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, $C_{16}H_{21}N_2O_3^+.I^-$, $M_r = 416{\cdot}26$, orthorhombic, $Pna2_1$, $a = 23{\cdot}293$ (7), $b = 10{\cdot}276$ (3), $c = 7{\cdot}971$ (1) Å, $V = 1908{\cdot}0$ (8) Å^3, $Z = 4$, $D_x = 1{\cdot}448$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 17{\cdot}1$ cm^{-1}, $F(000) = 832$, $T = 293$ K, $R = 0{\cdot}0610$ 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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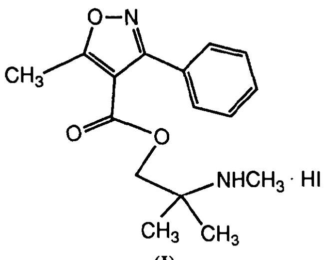
tions were collected up to $2\theta_{\max} = 55°$. The range of *hkl* values was $0 < h < 12$, $0 < k < 14$, $0 < l < 31$; two standard reflections (221 and 400) were monitored throughout the experiment with less than 3% variation in intensity. 2352 unique reflections, with 1846 having an intensity greater than 3σ, were observed. $R_{\rm int}$ from merging equivalent reflections was 0·0186 after absorption corrections for all 2541 measured reflections.



(I)

The structure was solved with the *SHELXTL* program package (Sheldrick, 1985) using the direct-methods routine *SOLV*. It was refined by block-diagonal matrix least squares on F^2. The origin was fixed with the I atom at 0·25 along the *z* axis. H-atom positions were calculated with the N—H and C—H bond distances constrained to 0·96 Å, and the thermal parameters were set at $1{\cdot}2U_{\rm eq}$ of the associated atom. The phenyl ring was constrained to a regular planar hexagon with bond distances set at 1·395 Å and the angles at 120°. Anisotropic thermal parameters were used for all other non-H atoms. The thermal parameters for the H atoms of the phenyl group were fixed at 0·08. 168 least-squares parameters were refined, using isotropic thermal parameters for the constrained phenyl ring and anisotropic thermal parameters for all other non-H atoms. The final *R* values were 0·0610, $wR = 0{\cdot}0683$ with $w = 1/[\sigma^2(F) + g(F^2)]$ and $g = 0{\cdot}00149$. The goodness of fit was 1·486.

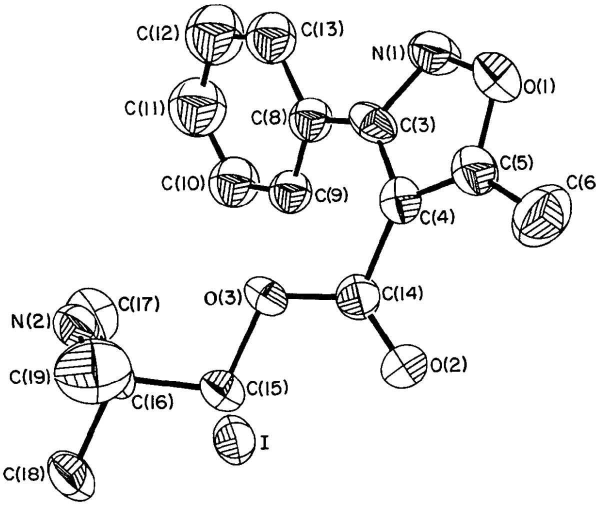


Fig. 1. Thermal ellipsoids with the atom-numbering scheme for 2′-*N*-methylamino-2′-methylpropyl 5-methyl-3-phenylisoxazole-4-carboxylate hydroiodide.

Table 1. *Atomic coordinates* (× 10^4) *and isotropic thermal parameters* (Å^2 × 10^3)

E.s.d.'s in the least significant digits are given in parentheses.

	x	*y*	*z*	$U_{\rm iso}$*
I	1752 (1)	951 (1)	2500	61 (1)
N(1)	−399 (4)	6083 (8)	4967 (16)	55 (3)
N(2)	1619 (3)	3247 (9)	9388 (12)	41 (3)
O(1)	−125 (3)	7308 (7)	5049 (13)	61 (3)
O(2)	1524 (4)	5603 (11)	4302 (16)	93 (4)
O(3)	1208 (3)	4314 (7)	6319 (11)	45 (2)
C(3)	14 (6)	5190 (10)	5170 (17)	41 (3)
C(4)	562 (5)	5832 (10)	5150 (16)	50 (3)
C(5)	463 (4)	7123 (10)	5157 (18)	56 (4)
C(6)	810 (6)	8284 (13)	5161 (23)	74 (5)
C(8)†	−132	3806	4983	44 (2)
C(9)†	225	2912	4178	50 (3)
C(10)†	75	1598	4132	67 (3)
C(11)†	−432	1178	4893	88 (5)
C(12)†	−788	2072	5699	88 (5)
C(13)†	−638	3386	5744	60 (3)
C(14)	1155 (4)	5245 (10)	5180 (16)	51 (4)
C(15)	1773 (4)	3693 (12)	6359 (16)	44 (3)
C(16)	1768 (4)	2664 (10)	7717 (20)	43 (3)
C(17)	1319 (5)	1628 (10)	7390 (23)	58 (4)
C(18)	2369 (5)	2073 (14)	7809 (19)	67 (5)
C(19)	1974 (6)	4340 (13)	10049 (24)	74 (5)

* The equivalent isotropic *U* for anisotropic atoms is defined as one third the trace of the orthogonalized U_{ij} tensor.

† Coordinates for these atoms were constrained and not refined.

Table 2. *Selected bond lengths* (Å) *and angles* (°)

E.s.d.'s in the least significant digits are given in parentheses.

N(1)—O(1)	1·412 (11)	C(4)—C(14)	1·508 (14)
N(1)—C(3)	1·340 (16)	C(5)—C(6)	1·442 (17)
N(2)—C(16)	1·501 (17)	C(9)—C(10)	1·395 (1)
N(2)—C(19)	1·491 (17)	C(9)—C(8)	1·395 (1)
O(1)—C(5)	1·385 (13)	C(10)—C(11)	1·395 (1)
O(2)—C(14)	1·167 (15)	C(11)—C(12)	1·395 (1)
O(3)—C(14)	1·324 (14)	C(12)—C(13)	1·395 (1)
O(3)—C(15)	1·464 (12)	C(13)—C(8)	1·395 (1)
C(3)—C(4)	1·439 (17)	C(15)—C(16)	1·513 (18)
C(3)—C(8)	1·470 (14)	C(16)—C(17)	1·515 (14)
C(4)—C(5)	1·347 (14)	C(16)—C(18)	1·527 (14)

O(1)—N(1)—C(3)	106·3 (8)	C(11)—C(12)—C(13)	120·0 (1)
C(16)—N(2)—C(19)	119·1 (10)	C(12)—C(13)—C(8)	120·0 (1)
N(1)—O(1)—C(5)	109·1 (7)	C(9)—C(8)—C(13)	120·0 (1)
C(14)—O(3)—C(15)	114·4 (8)	C(3)—C(8)—C(9)	123·1 (6)
N(1)—C(3)—C(4)	108·8 (10)	C(3)—C(8)—C(13)	116·8 (6)
N(1)—C(3)—C(8)	119·0 (11)	O(2)—C(14)—O(3)	124·8 (10)
C(4)—C(3)—C(8)	130·4 (11)	O(2)—C(14)—C(4)	122·6 (11)
C(3)—C(4)—C(5)	107·4 (10)	O(3)—C(14)—C(4)	112·6 (9)
C(3)—C(4)—C(14)	129·1 (9)	O(3)—C(15)—C(16)	108·3 (8)
C(5)—C(4)—C(14)	123·5 (9)	N(2)—C(16)—C(15)	110·9 (9)
O(1)—C(5)—C(4)	107·8 (9)	N(2)—C(16)—C(17)	105·9 (10)
O(1)—C(5)—C(6)	116·2 (10)	C(15)—C(16)—C(17)	111·9 (11)
C(4)—C(5)—C(6)	135·9 (11)	N(2)—C(16)—C(18)	109·2 (11)
C(10)—C(9)—C(8)	120·0 (1)	C(15)—C(16)—C(18)	107·8 (9)
C(9)—C(10)—C(11)	120·0 (1)	C(17)—C(16)—C(18)	111·2 (9)
C(10)—C(11)—C(12)	120·0 (1)		

Maximum Δ/σ for the final refinement cycle was 0·013 (0·003 mean). The maximum and minimum peaks on the final Fourier synthesis were 1·175 and −0·75 e Å^{-3}, respectively. The large maximum peak was found to be 0·8 Å from I. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).*

Fig. 1 shows the thermal ellipsoids with the atomic numbering scheme for (I). The atomic coordinates and isotropic temperature factors for all non-H atoms are shown in Table 1. Bond lengths and angles are listed in Table 2.

Related literature. The relationship between the isoxazole and phenyl rings for (I) compares favorably to the 3-phenylisoxazole dihedral angles found when the C(4) isoxazole substituent was an secondary carboxamide (31·2°; Verner, Oliver Schlicksupp & Natale, 1990) or a 4-substituted 1,4-dihydropyridine in an O-*endo* conformation (33·0°; Schauer, Anderson, Natale & Quincy, 1986), but is much smaller than that found when the C(4) group was a 4-substituted 1,4-dihydropyridine in an O-*exo* conformation (83·5°; McKenna, Schlicksupp, Natale, Willett, Maryanoff & Flaim, 1988). The dihedral angle between the ester moiety [defined by the mean plane containing C(4), C(14), O(2) and O(3)] and the isoxazole mean plane is 47·2 (7)° for the title compound [(I)], and again is fairly close to that of the previously cited secondary carboxamide (53·7°).

The Syntex $P2_1$ X-ray diffractometer and crystallographic computing system was purchased with funds provided by the National Science Foundation (Grant CHE-8408407) and The Boeing Company. NRN thanks the National Institutes of General Medical Sciences for Grant Number 1-R15-GM4209-01.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53767 (17 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 Ethyl 3′-Ethyl-6-methoxy-5′-oxo-1,2-cyclopent-1′,3′-dienonaphthalene-4′-acetate

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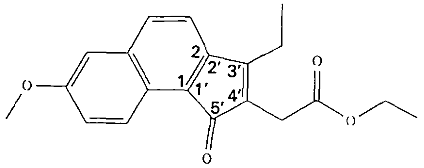
Abstract. $C_{20}H_{20}O_4$, $M_r = 324{\cdot}38$, monoclinic, $P2_1/c$, $a = 12{\cdot}514$ (1), $b = 15{\cdot}067$ (1), $c = 9{\cdot}588$ (1) Å, $\beta = 107{\cdot}47$ (1)°, $V = 1724{\cdot}4$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}249$ Mg m^{-3}, λ(Cu $K\bar{\alpha}$) = 1·54178 Å, $\mu = 0{\cdot}665$ mm^{-1}, $F(000) = 688$, $T = 294$ K, final $R = 0{\cdot}057$ for 1972 unique observed reflections with $I > 3\sigma(I)$. The cyclopentenonaphthalene ring system is planar (mean deviation, 0·014 Å). The crystal contains molecules held together by van der Waals forces (m.p. 390–391 K). Bond lengths and angles are internally consistent and the van der Waals interactions are normal. Averaged C—O bond lengths are: C(sp^2)—O = 1·338 (20); C(sp^3)—O = 1·428 (14) and C═O = 1·210 (1) Å.

Experimental. Clear bright-red needle-shaped crystals of the title compound were sequentially synthesized by a Friedel–Crafts reaction, a Stobbe condensation and cyclization (Hwang, 1962).



(1)

Crystal size 0·53 × 0·16 × 0·06 mm, glass-fiber mount, Enraf–Nonius CAD-4F four-circle diffrac-

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