The dihedral angles formed by the imidazole ring and the two bound phenyl rings are 73·7 (2)° for the N(2)-attached ring and 61·4 (2)° for the N(1)-attached ring, both values being similar to those encountered in the analogous compounds studied previously, ranging from 60 to 80°. The torsional angles describing the bonding are C(1)—N(1)—C(8)—C(9) = 63·5 (6) and C(1)—N(2)—C(16)—C(21) = 75·2 (7)°, respectively.

Phenyl rings. The N(2)-attached phenyl ring is planar, with atomic deviations from the mean plane within the standard deviations ($\chi^2 = 4{\cdot}52$). The average C—C bond length is 1·385 (8) Å and the mean C—C—C angle is 120·0 (5)°. The N(1)-attached phenyl ring, on the contrary, deviates significantly from the expected planar conformation ($\chi^2 = 29{\cdot}07$), with an average C—C bond length of 1·390 (7) Å and C—C—C angle of 120·0 (5)°.

The ethoxy group exhibits asymmetry in the C(10)—C(11)—O(1) = 115·1 (5) and C(12)—C(11)—O(1) = 124·4 (5)° angles, a common feature of the phenoxy group (Domiano, Nardelli, Balsamo, Macchia & Macchia, 1979).

Fig. 2 shows a view of the unit cell along the *b* axis. No evidence for hydrogen bonds exists and no intermolecular contacts appreciably shorter than the sum of the van der Waals radii have been detected.

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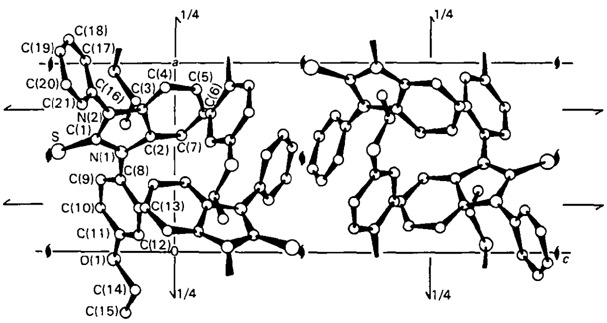

Fig. 2. Unit cell viewed along the *b* axis.

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2-(1,3-Dithiol-2-ylidene)-4-cyclopentene-1,3-dione, $C_8H_4O_2S_2$, and 2-(1,3-Benzodithiol-2-ylidene)cyclohexanone, $C_{13}H_{12}OS_2$

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Abstract. $C_8H_4O_2S_2$ (2), $M_r = 196{\cdot}25$, orthorhombic, $Pnma$, $a = 18{\cdot}080$ (2), $b = 11{\cdot}102$ (1), $c = 3{\cdot}8904$ (4) Å, $V = 780{\cdot}9$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}67$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 5{\cdot}549$ mm^{-1}, $F(000) = 400$, room temperature, $R = 0{\cdot}046$ based on 597 unique reflections. $C_{13}H_{12}OS_2$ (3), $M_r = 248{\cdot}24$, monoclinic, $P2_1/c$, $a = 6{\cdot}459$ (1), $b = 14{\cdot}263$ (3), $c = 13{\cdot}514$ (4) Å, $\beta = 112{\cdot}16$ (2)°, $V = 1153{\cdot}0$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}43$ Mg m^{-3}, Cu $K\alpha$, $\mu = 3{\cdot}90$ mm^{-1}, $F(000) = 520$, room temperature, $R = 0{\cdot}074$ based on 1429 unique reflections. Compound (2) is planar except for a slight 2° fold along the S···S line.

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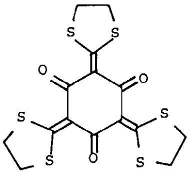
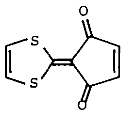
The molecules are packed in parallel sheets. Compound (3) is planar except for four atoms associated with the cyclohexanone system. The molecules pack in columns and are perpendicular to those in adjacent columns. There are short S···O intramolecular contacts of 2·944 (3) and 3·064 (8) Å in compounds (2) and (3), respectively.

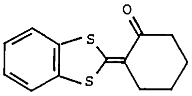
Introduction. Tetrathiafulvalene and its derivatives are of considerable interest because of the conducting charge-transfer complexes they form with tetracyano-*p*-quinodimethane (Garito & Heeger, 1974; Torrance, 1979). Our laboratories are interested in the synthesis of molecules with a variety of geometries and packing requirements which are capable of participating in the formation of strong charge-transfer complexes. Recently we reported the structure and properties of 2,4,6-tris(1,3-dithiolan-2-ylidene)-1,3,5-cyclohexanetrione (1) (Kimura, Watson & Nakayama, 1980), and now report the structures of compounds (2) and (3).



(1)



(2)



(3)

Experimental. All data collected on Syntex $P2_1$, θ:2θ scanning technique. Lattice parameters refined by least squares utilizing 15 medium-angle reflections (angles measured by a centering routine associated with the diffractometer system). Standard reflections: no change in intensity greater than $3\sigma(I)$. Direct methods program *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed the positions of most heavy atoms; alternate anisotropic (H isotropic) least-squares refinements and difference Fourier syntheses located all remaining atoms; function minimized $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\sigma^2(F_o)$.

Compound (2): crystal 0·52 × 0·25 × 0·20 mm, systematic absences ($k + l = 2n + 1$ for $0kl$ and $h = 2n + 1$ for $hk0$) consistent with space group *Pnma*; 770 independent reflections ($0 \leq h \leq 21$, $0 \leq k \leq 13$, $0 \leq l \leq 4$), $2\theta \leq 120°$, 597 observed with $I > 3\sigma(I)$; Lorentz, polarization and empirical absorption corrections; $R = 0{\cdot}046$, $R_w = 0{\cdot}052$, $S = 3{\cdot}7$; largest peak in final difference Fourier map 0·50 e Å^{-3} (associated with the S atoms); average and maximum Δ/σ 0·17 and 0·56, respectively.

Compound (3): crystal 0·5 × 0·5 × 0·3 mm, systematic absences ($l = 2n + 1$ for $h0l$ and $k = 2n + 1$ for $0k0$) consistent with space group $P2_1/c$; 1568 independent reflections ($0 \leq h \leq 7$, $0 \leq k \leq 15$, $-14 \leq l \leq 13$), $2\theta \leq 120°$, 1429 observed with $I > 3\sigma(I)$; Lorentz and polarization corrections; $R = 0{\cdot}074$, $R_w = 0{\cdot}078$, $S = 2{\cdot}8$; largest peak in final difference Fourier map 0·6 e Å^{-3} (associated with the S atoms); average and maximum Δ/σ 0·25 and 0·91, respectively.

Atomic scattering factors calculated by XRAY76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and corrected for anomalous dispersion. No correction for secondary extinction. Locally written programs used for data reduction and initial block-diagonal least-squares refinement. *MULTAN*78 used for direct-methods calculations and XRAY76 for all other computations.

Discussion. Tables 1 and 2 list atomic positional parameters and U_{eq} values for compounds (2) and (3).* Tables 3 and 4 list interatomic distances and valence angles.

Figs. 1 and 2 are *ORTEP* drawings (Johnson, 1971) of compounds (2) and (3). A perpendicular mirror plane containing atoms C(1) and C(3) bisects molecule (2). The molecule is essentially planar although there is a slight 2° fold along the S···S line. Compound (3) is planar except for atoms C(12) – C(14) and O(15). A least-squares plane fitted to the remaining atoms shows a maximum deviation of 0·017 (8) Å by S(3). O(15) lies 0·05 Å out of the plane. The planarity of the molecules leads to S···O intramolecular distances of 2·944 (3) and 3·064 (8) Å in compounds (2) and (3). An average value of 2·58 (2) Å was observed for the same six distances in compound (1) (Kimura, Watson & Nakayama, 1980). In compound (2) the endocyclic double bonds of 1·332 (4) and 1·341 (4) Å are shorter than the exocyclic C(1)—C(3) distance of 1·369 (4) Å. An average value of 1·398 (9) Å was found for the three exocyclic double bonds in compound (1). A shorter exocyclic bond length would lead to shorter S···O interactions or departures from planarity. In (3) the slight departure of O(15) from coplanarity leads to a longer S···O distance and a decrease in the exocyclic bond length to 1·321 (11) Å. In compound (2) the C(3)—C(4) bond of 1·469 (3) Å is shorter than the C(4)—C(5) bond of 1·495 (4) Å which indicates a greater conjugation between the carbonyl and the exocyclic double bond than between the carbonyl and the endocyclic double bond. This is consistent with the increased length of C(1)—C(3). The S—C(1) distance in (2) of 1·724 (2) Å is shorter than the S(1)—C(2) and S(3)—C(2) distances of 1·755 (7) and 1·762 (8) Å found in (3). The average value of the six equivalent distances in (1) is 1·73 (3) Å.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38893 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The planarity of compounds (2) and (3) makes them structurally suitable for the formation of charge-transfer complexes. Compound (2) packs in parallel sheets perpendicular to the *z* axis while the molecules of compound (3) are packed in columns. The molecules in adjacent columns are perpendicular.

We thank the TCU Research Foundation for their financial support. This is FASTBIOS publication No. 101.

Table 1. *Atomic positional parameters* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2) *for compound* (2)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* (\mathbf{a}_i.\mathbf{a}_j) \times 10^3.$$

	x	*y*	*z*	$U_{\rm eq}$
S	2775·7 (3)	1196·1 (4)	6723 (1)	41
O	4169 (1)	391 (1)	3534 (5)	58
C(1)	3245 (2)	2500	5700 (7)	32
C(2)	1994 (1)	1900 (3)	8263 (6)	45
C(3)	3928 (2)	2500	4180 (8)	34
C(4)	4356 (1)	1432 (2)	3177 (6)	41
C(5)	5055 (2)	1896 (3)	1607 (6)	49

Table 2. *Atomic positional parameters* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2) *for compound* (3)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* (\mathbf{a}_i.\mathbf{a}_j) \times 10^3.$$

	x	*y*	*z*	$U_{\rm eq}$
S(1)	2678 (3)	902 (1)	1520 (2)	50 (2)
C(2)	220 (12)	1452 (5)	672 (5)	41 (5)
S(3)	−2232 (3)	864 (1)	599 (2)	47 (1)
C(4)	−891 (13)	−56 (5)	1458 (6)	42 (5)
C(5)	1418 (14)	−42 (6)	1894 (6)	46 (6)
C(6)	2629 (15)	−738 (6)	2586 (7)	57 (7)
C(7)	1447 (18	−1455 (7)	2833 (7)	67 (7)
C(8)	−857 (17)	−1478 (6)	2396 (7)	61 (7)
C(9)	−2047 (15)	−781 (6)	1701 (7)	53 (6)
C(10)	285 (13)	2241 (5)	172 (6)	44 (5)
C(11)	2527 (15)	2687 (6)	330 (8)	58 (6)
C(12)	2334 (20)	3700 (9)	45 (13)	98 (11)
C(13)	520 (21)	3929 (9)	−960 (13)	112 (12)
C(14)	−1746 (17)	3585 (7)	−1069 (8)	70 (8)
C(15)	−1792 (15)	2670 (6)	−534 (7)	52 (6)
O(15)	−3586 (9)	2293 (4)	−696 (5)	69 (5)

Table 3. *Interatomic distances* (Å) *and valence angles* (°) *for compound* (2)

S—C(1)	1·724 (2)	C(3)—C(4)	1·469 (3)
S—C(2)	1·723 (2)	C(4)—O	1·213 (3)
C(2)—C(2′)	1·332 (4)	C(4)—C(5)	1·495 (4)
C(1)—C(3)	1·369 (4)	C(5)—C(5′)	1·341 (4)
C(1)—S—C(2)	95·9 (1)	C(4)—C(3)—C(4′)	107·6 (2)
S—C(1)—C(3)	122·9 (1)	O—C(4)—C(3)	126·3 (2)
S—C(1)—S′	114·2 (2)	O—C(4)—C(5)	127·7 (2)
S—C(2)—C(2′)	117·0 (2)	C(3)—C(4)—C(5)	106·1 (2)
C(1)—C(3)—C(4)	126·2 (1)	C(4)—C(5)—C(5′)	110·1 (2)

Table 4. *Interatomic distances* (Å) *and valence angles* (°) *for compound* (3)

S(1)—C(2)	1·755 (7)	C(7)—C(8)	1·379 (14)
S(1)—C(5)	1·744 (9)	C(8)—C(9)	1·385 (12)
C(2)—S(3)	1·762 (8)	C(10)—C(11)	1·521 (13)
C(2)—C(10)	1·321 (11)	C(10)—C(15)	1·456 (11)
S(3)—C(4)	1·750 (8)	C(11)—C(12)	1·489 (16)
C(4)—C(5)	1·381 (11)	C(12)—C(13)	1·457 (18)
C(4)—C(9)	1·386 (13)	C(13)—C(14)	1·497 (18)
C(5)—C(6)	1·386 (11)	C(14)—C(15)	1·498 (14)
C(6)—C(7)	1·389 (15)	C(15)—O(15)	1·220 (11)
C(2)—S(1)—C(5)	97·4 (4)	C(7)—C(8)—C(9)	120·4 (10)
S(1)—C(2)—S(3)	113·3 (4)	C(4)—C(9)—C(8)	119·0 (8)
S(1)—C(2)—C(10)	121·3 (6)	C(2)—C(10)—C(11)	119·8 (7)
S(3)—C(2)—C(10)	125·3 (6)	C(2)—C(10)—C(15)	119·6 (8)
C(2)—S(3)—C(4)	96·3 (4)	C(11)—C(10)—C(15)	120·6 (7)
S(3)—C(4)—C(5)	117·2 (7)	C(10)—C(11)—C(12)	112·8 (8)
S(3)—C(4)—C(9)	122·7 (6)	C(11)—C(12)—C(13)	114·7 (10)
C(5)—C(4)—C(9)	120·7 (7)	C(12)—C(13)—C(14)	115·5 (14)
S(1)—C(5)—C(4)	115·7 (6)	C(13)—C(14)—C(15)	115·8 (9)
S(1)—C(5)—C(6)	122·8 (7)	C(10)—C(15)—C(14)	120·1 (8)
C(4)—C(5)—C(6)	121·5 (8)	C(10)—C(15)—O(15)	120·7 (8)
C(5)—C(6)—C(7)	117·9 (8)	C(14)—C(15)—O(15)	119·2 (8)
C(6)—C(7)—C(8)	121·1 (9)		

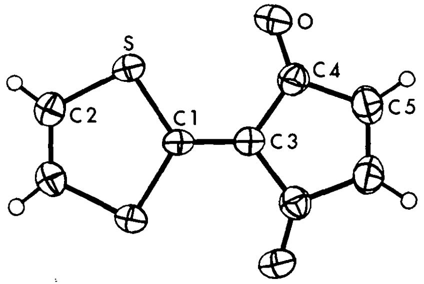

Fig. 1. *ORTEP* drawing of compound (2).

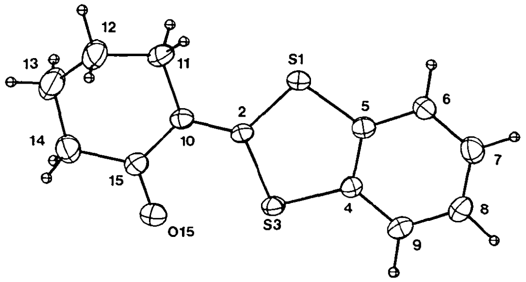

Fig. 2. *ORTEP* drawing of compound (3).

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Structure of 9-Annulene Derivatives. 3. 7*H*-Benzocyclononen-7-ol, $C_{13}H_{12}O$

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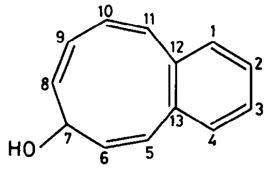
Abstract. $M_r = 184.2$, $F(000) = 392$, triclinic, $P\bar{1}$, $a = 5.688$ (2), $b = 12.878$ (4), $c = 14.138$ (6) Å, $\alpha = 80.85$ (4), $\beta = 85.52$ (3), $\gamma = 89.79$ (3)°, $U = 1019.3$ Å^3, $Z = 4$, $D_x = 1.20$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.080$ mm^{-1}, $T = 295$ K, final $R = 0.038$, 2139 observed reflections. The asymmetric unit contains two independent molecules. The nine-membered ring is in the tub conformation. The crystal structure is built up of $C_{13}H_{12}O$ units linked into tetramers by O···O′ hydrogen bonds.

Introduction. In this paper we investigate the structural properties of a 9-annulenol (I).



(I)

Experimental. Suitable crystals of the title compound grown at room temperature by slow evaporation from an ether solution. Crystal 0.38 × 0.55 × 0.41 mm. Enraf–Nonius CAD-4 diffractometer, take-off angle 5°, graphite-crystal-monochromatized Mo $K\alpha$ radiation. Cell dimensions: least-squares refinement of the setting angles of 25 accurately centred reflections ($\theta \leq 16°$). 3585 independent reflections ($R_{\rm int} = 0.015$), ω–2θ scans ($\theta \leq 25°$), background scans on each side of the peaks. Three standard reflections: no significant intensity variation. Automatic multisolution routine of *SHELX*76 (Sheldrick, 1976) used to generate a series of E maps, one of which contained recognizable fragments of two crystallographically independent molecules; successive difference syntheses phased on these fragments enabled all non-hydrogen atoms to be located. 2139 reflections considered observed $[I \geq 2\sigma(I)]$ and used in subsequent steps. Application of full-matrix least-squares refinement with isotropic temperature factors gave $R = 0.125$; incorporation of anisotropic temperature factors reduced R to 0.088. All H-atoms located at this stage from a difference map. Further refinement of non-H atoms anisotropically and H atoms isotropically reduced R to 0.040. No absorption correction. Refinement of an empirical extinction coefficient caused a significant reduction in R to 0.038. Weighting scheme based on counting statistics; $w = 1.10/(\sigma^2|F_o| + 7.46 \times 10^{-4} F_o^2)$; $\sum w\Delta F^2$ minimized; $R_w = 0.040$. $S = 1.18$. A final difference map showed no peaks or depressions larger than 0.13 e Å^{-3}. $(\Delta/\sigma)_{\rm max} = 0.0$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Fig. 1 shows the least-squares plane of the molecule in isometric projection, with 20% probability vibration ellipsoids for the non-H atoms and spheres of arbitrary radii 0.10 Å for H atoms. Fig. 2 shows the packing arrangement in the crystal. Both figures were produced using *ORTEP* (Johnson, 1976). Table 1 lists the fractional atomic coordinates and Table 2 lists bond distances and angles involving non-hydrogen atoms together with the endocyclic torsion angles for the nine-membered ring.†

The asymmetric unit in the present study contains two independent molecules. These molecules show some differences in their structural parameters, presumably due to their different intermolecular environments, with the largest deviation occurring in the absolute magnitude of the torsion angle C(8)—C(9)—C(10)—C(11) which is 30.6 (5)° in molecule A and 40.5 (5)° in molecule B.

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† Lists of coordinates and bond lengths and angles involving H atoms, and structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38901 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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