2,4,6-Tri-*tert*-butyl-1,3,5-triphosphinine (2a) was first prepared in 1995.2 Shortly thereafter, a simple one-pot synthesis comprising the cyclotrimerization of the phosphaalkynes 1 in the presence of a vanadium catalyst was developed that also made the isolation of other triphosphinines possible.1 Although their physicochemical properties (NMR spectroscopic data2,3, crystal structure analysis4) as well as theoretical investigations5 clearly demonstrated the aromatic character of the 1,3,5-triphosphinines 2, the compounds have been exhibiting a surprisingly high reactivity in, for example [4+2] cycloaddition reactions.6 When ethylene 3 was bubbled into a toluene solution of 2a, a [4+2] cycloaddition reaction occurred at room temperature to afford the 7,8-dihydro-1,3,5-triphosphabarrelenes 4.7 Various monosubstituted alkenes (acrylic acid derivatives, styrene), as well as some selected disubstituted alkenes (maleic acid derivatives, fumaric acid derivatives, norbornene, cyclopentadiene) also underwent Diels–Alder reactions with 2 to furnish comparable dihydrotriphosphabarrelenes. Alkenynes like acetylene 5 itself reacted with 2a to afford the novel triphospha cage compound 7 through a Diels–Alder/homo-Diels–Alder reaction sequence with the 1,3,5-triphosphabicyclo[2.2.2]octa-2,5,7-triene 6 as intermediate (Scheme 1).5

On account to their relatively weakly pronounced aromaticity, 1,3,5-triphosphinines should, in principle, be able to act as dipolarophiles in [3+2]-cycloaddition reactions. In order to test this assumption 1,3,5-triphosphinines 2a–c were allowed to react with 1,3-dipoles, namely phenyl- and mesitylnitrile oxides (Scheme 2). Dependent on the substituents R1 and R2, reactions of the 1,3,5-triphosphinines 2 with the nitrile oxides 8 in Et2O or toluene led either to the trisadducts 9 or proceeded with...
by a mesityl group (8b), the formation of trisadducts at all three P–C bonds of the 1,3,5-triphosphinine was no longer favored.

As shown in Figure 1, the reaction path branches presumably at the stage of the monocycloadduct A, because attack of the second nitrile oxide can occur from the C-5(\(\text{Si}\))-P-6(Re) or C-5(Re)-P-6(\(\text{Si}\)) side of the monoadduct. Addition of B to the double bond C-7-P-8 of A leads to the same types of intermediates and is therefore not further discussed here.

![Figure 1](image.png)

**Figure 1** Plausible intermediates in the 1,3-dipolar cycloaddition reaction of nitrile oxides towards 1,3,5-triphosphinines

When the C-5(\(\text{Si}\))/P-6(Re) side is accessible the reaction sequence leads to the cycloadduct B and ends at the tetracyclic species 9; when this side is shielded attack at the C-5(Re)/P-6(\(\text{Si}\)) side leads to the cycloadduct C with subsequent ring contraction under cleavage of the nitrile (\(\rightarrow\) D), and finally to the formation of product 11. With the 1-methylcyclopentyl and mesityl substituents both reaction paths are equally possible as an approximately 1:1 mixture of 9e and 11e was obtained.

The constitution of the trisadducts 9 could be deduced unambiguously from their spectroscopic data, which showed, in particular, that all three equivalents of nitrile oxides added to the 1,3,5-triphosphinine from the same side. Thus, all the 31P NMR spectra contained only one singlet signal confirming the high symmetry of the system 9. The chemical shifts were between \(\delta = 17.4\) and 30.9 ppm thus clearly indicating the location of three P-C double bonds. Compounds 9c and 9d were identified as minor products on the basis of their 31P NMR singlet resonances of \(\delta = 28.1\) and 29.0 ppm, respectively in the crude reaction mixtures. The \(J_P,P\) coupling constants of 9 were determined as simulation parameters of the 13C NMR data and amounted to between 1.8 and 9.4 Hz. The 13C NMR spectra contained only one signal between \(\delta = 97.6\) and 98.3 ppm for the three chemically equivalent atoms C-4a, C-8a, C-12a in agreement with the \(C_2\) symmetry. As shown by a simulation of the AA’AX’X spin system, all the signals of these skeletal carbon atoms possessed two characteristic \(J_{C,P}\) coupling constants of between 52.0 and 71.0 Hz, confirming the attachment to two phosphorus atoms. In addition, the chemically equivalent imine carbon atoms C-3, C-7, C-11 gave signals at typically low field between \(\delta = 156.4\) and 159.3 ppm, each with a characteristic \(J_{P,P}\) coupling constant ranging from 40.6 to 50.3 Hz, again demonstrating the direct attachment to a phosphorus atom and confirming the deduced direction of addition of the 1,3-dipole to the P-C double bond.

The cleavage of mesityl nitrile in the formation of the tricyclic compounds 11 was confirmed by correct elemental analysis results. Moreover, the IR spectrum of 11 revealed strong absorptions between 1671 cm\(^{-1}\) and 1674 cm\(^{-1}\) that could be assigned to the carbonyl vibrations of the newly formed P-acyl group. The phosphorus atoms P-4, P-5, P-9 of the central 1,2,4-triphosphophole appeared as an AMN spin system in the 31P NMR spectrum at about \(\delta = 50\) (P-4, P-5) ppm and \(\delta = 80\) (P-9) ppm. Further characteristic parameters were the large \(J_{P,P}\) coupling constants of between 255.8 and 264.1 Hz, demonstrating the direct attachment of P-4 and P-5, as well as typical \(J_{P,P}\) coupling constants of 10.7–23.4 Hz between P-4–P-9 on the one hand and P-5–P-9 on the other hand. Final confirmation of the structure of 11 was provided by an X-ray crystallographic analysis of 11c.

![Figure 2](image.png)

**Figure 2** Molecular structure of 11c; selected bond lengths [Å] and bond angles [°]: P4–P5 2.196(2), P4–C3 1.812(5), P5–C5a 1.895(5), C5–C51 1.888(6), C5–P9 1.848(5), P9–C9a 1.918(5), P9–C8 1.846(5), C9a–P 4 1.871(5), C9a–P4–P5 101.81(19), C9a–P4–C3 88.5(2), P4–P5–C5a 99.04(17), P4–P5–C51 86.93(18), C5a–P5–C51 104.1(2), C5a–P5–P9 114.2(3), C5a–P9–C9a 102.0(2), C5a–P9–C8 86.6(2), C9a–P9–C8 96.7(2), P9–C9a–P4 111.7(3)

Figure 2 clearly shows that the central 1,2,4-triphosphophole ring possessed a distorted envelope conformation, that the newly formed pivaloyl group on P-5 was located on the opposite side to the plane of the central five-membered ring as did the two condensed 4,5-dihydrooxazaphosphole rings, and that the oxygen of the carbonyl group was located below the five-membered ring.
In order to exclude the subsequent formation of 11 from 9 by cleavage of the nitrile, compound 9a was heated in toluene at 100 °C for 14 days. NMR spectroscopic control did not reveal any ring contraction, instead a quantitative [2+2+2]-cycloreversion to the previously known 5-tert-butyl-3-phenyl-1,2,4-oxazaphosphole (10)\(^{10,21}\) was observed.

When the tricyclic compounds 11 were subjected to a comparable thermolysis, an initial [3+2]-cycloreversion with liberation of mesitylnitrile oxide (8b) and formation of the bicyclic species 12 occurred with subsequent decomposition into the oxazaphospholes 13 and the oxadiphospholes 14 as indicated in Scheme 3.

Whereas the oxazaphospholes 13 are well known, oxadiphospholes 14 have as yet only been poorly investigated\(^{11,12}\) and this synthetic route has potential for exploitation as a specific access to this novel class of heterodiphospholes. Since control experiments showed that the oxadiphospholes 14 reacted with mesitylnitrile oxide under formation of compound 17 (Scheme 4), which reduced their yields or prevented their isolation, it seemed reasonable to remove the inevitably formed 1,3-dipole by means of a trapping reagent. It is well known that phosphaalkenes of the Becker type react with nitrile oxides via the 4,5-dihydro-1,2,4-oxazaphospholes 16 and cleavage of hexamethyldisiloxane to afford oxazaphospholes of the type 13,\(^{10,13,20}\) Thus, when the thermolysis of compounds 11 was performed in the presence of the phosphaalkene corresponding to the employed compound 11, we could indeed isolate the oxadiphospholes 14 in good yields of 68–71%. It was even possible to carry out the synthesis of 14 as a one-pot process starting with 2, three equivalents of 8b, and one equivalent of the respective phosphaalkene. As shown for the example of 14a it is possible to obtain this heterocyclic species by this procedure in yields between 60–65%.

The constitutions of the novel oxadiphospholes 14a–c were confirmed by correct elemental analysis results, the presence of molecular ion peaks in their EI-mass spectra, and examination of their spectroscopic data. Hence, each \(^{31}\)P NMR spectra each a signal at low field by \(\delta = 313.9\) and 316.4 ppm for the phosphorus atom P-2 directly attached to the oxygen atom, whereas the signal for P-4 was appreciably shifted to higher field and appeared between \(\delta = 122.0\) and 127.3 ppm. The \(^2J_{PP}\) coupling constants were on average 24.0 Hz. These and all other NMR data were in complete accord with those of the previously reported 3,5-dimesityl derivative (14, \(R =\) Mes)\(^{12}\) (see also experimental section). Finally, the selective reaction of 14a with mesitylnitrile oxide should be mentioned. Even under mild conditions two equivalents of mesitylnitrile oxide (8b) underwent addition to the two P–C double bonds of 14a to furnish in high diastereo- and regioselectivity the heterocyclic species 17 which was isolated in the form of colorless needles in 90% yield. The spectroscopic data of 17 were closely compatible with those of the already known analogue in which the oxygen atom in position 5 is replaced by sulfur.\(^{22}\) The only significant differences were observed for P-4 and C-5a, i.e., the atoms directly adjacent to the oxygen O-5 or sulfur S-5, respectively. The signals for both atoms in the corresponding spectra of 17 were markedly shifted to lower field (P-4: \(\delta = 156.7\) vs. 78.3 ppm; C-5a: \(\delta = 140.0\) vs. 125.8 ppm). The proposed relative stereochemistry in
which both equivalents of 8b added from the same side to 14a is plausible under consideration of the known angle-dependency of the J_{C,P} coupling constants.\textsuperscript{14} They were 23.5 and 24.8 Hz for the quaternary carbon atoms of the tert-butyl groups bonded to C-5a and C-9a, respectively, thus supporting the cis-orientation of these groups to the free electron pairs on the phosphorus atoms P-4 and P-9.

When 17 was subjected to thermolysis under the same conditions as employed for compounds 11 above, decomposition was complete within 75 minutes and the known compound 13a\textsuperscript{15} was formed quantitatively. The fate of the thus liberated oxygen was not followed.

All reactions were carried out under an Ar atmosphere in oven-dried glassware. The solvents were anhydrous and stored under Ar. \textsuperscript{1}H NMR spectra were measured on Bruker AMX-400 spectrometer and the chemical shifts are referenced to the solvent as internal standard. \textsuperscript{31}P NMR spectra were recorded on Bruker AC 200 spectrometer. MS (EI) was performed on Finnigan MAT 90 instrument. All reactions were carried out under an Ar atmosphere in oven-dried glassware.

To compound 2c (348 mg, 0.92 mmol) and Et₃N (279 mg, 2.76 mmol) in EtO (15 mL), was additionally dried with a solution of N-hydroxybenzene carboximidoyl chloride (429 mg, 2.76 mmol) in EtO (15 mL) under magnetic stirring at 0 °C. The solution was allowed to warm to r.t., the reaction mixture was filtered through Celite and the residue rinsed twice with toluene (10 mL). After removal of the solvent under oil pump vacuum (10⁻³ mmbar/25 °C), the residue was taken up in THF–EtO (1:1) and crystallization at −25 °C afforded 9b. Yield: 482 mg (0.66 mmol, 72%); colorless crystals; mp 173 °C (decomp).

IR (KBr): 3054 (w), 3026 (w), 2960 (vs), 2871 (s), 1598 (w), 1579 (m), 1459 (m), 1461 (m), 1434 (s), 1380 (s), 1323 (m), 1284 (w), 1268 (m), 1232 (m), 1202 (w), 1178 (w), 1118 (w), 1073 (m), 1049 (w), 1020 (m), 977 (w), 934 (w), 908 (m), 884 (s), 859 (s), 760 (vs), 695 (vs), 675 (m), 655 (m), 629 (m), 602 (s), 498 (m) cm⁻¹.

\textsuperscript{1}H NMR (CDCl₃): δ = 1.28 (s, 9 H, cyclohexyl-CH₂), 1.30–1.60 (m, 24 H, H-cyclohexyl), 7.42–7.48 (m, 9 H, H-phenyl), 7.67–7.73 (m, 6 H, H-phenyl).

\textsuperscript{13}C{\textsuperscript{1}H} NMR (CDCl₃): δ = 22.7, 23.4 (each s, C-3’-cyclohexyl, C-4’-cyclophenyl), 23.4 (AA’XX spin system, J_{C,P} = 15.7 Hz, 15.7 Hz), 17.6 (J_{C,P} = 9.0 Hz, 1’-CH₁-cyclohexyl), 36.0 (AA’XX spin system, J_{C,P} = 9.5 Hz, 7.3 Hz, J_{C,P} = 0.3 Hz, C-2’- or C-2’-cyclophenyl), 36.4 (AA’XX spin system, J_{C,P} = 9.8 Hz, 7.4 Hz, J_{C,P} = 0.2 Hz, C-2’- or C-2’-cyclophenyl), 54.4 (AA’XX spin system, J_{C,P} = 24.2 Hz, 21.8 Hz, J_{C,P} = 1.0 Hz, C-1’-cyclophenyl), 98.1 (AA’XX spin system, J_{C,P} = 62.9 Hz, 52.9 Hz, J_{C,P} = 0.5 Hz, C-4a, C-8a, C-12a), 128.4 (s, meta-C-phenyl), 129.2, 129.3 (each s, ortho-C-C-phenyl), 132.9 (AA’XX spin system, J_{C,P} = 18.8 Hz, J_{C,P} = 0.0 Hz, 0.0 Hz, ipso-C-phenyl), 159.3 (AA’XX spin system, J_{C,P} = 41.1 Hz, J_{C,P} = 0.9 Hz, 0.9 Hz, C-3, C-7, C-11); simulation parameter of AA’XX spin systems: J_{C,P} = 1.9 Hz.

\textsuperscript{31}P{\textsuperscript{1}H} NMR (CDCl₃): δ = 174.4 (s, P-4, P-8, P-12).

MS (FAB): m/z = 736 [M + H]\textsuperscript{+}.


Reaction of 2a–c with Mesityl nitride oxide (8b); General Procedure

To a solution of the 1,3,5-triphosphinines 2a, 2b, and 2c in toluene was added drop by drop a solution of a three-fold amount of mesitylnitrite oxide (8b) in toluene at −78 °C. After warming to r.t., all volatile materials were removed under oil pump vacuum (10⁻³ mmbar/25 °C) and the residue was worked up as described for the individual case.

5a,9a-Di-tert-butyl-3,8-dimesityl-5-(2,2-dimethylpropanoyl)-5,5a-dihydro[1,2,4]oxazaphosphole[4,5-\textit{d}]/[1,2,4]oxazaphosphole[11e]; Typical Procedure

The above-mentioned compound was prepared from 1,3,5-triphosphinines 2a (1.55 g, 5.17 mmol) in toluene (10 mL) and 8b (2.5 g, Synthesis 2004, No. 2, 241–248 © Thieme Stuttgart · New York.
15.5 mmol) in toluene (10 mL). After bulb-to-bulb distillation was performed (50–70 °C/10 mbar) to remove the formed mesitylinene, the oily crude product was worked up by column chromatography (silica gel, toluene) and crystallized from toluene–n-pentane (1:4) at −25 °C. Yield: 2.29 g (3.99 mmol, 77%); colorless to yellowish low-molecular-weight crystal powder; mp 154 °C (decomp).

IR (KBr): 2966 (vs), 2918 (s), 2879 (m), 1674 (s), 1470 (s), 1430 (s), 1398 (s), 1342 (s), 1324 (s) cm−1.

1H NMR (CD2Cl2): δ = 1.01, 1.12, 1.20 (each s, 3 H, C(CH3)3), 0.88 (t, JHC = 7.1 Hz, 3 H, CH2CH3CH2CH3), 0.95 (t, JHC = 7.7 Hz, 3 H, CH2CH3), 1.01, 1.07 (each s, 3 H, C(CH3)2CH2CH3), 1.23 (each s, 3 H, C(CH3)2CH2CH3), 1.30–1.90 (m, 6 H, C(CH3)2CH2CH3), 2.33, 2.46 (each s, 6 H, CH2CH3CH2CH3), 2.71, 2.74 (each s, 3 H, para-CH3-mesityl), 6.93, 7.04 (each s, 1 H, H-mesityl), 6.95 (s, 2 H, H-mesityl).

13C{1H} NMR (CD2Cl2): δ = 14.6 (s), 1443 (s), 1422 (m), 1393 (m), 1351 (s), 1324 (s), 1302 (s), 1219 (w), 978 (w), 941 (w), 918 (m), 811 (m), 796 (w), 776 (w), 733 (w) cm−1.

13C NMR (CD2Cl2): 15.5 (s), 1474 (s), 1459 (s), 1393 (m), 1364 (s), 1324 (s), 1302 (s), 1219 (w), 978 (w), 941 (w), 918 (m), 811 (m), 796 (w), 776 (w), 733 (w) cm−1.

11C NMR (CD2Cl2): 22.4 (s, ortho- or para-CH3-mesityl), 22.5 (broad s, C(CH3)2CH2CH3), 22.9 (d, JCP = 22.2 Hz, ortho- or para-CH3-mesityl), 23.8 (m, [C(CH3)2CH2CH3], 26.3 (m, [C(CH3)2CH2CH3], 26.6 (m, [C(CH3)2CH2CH3], 31.7 [br m, C(CH3)2CH2CH3], 31.4 [br m, C(CH3)2CH2CH3], 34.8 [br m, C(CH3)2CH2CH3], 43.3, 43.5, 43.9 [each m, (C(CH3)2CH2CH3), 54.5 [m, 1.0 JCH = 1.0 Hz, 13.8 Hz, iso-C5-mesityl], 129.1, 129.7, 130.7, 137.5, 138.6, 138.9 (each s, CH3-mesityl), 140.0 (d, JCP = 2.3 Hz, C5-mesityl), 155.9 (d, JCP = 34.5 Hz, C3 or C-8), 162.1 (d, JCP = 46.0 Hz, C3 or C-8), 223.4 (d, JCP = 52.9 Hz, CO).

MS (FAB): m/z = 681 [M + H]+.

11e IR (KBr): 2958 (vs), 2866 (s), 2866 (m), 1673 (s, C=O), 1609 (m), 1442 (br), 1420 (w), 1376 (m), 1294 (w), 1036 (w), 977 (m), 952 (m), 909 (w), 858 (w), 849 (m), 733 (m), 624 (w), 550 (w) cm⁻¹.

1H NMR (CDCl₃): δ = 1.17, 1.23, 1.26 (each s, 3 H, cyclopentyl-CH₃), 1.27–2.27 (24 m, H-4, H-5-cyclopentyl), 2.30, 2.31 (each s, 3 H, ortho-CH₃-mesityl), 2.42 (s, 6 H, ortho-CH₃-mesityl), 2.63, 2.69 (each s, 3 H, para-CH₃-mesityl), 6.91 (s, 1 H, H-mesityl), 6.92 (s, 2 H, H-mesityl), 7.01 (s, 1 H, H-mesityl).

13C [1H] NMR (CDCl₃): δ = 20.7 (d, JCP = 7.5 Hz, ortho- or para-CH₃-mesityl), 20.8 (s, ortho-CH₃-mesityl), 20.9, 22.2 (each s, ortho- or para-CH₃-mesityl), 22.9, 24.0, 24.7, 25.1, 25.3 (each s, C-3’- and C-4’-cyclopentyl), 23.0 (d, JCP = 22.4 Hz, ortho- or para-CH₃-mesityl), 24.1 (m, 1CH₃-cyclopentyl), 25.0 (dd, JCP = 10.8 Hz, JCP = 6.6 Hz, 1’-CH₃-cyclopentyl), 25.7 (pseudo t, JCP = 10.4 Hz, 1’-CH₃-cyclopentyl), 36.7, 36.9 (each pseudo t, JCP = 3.7 Hz, JCP = 4.1 Hz, respectively, C-2’- or C-5’-cyclopentyl), 37.0 (br m, C-3’- or C-5’-cyclopentyl), 37.3 (br s, C-2’- or C-5’-cyclopentyl), 38.5 (d, JCP = 5.0 Hz, C-2’- or C-5’-cyclopentyl), 40.7 (br s, C-2’- or C-5’-cyclopentyl), 51.6 (dd, JCP = 26.1 Hz, 24.5 Hz, C-1’-cyclopentyl), 53.5 (m, C-1’-cyclopentyl), 62.3 (JCP = 29.0 Hz, C-1’-cyclopentyl), 116.1 (br m, C-5a or C-9a), 122.2 (br m, C-5a or C-9a), 125.7 (d, JCP = 15.8 Hz, JCP = 14.4 Hz, ipso-c-mesityl), 126.7 (d, JCP = 14.1 Hz, ipso-c-mesityl), 129.0, 129.7, 130.6, 137.5, 138.7, 138.9 (each s, C-mesityl), 139.9 (d, JCP = 2.5 Hz, C-mesityl), 156.2 (d, JCP = 39.8 Hz, C-3 or C-8), 162.1 (d, JCP = 44.8 Hz, C-3 or C-8), 222.7 (d, JCP = 57.2 Hz, C≡O).

31P [1H] NMR (CDCl₃): δ = 53.5, 57.9 (AMN spin system, JCP = 255.8 Hz, JCP = 208.8 Hz, JCP = 10.7 Hz, P-4, P-5), 84.4 (AMN spin system, JCP = 20.8 Hz, JCP = 10.7 Hz, P-9).

MS (FAB): m/z = 717 [M + H]⁺.

Anal. Calcld for C₁₆H₂₉N₂O₃P₂: C, 63.89; H, 7.73; N, 3.91. Found: C, 63.91; H, 7.60; N, 3.88.

3.5-Di-tert-butyl-1,2,4-oxadiphosphole (14a): Typical Procedure

Variation A (thermolysis of 11e): A pressure-Schlenk tube was charged with a solution of 11e (433 mg, 0.67 mmol) in toluene (5 mL) and heated for 35 h at 100 °C. The work up was performed as described under variation A. Yield: 104 mg (0.48 mmol, 71%), colorless oil.

Variation B (co-thermolysis of 11c with the phoshakaene 15a): A pressure-Schlenk tube was charged with a solution of 11c (433 mg, 0.68 mmol) and phoshakaene 15a (178 mg, 0.68 mmol) in toluene (5 mL) and heated for 35 h at 100 °C. The work up was performed as described under variation A. Yield: 104 mg (0.48 mmol, 71%), colorless oil.

Variation C (one-pot synthesis starting from 2a): In a pressure-Schlenk tube a solution of 8b (2.04 g, 12.7 mmol) in toluene (7 mL) was added to a solution of 2a (1.27 g, 4.22 mmol) in toluene (8 mL) at −78 °C. The mixture was allowed to warm up to r.t. and was then stirred at this temperature for 1 h. To this mixture the phoshakaene 15a (1.11 g, 4.22 mmol) was added and the mixture was heated for 35 h at 100 °C. The work up was performed as described under variation A. Yield: 566 mg (2.62 mmol, 62% related to 2a), colorless oil.
1H NMR (CD₂Cl₂): δ = 1.39 (d, JHH = 1.2 Hz, 3 H, 5-CH₂-cyclopentyl), 1.40 (d, JHH = 1.7 Hz, 3CH₂-cyclopentyl), 1.49–1.81 (m, 12 H, CH₂-cyclopentyl), 2.09–2.32 (m, 6 H, CH₂-cyclopentyl).

13C{1H} NMR (C₆D₆): δ = 24.4 (dd, JCP = 6.4 Hz, 5a-C-2), 22.2 (s, 3H, para-C₃H₃-mesityl), 25.2 (d, JCP = 4.5 Hz, 9a-C-₃), 24.4 (dd, 3JCP = 31.3 (d, 2H, ortho-C₃H₃-mesityl), 32.1 (s, 9H, C(CH₃)₃), 32.0, 247.1 (d, JCP = 0.8 Hz, C₃-P₃), 32.1 (s, 9H, C(CH₃)₃), 44.2 (dd, 2JCP = 5.1 Hz, 3-C₂-cyclopentyl), 47.6 (dd, 2JCP = 15.7 Hz, JCP = 13.1 Hz, 3-C-1’-cyclopentyl), 51.8 (dd, 2JCP = 13.6 Hz, JCP = 2.1 Hz, 2-C₃-1’-cyclopentyl), 210.1 (dd, JCP = 69.1 Hz, JCP = 64.0 Hz, C-3), 222.9 (dd, 2JCP = 63.8 Hz, JCP = 6.1 Hz, C-5).

31P{1H} NMR (CD₂Cl₂): δ = 124.0 (d, JPH = 23.7 Hz, P-2), 315.1 (d, JPH = 3.6 Hz, P-4).


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(23) Crystal data (excluding structure factors) for the structure reported in this paper has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-211047. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, U.K. [Fax: +44(1223)336033; E-mail: deposit@chemcrys.cam.ac.uk].