$C_{21}H_{32}N_2O_5$

Table 3. *Hydrogen-bond-like interactions* (Å) *with e.s.d.'s* $\simeq 0.03$ Å

O1(O-Leu; mol. I)$\cdots$N(Val; mol. II)(x,y,z)	2·98
O1(O-Leu; mol. I)$\cdots$O(Val; mol. II)(x,y,z)	2·68
N(Val; mol. I)$\cdots$O1(O-Leu; mol. II)(x,y,z)	3·09
O(Val; mol. I)$\cdots$O1(O-Leu; mol. II)(x,y,z)	2·73
O(O-Leu; mol. II)$\cdots$N(Phe; mol. I)(1+x,y,z)	2·78
N(Phe; mol. II)$\cdots$O(O-Leu; mol. I)(1+x,y,z)	2·93

The arrangement of the molecules in the crystal resembles the well known antiparallel β-sheet structure. In the present case, however, the hydrogen-bonding pattern is very different. As seen in Fig. 2, at the level of the O-Leu and Val residues, the two classic NH$\cdots$O hydrogen bonds are replaced by four hydrogen-bond-type interactions. The hydroxyl O1 atoms of both molecules I and II participate in two hydrogen interactions (Table 3). The first is of NH$\cdots$O1 type (2·98 and 3·09 Å) and the second is of O1H$\cdots$O type (2·68 and 2·73 Å).

References

BENEDETTI, E., MORELLI, G., NEMETHY, G. & SCHERAGA, H. A. (1983). *Int. J. Pept. Protein Res.* 22, 1–15.

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 72–73. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

CRUSE, W. B. T., EGERT, E., VISWAMITRA, M. A. & KENNARD, O. (1982). *Acta Cryst.* B38, 1758–1764.

DANN, J. G., STAMMERS, D. K., HARRIS, C. J., ARROWSMITH, R. J., DAVIES, D. E., HARDY, G. W. & MORTON, J. A. (1986). *Biochem. Biophys. Res. Commun.*, 134(1), 71–77.

GEOFFRE, S., LEROY, F. & PRECIGOUX, G. (1986). *Int. J. Pept. Protein Res.* 27, 454–460.

GILMORE, C. J. (1984). *J. Appl. Cryst.* 17, 42–46.

IUPAC–IUB COMMISSION ON BIOCHEMICAL NOMENCLATURE (1970). *J. Mol. Biol.* 52, 1–17.

JANIN, J., WODAK, S., LEVITT, M. & MAIGRET, B. (1978). *J. Mol. Biol.* 125, 357–386.

LALITHA, V., SUBRAMANIAN, E. & PARTHASARATHY, R. (1986). *Int. J. Pept. Protein Res.* 27, 223–228.

PRECIGOUX, G., COTRAIT, M. & GEOFFRE, S. (1986). *Acta Cryst.* C42, 315–317.

PRECIGOUX, G., COURSEILLE, C., GEOFFRE, S. & LEROY, F. (1987). *J. Am. Chem. Soc.* 109, 7463–7465.

SKEGGS, L. T., KAHN, J. R., LENTZ, K. E. & SHUMWAY, N. P. J. (1957). *J. Exp. Med.* 106, 439–453.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.

SZELKE, M., LECKIE, B., HALLETT, A., JONES, D. M., SUIERAS, J., ATRASH, B. & LEVER, A. F. (1982). *Nature (London)*, 299, 555–557.

TEWKSBURY, D. A., DART, R. A. & TRAVIS, J. (1981). *J. Biochem. Biophys. Res. Commun.* 99, 1311–1315.

TREE, M., BROWN, J. J., LECKIE, B. J., LEVER, A. F., MANHEM, P., MORTON, J. J., ROBERTSON, J. I. S., SZELKE, M. & WEBB, D. (1984). *Biochem. Soc. Trans.* 12, 948–951.

Structure of 1-Mesitylsulfonyl-4-nitroimidazole

By ZENEI TAIRA AND JUNKOU UCHI

Faculty of Pharmaceutical Sciences, Tokushima Bunri University, Yamashiro-cho, Tokushima 770, Japan

Abstract. $C_{12}H_{13}N_3O_4S$, $M_r = 295\cdot31$, triclinic, $P\bar{1}$, $a = 15\cdot841$ (6), $b = 10\cdot774$ (5), $c = 4\cdot782$ (1) Å, $\alpha = 99\cdot26$ (3), $\beta = 79\cdot43$ (3), $\gamma = 59\cdot62$ (3)°, $V = 660\cdot4$ (5) Å^3, $Z = 2$, $D_x = 1\cdot485$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu(\text{Mo } K\alpha) = 0\cdot2634$ mm^{-1}, $F(000) = 308$, room temperature, final $R = 0\cdot034$ for 2174 unique observed reflections. The molecule, except for the nitro group, has a geometry which is nearly symmetrical with respect to the plane through the atoms C(3)–S(1)–N(13); the dihedral angle between the least-squares planes of the benzene and imidazole rings is 100·8°. The benzene ring and the sulfonyl group are strained as a result of steric hindrance.

Introduction. The present study is part of a series of structural studies on compounds available for the synthesis of oligodeoxyribonucleotides by the phosphotriester approach (Itakura, Katagiri, Bahl, Wightman & Narang, 1973). We report here the structure of the title compound (MSNI) which is a condensing reagent for this approach. Although arylsulfonates of imidazoles, triazoles and tetrazoles are used for such condensing reagents, an X-ray structure analysis has only been reported for 1-(mesitylsulfonyl)-3-nitro-1,2,4-triazole (MSNT) (Kuroda, Sanderson, Neidle & Reese, 1982). We are interested in elucidating the geometry around the sulfonyl group of MSNI.

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å²) *of the non-H atoms*

E.s.d.'s in parentheses are in the units of the last significant digit.

$$B_{eq} = \tfrac{4}{3}\sum_i\sum_j\beta_{ij}(a_i.a_j).$$

|	x	y	z	B_{eq}
S(1)	0·69759 (4)	0·9604 (1)	0·7624 (1)	4·77
C(2)	0·7011 (2)	1·0887 (2)	1·0316 (5)	5·08
C(3)	0·6102 (2)	1·2028 (3)	1·2601 (5)	5·56
C(4)	0·6178 (2)	1·2987 (3)	1·4703 (6)	5·78
C(5)	0·7096 (2)	1·2862 (3)	1·4612 (6)	7·70
C(6)	0·7969 (2)	1·1734 (3)	1·2332 (6)	7·82
C(7)	0·7961 (2)	1·0720 (3)	1·0161 (5)	6·32
C(8)	0·5045 (2)	1·2306 (3)	1·3026 (6)	5·78
C(9)	0·7150 (3)	1·3919 (3)	1·6929 (6)	9·70
C(10)	0·8993 (2)	0·9506 (3)	0·7875 (6)	6·53
O(11)	0·5948 (1)	1·0138 (2)	0·7667 (4)	6·77
O(12)	0·7755 (1)	0·9042 (2)	0·4879 (3)	6·66
N(13)	0·7339 (1)	0·8075 (2)	0·8906 (4)	4·50
C(14)	0·6715 (2)	0·7903 (3)	1·1003 (5)	5·09
N(15)	0·7227 (2)	0·6633 (2)	1·1587 (4)	5·83
C(16)	0·8208 (2)	0·5978 (2)	0·9793 (5)	4·55
C(17)	0·8315 (2)	0·6812 (3)	0·8114 (5)	4·59
N(18)	0·9031 (2)	0·4516 (2)	0·9752 (5)	6·88
O(19)	0·8857 (2)	0·3961 (2)	1·1602 (6)	15·23
O(20)	0·9859 (2)	0·3938 (2)	0·7873 (5)	8·12
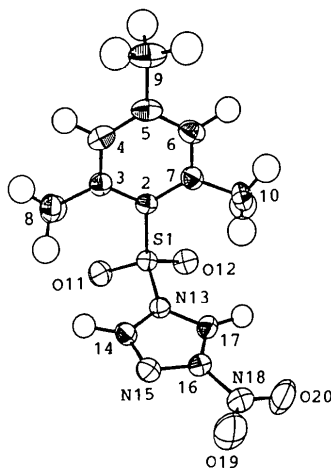
Fig. 1. *ORTEP* (Johnson, 1965) drawing of MSNI with the numbering scheme.

Experimental. MSNI was prepared by the reaction of mesitylenesulfonyl chloride and nitroimidazole (van Boom, Burgers, van der Marel, Verdegaal & Wille, 1977) and recrystallized by the evaporation of a benzene solution at room temperature. Crystal size: 0·3 × 0·3 × 0·3 mm. Cell parameters were determined from least-squares method for 25 reflections having 15° < 2θ < 25°; Rigaku four-circle AFC 6B diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. A total of 2513 reflections with $2\theta_{max}$ 50°, $0 \le h \le 11$, $-10 \le k \le 12$, $-5 \le l \le 5$, were collected with the ω-scan technique, scan speed 2° min⁻¹. Three reference reflections showed no significant change in intensities, 0·996 < $|F|/|F|_{initial}$ < 1·003. 2174 observed reflections ($F > 3\sigma_F$) were corrected for Lorentz–

Table 2. *Intramolecular bond lengths* (Å), *bond angles* (°) *and some torsion angles* (°)

S(1)–C(2)	1·761 (3)	S(1)–O(11)	1·421 (2)
S(1)–O(12)	1·417 (2)	S(1)–N(13)	1·708 (3)
C(2)–C(3)	1·407 (3)	C(2)–C(7)	1·408 (5)
C(3)–C(4)	1·389 (5)	C(3)–C(8)	1·511 (5)
C(4)–C(5)	1·383 (5)	C(5)–C(6)	1·378 (3)
C(5)–C(9)	1·503 (5)	C(6)–C(7)	1·388 (5)
C(7)–C(10)	1·517 (4)	N(13)–C(14)	1·378 (4)
N(13)–C(17)	1·370 (3)	C(14)–N(15)	1·301 (4)
N(15)–C(16)	1·362 (4)	C(16)–C(17)	1·348 (4)
C(16)–N(18)	1·444 (3)	N(18)–O(19)	1·210 (4)
N(18)–O(20)	1·215 (4)		

C(2)–S(1)–O(11)	111·0 (1)	C(2)–S(1)–O(12)	111·6 (1)
C(2)–S(1)–N(13)	104·5 (1)	O(11)–S(1)–O(12)	120·0 (1)
O(11)–S(1)–N(13)	103·8 (1)	O(12)–S(1)–N(13)	104·1 (1)
S(1)–C(2)–C(3)	119·7 (3)	S(1)–C(2)–C(7)	118·8 (2)
C(3)–C(2)–C(7)	121·5 (3)	C(2)–C(3)–C(4)	117·3 (3)
C(2)–C(3)–C(8)	126·4 (3)	C(4)–C(3)–C(8)	116·3 (2)
C(3)–C(4)–C(5)	122·8 (2)	C(4)–C(5)–C(6)	118·2 (3)
C(4)–C(5)–C(9)	121·4 (2)	C(6)–C(5)–C(9)	120·4 (3)
C(5)–C(6)–C(7)	122·6 (3)	C(2)–C(7)–C(6)	117·6 (2)
C(2)–C(7)–C(10)	126·2 (3)	C(6)–C(7)–C(10)	116·2 (3)
S(1)–N(13)–C(14)	126·5 (1)	S(1)–N(13)–C(17)	126·2 (2)
C(14)–N(13)–C(17)	107·2 (2)	N(13)–C(14)–N(15)	111·9 (2)
C(14)–N(15)–C(16)	103·6 (3)	N(15)–C(16)–C(17)	113·8 (2)
N(15)–C(16)–N(18)	121·3 (3)	C(17)–C(16)–N(18)	124·9 (2)
N(13)–C(17)–C(16)	103·5 (2)	C(16)–N(18)–O(19)	118·2 (2)
C(16)–N(18)–O(20)	117·3 (3)	O(19)–N(18)–O(20)	124·5 (2)

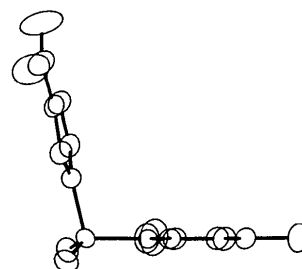|	
O(11)–S(1)–C(2)–C(3)	15·5 (3)
O(11)–S(1)–C(2)–C(7)	−166·0 (2)
O(12)–S(1)–C(2)–C(3)	152·2 (2)
O(12)–S(1)–C(2)–C(7)	−29·2 (2)
N(13)–S(1)–C(2)–C(3)	−95·9 (2)
N(13)–S(1)–C(2)–C(7)	82·7 (2)
C(2)–S(1)–N(13)–C(14)	82·8 (3)
C(2)–S(1)–N(13)–C(17)	−94·7 (2)
O(11)–S(1)–N(13)–C(14)	−33·7 (3)
O(11)–S(1)–N(13)–C(17)	148·8 (2)
O(12)–S(1)–N(13)–C(14)	−160·1 (2)
O(12)–S(1)–N(13)–C(17)	22·4 (3)

Fig. 2. A view showing the orientation of the benzene and imidazole rings.

polarization, but not for absorption. The structure was solved by direct methods (Main, Woolfson, Lessinger, Germain & Declercq, 1974), and atomic parameters were refined anisotropically by block-diagonal least squares, minimizing $\sum w(|F_o| - |F_c|)^2$; $w = 1\cdot0$. All H atoms were located on difference Fourier maps and refined isotropically. The final R value was 0·034 ($wR = 0\cdot037$, $S = 0\cdot516$). $(\Delta/\sigma)_{max} = 0\cdot45$, $(\Delta\rho)_{max} = |0\cdot41|$ e Å⁻³. Atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974).

All calculations were performed by application of the program packages: *RASA* (Rigaku Co., 1980), *X-STANP* (Taira, 1980), *MULTAN* (Main, Woolfson, Lessinger, Germain, & Declercq, 1974) and *ORTEP* (Johnson, 1965), on a PANAFACOM U1400 minicomputer, an NEC PC9801m2 personal computer at Tokushima Bunri University, and a FACOM-M360 computer at the Computer Center of the University of Tokushima.

Discussion. The fractional atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* The bond distances, bond angles and torsion angles are listed in Table 2. The molecular structure with the numbering scheme is given in Fig. 1. As shown in Fig. 2 and by the torsion angles around the bonds C(2)—S(1) and S(1)—N(13) in Table 2, the benzene and imidazole rings face each other, the dihedral angle between the least-squares planes of the rings being $100.8°$. The molecule, except for the nitro group, has a geometry which is nearly symmetrical with respect to the plane through the atoms C(3)—S(1)—N(13), and the bond lengths and angles are distorted by the steric hindrance between the groups overcrowded around the sulfonyl group. In particular the methyl groups at the

---

* 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 51137 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

ortho positions are bent away from the sulfonyl group, similar to the situation found for MSNT (Kuroda, Sanderson, Neidle & Reese, 1982). Although the length of the bond S(1)—C(2) is close to that of MSNT, 1.758 Å, S(1)—N(13) is significantly shorter than the 1.736 Å of MSNT, which is close to the upper limit reported for the S—N bond, $1.616–1.736$ Å (Kuroda, Sanderson, Neidle & Reese, 1982). Such a difference in bond length may be caused by the different electronic effects of the imidazole and triazole rings.

References

Boom, J. H. van, Burgers, P. M. J., van der Marel, G., Verdegaal, C. H. M. & Wille, G. (1977). *Nucleic Acids Res.* 4(4), 1047–1063.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Itakura, K., Katagiri, N., Bahl, C. P., Wightman, R. H. & Narang, S. A. (1973). *Can. J. Chem.* 51, 3649–3651.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Kuroda, R., Sanderson, M. R., Neidle, S. & Reese, C. B. (1982). *J. Chem. Soc. Perkin Trans.* 2, pp. 617–620.
Main, P., Woolfson, M. M., Lessinger, L., Germain, G. & Declercq, J.-P. (1974). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Rigaku Co. (1980). *RASA. Rigaku Automatic Structure Analysis Programs.* Tokyo, Japan.
Taira, Z. (1980). *X-STANP. X-ray Structure Analysis Programs for a PANAFACOM U1400 Minicomputer and an NEC PC9801 Personal Computer.* Tokushima Bunri Univ., Japan.

Structure of Bis[2,6-bis(trifluoromethyl)phenyl]diphosphene

By A. Dubourg

Laboratoire de Physique Industrielle Pharmaceutique, Faculté de Pharmacie, avenue Charles Flahaut, 34060 Montpellier, France

J.-P. Declercq

Laboratoire de Chimie Physique et de Cristallographie, Université Catholique de Louvain, place Pasteur 1, B-1348 Louvain-la-Neuve, Belgium

And H. Ranaivonjatovo, J. Escudié, C. Couret and M. Lazraq

Laboratoire de Chimie des Organominéraux, UA 477 du CNRS, Université P. Sabatier, 118 route de Narbonne, 31062 Toulouse CEDEX, France

Abstract. $C_{16}H_6F_{12}P_2$, $M_r = 488.15$, monoclinic, $P2_1/c$, $a = 4.937$ (3), $b = 15.994$ (5), $c = 11.492$ (4) Å, $\beta = 93.27$ (3)°, $V = 906.0$ (7) Å^3, $Z = 2$, $D_x = 1.79$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.39$ mm^{-1}, $F(000) = 480$, $T = 295$ K, final $R = 0.053$ for 1415 observed reflections. The title compound