

Acta Cryst. (1993). **C49**, 1637–1638

Structure of 2,7,7-Trimethyl-5,6,7,8-tetrahydro-4*H*-furo[3,2-*c*]azepine-4-thione

M. SORIANO-GARCÍA,* E. CORTÉS C., E. CORTÉS R., J. G. AVILA Z. AND A. DOMÍNGUEZ T.

Instituto de Química,† Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán 04510, México DF

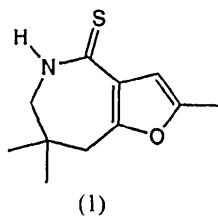
(Received 5 October 1992; accepted 17 February 1993)

Abstract

The asymmetric unit of the title compound contains two molecules with very similar geometry. The five-membered furan ring is planar and is *cis*-fused to the seven-membered azepine ring. The seven-membered ring adopts a twisted-sofa conformation TS1 [Boessenkool & Boeyens (1980). *J. Crystallogr. Spectrosc. Res.* **10**, 11–18]. N—H...S bonds link adjacent molecules in the crystal.

Comment

The title compound (1) has been prepared as part of an investigation of furo[3,2-*c*]azepine analogues with tranquilizer activity (Weiss, Gibs, Poletto & Remers, 1973).



† Contribution No. 1180 of the Instituto de Química, UNAM.

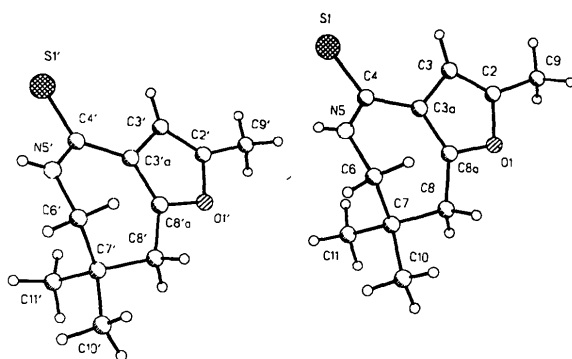


Fig. 1. The molecular structure of the title compound with atom numbering.

Adjacent molecules in the crystal are linked by hydrogen bonds; N(5)—H...S(1') is 3.367 (5) Å, N(5')—H...S(1) (1 - x, 1 - y, -z) is 3.432 Å. The C—S(*sp*²) bond lengths of 1.683 (4) and 1.680 (4) Å are in agreement with the value of 1.671 (24) Å given by Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987).

Experimental

Crystal data

C₁₁H₁₅NOS
M_r = 209.3
 Triclinic
P $\bar{1}$
a = 10.359 (5) Å
b = 10.861 (5) Å
c = 9.947 (4) Å
 α = 91.89 (3)°
 β = 99.97 (4)°
 γ = 89.65 (4)°
V = 1102 (1) Å³
Z = 4

D_x = 1.26 Mg m⁻³
 Cu K α radiation
 λ = 1.5418 Å
 Cell parameters from 25 reflections
 θ = 4.5–13.05°
 μ = 2.29 mm⁻¹
T = 293 K
 Cubes
 0.2 × 0.2 × 0.2 mm
 Colourless
 Crystal source: ethanol

Data collection

Nicolet P3/F diffractometer
 2 θ / θ scans
 Absorption correction:
 none
 24 211 measured reflections
 2257 independent reflections
 1898 observed reflections
 $[I > 2.5\sigma(I)]$
R_{int} = 0.021

θ_{\max} = 110°
h = -10 → 10
k = -10 → 10
l = 0 → 9
 2 standard reflections monitored every 50 reflections
 intensity variation: <3%

Refinement

Refinement on *F*
 Final *R* = 0.065
wR = 0.088
S = 1.06
 1898 reflections
 259 parameters
 $w = [\sigma^2(F) + 0.0054F^2]^{-1}$

$(\Delta/\sigma)_{\max}$ = 0.011
 $\Delta\rho_{\max}$ = 0.42 e Å⁻³
 $\Delta\rho_{\min}$ = -0.39 e Å⁻³
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
S(1)	0.0625 (1)	0.5151 (1)	0.2019 (1)	61 (1)
O(1)	-0.0795 (3)	0.7852 (3)	0.5506 (3)	55 (1)
C(2)	-0.1673 (4)	0.6945 (4)	0.4941 (4)	52 (2)
C(3)	-0.1106 (4)	0.6193 (4)	0.4134 (4)	49 (2)
C(3a)	0.0215 (4)	0.6629 (3)	0.4173 (4)	41 (1)
C(4)	0.1146 (4)	0.6074 (4)	0.3396 (4)	46 (2)
N(5)	0.2400 (4)	0.6338 (4)	0.3761 (4)	58 (2)
C(6)	0.3054 (4)	0.7043 (4)	0.4945 (5)	57 (2)
C(7)	0.2662 (4)	0.8403 (4)	0.5002 (4)	50 (2)
C(8)	0.1394 (4)	0.8533 (4)	0.5575 (5)	61 (2)
C(8a)	0.0354 (4)	0.7643 (4)	0.5024 (4)	47 (2)
C(9)	-0.2967 (4)	0.6995 (5)	0.5369 (5)	71 (2)
C(10)	0.3732 (4)	0.9071 (4)	0.6005 (5)	65 (2)
C(11)	0.2537 (5)	0.8957 (5)	0.3618 (5)	82 (2)

S(1')	0.4994 (1)	0.4893 (1)	-0.2602 (1)	56 (1)
O(1')	0.3478 (3)	0.8170 (3)	0.0228 (3)	55 (1)
C(2')	0.2532 (4)	0.7425 (4)	-0.0523 (4)	50 (2)
C(3')	0.3106 (4)	0.6526 (4)	-0.1154 (4)	49 (2)
C(3'a)	0.4505 (4)	0.6700 (3)	-0.0788 (4)	42 (1)
C(4')	0.5480 (4)	0.5909 (4)	-0.1306 (4)	46 (2)
N(5')	0.6725 (3)	0.6013 (3)	-0.0751 (4)	50 (1)
C(6')	0.7336 (4)	0.6711 (4)	0.0467 (4)	51 (2)
C(7')	0.7127 (4)	0.8105 (4)	0.0450 (4)	47 (2)
C(8')	0.5811 (4)	0.8399 (4)	0.0830 (5)	56 (2)
C(8'a)	0.4678 (4)	0.7712 (4)	0.0041 (4)	49 (2)
C(9')	0.1153 (4)	0.7752 (4)	-0.0444 (5)	67 (2)
C(10')	0.8186 (4)	0.8701 (4)	0.1525 (5)	61 (2)
C(11')	0.7223 (5)	0.8607 (4)	-0.0949 (5)	64 (2)

Table 2. Geometric parameters (Å, °)

S(1)—C(4)	1.683 (4)	O(1)—C(2)	1.383 (5)
O(1)—C(8a)	1.372 (5)	C(2)—C(3)	1.331 (6)
C(2)—C(9)	1.474 (7)	C(3)—C(3a)	1.445 (6)
C(3a)—C(4)	1.453 (6)	C(3a)—C(8a)	1.359 (5)
C(4)—N(5)	1.317 (5)	N(5)—C(6)	1.450 (6)
C(6)—C(7)	1.531 (6)	C(7)—C(8)	1.524 (6)
C(7)—C(10)	1.526 (6)	C(7)—C(11)	1.505 (7)
C(8)—C(8a)	1.473 (6)	S(1')—C(4')	1.680 (4)
O(1')—C(2')	1.375 (5)	O(1')—C(8'a)	1.376 (5)
C(2')—C(3')	1.335 (6)	C(2')—C(9')	1.485 (6)
C(3')—C(3'a)	1.443 (5)	C(3'a)—C(4')	1.472 (6)
C(3'a)—C(8'a)	1.345 (5)	C(4')—N(5')	1.316 (5)
N(5')—C(6')	1.457 (5)	C(6')—C(7')	1.529 (6)
C(7')—C(8')	1.506 (6)	C(7')—C(10')	1.524 (6)
C(7')—C(11')	1.530 (6)	C(8')—C(8'a)	1.485 (6)
C(2)—O(1)—C(8a)	107.6 (3)	O(1)—C(2)—C(3)	109.4 (4)
O(1)—C(2)—C(9)	115.7 (4)	C(3)—C(2)—C(9)	134.8 (4)
C(2)—C(3)—C(3a)	107.6 (4)	C(3)—C(3a)—C(4)	124.6 (3)
C(3)—C(3a)—C(8a)	106.1 (4)	C(4)—C(3a)—C(8a)	129.3 (4)
S(1)—C(4)—C(3a)	120.5 (3)	S(1)—C(4)—N(5)	120.6 (3)
C(3a)—C(4)—N(5)	118.9 (4)	C(4)—N(5)—C(6)	129.2 (4)
N(5)—C(6)—C(7)	115.2 (3)	C(6)—C(7)—C(8)	109.7 (4)
C(6)—C(7)—C(10)	107.3 (3)	C(8)—C(7)—C(10)	107.3 (4)
C(6)—C(7)—C(11)	111.1 (4)	C(8)—C(7)—C(11)	111.4 (4)
C(10)—C(7)—C(11)	109.8 (4)	C(7)—C(8)—C(8a)	115.8 (4)
O(1)—C(8a)—C(3a)	109.3 (3)	O(1)—C(8a)—C(8)	113.3 (3)
C(3a)—C(8a)—C(8)	137.4 (4)	C(2')—O(1')—C(8'a)	107.4 (3)
O(1')—C(2')—C(3')	109.4 (3)	O(1')—C(2')—C(9')	116.1 (4)
C(2')—C(3')—C(3'a)	134.5 (4)	C(2')—C(3')—C(3'a)	107.4 (3)
C(3')—C(3'a)—C(4')	123.9 (3)	C(3')—C(3'a)—C(8'a)	106.2 (4)
C(4')—C(3'a)—C(8'a)	129.8 (4)	S(1')—C(4')—C(3'a)	119.7 (3)
S(1')—C(4')—N(5')	120.8 (3)	C(3'a)—C(4')—N(5')	119.4 (4)
C(4')—N(5')—C(6')	129.8 (4)	N(5')—C(6')—C(7')	116.2 (3)
C(6')—C(7')—C(8')	109.0 (4)	C(6')—C(7')—C(10')	108.1 (3)
C(8')—C(7')—C(10')	108.5 (4)	C(6')—C(7')—C(11')	111.2 (4)
C(8')—C(7')—C(11')	110.8 (3)	C(10')—C(7')—C(11')	109.2 (4)
C(7')—C(8')—C(8'a)	116.0 (4)	O(1')—C(8'a)—C(3'a)	109.6 (3)
O(1')—C(8'a)—C(8')	114.0 (3)	C(3'a)—C(8'a)—C(8')	136.4 (4)

Data were corrected for Lorentz-polarization effects. The structure was solved by direct methods (*SHELXTL*; Sheldrick, 1985). The H-atom positions in the CH, CH₂ and CH₃ groups were calculated. Non-H atoms were refined anisotropically. The H atoms were assigned the fixed isotropic displacement parameter 0.060 Å² and coordinates of H atoms bonded to N were refined.

We are greatly indebted to Mr R. A. Toscano and Mrs Cynthia E. Lesh for technical assistance.

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71115 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1036]

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
- Boessenkool, I. K. & Boeyens, J. C. A. (1980). *J. Crystallogr. Spectrosc. Res.* **10**, 11–18.
- Sheldrick, G. M. (1985). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Revision 5.1. Univ. of Göttingen, Germany.
- Weiss, M. J., Gibb, G. J., Poletto, J. F. & Remers, W. A. (1973). US Patent 3 758 501.

Acta Cryst. (1993). **C49**, 1638–1640

Structure of a 2-Arylquinoline Dimer, a Compound with a New Ring System

HERMAN L. AMMON

Department of Chemistry and Biochemistry, University of Maryland, College Park, MD 20742, USA

THEODORE L. REID†

Department of Chemistry, American University, Washington, DC 20016, USA

(Received 2 June 1992; accepted 8 February 1993)

Abstract

The title compound, 6,15-dimethyltribenzo-*[c,f,j]*naphtho[1,2,3,4-*lmn*][2,7]phenanthroline, was obtained in low yield from the reaction of 2-(4-methylphenyl)quinoline and various aryl-lithiums. The compound is a 'dimer' in which two phenylquinoline units are joined by three bonds; the dimer has exact twofold symmetry. Non-bonded interactions produce substantial out-of-plane distortions. Bond lengths between the two phenylquinoline halves suggest that the two phenylquinoline π -electron systems are more-or-less undisturbed and linked by single bonds. Molecular-mechanics optimizations of models with inversion ($\bar{1}$) and twofold symmetry suggest that the latter conformation is the more stable of the two to a slight extent.

Comment

The reactions of certain 2-arylquinolines with aryl-lithiums (*e.g.* aryl = phenyl, 2-methylphenyl, 4-methylphenyl, 2,4,6-trimethylphenyl) give, in low yield, 'dimeric' products with molecular weights that are equal to twice that of the starting arylquinoline

† On leave from the National Science Foundation.