

O4—C17—C16	115.1 (6)	O4'—C17'—C16'	115.5 (6)
C16—C17—C19	111.6 (6)	C16'—C17'—C19'	110.0 (5)
O4—C17—C19	105.5 (5)	O4'—C17'—C19'	104.5 (5)
C10—C1—C2—C3	55.2 (8)	C10'—C1'—C2'—C3'	51.3 (8)
C1—C2—C3—C4	-60.2 (8)	C1'—C2'—C3'—C4'	-56.8 (8)
C2—C3—C4—C5	34.6 (9)	C2'—C3'—C4'—C5'	33.6 (8)
C3—C4—C5—C10	-6.8 (10)	C3'—C4'—C5'—C10'	-4.7 (9)
C4—C5—C10—C1	2.6 (10)	C4'—C5'—C10'—C1'	-0.2 (10)
C2—C1—C10—C5	-25.6 (9)	C2'—C1'—C10'—C5'	-22.5 (9)
C8—C9—C10—C5	-9.6 (10)	C8'—C9'—C10'—C5'	-5.0 (9)
C6—C5—C10—C9	-1.7 (10)	C6'—C5'—C10'—C9'	-0.3 (10)
C10—C5—C6—C7	-18.5 (9)	C10'—C5'—C6'—C7'	-24.2 (9)
C5—C6—C7—C8	50.3 (8)	C5'—C6'—C7'—C8'	53.0 (8)
C6—C7—C8—C9	-62.0 (8)	C6'—C7'—C8'—C9'	-57.6 (7)
C7—C8—C9—C10	39.7 (8)	C7'—C8'—C9'—C10'	33.3 (9)
C9—C11—C12—C13	-16.7 (10)	C9'—C11'—C12'—C13'	-13.9 (10)
C8—C9—C11—C12	0.0 (12)	C8'—C9'—C11'—C12'	-0.8 (11)
C14—C8—C9—C11	-13.6 (10)	C14'—C8'—C9'—C11'	-15.7 (9)
C9—C8—C14—C13	44.6 (8)	C9'—C8'—C14'—C13'	47.2 (8)
C12—C13—C14—C8	-62.4 (7)	C12'—C13'—C14'—C8'	-61.7 (7)
C11—C12—C13—C14	45.5 (8)	C11'—C12'—C13'—C14'	42.4 (8)
C14—C13—C17—C16	-41.3 (6)	C14'—C13'—C17'—C16'	-41.8 (6)
C17—C13—C14—C15	41.8 (6)	C17'—C13'—C14'—C15'	44.2 (6)
C13—C14—C15—C16	-27.1 (7)	C13'—C14'—C15'—C16'	-29.5 (7)
C14—C15—C16—C17	0.3 (7)	C14'—C15'—C16'—C17'	3.0 (7)
C15—C16—C17—C13	25.6 (7)	C15'—C16'—C17'—C13'	24.4 (7)

H atoms were located from difference Fourier maps; they were positioned geometrically and included as riding atoms with fixed isotropic displacement parameters in the structure-factor calculations.

Data collection: Siemens diffractometer software. Cell refinement: Siemens diffractometer software. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1212). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,4-Diphenylquinoline and its Sterically Hindered Derivative 3-Methyl-2,4-diphenylquinoline

MUSTAFA T. AHMET, JOHN R. MILLER, ALAN G. OSBORNE AND JOANNE F. WARMSLEY

Department of Chemistry and Biological Chemistry, University of Essex, Wivenhoe Park, Colchester CO4 3SQ, England

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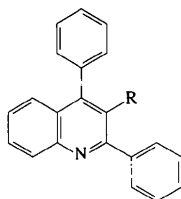
Abstract

The rotation of the phenyl groups in 2,4-diphenylquinoline, C₂₁H₁₅N, and its sterically hindered 3-methyl-2,4-diphenylquinoline derivative, C₂₂H₁₇N, are compared. The comparison demonstrates that the orientation of the phenyl substituents has a marked effect on the interpretation of both the ¹H and ¹³C NMR spectra of these compounds in solution.

Comment

We have recently reported the results of an ¹H and a ¹³C NMR spectral study of 3-methyl-2,4-diphenylquinoline (Osborne, Ahmet, Miller & Warmesley, 1995). The ¹H spectra were assigned by consideration of the anisotropic effects resulting from the rotation of the phenyl rings. The ¹³C NMR spectra obtained were interpreted by proposing the existence of a 'pseudo-*peri*-proximity' (PPP) effect. A normal *peri*-proximity effect (Wilson & Stothers, 1974) for the phenyl–methyl couple in the ¹³C NMR spectrum of 5,8-dimethyl-2,4-diphenylquinoline has been observed previously (Osborne, 1983, 1989).

Here we report the crystal and molecular structures of 2,4-diphenylquinoline, (1), and 3-methyl-2,4-diphenylquinoline, (2), and discuss the effect of the 3-methyl substituent on the crystal structure and ¹H NMR spectrum. Figs. 1 and 2 depict the molecular structures of (1) and (2), respectively, and show the labelling scheme used. Examination of selected bond angles presented in Tables 2 and 4 reveals that all the selected angles are close to 120°, indicating that there is little in-plane splaying of the phenyl rings. Furthermore, there is no out-of-plane distortion of the phenyl groups.



- (1) $R = \text{H}$
 (2) $R = \text{CH}_3$

From Table 5 it can be seen that the largest distance of an appended *ipso* substituent from the mean plane of the quinoline ring is only 0.26 Å. The quinoline ring is essentially planar, as has been noted previously for 2,3-diphenylquinoline (Carloni, Damiani, Greci, Stipa, Rizzoli, Sgarabotto & Uguzzoli, 1993). The dihedral angles between the mean plane of the quinoline ring and the mean planes of the phenyl substituents are listed in Table 5. Table 6 lists ¹H NMR spectral data for (1) and (2). The increase in the 2-phenyl/quinoline dihedral angle from 21° in (1) to 53° in (2) is a direct consequence of steric hindrance between the methyl H atoms (H30a–H30c) and the 2-phenyl H atoms, H(25) and H(21). This steric hindrance results in a reduced electrostatic field effect from the N-atom lone pair, which is manifested in the ¹H NMR spectrum of (2) by an upfield shift of the *ortho* 2-phenyl protons relative to

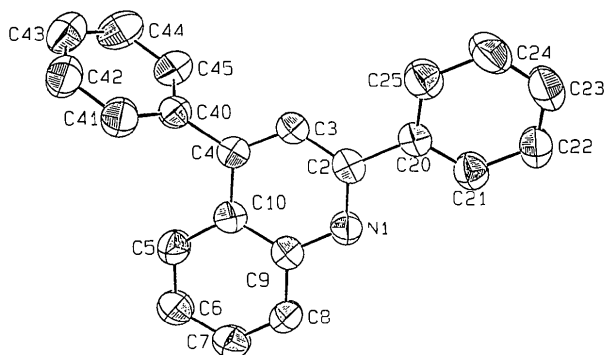


Fig. 1. ORTEP (Johnson, 1965) drawing of compound (1) with displacement ellipsoids drawn at the 50% probability level.

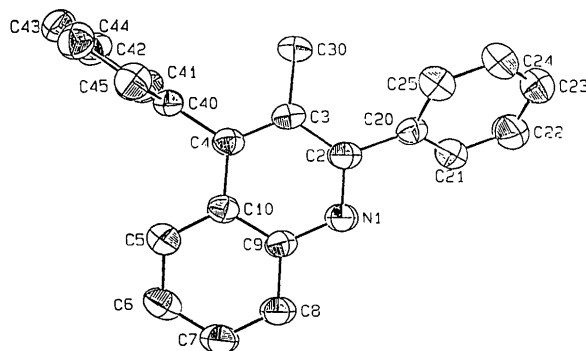


Fig. 2. ORTEP (Johnson, 1965) drawing of compound (2) with displacement ellipsoids drawn at the 50% probability level.

those of (1) (see Table 6). The near orthogonal dihedral angle (89°) between the 4-phenyl ring and the quinoline plane in (2), compared to 65° in (1), results in the H(5) and *ortho* 4-phenyl protons of (2) being anisotropically shielded; this is once again manifested by an upfield shift of these protons relative to those in (1).

The phenyl ring/quinoline plane dihedral angles of (1) and (2) calculated in this study have proved invaluable in the interpretation of the ¹³C NMR spectra of these compounds (Osborne, Ahmet, Miller & Warmesley, 1995). On the basis of the observed dihedral angles, a residual PPP effect has been proposed for the 2-phenyl ring of (2) (53°), while the near orthogonality of the 4-phenyl group of (2) (89°) minimizes the PPP effect.

Experimental

Compound (1)

Crystal data

C₂₁H₁₅N

$M_r = 281.36$

Monoclinic

$P2_1/n$

$a = 10.497(6) \text{ \AA}$

$b = 8.405(4) \text{ \AA}$

$c = 17.17(1) \text{ \AA}$

$\beta = 95.59(3)^\circ$

$V = 1507(3) \text{ \AA}^3$

$Z = 4$

$D_x = 1.235 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 16.44\text{--}21.76^\circ$

$\mu = 0.067 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Plate

$0.39 \times 0.25 \times 0.06 \text{ mm}$

Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer

ω - 2θ scans

Absorption correction: none

5677 measured reflections

2847 independent reflections

1126 observed reflections

$[I > 1.5\sigma(I)]$

$R_{\text{int}} = 0.027$

$\theta_{\text{max}} = 25^\circ$

$h = -12 \rightarrow 12$

$k = 0 \rightarrow 10$

$l = -20 \rightarrow 20$

3 standard reflections

frequency: 120 min

intensity decay: 2.4%

Refinement

Refinement on F

$R = 0.0349$

$wR = 0.0359$

$S = 0.253$

1126 reflections

259 parameters

H atoms refined

$w = 1/[\sigma^2(F) + (0.02F)^2 + 1]$

$(\Delta/\sigma)_{\text{max}} < 0.01$

$\Delta\rho_{\text{max}} = 0.1 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$

Atomic scattering factors

from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2) for (1)

B_{iso} for H atoms; $B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$ for others.				
	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{iso}}/B_{\text{eq}}$
N(1)	0.9862 (2)	0.2636 (3)	0.4378 (1)	3.98 (6)
C(2)	0.9385 (3)	0.3769 (4)	0.3900 (2)	3.88 (7)
C(3)	1.0109 (3)	0.5098 (4)	0.3706 (2)	3.94 (7)
C(4)	1.1377 (3)	0.5235 (4)	0.3973 (2)	3.64 (7)

C(5)	1.3200 (3)	0.4061 (4)	0.4841 (2)	4.67 (8)
C(6)	1.3635 (3)	0.2926 (4)	0.5361 (2)	5.37 (9)
C(7)	1.2830 (3)	0.1701 (4)	0.5563 (2)	5.29 (9)
C(8)	1.1601 (3)	0.1612 (4)	0.5233 (2)	4.68 (8)
C(9)	1.1111 (3)	0.2781 (4)	0.4693 (2)	3.73 (7)
C(10)	1.1917 (3)	0.4037 (4)	0.4494 (2)	3.78 (7)
C(20)	0.8032 (3)	0.3579 (4)	0.3564 (2)	3.91 (7)
C(21)	0.7194 (3)	0.2654 (4)	0.3944 (2)	4.63 (8)
C(22)	0.5931 (3)	0.2465 (4)	0.3647 (2)	5.18 (8)
C(23)	0.5490 (3)	0.3194 (5)	0.2960 (2)	5.60 (9)
C(24)	0.6298 (3)	0.4124 (5)	0.2571 (2)	6.0 (1)
C(25)	0.7563 (3)	0.4322 (5)	0.2872 (2)	5.47 (9)
C(40)	1.2148 (3)	0.6588 (4)	0.3715 (2)	3.67 (7)
C(41)	1.3169 (3)	0.6313 (4)	0.3276 (2)	5.01 (8)
C(42)	1.3856 (3)	0.7552 (5)	0.2993 (2)	5.75 (9)
C(43)	1.3551 (3)	0.9085 (5)	0.3160 (2)	5.80 (9)
C(44)	1.2559 (4)	0.9386 (4)	0.3600 (2)	6.1 (1)
C(45)	1.1843 (3)	0.8146 (4)	0.3877 (2)	4.95 (8)
H(3)	0.971 (2)	0.597 (3)	0.338 (1)	3.6 (6)
H(5)	1.375 (2)	0.492 (3)	0.470 (2)	5.5 (7)
H(6)	1.455 (3)	0.295 (4)	0.561 (2)	7.4 (9)
H(7)	1.318 (2)	0.087 (3)	0.595 (2)	5.5 (7)
H(8)	1.106 (2)	0.078 (3)	0.537 (1)	4.5 (7)
H(21)	0.748 (2)	0.217 (3)	0.444 (1)	4.5 (7)
H(22)	0.536 (3)	0.185 (3)	0.395 (2)	5.8 (8)
H(23)	0.463 (3)	0.306 (4)	0.273 (2)	6.1 (8)
H(24)	0.598 (3)	0.467 (4)	0.207 (2)	6.9 (8)
H(25)	0.812 (3)	0.493 (4)	0.259 (2)	6.5 (8)
H(41)	1.338 (3)	0.524 (4)	0.314 (2)	6.9 (8)
H(42)	1.458 (3)	0.731 (4)	0.261 (2)	9 (1)
H(43)	1.402 (3)	0.998 (4)	0.292 (2)	9 (1)
H(44)	1.232 (3)	1.042 (4)	0.373 (2)	9 (1)
H(45)	1.113 (3)	0.837 (4)	0.420 (2)	7.1 (9)

Table 2. Selected geometric parameters (\AA , $^\circ$) for (1)

N(1)—C(2)	1.323 (4)	C(40)—C(45)	1.383 (5)
N(1)—C(9)	1.374 (4)	C(41)—C(42)	1.381 (5)
C(2)—C(3)	1.409 (5)	C(42)—C(43)	1.365 (6)
C(2)—C(20)	1.489 (4)	C(43)—C(44)	1.369 (6)
C(3)—C(4)	1.369 (4)	C(44)—C(45)	1.395 (5)
C(4)—C(10)	1.427 (4)	C(5)—H(5)	0.98 (3)
C(4)—C(40)	1.488 (4)	C(6)—H(6)	1.01 (3)
C(5)—C(6)	1.356 (5)	C(7)—H(7)	1.01 (3)
C(5)—C(10)	1.419 (5)	C(8)—H(8)	0.95 (3)
C(6)—C(7)	1.396 (5)	C(21)—H(21)	0.96 (3)
C(7)—C(8)	1.359 (5)	C(22)—H(22)	0.98 (3)
C(8)—C(9)	1.413 (5)	C(23)—H(23)	0.96 (3)
C(9)—C(10)	1.416 (4)	C(24)—H(24)	1.01 (3)
C(20)—C(21)	1.384 (5)	C(25)—H(25)	0.95 (3)
C(20)—C(25)	1.389 (5)	C(41)—H(41)	0.96 (4)
C(21)—C(22)	1.382 (5)	C(42)—H(42)	1.07 (4)
C(22)—C(23)	1.368 (6)	C(43)—H(43)	1.01 (4)
C(23)—C(24)	1.373 (6)	C(44)—H(44)	0.93 (4)
C(24)—C(25)	1.387 (5)	C(45)—H(45)	1.00 (3)
C(40)—C(41)	1.388 (5)		
C(20)—C(2)—N(1)	116.9 (3)	C(40)—C(4)—C(3)	120.3 (3)
C(20)—C(2)—C(3)	120.6 (3)	C(40)—C(4)—C(10)	122.1 (3)

Compound (2)*Crystal data*C₂₂H₁₇N $M_r = 295.39$

Triclinic

 $P\bar{1}$ $a = 8.273 (2) \text{\AA}$ $b = 10.248 (3) \text{\AA}$ $c = 11.113 (2) \text{\AA}$ $\alpha = 111.87 (2)^\circ$ $\beta = 103.61 (1)^\circ$ $\gamma = 101.35 (2)^\circ$ $V = 806.5 (8) \text{\AA}^3$ $Z = 2$ $D_x = 1.216 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation $\lambda = 0.71073 \text{\AA}$

Cell parameters from 25

reflections

 $\theta = 17.11\text{--}21.28^\circ$ $\mu = 0.065 \text{ mm}^{-1}$ $T = 293 \text{ K}$

Plate

 $0.28 \times 0.14 \times 0.08 \text{ mm}$

Colourless

Data collection

Enraf–Nonius CAD-4

diffractometer

 ω – 2θ scans

Absorption correction:

 ψ scan $T_{\min} = 0.9717$, $T_{\max} =$

0.9998

3041 measured reflections

2827 independent reflections

1521 observed reflections

 $[I > 1.5\sigma(I)]$ $R_{\text{int}} = 0.032$ $\theta_{\max} = 25^\circ$ $h = 0 \rightarrow 9$ $k = -12 \rightarrow 12$ $l = -13 \rightarrow 13$

3 standard reflections

frequency: 120 min

intensity decay: 2.38%

*Refinement*Refinement on F^2 $R = 0.0600$ $wR = 0.0631$ $S = 0.478$

1521 reflections

276 parameters

H atoms refined

 $w = 1/[\sigma^2(F) + (0.02F)^2 + 1]$ $(\Delta/\sigma)_{\max} < 0.01$ $\Delta\rho_{\max} = 0.21 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.11 \text{ e \AA}^{-3}$

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV)

Table 3. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2) for (2)

N(1)	B_{iso} for H atoms; $B_{\text{eq}} = (4/3)\sum_i \beta_{ij} a_i \cdot a_j$ for others.			
	x	y	z	$B_{\text{iso}}/B_{\text{eq}}$
N(1)	0.3768 (4)	0.9862 (4)	0.6434 (3)	3.46 (8)
C(2)	0.2363 (5)	0.9526 (4)	0.6780 (4)	3.4 (1)
C(3)	0.2308 (5)	1.0251 (4)	0.8140 (4)	3.3 (1)
C(4)	0.3744 (5)	1.1421 (4)	0.9127 (4)	3.2 (1)
C(5)	0.6876 (5)	1.2850 (5)	0.9774 (4)	4.0 (1)
C(6)	0.8350 (6)	1.3085 (5)	0.9412 (5)	4.6 (1)
C(7)	0.8263 (5)	1.2325 (5)	0.8053 (4)	4.5 (1)
C(8)	0.6746 (5)	1.1292 (5)	0.7074 (4)	3.9 (1)
C(9)	0.5236 (5)	1.0979 (4)	0.7440 (4)	3.22 (9)
C(10)	0.5297 (5)	1.1782 (4)	0.8794 (4)	3.12 (9)
C(20)	0.0849 (5)	0.8297 (4)	0.5638 (4)	3.4 (1)
C(21)	0.1041 (6)	0.6959 (5)	0.4887 (4)	4.4 (1)
C(22)	−0.0360 (6)	0.5805 (6)	0.3835 (5)	5.6 (1)
C(23)	−0.2000 (6)	0.6001 (6)	0.3477 (5)	5.3 (1)
C(24)	−0.2192 (6)	0.7335 (5)	0.4200 (5)	5.1 (1)
C(25)	−0.0809 (6)	0.8471 (5)	0.5259 (4)	4.4 (1)
C(30)	0.0740 (6)	0.9702 (5)	0.8505 (4)	4.7 (1)
C(40)	0.3702 (5)	1.2285 (4)	1.0526 (4)	3.2 (1)
C(41)	0.4122 (6)	1.1855 (5)	1.1578 (4)	4.3 (1)
C(42)	0.3987 (6)	1.2631 (5)	1.2845 (4)	4.7 (1)
C(43)	0.3501 (6)	1.3885 (6)	1.3108 (4)	4.7 (1)
C(44)	0.3133 (6)	1.4360 (5)	1.2098 (5)	4.8 (1)
C(45)	0.3255 (6)	1.3576 (5)	1.0824 (4)	4.3 (1)
H(5)	0.699 (5)	1.344 (4)	1.077 (4)	5 (1)
H(6)	0.943 (5)	1.393 (4)	1.020 (4)	5 (1)
H(7)	0.926 (5)	1.253 (4)	0.786 (4)	6 (1)
H(8)	0.661 (5)	1.067 (4)	0.605 (4)	5 (1)
H(21)	0.216 (6)	0.695 (5)	0.532 (4)	8 (1)
H(22)	−0.024 (7)	0.476 (6)	0.322 (5)	11 (2)
H(23)	−0.292 (6)	0.497 (5)	0.265 (5)	9 (2)
H(24)	−0.329 (5)	0.739 (4)	0.386 (4)	6 (1)
H(25)	−0.087 (6)	0.952 (5)	0.578 (4)	7 (1)
H(30a)	0.004 (6)	0.864 (5)	0.786 (4)	7 (1)
H(30b)	0.117 (6)	1.006 (5)	0.965 (5)	9 (2)
H(30c)	−0.005 (8)	1.018 (7)	0.845 (6)	13 (2)
H(41)	0.422 (5)	1.076 (4)	1.119 (4)	6 (1)
H(42)	0.422 (5)	1.223 (4)	1.346 (4)	7 (1)
H(43)	0.351 (4)	1.446 (4)	1.405 (3)	3.8 (9)
H(44)	0.295 (6)	1.543 (5)	1.245 (4)	8 (1)
H(45)	0.300 (5)	1.388 (4)	1.012 (4)	5 (1)

Table 4. Selected geometric parameters (Å, °) for (2)

N(1)—C(2)	1.330 (5)	C(41)—C(42)	1.379 (7)
N(1)—C(9)	1.376 (5)	C(42)—C(43)	1.371 (8)
C(2)—C(3)	1.431 (6)	C(43)—C(44)	1.376 (8)
C(2)—C(20)	1.485 (6)	C(44)—C(45)	1.383 (7)
C(3)—C(4)	1.377 (6)	C(5)—H(5)	1.02 (4)
C(3)—C(30)	1.514 (6)	C(6)—H(6)	1.04 (5)
C(4)—C(10)	1.437 (6)	C(7)—H(7)	0.90 (5)
C(4)—C(40)	1.487 (6)	C(8)—H(8)	1.04 (5)
C(5)—C(6)	1.377 (7)	C(21)—H(21)	0.94 (6)
C(5)—C(10)	1.403 (6)	C(22)—H(22)	1.08 (7)
C(6)—C(7)	1.393 (8)	C(23)—H(23)	1.08 (6)
C(7)—C(8)	1.362 (7)	C(24)—H(24)	0.92 (5)
C(8)—C(9)	1.418 (6)	C(25)—H(25)	1.03 (5)
C(9)—C(10)	1.402 (6)	C(30)—H(30a)	1.00 (6)
C(20)—C(21)	1.378 (7)	C(30)—H(30b)	1.12 (6)
C(20)—C(25)	1.404 (6)	C(30)—H(30c)	0.89 (8)
C(21)—C(22)	1.378 (7)	C(41)—H(41)	1.07 (5)
C(22)—C(23)	1.398 (8)	C(42)—H(42)	0.92 (5)
C(23)—C(24)	1.366 (8)	C(43)—H(43)	0.99 (4)
C(24)—C(25)	1.367 (7)	C(44)—H(44)	1.08 (6)
C(40)—C(41)	1.391 (6)	C(45)—H(45)	0.94 (5)
C(40)—C(45)	1.387 (6)		
C(20)—C(2)—N(1)	114.3 (4)	C(40)—C(4)—C(10)	120.3 (4)
C(20)—C(2)—C(3)	122.2 (4)	C(30)—C(3)—C(2)	120.7 (4)
C(40)—C(4)—C(3)	120.7 (4)	C(30)—C(3)—C(4)	120.6 (4)

Table 5. Least-squares planes data

(a) Angles (°) between planes in (1) and (2); (b) distances (Å) of relevant atoms from the mean quinoline planes of (1) and (2).

	(1)	(2)
(a)		
Mean quinoline plane / 2-phenyl plane	21.1 (2)	52.6 (1)
Mean quinoline plane / 4-phenyl plane	64.9 (1)	88.9 (1)
(b)		
C(20) 2-phenyl ipso C atom	0.034 (3)	-0.081 (4)
C(40) 4-phenyl ipso C atom	0.036 (3)	0.127 (4)
C(30) methyl C atom	-	-0.258 (6)

Table 6. 270 MHz ¹H NMR spectra of compounds (1) and (2)

Assignment	(1)		(2)	
	δH (p.p.m.)	Multiplicity	δH (p.p.m.)	Multiplicity
H(3)	7.819	ddd	-	-
H(5)	7.905	ddd	7.386	ddd
H(6)	7.469	ddd	7.351	ddd
H(7)	7.728	ddd	7.619	ddd
H(8)	8.249	ddd	8.175	ddd
2-Phenyl				
o-	8.19	m	7.61	m
m-/p-	7.46-7.53	m	7.42-7.52	m
4-Phenyl				
o-	7.55-7.57	m	7.27-7.31	m
m-/p-	7.55-7.57	m	7.45-7.55	m
CH ₃	-	-	2.143	s

Accurate unit-cell dimensions and the crystal-orientation matrices for (1) and (2) were determined by a least-squares refinement of the setting angles of 25 centred reflections using the automatic indexing routine of the diffractometer. The space group for (1) was determined by systematic absences and from Harker planes in the Patterson function. Inversion symmetry was detected statistically from *E* maps. The usual corrections for Lorentz and polarization factors were made. For structure (2), an empirical absorption correction was made after scanning nine reflections, each at 36 different azimuthal angles (ψ scan). In the case of structure (1), the data were collected using the *FLAT* procedure (Enraf-Nonius, 1990) in which each collection was carried out at an azimuthal angle ψ

calculated to minimize absorption by minimizing the path of the X-rays through the crystal. No further absorption corrections were applied. Both structures were determined by direct methods using *SHELXS86* (Sheldrick, 1985); subsequent full-matrix least-squares refinement and difference Fourier synthesis using *MolEN* (Fair, 1990) located all remaining atoms, including H atoms. All non-H atoms were refined anisotropically, while the H atoms were refined with isotropic displacement parameters. Killean & Lawrence (1969) weights were used in the refinement. All computations were performed on a MicroVAX computer. Diagrams were drawn using *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1097). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[3,3-Dimethyl-1-(2-pyridylthio)butyl]-methyl(2,4,6-trimethylphenyl)phosphine Oxide

WANDA WIECZOREK

*Institute of General and Ecological Chemistry,
Technical University of Łódź, Żwirki 36, 90-924 Łódź,
Poland*

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Abstract

In the molecule of [3,3-dimethyl-1-(2-pyridylthio)-butyl]methyl(2,4,6-trimethylphenyl)phosphine oxide, C₂₁H₃₀NOPS, the P=O bond is nearly coplanar with