenylsulfonyl)dithioformate **2** (R^1 = $PhSO_2$, R^2 = C_6Cl_5S) forms the corresponding Friedel–Crafts product **4** without any observable Diels–Alder intermediate **3**. As a working hypothesis one may assume that a particularly bulky R^2 group such as C_6Cl_5S destabilizes **3** relative to **4** so that the cycloadduct **3** corresponding to **4b** remains unobserved.

Our experimental results show that *C*-sulfonyldithioformates **2** readily react with anthracene and with 9-methylanthracene and also support the hypothesis that all such reactions take place as Diels–Alder additions. As a consequence of the presence of a nucleofugal group on C-12 of **3** so formed this may or may not be subject to a facile uncatalyzed elimination which regenerates the unperturbed anthracene system **4**. The only 9-anthracenecarbodithioic acid derivative that was known prior to our investigations is 9,10-anthracenedicarbodithioic acid, prepared by treatment of 9,10-bis(chloromethyl)anthracene with sulfur and sodium methoxide in methanol [5].

The structure of **3b** was determined by a single-crystal X-ray study (cf. Fig. 1). It should be noted that the structure of all earlier observed unsymmetrical adducts **3** was assigned in a reasonable, but not totally compelling way by circumstantial spectroscopic evidence [1b]. Our structural data for **3b** confirm these early assignments and put this chain of reasoning on a firm and unambiguous basis. Considering the near quantitative yield of **3b** and **3c** the corresponding unsymmetrical Diels–Alder reactions are in all likelihood regiospecific rather than just highly regioselective. As mentioned earlier this is most likely determined by steric effects.

EXPERIMENTAL

All syntheses were carried out under an atmosphere of dry nitrogen. All NMR experiments (TMS as internal standard) were carried out with a Varian Unity 400 MHz spectrometer. The mass spectra were obtained in the EI mode (70 eV, direct inlet) with a Finnigan MAT GCQ PLUS system. The infrared spectra (KBr disks) were run on a Perkin-Elmer FT/IR-1760 spectrometer. The melting points were taken



SCHEME 2

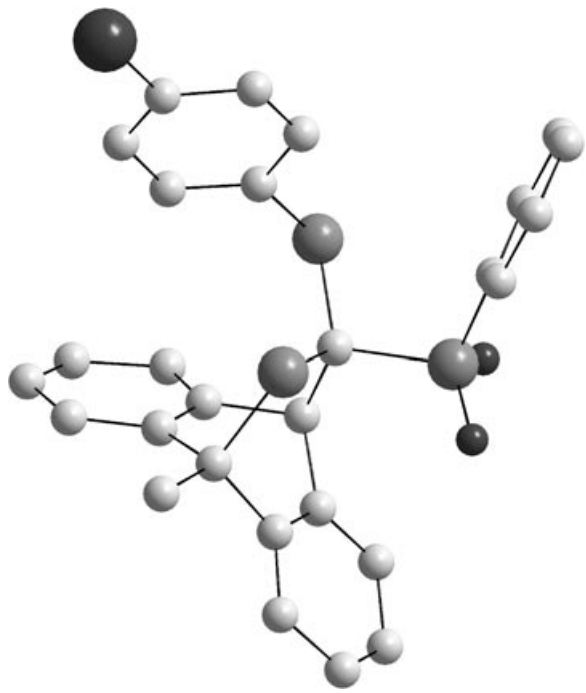


FIGURE 1 The molecular structure of **3b**.

with an SMP3 apparatus. The elemental analyses were carried out at the Microanalytical Department, Department of Chemistry, Copenhagen University, Copenhagen, Denmark.

The starting compounds **2** with (a) $R^1 = PhSO_2$, $R^2 = 4\text{-}ClC_6H_4S$, (b) $R^1 = 4\text{-}MeC_6H_4SO_2$, $R^2 = PhS$, and (c) $R^1 = PhSO_2$, $R^2 = C_6Cl_5S$ were prepared according to a literature procedure by tetrabutylammonium hydrogen sulfate catalyzed reaction of the appropriate chlorodithioformates with the appropriate sodium arenesulfinates [3b].

*(±)-9,10-Dihydro-12-(4-methylphenylsulfonyl)-12-(phenylthio)-10,9-(epithiomethano)anthracene (**3a**) (**3**, $R^1 = 4\text{-}MeC_6H_4SO_2$, $R^2 = PhS$, $R^3 = H$)*

A mixture of **2** ($R^1 = 4\text{-}MeC_6H_4SO_2$, $R^2 = PhS$) (100 mg, 0.32 mmol) and **1** ($R^3 = H$) (70 mg, 0.40 mmol) in $CDCl_3$ (0.8 ml) was left in an NMR tube at room temperature until the bright red color of **2** had disappeared (2 days). The ^1H and ^{13}C NMR spectra of the crude reaction mixture showed that the cycloadduct **3a** had formed in quantitative yield. Under TLC conditions **4a** was the only observable compound (vide infra). ^1H NMR (400 MHz, $CDCl_3$): δ 2.40 (s, 3H), 5.12 (s, 1H), 5.46 (s, 1H), 6.95–7.05 (m, 2H), 7.17–7.30 (m, 10H), 7.45–7.51 (m, 3H), 7.86 (d, 2H, ^3J = 8.4 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.66 (q), 48.41 (d), 53.49 (d), 94.22 (s), 125.22 (d), 125.90 (d), 126.60 (d), 126.73 (d), 127.42 (d), 127.86 (d), 128.01 (d), 128.57 (d), 129.28 (d), 129.87 (s), 130.74 (d), 131.03 (d), 131.79 (d), 133.91 (s), 137.16 (d), 138.48 (s), 140.06 (s), 141.83 (s), 144.24 (s), 145.94 (s) ppm.

*9-Anthracenecarbodithioic Acid Phenyl Ester (**4a**) (**4**, $R^2 = PhS$, $R^3 = H$)*

Upon attempted isolation of the above cycloadduct **3a** by evaporation of the solvent under reduced pressure and washing with ether, *p*-toluenesulfinic acid was eliminated with formation of **4a** as red crystals. Yield 76 mg (72%), m.p. 130–131°C. IR (KBr): ν 3051, 1622, 1339, 1141, 995, 870, 743, 692, 608 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.11–7.57 (m, 11H), 7.90–8.12 (m, 2H), 8.44 (s, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 125.23 (d), 125.94 (d), 126.73 (d), 127.32 (d), 127.43 (d), 128.28 (d), 129.30 (d), 130.20 (d), 130.65 (s), 131.10 (s), 135.44 (s), 136.41 (s), 224.75 (s, C=S) ppm; MS: *m/z* 330 (M, $C_{21}H_{14}S_2$, 18%), 221 ($C_{15}H_9S$, 100%), 176 ($C_{14}H_8$, 13%), 153 ($C_7H_5S_2$, 2%), 109 (C_6H_5S, 2%). Calc. for $C_{21}H_{14}S_2$: C, 76.82; H, 4.12; S, 19.56. Found: C, 76.36; H, 4.24; S, 19.40%.

*9-Anthracenecarbodithioic Acid Pentachlorophenyl Ester (**4b**) (**4**, $R^2 = C_6Cl_5S$, $R^3 = H$)*

A mixture of **2** ($R^1 = PhSO_2$, $R^2 = C_6Cl_5S$) (300 mg, 0.64 mmol) and **1** ($R^3 = H$) (115 mg, 0.64 mmol), dissolved in dry trichloromethane (2 ml), was heated in a sealed tube at 70°C until the purple color of **2** had changed to red (30 h). After removal of the solvent under reduced pressure the red residue was crystallized from trichloromethane/ether (1:3) to give red crystals. Yield 270 mg (84%), m.p. 148–149°C. IR (KBr): ν 3055, 1623, 1474, 1445, 1306, 1144, 996, 743, 687, 590, 547 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.54–7.57 (m, 4H), 8.04 (d, 2H, ^3J = 6.0 Hz), 8.27 (d, 2H, ^3J = 7.6 Hz), 8.55 (s, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 124.76 (d), 125.58 (d), 127.07 (d), 127.63 (s), 128.51 (d), 129.30 (d), 131.01 (s), 131.09 (s), 133.11 (s), 137.13 (s), 138.14 (s), 138.63 (s), 225.50 (s, C=S) ppm; MS: *m/z* 465 ($C_{21}H_9Cl_4S_2$, 1%), 279 (C_6Cl_5S, 1%), 247 (C_6Cl_5, 6%), 221 ($C_{15}H_9S$, 100%), 177 ($C_{14}H_9$, 5%), 176 ($C_{14}H_8$, 10%). Calc. for $C_{21}H_9Cl_5S_2$: C, 50.15; H, 1.79; S, 12.73. Found: C, 50.07; H, 1.54; S, 13.10%.

*(±)-12-(4-Chlorophenylthio)-9,10-dihydro-10-methyl-12-(phenylsulfonyl)-10,9-(epithiomethano)anthracene (**3b**) (**3**, $R^1 = PhSO_2$, $R^2 = 4\text{-}ClC_6H_4S$, $R^3 = Me$)*

To a stirred solution of **2** ($R^1 = 4\text{-}ClC_6H_4S$, $R^2 = PhSO_2$) (0.40 g, 1.22 mmol) in dry trichloromethane (15 ml) was added **1** ($R^3 = Me$) (235 mg, 1.22 mmol). The mixture was stirred at room temperature until the bright red color of **2** had disappeared (2 h). After removal of the solvent under reduced pressure the colorless residue was washed with ether and recrystallized from trichloromethane/ether (1:3) to afford the cycloadduct **3b** as colorless needles. Yield 0.59 g (93%), m.p. 113–114°C. IR (KBr): ν 3065, 2972, 1626, 1572, 1474, 1306, 1145, 1080, 750, 688, 590, 569, 545 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 2.15 (s, 3H), 5.40 (s, 1H), 6.80–6.90 (m, 2H), 7.17–7.28 (m, 8H), 7.40–7.45 (m, 2H), 7.54–7.60 (m, 3H), 7.90 (d, 2H, ^3J = 8.4 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 16.34 (q), 52.34 (s), 53.66 (d), 95.42 (s), 125.84 (d), 126.52 (d), 126.85 (d), 126.99 (d), 127.43 (d), 127.98 (d), 128.07 (d), 128.48 (d), 129.18 (d), 129.80 (d), 130.60 (d), 131.82 (d), 133.23 (s), 135.97 (s), 138.10 (s), 138.34 (d), 138.61 (s), 140.30 (s), 144.26 (s), 145.14 (s); MS: *m/z* 379 ($C_{22}H_{16}ClS_2$, 10%), 235 ($C1_6H_{11}S$, 3%), 192 ($C_{15}H_{12}$, 100%), 187 ($C_7H_4ClS_2$, 7%), 111 (C_6H_4Cl, 2%). Calc. for $C_{28}H_{21}ClO_2S_3$: C, 64.55; H, 4.03; S, 18.44. Found: C, 64.39; H, 3.94; S, 17.99%.

*(±)-9,10-Dihydro-10-methyl-12-(4-methylphenylsulfonyl)-12-(phenylthio)-10,9-(epithiomethano)anthracene (**3c**) (**3**, R^1 = 4-$MeC_6H_4SO_2$, R^2 = PhS, R^3 = Me)*

To a stirred solution of **2** (R^1 = 4-$MeC_6H_4SO_2$, R^2 = PhS) (0.50 g, 1.62 mmol) in dry trichloromethane (15 ml) was added **1** (R^3 = Me) (312 mg, 1.62 mmol). The mixture was stirred at room temperature until the bright red color of **2** had disappeared (4 h). After removal of the solvent under reduced pressure the colorless residue was washed with ether and recrystallized from trichloromethane/ether (1:3) to afford the cycloadduct **3c** as colorless needles. Yield 0.76 g (94%). IR (KBr): ν 3053, 2927, 1622, 1594, 1440, 1328, 1144, 811, 733, 654, 584 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 2.13 (s, 3H), 2.42 (s, 3H), 5.40 (s, 1H), 6.90–6.04 (m, 2H), 7.13–7.27 (m, 10H), 7.53–7.58 (m, 3H), 7.81 (d, 2H, ^3J = 8.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 16.37 (q), 21.63 (q), 52.13 (s), 53.60 (d), 95.58 (s), 125.73 (d), 126.40 (d), 126.75 (d), 126.78 (d), 127.79 (d), 128.57 (d), 129.30 (d), 129.90 (d), 130.16 (d), 130.89 (d), 131.85 (d), 134.03 (s), 135.20 (s), 137.05 (d), 138.93 (s), 140.45 (s), 144.18 (s), 144.38 (s), 145.11 (s) ppm; MS: *m/z* 345 ($C_{22}H_{17}S_2$, 3%), 192 ($C_{15}H_{12}$, 100%), 153 ($C_7H_5S_2$, 4%) 77 (C_6H_5, 2%). Calc. for $C_{29}H_{24}O_2S_3$: C, 69.60; H, 4.80; S, 19.29. Found: C, 68.90; H, 4.89; S, 19.70%.

*10-Methylanthracene-9-carbodithioic Acid 4-Chlorophenyl Ester (**4c**) (**4**, R^2 = 4-ClC_6H_4S, R^3 = Me)*

A solution of **3b** (200 mg, 0.38 mmol) in dry trichloromethane (1 ml) was heated overnight in a sealed tube at 80°C, the resulting mixture evaporated, and the red residue crystallized from ether to afford **4c** as red crystals. Yield 118 mg (82%), m.p. 139–142°C. IR (KBr): ν 3065, 2927, 1620, 1333, 1152, 979, 798, 742, 687 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 3.12 (s, 3H), 7.42–7.56 (m, 8H), 8.15 (d, 2H, ^3J = 8.4 Hz), 8.30 (d, 2H, ^3J = 8.4 Hz) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.47 (q), 124.82 (d), 125.26 (d), 125.49 (d), 126.17 (d), 126.22 (d), 129.46 (s), 129.68 (d), 129.80 (s), 132.76 (s), 135.78 (s), 136.85 (s), 138.39 (s), 226.19 (s, C=S) ppm; MS: *m/z* 378 (M, $C_{22}H_{15}ClS_2$, 2%), 235 ($C_{16}H_{11}S$, 100%), 187 ($C_7H_4ClS_2$, 3%), 143 (C_6H_4ClS, 4%), 108 (C_6H_4S, 7%). Calc. for $C_{22}H_{15}ClS_2$: C, 69.75; H, 3.96; S, 16.90. Found: C, 69.86; H, 3.79; S, 17.23%.

*10-Methylanthracene-9-carbodithioic Acid Pentachlorophenyl Ester (**4d**) (**4**, R^2 = C_6Cl_5S, R^3 = Me)*

A mixture of **2** (R^1 = $PhSO_2$, R^2 = C_6Cl_5S) (200 mg, 0.42 mmol) and **1** (R^3 = Me) (81 mg, 0.42 mmol), dissolved in dry trichloromethane (2 ml), was stirred at room temperature until the purple color of **2** had changed to red (7 days). After removal of the solvent under reduced pressure the red residue was crystallized from trichloromethane/ether (1:3) to give red crystals of **4d**. Yield 180 mg (83%), m.p. 150–151°C. IR (KBr): ν 3085, 2930, 1622, 1338, 1155, 989, 802, 745, 691 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 3.17 (s, 3H), 7.54–7.59 (m, 4H), 8.30–8.36 (m, 4H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.58 (q), 124.85 (d), 125.40 (d), 125.52 (d), 126.38 (d), 128.98 (s), 129.56 (d), 133.01 (s), 133.55 (s), 136.84 (s), 136.99 (s), 138.58 (s), 139.10 (s), 226.24 (s, C=S) ppm; MS: *m/z* 482 ($C_{22}H_{11}Cl_5S$, 0.5%), 379 ($C_{22}H_{16}ClS_2$, 10%), 323 ($C_7Cl_5S_2$, 0.5%), 247 (C_6Cl_5, 4%), 235 ($C_{16}H_{11}S$, 100%), 191 ($C_{15}H_{11}$, 14%). Calc. for $C_{22}H_{11}Cl_5S_2$: C, 51.11; H, 2.13; S, 12.39. Found: C, 50.86; H, 1.79; S, 12.58%.

Structure Determination of **3b**

Diffraction data were collected using a Bruker–Nonius KappaCCD diffractometer. The compound crystallizes monoclinic, space group *C*2/*c*, a = 3747.6(2) pm, b = 682.95(4) pm, c = 1957.7(1) pm, β = 101.939(3)°. R-factors: R_1 = 0.0611 ($I > 2\sigma(I)$), wR2 = 0.126, GooF = 1.054 for 4127 unique reflections and 307 parameters.

Crystallographic data excluding structure factors have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 191484. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax: +44(0)-1223-336033 or e-mail: deposit@ccdc.cam.ac.uk].

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