

C(41)	0.32296 (16)	-0.3515 (13)	0.1396 (3)	0.0590 (21)
O(41)	0.32470 (17)	-0.5756 (11)	0.1651 (3)	0.0797 (22)
O(42)	0.33030 (14)	-0.2649 (11)	0.04534 (23)	0.0681 (18)
C(43)	0.34400 (20)	-0.444449 †	-0.0377 (3)	0.069 (3)
C(44)	0.3019 (3)	-0.6293 (14)	-0.0431 (5)	0.091 (3)
C(45)	0.3915 (3)	-0.5871 (19)	-0.0270 (6)	0.096 (4)
C(46)	0.3515 (4)	-0.2714 (16)	-0.1290 (5)	0.114 (4)
C(51)	0.40077 (22)	-0.1074 (17)	0.2957 (5)	0.089 (4)
C(52)	0.4240 (4)	-0.316 (3)	0.3300 (13)	0.146 (9)
C(53)	0.4696 (5)	-0.378 (4)	0.2742 (15)	0.171 (12)
C(54)	0.4904 (8)	-0.260 (5)	0.2029 (20)	0.242 (22)
C(55)	0.4647 (5)	-0.036 (5)	0.1630 (11)	0.171 (12)
C(56)	0.4220 (4)	0.029 (3)	0.2139 (9)	0.136 (8)

† The y coordinate of atom C(43) was fixed at the given value in order to fix the origin along y in space group C2.

Table 2. Selected geometric parameters (Å, °)

C(1)—O(1)	1.185 (13)	C(5)—C(51)	1.489 (10)
C(1)—C(2)	1.462 (12)	O(11)—C(12)	1.529 (20)
C(1)—O(11)	1.316 (13)	C(12)—C(13)	1.30 (3)
C(2)—O(2)	1.450 (10)	C(41)—O(41)	1.222 (7)
C(2)—C(3)	1.472 (10)	C(41)—O(42)	1.331 (7)
O(2)—C(3)	1.415 (9)	O(42)—C(43)	1.461 (6)
C(3)—C(4)	1.500 (8)	C(43)—C(44)	1.530 (8)
C(4)—N(4)	1.455 (7)	C(43)—C(45)	1.545 (9)
C(4)—C(5)	1.544 (9)	C(43)—C(46)	1.512 (10)
N(4)—C(41)	1.356 (7)		
C(2)—C(1)—O(1)	124.9 (9)	C(51)—C(5)—C(4)	112.0 (6)
O(11)—C(1)—O(1)	123.5 (10)	C(12)—O(11)—C(1)	112.2 (10)
O(11)—C(1)—C(2)	111.5 (8)	C(13)—C(12)—O(11)	101.0 (17)
O(2)—C(2)—C(1)	115.7 (7)	O(41)—C(41)—N(4)	123.8 (5)
C(3)—C(2)—C(1)	120.3 (7)	O(42)—C(41)—N(4)	110.4 (5)
C(3)—C(2)—O(2)	57.9 (5)	O(42)—C(41)—O(41)	125.9 (5)
C(3)—O(2)—C(2)	61.8 (5)	C(43)—O(42)—C(41)	119.5 (4)
O(2)—C(3)—C(2)	60.3 (5)	C(44)—C(43)—O(42)	109.6 (4)
C(4)—C(3)—C(2)	123.8 (6)	C(45)—C(43)—O(42)	110.9 (4)
C(4)—C(3)—O(2)	116.3 (5)	C(45)—C(43)—C(44)	112.0 (5)
N(4)—C(4)—C(3)	112.5 (5)	C(46)—C(43)—O(42)	103.0 (5)
C(5)—C(4)—C(3)	108.4 (5)	C(46)—C(43)—C(44)	110.0 (5)
C(5)—C(4)—N(4)	110.9 (5)	C(46)—C(43)—C(45)	110.9 (5)
C(41)—N(4)—C(4)	121.3 (5)		
O(1)—C(1)—C(2)—O(2)	-24.0 (13)		
O(1)—C(1)—C(2)—C(3)	42.2 (13)		
O(11)—C(1)—C(2)—O(2)	160.1 (7)		
O(11)—C(1)—C(2)—C(3)	-133.5 (8)		
O(1)—C(1)—O(11)—C(12)	10.9 (15)		
C(2)—C(1)—O(11)—C(12)	-173.2 (10)		
C(1)—C(2)—O(2)—C(3)	111.1 (18)		
C(1)—C(2)—C(3)—O(2)	-102.9 (8)		
C(1)—C(2)—C(3)—C(4)	153.6 (7)		
O(2)—C(2)—C(3)—C(4)	-103.3 (7)		
C(2)—O(2)—C(3)—C(4)	115.6 (6)		
C(2)—C(3)—C(4)—N(4)	19.5 (8)		
C(2)—C(3)—C(4)—C(5)	142.6 (6)		
O(2)—C(3)—C(4)—N(4)	-50.9 (7)		
O(2)—C(3)—C(4)—C(5)	72.1 (7)		
C(3)—C(4)—N(4)—C(41)	-124.7 (5)		
C(5)—C(4)—N(4)—C(41)	113.6 (6)		
C(3)—C(4)—C(5)—C(51)	177.4 (5)		
N(4)—C(4)—C(5)—C(51)	-58.5 (7)		
C(4)—N(4)—C(41)—O(41)	12.9 (8)		
C(4)—N(4)—C(41)—O(42)	-168.5 (5)		
C(4)—C(5)—C(51)—C(52)	-84.1 (10)		
C(4)—C(5)—C(51)—C(56)	95.4 (10)		
C(1)—O(11)—C(12)—C(13)	169.3 (15)		
N(4)—C(41)—O(42)—C(43)	178.5 (4)		
O(41)—C(41)—O(42)—C(43)	-3.0 (8)		
C(41)—O(42)—C(43)—C(44)	66.1 (6)		
C(41)—O(42)—C(43)—C(45)	-58.0 (6)		
C(41)—O(42)—C(43)—C(46)	-176.8 (5)		
C(5)—C(51)—C(52)—C(53)	178.6 (11)		

Program used to solve the structure: *SHELX86* (Sheldrick, 1985). Program used for refinement: *SHELX76* (Sheldrick, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1090). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Gould, R. O. & Smith, D. E. (1986). *CADABS*. Univ. of Edinburgh, Scotland.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Reetz, M. T. & Lauterbach, E. H. (1991a). *Tetrahedron Lett.* **32**, 4477–4480.
- Reetz, M. T. & Lauterbach, E. H. (1991b). *Tetrahedron Lett.* **32**, 4481–4482.
- Sandoz (1993). European Patent Application 92/01 571.
- Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
- Sheldrick, G. M. (1985). *SHELX86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.

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Cyclic (1*R*,3*R*)-1,3-Dimethyltrimethylene [(5*R*)-2-Hydroxy-5-methyl-5-(2-methyl-1,3-dioxolan-2-yl)-1-cyclohexen-1-yl]-phosphonate, a Stable Enol

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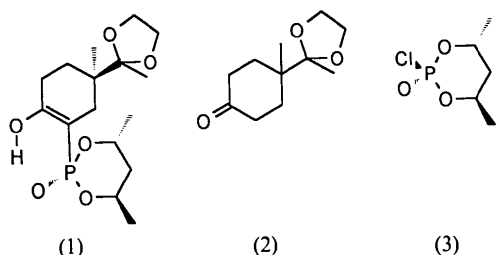
Abstract

The title compound, C₁₆H₂₇O₆P, was obtained from the rearrangement of the corresponding vinyl phosphate. ³¹P NMR experiments in solution have shown that this compound equilibrates to a mixture of three isomers in nearly equal proportions. In the crystalline state a single isomer is found which diffraction analysis identified as the enol form of the β-keto phosphonate having *R* stereochemistry at atom C(8).

Comment

The vinyl phosphate derivatives of some cyclic ketones are known to undergo rearrangement to the corresponding β-keto phosphonates upon treatment with strong base (Calogeropoulou, Hammond & Wiemer, 1987; An & Wiemer, 1992). When this reaction

sequence was explored with 4-methyl-4-(2-methyl-1,3-dioxolan-2-yl)cyclohexanone, (2), and 2-chloro-4,6-dimethyl-1,3,2-dioxaphosphorinane 2-oxide, (3), derived from (2*R*,4*R*)-pentanediol, a mixture of six products resulted. Identification of the structure and stereochemistry of these isomers was fundamental to an increased understanding of the rearrangement mechanism, but could not be accomplished by routine spectral methods.



C(2) bond distance of 1.352(5) Å and the C(1)—O(1) bond distance of 1.352(5) Å closely match the average literature values (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987) reported for enol compounds based on more than 50 examples. The H atom of the enol is hydrogen bonded to the O(2) atom forming a six-membered ring. Data to support this conclusion can be found in the bond angle and the bond distances [O(1)—H(O1)···O(2) 157(6)°, O(1)—H(O1) 0.76(4), O(2)···H(O1) 1.91(5), O(1)···O(2) 2.629(6) Å]. Finally, given that the stereochemistries of atoms C(21) and C(23) were known to be *R* by selection of the starting material, diffraction analysis allowed identification of the stereochemistry at C(8) as *R*.

Experimental

A mixture of the phosphonates obtained upon rearrangement was purified by flash column chromatography. The resulting mixture of diastereomers was dissolved in ether at room temperature. This solution was allowed to stand for 2 d at room temperature open to the air. Crystal formation resulted as the solution concentrated. After filtration, the crystals were washed with cold ether (277–278 K) and dried in air.

Crystal data

C₁₆H₂₇O₆P
M_r = 346.36
 Orthorhombic
*P*2₁2₁
a = 10.677 (7) Å
b = 16.763 (8) Å
c = 10.015 (5) Å
V = 1793 (3) Å³
Z = 4
D_x = 1.28 Mg m⁻³

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 20–30°
 μ = 0.17 mm⁻¹
T = 295 K
 Irregular platelet
 0.60 × 0.40 × 0.20 mm
 Clear

Data collection

Enraf–Nonius CAD-4 diffractometer
 θ -2 θ scans
 Absorption correction: empirical
 T_{\min} = 0.897, T_{\max} = 0.998
 4495 measured reflections
 1804 independent reflections
 1274 observed reflections
 $[I > 2\sigma(I)]$

R_{int} = 0.034
 θ_{max} = 25°
 h = -12 → 8
 k = -19 → 19
 l = -11 → 1
 4 standard reflections monitored every 500 reflections (for orientation) and every 240 min (for intensity)
 intensity decay: <0.3%

Refinement

Refinement on *F*
R = 0.037
 wR = 0.048
S = 1.09
 1274 reflections
 212 parameters
 $w = 1/[\sigma^2(F) + (0.06F)^2]$

$(\Delta/\sigma)_{\text{max}}$ = 0.02
 $\Delta\rho_{\text{max}}$ = 0.159 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -0.187 e Å⁻³
 Atomic scattering factors from Cromer & Waber (1974)

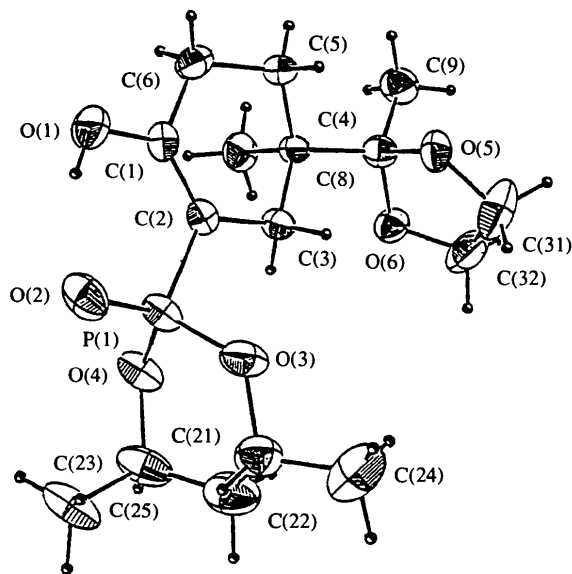


Fig. 1. An ORTEP (Johnson, 1965) drawing of the title compound. Displacement ellipsoids are plotted at the 35% probability level.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
P(1)	0.40304 (8)	0.99431 (6)	0.6848 (1)	0.0470 (2)
O(1)	0.5369 (3)	1.1316 (2)	0.5460 (3)	0.0667 (9)
O(2)	0.3551 (3)	1.0766 (2)	0.6973 (3)	0.0674 (8)
O(3)	0.4340 (2)	0.9525 (2)	0.8221 (3)	0.0618 (8)
O(4)	0.3094 (2)	0.9365 (2)	0.6120 (3)	0.0598 (8)
O(5)	0.8547 (2)	0.8475 (1)	0.6444 (3)	0.0528 (8)
O(6)	0.7325 (3)	0.7666 (1)	0.5160 (3)	0.0516 (6)
C(1)	0.5903 (3)	1.0585 (2)	0.5400 (4)	0.0462 (9)
C(2)	0.5447 (3)	0.9907 (2)	0.5947 (3)	0.0423 (9)
C(3)	0.6115 (3)	0.9114 (2)	0.5849 (4)	0.0438 (9)
C(4)	0.7177 (3)	0.9085 (2)	0.4814 (3)	0.0388 (9)
C(5)	0.7917 (3)	0.9871 (2)	0.4891 (4)	0.0480 (9)
C(6)	0.7112 (4)	1.0599 (2)	0.4641 (4)	0.052 (1)
C(7)	0.6609 (4)	0.8985 (2)	0.3418 (4)	0.059 (1)
C(8)	0.8058 (4)	0.8388 (2)	0.5126 (4)	0.0441 (9)
C(9)	0.9142 (4)	0.8285 (3)	0.4161 (5)	0.063 (1)
C(21)	0.3467 (4)	0.9047 (3)	0.8969 (5)	0.068 (1)
C(22)	0.2709 (4)	0.8519 (3)	0.8040 (6)	0.089 (1)
C(23)	0.2117 (4)	0.8959 (3)	0.6891 (5)	0.072 (1)
C(24)	0.4218 (7)	0.8590 (4)	0.9952 (7)	0.144 (2)
C(25)	0.1109 (4)	0.9518 (3)	0.7309 (6)	0.092 (1)
C(31)	0.8497 (6)	0.7732 (3)	0.7101 (5)	0.091 (1)
C(32)	0.7779 (6)	0.7204 (3)	0.6224 (6)	0.090 (1)
H(O1)	0.474 (3)	0.124 (2)	0.578 (4)	0.07 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

P(1)—O(2)	1.477 (3)	C(2)—C(3)	1.511 (6)
P(1)—O(3)	1.579 (3)	C(3)—C(4)	1.538 (5)
P(1)—O(4)	1.571 (3)	C(4)—C(5)	1.537 (5)
P(1)—C(2)	1.762 (4)	C(4)—C(7)	1.533 (6)
O(1)—C(1)	1.352 (5)	C(4)—C(8)	1.533 (5)
O(3)—C(21)	1.440 (6)	C(5)—C(6)	1.513 (6)
O(4)—C(23)	1.466 (6)	C(8)—C(9)	1.518 (6)
O(5)—C(8)	1.427 (5)	C(21)—C(22)	1.518 (8)
O(5)—C(31)	1.410 (6)	C(21)—C(24)	1.483 (9)
O(6)—C(8)	1.441 (5)	C(22)—C(23)	1.507 (9)
O(6)—C(32)	1.405 (7)	C(23)—C(25)	1.486 (7)
C(1)—C(2)	1.352 (5)	C(31)—C(32)	1.464 (8)
C(1)—C(6)	1.498 (6)		
O(2)—P(1)—O(3)	114.4 (2)	C(5)—C(4)—C(7)	110.1 (3)
O(2)—P(1)—O(4)	113.3 (2)	C(7)—C(4)—C(8)	110.2 (3)
O(2)—P(1)—C(2)	111.9 (2)	C(4)—C(5)—C(6)	113.0 (3)
O(3)—P(1)—O(4)	105.3 (2)	C(1)—C(6)—C(5)	113.1 (3)
O(3)—P(1)—C(2)	104.5 (2)	O(5)—C(8)—O(6)	105.3 (3)
O(4)—P(1)—C(2)	106.7 (2)	O(5)—C(8)—C(4)	109.6 (3)
P(1)—O(3)—C(21)	124.4 (3)	O(5)—C(8)—C(9)	108.7 (4)
P(1)—O(4)—C(23)	119.7 (3)	O(6)—C(8)—C(4)	108.1 (3)
C(8)—O(5)—C(31)	109.1 (3)	O(6)—C(8)—C(9)	109.5 (3)
C(8)—O(6)—C(32)	107.1 (4)	C(4)—C(8)—C(9)	115.1 (4)
O(1)—C(1)—C(2)	126.3 (4)	O(3)—C(21)—C(22)	110.6 (5)
O(1)—C(1)—C(6)	111.8 (4)	O(3)—C(21)—C(24)	106.5 (5)
C(2)—C(1)—C(6)	121.9 (4)	C(22)—C(21)—C(24)	113.2 (6)
P(1)—C(2)—C(1)	119.1 (3)	C(21)—C(22)—C(23)	113.9 (4)
P(1)—C(2)—C(3)	118.0 (3)	O(4)—C(23)—C(22)	109.4 (4)
C(1)—C(2)—C(3)	122.9 (3)	O(4)—C(23)—C(25)	111.8 (4)
C(2)—C(3)—C(4)	114.8 (3)	C(22)—C(23)—C(25)	113.4 (5)
C(3)—C(4)—C(5)	108.5 (3)	O(5)—C(31)—C(32)	105.9 (4)
C(3)—C(4)—C(7)	109.1 (3)	O(6)—C(32)—C(31)	107.6 (4)
C(3)—C(4)—C(8)	109.8 (3)	C(1)—O(1)—H(O1)	104 (4)
O(2)—P(1)—O(3)—C(21)	91.4 (3)		
O(4)—P(1)—O(3)—C(21)	-33.6 (3)		
C(2)—P(1)—O(3)—C(21)	-145.8 (3)		
O(2)—P(1)—O(4)—C(23)	-86.2 (3)		
O(3)—P(1)—O(4)—C(23)	39.5 (3)		
C(2)—P(1)—O(4)—C(23)	150.2 (3)		
O(2)—P(1)—C(2)—C(1)	-4.3 (4)		
O(2)—P(1)—C(2)—C(3)	174.3 (3)		
O(3)—P(1)—C(2)—C(1)	-128.6 (3)		
O(3)—P(1)—C(2)—C(3)	49.9 (3)		

O(4)—P(1)—C(2)—C(1)	120.1 (3)
O(4)—P(1)—C(2)—C(3)	-61.3 (3)
P(1)—O(3)—C(21)—C(22)	40.7 (5)
P(1)—O(3)—C(21)—C(24)	164.1 (3)
P(1)—O(4)—C(23)—C(22)	-53.9 (4)
P(1)—O(4)—C(23)—C(25)	72.6 (4)
C(31)—O(5)—C(8)—O(6)	19.5 (4)
C(31)—O(5)—C(8)—C(4)	135.6 (4)
C(31)—O(5)—C(8)—C(9)	-97.7 (4)
C(8)—O(5)—C(31)—C(32)	-8.6 (5)
C(32)—O(6)—C(8)—O(5)	-23.0 (4)
C(32)—O(6)—C(8)—C(4)	-140.1 (3)
C(32)—O(6)—C(8)—C(9)	93.7 (4)
O(1)—C(1)—C(2)—P(1)	-0.6 (5)
O(1)—C(1)—C(2)—C(3)	-179.1 (3)
C(6)—C(1)—C(2)—P(1)	179.0 (3)
C(6)—C(1)—C(2)—C(3)	0.5 (6)
O(1)—C(1)—C(6)—C(5)	163.0 (3)
C(2)—C(1)—C(6)—C(5)	-16.6 (5)
P(1)—C(2)—C(3)—C(4)	167.5 (2)
C(1)—C(2)—C(3)—C(4)	-14.0 (5)
C(2)—C(3)—C(4)—C(5)	41.2 (4)
C(2)—C(3)—C(4)—C(7)	-78.7 (4)
C(2)—C(3)—C(4)—C(8)	160.5 (3)
C(3)—C(4)—C(5)—C(6)	-58.4 (4)
C(7)—C(4)—C(5)—C(6)	60.9 (4)
C(8)—C(4)—C(5)—C(6)	-178.1 (3)
C(3)—C(4)—C(8)—O(5)	-57.9 (4)
C(3)—C(4)—C(8)—O(6)	56.3 (4)
C(3)—C(4)—C(8)—C(9)	179.1 (3)
C(5)—C(4)—C(8)—O(5)	60.9 (4)
C(5)—C(4)—C(8)—O(6)	175.2 (3)
C(5)—C(4)—C(8)—C(9)	-62.0 (4)
C(7)—C(4)—C(8)—O(5)	-178.1 (3)
C(7)—C(4)—C(8)—O(6)	-63.8 (4)
C(7)—C(4)—C(8)—C(9)	59.0 (4)
C(4)—C(5)—C(6)—C(1)	46.4 (4)
O(3)—C(21)—C(22)—C(23)	-50.7 (5)
C(24)—C(21)—C(22)—C(23)	-170.1 (5)
C(21)—C(22)—C(23)—O(4)	57.6 (5)
C(21)—C(22)—C(23)—C(25)	-68.0 (5)
O(5)—C(31)—C(32)—O(6)	-5.9 (6)

Data collection used a scan range of $(0.6 + 0.35\text{tan}\theta)^\circ$ and a scan speed in the range 0.75 to $6.7^\circ \text{min}^{-1}$. Net intensity was determined by profiling scan using the method of Lehman & Larsen (1974). All H atoms were found on an electron density difference map. Atom H(O1) was refined but all other H atoms were placed in calculated positions with a C—H distance of 0.95\AA . The structure was determined by direct methods. The absolute stereochemistry of the 2,4-pentanediol was known in advance and so no attempt was made to determine the absolute configuration from the X-ray diffraction data. The scattering factors f' and f'' were included in the structure-factor calculations.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *MolEN*. Software used to prepare material for publication: *MolEN*.

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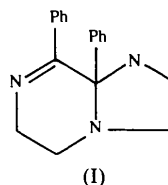
Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry including torsion angles have been deposited with the IUCr (Reference: BK1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
- An, Y. Z. & Wiemer, D. F. (1992). *J. Org. Chem.* **57**, 317–321.
- Calogeropoulou, T., Hammond, G. B. & Wiemer, D. F. (1987). *J. Org. Chem.* **52**, 4185–4190.
- Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Lehman, M. S. & Larsen, F. K. (1974). *Acta Cryst.* **A30**, 580–584.

sedatives. However, crystal structure analyses of only a few derivatives have been carried out (Lumma & Springer, 1981).

The title compound, (I), was synthesized for the first time in good yield by the reaction of 3-aza-1,5-diaminopentane with benzil in the presence of HgCl₂ under refluxing conditions in methanol (Parihar, 1993).



An *ORTEP* (Johnson, 1965) diagram of the molecule is shown in Fig. 1. The molecule consists of a nine-membered fused bicyclic ring system containing five- and six-membered heterocycles which are joined at C(6) and N(2). C(6) is a chiral centre with typical tetrahedral bond angles. The geometry about N(2) is distorted tetrahedral. The values of the N(2)—C(4)—C(5), C(4)—C(5)—N(3), C(5)—N(3)—C(6), N(3)—C(6)—N(2) and C(6)—N(2)—C(4) bond angles are typical of a strained saturated five-membered ring system.

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An Imidazo[1,2-*a*]pyrazine Derivative, C₁₈H₁₉N₃

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Abstract

Interaction of benzil in methanol with 3-aza-1,5-diaminopentane in the presence of HgCl₂ yields the title compound, 1,2,3,5,6,8a-hexahydro-8,8a-diphenylimidazo[1,2-*a*]pyrazine. The molecule consists of a bicyclic system containing five- and six-membered heterocycles which are joined at C(6) and N(2). C(6) is a chiral centre and N(2) has distorted tetrahedral geometry around it. The molecule is puckered at the C(6)—N(2) bond.

Comment

The first derivative of imidazo[1,2-*a*]pyrazine, a bicyclic system, was reported in 1957 as a perchlorate salt in low yields (Martin & Tarasiejska, 1957). More recently, several derivatives of imidazo[1,2-*a*]pyrazine have been reported (Bradac *et al.*, 1977; Abignente *et al.*, 1981; Bonnet, Sablayrallis & Chapat, 1984; Katsunori, Minoru, Tetsuya & Toshio, 1992). These derivatives have been attracting increasing attention (Lumma, 1980; Lumma *et al.*, 1983; Sablayrallis, Bonnet, Cros, Chapat & Bouchard, 1988; Zaitsev, Glushkov, Mashkovskii & Andreova, 1989) because of their interesting structural features, as well as a wide range of applications, *e.g.* as antispasmodics, uterine relaxants, bronchodilators and

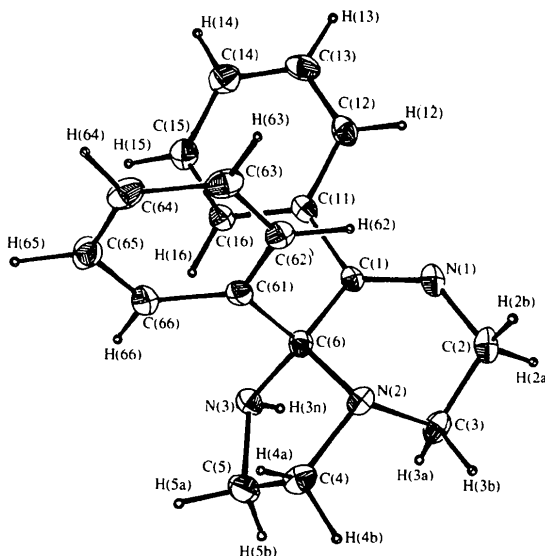


Fig. 1. View of the molecule with displacement ellipsoids drawn at the 50% probability level and H atoms as small spheres of arbitrary size.

The C(1)—N(1) bond distance of 1.274 (4) Å is comparable to the reported value of C=N (1.307 Å) in 3-(1-hydroxyethyl)imidazo[1,2-*a*]pyrazine (Lumma & Springer, 1981). The average bond distance of C(2)—N(1), C(3)—N(2), C(4)—N(2), C(6)—N(2), C(6)—N(3) and C(5)—N(3) is 1.474 (4) Å, which is very