C8	0.3950 (4)	0.7889 (3)	−0.01781 (13)	0.0595 (7)
C11	0.7066 (4)	0.9000 (3)	−0.24448 (13)	0.0542 (6)
C1	0.3199 (4)	0.6129 (3)	0.20974 (13)	0.0477 (5)
C2	0.5536 (5)	0.4992 (3)	0.2388 (2)	0.0610 (7)
C13	0.7660 (5)	1.0094 (3)	−0.39804 (13)	0.0546 (6)
C4	0.3365 (6)	0.4897 (4)	0.3763 (2)	0.0809 (9)
C6	0.0973 (5)	0.6655 (3)	0.26476 (15)	0.0641 (7)
C5	0.1047 (5)	0.6039 (4)	0.3476 (2)	0.0759 (8)
C3	0.5590 (6)	0.4386 (4)	0.3220 (2)	0.0775 (8)

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

Compound (I)

Cl—C4	1.742 (2)	C9—C8	1.511 (3)
N1—C7	1.343 (3)	C2—C3	1.376 (3)
N1—C1	1.416 (3)	C7—C8	1.510 (3)
O122—C12	1.272 (3)	C11—C12	1.494 (3)
C10—C9	1.513 (3)	C3—C4	1.370 (4)
C10—C11	1.513 (3)	O121—C12	1.227 (3)
O7—C7	1.224 (3)	C6—C5	1.383 (3)
C1—C6	1.375 (3)	C4—C5	1.364 (4)
C1—C2	1.380 (3)		
C7—N1—C1	125.4 (2)	C4—C3—C2	119.7 (2)
C9—C10—C11	112.1 (2)	C1—C6—C5	120.5 (2)
C6—C1—C2	119.1 (2)	C5—C4—C3	120.7 (2)
C6—C1—N1	119.9 (2)	C5—C4—Cl	119.9 (2)
C2—C1—N1	121.1 (2)	C3—C4—Cl	119.4 (2)
C10—C9—C8	113.1 (2)	O121—C12—O122	122.7 (2)
C3—C2—C1	120.4 (2)	O121—C12—C11	121.7 (2)
O7—C7—N1	122.8 (2)	O122—C12—C11	115.5 (2)
O7—C7—C8	121.5 (2)	C7—C8—C9	113.3 (2)
N1—C7—C8	115.6 (2)	C4—C5—C6	119.5 (2)
C12—C11—C10	115.1 (2)		
O121···O122^{i}	2.617	N1···O7^{ii}	2.958

Symmetry codes: (i) $1 - x, -y - 1, 2 - z$; (ii) $1 + x, y, z$.

Compound (II)

N1—C7	1.349 (3)	C12—C13	1.483 (3)
N1—C1	1.410 (3)	C12—C11	1.512 (3)
O7—C7	1.224 (2)	C1—C6	1.376 (3)
C9—C10	1.504 (3)	C1—C2	1.379 (3)
C9—C8	1.509 (3)	C2—C3	1.378 (4)
C7—C8	1.499 (3)	C4—C3	1.364 (4)
O132—C13	1.209 (3)	C4—C5	1.372 (4)
C10—C11	1.514 (3)	C6—C5	1.374 (3)
O131—C13	1.304 (3)		
C7—N1—C1	127.8 (2)	C6—C1—N1	118.0 (2)
C10—C9—C8	111.8 (2)	C2—C1—N1	122.8 (2)
O7—C7—N1	122.9 (2)	C3—C2—C1	119.6 (3)
O7—C7—C8	123.0 (2)	O132—C13—O131	122.2 (2)
N1—C7—C8	114.1 (2)	O132—C13—C12	123.5 (2)
C9—C10—C11	114.9 (2)	O131—C13—C12	114.3 (2)
C13—C12—C11	115.1 (2)	C3—C4—C5	119.6 (3)
C7—C8—C9	115.8 (2)	C5—C6—C1	120.7 (2)
C12—C11—C10	112.2 (2)	C6—C5—C4	120.0 (3)
C6—C1—C2	119.2 (2)	C4—C3—C2	121.0 (2)
O131···O132^{i}	2.644	N1···O7^{ii}	2.988

Symmetry codes: (i) $2 - x, 2 - y, -1 - z$; (ii) $x - 1, y, z$.

The coordinates of the carboxylic acid H atoms could not be located in sensible positions. All other H atoms were fixed geometrically.

Data collection, cell refinement and data reduction: *CAD-4 Software* (Enraf-Nonius, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1070). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Feeder, N. & Jones, W. (1992). *Mol. Cryst. Liq. Cryst.* **211**, 111–124.
Feeder, N. & Jones, W. (1993). *Acta Cryst.* B**49**, 541–546.
Feeder, N. & Jones, W. (1994). *Acta Cryst.* C**50**, 816–820.
Haisa, M., Kashino, S., Ueno, T., Shinozaki, N. & Matsuzaki, Y. (1980). *Acta Cryst.* B**36**, 2306–2311.
Kariuki, B. M. & Jones, W. (1990). *Mol. Cryst. Liq. Cryst.* **186**, 45–52.
Leiserowitz, L. (1976). *Acta Cryst.* B**32**, 775–802.
Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
Sheldrick, G. M. (1994). *J. Appl. Cryst.* In preparation.

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Two Nitrogen-Containing Analogues of Chorismic Acid

M. F. Mackay

School of Chemistry, La Trobe University, Bundoora, Victoria 3083, Australia

M. J. McLeish and M. Campbell

School of Pharmaceutical Chemistry,
Victorian College of Pharmacy, Monash University,
381 Royal Parade, Parkville, Victoria 3052,
Australia

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Abstract

The cyclohexane rings in both the title molecules, methyl 3-[(*tert*-butyloxycarbonyl)amino]-4-hydroxy-1-cyclohexanecarboxylate (3), $C_{13}H_{23}NO_5$, and methyl 4-[(*tert*-butyloxycarbonyl)amino]-3-hydroxy-1-cyclohexanecarboxylate (6), $C_{13}H_{23}NO_5$, have regular chair forms. The urethane side chains are extended, adopting the type b *trans–trans* conformation, as is generally observed for urethanes containing a secondary N atom. In the crystals, the *trans* planar NH—CO moieties of the urethane side chains and the hydroxyl substituents participate in extensive hydrogen bonding.

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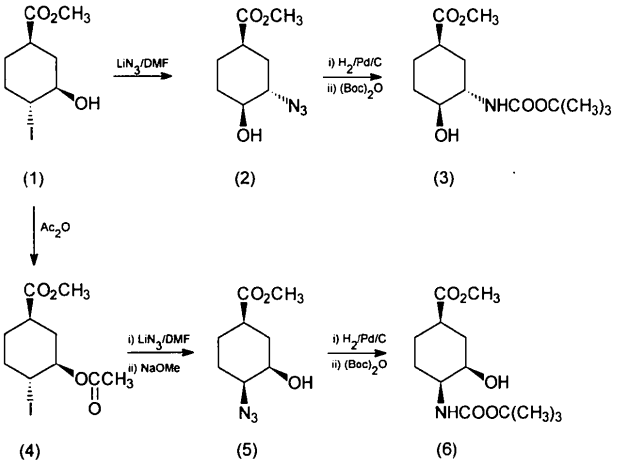
Comment

As part of our investigation into the biosynthesis of *p*-aminobenzoic acid, it has been necessary to synthesize nitrogen-containing analogues of chorismic acid (Campbell & McLeish, 1993). The preparation of methyl 4-amino-3-hydroxy-1-cyclohexanecarboxylic acid, a precursor to many of these compounds, was expected to be straightforward, requiring a simple azido displacement of the iodo group in (1) to give (5), followed by reduction to give the amino alcohol. Somewhat unexpectedly, it was observed that the azido displacement led to (2) rather than (5).



Determination of the stereochemistry of (2) using standard spectroscopic techniques proved difficult, with results being inconclusive. Similarly, the stereochemistry of (5), even when prepared using an unambiguous synthesis as shown in the scheme, was difficult to establish by the usual spectroscopic methods. In order to obtain an unambiguous assignment of their stereochemistries, the azido alcohols (2) and (5) were converted to their corresponding *tert*-butyloxycarbonylamino derivatives (3) and (6), respectively, for which crystals suitable for X-ray study were obtained.

The molecular structures are illustrated in Fig. 1. The cyclohexane ring adopts a regular chair form in both structures as shown by the asymmetry parameters (Duax & Norton, 1975) ΔC_s^{1} and $\Delta C_2^{1,2}$ of 2.6 and 2.3°, respectively, for (3), and ΔC_s^{2} and $\Delta C_2^{2,3}$ of 0.6° for (6). In (3), the methyl carboxylate substituent at C1 occupies the axial position while the *N*-butyloxycarbonyl group at C3 and the hydroxyl group at C4 are equatorial, whereas in (6) the *N*-butyloxycarbonyl group at C4 is axial and the methyl carboxylate group at C1 and the hydroxyl group at C3 are in equatorial positions. The urethane moieties adopt the type b *trans–trans* conformation (Bendetti, Pedone, Toniolo, Némethy, Pottle & Scheraga, 1980), as is generally observed for urethanes containing a secondary N atom, with torsion angles

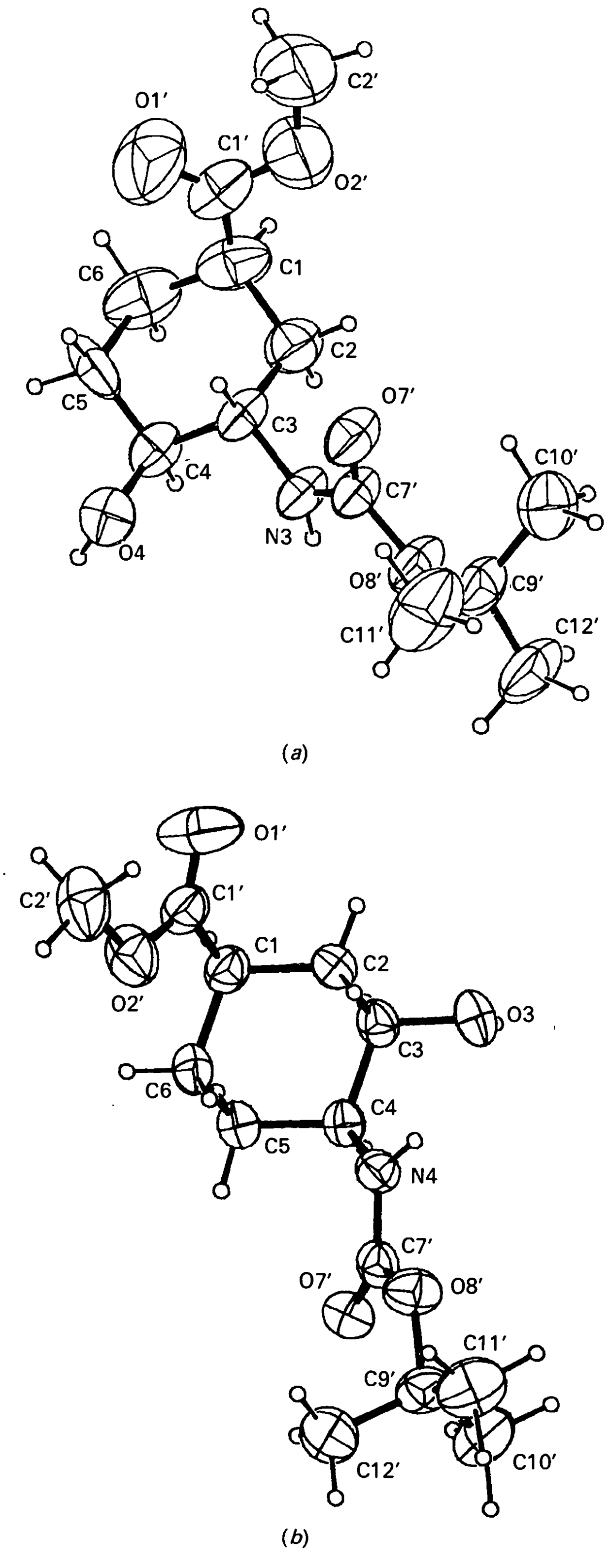


Fig. 1. Perspective views of the molecular structures showing (*a*) the 1*R*,3*S*,4*S* enantiomer of (3) and (*b*) the 1*R*,3*R*,4*S* enantiomer of (6) with displacement ellipsoids scaled to 50% probability. H atoms are denoted by spheres of arbitrary size.

C3—N3—C7′—O8′ −169.3 (3) and N3—C7′—O8′—C9′ −175.2 (3)° for compound (3) and C4—N4—C7′—O8′ 179.2 (2) and N4—C7′—O8′—C9′ −178.1 (2)° for compound (6). The methyl C atoms are staggered with respect to the C7—O7′ bonds and there is no intramolecular hydrogen bonding in either structure. The bond lengths and angles are in good agreement with those reported for comparable structures. The N—C7′ bonds are on average 0.11 Å shorter than the N—C3,C4 bonds and the C7′—O8′ bonds are on average 0.13 Å shorter than the C9′—O8′ bonds, reflecting the different covalent radii of sp^2 and sp^3 C atoms. The N—C7′—O8′ angles are contracted by about 10° from the regular trigonal value whereas the N—C7′—O7′ and O8′—C7′—O7′ angles are, on average, expanded by 5° [*cf.* values reported by Ball, Wong, Capuano, Gulbis, Mackay & Alewood (1990) for an N-butyloxycarbonyl subtituent].

The crystal packing of both structures is illustrated in Fig. 2. The *trans* planar NH—CO moieties and the hydroxyl substituents in each structure are involved in intermolecular hydrogen bonding. The NH groups donate their protons to the hydroxyl O atoms, while the hydroxyl groups donate their protons to the carbonyl O atoms, O7′. In (3), the N3···O4($\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$), N3—HN3 and HN3···O4 distances are 2.934 (5), 0.86 (3) and 2.08 (3) Å, respectively, with the N3—HN3···O4 angle having a value of 173 (2)°, while the O4···O7′($\frac{1}{2}-x$, $y+\frac{1}{2}$, $\frac{1}{2}-z$), O4—HO4 and HO4···O7′ distances are 2.826 (4), 0.78 (4) and 2.07 (4) Å, respectively, with an angle at HO4 of 163 (3)°. These interactions link screw-related molecules into infinite helices along the [010] direction in the crystal. In (6), the N4···O3($1-x$, $2-y$, $-z$), N4—HN4 and HN4···O3 distances are 2.969 (3), 0.92 (4) and 2.05 (4) Å, respectively, with an N4—HN4···O3 angle of 173 (3)°, while the O3···O7′($-x$, $2-y$, $-z$), O3—HO3 and HO3···O7′ distances are 2.739 (3), 0.95 (4) and 1.80 (4) Å, respectively, with an angle at HO3 of 170 (2)°. Both these interactions link the molecules across inversion centres into layers parallel to the (110) planes. There are no other unusually short intermolecular approaches in either crystal.

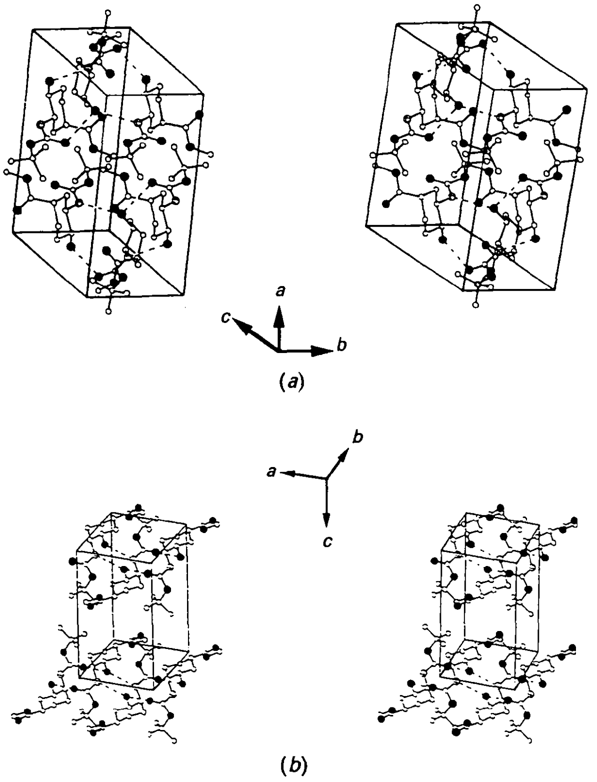


Fig. 2. Stereoviews of the crystal packing showing (*a*) structure (3) and (*b*) structure (6).

Experimental

Compound (3)

Crystal data

$C_{13}H_{23}NO_5$
$M_r = 273.3$
Monoclinic
$P2_1/n$
$a = 14.214$ (1) Å
$b = 6.593$ (1) Å
$c = 16.543$ (1) Å
$\beta = 99.51$ (1)°
$V = 1529.0$ (5) Å^3
$Z = 4$
$D_x = 1.187$ Mg m^{-3}
$D_m = 1.189$ (5) Mg m^{-3}
D_m measured by flotation

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 22$–40°
$\mu = 0.72$ mm^{-1}
$T = 291$ (1) K
Lathe
0.51 × 0.49 × 0.21 mm
Colourless

Data collection

Rigaku AFC diffractometer
$\omega/2\theta$ scans
Absorption correction: Gaussian (*SHELX*76; Sheldrick, 1976)
$T_{\min} = 0.656$, $T_{\max} = 0.801$
2685 measured reflections
2134 independent reflections
1728 observed reflections [$F > 6.0\sigma(F)$]

$R_{\rm int} = 0.018$
$\theta_{\max} = 65.0°$
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 7$
$l = 0 \rightarrow 19$
3 standard reflections monitored every 50 reflections intensity variation: <1.5%

Refinement

Refinement on F
$R = 0.070$
$wR = 0.075$
$S = 3.79$
1728 reflections
219 parameters
$w = 1/[\sigma^2(F) + 0.000045F^2]$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Extinction correction: *SHELX*76 (Sheldrick, 1976)
Extinction coefficient: 1.3 (1) × 10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (6)

Crystal data

$C_{13}H_{23}NO_5$
$M_r = 273.3$
Triclinic
$P\bar{1}$
$a = 6.843$ (1) Å
$b = 10.205$ (1) Å
$c = 11.642$ (1) Å
$\alpha = 94.81$ (1)°
$\beta = 100.10$ (1)°
$\gamma = 105.96$ (1)°
$V = 762.0$ (3) Å^3
$Z = 2$
$D_x = 1.191$ Mg m^{-3}
$D_m = 1.201$ (5) Mg m^{-3}
D_m measured by flotation

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 22$–40°
$\mu = 0.72$ mm^{-1}
$T = 291$ (1) K
Prismatic
0.51 × 0.33 × 0.31 mm
Colourless

Data collection

Rigaku AFC diffractometer
$\omega/2\theta$ scans
Absorption correction: Gaussian (*SHELX*76; Sheldrick, 1976)
$T_{\min} = 0.723$, $T_{\max} = 0.850$
2722 measured reflections
2460 independent reflections
2008 observed reflections $[F > 6.0\sigma(F)]$

$R_{\text{int}} = 0.025$
$\theta_{\max} = 65.0°$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 13$
3 standard reflections monitored every 50 reflections
intensity variation: <1.1%

Refinement

Refinement on F
$R = 0.054$
$wR = 0.075$
$S = 3.14$
2008 reflections
219 parameters
$w = 1/[\sigma^2(F) + 0.00055F^2]$
$(\Delta/\sigma)_{\max} = 0.012$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Extinction correction: *SHELX*76 (Sheldrick, 1976)
Extinction coefficient: 8.1 (6) × 10^{-6}
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)

$$B_{\text{eq}} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Compound (3)				
C1	0.3059 (4)	−0.0170 (8)	0.5034 (3)	6.7 (2)
C2	0.3630 (4)	0.0489 (9)	0.4356 (3)	6.0 (2)
C3	0.3186 (3)	−0.0193 (6)	0.3494 (2)	4.3 (1)
C4	0.2151 (3)	0.0474 (6)	0.3305 (2)	4.3 (1)
C5	0.1604 (3)	−0.0413 (8)	0.3931 (3)	5.7 (1)
C6	0.2005 (5)	0.0308 (10)	0.4784 (3)	7.9 (2)
C1′	0.3228 (5)	−0.2366 (8)	0.5275 (4)	7.5 (2)
O1′	0.2652 (3)	−0.3616 (7)	0.5267 (4)	13.8 (2)
O2′	0.4104 (3)	−0.2748 (6)	0.5565 (3)	10.6 (2)
C2′	0.4352 (4)	−0.4687 (8)	0.5953 (4)	10.6 (2)
O4	0.1748 (2)	−0.0127 (5)	0.2491 (2)	4.9 (1)
N3	0.3733 (2)	0.0620 (5)	0.2903 (2)	4.7 (1)
C7′	0.4465 (2)	−0.0354 (6)	0.2673 (2)	4.4 (1)
O7′	0.4679 (2)	−0.2113 (4)	0.2827 (2)	5.4 (1)
O8′	0.4937 (2)	0.0905 (4)	0.2232 (2)	5.4 (1)
C9′	0.5734 (3)	0.0200 (6)	0.1842 (3)	5.5 (2)
C10′	0.6544 (3)	−0.0529 (9)	0.2491 (3)	8.6 (2)
C11′	0.5398 (4)	−0.1411 (9)	0.1222 (3)	9.7 (2)
C12′	0.6011 (3)	0.2108 (7)	0.1419 (3)	7.5 (2)
HO4	0.133 (3)	0.060 (6)	0.231 (3)	
Compound (6)				
C1	0.3680 (4)	0.7188 (2)	0.2063 (2)	4.00 (4)
C2	0.4667 (4)	0.8588 (2)	0.1668 (2)	3.86 (4)
C3	0.3083 (4)	0.9366 (2)	0.1425 (2)	3.61 (4)
C4	0.1132 (4)	0.8544 (2)	0.0519 (2)	3.52 (4)
C5	0.0174 (4)	0.7168 (2)	0.0922 (2)	3.98 (4)
C6	0.1751 (4)	0.6353 (2)	0.1156 (2)	4.15 (4)
C1′	0.5302 (4)	0.6460 (3)	0.2360 (2)	4.66 (5)
O1′	0.6871 (4)	0.6929 (3)	0.3094 (2)	8.62 (6)
O2′	0.4875 (3)	0.5266 (2)	0.1691 (2)	6.08 (4)
C2′	0.6265 (5)	0.4443 (3)	0.1954 (4)	7.68 (8)
O3	0.4055 (3)	1.0649 (2)	0.1073 (2)	4.30 (3)
N4	0.1670 (3)	0.8380 (2)	−0.0634 (2)	3.63 (3)
C7′	0.0185 (4)	0.8009 (2)	−0.1630 (2)	3.67 (4)
O7′	−0.1670 (3)	0.7744 (2)	−0.1664 (1)	4.32 (3)
O8′	0.1054 (3)	0.7943 (2)	−0.2570 (1)	4.79 (3)
C9′	−0.0221 (4)	0.7599 (3)	−0.3780 (2)	4.74 (5)
C10′	−0.1440 (6)	0.8619 (3)	−0.4016 (3)	6.64 (7)
C11′	−0.1609 (6)	0.6132 (3)	−0.3962 (3)	6.30 (6)
C12′	0.1406 (6)	0.7761 (4)	−0.4533 (3)	7.14 (7)
HO3	0.336 (5)	1.127 (4)	0.134 (3)	

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

	(3)	(6)
C1—C2	1.551 (8)	1.548 (3)
C1—C6	1.520 (9)	1.525 (3)
C1—C1′	1.510 (7)	1.505 (4)
C2—C3	1.529 (6)	1.513 (4)
C3—C4	1.518 (6)	1.530 (3)
C3—N3	1.449 (5)	
C3—O3		1.425 (3)
C4—C5	1.512 (6)	1.528 (3)
C4—O4	1.430 (5)	
C4—N4		1.461 (4)
C5—C6	1.509 (7)	1.536 (4)
C1′—O1′	1.160 (8)	1.200 (3)
C1′—O2′	1.283 (8)	1.320 (3)
O2′—C2′	1.448 (7)	1.444 (4)
N3—C7′	1.331 (5)	
N4—C7′		1.350 (3)
C7′—O7′	1.215 (5)	1.216 (3)
C7′—O8′	1.354 (5)	1.339 (3)
O8′—C9′	1.469 (6)	1.479 (2)
C9′—C10′	1.517 (6)	1.517 (5)
C9′—C11′	1.499 (7)	1.511 (5)
C9′—C12′	1.523 (6)	1.517 (4)
C2—C1—C6	110.8 (4)	110.7 (2)
C2—C1—C1′	112.3 (4)	109.8 (2)
C6—C1—C1′	112.2 (4)	114.8 (2)
C1—C2—C3	114.0 (4)	110.5 (2)
C2—C3—C4	110.1 (3)	113.0 (2)
C2—C3—N3	109.6 (3)	
C4—C3—N3	111.3 (3)	
C2—C3—O3		108.7 (2)
C4—C3—O3		111.7 (2)
C3—C4—C5	109.7 (3)	109.7 (2)
C3—C4—O4	109.7 (3)	
C5—C4—O4	111.5 (3)	
C3—C4—N4		109.1 (2)
C5—C4—N4		112.7 (2)
C4—C5—C6	111.2 (4)	111.6 (2)
C1—C6—C5	113.1 (4)	111.1 (2)
C1—C1′—O1′	126.6 (5)	124.1 (2)
C1—C1′—O2′	112.9 (4)	113.1 (2)
O1′—C1′—O2′	120.3 (4)	122.7 (2)
C1′—O2′—C2′	119.3 (4)	117.5 (2)
C3—N3—C7′	123.1 (3)	
N3—C7′—O7′	125.5 (2)	
N3—C7′—O8′	109.8 (2)	

C4—N4—C7′		120.9 (2)
N4—C7′—O7′		124.9 (2)
N4—C7′—O8′		110.0 (2)
O7′—C7′—O8′	124.7 (2)	125.1 (2)
C7′—O8′—C9′	122.0 (3)	121.2 (2)
O8′—C9′—C10′	109.9 (3)	110.5 (2)
O8′—C9′—C11′	110.1 (3)	109.7 (2)
O8′—C9′—C12′	102.5 (3)	102.5 (2)
C10′—C9′—C11′	112.2 (3)	112.4 (2)
C10′—C9′—C12′	111.3 (3)	110.1 (2)
C11′—C9′—C12′	110.4 (3)	111.2 (2)
C1—C6—C5—C4	−56.7 (6)	56.8 (3)
C6—C5—C4—C3	60.9 (5)	−55.4 (3)
C5—C4—C3—C2	−58.5 (4)	55.8 (3)
C4—C3—C2—C1	53.1 (5)	−55.8 (3)
C3—C2—C1—C6	−47.7 (6)	55.1 (3)
C2—C1—C6—C5	48.9 (6)	−55.8 (3)
C6—C1—C1′—O1′	1.4 (9)	−176.2 (3)
C1—C1′—O2′—C2′	169.4 (5)	−176.1 (2)
O4—C4—C3—N3	56.8 (4)	
C4—C3—N3—C7′	−147.7 (3)	
C2—C3—N3—C7′	90.2 (4)	
C3—N3—C7′—O8′	−169.3 (3)	
N3—C7′—O8′—C9′	−175.2 (3)	
O3—C3—C4—N4		54.9 (3)
C3—C4—N4—C7′		−163.1 (2)
C5—C4—N4—C7′		74.7 (3)
C4—N4—C7′—O8′		179.2 (2)
N4—C7′—O8′—C9′		−178.1 (2)
C7′—O8′—C9′—C12′	179.2 (3)	175.5 (2)

The structures were solved by direct methods and refined by full-matrix least squares. The methyl H-atom coordinates were calculated and the atoms given a common isotropic temperature factor. The coordinates of the other H atoms were refined and the atoms were given individual isotropic temperature factors. Calculations were performed on a VAX 8800 computer.

For both compounds, data collection: Rigaku AFC software; cell refinement: Rigaku AFC software; data reduction: Rigaku AFC software; program(s) used to solve structures: *SHELX*76 (Sheldrick, 1976); program(s) used to refine structures: *SHELX*76; molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *Xtal*3.0 (Hall & Stewart, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1005). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Ball, J. B., Wong, M. G., Capuano, B., Gulbis, J. M., Mackay, M. F. & Alewood, P. F. (1990). *J. Heterocycl. Chem.* **26**, 161–169.

Bendetti, E., Pedone, C., Toniolo, C., Némethy, G., Pottle, M. S. & Scheraga, H. A. (1980). *Int. J. Pept. Protein Res.* **16**, 156–17.

Campbell, M. & McLeish, M. J. (1993). *J. Chem Res.* (*S*), pp. 148–149.

Duax, W. L. & Norton, D. A. (1975). *Atlas of Steroid Structures*, Vol. 1, pp. 18–19. New York: Plenum.

Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal*3.0 *Reference Manual*. Univs. of Western Australia, Australia, and Maryland, USA.

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

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. Univ. of Cambridge, England.

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Benzoylmethylenetriphenylphosphorane, $C_{26}H_{21}OP$

M. Kalyanasundari and K. Panchanatheswaran*

Department of Chemistry, Bharathidasan University, Tiruchirappalli 620024, India

V. Parthasarathi

Department of Physics, Bharathidasan University, Tiruchirappalli 620024, India

Ward T. Robinson and Huo Wen

Department of Chemistry, University of Canterbury, Christchurch, New Zealand

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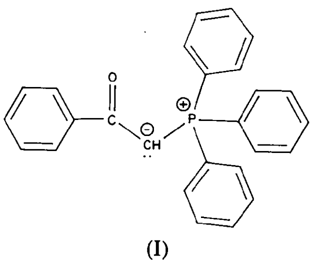
Abstract

The air-stable phosphorane (2-oxo-2-phenyltriphenylphosphoniumethylide) is shown to be a resonance hybrid of an ylene, ylidic and enolate canonical assembly. The geometry around the P atom is nearly tetrahedral. The O atom is oriented *cis* to the P atom. The phenyl ring of the benzoyl group is twisted with respect to the plane containing the carbonyl group.

Comment

Phosphoranes of the type $R_3PCHCOC_6H_5$ (R = aryl) can coordinate to metals through either C or O atoms. We have recently prepared metal derivatives of the title ylide. The crystal and molecular structure of the benzene solvate (Shao, Jin, Tang, Huang & Huang, 1982) of the title compound, which crystallized in space group $P2_1/c$, was reported without e.s.d.'s for the bond parameters. This structural investigation has been undertaken to obtain more accurate metrical parameters for the title compound, (I), and to see how they vary with a change in delocalization in the metal derivatives as well as in other resonance-stabilized ylides.



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