601 inobservées [$I < 2\sigma(I)$]. Méthodes directes, programme *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) et série de Fourier des ΔF. Affinement basé sur les F, programme à matrice complète. Facteurs de diffusion des *International Tables for X-ray Crystallography* (1974, Tome IV, Tableaux 2.2B, p. 99 et 2.3.1, p. 149). Paramètres affinés: x, y, z de tous les atomes et β_{ij} de C et N. B(H) = $B_{\text{éq}}$ de l'atome lié à H + 1 Å^2.R = 0,035, wR = 0,038, $w = 1/\sigma^2(F)$, S = 1,31, $(\Delta/\sigma)_{\max}$ = 0,01, $|\Delta\rho|_{\max}$ = 0,09 (2) e Å^{-3}. Coefficient d'extinction secondaire isotrope: g = 1,8 (3) × 10^{-6}. Programmes de calcul du système *SDP* (B. A. Frenz & Associates, Inc., 1982). Angles de torsion: *ORFFE* (Busing, Martin & Levy, 1964). Fig. 1 et 2: *ORTEP*II (Johnson, 1976).

Le composé étudié est un intermédiaire de synthèse. Sa molécule contient deux atomes de carbone asymétriques qui ont la même configuration. Celle-ci est R pour l'un d'entre eux dans le réactif utilisé au cours de la synthèse. La résolution de la structure permet donc d'affirmer que le deuxième atome de carbone présente aussi la configuration R.

Les coordonnées relatives et les facteurs de température isotropes équivalents des atomes de carbone et d'azote sont rapportés dans le Tableau 1,* les longueurs et les angles des liaisons dans le Tableau 2. Les numéros attribués aux atomes sont indiqués sur la Fig. 1. La Fig. 2 représente la structure vue selon [010].

Littérature associée. α-Phényl-α-éthylacétate de phényl-1 éthylammonium n (Brianso, 1978). Mandélate de phényl-1 éthylammonium (Brianso, Leclercq & Jacques, 1979). Structures atomiques et moléculaires des sels diastéréoisomers des α-phényl-α-méthylacétates de phényl-1 éthylammonium p et n (Brianso, 1976). Fumarate de bis{[(1H-benzimidazolyl-1)-3 méthyl-1 propyl] [hydroxy-2 (hydroxy-4 méthoxy-3 phényl)-2 éthyl] ammonium} hexahydrate (Toffoli, Rodier, Céolin, Dao Duong & Joannic, 1990).

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* Les listes des facteurs de structure observés et calculés, des coefficients d'agitation thermique anisotrope, des paramètres des atomes d'hydrogène, des distances C—H et N—H, des distances des atomes aux plans moyens, des distances interatomiques intermoléculaires et des angles de torsion ont été déposées aux archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 53700:15 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

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Structure of 6,7-Dimethyl-2,3-diphenylquinoxaline

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Abstract. $C_{22}H_{18}N_2$, M_r = 310·4, orthorhombic, $Pbca$, a = 8·013 (2), b = 20·003 (4), c = 20·846 (4) Å, V = 3341 Å^3, Z = 8, D_x = 1·234 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·677 cm^{-1}, F(000) = 1312, T = 291 K, R = 0·0667 for 1855 unique reflections. The molecule has dihedral angles of 22·2 (1) and 48·1 (1)° between the best planes of the phenyl rings and the quinoxaline parent. Energy calculations, taking into account the mutual dependence of the twist out of the quinoxaline plane by the two phenyl groups, give the optimal conformation when both groups have torsion angles that are 60°. The twists from this calculated conformation to the experimental one incur about 4·0 kcal mol^{-1}. They are a result of packing forces which are usually estimated to 2 kcal mol^{-1}. (1 kcal mol^{-1}≡4·2 kJ mol^{-1}.)

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Table 1. *Final fractional coordinates* ($\times 10^4$) *and equivalent isotropic temperature coefficients* ($\times 10^3$ Å^2), *for non-*H *atoms*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
N1	8871 (4)	2184 (2)	7346 (1)	50 (1)
N2	7532 (4)	1205 (2)	8160 (1)	51 (1)
C1	8771 (5)	2280 (2)	7977 (2)	45 (1)
C2	8209 (5)	1752 (2)	8391 (2)	44 (1)
C3	6624 (6)	0589 (2)	7235 (2)	59 (2)
C4	6586 (5)	0506 (2)	6581 (2)	58 (2)
C5	7481 (6)	0946 (2)	6178 (2)	58 (1)
C6	8277 (6)	1488 (2)	6433 (2)	55 (2)
C7	8243 (5)	1611 (2)	7109 (2)	47 (1)
C8	7465 (5)	1137 (2)	7503 (2)	47 (1)
C9	5555 (9)	−0055 (3)	6302 (3)	83 (3)
C10	7533 (10)	0842 (4)	5458 (2)	79 (2)
C1*A*	8362 (5)	1769 (2)	9109 (2)	48 (1)
C2*A*	9576 (6)	2136 (2)	9420 (2)	57 (2)
C3*A*	9683 (7)	2128 (2)	10089 (2)	64 (2)
C4*A*	8576 (7)	1763 (2)	10442 (2)	70 (2)
C5*A*	7383 (7)	1389 (3)	10139 (2)	68 (2)
C6*A*	7258 (6)	1390 (2)	9471 (2)	47 (2)
C1*B*	9158 (5)	2967 (2)	8185 (2)	47 (1)
C2*B*	8097 (6)	3323 (3)	8588 (2)	61 (2)
C3*B*	8402 (7)	3984 (3)	8734 (2)	72 (2)
C4*B*	9755 (8)	4304 (3)	8475 (2)	78 (2)
C5*B*	10806 (8)	3971 (3)	8077 (2)	77 (2)
C6*B*	10497 (6)	3317 (2)	7927 (2)	64 (2)

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

N1—C1	1·327 (5)	C7—C8	1·402 (6)
N1—C7	1·346 (6)	C1*A*—C2*A*	1·381 (6)
C1—C2	1·438 (6)	C1*A*—C6*A*	1·388 (6)
C1—C1*B*	1·475 (6)	C2*A*—C3*A*	1·395 (6)
C2—N2	1·316 (5)	C3*A*—C4*A*	1·365 (7)
C2—C1*A*	1·438 (6)	C4*A*—C5*A*	1·368 (8)
N2—C8	1·375 (5)	C5*A*—C6*A*	1·396 (6)
C3—C8	1·399 (6)	C1*B*—C2*B*	1·396 (6)
C3—C4	1·377 (6)	C1*B*—C6*B*	1·391 (7)
C4—C9	1·507 (8)	C2*B*—C3*B*	1·373 (8)
C4—C5	1·415 (6)	C3*B*—C4*B*	1·373 (8)
C5—C6	1·365 (6)	C4*B*—C5*B*	1·361 (8)
C5—C10	1·516 (6)	C5*B*—C6*B*	1·365 (8)
C6—C7	1·428 (6)		

N1—C1—C2	120·7 (4)	C6—C7—N1	120·2 (4)
N1—C1—C1*B*	114·4 (4)	C8—C7—N1	121·6 (4)
C2—C1—C1*B*	124·8 (4)	C1—N1—C7	117·6 (4)
C1—C2—N2	121·2 (4)	C2*A*—C1*A*—C6*A*	118·7 (4)
C1—C2—C1*A*	124·0 (4)	C1*A*—C2*A*—C3*A*	120·7 (5)
N2—C2—C1*A*	114·9 (4)	C2*A*—C3*A*—C4*A*	120·4 (4)
C2—N2—C8	117·6 (4)	C3*A*—C4*A*—C5*A*	119·7 (4)
N2—C8—C3	119·4 (4)	C4*A*—C5*A*—C6*A*	120·6 (4)
N2—C8—C7	120·2 (4)	C1*A*—C6*A*—C5*A*	120·0 (4)
C3—C8—C7	120·5 (4)	C2—C1*A*—C2*A*	122·7 (4)
C8—C3—C4	120·5 (4)	C2—C1*A*—C6*A*	118·6 (4)
C3—C4—C5	119·7 (4)	C1—C1*B*—C2*B*	122·0 (4)
C3—C4—C9	119·3 (4)	C1—C1*B*—C6*B*	121·2 (4)
C5—C4—C9	121·0 (4)	C2*B*—C1*B*—C6*B*	116·4 (4)
C4—C5—C10	120·7 (4)	C1*B*—C2*B*—C3*B*	121·0 (5)
C6—C5—C10	119·2 (4)	C2*B*—C3*B*—C4*B*	120·5 (5)
C4—C5—C6	120·1 (4)	C3*B*—C4*B*—C5*B*	119·7 (5)
C5—C6—C7	120·8 (4)	C4*B*—C5*B*—C6*B*	120·0 (5)
C6—C7—C8	118·1 (4)	C5*B*—C6*B*—C1*B*	122·3 (5)

Experimental. Crystals of 6,7-dimethyl 2,3-diphenylquinoxaline (DDQ) were crystallized from acetonitrile. An Enraf–Nonius CAD-4 diffractometer was used with graphite-monochromatized Mo $K\alpha$ radiation. The crystal size was 0·025 × 0·30 × 0·35 mm. Unit-cell parameters were obtained by least-squares fit of the setting angles of 25 reflections in the range $3 < 2\theta < 15°$. The intensities of 3360 reflections were measured ($\sin\theta/\lambda < 0{\cdot}60$ Å^{-1}, $0 \le h \le 9$, $0 \le k \le 23$, $0 \le l \le 24$, ω–2θ scan mode). No significant variation (< 4%) was found; intensity control reflections 231, 144 and 024. The data were corrected for Lorentz and polarization effect, no absorption correction was applied. 1855 unique reflections with $|F| \ge 3\sigma(F)$ were used in the calculations, $R_{\rm int} = 0{\cdot}024$. The structure was solved with multisolution direct methods (*SHELXS*86; Sheldrick, 1990), and refined using full-matrix least-squares methods (*SHELX*; Sheldrick, 1976) minimizing $\sum w(|F_o| - |F_c|)^2$, $w = \{6{\cdot}9672/[\sigma^2(F) + 0{\cdot}000038F^2]\}$. Heavy atoms were refined with anisotropic and H atoms with isotropic temperature factors; 289 parameters were varied. The refinement converged at $R = 0{\cdot}066$, $wR = 0{\cdot}0586$. $(\Delta/\sigma)_{\max} = 0{\cdot}040$, $(\Delta/\sigma)_{\rm mean} = 0{\cdot}010$, $\Delta\rho_{\max} = +0{\cdot}25$, $\Delta\rho_{\min} = -0{\cdot}25$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Fractional coordinates and equivalent isotropic temperature coeffi-

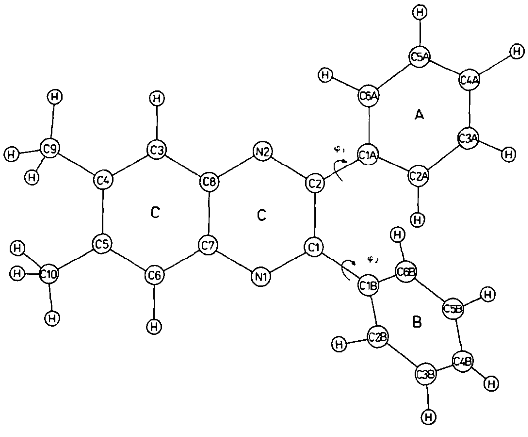

Fig. 1. Numbering of atoms in the title compound.

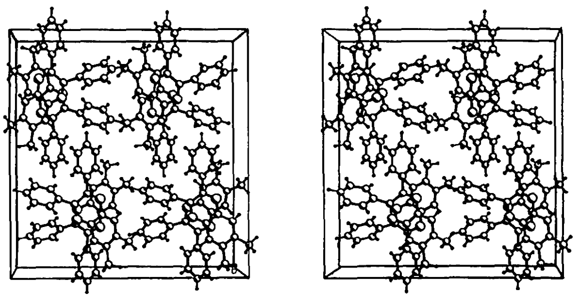

Fig. 2. Stereoscopic projection of the unit-cell contents. **c** is vertical, **b** is horizontal and **a** is out of the plane of the paper.

cients for non-H atoms are given in Table 1.* The molecular geometry is given in Table 2 and atom labelling in Fig. 1. The packing of the molecules in the unit cell is given in Fig. 2. The figures have been drawn with *PLUTO* (Motherwell, 1972). Due to overcrowding in the region of *o*-substitution the expected C_{2v} symmetry of DDQ is broken. Both phenyl rings, *A* and *B*, differ in their twist angles with respect to the quinoxaline plane [22·2 (1) and 48·1 (1)°, respectively], hence one may expect different effects on the molecular geometry from this part of DDQ.

Related literature. Crystal structures containing 2,3-disubstituted derivatives of quinoxaline have aroused considerable interest because of the repulsion between the neighbouring substituents (Visser, Vos, de Grooth & Wynberg, 1968; Visser & Vos, 1971; Lipkowski, Herbich & Andreetti, 1985; Krigier, Kocak & Bekaroglu, 1985; Woźniak, Krygowski, Kariuki & Jones, 1990). The conformation of DDQ was calculated usig the *MM2*/*MMP2* program (Allinger & Yuh, 1987).

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* Lists of structure factors, anisotropic thermal parameters, least-squares planes, dihedral angles and H-atom parameters and an energy map as a function of φ_1 and φ_2 have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53764 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of an Isoxazole Amino Ester

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Abstract. 2′-(*N*-Methylamino)-2-methylpropyl 5-methyl-3-phenylisoxazole-4-carboxylate hydroiodide, $C_{16}H_{21}N_2O_3^+.I^-$, $M_r = 416·26$, orthorhombic, $Pna2_1$, $a = 23·293$ (7), $b = 10·276$ (3), $c = 7·971$ (1) Å, $V = 1908·0$ (8) Å^3, $Z = 4$, $D_x = 1·448$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 17·1$ cm^{-1}, $F(000) = 832$, $T = 293$ K, $R = 0·0610$ for 1846 unique observed reflections with $F > 3\sigma$. In the title compound, the isoxazole ring is distorted only slightly from planarity, with C(3) being 0·045 Å out of the mean plane. Steric interactions force the isoxazole ring out of planarity with both the phenyl ring and the ester carbonyl O(2), which show dihedral angles of 34·9 (9)° and 47·2 (7)°, respectively. The N(2) amino methyl group is oriented approximately *anti* to the ester carbonyl O(2). The minimum iodide to nitrogen distance is from I to N(2)′, 3·46 (2) Å.

Experimental. The title compound, (I), was isolated as a by-product from the methiodide salt formation of 4,4-dimethyl-2-(5′-methyl-3′-phenylisoxazol-4′-yl)-2-oxazoline (Natale, McKenna, Niou, Borth & Hope, 1985). Suitable crystals (0·45 × 0·25 × 0·2 mm) were obtained by slow evaporation from acetonitrile–ethyl ether, as needles. The data were collected on an upgraded Syntex $P2_1$ diffractometer, using the ω-scan technique (Campana, Shepard & Litchman, 1980). The measured intensities were corrected for Lorentz and polarization effects. Azimuthal absorption correction was applied using empirical absorption correction with maximum and minimum transmission factors 0·783 and 0·709, respectively. Cell constants were obtained by a least-squares fitting of setting angles of the diffractometer from 25 reflections with $23 < 2\theta < 32°$. 2541 reflec-

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