Table 2. *Selected geometric parameters (Å, °)*

O(1)—C(11)	1.250 (5)	C(3)—C(4)	1.421 (5)
O(2)—C(11)	1.254 (5)	C(4)—C(10)	1.446 (5)
O(4)—C(4)	1.261 (4)	C(5)—C(6)	1.370 (6)
O(8)—C(8)	1.356 (5)	C(5)—C(10)	1.410 (5)
N(1)—C(2)	1.337 (5)	C(6)—C(7)	1.393 (6)
N(1)—C(9)	1.370 (4)	C(7)—C(8)	1.382 (6)
C(2)—C(3)	1.356 (5)	C(8)—C(9)	1.405 (5)
C(2)—C(11)	1.517 (5)	C(9)—C(10)	1.403 (5)
C(2)—N(1)—C(9)	121.9 (3)	O(8)—C(8)—C(9)	117.0 (3)
N(1)—C(2)—C(3)	121.3 (3)	C(7)—C(8)—C(9)	118.5 (4)
N(1)—C(2)—C(11)	114.9 (3)	N(1)—C(9)—C(8)	119.0 (3)
C(3)—C(2)—C(11)	123.8 (3)	N(1)—C(9)—C(10)	119.6 (3)
C(2)—C(3)—C(4)	121.5 (3)	C(8)—C(9)—C(10)	121.5 (3)
O(4)—C(4)—C(3)	122.6 (4)	C(4)—C(10)—C(5)	122.4 (3)
O(4)—C(4)—C(10)	121.2 (3)	C(4)—C(10)—C(9)	119.5 (3)
C(3)—C(4)—C(10)	116.2 (3)	C(5)—C(10)—C(9)	118.0 (3)
C(6)—C(5)—C(10)	120.4 (4)	O(1)—C(11)—O(2)	127.0 (4)
C(5)—C(6)—C(7)	120.8 (4)	O(1)—C(11)—C(2)	115.7 (4)
C(6)—C(7)—C(8)	120.7 (4)	O(2)—C(11)—C(2)	117.2 (3)
O(8)—C(8)—C(7)	124.5 (4)		

The H atoms bonded to the water O atom could not be located on a difference Fourier map. The relatively large *R* value of 0.072 may be a result of the crystal being extremely thin.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELX*86 (Sheldrick, 1985) and *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1218). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors.* Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kotake, Y., Sotokawa, T., Murakami, E., Hisatake, A., Abe, M. & Ikeda, Y. (1968). *J. Biochem.* **63**, 578–581.

Martin, D. W., Mayes, P. A. & Rodwell, V. W. (1983). *Harper's Review of Biochemistry*, pp. 464–479. Tokyo: Maruzen Asia.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Murakami, E. (1968). *J. Biochem.* **63**, 573–577.

Seifert, J. & Pewnim, T. (1992). *Biochem. Pharmacol.* **44**, 2243–2250.

Sheldrick, G. M. (1985). *SHELXS86. Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.

Acta Cryst. (1996). C**52**, 665–667

3,4-Diphenylpyrrole-2,5-dicarboxylic Acid Acetic Acid Disolvate

PAULA PRAYZNER,[a] EMMANUEL C. A. OJADI,[a] JAMES A. GOLEN[a] AND PAUL G. WILLIARD[b]

[a]*Department of Chemistry, University of Massachusetts Dartmouth, North Dartmouth, MA 02747, USA, and*
[b]*Department of Chemistry, Brown University, Providence, RI 02912, USA*

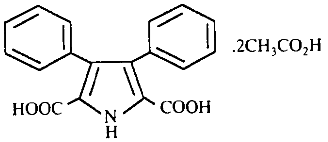
(*Received 30 August 1994; accepted 25 July 1995*)

Abstract

3,4-Diphenylpyrrole-2,5-dicarboxylic acid crystallizes as the acetic acid disolvate, $C_{18}H_{13}NO_4.2CH_3CO_2H$, and exhibits extensive hydrogen bonding involving the carboxylic acid functional groups, the amine group and the acetic acid moieties. The phenyl rings were found to lie at an angle of nearly 50° with respect to the planar pyrrole ring.

Comment

As part of our investigations of substituted porphyrins, the title compound, (I) (Friedman, 1965), was synthesized.



(I)

Analysis of the structural information shows the phenyl rings (Fig. 1) to have torsion angles C(1)—C(2)—C(7)—C(8) and C(4)—C(3)—C(13)—C(18) of 52.8 (9) and 57.0 (8)°, respectively, with respect to the the planar pyrrole ring, rather than being perpendicular to it (Medforth, Senge, Smith, Sparks & Shelnut, 1992).

The carboxylic acid functionality of the substituted pyrrole ring and the acetic acid solvate molecules participate in extensive hydrogen bonding (Fig. 2). The hydrogen-bonding distances between the pyrrole dicarboxylic acid groups and the acetic acid molecules are O(3^{i})···H—O(8) 2.626 (6), O(4)—H···O(7^{i}) 2.837 (6) [symmetry code: (i) $1-x$, $2-y$, $1-z$] and O(2)—H···O(5) 2.630 (6) Å, while the O(6)—H···O(7) distance between the acetic acid molecules is 2.686 (7) Å. Hydrogen bonding between the amine H atom and carboxy O atom of an adjacent pyrrole molecule [N(1)—H···O(1^{i}) 2.969 (7) Å] results in a dimer-like structure having a center of symmetry between the N(1) and N(1^{i}) atoms. The O—H···O and N—H···O hydrogen-bond distances are normal (Huheey, 1983). Other bond distances are compatible with those reported in the literature for related pyrroles (Viossat, Nguyen-Huy, Lehuede, Vierfond, 1994; Boukhari, Eddaif, Laurent, Mison & Faure, 1989).

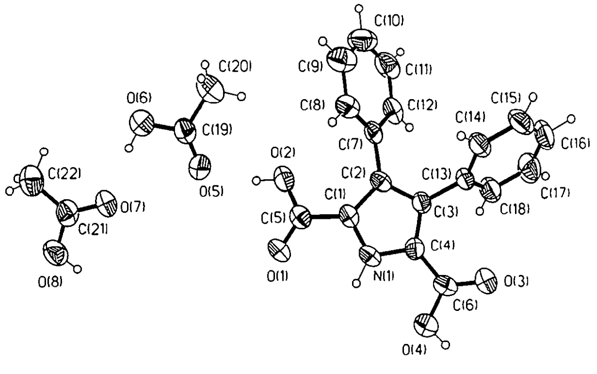


Fig. 1. View of $C_{18}H_{13}NO_4.2CH_3CO_2H$ showing the labeling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

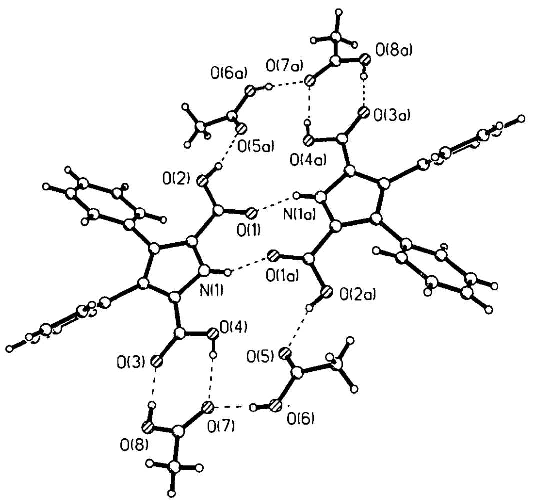


Fig. 2. View of the dimer-like structure formed by extensive hydrogen bonding. The dotted lines indicate the intermolecular hydrogen bonding.

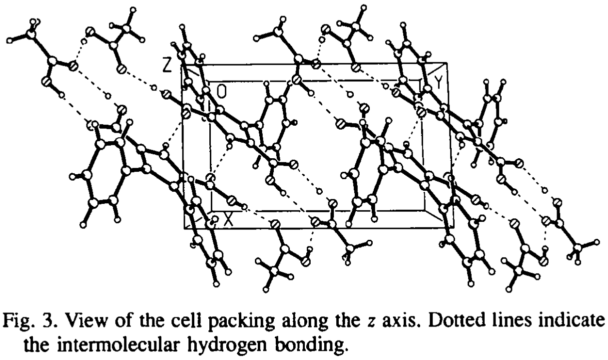


Fig. 3. View of the cell packing along the z axis. Dotted lines indicate the intermolecular hydrogen bonding.

Experimental

The synthesis of the title compound was carried out by reacting N-acetyliminodiacetate dimethyl ester with sodium methoxide in dry methanol solution, followed by the addition of benzil and then hydrolysis of the pyrrole ester. The product was recrystallized from glacial acetic acid as a disolvate (Prayzner, 1994). The crystal used for data collection was mounted with epoxy on a glass fiber and coated with epoxy to avoid loss of solvent.

Crystal data

$C_{18}H_{13}NO_4.2C_2H_4O_2$
$M_r = 427.4$
Triclinic
$P\bar{1}$
$a = 6.4630\,(10)$ Å
$b = 10.665\,(2)$ Å
$c = 15.623\,(3)$ Å
$\alpha = 87.07\,(3)°$
$\beta = 82.13\,(3)°$
$\gamma = 88.24\,(3)°$
$V = 1065.0\,(3)$ Å^3
$Z = 2$
$D_x = 1.333$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12–12.5°$
$\mu = 0.102$ mm^{-1}
$T = 297$ K
Wedge
0.44 × 0.22 × 0.22 mm
Colorless

Data collection

Siemens $R3m/V$ diffractometer
ω scans
Absorption correction: none
3667 measured reflections
2778 independent reflections
1406 observed reflections [$F > 4\sigma(F)$]

$R_{\rm int} = 0.0283$
$\theta_{\max} = 22.5°$
$h = -1 \rightarrow 6$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$
3 standard reflections monitored every 97 reflections
intensity decay: 7.2%

Refinement

Refinement on F
$R = 0.0578$
$wR = 0.0575$
$S = 1.32$
1406 reflections
280 parameters
H atoms refined as riding with fixed isotropic U

$w = 1/[\sigma^2(F) + 0.0005F^2]$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N(1)	0.4419 (8)	1.1262 (4)	0.3724 (3)	0.045 (2)
O(1)	0.2780 (7)	0.9499 (4)	0.4975 (3)	0.065 (2)
O(2)	0.1318 (7)	0.8547 (4)	0.3977 (2)	0.065 (2)
O(3)	0.6397 (7)	1.3980 (4)	0.2476 (3)	0.069 (2)
O(4)	0.7444 (7)	1.2961 (4)	0.3640 (3)	0.065 (2)
O(5)	−0.0391 (8)	0.6808 (4)	0.5083 (3)	0.070 (2)
O(6)	−0.3037 (7)	0.5574 (4)	0.5562 (3)	0.074 (2)
O(7)	−0.0419 (7)	0.5118 (4)	0.6743 (3)	0.069 (2)
O(8)	0.0728 (8)	0.4323 (4)	0.7930 (3)	0.074 (2)
C(1)	0.2940 (10)	1.0447 (5)	0.3573 (3)	0.042 (2)
C(2)	0.2368 (9)	1.0742 (5)	0.2749 (3)	0.038 (2)

C(3)	0.3536 (10)	1.1787 (5)	0.2414 (3)	0.041 (2)
C(4)	0.4805 (10)	1.2099 (5)	0.3019 (3)	0.044 (2)
C(5)	0.2336 (10)	0.9459 (6)	0.4251 (4)	0.049 (3)
C(6)	0.6269 (10)	1.3088 (6)	0.3008 (4)	0.049 (3)
C(7)	0.0897 (10)	1.0087 (5)	0.2296 (4)	0.038 (2)
C(8)	−0.1108 (11)	0.9839 (5)	0.2672 (4)	0.052 (3)
C(9)	−0.2498 (12)	0.9257 (6)	0.2234 (5)	0.072 (3)
C(10)	−0.1864 (14)	0.8898 (7)	0.1401 (6)	0.076 (4)
C(11)	0.0112 (15)	0.9130 (6)	0.1009 (4)	0.069 (3)
C(12)	0.1513 (11)	0.9718 (5)	0.1448 (4)	0.056 (3)
C(13)	0.3450 (12)	1.2437 (5)	0.1545 (4)	0.047 (3)
C(14)	0.1602 (11)	1.2915 (5)	0.1326 (4)	0.054 (3)
C(15)	0.1515 (13)	1.3512 (6)	0.0513 (5)	0.068 (3)
C(16)	0.3280 (15)	1.3618 (7)	−0.0060 (4)	0.074 (4)
C(17)	0.5133 (14)	1.3142 (7)	0.0142 (4)	0.076 (3)
C(18)	0.5244 (11)	1.2542 (6)	0.0947 (4)	0.060 (3)
C(19)	−0.2098 (12)	0.6445 (6)	0.5015 (4)	0.050 (3)
C(20)	−0.3436 (11)	0.6888 (6)	0.4351 (4)	0.076 (3)
C(21)	−0.0672 (12)	0.4413 (6)	0.7393 (4)	0.056 (3)
C(22)	−0.2501 (12)	0.3625 (6)	0.7625 (4)	0.078 (3)

Table 2. *Selected geometric parameters (Å, °)*

N(1)—C(1)	1.363 (8)	N(1)—C(4)	1.382 (7)
O(1)—C(5)	1.208 (8)	O(2)—C(5)	1.312 (8)
O(3)—C(6)	1.229 (7)	O(4)—C(6)	1.324 (8)
O(5)—C(19)	1.200 (10)	O(6)—C(19)	1.332 (7)
O(7)—C(21)	1.228 (7)	O(8)—C(21)	1.313 (9)
C(1)—C(2)	1.405 (8)	C(1)—C(5)	1.477 (8)
C(2)—C(3)	1.404 (8)	C(2)—C(7)	1.473 (9)
C(3)—C(4)	1.393 (9)	C(3)—C(13)	1.499 (8)
C(4)—C(6)	1.436 (9)	C(7)—C(8)	1.375 (9)
C(19)—C(20)	1.490 (10)	C(21)—C(22)	1.465 (10)
C(1)—N(1)—C(4)	109.0 (5)	N(1)—C(1)—C(2)	108.8 (5)
N(1)—C(1)—C(5)	117.2 (5)	C(2)—C(1)—C(5)	133.9 (6)
C(1)—C(2)—C(3)	106.2 (5)	C(1)—C(2)—C(7)	128.3 (5)
C(3)—C(2)—C(7)	125.5 (5)	C(2)—C(3)—C(4)	108.3 (5)
C(2)—C(3)—C(13)	125.6 (6)	C(4)—C(3)—C(13)	126.1 (5)
N(1)—C(4)—C(3)	107.6 (5)	N(1)—C(4)—C(6)	121.2 (5)
C(3)—C(4)—C(6)	131.2 (5)	O(1)—C(5)—O(2)	124.6 (5)
O(1)—C(5)—C(1)	122.3 (6)	O(2)—C(5)—C(1)	113.1 (5)
O(3)—C(6)—O(4)	123.0 (6)	O(3)—C(6)—C(4)	123.5 (6)
O(4)—C(6)—C(4)	113.5 (5)	C(2)—C(7)—C(8)	122.0 (5)
C(2)—C(7)—C(12)	120.0 (6)	C(8)—C(7)—C(12)	118.0 (6)
C(7)—C(12)—C(11)	120.2 (6)	C(3)—C(13)—C(14)	120.4 (6)
C(3)—C(13)—C(18)	120.5 (6)	C(14)—C(13)—C(18)	119.1 (5)
O(5)—C(19)—O(6)	121.9 (6)	O(5)—C(19)—C(20)	126.4 (6)
O(6)—C(19)—C(20)	111.6 (6)	O(7)—C(21)—O(8)	120.8 (6)
O(7)—C(21)—C(22)	123.5 (7)	O(8)—C(21)—C(22)	115.7 (5)

Data collection, structure solution and refinement, and preparation of drawings were performed using *SHELXTL-Plus* programs (Sheldrick, 1991).

The authors wish to acknowledge the University of Massachusetts Dartmouth for financial support and the Chemistry Department of Brown University for the use of their X-ray facilities. J. Loehlin of Wellesley University is also thanked for his help with the standard-deviation calculations.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1169). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Boukhari, A., Eddaif, A., Laurent, A., Mison, P. & Faure, R. (1989). *J. Crystallogr. Spectrosc. Res.* **19**, 603–621.

Friedman, M. (1965). *J. Org. Chem.* **30**, 861–862.

Huheey, J. (1983). *Inorganic Chemistry: Principles of Structure and Reactivity*, 3rd ed. p. 268. New York: Harper & Row.

Medforth, C. J., Senge, M. O., Smith, M., Sparks, L. D. & Shelnut, J. A. (1992). *J. Am. Chem. Soc.* **114**, 9860–9861.

Prayzner, P. (1994). MS thesis, University of Massachusetts Dartmouth, North Dartmouth, Massachusetts, USA.

Sheldrick, G. M. (1991). *SHELXTL-Plus. Structure Determination Software Programs.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Viossat, B., Nguyen-Huy, D., Lehuede, J. & Vierfond, J. M. (1994). *Acta Cryst.* C**50**, 211–214.

Acta Cryst. (1996). C**52**, 667–669

Diethyl (1-Hydroxy-2-butynyl)phosphonate

TANYA C. SANDERS,[a] GERALD B. HAMMOND,[a] JAMES A. GOLEN[a] AND PAUL G. WILLIARD[b]

[a]*Department of Chemistry, University of Massachusetts Dartmouth, North Dartmouth, MA 02747, USA, and* [b]*Department of Chemistry, Brown University, Providence, RI 02912, USA*

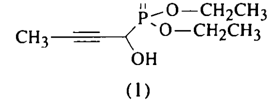
(*Received 18 October 1994; accepted 4 September 1995*)

Abstract

The structure of diethyl (1-hydroxy-2-butynyl)phosphonate, $C_8H_{15}O_4P$, exhibits nearly tetrahedral geometry around the P atom, in addition to intermolecular and intramolecular hydrogen bonding between the hydroxy group and the double-bonded phosphoryl O atom (O—H···O=P).

Comment

Mono- and difluorinated compounds of phosphorus and their derivatives have found use as markers in many biological phosphate systems (Blackburn, Brown, Martin & Parratt, 1987; Halazy & Gross-Berges, 1992). In the course of our studies on the regiospecific fluorination of hydroxy phosphonates, the title compound, (1), was isolated (Sanders & Hammond, 1993).



$CH_3-C\equiv C-CH(OH)-P(=O)(O-CH_2CH_3)(O-CH_2CH_3)$

(1)

Analysis of the structural data indicates that the P—O distances [1.557 (6) and 1.571 (5) Å] in the P—O—C linkages (Fig. 1) differ by 0.014 Å, but the bond distances are compatible with known values: a P—O bond distance of 1.586 Å in substituted dioxaphosphocine (Naidu, Krishnaiah & Sivakumar, 1992) and P—

Acta Crystallographica Section C
ISSN 0108-2701 ©1996