

Fig. 1. ORTEP view of  $C_{12}H_{16}N_4S_6$ , 50% ellipsoids.

macrocycles: Bottino & Pappalardo (1981), Pappalardo, Bottino & Tringali (1984), Pappalardo, Bottino, Tringali & Fronczek (1987); structure of a dimeric thiadiazole macrocycle and its polymeric  $Cu(NO_3)_2$  complex: Evans, Fronczek, Jarrett, Moorefield, Oliver, Watkins & Pappalardo (1987); structures of thiadiazole rings previously reported and refined to comparable precision: Mathew & Palenik (1974), Bats (1976), Kornis, Marks & Chidester (1980), Foresti, Riva di Sanseverino & Sabatino (1985).

#### References

BATS, J. W. (1976). *Acta Cryst.* B32, 2866–2870.

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## Structure of a 7-Oxa-2,8-diazatricyclo[3.2.2.0<sup>2,4</sup>]non-8-en-6-one Intermediate in a 4H-1,3-Diazepine Synthesis

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**Abstract.** 3-Isopropyl-9-methyl-4-phenyl-1-(trifluoromethyl)-7-oxa-2,8-diazatricyclo[3.2.2.0<sup>2,4</sup>]non-8-en-6-one (3),  $C_{17}H_{17}F_3N_2O_2$ ,  $M_r = 338.34$ , triclinic,  $P\bar{1}$ ,  $a = 13.031$  (4),  $b = 14.833$  (7),  $c = 10.304$  (4) Å,  $\alpha = 113.96$  (3),  $\beta = 103.37$  (3),  $\gamma = 100.40$  (3)°,  $V = 1684$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.334$  g cm<sup>-3</sup>,  $\lambda(Cu K\alpha) =$

- BOTTINO, F. & PAPPALARDO, S. (1981). *Org. Magn. Reson.* 16, 1–6.
- EVANS, D. W., FRONCZEK, F. R., JARRETT, W. L., MOOREFIELD, C. N., OLIVER, M. E., WATKINS, S. F. & PAPPALARDO, S. (1987). *J. Chem. Soc. Perkin Trans. 2*. Submitted.
- FORESTI, E., RIVA DI SANSEVERINO, L. & SABATINO, P. (1985). *Acta Cryst.* C41, 240–243.
- FRENZ, B. A. (1978). *The Enraf-Nonius CAD-4 Structure Determination Package – A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography*, edited by H. SCHENK, R. OLTHOFF-HAEZKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 64–71. Delft Univ. Press.
- GAJENDRAGAD, M. R. & AGARWALA, U. (1975). *Aust. J. Chem.* 28, 763–771, and references therein.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- JOHNSON, C. K. (1965). ORTEP. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- KORNIS, G., MARKS, P. J. & CHIDESTER, C. G. (1980). *J. Org. Chem.* 45, 4860–4863.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MATHEW, M. & PALENIK, G. J. (1974). *J. Chem. Soc. Perkin Trans. 2*, pp. 532–536.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO78*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- PAPPALARDO, S., BOTTINO, F. & TRINGALI, C. (1984). *Heterocycles*, 22, 1339–1342.
- PAPPALARDO, S., BOTTINO, F. & TRINGALI, C. (1987). *J. Org. Chem.* 52, 405–412.
- PAPPALARDO, S., BOTTINO, F., TRINGALI, C. & FRONCZEK, F. R. (1987). *J. Org. Chem.* 52, 3409–3413.
- SHELDRIK, G. M. (1976). *SHELX76*. A program for crystal structure determination. Univ. of Cambridge, England.

$1.54178$  Å,  $\mu = 9.70$  cm<sup>-1</sup>,  $F(000) = 704$ ,  $T = 295$  K,  $R = 0.057$  for 2949 observed reflections. There are two independent molecules in the unit cell and all chemically equivalent bonds are statistically equivalent except for those around C(4) and those involving the trifluoromethyl group. The molecules are held together by van der Waals interactions with the methyl and trifluoromethyl groups exhibiting large thermal motion.

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Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^3$ )

Molecule (I)	x	y	z	$U_{eq}^*$
C(1)	2813 (4)	3635 (3)	3891 (5)	65 (2)
N(2)	1995 (3)	2687 (3)	2578 (4)	60 (2)
C(3)	2317 (3)	1726 (3)	2129 (5)	56 (2)
C(4)	2485 (3)	2372 (3)	1359 (5)	52 (2)
C(5)	3656 (3)	3171 (3)	1920 (5)	54 (2)
C(6)	3417 (4)	4191 (3)	2304 (5)	69 (2)
O(7)	2915 (3)	4422 (2)	3380 (4)	74 (2)
N(8)	3876 (3)	3541 (3)	4467 (4)	64 (2)
C(9)	4309 (4)	3284 (3)	3422 (6)	62 (2)
C(10)	5384 (4)	3050 (4)	3609 (5)	81 (2)
C(11)	1788 (3)	2111 (3)	-206 (5)	54 (2)
C(12)	920 (4)	2501 (4)	-450 (6)	77 (2)
C(13)	339 (4)	2304 (5)	-1893 (6)	89 (3)
C(14)	606 (4)	1697 (4)	-3104 (6)	80 (2)
C(15)	1457 (4)	1307 (4)	-2865 (5)	78 (2)
C(16)	2058 (4)	1505 (3)	-1414 (5)	66 (2)
C(17)	1359 (3)	733 (3)	1405 (5)	64 (2)
C(18)	1727 (4)	-183 (4)	618 (6)	92 (3)
C(19)	940 (4)	651 (4)	2636 (7)	102 (3)
C(20)	2296 (5)	4015 (4)	5111 (6)	81 (3)
F(21)	1379 (3)	4232 (3)	4662 (4)	108 (2)
F(22)	2035 (2)	3290 (2)	5529 (3)	93 (2)
F(23)	2997 (3)	4859 (2)	6348 (3)	107 (2)
O(24)	3591 (3)	4768 (2)	1781 (4)	84 (2)

Molecule (II)	x	y	z	$U_{eq}^*$
C(1)	5334 (4)	1625 (4)	-1602 (6)	73 (2)
N(2)	6492 (3)	2038 (3)	-1453 (4)	73 (2)
C(3)	7021 (4)	3168 (3)	-574 (5)	67 (2)
C(4)	6511 (3)	2705 (3)	-2246 (5)	59 (2)
C(5)	5313 (4)	2674 (3)	-2929 (5)	60 (2)
C(6)	4673 (4)	1533 (4)	-3998 (6)	76 (2)
O(7)	4739 (2)	953 (2)	-3233 (4)	81 (2)
N(8)	4793 (3)	2391 (3)	-1011 (4)	67 (2)
C(9)	4771 (3)	2914 (3)	-1739 (5)	63 (2)
C(10)	4185 (4)	3708 (4)	-1492 (6)	82 (3)
C(11)	7167 (3)	2534 (4)	-3306 (5)	62 (2)
C(12)	7735 (4)	3371 (4)	-3400 (5)	71 (2)
C(13)	8284 (4)	3198 (5)	-4466 (7)	89 (3)
C(14)	8244 (5)	2220 (6)	-5396 (7)	94 (3)
C(15)	7676 (5)	1402 (5)	-5298 (6)	95 (3)
C(16)	7139 (4)	1548 (4)	-4249 (6)	80 (3)
C(17)	8268 (4)	3520 (4)	220 (6)	84 (3)
C(18)	8734 (5)	4659 (6)	760 (7)	123 (3)
C(19)	8498 (5)	3254 (5)	1526 (6)	109 (3)
C(20)	5233 (5)	876 (5)	-948 (8)	89 (3)
F(21)	5601 (3)	108 (2)	-1553 (4)	106 (2)
F(22)	4237 (3)	538 (3)	-975 (5)	138 (2)
F(23)	5881 (3)	1398 (3)	515 (4)	119 (2)
O(24)	4157 (3)	1120 (3)	-5328 (4)	97 (2)

\* Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

Table 2. Bond distances ( $\text{\AA}$ )

	Molecule (I)	Molecule (II)
C(1)–N(2)	1.480 (4)	1.469 (7)
C(1)–O(7)	1.461 (7)	1.475 (6)
C(1)–N(8)	1.431 (7)	1.444 (7)
C(1)–C(20)	1.518 (9)	1.517 (12)
N(2)–C(3)	1.481 (6)	1.472 (5)
N(2)–C(4)	1.485 (7)	1.518 (8)
C(3)–C(4)	1.489 (8)	1.492 (6)
C(3)–C(17)	1.516 (5)	1.521 (7)
C(4)–C(5)	1.565 (5)	1.541 (7)
C(4)–C(11)	1.505 (7)	1.508 (7)
C(5)–C(6)	1.511 (7)	1.525 (6)
C(5)–C(9)	1.509 (8)	1.505 (8)
C(6)–O(7)	1.372 (7)	1.384 (8)
C(6)–O(24)	1.198 (7)	1.204 (6)
N(8)–C(9)	1.284 (8)	1.279 (8)
C(9)–C(10)	1.492 (8)	1.485 (8)
C(11)–C(12)	1.378 (8)	1.378 (9)
C(11)–C(16)	1.374 (7)	1.376 (7)
C(12)–C(13)	1.384 (8)	1.409 (9)
C(13)–C(14)	1.375 (8)	1.359 (10)
C(14)–C(15)	1.359 (8)	1.356 (12)
C(15)–C(16)	1.398 (7)	1.385 (9)
C(17)–C(18)	1.501 (7)	1.498 (10)
C(17)–C(19)	1.531 (9)	1.531 (10)
C(20)–F(21)	1.323 (8)	1.294 (8)
C(20)–F(22)	1.330 (8)	1.294 (8)
C(20)–F(23)	1.334 (5)	1.346 (7)

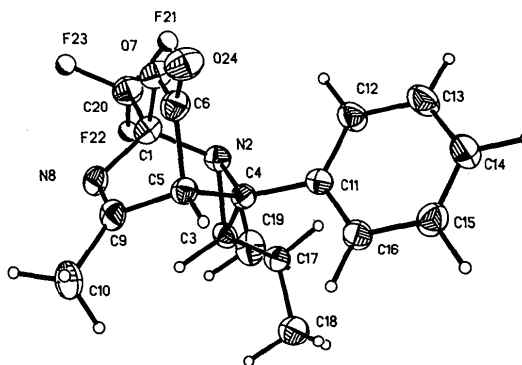
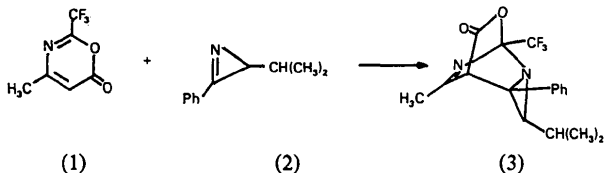


Fig. 1. Drawing of compound (3). Thermal ellipsoids are drawn at the 15% probability level. H and F atoms are shown as spheres of arbitrary size.

**Experimental.** Compound (3) is formed by the reaction of 4-methyl-2-(trifluoromethyl)-6*H*-1,3-oxazin-6-one (1) with 3-isopropyl-2-phenylazirine (2).



A transparent light-amber rectangular prism of dimensions  $0.50 \times 0.38 \times 0.28$  mm was mounted in a capillary; the crystal turns dark brown upon exposure to X-rays; Syntex  $P2_1$  diffractometer; data collected by  $\theta:2\theta$  technique ( $4 \leq 2\theta \leq 120^\circ$ ), variable scan rate, graphite-monochromated  $\text{Cu } K\alpha$  radiation; lattice

parameters from a least-squares refinement of 15 reflections ( $7.33 \leq 2\theta \leq 26.08^\circ$ ), angles measured by a centering routine, crystal deterioration prevented a more accurate determination of unit-cell dimensions and the collection of  $\psi$ -scan absorption-correction data; no systematic absences, Laue symmetry  $\bar{1}$ , and statistics consistent with space group  $P\bar{1}$ ; monitored reflection ( $2\bar{1}2$ ) showed about a 30% decrease in intensity during data collection; 4462 independent reflections measured ( $0 \leq h \leq 12$ ,  $-15 \leq k \leq 15$ ,  $-10 \leq l \leq 10$ ),  $2949 \geq 3\sigma(I)$ ; Lorentz-polarization corrections applied, no absorption correction; structure solved by direct methods, H atoms located in difference maps, but constrained at  $1.08 \text{ \AA}$  with fixed isotropic temperature factors, full-matrix anisotropic least-squares refinement gave  $R = 0.0571$ ,  $wR = 0.0601$  for 2949 reflections and 433 parameters,  $S = 1.91$ ,  $(\Delta/\sigma)_{\text{max}} = 0.37$ ,  $(\Delta/\sigma)_{\text{av}} = 0.08$ ; largest

Table 3. Bond angles ( $^{\circ}$ )

	Molecule (I)	Molecule (II)
N(2)C(1)O(7)	105.0 (4)	105.7 (4)
N(2)C(1)N(8)	115.7 (4)	115.6 (4)
N(2)C(1)C(20)	108.4 (4)	110.7 (5)
O(7)C(1)N(8)	110.8 (4)	109.8 (4)
O(7)C(1)C(20)	104.7 (4)	103.4 (4)
N(8)C(1)C(20)	111.6 (4)	110.9 (5)
C(1)N(2)C(3)	116.7 (4)	117.6 (4)
C(1)N(2)C(4)	107.6 (4)	107.5 (4)
C(3)N(2)C(4)	60.3 (3)	59.9 (3)
N(2)C(3)C(4)	60.0 (3)	61.6 (3)
N(2)C(3)C(17)	114.8 (3)	115.4 (3)
C(4)C(3)C(17)	121.9 (4)	122.4 (5)
N(2)C(4)C(3)	59.7 (3)	58.5 (3)
N(2)C(4)C(5)	109.0 (3)	108.6 (4)
N(2)C(4)C(11)	118.5 (3)	116.8 (4)
C(3)C(4)C(5)	118.4 (4)	119.3 (4)
C(3)C(4)C(11)	125.5 (3)	123.9 (4)
C(5)C(4)C(11)	113.0 (4)	114.6 (4)
C(4)C(5)C(6)	103.5 (3)	105.6 (4)
C(4)C(5)C(9)	107.7 (4)	109.4 (4)
C(6)C(5)C(9)	104.4 (3)	101.8 (4)
C(5)C(6)O(7)	111.6 (4)	110.6 (4)
C(5)C(6)O(24)	128.7 (5)	129.1 (6)
O(7)C(6)O(24)	119.7 (4)	120.2 (5)
C(1)O(7)C(6)	110.7 (4)	111.0 (4)
C(1)N(8)C(9)	109.2 (4)	108.5 (2)
C(5)C(9)N(8)	117.2 (5)	118.4 (4)
C(5)C(9)C(10)	119.5 (5)	119.6 (5)
N(8)C(9)C(10)	123.2 (5)	122.0 (5)
C(4)C(11)C(12)	121.7 (4)	119.2 (4)
C(4)C(11)C(16)	118.9 (4)	120.7 (5)
C(12)C(11)C(16)	119.4 (5)	120.0 (5)
C(1)C(12)C(13)	120.5 (5)	118.9 (5)
C(12)C(13)C(14)	120.3 (6)	120.5 (7)
C(13)C(14)C(15)	119.3 (5)	120.0 (7)
C(14)C(15)C(16)	121.2 (5)	120.9 (6)
C(1)C(16)C(15)	119.4 (5)	119.7 (6)
C(3)C(17)C(18)	110.1 (4)	109.7 (5)
C(3)C(17)C(19)	108.5 (3)	108.9 (5)
C(18)C(17)C(19)	111.2 (5)	111.6 (5)
C(1)C(20)F(21)	112.9 (5)	113.7 (7)
C(1)C(20)F(22)	109.8 (5)	112.9 (6)
C(1)C(20)F(23)	111.9 (5)	107.5 (5)

peaks in the final difference map of  $+0.29$  and  $-0.28 \text{ e } \text{\AA}^{-3}$ ,  $\sum w(|F_o| - |F_c|)^2$  minimized with  $w = 1/\sigma^2(F_o)$ ; IBM 4341 computer, locally written programs for data collection, *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) for

direct-methods calculations and *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) for all others; atomic scattering factors for C and O from Cromer & Mann (1968), those for H from Stewart, Davidson & Simpson (1965). Table 1 lists atomic positional parameters and  $U_{eq}$  values while Tables 2 and 3 give bond lengths and bond angles.\* Fig. 1 shows the atom numbering.

**Related literature.** The synthesis and chemistry of compound (3) is given in a paper by Steglich, Jeschke & Buschmann (1986). No crystal structure with a similar tricyclic skeleton could be found in the literature.

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\* Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44600 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

#### References

- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.  
 MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.  
 STEGLICH, W., JESCHKE, R. & BUSCHMANN, E. (1986). *Gazz. Chim. Ital.* **116**, 361–372.  
 STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. & FLACK, H. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.  
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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*Acta Cryst.* (1988). **C44**, 773

**$\mu$ -[Ethylenebis(dimethylphosphine)]-bis[tris(cyclopentadienyl)uranium(IV)]. Erratum.** By ALLAN ZALKIN, JOHN G. BRENNAN and RICHARD A. ANDERSEN, *Materials and Molecular Research Division, Lawrence Berkeley Laboratory and Department of Chemistry, University of California, Berkeley CA 94720, USA*

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#### Abstract

In the title to the paper by Zalkin, Brennan & Andersen [*Acta Cryst.* (1987). **C43**, 1706–1708] the oxidation state of

uranium was incorrectly stated as (IV) instead of (III).

All relevant information is given in the *Abstract*.