

Structure of *N*-(2-Hydroxyethyl)-2-thiofuramide

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**Abstract.**  $C_7H_9NO_2S$ ,  $M_r = 171.21$ , monoclinic,  $P2_1/c$ ,  $a = 8.346$  (2),  $b = 12.329$  (4),  $c = 8.568$  (3) Å,  $\beta = 110.87$  (4)°,  $V = 823.8$  (5) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.37$ ,  $D_x = 1.380$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 3.26$  cm<sup>-1</sup>,  $F(000) = 360$ ,  $T = 293$  K, final  $R = 0.043$  for 1217 observed reflections. In accord with a common feature of 2- or 5-furyl bonds the exocyclic angles at the furan C(2) atom are different, C–C–C 132.8 (3) being larger than C–C–O 117.6 (2)°. The non-H atoms of the furan ring and thioamide group together with the hydroxyethyl C atom attached to it deviate within  $\pm 0.018$  (2) Å from their mean plane. The N–C( $sp^2$ ), N–C( $sp^3$ ) and C=S bond lengths are 1.317 (4), 1.460 (3) and 1.673 (3) Å, respectively. The molecules are connected by N–H...O(H) and O–H...S hydrogen bonds of 2.846 (4) and 3.255 (3) Å, respectively.

**Introduction.** It is well known that furans exhibit pharmacological activity and many of them are in use as pharmaceuticals (Martorana, Share & Zimmerman, 1985; Hammarlund, Odland & Paalzow, 1985), herbicides (Melhus, 1935), fungicides (Koschucharoff & Harisanowa, 1962), bactericides (Ascoli, Casini, Ferappi & Tubaro, 1967), etc.

As part of our work on furan chemistry, we synthesized some furan derivatives (Vlahov, 1976) and tested them under *in vitro* as well as *in vivo* experimental conditions on mice of various strains (Vlahov & Volf, 1983). The structure of one such potentially pharmacologically active compound is presented.

**Experimental.** The compound was obtained by hydrolysis of *N*-(2-acetoxyethyl)-2-thiofuramide with 1M NaOH and characterized using elemental microanalysis and spectroscopic methods (Vlahov, 1976, 1982). Crystals suitable for X-ray examination were obtained by recrystallization from benzene.  $D_m$  by flotation in a mixture of CCl<sub>4</sub> and petroleum ether.

Intensity data collected from a crystal of dimensions 0.10 × 0.33 × 0.45 mm on a Philips PW 1100 four-circle diffractometer,  $\theta$ – $2\theta$  scanning technique, scan width 1.80°, scan rate 0.06° s<sup>-1</sup>. Unit-cell parameters obtained from least-squares analysis of 20 reflections with  $2\theta$  values ranging from 13 to 20°. By absent reflections  $h0l$ ,  $l \neq 2n$  and  $0k0$ ,  $k \neq 2n$ , space group  $P2_1/c$  was determined. Out of 1511 reflections scanned within a quadrant  $\pm h, k, l$  ( $h$  10, –11;  $k$  17;  $l$  11) up to  $\sin\theta/\lambda \leq 0.70$  Å<sup>-1</sup>, 1410 unique reflections classified as observed. Three standard reflections ( $\bar{1}41$ ,  $40\bar{2}$ ,  $\bar{3}10$ ) measured every 2 h showed an averaged variation of 1%. Corrections applied for Lorentz and polarization effects. No correction for absorption or extinction. Structure solved by direct methods (*MULTAN80*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The  $E$  map calculated with 244 largest  $E$  values ( $E \geq 1.33$ ) revealed the positions of all non-H atoms except one furan C atom. Subsequent calculations performed mainly with *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). The remaining C atom and all H atoms located from difference maps. In final calculations four strong low-order reflections (100, 200,  $\bar{1}21$ , 031) strongly affected by extinction were rejected. Full-matrix least-squares refinement on  $F$  for 1217 remaining reflections with  $I > 2.5\sigma(I)$  and anisotropic thermal parameters for non-H and isotropic for H atoms (136 parameters refined) gave  $R = 0.043$  and  $wR = 0.052$ ; function minimized  $\sum w(|F_o| - |F_c|)^2$  with  $w = 1.00/\sigma^2(F_o)$  giving  $S = 1.51$ ; parameter shifts were  $< 0.1$ . Final difference map revealed residuals between  $-0.25$  and  $0.21$  e Å<sup>-3</sup>. Scattering factors of Cromer & Mann (1968) for non-H and those of Stewart, Davidson & Simpson (1965) for H atoms; anomalous-dispersion factors from Cromer & Liberman (1970). All calculations performed on a Univac 1110 computer at the University Computing Centre in Zagreb.

**Discussion.** The final atomic parameters are listed in Table 1\* and bond lengths and angles in Table 2. Fig. 1 shows a pair of the molecules connected by N—H...O(H) hydrogen bonds and the atom numbering.

\* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond lengths and angles involving H atoms and mean planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43393 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional positional parameters ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\times 10^2$ ) for non-H atoms

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
S	2902 (1)	1497 (1)	3855 (1)	7.15 (4)
O(1)	7688 (2)	1010 (2)	4156 (2)	5.64 (8)
O(2)	3121 (3)	869 (2)	-1237 (3)	6.51 (9)
N	4546 (3)	348 (2)	2288 (3)	4.75 (8)
C(1)	4617 (3)	1048 (2)	3472 (3)	4.53 (9)
C(2)	6326 (3)	1422 (2)	4495 (3)	4.53 (9)
C(3)	6894 (5)	2117 (3)	5786 (4)	6.4 (1)
C(4)	8697 (5)	2144 (3)	6286 (5)	7.6 (2)
C(5)	9122 (4)	1473 (3)	5298 (4)	6.8 (1)
C(6)	2974 (4)	-128 (3)	1118 (4)	6.2 (1)
C(7)	2055 (4)	617 (3)	-319 (4)	6.5 (1)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) of non-H atoms

S—C(1)	1.673 (3)	C(1)—C(2)	1.458 (3)
O(1)—C(2)	1.368 (4)	C(2)—C(3)	1.345 (4)
O(1)—C(5)	1.372 (3)	C(3)—C(4)	1.411 (6)
O(2)—C(7)	1.416 (5)	C(4)—C(5)	1.319 (6)
N—C(1)	1.317 (4)	C(6)—C(7)	1.508 (5)
N—C(6)	1.460 (3)		
C(2)—O(1)—C(5)	105.9 (2)	C(1)—C(2)—C(3)	132.8 (3)
C(1)—N—C(6)	125.1 (3)	C(2)—C(3)—C(4)	106.9 (3)
S—C(1)—N	124.2 (2)	C(3)—C(4)—C(5)	107.0 (3)
S—C(1)—C(2)	119.9 (2)	O(1)—C(5)—C(4)	110.6 (3)
N—C(1)—C(2)	115.9 (3)	N—C(6)—C(7)	112.8 (3)
O(1)—C(2)—C(1)	117.6 (2)	O(2)—C(7)—C(6)	110.4 (3)
O(1)—C(2)—C(3)	109.6 (2)		

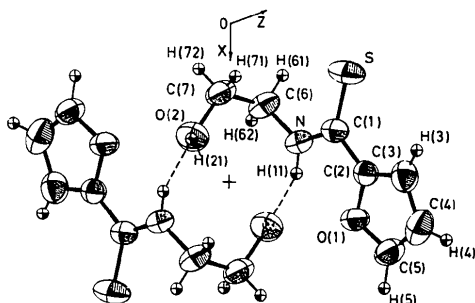


Fig. 1. An ORTEP diagram (Johnson, 1965) showing a pair of the mutually centrosymmetric hydrogen-bonded molecules viewed along **b** and the atom numbering. The symmetry centre at  $\frac{1}{2}, 0, 0$  is denoted by +.

Table 3. Pairs of exocyclic  $R'-C-C$  and  $R'-C-O$  angles ( $^\circ$ ) with their differences  $\Delta$  ( $^\circ$ ) for furan and some selected derivatives ( $R'$  denotes the atom of a substituent directly attached to the ring at positions 2 or/and 5)

	$R'-C-C$	$R'-C-O$	$\Delta$	$R'$	Reference
(a)	134 (2)	115 (2)	19	H	Fourme (1972)
(b)	133.37 (3)	115.98 (3)	17.39	H	Mata, Martin & Sørensen (1978)
(c)	131.0 (4)	118.7 (4)	12.3	C	Carpy, Leger & Nuhrich (1978)
	130.7 (4)	116.6 (4)	14.1	N	
(d)	137.7 (4)	112.2 (4)	25.5	C	Eilingsfeld, Patsch & Hädicke (1980)
	134.3 (5)	115.2 (5)	19.1	N	
(e)	136.5 (2)	113.8 (1)	22.7	C	Meunier & Pascard (1980)
	134.2 (2)	115.6 (2)	18.6	C	
	132.6 (2)	117.3 (2)	15.3	C	
(f)	134.9 (5)	117.1 (4)	17.8	Hg	Sikirica, Grdenić & Cimaš (1982)
(g)	135.3 (2)	114.7 (1)	20.6	C	Jaulmes, Cassanas & Laruelle (1982)
(h)	131.9 (1)	117.6 (1)	14.3	C	Gilmore, Mallinson & Speakman (1983)
(i)	132.3 (5)	116.8 (5)	15.1	C	Fodor, Sussangkarn, Mathelier, Arnold, Karle & George (1984)
(j)	132.8 (3)	117.6 (2)	15.2	C	This work
	138 (2)	112 (2)	26	H	

(a) Furan (X-ray determination of the tetragonal crystal form at 123 K); (b) furan (microwave spectra determination at room temperature); (c) 1-phenyl-3-(5-nitro-2-furyl)-2-propen-1-one; (d) 5,5'-diamino-4-cyano-3,3'-dimethyl-2',3'-dihydro-2,3'-bifuryl-4'-thiocarbamide; (e) 2,4,5-tri(3-methoxycarbonylfuran-2-yl)- $\Delta^2$ -imidazole; (f) di(2-furyl)mercury; (g) potassium 2,5-furandi-carboxylate; (h) 2-furoic acid; (i) 2-(5-methyl-2-furyl)-3-keto-L-gulonolactone 3,6-hemiketal.

Among the features common to the geometry of unsubstituted or substituted furan rings there are two which stand out. First, the C atoms are linked by two lateral bonds of practically double-bond length and by a longer bond opposite to the O atom which has partial double-bond character. In the title compound the relevant bond lengths are 1.319 (6), 1.345 (4) and 1.411 (6)  $\text{\AA}$ . The other feature is related to the exocyclic angles at the C atoms at positions 2 and 5: the  $R'-C-O$  angles are always considerably less than  $R'-C-C$  angles, where  $R'$  denotes the atom of a substituent  $R$  which is directly attached to the ring. It is difficult to discern any clear correlation between different substituents  $R$  and the magnitude of these angles (Table 3). Leiserowitz & Rabinovich (1969) assumed that the difference in angle is due to asymmetrical release of the  $R'\cdots O$  and  $R'\cdots C(3)/C(4)$  repulsions. Sikirica, Grdenić & Cimaš (1982) consider the similar difference in di(2-furyl)mercury to be appropriate to the 2-furyl bond but not as a consequence of an  $Hg\cdots O$  attraction. That steric reasons are not predominantly responsible for this feature is indicated particularly by its appearing in the crystal structure (a tetragonal form at 123 K) of unsubstituted furan (Fourme, 1972). Although the H atoms involved are determined with lower accuracy, the difference of

19° between the angles [115 (2) and 134 (2)°] is reliable enough and could be compared with the difference between H(5)—C(5)—O(1) and H(5)—C(5)—C(4) of 112 (2) and 138 (2)° respectively, in this compound. A determination on the basis of microwave spectra measurements at room temperature gave angles of 115.98 (3) and 133.37 (3)° (Mata, Martin & Sørensen, 1978).

The partially double C(sp<sup>2</sup>)—O bond lengths are 1.368 (4) and 1.372 (3) Å. The furan ring is planar within ±0.003 (4) Å. The thioamide group with the furan C(2) atom attached to it is planar within ±0.004 (3) Å. Nevertheless, it is indicated that the group is slightly pyramidal because C(1) is 0.006 (3) Å above the corresponding basal plane. Most of the molecule does not deviate appreciably from planarity because the non-H atoms of the furan ring, thioamide group and the C(6) atom deviate from their common mean plane within ±0.018 (2) Å. This enables a conjugation in the system involving also the N lone electron pair. It results in the very short C(1)—N of 1.317 (4) Å and rather short C(1)—C(2) bond length of 1.458 (3) Å. Also, the C(1)=S bond length of 1.673 (3) Å compared with the values of 1.685 (5) and 1.690 (5) Å reported for 4-(2'-hydroxythiobenzoyl) morpholine (Sarkar & Sen Gupta, 1980) and the C(sp<sup>3</sup>)—N bond length of 1.460 (3) Å seem to be slightly shortened.

The perfect synclinal N—C(6)—C(7)—O(2) torsion angle of 60.1 (4)° shows an unstrained atom arrangement of this part of the molecule. The corresponding N...O(2) contact of 2.897 (3) Å is obviously not a hydrogen bond (Table 4).

The N—C(1)—C(2)—O(1) torsion angle of -0.8 (4)° is in accord with the planarity mentioned above. The corresponding N...O(1) contact of 2.667 (3) Å might be considered as an intramolecular hydrogen bond in spite of the small N—H(11)...O(1) angle of 108 (2)°. Its geometry (Table 4) is in agreement with the observations of Taylor, Kennard & Versichel (1984), given for N—H...O=C intramolecular hydrogen bonds. They found that their H...O distances tend to be longer, the N...O distances shorter and consequently the N—H...O angles smaller than in intermolecular hydrogen bonds. So the H(11) atom would be involved in a 'bifurcated' hydrogen bond [three-centre hydrogen bond: Jeffrey & Maluszynska (1982)], because the neighbouring molecules related by the symmetry centres at  $\frac{1}{2}, 0, 0$  and  $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$  are self-paired by mutual N—H...O(H) hydrogen bonds of 2.846 (4) Å showing an angle at the H atom of 152 (2)° (Fig. 1, Table 4). The angle of elevation, defined as the angle between the plane containing the lone-pair orbitals of the O atom and the H...O vector (Taylor & Kennard, 1982) is rather large (47.1°) for the H(11)...O(1) intramolecular contact and favourably small (4.0°) for the H(11)...O(2<sup>i</sup>) intermolecular contact [instead of the

Table 4. *Geometry of the intermolecular hydrogen bonds and the intramolecular N(H)...O(H) contacts*

X	H	Y	X...Y (Å)	X—H (Å)	H...Y (Å)	X—H...Y (°)
N	H(11)	O(2 <sup>i</sup> )	2.846 (4)	0.87 (3)	2.05 (3)	152 (2)
O(2)	H(21)	S <sup>ii</sup>	3.255 (3)	0.76 (4)	2.50 (3)	168 (4)
N	H(11)	O(1)	2.667 (3)	0.87 (3)	2.26 (2)	108 (2)
N	H(11)	O(2)	2.897 (3)	0.87 (3)	3.04 (2)	72 (2)
O(2)	H(21)	N		0.76 (4)	3.19 (3)	61 (2)

Symmetry code: (i)  $-x+1, -y, -z$ ; (ii)  $x, -y+\frac{1}{2}, z-\frac{1}{2}$ .

elevation angle, its complement  $\theta_H$  is afterwards introduced as a spherical polar coordinate (Taylor, Kennard & Versichel, 1983)].

The pairs of hydrogen-bonded molecules are connected in layers parallel to (100) by O—H...S hydrogen bonds of 3.255 (3) Å (Table 4). This distance is in accord with the distances reported for such hydrogen bonds (Srinivasan & Chandrasekharan, 1968; Sarkar & Sen Gupta, 1980; Ravichandran & Chacko, 1985).

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#### References

- ASCOLI, F., CASINI, G., FERAPPI, M. & TUBARO, E. (1967). *J. Med. Chem.* **10**, 97–99.
- CARPY, A., LEGER, J. M. & NUHRICH, A. (1978). *Cryst. Struct. Commun.* **7**, 187–190.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- EILINGSFELD, H., PATSCH, M. & HÄDICKE, E. (1980). *Ann. Chem. Liebigs*, pp. 1952–1959.
- FODOR, G., SUSSANGKARN, K., MATHÉLIER, H., ARNOLD, R., KARLE, I. & GEORGE, C. (1984). *J. Org. Chem.* **49**, 5064–5069.
- FOURME, R. (1972). *Acta Cryst.* **B28**, 2984–2991.
- GILMORE, C. J., MALLINSON, P. R. & SPEAKMAN, J. C. (1983). *Acta Cryst.* **C39**, 1111–1113.
- HAMMARLUND, M. M., ODLIND, B. & PAALZOW, L. K. (1985). *J. Pharmacol. Exp. Ther.* **233**, 447–453.
- JAULMES, S., CASSANAS, G. & LARUELLE, P. (1982). *Acta Cryst.* **B38**, 279–281.
- JEFFREY, G. A. & MALUSZYNSKA, H. (1982). *Int. J. Biol. Macromol.* **4**, 173–185.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- KOSCHUCHAROFF, P. & HARISANOWA, T. (1962). *Pharmazie*, **17**, 134–137.
- LEISEROWITZ, L. & RABINOVICH, D. (1969). *J. Chem. Soc. A*, pp. 2367–2371.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MARTORANA, P. A., SHARE, N. N. & ZIMMERMAN, M. (1985). *Eur. J. Respir. Dis.* **66**, 297–301.

- MATA, F., MARTIN, M. C. & SØRENSEN, G. O. (1978). *J. Mol. Struct.* **48**, 157–163.
- MELHUS, I. E. (1935). US Patent No. 2007433.
- MEUNIER, B. & PASCARD, C. (1980). *Cryst. Struct. Commun.* **9**, 121–128.
- RAVICHANDRAN, V. & CHACKO, K. K. (1985). *Acta Cryst.* **C41**, 1470–1473.
- SARKAR, P. B. & SEN GUPTA, S. P. (1980). *Cryst. Struct. Commun.* **9**, 925–932.
- SIKIRICA, M., GRDENIĆ, D. & CIMAŠ, Š. (1982). *Acta Cryst.* **B38**, 926–927.
- SRINIVASAN, R. & CHANDRASEKHARAN, R. (1986). *Acta Cryst.* **B24**, 1698–1700.
- 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.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- TAYLOR, R. & KENNARD, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.
- TAYLOR, R., KENNARD, O. & VERSICHEL, W. (1983). *J. Am. Chem. Soc.* **105**, 5761–5766.
- TAYLOR, R., KENNARD, O. & VERSICHEL, W. (1984). *Acta Cryst.* **B40**, 280–288.
- VLAHOV, A. (1976). *Synthesis of Some N-Hydroxyalkyl- and N-Chloroalkylamides Derived from Thiobenzoic and 2-Furanthiocarboxylic Acids*. MSc Thesis, Univ. of Zagreb.
- VLAHOV, A. (1982). *The Effect of Newly Synthesized Derivatives of 2-Furancarboxylic Acid on Functional Capacity of Lymphohematopoietic Cells*. PhD Thesis, Univ. of Zagreb.
- VLAHOV, A. & VOLF, D. (1983). *Topics in Furan Chemistry*. Proc. Fourth Symp. Furan Chem., edited by J. KOVAC, pp. 237–239. Bratislava.

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## Structure of 2,2',4,4',5,5'-Hexamethoxybiphenyl

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**Abstract.** C<sub>18</sub>H<sub>22</sub>O<sub>6</sub>,  $M_r = 334.4$ , monoclinic,  $C2/c$ ,  $a = 24.801$  (3),  $b = 5.3654$  (5),  $c = 15.428$  (2) Å,  $\beta = 126.18$  (1)°,  $V = 1657.1$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.340$ ,  $D_m = 1.320$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu(\text{Cu } K\alpha) = 0.79$  mm<sup>-1</sup>,  $F(000) = 712$ ,  $T = 293$  K,  $R = 0.053$  for 1168 observed reflections. The crystallographic diad coincides with the molecular diad with an inter-ring twist angle of 81.2 (1)°. The methoxy groups are coplanar with the phenyl ring and the crystal structure is stabilized by van der Waals interactions.

**Introduction.** Structure analyses of the biphenyl molecule in different states have revealed that interesting conformational changes in the molecule take place on crystallization. The inter-ring twist angle is 42° in the gas phase (Bastiansen & Trætteberg, 1962) whereas X-ray diffraction analysis (Trotter, 1961; Hargreaves & Rizvi, 1962; Charbonneau & Delugeard, 1976, 1977) has shown the molecule to be planar. We are interested in studying the molecular geometry and packing arrangements of substituted biphenyls. The

present work reports the crystal structure analysis of 2,2',4,4',5,5'-hexamethoxybiphenyl.

**Experimental.** Crystals from ethanol by slow evaporation; density measured by flotation in benzene–bromofrom mixture, crystal size 0.28 × 0.38 × 0.15 mm; intensity data collected on rotating-anode Rigaku AFC-5 four-circle diffractometer at 40 kV, 200 mA with Ni-filtered Cu  $K\alpha$  radiation; lattice parameters refined by the least-squares fit of the setting angles of 19 reflections in the range  $17 < 2\theta < 34^\circ$ ; 1324 independent reflections collected with  $2\theta_{\text{max}} = 125.0^\circ$  ( $h = -28 \rightarrow 28$ ,  $k = 0 \rightarrow 6$ ,  $l = 0 \rightarrow 17$ ); scan speed  $4^\circ \text{ min}^{-1}$  in  $\omega$ , scan range  $(1.2^\circ + 0.15^\circ \tan\theta)$ , background measured for 4 s on either side of the peak; three standard reflections (600,  $\bar{1}11$ ,  $11\bar{2}$ ) recorded after every 57 reflections to ensure stability of the crystal quality and position; variation in  $I_o$  within 0.8%; data corrected for  $L_p$  but not for absorption; intensity statistics and successful refinement confirmed space group  $C2/c$ , structure solved by direct methods (*MULTAN78*;