

Fig. 2. The asymmetric unit showing the base pairing.

parameters are given in Table 1,* bond lengths and angles are given in Table 2. Fig. 2 shows the base-paired asymmetric unit.

Related literature. The related compound 3',5'-di-*O*-acetylthymidine has been studied recently (Wilson, Low, Tollin & Wilson, 1984). The conformations of substituted 2'-deoxyuridines are discussed by Kálmán, Czugler & Simon (1982) and by Párkányi, Kálmán, Czugler, Kovács & Walker (1987). Conformational parameters for nucleosides are discussed by Saenger (1984).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44669 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 2,2',5,5'-Bis(tetramethylenedithio)di-1,3,4-thiadiazole

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Abstract. $C_{12}H_{16}N_4S_6$, $M_r = 408.67$, triclinic, $P\bar{1}$, $a = 5.731$ (1), $b = 8.246$ (1), $c = 9.913$ (1) Å, $\alpha = 100.23$ (1), $\beta = 103.77$ (1), $\gamma = 101.33$ (1)°, $V = 433.6$ (1) Å³, $Z = 1$, $D_x = 1.565$, $D_m =$

Note added in proof: After this paper had been accepted for publication it was noted that the structure had already been solved and published independently by Gurskaya, Tsapkina, Skaptsova, Kraevskil, Lindeman & Struchkov (1986), Birnbaum, Giziewicz, Gabe, Lin & Prusoff (1987) and Camerman, Mastropaolo & Camerman (1987).

References

- BIRNBAUM, G. I., GIZIEWICZ, J., GABE, E. J., LIN, T. & PRUSOFF, W. H. (1987). *Can. J. Chem.* **65**, 2135.
 CAMERMAN, A., MASTROPAOLO, D. & CAMERMAN, N. (1987). *Proc. Natl Acad. Sci. USA*, **84**, 8239–8242.
 GURSKAYA, G. V., TSAPKINA, E. N., SKAPTSOVA, N. V., KRAEVSKIL, A. A., LINDEMAN, S. V. & STRUCHKOV, YU. T. (1986). *Dokl. Akad. Nauk SSSR*, **291**, 854.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
 IUPAC–IUB JOINT COMMISSION ON BIOCHEMICAL NOMENCLATURE (1983). *Pure Appl. Chem.* **55**, 1273–1280.
 KÁLMÁN, A., CZUGLER, M. & SIMON, K. (1982). *Molecular Structure and Biological Activity*, edited by J. F. GRIFFIN & W. L. DUAX, pp. 367–376. New York: Elsevier.
 MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
 PÁRKÁNYI, L., KÁLMÁN, A., CZUGLER, M., KOVÁCS, T. & WALKER, R. T. (1987). *Nucleic Acids Res.* **15**, 4111–4121.
 ROBERTS, P. & SHELDRIK, G. M. (1975). *XANADU*. Program for torsion angle, mean plane and libration correction calculations. Univ. of Cambridge, England.
 SAENGER, W. (1984). In *Principles of Nucleic Acid Structure*. New York: Springer Verlag.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRIK, G. M. (1986). *SHELXS86*. Program for the automatic solution of crystal structures. Univ. of Göttingen, Federal Republic of Germany.
 WILSON, C. C., LOW, J. N., TOLLIN, P. & WILSON, H. R. (1984). *Acta Cryst.* **C40**, 1712–1715.
 WILSON, C. C. & TOLLIN, P. (1987). *Nucleosides Nucleotides*, **6**, 643–653.

1.59 (2) g cm⁻³, $\lambda(\text{Cu K}\alpha) = 1.54184$ Å, $\mu = 72.1$ cm⁻¹, $F(000) = 212$, $T = 296$ (1) K, 1787 unique reflections measured, final $R = 0.049$ for 1585 reflections having $F_o > 6.0\sigma(F_o)$. The centrosymmetric macrocyclic molecule consists of two 1,3,4-thiadiazole rings S-bonded at the 2 and 5 positions by two fully

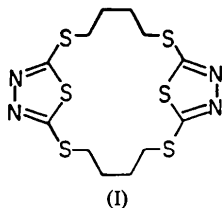
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Table 1. *Experimental details*

| | |
|---------------------------------------|--|
| Crystal | 0.08 × 0.12 × 0.60 mm |
| Instrument | Enraf-Nonius CAD-4 |
| Monochromator | Incident-beam graphite |
| Lattice constants | 25 reflections (25–30° θ) |
| Mode | ω–2θ |
| Scan rates | Variable (0.74–3.30° min ⁻¹) |
| Standards | 300, 030, 003 |
| Decay | None |
| Corrections | Lorentz–polarization Empirical absorption (0.749–0.994 on <i>I</i>) Extinction (3.1 × 10 ⁻⁶) |
| 2θ | 4–150° |
| <i>hkl</i> | <i>h</i> = 0 to 7, <i>k</i> = –10 to 10, <i>l</i> = –12 to 12 |
| Reflections | 1787 unique 1585 observed [<i>F</i> _o > 6σ(<i>F</i> _o)] |
| Solution | Direct methods |
| Function minimized: | $\sum w(F_o - F_c)^2$ |
| Weights | $4F_o^2 L_p^2 / [S^2(C + R^2B) + (0.02F_o^2)^2]$ <i>S</i> = scan rate, <i>C</i> = integrated count, <i>R</i> = scan time/background time, <i>B</i> = background count |
| Parameters refined | 133 |
| <i>R</i> , <i>wR</i> , <i>R</i> (all) | 0.049, 0.064, 0.054 |
| Goodness of fit | 3.28 |
| Maximum Δ/ <i>σ</i> | 0.01 |
| Δ <i>p</i> | 0.58 (10), –0.75 (10) e Å ⁻³ |

extended tetramethylenedithio chains. The five-membered rings are in the envelope conformation, with S 0.016 (1) Å out-of-plane. The best planes of the thiadiazole and macrocyclic rings form a dihedral angle of 41.4°. Attachment of the tetramethylenedithio chains to the heterocyclic rings forms S–C–S–C torsion angles of ±29.9 (2) and ±31.0 (2)°.

Experimental. Crystals of the title compound (I) were previously prepared and kindly supplied by Professor S. Pappalardo (Pappalardo, Bottino & Tringali, 1987). A colorless prismatic needle was mounted on a glass fiber in random orientation with epoxy. The density was measured by flotation in aqueous KI. Details of data collection and structural refinement are given in Table 1.



The structure was solved by direct methods. Ten of the eleven non-H atoms were located from an *E* map, and the remaining atom and all H atoms were located from difference Fourier maps. The structure was refined by full-matrix least squares, with H atoms refined isotropically. Calculated structure factors of all non-H atoms included anisotropic thermal parameters and anomalous dispersion (*International Tables for X-ray Crystallography*, 1974). All scattering factors from

Table 2. *Coordinates and equivalent isotropic thermal parameters*

| | <i>x</i> | <i>y</i> | <i>z</i> | <i>B</i> _{eq} * (Å ²) |
|----|------------|-------------|--------------|--|
| S1 | 0.8660 (1) | 0.3053 (1) | 0.03149 (8) | 3.27 (2) |
| S2 | 0.8267 (1) | 0.36214 (9) | 0.33834 (8) | 3.10 (1) |
| S3 | 0.5737 (1) | 0.2864 (1) | –0.27561 (8) | 3.33 (2) |
| N1 | 0.4039 (5) | 0.2927 (4) | –0.0471 (3) | 3.31 (6) |
| N2 | 0.4619 (5) | 0.3098 (4) | 0.0984 (3) | 3.32 (6) |
| C1 | 0.5933 (5) | 0.2891 (4) | –0.0971 (3) | 2.62 (5) |
| C2 | 0.6971 (5) | 0.3194 (4) | 0.1544 (3) | 2.54 (5) |
| C3 | 1.0922 (6) | 0.2706 (4) | 0.3483 (3) | 3.01 (6) |
| C4 | 1.0202 (6) | 0.0773 (4) | 0.3133 (3) | 3.17 (6) |
| C5 | 1.2458 (6) | 0.0049 (4) | 0.3116 (3) | 3.13 (6) |
| C6 | 1.1811 (6) | –0.1855 (4) | 0.3019 (3) | 2.99 (6) |

* The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation: $\frac{1}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + abB_{12}\cos\gamma + acB_{13}\cos\beta + bcB_{23}\cos\alpha]$.

Table 3. *Selected bond distances (Å), angles (°) and torsion angles (°)*

| | | | |
|-------------|------------|-------------|------------|
| S1 C1 | 1.731 (3) | N1 C1 | 1.299 (4) |
| S1 C2 | 1.730 (3) | N2 C2 | 1.311 (4) |
| S2 C2 | 1.739 (3) | C3 C4 | 1.519 (4) |
| S2 C3 | 1.817 (4) | C4 C5 | 1.529 (5) |
| S3 C1 | 1.743 (3) | C5 C6 | 1.521 (4) |
| N1 N2 | 1.375 (4) | | |
| C1 S1 C2 | 86.7 (1) | S1 C2 S2 | 124.0 (2) |
| C2 S2 C3 | 101.1 (1) | S1 C2 N2 | 114.1 (2) |
| N2 N1 C1 | 113.0 (2) | S2 C2 N2 | 121.6 (3) |
| N1 N2 C2 | 112.1 (3) | S2 C3 C4 | 112.9 (2) |
| S1 C1 S3 | 124.0 (2) | C3 C4 C5 | 111.3 (2) |
| S1 C1 N1 | 114.0 (2) | C4 C5 C6 | 110.9 (3) |
| S3 C1 N1 | 121.8 (2) | | |
| C1 N1 N2 | 0.27 (40) | N2 N1 C1 S1 | 0.37 (36) |
| C2 S1 C1 N1 | –0.66 (25) | N1 N2 C2 S1 | –0.78 (36) |
| C1 S1 C2 N2 | 0.81 (25) | | |

International Tables for X-ray Crystallography (1974). The final cycle of refinement included 133 variable parameters and converged to *R* = 0.049. Programs used were *MULTAN11/82* (Main, Fiske, Hull, Lesinger, Germain, Declercq & Woolfson, 1982), *SHELX76* (Sheldrick, 1976), *ORTEP* (Johnson, 1965), *PLUTO78* (Motherwell & Clegg, 1978), and *SDP* (VAX) (Frenz, 1978).

Final positional and equivalent isotropic thermal parameters are in Table 2, and selected bond lengths, bond angles and torsion angles are in Table 3.* Fig. 1 shows the molecule and the atomic numbering system.

Related literature. The 2,5-dimercapto-1,3,4-thiadiazole ligand as a chelate: Gajendragad & Agarwala (1975); preparation and structure of thiadiazole-containing

* Lists of structure factors, anisotropic thermal parameters, H-atom positions, bond lengths, bond angles, torsion angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44611 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

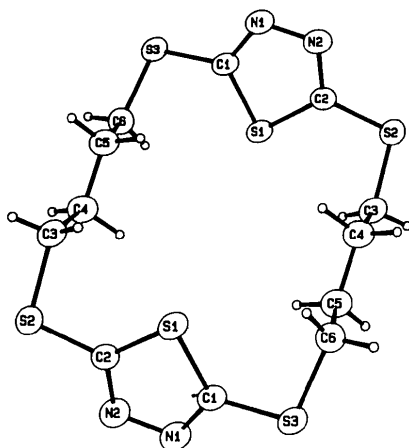


Fig. 1. ORTEP view of $C_{12}H_{16}N_4S_6$, 50% ellipsoids.

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^{2,4}]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^{2,4}]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) Å³, $Z = 4$, $D_x = 1.334$ g cm⁻³, $\lambda(Cu K\alpha) =$

- 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). *MULTAN*11/82. *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). *PLUTO*78. 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). *SHELX*76. A program for crystal structure determination. Univ. of Cambridge, England.

1.54178 Å, $\mu = 9.70$ cm⁻¹, $F(000) = 704$, $T = 295$ 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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