Fig. 1. An *ORTEP*II diagram of the molecule using 40% probability ellipsoids for C and O atoms and showing the labeling scheme. Atoms with primed labels are at the symmetry related positions $-x$, $-y$, $-z$ of corresponding unprimed atoms. H atoms are shown as open spheres of arbitrary radius.

from 0.051, wR was unchanged from 0.060, the weighting factor was $1/[\sigma^2(F_o) + 0.001962(F_o)^2]$, the goodness of fit ($S$) was 1.17, $(\Delta/\sigma)_{max} < |0.01|$, and $\Delta\rho_{max}$ and $\Delta\rho_{min}$ were 0.13 and -0.19 e Å^{-3} respectively. Corrections for effects of extinction were deemed unnecessary. Final parameters are given in Table 1,* and Table 2 contains bond distances and angles. An *ORTEP*II (Johnson, 1976) diagram of the

molecule is shown in Fig. 1. In Table 2 and Fig. 1, atoms with primed labels are at the symmetry related positions $-x$, $-y$, $-z$ of corresponding unprimed atoms.

Related literature. Isolation and identification of the tetraester precursor to the title compound, and preliminary structural information on a stereospecific transannular cycloaddition product that it forms has been reported by Gipson, Guin, Simonsen, Skinner & Shive (1966). A boat conformation for crystals of *cis,cis*-3,8-cyclodecadiene-1,6-dione was deduced from space-group considerations and reported by Carrell, Roberts, Donohue & Vollmer (1968). Conformational features of the *cis,cis*-1,6-cyclodecadiene system have been reported by Allinger, Tribble & Sprague (1972).

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* Lists of U_{ij} values, H-atom coordinates and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54906 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0550]

References

Allinger, N. L., Tribble, M. T. & Sprague, J. T. (1972). *J. Org. Chem.* **37**, 2423–2424.
Carrell, H. L., Roberts, B. W., Donohue, J. & Vollmer, J. J. (1968). *J. Am. Chem. Soc.* **90**, 5263–5264.
Gilbert, K. E. & Gajewski, J. J. (1987). *PCMODEL*. Molecular modeling software for the IBM PC/XT/AT and compatibles. Serena Software, Box 3076, Bloomington, Indiana, USA.
Gipson, R. M., Guin, H. W., Simonsen, S. H., Skinner, C. G. & Shive, W. (1966). *J. Am. Chem. Soc.* **88**, 5366–5367.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Krapcho, A. P. (1982). *Synthesis*, pp. 805–822.
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.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Structure of *r*-5-Cyano-5-methyl-*t*-4-methyl-*t*-7-oxo-7-phenyl-8-oxa-7-phosphabicyclo[4.3.0]non-2-ene Monohydrate

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Abstract. 6,7-Dimethyl-1-oxo-1-phenyl-1,3,6,7-tetrahydro-2,1-benzoxaphosphole-7-carbonitrile monohydrate, $C_{16}H_{18}NO_2P.H_2O$, $M_r = 305.3$, monoclinic, Ia, $a = 12.058$ (4), $b = 7.791$ (1), $c = 17.006$ (3) Å, $\beta = 91.74$ (3)°, $V = 1596.9$ Å^3, $Z = 4$, $D_m = 1.26$, $D_x = 1.269$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 1.75$ cm^{-1},

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$F(000) = 648$, $T = 293$ K, $R = 0.026$, $wR = 0.024$ for 1420 observed reflections. The H atoms belonging to the ring connection C(1)—C(6) are *trans*. Both rings are considerably puckered. One molecule of water is present in the asymmetric unit.

Experimental. Colourless crystals grown by slow evaporation from chloroform–ethyl acetate (1/1); crystal used in analysis shaped to ellipsoid of 0.45 × 0.3 mm by immersion in *n*-hexane/chloroform mixture; D_m by flotation in aqueous NaI; Stoe–Siemens AED four-circle automated diffractometer with graphite monochromator; ω–θ scan mode; 20 reflections used for lattice parameter determination, θ range 3.38–49.95°; 1420 reflections with $F_o > 3\sigma(F_o)$; 2θ 3.38 to 49.95°; h −14 to 14, k −9 to 9, l 0 to 20; 2 standard reflections, no intensity variation; Lp but no absorption correction applied; structure solved by Patterson synthesis and refined using F values by standard full-matrix least-squares methods and difference Fourier syntheses; all H atoms except H(80) to H(82) and H(90) to H(92) located from ΔF maps, these methyl H atoms calculated geometrically and refined in the riding model; all H atoms refined isotropically; final conventional $R = 0.0264$, $wR = 0.0245$ for observed reflections; $R = 0.0260$, $wR = 0.0241$ when all coordinates were reversed; maximum $\Delta/\sigma = 0.073$ for y of P; largest peak in final ΔF map 0.11, largest minimum -0.21 e Å^{-3}; $w = 1/\sigma^2(F_o)$; number of reflections to number of parameters ratio 5.6; scattering factors as incorporated in *SHELX*76; all calculations performed using programs written by Sheldrick (1976) on an ODRA-1305 mainframe computer. Atomic coordinates and equivalent isotropic thermal parameters for non-H atoms are listed in Table 1.* Bond distances and angles are given in Table 2. Fig. 1. is an *ORTEP*II (Johnson, 1976) plot of the compounds.

Related literature. The title compound was synthesized by the intramolecular Diels–Alder (4 + 2) cycloaddition. The tetracoordinated P atom is isosteric with C(sp^3). The essence of the method is the introduction of a chiral P atom as a centre of initial control in the asymmetric synthesis. The ultimate goal is to obtain chiranes for the synthesis of steroids. They are potentially useful in a large number of syntheses (Bodalski & Krawczyk, 1991). For a recent crystal structure analysis of the *cis* diastereoisomer see Główka, Krawczyk, Bodalski &

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^3$)

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
P	−7500 (0)	−6379 (1)	−2500 (0)	46 (1)
O(1)	−6333 (2)	−6887 (3)	−2856 (1)	62 (1)
O(2)	−7780 (2)	−4544 (2)	−2525 (1)	64 (1)
C(1)	−7170 (2)	−7297 (3)	−1537 (1)	38 (1)
C(2)	−7743 (2)	−6796 (3)	−778 (1)	42 (1)
C(3)	−7259 (2)	−7965 (4)	−101 (2)	51 (1)
C(4)	−6027 (3)	−8168 (4)	−152 (2)	59 (1)
C(5)	−5423 (3)	−7750 (4)	−758 (2)	58 (1)
C(6)	−5919 (2)	−6928 (3)	−1475 (2)	47 (1)
C(7)	−5511 (3)	−7506 (4)	−2270 (2)	58 (1)
C(8)	−7818 (3)	−9722 (4)	−47 (2)	65 (1)
C(9)	−7574 (3)	−4878 (3)	−565 (2)	54 (1)
C(10)	−8940 (2)	−7093 (3)	−886 (2)	49 (1)
N	−9867 (2)	−7297 (4)	−994 (2)	72 (1)
C(11)	−8505 (2)	−7644 (3)	−3039 (1)	49 (1)
C(12)	−8294 (3)	−9347 (4)	−3219 (2)	56 (1)
C(13)	−9091 (3)	−10282 (4)	−3636 (2)	68 (1)
C(14)	−10067 (4)	−9539 (5)	−3875 (2)	81 (1)
C(15)	−10280 (4)	−7864 (6)	−3690 (2)	89 (2)
C(16)	−9497 (3)	−6900 (5)	−3275 (2)	69 (1)
O(3)	−6886 (3)	−11486 (3)	−1895 (2)	83 (1)

Table 2. *Bond lengths* (Å) *and angles* (°)

O(1)—P	1.598 (2)	O(2)—P	1.470 (2)
C(1)—P	1.821 (2)	C(11)—P	1.792 (3)
C(7)—O(1)	1.466 (4)	C(2)—C(1)	1.532 (3)
C(6)—C(1)	1.535 (3)	C(3)—C(2)	1.567 (3)
C(9)—C(2)	1.550 (3)	C(10)—C(2)	1.468 (4)
C(4)—C(3)	1.499 (4)	C(8)—C(3)	1.530 (4)
C(5)—C(4)	1.320 (4)	C(6)—C(5)	1.487 (4)
C(7)—C(6)	1.521 (4)	N—C(10)	1.139 (3)
C(12)—C(11)	1.387 (4)	C(16)—C(11)	1.377 (4)
C(13)—C(12)	1.384 (4)	C(14)—C(13)	1.363 (6)
C(15)—C(14)	1.368 (6)	C(16)—C(15)	1.382 (5)

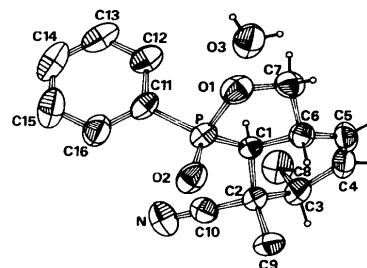|			
O(2)—P—O(1)	115.7 (1)	C(1)—P—O(1)	94.2 (1)
C(1)—P—O(2)	116.8 (1)	C(11)—P—O(1)	105.1 (1)
C(11)—P—O(2)	111.7 (1)	C(11)—P—C(1)	111.8 (1)
C(7)—O(1)—P	114.1 (2)	C(2)—C(1)—P	124.5 (2)
C(6)—C(1)—P	100.1 (2)	C(6)—C(1)—C(2)	111.2 (2)
C(3)—C(2)—C(1)	107.7 (2)	C(9)—C(2)—C(1)	112.5 (2)
C(9)—C(2)—C(3)	110.2 (2)	C(10)—C(2)—C(1)	108.8 (2)
C(10)—C(2)—C(3)	110.1 (2)	C(10)—C(2)—C(9)	107.6 (2)
C(4)—C(3)—C(2)	111.5 (2)	C(8)—C(3)—C(2)	114.2 (2)
C(8)—C(3)—C(4)	110.4 (2)	C(5)—C(4)—C(3)	126.2 (3)
C(6)—C(5)—C(4)	121.9 (3)	C(5)—C(6)—C(1)	110.2 (3)
C(7)—C(6)—C(1)	103.2 (2)	C(7)—C(6)—C(5)	117.9 (2)
C(6)—C(7)—O(1)	106.0 (2)	N—C(10)—C(2)	177.7 (3)
C(12)—C(11)—P	120.8 (2)	C(16)—C(11)—P	119.1 (2)
C(16)—C(11)—C(12)	120.1 (3)	C(13)—C(12)—C(11)	119.2 (3)
C(14)—C(13)—C(12)	120.6 (3)	C(15)—C(14)—C(13)	120.2 (3)
C(16)—C(15)—C(14)	120.4 (4)	C(15)—C(16)—C(11)	119.6 (3)

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the asymmetric unit showing the numbering scheme.

$C_{16}H_{18}NO_2P.H_2O$

Bertolasi (1989). For a related compound, 1,2,7a-trihydroxy-2-methylperhydro-1-phosphaindene 1-oxide, see Bartczak & Yagbasan (1991).

References

BARTCZAK, T. J. & YAGBASAN, R. (1991). *Acta Cryst.* C47, 1750–1752.
BODALSKI, R. & KRAWCZYK, H. (1991). Personal communication.
GŁÓWKA, M. L., KRAWCZYK, H., BODALSKI, R. & BERTOLASI, V. (1989). *J. Crystallogr. Spectrosc. Res.* 19, 701–705.
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.

3-Chloro-6-methoxy-5-tosylmethylpyridazine

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Abstract. $C_{13}H_{13}ClN_2O_3S$, $M_r = 312.77$, monoclinic, $P2_1/c$, $a = 12.365$ (2), $b = 5.524$ (1), $c = 21.646$ (4) Å, $\beta = 106.98$ (1)°, $V = 1414.0$ (4) Å^3, $Z = 4$, $D_x = 1.47$ (1) g cm^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 38.67$ cm^{-1}, $F(000) = 648$, $T = 293$ K, $R = 0.049$, $wR = 0.049$ for 1470 unique observed reflections. The conformation of the molecule as a whole can be described as *cis*-staggered. The tosylmethyl substituent is at C(5) in the pyridazine ring. The C(phenyl)—S—CH$_2$—C(pyridazine) torsion angle is 62.5 (2)°.

Experimental. Crystals of the title compound (I) were obtained from ethanol solution as small colourless elongated prisms. The crystal selected for data collec-

(I)
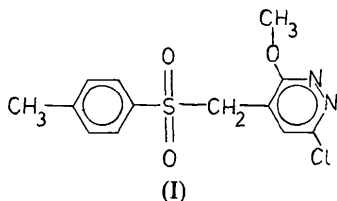
tion had dimensions $0.37 \times 0.18 \times 0.05$ mm. Measurements were carried out on a Syntex $P2_1$ diffractometer using graphite-monochromated Cu $K\alpha$ radiation. The unit-cell parameters were determined by least-squares fit to ten automatically centred reflections (2θ range 12.90–27.74°). Two control reflections were monitored after every 100 intensity measurements and showed no systematic variation in intensity throughout the data collection. The θ–2θ scan method with a variable scan speed (1.9–29.3° min^{-1}) was used. 2336 reflections were col-

lected up to $2\theta = 115.0°$, with indices h $0 \rightarrow 14$, k $0 \rightarrow 7$, l $-24 \rightarrow 24$. The background and integrated intensity for each reflection were calculated according to the profile-analysis method of Lehmann & Larsen (1974). Of 1874 unique reflections, 1471 reflections had $I \geq 1.96\sigma(I)$ and were used in the structure refinement. Only Lp corrections were applied. The structure was solved using *SHELXS*86 (MS DOS version) (Sheldrick, 1986). After the refinement of the non-H atoms with anisotropic temperature factors ($R = 0.073$) eight H atoms (of the two methyl groups and the methylene) were calculated from the geometry of the molecule (C—H = 1.08 Å) and assigned isotropic temperature factors of 0.06 Å^2 – their coordinates were recalculated after each cycle of refinement following the shifts of their carriers. The other H atoms were located from a ΔF map. They were included in the refinement with isotropic temperature factors. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$ where $w = [\sigma^2(F_o)]^{-1}$. The final cycles of the full-matrix least-squares refinement gave $R = 0.049$, $wR = 0.049$ and $S = 1.7$, for 1470 reflections – one reflection with a large $\Delta F/\sigma$ value was excluded from the last cycles. For all the refined parameters the final Δ/σ was smaller than 0.1; the maximum peak on the final ΔF map was 0.31, the minimum -0.32 e Å^{-3}. The final atomic parameters are listed in Table 1.* Atomic scattering factors incorporated in *SHELX*76 (Sheldrick, 1976) were