

Table 4. Selected geometric parameters (Å, °) for (II)

N—S	1.631 (2)	C(9)—C(8)	1.520 (3)
O(1)—S	1.418 (2)	C(15)—C(8)	1.534 (3)
O(2)—S	1.441 (2)	C(10)—C(9)	1.384 (4)
C(16)—S	1.751 (3)	C(14)—C(9)	1.378 (3)
C(1)—N	1.492 (3)	C(11)—C(10)	1.395 (5)
C(8)—N	1.459 (3)	C(12)—C(11)	1.376 (6)
C(15)—O(3)	1.303 (3)	C(13)—C(12)	1.378 (6)
C(15)—O(4)	1.222 (3)	C(14)—C(13)	1.384 (5)
C(2)—C(1)	1.498 (3)	C(17)—C(16)	1.399 (4)
C(3)—C(2)	1.376 (3)	C(21)—C(16)	1.379 (4)
C(7)—C(2)	1.380 (3)	C(18)—C(17)	1.366 (5)
C(4)—C(3)	1.401 (4)	C(19)—C(18)	1.364 (7)
C(5)—C(4)	1.361 (5)	C(20)—C(19)	1.391 (6)
C(6)—C(5)	1.334 (5)	C(22)—C(19)	1.511 (5)
C(7)—C(6)	1.403 (4)	C(21)—C(20)	1.378 (5)
O(1)—S—N	105.9 (1)	C(10)—C(9)—C(8)	122.9 (2)
O(2)—S—N	106.4 (1)	C(14)—C(9)—C(8)	117.4 (2)
O(2)—S—O(1)	120.5 (1)	C(14)—C(9)—C(10)	119.5 (2)
C(16)—S—N	107.3 (1)	C(11)—C(10)—C(9)	119.6 (3)
C(16)—S—O(1)	107.9 (1)	C(12)—C(11)—C(10)	120.4 (4)
C(16)—S—O(2)	108.1 (1)	C(13)—C(12)—C(11)	119.8 (3)
C(1)—N—S	118.9 (2)	C(14)—C(13)—C(12)	119.9 (3)
C(8)—N—S	118.2 (1)	C(13)—C(14)—C(9)	120.7 (3)
C(8)—N—C(1)	119.5 (2)	O(4)—C(15)—O(3)	123.8 (2)
C(2)—C(1)—N	115.8 (2)	C(8)—C(15)—O(3)	112.7 (2)
C(3)—C(2)—C(1)	118.1 (2)	C(8)—C(15)—O(4)	123.4 (2)
C(7)—C(2)—C(1)	124.8 (2)	C(17)—C(16)—S	119.7 (2)
C(7)—C(2)—C(3)	117.1 (2)	C(21)—C(16)—S	120.4 (2)
C(4)—C(3)—C(2)	121.5 (3)	C(21)—C(16)—C(17)	119.9 (3)
C(5)—C(4)—C(3)	119.6 (3)	C(18)—C(17)—C(16)	119.0 (4)
C(6)—C(5)—C(4)	120.0 (3)	C(19)—C(18)—C(17)	122.6 (4)
C(7)—C(6)—C(5)	121.1 (3)	C(20)—C(19)—C(18)	117.7 (3)
C(6)—C(7)—C(2)	120.6 (3)	C(22)—C(19)—C(18)	122.7 (5)
C(9)—C(8)—N	110.8 (2)	C(22)—C(19)—C(20)	119.6 (5)
C(15)—C(8)—N	111.9 (2)	C(21)—C(20)—C(19)	121.7 (4)
C(15)—C(8)—C(9)	113.8 (2)	C(20)—C(21)—C(16)	119.1 (4)

All H-atom positions in (I) were calculated and refined with an overall isotropic temperature factor using a riding model [$U_{\text{iso}} = 0.108(4) \text{ \AA}^2$]. The positions of four H atoms in (II) were calculated and the remainder were located from a difference synthesis. All were refined with an overall isotropic temperature factor, using a riding model for calculated H atoms [$U_{\text{iso}} = 0.106(3) \text{ \AA}^2$]. Program used to solve structures: *SHELXS86* (Sheldrick, 1990). Program used to refine structures: *SHELX76* (Sheldrick, 1976). Molecular graphics: *SCHAKAL* (Keller, 1988). Most calculations were performed using *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: KA1061). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A New Class of Substituted 1,2,4-Triazolo-1,3,4-thiadiazepines

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Abstract

A series of 3-aryloxymethyl-(5-nitro-2-furyl)-6-phenyl-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazepine compounds have been synthesized recently by a new route. Reported here are the structures of two such compounds with *para*-substituted aryloxymethyl groups: one has a chloro group, 3-(4-chlorophenyl-oxymethyl)-8-(5-nitro-2-furyl)-6-phenyl-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazepine, C₂₂H₁₄ClN₅O₄S, TD1, and the other a methyl group, 3-(*p*-tolyl-oxymethyl)-8-(5-nitro-2-furyl)-6-phenyl-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazepine, C₂₃H₁₇N₅O₄S, TD7. The nitrofuryl and phenyl groups on the thiadiazepine ring are each found to adopt a similar conformation in the two structures, whereas the aryloxymethyl substituents on the triazole rings are conformationally different from each other. Each thiadiazepine ring adopts a boat conformation with the S atom at the apex. The interplanar angle between the

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triazole ring and the thiadiazepine ring is 30° for both compounds. The conformation of the aryloxy-methyl group is dependent on the intermolecular interactions that arise as a result of the polarity of the *para* substituent. The Cl group in TD1 is involved in a C—Cl \cdots O non-bonded interaction with a Cl \cdots O distance of 3.100 (3) Å and a C—Cl \cdots O angle of $138.4 (1)^\circ$. TD7 has a stacking interaction involving the nitrofuryl groups.

Comment

The synthesis of a triazolothiadiazepine was first reported by Heindel & Reid (1980). A new synthetic scheme has been reported recently by Shridhara (1991) using a nitrofurylacetylenic ketone as a starting compound. Initially there was ambiguity as to whether a six- or seven-membered ring was formed in the final product. Physical measurements (IR, NMR and mass spectroscopy) did not conclusively determine the ring size. Earlier results from similar reactions involving condensation of aminomercapto-triazoles and nitrofuryl α -bromopropenones had resulted in only a six-membered thiadiazine ring (Holla *et al.*, 1994). Condensation of aminomercapto-triazole and α -bromo-nitrophenylfuryl-propenones also produces a thiadiazine derivative rather than a thiadiazepine ring (Akberali, 1991). However, when the starting material was nitrofurylacetylenic ketone the formation of the thiadiazepine ring was observed.

Fig. 1 presents ORTEPII drawings (Johnson, 1976) of molecules of TD1 and TD7 in the same relative orientation. Fig. 2 presents packing diagrams of the compounds showing the different intermolecular interactions. Examination of Table 2 shows that the C1—C6 bond distance between the thiadiazepine and nitrofuryl groups has an average length of

1.447 (3) Å and that the C3—C10 bond distance between the thiadiazepine and aryl groups has an average length of 1.485 (3) Å for the two compounds. This indicates that any conjugation in the aryl and nitrofuryl rings is localized within the rings and does not extend into the thiadiazepine ring. The S atom of the thiadiazepine and the O atom of the furyl ring are *trans* to each other. The central 1,2,4-triazolothiadiazepine moiety, and the aryl and nitrofuryl parts of TD1 and TD7 have a high degree of similarity as shown by the close agreement in the corresponding bond distances, bond angles and torsion angles. In contrast, the aryloxymethyl group orientation is dependent on the polarity of the substituents on the phenyl ring. Examination of the torsion angles about the C5—C16—O2 and N4—C5—C16—O2, are $-179.0 (2)$ and $-0.8 (4)^\circ$, respectively, for TD1, and $80.9 (3)$ and $-102.9 (3)^\circ$, respectively, for TD7. TD1, with a polar *p*-chlorophenyl moiety, exhibits an electrophilic-

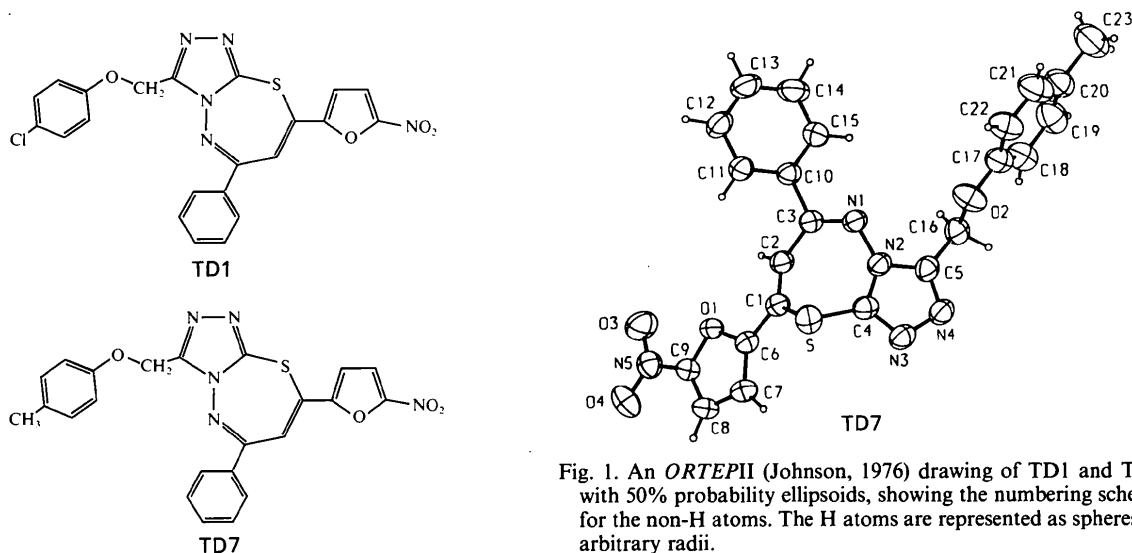


Fig. 1. An ORTEPII (Johnson, 1976) drawing of TD1 and TD7 with 50% probability ellipsoids, showing the numbering scheme for the non-H atoms. The H atoms are represented as spheres of arbitrary radii.

nucleophilic interaction of the type C—Cl...O, as described by Ramasubbu, Parthasarathy & Murray-Rust (1986), with the nitro group of a symmetry-related molecule. The Cl...O distance of 3.100 (3) Å and the C—Cl...O angle of 138.4 (1)° are within the area mapped out for this type of interaction. TD7, which has a non-polar tolyl moiety, exhibits a stacking interaction involving the nitrofuryl groups along the crystallographic *a* axis. The interplanar angles are 0.0° and the distances between the planes are 3.16 (3) and 3.41 (3) Å for the two pairs of symmetry-related molecules involved in the interaction.

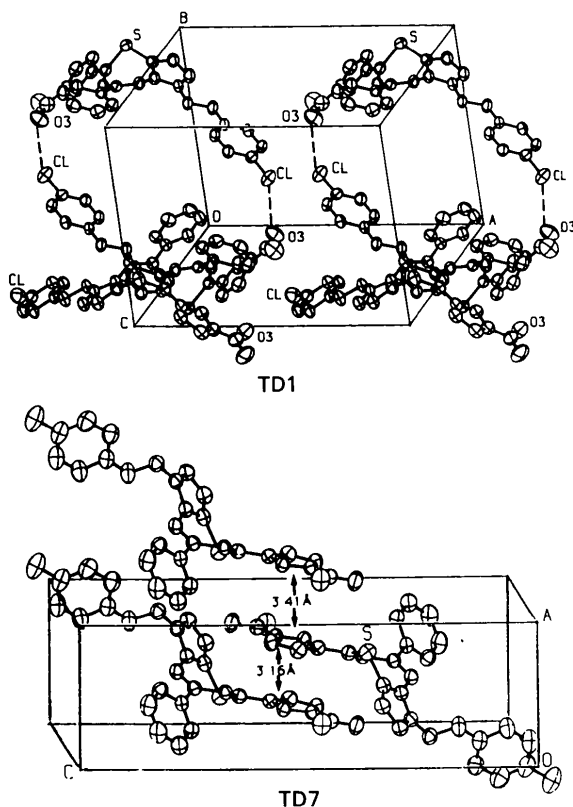


Fig. 2. ORTEP (Johnson, 1976) drawing of a unit cell of each compound showing the intermolecular interactions described in the text. The H atoms are not shown.

One other interaction that is of interest involves the phenyl ring on the thiadiazepine ring and the furan ring of a symmetry-related molecule in TD1. This aromatic-aromatic interaction appears to be quite strong. The centre-centre distance is 4.1 (2) Å with an interplanar angle of 18.0 (3)°. This interaction is similar to the phenyl-phenyl interactions of peptides and proteins described by Burley & Petsko (1985), and also by Williams (1980) for crystalline benzene.

Experimental

TD1

Crystal data

C₂₂H₁₄ClN₅O₄S
M_r = 479.90
 Monoclinic
*P*2₁/*c*
a = 15.444 (5) Å
b = 12.664 (1) Å
c = 10.996 (1) Å
 β = 99.61 (2)°
V = 2120.4 (11) Å³
Z = 4
D_x = 1.50 Mg m⁻³

Data collection

Enraf-Nonius CAD-4 diffractometer
 ω -2 θ scans
 Absorption correction: none
 4508 measured reflections
 4337 independent reflections
 2947 observed reflections
 [*I* > 2 σ (*I*)]

Refinement

Refinement on *F*
R = 0.044
wR = 0.052
S = 1.36
 2947 reflections
 355 parameters
 All H-atom parameters refined
w = 1/ σ^2 (*F*)
 (Δ/σ)_{max} = 0.01

TD7

Crystal data

C₂₃H₁₇N₅O₄S
M_r = 459.49
 Triclinic
P $\bar{1}$
a = 6.755 (1) Å
b = 7.614 (1) Å
c = 21.537 (2) Å
 α = 89.25 (1)°
 β = 88.82 (1)°
 γ = 78.95 (1)°
V = 1086.8 (7) Å³
Z = 2
D_x = 1.40 Mg m⁻³

Data collection

Enraf-Nonius CAD-4 diffractometer
 ω -2 θ scans
 Absorption correction: none

Cu *K* α radiation

λ = 1.5418 Å

Cell parameters from 25 reflections

θ = 20–30°

μ = 2.87 mm⁻¹

T = 293 K

Rectangular plates

0.7 × 0.3 × 0.1 mm

Yellow

*R*_{int} = 0.033

θ _{max} = 75°

h = -13 → 13

k = 0 → 15

l = 0 → 19

3 standard reflections

frequency: 60 min

intensity variation: -3.1%

$\Delta\rho$ _{max} = 0.283 e Å⁻³

$\Delta\rho$ _{min} = -0.203 e Å⁻³

Extinction correction:

Zachariasen (1967)

Atomic scattering factors

from Cromer & Waber

(1974)

Cu *K* α radiation

λ = 1.5418 Å

Cell parameters from 25 reflections

θ = 20–30°

μ = 1.63 mm⁻¹

T = 298 K

Rectangular plate

0.5 × 0.3 × 0.1 mm

Yellow

*R*_{int} = 0.022

θ _{max} = 75°

h = -8 → 8

k = -9 → 9

l = 0 → 27

4598 measured reflections
 4479 independent reflections
 3037 observed reflections
 [$I > 2\sigma(I)$]

3 standard reflections
 frequency: 60 min
 intensity variation: 0.1%

Refinement

Refinement on F^2
 $R = 0.043$
 $wR = 0.054$
 $S = 1.513$
 3037 reflections
 367 parameters
 All H-atom parameters
 refined
 $w = 1/\sigma^2(F)$
 $(\Delta/\sigma)_{\max} = 0.73$ (for H
 atoms)

$\Delta\rho_{\max} = 0.218 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.244 \text{ e } \text{\AA}^{-3}$
 Extinction correction:
 Zachariasen (1967)
 Atomic scattering factors
 from Cromer & Waber
 (1974)

C1	0.7179 (3)	0.2792 (3)	0.3945 (1)	0.046 (5)
C2	0.6477 (3)	0.4157 (3)	0.3560 (1)	0.045 (5)
C3	0.6195 (3)	0.4072 (3)	0.2886 (1)	0.043 (5)
C4	0.5652 (4)	0.0397 (3)	0.3348 (1)	0.048 (5)
C5	0.3109 (3)	0.0879 (3)	0.2725 (1)	0.049 (5)
C6	0.7467 (3)	0.3018 (3)	0.4601 (1)	0.044 (5)
C7	0.7708 (4)	0.1876 (3)	0.5098 (1)	0.055 (6)
C8	0.7920 (4)	0.2894 (3)	0.5625 (1)	0.054 (6)
C9	0.7788 (3)	0.4581 (3)	0.5414 (1)	0.045 (5)
C10	0.6784 (3)	0.5544 (3)	0.2510 (1)	0.044 (5)
C11	0.8453 (4)	0.6229 (3)	0.2672 (1)	0.051 (6)
C12	0.9052 (4)	0.7584 (3)	0.2318 (1)	0.065 (7)
C13	0.7928 (5)	0.8282 (3)	0.1817 (1)	0.074 (8)
C14	0.6252 (5)	0.7628 (4)	0.1658 (1)	0.074 (8)
C15	0.5668 (4)	0.6243 (3)	0.1995 (1)	0.057 (6)
C16	0.1643 (4)	0.1717 (3)	0.2260 (1)	0.054 (6)
C17	0.1277 (4)	0.1837 (3)	0.1158 (1)	0.060 (7)
C18	-0.0710 (5)	0.2663 (4)	0.1200 (1)	0.070 (8)
C19	-0.1749 (5)	0.3165 (4)	0.0648 (1)	0.082 (9)
C20	-0.0863 (5)	0.2840 (4)	0.0076 (1)	0.078 (8)
C21	0.1132 (5)	0.2002 (4)	0.0053 (1)	0.09 (1)
C22	0.2207 (5)	0.1510 (4)	0.0583 (1)	0.078 (9)
C23	-0.2037 (6)	0.3419 (5)	-0.0514 (2)	0.11 (1)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for compounds

TD1 and TD7				
$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$				
	x	y	z	U_{eq}
TD1				
S	0.1806 (1)	0.0513 (1)	0.9231 (1)	0.052 (2)
C1	-0.4589 (1)	0.4282 (1)	0.3381 (1)	0.069 (2)
O1	0.3716 (1)	0.2501 (2)	0.8664 (2)	0.047 (4)
O2	-0.1730 (1)	0.2595 (2)	0.7039 (2)	0.052 (5)
O3	0.5018 (2)	0.3684 (2)	0.8169 (2)	0.081 (7)
O4	0.5535 (2)	0.3873 (2)	1.0104 (2)	0.094 (8)
N1	0.0654 (1)	0.1085 (2)	0.6539 (2)	0.041 (5)
N2	0.0441 (1)	0.1402 (2)	0.7670 (2)	0.040 (5)
N3	0.0309 (1)	0.1495 (2)	0.9628 (2)	0.050 (6)
N4	-0.0449 (1)	0.1917 (2)	0.8913 (2)	0.051 (6)
N5	0.5006 (2)	0.3518 (2)	0.9256 (2)	0.061 (7)
C1	0.2434 (2)	0.1386 (2)	0.8454 (2)	0.043 (6)
C2	0.2212 (2)	0.1574 (2)	0.7238 (2)	0.043 (6)
C3	0.1462 (2)	0.1149 (2)	0.6387 (2)	0.038 (6)
C4	0.0821 (2)	0.1192 (2)	0.8868 (2)	0.043 (6)
C5	-0.0358 (2)	0.1834 (2)	0.7757 (2)	0.041 (6)
C6	0.3192 (2)	0.1849 (2)	0.9218 (2)	0.044 (6)
C7	0.3510 (2)	0.1785 (3)	1.0457 (3)	0.053 (7)
C8	0.4252 (2)	0.2440 (3)	1.0684 (3)	0.056 (8)
C9	0.4342 (2)	0.2848 (2)	0.9589 (3)	0.051 (7)
C10	0.1630 (2)	0.0853 (2)	0.5139 (2)	0.039 (6)
C11	0.2445 (2)	0.0457 (3)	0.4974 (3)	0.049 (7)
C12	0.2591 (2)	0.0184 (3)	0.3813 (3)	0.058 (8)
C13	0.1947 (2)	0.0323 (3)	0.2800 (3)	0.057 (8)
C14	0.1138 (2)	0.0707 (3)	0.2948 (3)	0.054 (8)
C15	0.0972 (2)	0.0964 (2)	0.4110 (3)	0.047 (7)
C16	-0.0974 (2)	0.2161 (2)	0.6646 (3)	0.046 (6)
C17	-0.2377 (2)	0.2964 (2)	0.6133 (2)	0.041 (6)
C18	-0.3072 (2)	0.3472 (2)	0.6543 (3)	0.048 (6)
C19	-0.3749 (2)	0.3879 (2)	0.5710 (3)	0.050 (7)
C20	-0.3735 (2)	0.3777 (2)	0.4457 (3)	0.046 (7)
C21	-0.3045 (2)	0.3289 (2)	0.4044 (2)	0.047 (7)
C22	-0.2364 (2)	0.2871 (2)	0.4885 (2)	0.045 (6)
TD7				
S	0.7924 (1)	0.0559 (1)	0.3678 (1)	0.058 (2)
O1	0.7508 (2)	0.4727 (2)	0.4793 (1)	0.045 (3)
O2	0.2454 (3)	0.1229 (2)	0.1660 (1)	0.070 (5)
O3	0.8267 (3)	0.6045 (3)	0.6285 (1)	0.074 (5)
O4	0.7666 (3)	0.7576 (2)	0.5432 (1)	0.080 (6)
N1	0.5408 (3)	0.2940 (2)	0.2574 (1)	0.048 (5)
N2	0.4756 (3)	0.1541 (2)	0.2892 (1)	0.046 (4)
N3	0.4614 (3)	-0.0860 (2)	0.3455 (1)	0.055 (5)
N4	0.2979 (3)	-0.0541 (3)	0.3061 (1)	0.055 (5)
N5	0.7915 (3)	0.6178 (3)	0.5724 (1)	0.054 (5)

Table 2. Selected geometric parameters (\AA , $^\circ$)

	TD1	TD7
S—C4	1.735 (3)	1.731 (3)
S—C1	1.781 (3)	1.777 (2)
C1—C2	1.346 (4)	1.341 (3)
C2—C3	1.465 (3)	1.471 (3)
N1—C3	1.289 (3)	1.297 (3)
N1—N2	1.398 (3)	1.395 (3)
N2—C4	1.376 (3)	1.373 (3)
N2—C5	1.368 (3)	1.373 (3)
N3—N4	1.403 (3)	1.363 (3)
N3—C4	1.300 (4)	1.389 (3)
N4—C5	1.306 (4)	1.307 (3)
C1—C6	1.446 (3)	1.308 (3)
O1—C6	1.368 (3)	1.448 (3)
O1—C9	1.355 (3)	1.375 (3)
C6—C7	1.371 (4)	1.357 (3)
C7—C8	1.404 (4)	1.363 (3)
C8—C9	1.339 (4)	1.408 (3)
C21—C22	1.384 (3)	1.345 (3)
C20—C23	—	1.374 (4)
N5—C9	1.425 (4)	1.413 (3)
O3—N5	1.217 (3)	1.234 (3)
O4—N5	1.218 (3)	1.214 (3)
C3—C10	1.486 (4)	1.484 (3)
C10—C11	1.396 (3)	1.382 (4)
C10—C15	1.395 (4)	1.395 (3)
C11—C12	1.383 (4)	1.391 (4)
C12—C13	1.375 (4)	1.373 (4)
C13—C14	1.375 (4)	1.373 (5)
C14—C15	1.375 (4)	1.387 (4)
C5—C16	1.477 (3)	1.174 (3)
O2—C16	1.419 (3)	1.419 (3)
O2—C17	1.371 (3)	1.378 (3)
C17—C18	1.389 (4)	1.370 (4)
C17—C22	1.381 (4)	1.380 (4)
C18—C19	1.371 (4)	1.404 (4)
C19—C20	1.387 (4)	1.363 (4)
C20—C21	1.374 (4)	1.376 (5)
C1—C20	1.739 (3)	—
C1—S—C4	96.2 (1)	96.8 (1)
S—C1—C2	121.2 (2)	122.2 (2)
C1—C2—C3	137.9 (3)	127.2 (2)
N1—C3—C2	128.3 (2)	129.1 (2)
N2—N1—C3	118.3 (2)	118.9 (2)
N1—N2—C4	132.1 (2)	131.1 (2)
S—C4—N2	122.3 (2)	123.5 (2)
N1—N2—C5	121.8 (2)	123.1 (2)
C4—N2—C5	104.9 (2)	105.2 (2)
N4—N3—C4	107.0 (2)	107.1 (2)
N3—N4—C5	107.5 (2)	107.8 (2)

S—C4—N3	127.0 (2)	126.3 (2)
N2—C4—N3	110.5 (2)	110.1 (2)
N2—C5—N4	110.2 (2)	109.9 (2)
N2—C5—C16	121.2 (2)	124.4 (2)
N4—C5—C16	128.6 (2)	125.7 (2)
O2—C16—C5	107.7 (2)	108.4 (2)
C16—O2—C17	116.6 (2)	117.5 (2)
O2—C17—C18	115.6 (2)	124.4 (2)
O2—C17—C22	124.3 (2)	115.5 (2)
C18—C17—C22	120.1 (2)	120.0 (3)
C17—C18—C19	120.1 (3)	118.4 (3)
C18—C19—C20	119.5 (3)	122.5 (3)
C19—C20—C21	120.8 (3)	117.3 (3)
C20—C21—C22	119.7 (3)	121.9 (3)
C17—C22—C21	119.7 (3)	119.8 (3)
C19—C20—C23	—	121.0 (3)
N1—C3—C10	115.2 (2)	114.9 (2)
C2—C3—C10	116.2 (2)	115.8 (2)
C3—C10—C11	120.5 (2)	119.7 (2)
C3—C10—C15	120.8 (2)	120.6 (2)
C11—C10—C15	118.8 (2)	119.7 (2)
C10—C11—C12	120.2 (3)	120.5 (2)
C11—C12—C13	120.3 (2)	119.4 (3)
C12—C13—C14	119.9 (3)	120.5 (3)
C13—C14—C15	120.7 (3)	120.7 (3)
C10—C15—C14	120.1 (2)	119.1 (3)
S—C1—C6	115.2 (1)	114.6 (2)
C2—C1—C6	123.6 (2)	123.2 (2)
O1—C6—C1	117.8 (2)	116.5 (2)
O1—C6—C7	109.8 (2)	109.8 (2)
C6—O1—C9	105.2 (2)	105.0 (2)
C1—C6—C7	132.3 (3)	133.7 (2)
C6—C7—C8	106.7 (3)	107.3 (2)
C7—C8—C9	105.8 (2)	105.3 (2)
O1—C9—C8	112.4 (3)	112.6 (2)
O1—C9—N5	117.1 (2)	116.2 (2)
N5—C9—C8	130.4 (3)	131.2 (2)
O3—N5—O4	124.8 (3)	124.3 (2)
O3—N5—C9	119.0 (3)	116.7 (2)
O4—N5—C9	116.3 (3)	119.0 (2)
C1—C20—C19	120.4 (2)	—
C1—C20—C21	118.8 (2)	—
C21—C20—C23	—	121.7 (3)
N2—C5—C16—O2	−179.0 (2)	80.9 (3)
N4—C5—C16—O2	−0.8 (4)	−102.9 (3)
C5—C16—O2—C17	178.7 (2)	178.0 (2)
C16—O2—C17—C18	−174.1 (2)	−9.5 (4)
C16—O2—C17—C22	5.2 (4)	172.7 (2)
S—C1—C6—O1	177.9 (2)	160.9 (2)
C2—C1—C6—O1	−1.0 (4)	−16.8 (3)
S—C1—C6—C7	−3.4 (4)	−19.6 (4)
C2—C1—C6—C7	177.7 (3)	162.7 (3)
O1—C9—N5—O3	−5.1 (4)	−176.6 (2)
O1—C9—N5—O4	175.9 (3)	3.8 (3)
C8—C9—N5—O3	170.5 (3)	2.5 (4)
C8—C9—N5—O4	−8.5 (5)	−177.1 (3)
C2—C3—C10—C11	33.2 (4)	36.9 (3)
C2—C3—C10—C15	−146.9 (3)	−143.0 (2)
N1—C3—C10—C11	−152.7 (3)	−146.4 (2)
N1—C3—C10—C15	27.2 (4)	33.7 (3)

The structures were solved by direct methods using *MULTAN11/82* (Main *et al.*, 1982). All calculations were performed on a MicroVAX II using the *SDP/VAX* structure determination package (Frenz, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry have been deposited with the IUCr (Reference: CR1075). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tuberostemonine L-G

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Abstract

The tuberostemonine molecule, C₂₂H₃₃NO₄, consists of a pyrrolidine, a cyclohexane, an azepine and two furan rings. Each furan ring adopts the envelope conformation while the pyrrolidine ring takes that of a twist-chair. The cyclohexane and the azepine rings have chair conformations. In the title compound, the vicinal rings are *cis*-fused, whereas in a previously investigated tuberostemonine (m.p. 359–361 K) a