those incorporated in *SHELX76*. The final atomic parameters are given in Table 1.* Bond lengths and angles are given in Table 2. A view of the molecule with atomic numbering is shown in Fig. 1.

Related literature. Bonati & Bovio (1990) have recently compiled the known data on pyrazoles and derived a set of empirical rules for the bond angles about the ring and its substituents. Our results agree completely with their rules.

The authors acknowledge support from the Auckland Division of the Cancer Society of New Zealand and from the Health Research Council of New Zealand.

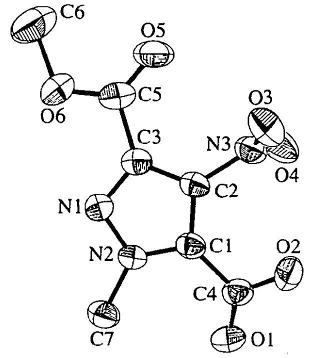

Fig. 1. *ORTEP* (Johnson, 1965) plot and atomic numbering scheme of the title compound.

References

Bonati, F. & Bovio, B. (1990). *J. Crystallogr. Spectrosc. Res.* **20**, 233–244.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Sheldrick, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71059 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1001]

Acta Cryst. (1993). C**49**, 1430–1432

Structure of 5,5′-Dimethylsilanediyldi(phthalic anhydride):* a Model System for Aromatic Polyimides

By Joseph W. Ziller

Department of Chemistry, University of California, Irvine, California 92717, *USA*

and Peter M. Schaber† and Frank J. Dinan

Department of Chemistry, Canisius College, Buffalo, New York 14208, *USA*

(*Received* 18 *May* 1992; *accepted* 10 *May* 1993)

Abstract. 5,5′-Dimethylsilanediylbis(1,3-isobenzofurandione), $C_{18}H_{12}O_6Si$, $M_r = 352.4$, triclinic, $P\bar{1}$, $a = 9.0405\,(16)$, $b = 9.1893\,(15)$, $c = 10.6043\,(19)$ Å, $\alpha = 93.285\,(14)$, $\beta = 106.558\,(13)$, $\gamma = 101.730\,(13)°$, $V = 820.5\,(2)$ Å^3, $Z = 2$, $D_m = 1.43$, $D_x = 1.43$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.710730 Å, $\mu = 0.167$ mm^{-1}, $F(000) = 364$, $T = 296\,(1)$ K, $R = 0.045$ for 2779 observed reflections with $|F_o| > 0$. This monomeric molecule is structurally similar to the repeat unit present in 'high peformance' polyimides. Of particular importance in terms of π-transfer complex formation is the overlap between the five- and six-membered rings. The separation between different molecules is 3.51 Å for the ring system defined by C(11)—C(12)—C(13)—C(14)—O(4)—C(15)—C(16)—C(17)—C(18) (perpendicular distance between least-squares ring planes). The closest contact between atoms is 2.45 Å which is the interatomic separation between H(18) and O(2) at the equipoint (x, $1 + y$, z).

Experimental. Colorless triclinic crystals of the title compound were grown from saturated acetonitrile solution *via* vapor diffusion of diethyl ether. D_m was determined by neutral buoyancy in a benzene/CCl_4 solution. Melting point was obtained using a Fisher–Jones Melting Point Apparatus (stage technique); m.p. 450–451 K. A crystal of approximate dimensions 0.27 × 0.50 × 0.58 mm was secured with epoxy to a glass fiber, mounted and aligned on a Syntex

---

* Alternative name: bis(3,4-dicarboxyphenyl)dimethylsilane dianhydride (SiDA).

† Author to whom correspondence should be addressed.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^4$) *with e.s.d.'s in parentheses*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Si(1)	2807 (1)	6725 (1)	8224 (1)	423 (2)
C(1)	4696 (3)	7710 (3)	9472 (2)	582 (9)
C(2)	2009 (3)	4851 (3)	8650 (3)	624 (10)
C(3)	1285 (2)	7900 (2)	8086 (2)	428 (6)
C(4)	1276 (2)	9094 (2)	7318 (2)	477 (7)
C(5)	165 (2)	9935 (2)	7278 (2)	497 (7)
C(6)	− 123 (3)	11233 (2)	6558 (3)	625 (9)
C(7)	− 1901 (3)	10759 (3)	7729 (3)	710 (10)
C(8)	− 932 (2)	9630 (2)	7964 (2)	535 (7)
C(9)	− 969 (3)	8455 (3)	8714 (2)	600 (9)
C(10)	142 (2)	7610 (3)	8759 (2)	524 (8)
C(11)	3127 (2)	6494 (2)	6548 (2)	405 (6)
C(12)	4293 (2)	7519 (2)	6214 (2)	436 (7)
C(13)	4522 (2)	7255 (2)	4997 (2)	434 (6)
C(14)	5655 (2)	8125 (2)	4396 (2)	530 (8)
C(15)	4237 (3)	6058 (3)	2940 (2)	576 (9)
C(16)	3649 (2)	6015 (2)	4108 (2)	459 (7)
C(17)	2485 (3)	4982 (2)	4395 (2)	527 (7)
C(18)	2242 (2)	5244 (2)	5613 (2)	488 (7)
O(1)	− 1333 (2)	11724 (2)	6922 (2)	760 (7)
O(2)	473 (2)	11853 (2)	5816 (2)	832 (8)
O(3)	− 2987 (2)	10923 (2)	8110 (2)	998 (10)
O(4)	5449 (2)	7339 (2)	3171 (1)	606 (6)
O(5)	6600 (2)	9281 (2)	4783 (2)	716 (7)
O(6)	3840 (2)	5210 (2)	1943 (2)	828 (8)

Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Si(1)—C(1)	1.853 (2)	Si(1)—C(2)	1.860 (3)
Si(1)—C(3)	1.894 (2)	Si(1)—C(11)	1.888 (2)
C(3)—C(4)	1.403 (3)	C(3)—C(10)	1.407 (3)
C(4)—C(5)	1.380 (3)	C(5)—C(6)	1.480 (3)
C(5)—C(8)	1.382 (3)	C(6)—O(1)	1.404 (3)
C(6)—O(2)	1.183 (3)	C(7)—C(8)	1.476 (4)
C(7)—O(1)	1.391 (3)	C(7)—O(3)	1.195 (4)
C(8)—C(9)	1.378 (3)	C(9)—C(10)	1.381 (4)
C(11)—C(12)	1.404 (3)	C(11)—C(18)	1.409 (2)
C(12)—C(13)	1.381 (3)	C(13)—C(14)	1.480 (3)
C(13)—C(16)	1.377 (2)	C(14)—O(4)	1.395 (3)
C(14)—O(5)	1.187 (2)	C(15)—C(16)	1.480 (3)
C(15)—O(4)	1.391 (3)	C(15)—O(6)	1.195 (3)
C(16)—C(17)	1.382 (3)	C(17)—C(18)	1.386 (3)
C(1)—Si(1)—C(2)	112.2 (1)	C(1)—Si(1)—C(3)	109.5 (1)
C(2)—Si(1)—C(3)	109.1 (1)	C(1)—Si(1)—C(11)	109.4 (1)
C(2)—Si(1)—C(11)	109.1 (1)	C(3)—Si(1)—C(11)	107.3 (1)
Si(1)—C(3)—C(4)	120.9 (2)	Si(1)—C(3)—C(10)	121.0 (2)
C(4)—C(3)—C(10)	118.1 (2)	C(3)—C(4)—C(5)	118.4 (2)
C(4)—C(5)—C(6)	130.1 (2)	C(4)—C(5)—C(8)	122.0 (2)
C(6)—C(5)—C(8)	107.9 (2)	C(5)—C(6)—O(1)	106.8 (2)
C(5)—C(6)—O(2)	132.2 (2)	O(1)—C(6)—O(2)	121.0 (2)
C(8)—C(7)—O(1)	107.5 (2)	C(8)—C(7)—O(3)	131.1 (3)
O(1)—C(7)—O(3)	121.4 (3)	C(5)—C(8)—C(7)	107.7 (2)
C(5)—C(8)—C(9)	121.1 (2)	C(7)—C(8)—C(9)	131.2 (2)
C(8)—C(9)—C(10)	117.1 (2)	C(3)—C(10)—C(9)	123.2 (2)
Si(1)—C(11)—C(12)	121.4 (1)	Si(1)—C(11)—C(18)	120.7 (1)
C(12)—C(11)—C(18)	117.9 (2)	C(11)—C(12)—C(13)	118.8 (1)
C(12)—C(13)—C(14)	130.3 (2)	C(12)—C(13)—C(16)	122.0 (2)
C(14)—C(13)—C(16)	107.7 (2)	C(13)—C(14)—O(4)	107.4 (2)
C(13)—C(14)—O(5)	131.3 (2)	O(4)—C(14)—O(5)	121.3 (2)
C(16)—C(15)—O(4)	107.6 (2)	C(16)—C(15)—O(6)	130.8 (2)
O(4)—C(15)—O(6)	121.6 (2)	C(13)—C(16)—C(15)	107.6 (2)
C(13)—C(16)—C(17)	121.1 (2)	C(15)—C(16)—C(17)	131.2 (2)
C(16)—C(17)—C(18)	117.2 (2)	C(11)—C(18)—C(17)	123.0 (2)
C(6)—O(1)—C(7)	109.8 (2)	C(14)—O(4)—C(15)	109.6 (2)

$P2_1$ automated four-circle diffractometer (graphite-monochromated Mo $K\alpha$ radiation). Laue symmetry, unit-cell parameters and the orientation matrix were determined by previously described techniques (Churchill, Lashewycz & Rotella, 1977). A total of 3104 reflections, all corrected for Lorentz and polarization effects and placed on an approximate scale, were collected at room temperature (296 K) using θ–2θ scans for 2θ from 4.0 to 50.0° ($0 \leq h \leq 10$, $-10 \leq k < 10$, $-12 \leq l \leq -12$), a constant scan speed of 2.00° $\min^{-1}$ (in ω), and a scan range (ω) of 1.20° plus $K\alpha$ separation. Background measurements were estimated from a 96-step profile. The intensity of three standards (700, 272, 027) measured after every 97 reflections varied by less than 5%; no absorption correction was necessary. All crystallographic calculations were carried out using a modified version of the *UCLA Crystallographic Computing Package* (Strouse, 1981) or the *SHELXTL-Plus* program set (Sheldrick, 1988). Analytical scattering factors for neutral atoms were used throughout the analysis (*International Tables for X-ray Crystallography*, 1974, Vol. IV); both real (f') and imaginary (if'') components of anomalous dispersion were included. The quantity minimized during least-squares analysis was $\sum w(|F_o| - |F_c|)^2$ with $w^{-1} = \sigma^2(|F_o|) + 0.0008(|F_o|)^2$. An extinction correction was applied; $\chi = 0.0009$ (6), where $F^* = F[1 + 0.002\chi F^2/\sin(2\theta)]^{-1/4}$.

The structure was solved by direct methods (*SHELXTL-Plus*; Sheldrick, 1988); all non-H atoms were located from a single E map. The 12 H atoms were located from a difference Fourier synthesis following isotropic refinement of the model. $R_{\text{int}} = 0.049$ for merging equivalent reflections. Full-matrix least-squares refinement of positional and anisotropic thermal parameters (C, O, Si) led to convergence with $R = 0.045$, $wR = 0.059$ and $S = 1.55$ for 275 variables refined against all 2779 data with $|F_o| > 0$ [$R = 0.038$, $wR = 0.056$ for those 2406 data with $|F_o| > 6.0\sigma(|F_o|)$]. A final difference Fourier synthesis showed no significant features. Residual electron density was within −0.21 to 0.34 e $Å^{-3}$. Maximum Δ/σ was 0.002 (mean < 0.001). Table 1* lists the atomic coordinates and equivalent isotropic displacement coefficients and Table 2 gives the interatomic distances and angles. Fig. 1 is an *ORTEP* (Johnson, 1965) plot showing the atom-labeling scheme and 50% probability thermal ellipsoids. Fig. 2 gives a stereoview of the unit-cell packing.

---

* Lists of anisotropic displacement coefficients, H-atom parameters and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71136 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1023]

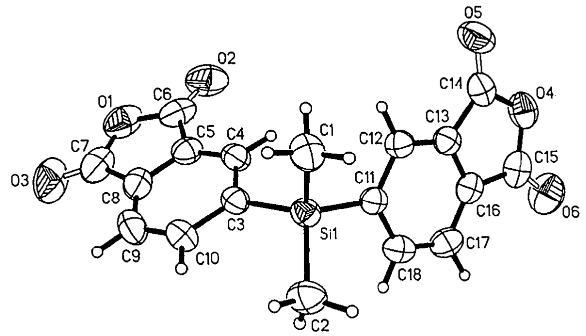

Fig. 1. *ORTEP* plot of the title compound showing the atom-labeling scheme and 50% probability thermal ellipsoids.

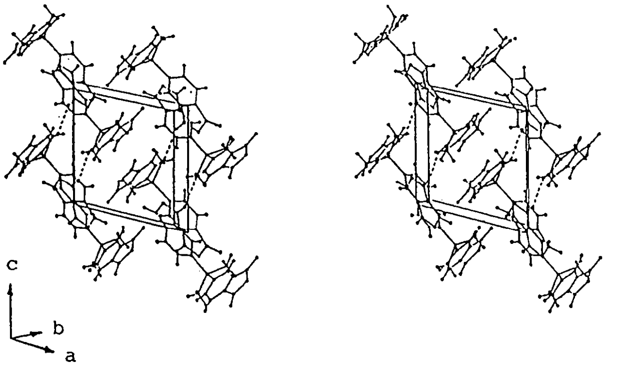

Fig. 2. Stereoview of the unit-cell packing of the title compound.

Related literature. The demand for materials which possess high thermal stability coupled with exceptional physical properties and, especially, outstanding electrical resistance has greatly increased in recent times. Aromatic polyimides are a class of compounds which possess these characteristics and have been extensively studied by Mittal (1984) and Bessonov, Kolton, Kudryavtsev & Louis (1987). Spectroscopic investigation of polyimides is, at best, difficult. Thus, the study of monomeric molecules, which are structurally similar to the repeat unit present in polyimides, has been undertaken. Solid-state ^{13}C NMR data, analyzed by Dinan, Schwartz, Wolfe, Pratt & St Clair (1989) and Dinan, Wolfe, Schwartz, Pratt & St Clair (1990), indicate that alignment of monomeric species occurs in a manner which is consistent with strong electrical interactions between electron-rich and electron-deficient portions of these molecules.

The authors wish to thank Dr J. K. Pratt, NASA Langley Research Center, for kindly supplying the title compound used in this study. Funds for the purchase of the Nicolet *R*3*m*/*V* diffractometer system were made available by the National Science Foundation under Grant CHE-85-14495 (UC Irvine).

References

Bessonov, M. I., Kolton, M. M., Kudryavtsev, V. V. & Louis, L. A. (1987). *Polyimides. Thermally Stable Polymers.* New York: Consultants Bureau.

Churchill, M. R., Lashewycz, R. A. & Rotella, F. J. (1977). *Inorg. Chem.* **16**, 265–271.

Dinan, F. J., Schwartz, W. T., Wolfe, R. A., Pratt, J. R. & St Clair, T. (1989). *The First Pacific Polymer Conference*, edited by B. C. Anderson, pp. 281–282. Pacific Polymer Reprints.

Dinan, F. J., Wolfe, R. A., Schwartz, W. T., Pratt, J. R. & St Clair, T. (1990). Presented at *The Interdisciplinary Symposium on Recent Advances in Polyimide and Other High Performance Polymers*, San Diego, CA.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Mittal, K. L. (1984). *Polyimides*, Vols. I and II, edited by K. L. Mittal. New York: Plenum.

Sheldrick, G. M. (1988). *SHELXTL-Plus Structure Determination Software Programs.* Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Strouse, C. (1981). *UCLA Crystallographic Computing Package.* Univ. of California, Los Angeles, USA.

Acta Cryst. (1993). C**49**, 1432–1434

Structure of Ethyl 2-Cyano-3-(3-hydroxy-4-methoxyphenyl)propenoate

By He Youping, Shi Jianqiu and Su Genbo

Fujian Institute of Research on the Structure of Matter, Academia Sinica, Fuzhou 350002,
People's Republic of China

(*Received* 3 *December* 1992; *accepted* 8 *April* 1993)

Abstract. $C_{13}H_{13}NO_4$, $M_r = 247.25$, monoclinic, $P2_1/n$, $a = 9.755$ (5), $b = 12.849$ (9), $c = 10.420$ (8) Å, $\beta = 104.16$ (4)°, $V = 1266$ (2) Å^3, $Z = 4$, $D_x = 1.30$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.91$ cm^{-1}, $F(000) = 520$, $T = 296$ K, $R = 0.052$ for 1690 observed unique reflections with $I > 3\sigma(I)$. The molecules are linked by O—H···N hydrogen bonds [O···N($\frac{1}{2} - x$, $\frac{1}{2} + y$, $\frac{3}{2} - z$) 2.88 Å, O—H···N 159°].

Acta Crystallographica Section C
ISSN 0108-2701 ©1993