Synthesis and structure of cyclic phosphine–boranes

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Abstract

In a search for cyclic phosphine–boranes, which can be expected to undergo ring opening polymerisation, or decomposition, to leave boron phosphide, the hydroboration reaction of ω-alkenyl-diphenylphosphines with boranes was investigated. Addition of one equivalent of borane (introduced as its tetrahydrofuran adduct) to phosphines $H_2C=CH-(CH_2)_n-PPh_2$ ($n = 0$, 1, 2) gave the corresponding phosphine–boranes $H_2C=CH-(CH_2)_n-PPh_2BH_3$ without any further cyclisation. With an excess of $(thf)BH_3$, hydroboration of the olefinic function occurred, but again no ring closure. Treatment of the alkenylphosphines with 9-borabicyclononane gave the expected cyclic phosphine–boranes $(C_8H_{14})B-(CH_2)_n-PPh_2$ ($n = 3$, 4) in good yield as crystalline products. The structures of the two heterocycles have been determined by X-ray diffraction methods. They show the expected strain-free ring systems with short P–B bonds, comparable with the isoelectronic Si–C systems.

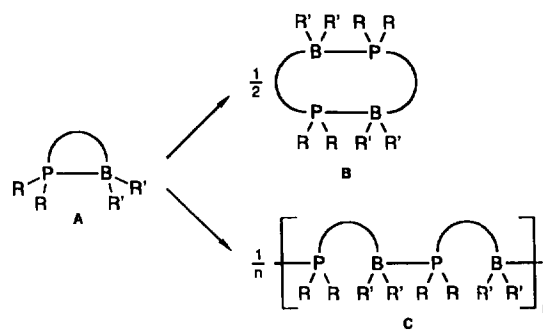*Keywords:* Phosphine–boranes; Hydroboration; Alkenylphosphines; Borabicyclononane; 1,2-Boraphosphacycloalkanes

1. Introduction

Phosphine–boranes of the general formula R_3P-BH_3 are an intriguing class of compounds [1]. It has already been noted in the earliest studies that the P–B donor–acceptor bond is unusually strong and that the B–H bonds appear to show signs of an 'Umpolung' [2]. The adducts of tertiary phosphines with monoborine BH_3 are stable to oxidation and hydrolysis even in strongly acidic media. Concentrated nitric acid is required for a total destruction of the compounds.

These properties are modified in derivatives with different numbers and types of substituents at phosphorus and/or boron ($R_3P-BH_mR'_{3-m}$), and generally more reactive species are obtained. These were found to have extensive synthetic potential in organic chemistry, and a large number of very useful preparations have been developed with phosphine–boranes as key reagents [3]. The role of phosphine–boranes as ligands in inorganic and organometallic chemistry is more limited, but is currently attracting growing interest [4].

The present study was initiated in order to provide access to cyclic phosphine–boranes, which in the past were considered only sporadically [5–8]. Heterocycles of this type (A) can be expected to be stable regarding

the underlying P–B bond (above), but these bonds may also re-form in thermal ring polymerisation reactions to give large ring oligomers or polymers (B, C). More drastic thermolysis could lead to boron phosphides $(BP)_n$, provided that the organic components of the heterocycle or polymer are lost in an efficient process of elimination of olefins and other hydrocarbons.

2. Results

The most promising general synthetic concept for the formation of phosphine–borane heterocycles is offered

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by an internal hydroboration of a phosphine–borane bearing an organic group with a terminal olefinic function at phosphorus. These compounds should be accessible through BH_3 addition to the corresponding ω-alkenylphosphine. Since a maximum stability is expected for five- and six-membered rings, 2-propenyl- (1) and 3-butenyl-diphenylphosphine (2) were chosen as the synthons. Both phosphines can be obtained via standard Grignard reactions from chlorodiphenylphosphine (1) [9] or from the bromoalkene and potassium diphenylphosphide (2) [10]. The vinyl derivative (3), a possible precursor for the four-membered ring, is also generated via the Grignard route [11].

All three alkenyl phosphines are readily converted into their phosphine–boranes by treatment with $(thf)BH_3$ [12].

The products (4–6) have been characterised by their analytical and spectroscopic data, confirming or complementing literature values where available [3].

All attempts to induce internal hydroboration in compounds 4–6 were unsuccessful. No cyclic product could be obtained even on prolonged thermal treatment. Under mild conditions no reactions took place, while under more forcing conditions extensive decomposition finally ensued. There was also no evidence for a clean formation of phosphine–borane polymers.

Treatment of compound 5 with an excess of $(thf)BH_3$ led only to the formation of the hydroboration product (7), with one equivalent of BH_3 added (anti-Markovnikoff) to the terminal double bond of the 3-butenyl group (Eq. (1)). Addition of additional BH_3 (from $(thf)BH_3$) is clearly preferred over internal hydroboration (from the $-PPh_2BH_3$ group in 5).

Ph₂P⌒⌒⧵⧵ + H₃B-THF ⟶ Ph₂P⌒⌒⌒⌒-BH₂-THF
  |BH₃   5 |BH₃   7

Ph₂P⌒⌒⌒⌒-BH₂-THF ⫽ ⬡ + H₃B-THF
  |BH₃   7   Ph₂P–BH₂

$$(1)$$

Based on the idea that a bulky and inflexible set of substituents at boron would reduce the acceptor properties at boron and favour ring systems over polymer structures, the attempted hydroboration reactions were extended to the cage-type 9-borabicyclononane (9-BBN) system. Since 9-BBN is known to be a good hydroborating reagent [12], the hydroborative cyclisation of 1 and 2 should take the expected course: instead of an irreversible initial step of complexation at the phosphorus atom with BH_3 (with no internal hydroboration to follow), this complexation could be reversible with 9-BBN, and the irreversible hydroboration at the olefin end of the substrate molecules could become reaction

determining. Internal P–B complexation (cyclisation) would simply be the last, entropy-favoured step, irrespective of the lower enthalpy (and reversible nature) of the donor–acceptor reaction.

Equimolar quantities of 1 or 2 and 9-BBN were found to react virtually quantitatively in boiling tetrahydrofuran (thf) in a matter of less than 1 h (Eq. (2)).

Ph₂P⌒⌒⫽ + 9-BBN ⟶ [structure 8]

Ph₂P⌒⌒⧵⧵ + 9-BBN ⟶ [structure 9]

$$(2)$$

The products can be isolated as colourless crystals in ca. 90% yield, m.p. 115 °C (8), m.p. 140 °C (9). They are soluble in diethyl ether, thf and di- or trichloromethane. The solutions are not very sensitive to air and moisture, and the crystals decompose only very slowly when exposed to the atmosphere. The compounds are readily identified by their analytical data, as well as by mass spectrometry and ^1H, ^{11}B, ^{13}C, and ^{31}P NMR spectroscopy (Section 3). The reduced number of inequivalent carbon atoms of the bicyclononane skeleton is indicative of a ring opening and recombination process (at the P–B bond) rising the virtual local symmetry level from point group C_s to C_{2v}.

Details of the molecular structures of both compounds were elucidated by single crystal X-ray diffraction methods.

Crystals of compound 8 (from diethyl ether) are orthorhombic, space group $Pna2_1$ with $Z = 4$ formula units in the unit cell. The molecules show no unusual intermolecular interactions. The five-membered ring is in an envelope conformation, with the middle carbon atom (C10, Fig. 1) out of the plane of the other four ring atoms. The P–B bond length of 2.072(3) Å is longer than comparable parameters of open-chain phosphine–boranes, suggesting a weakening of this bond through conformational strain. The ring C–C bonds have standard single bond distances (C9–C10 1.520(4) Å, C10–C11 1.541(4) Å), and the C–B bond (C11–B 1.628(4) Å) is not unusual for tetracoordinate boron atoms. It thus appears that the ring system of compound 8 is slightly strained and the strain is affecting predominantly the P–B donor–acceptor bond. This bond is clearly elongated compared with standard reference data [1] and in the homologous six-membered ring compound (9, below).
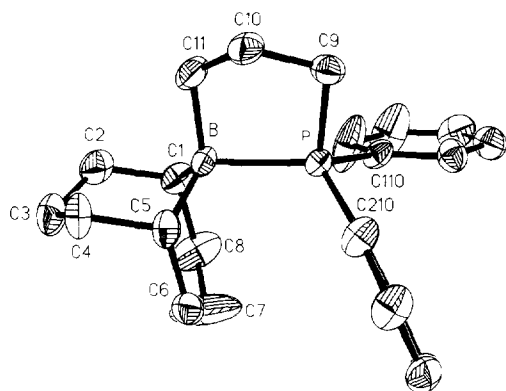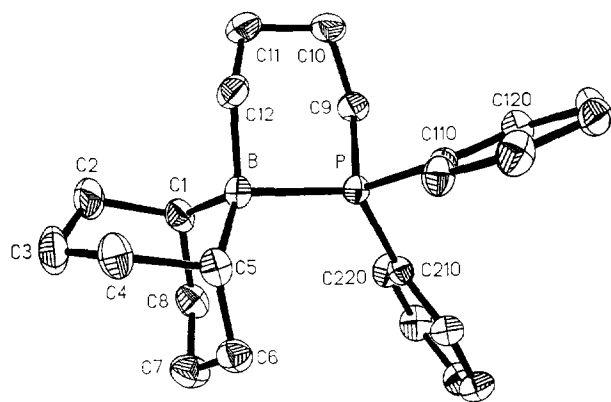
Fig. 1. Molecular structure of compound **8** with atomic numbering (ORTEP; 50% probability ellipsoids). Selected bond lenghts (Å) and angles (deg) are as follows: P–B, 2.072(3); P–C9, 1.828(2); P–C110, 1.816(2); P–C210, 1.812(2); C9–C10, 1.520(4); C10–C11, 1.541(4); B–C11, 1.628(4); B–C1, 1.621(3); B–C5, 1.628(4); C1–C2, 1.543(4); C2–C3, 1.521(5); C3–C4, 1.563(4); C4–C5, 1.543(3); C5–C6, 1.543(4); C6–C7, 1.463(5); C7–C8, 1.429(5); C8–C1, 1.530(4); C110–P–C210, 104.6(1); C110–P–C9, 104.9(1); C110–P–B, 123.2(1); C9–P–C210, 106.7(1); C210–P–B, 118.1(1); C9–P–B, 96.9(1); C1–B–C5, 105.6(2); C1–B–C11, 115.4(2); C5–B–C11, 116.5(2); C1–B–P, 117.3(2); C5–B–P, 107.1(2); C11–B–P, 94.8(2); C1–C2–C3, 114.6(2); C2–C3–C4, 113.8(2); C3–C4–C5, 113.4(2); C4–C5–C6, 111.2(2); C5–C6–C7, 117.3(2); C6–C7–C8, 125.0(3); C7–C8–C1, 118.0(3); C8–C1–C2, 112.2(2); P–C9–C10, 106.3(2); C9–C10–C11, 106.7(2); C10–C11–B, 111.8(2).

P–B bonds in phosphine–boranes are generally longer than the bonds in isoelectronic Si–C systems [13]; this is surprising, since with almost equal sums of

Fig. 2. Molecular structure of compound **9** with atomic numbering (ORTEP; 50% probability ellipsoids). Selected bond lenghts (Å) and angles (deg) are as follows: P–B, 2.029(2); P–C9, 1.824(1); P–C110, 1.821(1); P–C210, 1.826(1); C9–C10, 1.530(2); C10–C11, 1.524(2); C11–C12, 1.535(2); B–C12, 1.643(2); B–C1, 1.632(2); B–C5, 1.625(2); C1–C2, 1.542(2); C2–C3, 1.535(2); C3–C4, 1.533(2); C4–C5, 1.547(2); C5–C6, 1.531(2); C6–C7, 1.532(2); C7–C8, 1.534(2); C8–C1, 1.539(2); C110–P–C210, 102.24(6); C110–P–C9, 104.51(7); C110–P–B, 115.67(6); C9–P–C210, 104.23(7); C210–P–B, 125.17(6); C9–P–B, 102.90(7); C1–B–C5, 104.9(1); C1–B–C12, 114.7(1); C5–B–C12, 115.0(1); C1–B–P, 108.5(1); C5–B–P, 115.7(1); C12–B–P, 98.22(9); C1–C2–C3, 114.0(1); C2–C3–C4, 114.9(1); C3–C4–C5, 116.0(1); C4–C5–C6, 112.8(1); C5–C6–C7, 114.7(1); C6–C7–C8, 114.8(1); C7–C8–C1, 116.3(1); C8–C1–C2, 112.4(1); P–C9–C10, 111.8(1); C9–C10–C11, 113.9(1); C10–C11–C12, 113.4(1); C11–C12–B, 117.7(1).

the covalent radii (P–B vs. Si–C) the formal charges at the phosphorus and carbon atoms could mean a strengthening (and shortening) of the $^{(+)}P–B^{(-)}$ linkage. Obviously, contributions from such 'semipolar double bonds' appear to be small, or at least do not show in the structural parameters.

Crystals of compound **9** are triclinic, space group $P\bar{1}$, with $Z = 2$ formula units per unit cell. There are no unusual intermolecular contacts in the lattice. The six-membered ring has a chair conformation (Fig. 2) with the B- and P-bonded carbon atoms (C12, C9) above and below the plane of the atoms B, P, C10, C11. The P–B bond (2.029(2) Å) is shorter than in the lower homologue **8**, while the endocyclic B–C bond (B–C12 1.643(3) Å) is longer than the corresponding bond in **8**. The ring C–C bonds show no such anomalies.

The reaction of 9-BBN with diphenyl(vinyl)phosphine did not produce the expected boraphosphetane. The ^{31}P NMR spectrum of the reaction mixture (in thf) showed peaks at $\delta = 36$ and 7.6 ppm, which could be assigned to phosphine–boranes of various ring sizes, but attempts to isolate the products met with failure owing to extensive decomposition (Eq. (3)).

(3)
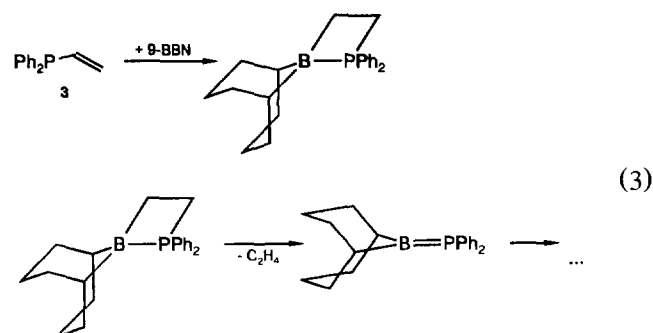
The observed low-field shift of the ^{31}P NMR signals of the decomposed material could arise from phosphino–boranes generated by loss of olefin from the boraphosphetane intermediate [14].

3. Experimental

3.1. General

All experiments were carried out routinely in an atmosphere of purified dry nitrogen. Solvents were dried and kept under nitrogen, and glassware was oven-dried and filled with nitrogen. Starting materials were either commercially available or were prepared following literature methods.

3.2. Allyl(diphenyl)phosphine–borane, 4

A solution of allyl(diphenyl)phosphine (**1**, 0.57 g, 2.5 mmol) in thf (3 ml) is treated with 2.5 ml of a 1 M

solution of $(C_4H_8O)BH_3$ in thf. After 1 h at 20 °C the solvent is removed from the reaction mixture in a vacuum. The residue is washed twice with pentane to remove unreacted phosphine. Yield 0.51 g (85%), colourless oil. ^1H NMR $(CDCl_3, 23 °C)$: $\delta = 3.06$, dd, $J(P,H) = 13$ Hz, $J(H,H) = 7$ Hz, 2H, PCH_2; 5.08, m, 2H, CH_2 =C; 5.78, m, 1H, CCHC; 7.45–7.67, m, 10H, Ph. $^{11}B\{^1H\}$ NMR (v.s.): $\delta = -39.6$, d, $J(P,B) = 49$ Hz. $^{31}P\{^1H\}$ NMR (v.s.): $\delta = 15.9$, br s. $^{13}C\{^1H\}$ NMR (v.s.): $\delta = 31.8$, d, $J(P,C) = 35$ Hz, PCH_2; 120.3, d, $J(P,C) = 11$ Hz, CCH_2; 128.2, d, $J(P,C) = 6$ Hz, CH; 128.6 (10), 128.7 (55), 131.2 (3), 132.3 (8) for $\delta(J)$ of meta-, ipso-, para-, and ortho-Ph carbon atoms. MS (EI): $m/z = 226$, M − BH_3; 183, M − $BH_3 - C_3H_6$. Anal. Found: C, 74.58; H, 7.92. $C_{15}H_{18}BP$ (240.09). Calc.: C, 75.04; H, 7.56%.

3.3. 3-Butenyl(diphenyl)phosphine–borane, 5

As described for **4**, with 3-butenyl(diphenyl)phosphine (**2**, 1.06 g, 4.16 mmol) and 4.2 ml of 1 M (thf)BH_3 in thf solution (10 ml). Yield 0.98 g (85%), colourless oil. ^1H NMR (v.s.): $\delta = 2.20$, m, 2H, $PCCH_2$; 2.37, dt, $J(P,H) = 11$ Hz, $J(H,H) = 7$ Hz, 2H, PCH_2; 4.80, m, 2H, CH_2 =C; 5.62, m, 1H, CH. $^{11}B\{^1H\}$ NMR (v.s.): $\delta = -40.2$, d, $J(P,B) = 52$ Hz. $^{31}P\{^1H\}$ NMR (v.s.): $\delta = 16.4$, br s. MS(EI): $m/z = 239$, M − BH_3; 183, M − $BH_3 - C_4H_8$. Anal. Found: C, 75.33; H, 7.74. $C_{16}H_{20}BP$ (254.12). Calc.: C, 75.63; H, 7.93%.

3.4. Diphenyl(vinyl)phosphine–borane, 6

This compound was prepared from phosphine **3** and (thf)BH_3. It has been fully characterised in previous studies [3].

3.5. 3-Boranylbutyl(diphenyl)phosphine–borane(thf), 7

Compound **5** (1.06 g, 4.16 mmol) is dissolved in thf (10 ml) and treated at −50 °C with 4.2 ml of a 1 M solution of (thf)BH_3. The reaction mixture is allowed to warm to room temperature over a period of 2 h, and the solvent is removed in a vacuum. The residue is dissolved in dichloromethane and precipitated by addition of pentane. Yield 0.66 g (47%), colourless oil, b.p. 135 °C/0.05 Torr. ^1H NMR $(CDCl_3, 23 °C)$: $\delta = 2.20$, br s, 2H, PCH_2; 7.42–7.66, m, 10H, Ph; 3.64, m, 4H, OCH_2; 0.9–1.8, m, 10H. $^{11}B\{^1H\}$ NMR (v.s.): $\delta = -40.1$, br s, BH_3; 19.0, br s, BH_2R. $^{31}P\{^1H\}$ NMR (v.s.): $\delta = 15.7$, br s. MS (EI): $m/z = 253$, M − BH_3 − thf; 183, M − BH_3 − thf − C_4H_8. Anal. Found: C, 69.97; H, 9.75. $C_{16}H_{23}B_2P \cdot C_4H_8O$ (340.08). Calc.: C, 70.64; H, 9.20%.

3.6. cyclo-[(9-Borabicyclo[3.3.1]nonanyl)propyl(diphenyl)phosphine], 8

9-BBN (4.7 ml of 0.5 M thf solution) was added to a solution of allyl (diphenyl) phosphine (**1**, 0.53 g, 2.34 mmol) in 5 ml of thf. After refluxing for 1 h the solvent was removed under reduced pressure and the product was recrystallised from diethyl ether. Yield 0.75 g (93%) colourless crystals, m.p. 115 °C. ^1H NMR $(CD_2Cl_2, 23 °C)$: $\delta = 1.06$, br m, 4H, CH_2CH_2B; 1.4–2.0, m, 14H, 9-BBN–H; 2.36, dt, $J(P,H) = 17$ Hz, $J(H,H) = 7$ Hz, 2H, PCH_2; 7.43–7.59, m, 10H, Ph. $^{11}B\{^1H\}$ NMR (v.s.): $\delta = 0.0$, br s. $^{31}P\{^1H\}$ NMR (v.s.): $\delta = 10.3$, br s. $^{13}C\{^1H\}$ NMR (v.s.): $\delta = 23.7$, br s, 9-BBN–C^α; 24.8, s, 9-BBN–C^γ; 33.0, s, 9-BBN–C^β; 33.1, s, 9-BBN–C^b; 24.1, d, $J(P,C) = 17$ Hz, PCH_2CH_2; 26.5, br s, BCH_2; 32.1, d, $J(P,C) = 38$ Hz, PCH_2; 129.0 (8), 130.7 (2), 132.8 (34), 133.7 (9) for $\delta(J)$ of meta-, para-, ipso- and ortho-Ph carbon atoms. MS (EI): $m/z = 348$, M; 239, M + 1 − C_8H_{14}. Anal. Found: C, 78.66; H, 8.85. $C_{23}H_{30}BP$ (348.28). Calc.: C, 79.32; H, 8.68%.

3.7. cyclo-[(9-Borabicyclo[3.3.1]nonanyl)butyl(diphenyl)phosphine], 9

As described for **8**, with 3-butenyl(diphenyl)phosphine (**2**, 0.94 g, 3.91 mmol) and 7.9 ml of 0.5 M 9-BBN in thf solution (10 ml). Yield 1.23 g (87%) colourless crystals, m.p. 140 °C. ^1H NMR $(CD_2Cl_2, 23 °C)$: $\delta = 1.0–1.3$, m, 6H, $CH_2CH_2CH_2B$; 1.4–2.0, m, 14H, 9-BBN–H; 2.39, m, 2H, PCH_2; 7.37–7.52, m, 10H, Ph. $^{11}B\{^1H\}$ NMR (v.s.): $\delta = -5.0$, br s. $^{31}P\{^1H\}$ NMR (v.s.): $\delta = -1.8$, br s. $^{13}C\{^1H\}$ NMR (v.s.): $\delta = 24.3$, br s, 9-BBN–C^α; 24.9, s, 9-BBN–C^γ; 33.2, br s, 9-BBN–C^β; 22.0, br s, BCH_2; 22.0, d, $J(P,C) = 27$ Hz, PCH_2; 23.9, d, $J(P,C) = 2$ Hz, CH_2CH_2B; 25.0, d, $J(P,C) = 4$ Hz, PCH_2CH_2; 128.6 (8), 130.1 (3), 133.0 (9), 133.6 (33) for $\delta(J)$ of meta-, para-, ortho- and ipso-Ph carbon atoms. MS (EI): $m/z = 363$, M + 1; 253, M + 1 − C_8H_{14}. Anal. Found: C, 79.39; H, 9.03; P, 8.27. $C_{24}H_{32}BP$ (362.30). Calc.: C, 79.56; H, 8.90; P, 8.55%.

3.8. Crystal structure determinations

Suitable single crystals of **8** and **9** were sealed in glass capillaries and used for measurement of precise cell constants and intensity data collection. During data collection, three standard reflections were measured periodically as a general check of crystal and instrument stability. No significant changes were observed for either compound. Diffraction intensities were corrected for Lorentz and polarisation effects but not for absorption. The structures were solved by direct methods and refined by full matrix least squares calculations against F^2. The thermal motion of all non-hydrogen atoms was

Table 1
Crystal data, data collection, and structure refinement for compounds **8** and **9**

	8	9
Crystal data		
Formula	$C_{23}H_{30}BP$	$C_{24}H_{32}BP$
M_r	348.25	362.28
Crystal system	orthorhombic	triclinic
Space group	$Pna2_1$ (No. 33)	$P\bar{1}$ (No. 2)
a (Å)	8.619(1)	9.116(1)
b (Å)	15.352(1)	9.735(1)
c (Å)	14.674(1)	12.831(1)
α (deg)	90	69.28(1)
β (deg)	90	86.99(1)
γ (deg)	90	71.36(1)
V (Å³)	1941.6(3)	1006.8(2)
ρ_{calc} (g cm⁻³)	1.191	1.195
Z	4	2
$F(000)$ (e)	752	392
$\mu(MoK\alpha)$ (cm⁻¹)	1.44	1.42
Data collection		
Diffractometer	Enraf Nonius CAD4	Enraf Nonius CAD4
Radiation	MoKα, 0.71069 Å	MoKα, 0.71069 Å
T (°C)	−62	−68
Scan mode	$\omega-\theta$	$\omega-\theta$
hkl range	+10/+19/±18	±11/±11/+15
$\sin(\theta/\lambda)_{max}$ (Å⁻¹)	0.64	0.62
Measured refl.	4024	3696
Unique refl.	4024	3691
Observed refl.	3681	3371
$I \geq$	$2\sigma I$	$2\sigma I$
Refinement		
Refined parameters	225	235
H atoms (found/calc.)	0/30	0/32
$R1$ [a]	0.0422	0.0331
$wR2$ [b]	0.1064	0.0830
$(Shift/error)_{max}$	0.000	0.000
$\rho_{fin}(max/min)$ (e Å⁻³)	+0.277/−0.259	+0.283/−0.230

[a] $R1 = \Sigma(\|F_o|-|F_c\|)/\Sigma|F_o|$.
[b] $wR2 = \{[\Sigma w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$. $-w = q/\sigma^2(F_o^2)+(ap)^2 + bp$; $p = (F_o^2 + 2F_c^2)/3$; $a = 0.0592$ (**8**), 0.0377 (**9**); $b = 0.96$ (**8**), 0.46 (**9**).

treated anisotropically. All hydrogen atoms were placed in idealised calculated positions and allowed to ride on their corresponding carbon atoms with fixed isotropic contributions ($U_{iso(fix)} = 1.5U_{eq}$ of the corresponding C

atom). Further information on crystal data, data collection, and structure refinement are summarised in Table 1. Important interatomic distances and angles are shown in the corresponding figure captions.

Anisotropic thermal parameters, tables of interatomic distances and angles, and tables of observed and calculated structure factors have been deposited with the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschafttlich-technische Information mbH, D-76344 Eggenstein-Leopoldshafen. The data are available on request on quoting CSD No. 405268, 405269.

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