ordering can be found using simple 6 − exp potentials such as those to be found for Br in Burgos & Bonadeo (1977) and C and H in Williams (1966). Our results indicate an approach in which additional electrostatic effects associated more locally with the Br and methyl groups would be more fruitful. One possibility is to include a dipole–dipole interaction as done, for example, by Reynolds (1975) for the carbonyl dipole in anthrone, and we are proceeding along these lines. Work is in progress on a number of similar compounds involving disorder between Br and methyl substituents.

We are grateful to M. Puza for the preparation of the material used in this work.

References

BURGOS, E. & BONADEO, H. (1977). *Chem. Phys. Lett.* **49**, 475–478.

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

ELCOMBE, M. M., COX, G. W., PRYOR, A. W. & MOORE, F. H. (1971). *Programs for the Management and Processing of Neutron Diffraction Data*. Report AAEC/TM578. Australian Atomic Energy Commission.

FLACK, H. D. (1970). *Philos. Trans. R. Soc. London Ser. A*, **266**, 559–591.

GLAZER, A. M. (1970). *Philos. Trans. R. Soc. London Ser. A*, **266**, 593–639.

HELLER, E. & SCHMIDT, G. M. J. (1971). *Isr. J. Chem.* **9**, 449–462.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

JONES, R. D. G. & WELBERRY, T. R. (1980). *Acta Cryst.* B**36**, 852–857.

JONES, R. D. G. & WELBERRY, T. R. (1981). *Acta Cryst.* B**37**, 1125–1126.

LEHMANN, M. S. & PAWLEY, G. S. (1972). *Acta Chem. Scand.* **26**, 1996–2004.

MASON, R. (1964). *Acta Cryst.* **17**, 547–555.

MOORE, F. H. (1972). *Acta Cryst.* A**28**, S256.

PRAT, M. T. (1961). *Acta Cryst.* **14**, 110–112.

RAMDAS, S. & THOMAS, J. M. (1978). *Chem. Phys. Solids Surfaces*, **7**, 31–58.

REYNOLDS, P. A. (1975). *Acta Cryst.* A**31**, 80–83.

SHELDRICK, G. M. (1976) *SHELX*. A program for crystal structure determination. Univ. of Cambridge, England.

WELBERRY, T. R. & JONES, R. D. G. (1980). *J. Appl. Cryst.* **13**, 244–251.

WELBERRY, T. R., JONES, R. D. G. & EPSTEIN, J. (1982). To be published.

WELBERRY, T. R., MILLER, G. H. & PICKARD, D. K. (1979). *Proc. R. Soc. London Ser. A*, **367**, 175–192.

WILLIAMS, D. E. (1966). *J. Chem. Phys.* **45**, 3770–3778.

WILSON, A. J. C. (1962). *X-ray Optics*. London: Methuen.

Acta Cryst. (1982). B**38**, 1525–1529

2-[3-(*o*-Nitrophenylthio)-1-indenylidene]-1,3-dithiolane, 2-[3-(*o*-Nitrophenylthio)-1-indenylidene]-1,3-dithiole and 2-{3-[1-(1,3-Dithiolan-2-ylidene)-2,3-dihydro-2-indenyl]-1-indenylidene}-1,3-dithiolane

BY JOHN C. BARNES AND JOHN D. PATON

*Chemistry Department, The University, Dundee DD*1 4*HN, Scotland*

AND BRIAN H. NICHOLLS

The Wakeman School, Shrewsbury, Shropshire, England

(*Received* 5 *August* 1981; *accepted* 3 *December* 1981)

Abstract

The crystal and molecular structures of the title compounds have been determined. (I) $C_{18}H_{13}NO_2S_3$ is monoclinic, space group $P2_1/c$ with a = 11·584 (6), b = 10·935 (7), c = 16·173 (8) Å, β = 121·5 (3)°, U = 1746 Å^3, Z = 4, D_c = 1·36 g cm^{-3}, $F(000)$ = 768, μ(Cu $K\alpha$) = 38 cm^{-1}. (II) $C_{18}H_{11}NO_2S_3$ is triclinic, space group $P\bar{1}$, with a = 10·782 (5), b = 8·40 (1), c = 10·501 (6) Å, α = 107·95 (5), β = 69·23 (1), γ = 109·0 (1)°, U = 820·6 Å^3, Z = 2, D_c = 1·40 g cm^{-3}, $F(000)$ = 380, μ(Cu $K\alpha$) = 41 cm^{-1}. (III) $C_{24}H_{20}S_4$ is monoclinic, space group $P2_1/n$, with a = 12·23 (1), b = 7·58 (1), c = 23·43 (2) Å, β = 105·96°, U = 2088·6 Å^3, Z = 4, D_c = 1·39 g cm^{-3}, $F(000)$ = 912, μ(Cu $K\alpha$) = 40 cm^{-1}. Refinement for (I), (II) and (III) converged at R = 0·047, 0·069 and 0·057 for, respectively, 1852, 1486 and 1600 unique reflexions above background.

0567-7408/82/051525-05$01.00

In each case the five-membered dithio ring is twisted slightly out of the plane of the indene. The nitrophenylthio group in (I) and (II) and the second indene group in (III) are twisted by 72 ± 5° from the indene plane.

Introduction

The reaction of 2-(1-indenylidene)-1,3-dithiolane with *o*-nitrophenylsulphenyl chloride gave a monomer (I) and two dimeric products (III) and (IV) (Bigg & Suschitzky, 1982). In the present work single-crystal X-ray analysis has shown the monomer to be 2-[3-(*o*-nitrophenylthio)-1-indenylidene]-1,3-dithiolane (I) and one dimer to be 2-{3-[1-(1,3-dithiolan-2-ylidene)-2,3-dihydro-2-indenyl]-1-indenylidene}-1,3-dithiolane (III). (I) is oxidized by $HgCl_2$ to the corresponding dithiole (II), the crystal structure of which has also been determined.

Experimental

(I) (0·1 g) was reacted with an equimolar quantity of $HgCl_2$ in ethyl acetate at room temperature for 72 h. Slow removal of solvent gave roughly equal yields of unchanged (I) and of (II) as pale-orange triclinic tablets. These products were separated by hand under a binocular microscope.

Compound (I)

After preliminary oscillation and Weissenberg photographs, data were collected on equi-inclination Weissenberg photographs for levels $h(0–7)l$ and $hk(0–10)$ on rectangular prisms 0·2 × 0·2 × 0·5 mm. Intensities were measured by the SRC Microdensitometer Service, Daresbury Laboratory.

All calculations were performed using Sheldrick's (1976) program system *SHELX* 76 and his molecular geometry program *XANADU*. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Intensities were not corrected for absorption. 1852 unique reflexions were observed above background. The direct-methods routine *TANG* revealed the three S atoms and ten C atoms on the best E map. A structure factor calculation on these atoms gave $R = 0·36$. The structure was extended by conventional least-squares and Fourier methods to $R = 0·086$ with anisotropic S atoms and isotropic C, N and O. At this point a Fourier map revealed all the H atoms which were then refined with grouped isotropic thermal parameters. Anisotropic thermal parameters were used for all non-hydrogen atoms in the final stages. Convergence occurred at $R = 0·047$ using unit weights.

Compound (II)

Data were collected as above for layers $0kl$, $h(0–6)l$, and $hk(0–4)$, giving 1492 unique reflexions above background. The structure was solved by the direct methods routine *TANG* with some difficulty, since the compound was initially believed to be a mercury complex of (I). This belief was reinforced by the best E maps giving a very large origin peak. However, calculations based on an Hg atom at the origin gave no developing structure. Examination of the fourth E map revealed a molecule which was indistinguishable from (I) at this stage. All non-hydrogen atoms except the NO_2 group appeared in the E map. This structure developed normally, refining to $R = 0·12$ with anisotropic S atoms and all other non-hydrogen atoms isotropic. At this point it was clear that C(12)—C(13) (1·39 Å) could not be a single bond. Accordingly all H atoms except those on C(12) and C(13) were introduced on calculated positions using a single refinable isotropic thermal parameter for the indene protons and a second such parameter for the phenyl protons. A Fourier map then showed a single hydrogen on each of C(12) and C(13). These two H atoms were given individual isotropic thermal parameters which were refined along with anisotropic parameters for all non-hydrogen atoms to convergence at $R = 0·069$ for unit weights after six reflexions for which $F_o > 2F_c$ were omitted.

Compound (III)

Two sets of data were collected. Photographic data as above for layers $h(0–5)l$ and $(0–6)kl$ gave 1600 unique reflexions above background. In addition the same crystals were used on a Wooster four-circle diffractometer. This instrument had been rebuilt locally using an LSI 11 microprocessor to drive CAMAC electronics. 2406 unique reflexions were collected on the diffractometer as a test of its performance in the fixed-χ mode.

Neither set of data gave a satisfactory solution. E maps were in general agreement on the S-atom positions but the structure would not extend to the C atoms. It was found that the diffractometer data were generally unreliable because of mechanical inaccuracies. The problem with the photographic data was that the most intense reflexions were off-scale on all the films. The situation was rescued by merging the most reliable levels of the diffractometer data ($\chi = 180$, 170°) with the photographic data to give 2260 unique reflexions. The best E map then gave four S atoms and a structure factor calculation phased by these showed all the C atoms. Refinement continued to $R = 0·27$ with all atoms isotropic and $R = 0·10$ with all non-hydrogen atoms anisotropic.

At this point the photographic data and the diffractometer data were examined separately. The photographic data immediately refined to $R = 0{\cdot}079$, revealing all the H atoms. The diffractometer data alone gave $R = 0{\cdot}13$ and were not explored further.

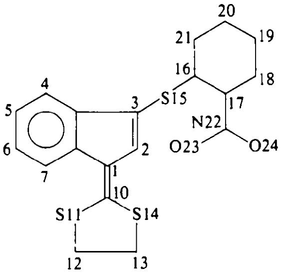
Final convergence for the photographic data was obtained at $R = 0{\cdot}057$ with unit weights; H atom positions were refined with average thermal parameters for each ring system, as for compound (II).*

Compound (IV)

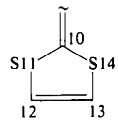
Crystals of the second dimeric material (pale yellow) were not suitable for data collection. Preliminary photographs showed a monoclinic cell $a = 14{\cdot}58$, $b = 6{\cdot}56$, $c = 22{\cdot}90$ Å, $\beta = 104°$. This indicates a molecular volume for $Z = 4$ not significantly different from (III), consistent with the assignation of (IV) as an isomer of (III).

Results and discussion

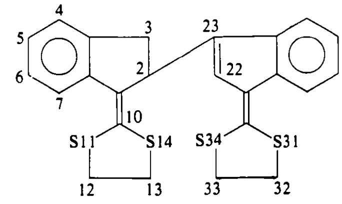
Atomic coordinates for the three structures are given in Tables 1–3. Interatomic distances and angles are summarized in Tables 4 and 5. The structures were found to be:



(I)



(II) [as (I) except where shown]



(III)

Throughout this paper atoms are referred to by the crystallographic numbering scheme shown. All the crystals studied showed centrosymmetric space groups and are therefore racemic mixtures. The double bonds shown were identified by length and by the location of all the conformationally significant H atoms. Tables 4

Table 1. *Atomic coordinates* ($\times 10^4$; *for* H $\times 10^3$) *with e.s.d.'s for compound* (I)

The equivalent isotropic thermal parameter is defined by

$$U_{eq} = \tfrac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	U_{eq} (Å^2)
C(1)	5020 (4)	6161 (4)	−877 (3)	40 (2)
C(2)	6410 (4)	5678 (4)	−340 (3)	43 (2)
C(3)	−3438 (4)	4797 (4)	−871 (3)	42 (2)
C(4)	4875 (4)	3807 (4)	−2567 (3)	48 (2)
C(5)	3514 (5)	3821 (5)	−3353 (3)	59 (3)
C(6)	2589 (5)	4639 (5)	−3354 (3)	58 (3)
C(7)	2969 (4)	5461 (4)	−2585 (3)	50 (3)
C(8)	−5697 (3)	5451 (3)	−1804 (3)	38 (2)
C(9)	5258 (4)	4620 (3)	−1798 (3)	39 (2)
C(10)	4562 (4)	7072 (4)	−542 (3)	41 (2)
S(11)	2882 (1)	7589 (1)	−1144 (1)	53 (1)
C(12)	3162 (6)	8884 (5)	−337 (4)	68 (3)
C(13)	4343 (5)	8581 (5)	670 (4)	64 (3)
S(14)	5662 (1)	7863 (1)	548 (1)	60 (1)
S(15)	8015 (1)	3854 (1)	9575 (1)	48 (1)
C(16)	−1344 (4)	4291 (4)	−1184 (3)	45 (2)
C(17)	−369 (4)	3612 (5)	−1242 (3)	55 (3)
C(18)	121 (5)	3934 (7)	−1843 (4)	77 (3)
C(19)	−341 (6)	5009 (8)	−2383 (4)	88 (4)
C(20)	−1270 (5)	5707 (6)	−2315 (4)	79 (3)
C(21)	−1787 (4)	5375 (5)	−1739 (4)	60 (3)
N(22)	195 (4)	2465 (5)	−665 (3)	65 (3)
O(23)	−47 (4)	2240 (4)	−27 (3)	88 (3)
O(24)	901 (5)	1805 (5)	−839 (3)	116 (4)
H(2)	714 (5)	595 (4)	30 (4)	60 (13)
H(4)	560 (4)	326 (4)	−262 (3)	52 (7)
H(5)	330 (4)	328 (4)	−386 (3)	52 (7)
H(6)	156 (5)	462 (4)	−389 (3)	52 (7)
H(7)	235 (4)	598 (4)	−262 (3)	52 (7)
H(121)	229 (6)	895 (5)	−32 (4)	91 (9)
H(122)	331 (6)	975 (6)	−58 (4)	91 (9)
H(131)	468 (6)	929 (6)	102 (4)	91 (9)
H(132)	413 (6)	797 (6)	110 (4)	91 (9)
H(18)	89 (7)	346 (6)	−176 (5)	99 (10)
H(19)	11 (6)	527 (6)	−270 (5)	99 (10)
H(20)	−162 (6)	637 (6)	−272 (5)	99 (10)
H(21)	−245 (6)	586 (6)	−169 (5)	99 (10)

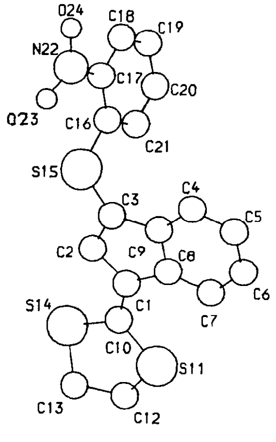


Fig. 1. Molecule (I) viewed perpendicular to the *ac* plane.

---

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36659 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Atomic coordinates* (×10^4; *for* H ×10^3) *with e.s.d.'s for compound* (II)

	x	y	z	U_{eq} (Å^2)
C(1)	5261 (8)	5009 (10)	7699 (9)	29 (5)
C(2)	5173 (9)	3608 (11)	6493 (9)	35 (5)
C(3)	6376 (8)	3169 (11)	5872 (9)	33 (5)
C(4)	8704 (9)	4347 (11)	6445 (9)	33 (5)
C(5)	9395 (9)	5578 (12)	7340 (10)	41 (6)
C(6)	8705 (10)	6687 (13)	8495 (9)	43 (6)
C(7)	7350 (9)	6578 (10)	8746 (8)	30 (4)
C(8)	6654 (8)	5377 (10)	7813 (8)	27 (5)
C(9)	7344 (8)	4248 (10)	6681 (8)	27 (4)
C(10)	4250 (8)	5851 (10)	8479 (8)	28 (4)
S(11)	4378 (2)	7628 (3)	9856 (2)	40 (1)
C(12)	2841 (13)	8106 (19)	96 (18)	115 (12)
C(13)	1901 (11)	6775 (16)	9513 (12)	70 (8)
S(14)	2692 (2)	5270 (3)	8100 (3)	53 (2)
S(15)	6818 (2)	1740 (3)	4243 (2)	35 (1)
C(16)	7340 (8)	298 (10)	4692 (8)	27 (5)
C(17)	8317 (8)	−618 (10)	3746 (8)	28 (5)
C(18)	8778 (9)	−1672 (11)	4128 (10)	41 (5)
C(19)	8218 (11)	−1925 (11)	5465 (9)	37 (6)
C(20)	7240 (10)	−1094 (11)	6423 (10)	41 (6)
C(21)	6803 (9)	23 (11)	6053 (9)	37 (5)
N(22)	1129 (8)	496 (10)	7710 (7)	42 (5)
O(23)	1829 (8)	110 (10)	8193 (7)	58 (5)
O(24)	9944 (7)	−876 (10)	1611 (7)	62 (5)
H(13)	120 (9)	681 (11)	940 (8)	64 (9)
H(12)	261 (10)	881 (12)	1078 (10)	71 (10)

Table 3. *Atomic coordinates* (×10^4; *for* H ×10^3) *with e.s.d.'s in parentheses for compound* (III)

	x	y	z	U_{eq} (Å^2)
C(1)	1492 (6)	1344 (11)	−877 (3)	31 (4)
C(2)	1610 (7)	529 (10)	−253 (3)	35 (4)
C(3)	2139 (10)	−1347 (11)	−296 (4)	57 (6)
C(4)	2979 (9)	−2704 (12)	−1099 (4)	61 (6)
C(5)	3099 (9)	−2521 (14)	−1664 (5)	68 (7)
C(6)	2682 (8)	−1054 (15)	−2005 (4)	57 (6)
C(7)	2112 (7)	242 (11)	−1784 (3)	39 (5)
C(8)	2004 (6)	137 (10)	−1221 (3)	30 (4)
C(9)	2414 (7)	−1360 (11)	−878 (3)	43 (5)
C(10)	932 (6)	2857 (10)	−1044 (3)	30 (4)
S(11)	616 (2)	3749 (3)	−1764 (1)	46 (1)
C(12)	−94 (9)	5730 (12)	−1612 (5)	54 (6)
C(13)	−594 (9)	5318 (16)	−1115 (6)	65 (7)
S(14)	418 (2)	4119 (3)	−549 (1)	48 (1)
C(21)	3489 (6)	3500 (10)	901 (3)	31 (4)
C(22)	3045 (6)	2936 (10)	297 (3)	32 (4)
C(23)	2330 (6)	1538 (9)	264 (3)	28 (4)
C(24)	1698 (7)	−159 (11)	1076 (3)	42 (5)
C(25)	1818 (8)	−252 (13)	1684 (3)	51 (5)
C(26)	2526 (8)	884 (13)	2072 (3)	52 (5)
C(27)	3136 (7)	2141 (12)	1863 (3)	40 (5)
C(28)	3043 (6)	2290 (10)	1272 (3)	30 (4)
C(29)	2311 (6)	1108 (10)	870 (3)	32 (4)
C(30)	4131 (6)	5011 (11)	1093 (3)	39 (4)
S(31)	4467 (2)	5793 (3)	1817 (1)	59 (2)
C(32)	5283 (14)	7648 (18)	1720 (6)	96 (11)
C(33)	5044 (14)	8172 (18)	1083 (6)	102 (10)
S(34)	4667 (2)	6275 (3)	610 (1)	49 (1)
H(21)	164 (8)	95 (13)	−58 (4)	56 (32)
H(31)	270 (8)	−176 (12)	8 (4)	79 (32)
H(32)	137 (8)	−238 (13)	−32 (4)	86 (34)
H(41)	356 (6)	−380 (9)	−76 (3)	41 (12)
H(51)	344 (6)	−335 (10)	−185 (3)	41 (12)
H(61)	277 (6)	−97 (10)	−236 (3)	41 (12)
H(71)	186 (7)	88 (10)	−202 (3)	41 (12)
H(121)	440 (7)	−116 (10)	299 (3)	48 (13)
H(122)	542 (7)	−148 (11)	356 (3)	48 (13)
H(131)	428 (6)	−137 (10)	417 (3)	48 (13)
H(132)	397 (8)	18 (14)	384 (5)	48 (13)
H(221)	318 (5)	336 (7)	0 (2)	1 (14)
H(241)	109 (6)	−91 (9)	76 (3)	40 (12)
H(251)	133 (6)	−100 (10)	179 (3)	40 (12)
H(261)	256 (6)	91 (9)	249 (3)	40 (12)
H(271)	361 (7)	262 (11)	205 (3)	40 (12)
H(321)	579 (10)	811 (16)	86 (5)	101 (19)
H(322)	544 (10)	938 (15)	91 (5)	101 (19)
H(331)	540 (9)	830 (14)	212 (5)	101 (19)
H(332)	510 (12)	862 (20)	140 (7)	101 (19)

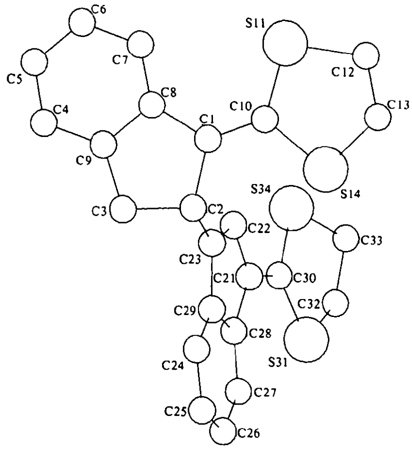


Fig. 2. Molecule (III) viewed perpendicular to the plane C(2), C(4), C(7).

and 5 show that all the bond lengths and angles have typical values. Figs. 1 and 2 are perspective views of compounds (I) and (III).

The details of the structures are explored in Table 6. In the indene fragment C(1), C(2) and C(3) are always out of the plane of the benzene ring. In (I), (II) and (III*b*) where C(2)—C(3) is a double bond, C(1), C(2) and C(3) all lie on one side of the benzene-ring plane giving a dihedral angle of 2–3° between the six- and five-membered rings. However, in (III*a*) C(2)—C(3) is a single bond and the resulting ring strain is relieved by twisting C(3) to the other side of the plane of the benzene ring to give a less planar indene unit. For similar reasons the dithiole ring in (II) is much closer to planar than the dithiolane ring in the other compounds. The dithiole ring has local C_2 symmetry,

The dithiolane ring is not exactly coplanar with the indene unit in any of these structures. The angle between the normals to the planes ranges from 3·8 (7) (I) to 9·3 (6)° (III*b*) (average 7·4°). This twist acts to reduce the contacts S(11)···C(7), S(11)···C(8) and

Table 4. *Interatomic distances* (Å) *with e.s.d.'s*

	(I)	(II)	(III*a*)	(III*b*)*
(*a*) Indene fragment				
C(1)–C(2)	1·471 (5)	1·446 (11)	1·558 (10)	1·435 (9)
C(2)–C(3)	1·361 (7)	1·344 (13)	1·577 (12)	1·363 (10)
C(3)–C(9)	1·483 (4)	1·473 (12)	1·493 (13)	1·464 (10)
C(9)–C(8)	1·428 (6)	1·415 (10)	1·401 (10)	1·424 (9)
C(8)–C(1)	1·496 (5)	1·472 (13)	1·469 (11)	1·468 (11)
C(9)–C(4)	1·400 (6)	1·376 (13)	1·407 (13)	1·384 (12)
C(4)–C(5)	1·417 (5)	1·380 (13)	1·378 (15)	1·394 (11)
C(5)–C(6)	1·395 (8)	1·417 (12)	1·382 (15)	1·373 (12)
C(6)–C(7)	1·406 (7)	1·366 (14)	1·385 (14)	1·380 (13)
C(7)–C(8)	1·394 (5)	1·400 (12)	1·362 (11)	1·361 (10)
C(2)–C(23)			1·496 (9)	
(*b*) Dithiolane fragment				
C(1)–C(10)	1·367 (7)	1·367 (12)	1·338 (11)	1·392 (11)
C(10)–S(14)	1·769 (4)	1·743 (10)	1·749 (8)	1·744 (9)
S(14)–C(13)	1·819 (7)	1·782 (11)	1·794 (11)	1·797 (13)
C(13)–C(12)	1·519 (7)	1·390 (18)	1·488 (19)	1·495 (19)
C(12)–S(11)	1·836 (6)	1·746 (17)	1·818 (10)	1·775 (15)
S(11)–C(10)	1·754 (4)	1·737 (7)	1·757 (7)	1·737 (7)
(*c*) Nitrophenylthio fragment				
C(3)–S(15)	1·775 (4)	1·767 (8)		
S(15)–C(16)	1·800 (6)	1·729 (11)		
C(16)–C(17)	1·395 (8)	1·416 (11)		
C(17)–C(18)	1·402 (10)	1·356 (17)		
C(18)–C(19)	1·394 (10)	1·371 (14)		
C(19)–C(20)	1·368 (11)	1·380 (14)		
C(20)–C(21)	1·393 (11)	1·377 (18)		
C(21)–C(16)	1·412 (7)	1·400 (12)		
C(17)–N(22)	1·495 (7)	1·454 (12)		
N(22)–O(23)	1·226 (9)	1·209 (15)		
N(22)–O(24)	1·229 (8)	1·211 (15)		

* Atoms numbered C(20 + *n*) *etc.*

Table 5. *Interatomic angles* (°) *with e.s.d.'s*

	(I)	(II)	(III*a*)	(III*b*)*
(*a*) Indene fragment				
C(1)–C(2)–C(3)	110·1 (4)	110·5 (8)	103·4 (6)	110·5 (7)
C(2)–C(3)–C(9)	109·3 (3)	108·7 (7)	105·3 (6)	107·4 (6)
C(3)–C(9)–C(8)	107·5 (3)	107·5 (8)	112·6 (7)	109·1 (6)
C(9)–C(8)–C(1)	107·5 (3)	107·3 (7)	109·2 (7)	105·6 (6)
C(8)–C(1)–C(2)	105·6 (4)	106·0 (7)	108·9 (6)	107·3 (6)
C(2)–C(1)–C(10)	124·0 (3)	124·7 (9)	121·5 (7)	126·2 (7)
C(8)–C(1)–C(10)	130·4 (3)	129·2 (7)	129·4 (7)	126·2 (6)
C(2)–C(3)–S(15)	124·3 (2)	126·4 (7)		
C(9)–C(3)–S(15)	125·7 (3)	124·5 (6)		
C(1)–C(2)–C(23)			116·1 (6)	
C(3)–C(2)–C(23)			110·6 (6)	
C(29)–C(23)–C(2)			120·6 (6)	
C(22)–C(23)–C(2)			132·0 (6)	
(*b*) Dithiolane fragment				
C(1)–C(10)–S(11)	124·2 (3)	125·4 (7)	124·8 (6)	123·9 (6)
C(1)–C(10)–S(14)	121·6 (3)	121·6 (6)	121·5 (6)	121·5 (6)
S(11)–C(10)–S(14)	114·1 (3)	112·9 (5)	113·7 (4)	114·6 (4)
C(10)–S(14)–C(13)	96·4 (2)	97·3 (5)	94·9 (5)	96·8 (5)
S(14)–C(13)–C(12)	108·3 (5)	112·5 (9)	109·7 (7)	110·5 (10)
C(13)–C(12)–S(11)	108·7 (4)	115·3 (11)	107·5 (7)	112·1 (10)
C(12)–S(11)–C(10)	96·9 (2)	97·4 (6)	96·9 (4)	97·8 (6)
(*c*) Nitrophenylthio fragment				
C(3)–S(15)–C(16)	102·1 (2)	101·3 (5)		
S(15)–C(16)–C(21)	120·9 (4)	119·6 (7)		
S(15)–C(16)–C(17)	122·8 (4)	123·0 (7)		
C(17)–C(16)–C(21)	116·3 (5)	117·4 (10)		
C(16)–C(17)–C(18)	123·2 (5)	122·6 (8)		
C(16)–C(17)–N(22)	120·6 (5)	121·3 (8)		
C(18)–C(17)–N(22)	116·1 (5)	116·1 (8)		
C(17)–N(22)–O(23)	118·5 (5)	116·8 (8)		
C(17)–N(22)–O(24)	118·4 (6)	119·4 (8)		
O(23)–N(22)–O(24)	123·1 (5)	123·7 (8)		

* Atom C(20 + *n*) *etc.*

Table 6. *Mean-plane calculations*

	(I)	(II)	(III*a*)	(III*b*)
(*a*) Deviations of neighbouring atoms from the mean plane of the benzene ring of the indene fragment ($\times 10^3$, Å)				
C(1)	−43 (3)	114 (7)	54 (5)	−86 (4)
C(2)	−94 (5)	108 (6)	77 (4)	−64 (5)
C(3)	−46 (3)	65 (7)	−87 (5)	−34 (4)
C(10)	−5 (3)	275 (7)	−9 (5)	−279 (6)
S(11)	−72 (2)	398 (4)	−227 (3)	−276 (5)
S(14)	75 (2)	462 (4)	95 (3)	−538 (3)
S(15)	−290 (2)	270 (3)		
C(2)*				−73 (5)
C(23)*			1305 (5)	
(*b*) R.m.s. deviations of ring atoms from the mean plane of the ring ($\times 10^3$, Å)				
Benzene ring of indene	9 (2)	11 (4)	11 (3)	2 (3)
Indene	19 (3)	26 (6)	37 (3)	18 (4)
(*c*) Deviations in dithiolane ring from mean plane of C(10), S(11), S(14) ($\times 10^3$, Å)				
C(12)	194 (5)	163 (4)	68 (4)	26 (5)
C(13)	−437 (4)	−162 (6)	−572 (5)	−424 (6)
(*d*) Angle between normals to mean planes (°)				
Indene and [C(10), S(11), S(14)]	3·79 (5)	8·41 (7)	7·86 (5)	9·26 (6)
Indene and phenyl group (or other indene)	76·97 (10)	70·63 (15)	68·61 (12)	
Phenyl ring and nitro group	10·94 (10)	24·63 (17)		

* Atom in adjacent indene fragment.

S(14)···C(2). The average distances for these contacts are 3·29, 3·28 and 3·11 Å respectively compared with 3·43 Å for the less constrained contact S(15)···C(4) and 3·50 Å for the sum of the van der Waals radii.

In (I) and (II) the angle between the normals to the indene unit and the thiophenyl group is 77·0 (8) and 70·6 (7)° respectively. The two indene units in (III) lie at the similar angle of 68·6 (9)°. This angle gives efficient packing with no short interactions between the nitro group and the dithiolane (dithiole) in (I) and (II) or between the two dithiolane groups in (III).

The nitro group is 10·9 (7)° out of the plane of the phenyl ring in (I), 24·6 (5)° in (II). In each case this gives a very short S(15)···O(23) contact, 2·66 (1), 2·73 (1) Å respectively compared with a van der Waals distance of 3·32 Å. This contact could easily have been avoided by twisting the nitro group further so it appears that there is a weak S···O interaction.

The chemistry of these compounds will be described in a forthcoming paper (Bigg & Suschitzky, 1982).

We thank Professor J. Iball for assistance and encouragement with the diffractometer, Professor H. Suschitzky for introducing us to the problem and supplying the samples of (I) and (III) and the Science Research Council for densitometer facilities.

References

Bigg, M. G. & Suschitzky, H. (1982). To be published.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Sheldrick, G. M. (1976). *SHELX* 76. Program for crystal structure determination, Univ. of Cambridge, England.