Stavaux, Lozac'h & Hordvik, 1985) and bis(dithiolo-tetrahydrodibenzodiazocine) (McKinnon, Secco & Duncan, 1987) with C—N distances of 1·29 and 1·42 Å, respectively. The corresponding negative charge is expected to be located at the S(3) atom as the length of the C(2)—S(3) bond [1·72 (1) Å] indicates a single bond. The geometry of the dithiole ring is in agreement with that commonly observed, the C—S distances range from 1·70 (1)–1·77 (1) Å, the C(1)—C(2) double bond is 1·38 (2) Å, and the ring is planar within 0·02 Å. The aliphatic hexadecane substituent with bond angles ranging from 112·9 (9)–116·0 (11)° indicates no strain, in contrast to the dodecanoic acid substituent of 12-(tetrathiafulvenyl)-11-dodecanoic acid (Rindorf, Thorup, Lerstrup & Bechgaard, 1988). The C atoms of the alkyl chain and the ring are almost coplanar with a dihedral angle between the two least-squares planes of 10°. No close contacts or hydrogen bonds are observed, and the packing of the molecules (cf. Fig. 2) is then believed to be dominated by electrostatic interactions between the positive N atom and the negative S atom on the heterocyclic fragment of neighbouring molecules. This work is part of a research project on new materials with potential use for Langmuir–Blodgett films exhibiting high conductivity or non-linear optical properties (Lerstrup, Jørgensen & Christensen, 1990).

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Structure of an 8a-Trifluoromethyl-8aH-cycloheptatrieno[b]pyrrole

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Abstract. 6-Methoxy-3-oxo-2-phenyl-8a-trifluoromethyl-8aH-cyclohepta-3a,5,7-trieno[b]pyrrole (3), $C_{17}H_{12}F_3NO_2$, $M_r = 319\cdot29$, triclinic, $P\bar{1}$, $a = 7\cdot985$ (1), $b = 9\cdot934$ (2), $c = 10\cdot550$ (2) Å, $\alpha = 65\cdot61$ (1), $\beta = 82\cdot66$ (1), $\gamma = 72\cdot46$ (1)°, $V = 726\cdot7$ Å^3, $Z = 2$, $D_x = 1\cdot459$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 1\cdot2$ cm^{-1}, $F(000) = 328$, $T = 238$ (2) K, $R = 0\cdot057$ for 2283 unique reflexions and 211 refined parameters. The seven-membered ring of the cycloheptatrieno[b]pyrrole system is noticeably folded, mostly as a result of the saturated C atom, 8a, which also bears the trifluoromethyl group. This ring fold is realized by twists around the C—C single bonds, whereas the double bonds are only twisted by, at most, $-5\cdot7$ (3)°. The pyrrole ring is essentially planar and forms a dihedral angle of 25·0 (3)° with the phenyl substituent. The C—C and C—N bond lengths of the entire fused ring system indicate largely localized double bonds. The trifluoromethyl group shows no sign of appreciable disorder.

Experimental. A suitable single crystal of (3), 0·25 × 0·30 × 0·45 mm, was grown from trichloromethane/hexane. Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Cell parameters by least-squares procedures on 15 selected reflexions

$(7\cdot5 \le \theta \le 18°)$ automatically centered on the diffractometer. Reduced-cell calculations (Zimmermann & Burzlaff, 1985) did not indicate symmetry higher than triclinic. Integrated intensities of 2828 reflexions were recorded up to $(\sin\theta/\lambda)_{max} = 0\cdot572$ Å^{-1} (*hkl* range 0– + 9, ± 11, ± 12; ω scans, $\Delta\omega = 0\cdot9°$, scan speed $0\cdot7$–$29\cdot3°$ min^{-1}). Only random fluctuations in the net intensities of three reference reflexions (010, 003, 200), measured every 50 reflexions. No correction for decay was therefore applied and no correction was made for absorption. 2283 unique reflexions ($R_{int} = 0\cdot018$), 1877 of which satisfied the criterion $F_o \ge 4\cdot0\sigma(F_o)$. Structure solved by direct methods (Sheldrick, 1985) and completed by difference Fourier techniques. After anisotropic refinement of the non-H atoms all 12 H atoms could be located on difference maps. They were kept constant in final refinement cycles with the exception of the methyl H atoms which were treated together with their respective C atoms as rigid groups. All non-H atoms were refined with anisotropic displacement parameters by full-matrix least-squares techniques based on F (211 refined parameters on all unique reflexions). At convergence (max. $\Delta/\sigma = 0\cdot001$) $R = 0\cdot057$, $wR = 0\cdot047$, $w = 1/\sigma^2(F_o)$, function minimized: $\Sigma w(|F_o| - |F_c|)^2$, no extinction correction (Sheldrick, 1976). The final difference map was qualitatively featureless with $\Delta\rho_{fin}(max./min.) = 0\cdot23/-0\cdot27$ e Å^{-3}. Scattering factors for neutral, isolated atoms (except for H) from Cromer & Waber (1965), those for H atoms based on a bonded spherical-atom model as given by Stewart, Davidson & Simpson (1965), corrections for f' and f'' applied for all except the H atoms (*International Tables for X-ray Crystallography*, 1974, Vol. IV). Table 1* contains the atomic parameters of the non-H atoms, Table 2 bond distances, angles and dihedral angles. The molecular structure is shown in Fig. 1 together with the adopted atom numbering.

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53883 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
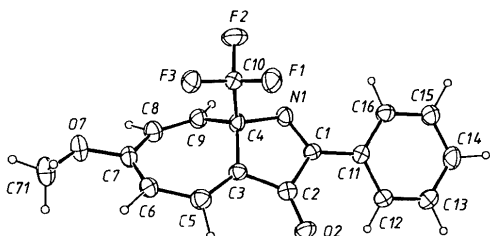

Fig. 1. Molecular structure (*ORTEP*; Johnson, 1976) of (3) and crystallographic numbering scheme adopted. Displacement ellipsoids are drawn at the 50% probability level, H atoms with arbitrary radii.

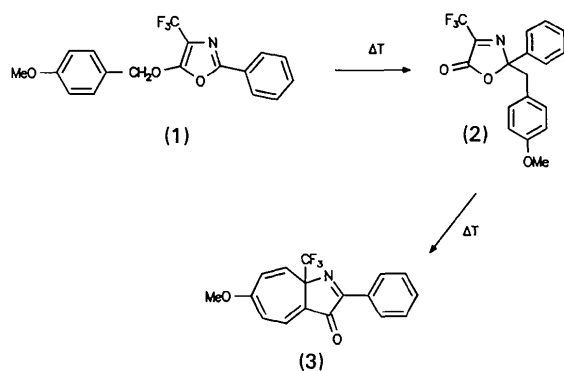Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters for the non-H atoms*

$$U_{eq} = (1/3)\Sigma_i \Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
F1	$-0\cdot2163$ (2)	$-0\cdot2253$ (2)	$-0\cdot2795$ (1)	$0\cdot041$
F2	$-0\cdot4191$ (2)	$-0\cdot0131$ (2)	$-0\cdot3171$ (2)	$0\cdot048$
F3	$-0\cdot1548$ (2)	$-0\cdot0097$ (2)	$-0\cdot3867$ (1)	$0\cdot038$
O2	$0\cdot1029$ (2)	$-0\cdot4246$ (2)	$0\cdot0767$ (2)	$0\cdot033$
O7	$0\cdot0261$ (2)	$0\cdot2964$ (2)	$-0\cdot3565$ (2)	$0\cdot037$
N1	$-0\cdot3177$ (3)	$-0\cdot1843$ (3)	$-0\cdot0380$ (2)	$0\cdot026$
C1	$-0\cdot2162$ (3)	$-0\cdot3112$ (3)	$0\cdot0441$ (2)	$0\cdot022$
C2	$-0\cdot0213$ (3)	$-0\cdot3198$ (3)	$0\cdot0159$ (2)	$0\cdot024$
C3	$-0\cdot0220$ (3)	$-0\cdot1724$ (3)	$-0\cdot0997$ (2)	$0\cdot023$
C4	$-0\cdot2086$ (3)	$-0\cdot0887$ (3)	$-0\cdot1415$ (2)	$0\cdot024$
C5	$0\cdot1246$ (3)	$-0\cdot1253$ (3)	$-0\cdot1465$ (3)	$0\cdot030$
C6	$0\cdot1374$ (3)	$0\cdot0236$ (3)	$-0\cdot2438$ (2)	$0\cdot028$
C7	$0\cdot0080$ (3)	$0\cdot1538$ (3)	$-0\cdot2692$ (2)	$0\cdot027$
C8	$-0\cdot1673$ (3)	$0\cdot1738$ (3)	$-0\cdot2050$ (3)	$0\cdot031$
C9	$-0\cdot2609$ (3)	$0\cdot0718$ (3)	$-0\cdot1449$ (2)	$0\cdot028$
C10	$-0\cdot2495$ (3)	$-0\cdot0846$ (3)	$-0\cdot2815$ (3)	$0\cdot029$
C11	$-0\cdot2827$ (3)	$-0\cdot4337$ (3)	$0\cdot1515$ (2)	$0\cdot024$
C12	$-0\cdot1829$ (3)	$-0\cdot5397$ (3)	$0\cdot2709$ (3)	$0\cdot031$
C13	$-0\cdot2462$ (4)	$-0\cdot6560$ (3)	$0\cdot3689$ (3)	$0\cdot037$
C14	$-0\cdot4088$ (4)	$-0\cdot6698$ (3)	$0\cdot3524$ (3)	$0\cdot037$
C15	$-0\cdot5094$ (3)	$-0\cdot5656$ (3)	$0\cdot2346$ (3)	$0\cdot036$
C16	$-0\cdot4456$ (3)	$-0\cdot4491$ (3)	$0\cdot1360$ (3)	$0\cdot029$
C71	$0\cdot1908$ (4)	$0\cdot3025$ (3)	$-0\cdot4290$ (3)	$0\cdot041$

Table 2. *Bond lengths* (Å), *angles* (°) *and some dihedral and torsion angles* (°) *with e.s.d.'s in parentheses*

F1—C10	$1\cdot334$ (3)	F2—C10	$1\cdot345$ (3)
F3—C10	$1\cdot342$ (3)	O2—C2	$1\cdot211$ (3)
O7—C7	$1\cdot374$ (3)	O7—C71	$1\cdot439$ (3)
N1—C1	$1\cdot287$ (3)	N1—C4	$1\cdot501$ (3)
C1—C2	$1\cdot530$ (3)	C1—C11	$1\cdot464$ (3)
C2—C3	$1\cdot468$ (3)	C3—C4	$1\cdot492$ (3)
C3—C5	$1\cdot350$ (3)	C4—C9	$1\cdot508$ (3)
C4—C10	$1\cdot534$ (3)	C5—C6	$1\cdot436$ (3)
C6—C7	$1\cdot338$ (3)	C7—C8	$1\cdot469$ (3)
C8—C9	$1\cdot339$ (3)	C11—C12	$1\cdot409$ (3)
C11—C16	$1\cdot391$ (3)	C12—C13	$1\cdot375$ (3)
C13—C14	$1\cdot386$ (3)	C14—C15	$1\cdot397$ (3)
C15—C16	$1\cdot382$ (3)		
C7—O7—C71	$117\cdot3$ (2)	C1—N1—C4	$109\cdot5$ (2)
N1—C1—C2	$112\cdot9$ (2)	N1—C1—C11	$122\cdot9$ (2)
C2—C1—C11	$124\cdot2$ (2)	O2—C2—C1	$127\cdot3$ (2)
O2—C2—C3	$128\cdot8$ (2)	C1—C2—C3	$103\cdot8$ (2)
C2—C3—C4	$107\cdot3$ (2)	C2—C3—C5	$123\cdot0$ (2)
C4—C3—C5	$129\cdot5$ (2)	N1—C4—C3	$106\cdot2$ (2)
N1—C4—C9	$110\cdot9$ (2)	C3—C4—C9	$112\cdot8$ (2)
N1—C4—C10	$104\cdot3$ (2)	C3—C4—C10	$112\cdot0$ (2)
C9—C4—C10	$110\cdot2$ (2)	C3—C5—C6	$128\cdot0$ (2)
C5—C6—C7	$124\cdot3$ (2)	O7—C7—C6	$123\cdot4$ (2)
O7—C7—C8	$108\cdot4$ (2)	C6—C7—C8	$128\cdot0$ (2)
C7—C8—C9	$129\cdot4$ (2)	C4—C9—C8	$124\cdot9$ (2)
F1—C10—F2	$107\cdot0$ (2)	F1—C10—F3	$106\cdot5$ (2)
F2—C10—F3	$106\cdot3$ (2)	F1—C10—C4	$112\cdot0$ (2)
F2—C10—C4	$112\cdot0$ (2)	F3—C10—C4	$112\cdot6$ (2)
C1—C11—C12	$121\cdot1$ (2)	C1—C11—C16	$120\cdot2$ (2)
C12—C11—C16	$118\cdot6$ (2)	C11—C12—C13	$120\cdot2$ (2)
C12—C13—C14	$120\cdot6$ (2)	C13—C14—C15	$119\cdot9$ (2)
C14—C15—C16	$119\cdot4$ (2)	C11—C16—C15	$121\cdot2$ (2)
N1—C1—C2—C3	$0\cdot0$ (3)	C5—C3—C4—C9	$-48\cdot1$ (3)
C4—C3—C5—C6	$4\cdot5$ (3)	C3—C5—C6—C7	$23\cdot6$ (3)
C5—C6—C7—C8	$1\cdot1$ (3)	C6—C7—C8—C9	$-24\cdot6$ (3)
C7—C8—C9—C4	$-5\cdot7$ (3)	C3—C4—C9—C8	$47\cdot3$ (3)

C11, C12, C13, C14, C15, C16/N1, C1, C2, C3, C4 $25\cdot0$ (3)

N1, C1, C2, C3/C3, C4, N1 $5\cdot7$ (3)

1,3-dipolar species surprisingly acts as a carbene and inserts into the donor substituted aryl moiety to form a cycloheptatriene ring system *via* [2 + 1] cyclo-addition and electrocyclic ring opening. Finally, during work-up the 1-pyrroline substructure undergoes an autoxidation process to give compound (3).

Related literature. (3) was prepared by thermolysis of 2-(methoxybenzyl)-2-phenyl-4-trifluoromethyl-5(2H)-oxazolone, (2), which in turn is available from 5-(4-methoxybenzyloxy)-2-phenyl-4-trifluoromethyloxazole, (1), by a thermally induced rearrangement (Burger, Schierlinger, Gaa, Geith, Sewald & Müller, 1989). The reaction sequence (1) > (2) > (3) is a unique combination of remarkable reaction steps: the rearrangement (1) > (2) represents a non-concerted 1,5-benzyl-group migration from oxygen to carbon (Burger, Gaa, Geith & Schierlinger, 1989). The transformation (2) > (3) involves a nitrile ylide intermediate which is formed by [3 + 2] cyclo-elimination of carbon dioxide from (2). The

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Structure of the 1:1 Adduct of 2-Phenyl-4-(4,6-dimethyl-2-pyrimidinyl)aminomethylene-5(4H)-oxazolone and Acetic Acid

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Abstract. $C_{16}H_{14}N_4O_2.C_2H_4O_2$, $M_r = 354.4$, monoclinic, $P2_1/c$, $a = 12.768$ (2), $b = 7.416$ (3), $c = 19.206$ (3) Å, $\beta = 98.71$ (1)°, $V = 1797.6$ (8) Å^3, $Z = 4$, $D_x = 1.309$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.887$ cm^{-1}, $F(000) = 744$, $T = 293$ (2) K, final $R = 0.044$ and $wR = 0.069$ for 1441 observed reflections. The phenyl, pyrimidinyl and oxazolone rings are planar to within 0.010 (5), 0.009 (4) and 0.009 (4) Å, respectively and the whole molecule to within 0.117 (4) Å. The molecule of acetic acid seems to stabilize the planar shape *via* two hydrogen bonds of the type O—H···N [2.756 (5) Å] and O···H—N [2.891 (5) Å]. The bond lengths and angles agree with expected values.

Experimental. The crystals of the title compound were prepared in an acetic acid solution. A plate-like colourless single crystal with poor scattering properties (0.80 × 0.60 × 0.16 mm) was sealed, together with a drop of mother liquor, into the glass capillary (crystals without the liquid decomposed when exposed to air). Cell dimensions were determined from the angular settings of 25 reflections with 9 < θ < 12° measured on an Enraf–Nonius CAD-4 diffrac-