Chemistry of 3-Oxygenated Isoxazoles. Formation and X-Ray Analysis of Ethyl (*RS*)-6-*tert*-Butyl-5-methyl-4-oxo-2,3-dihydro-4*H*-1,3-oxazine-2-carboxylate, an Isoxazol-3(2*H*)-one Rearrangement Product

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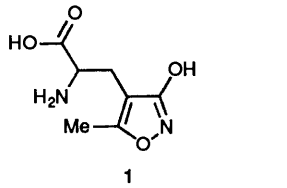
Treatment of 5-*tert*-butyl-4-methylisoxazole-3-ol **8** with ethyl chloroacetate under basic conditions gave ethyl (5-*tert*-butyl-4-methylisoxazol-3-yloxy)acetate **9** and the unexpected products ethyl (*RS*)-6-*tert*-butyl-5-methyl-4-oxo-2,3-dihydro-4*H*-1,3-oxazine-2-carboxylate **14** and (*RS*)-2,4,4-trimethyl-3-oxopentanamide **15**. The structure of **14** was determined by an X-ray analysis. It is proposed that formation of **14** proceeds through the isoxazol-3-(2*H*)-one intermediate **10**, the acetate carbanion **11** and the dioxo imine intermediate **12**. Compound **15**, which does not seem to be a degradation product of **14**, may be formed by hydrolysis of the dioxo imine **12**.

The neuroexcitatory actions of (*S*)-glutamic acid, the major excitatory amino acid (EAA) neurotransmitter in the mammalian central nervous system,[1–3] are mediated by multiple receptors, notably the *N*-methyl-D-aspartic acid (NMDA), the 2-amino-3-(3-hydroxy-5-methylisoxazol-4-yl)propionic acid (AMPA) and the kainic acid receptors.[1–5] These EAA receptor subtypes are named after the respective selective agonists, NMDA, AMPA **1**, and kainic acid.

Hyperactivation of central EAA receptors appears to play an important role in the destruction of neurones in certain neurodegenerative disorders such as Alzheimer's disease and Huntington's chorea.[6] There is a therapeutic interest in antagonists for the central EAA receptors.[1–5] Furthermore, selective antagonists for EAA receptor subtypes are indispensable for studies of EAA neurotransmitter mechanisms in the brain.



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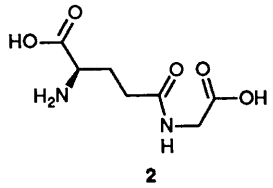

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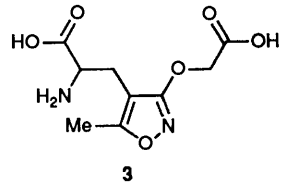

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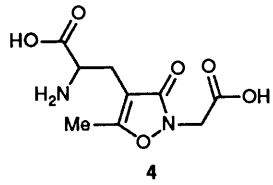
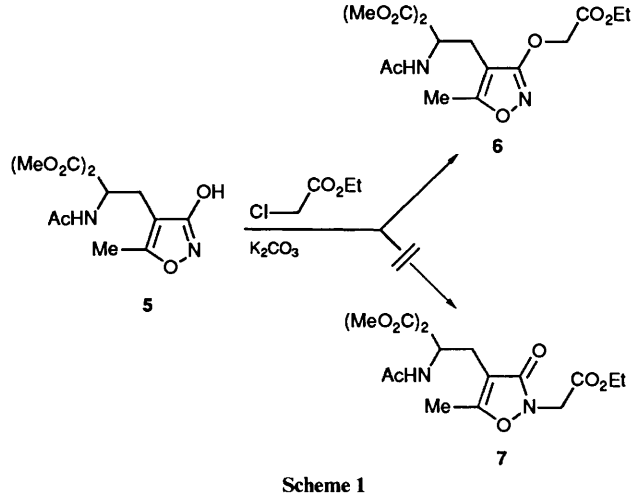


4

A number of compounds have been shown to block NMDA receptors competitively[4,7] or non-competitively.[2,3,5] Certain quinoxalinediones, which are structurally unrelated to AMPA **1** or kainic acid, antagonize neuronal excitation mediated by AMPA and kainic acid receptors.[4,8]

Furthermore, certain compounds derived from glutamic acid, *e.g.* γ-D-glutamylglycine **2**, are non-selective antagonists at EAA receptors.[4,7] By using **2** and the highly selective AMPA receptor agonist, AMPA **1**, as lead structures we have recently developed the selective non-NMDA antagonist (*RS*)-2-amino-3-[3-(carboxymethoxy)-5-methylisoxazol-4-yl]propionic acid (AMOA, **3**).[9] Within the framework of this EAA receptor antagonist project, the isomeric compound (*RS*)-2-amino-3-[2-(carboxymethyl)-5-methyl-3(2*H*)-oxoisoxazol-4-yl]propionic acid **4**, which is a heterocyclic bioisostere of **2**, was considered a molecule of primary interest.



Scheme 1

So far, attempts to synthesize compound **4** have been unsuccessful.[9] Treatment of the protected form **5** (Scheme 1) of AMPA **1** with ethyl chloroacetate gave only **6**, whereas the expected second reaction product **7**, considered an intermediate for the preparation of **4**, could not be isolated from the reaction mixture. Compound **6** was easily deprotected to give AMOA **3**. Compound **6** was only obtained in relatively low yield (28%),[9] and alkylation of isoxazol-3-ols has previously been shown to give mixtures of *O*- and *N*-alkylated products.[10–13] Thus, it is likely that the expected *N*-alkylated compound **7** is formed during this reaction, but impossible to isolate due to instability. In this paper we describe studies on a similar reaction between chloroacetate and the simple isoxazol-3-ol **8**, which may explain why the expected *N*-alkylated product **7** (Scheme 1) could not be isolated from the reaction mixture.

Results and Discussion

Treatment of a solution of 5-*tert*-butyl-4-methylisoxazol-3-ol **8** in acetone with ethyl chloroacetate in the presence of anhydrous potassium carbonate gave the expected *O*-alkylated reaction product ethyl (5-*tert*-butyl-4-methylisoxazol-3-yloxy)acetate **9** and the unexpected product ethyl (*RS*)-6-*tert*-butyl-5-methyl-4-oxo-2,3-dihydro-4*H*-1,3-oxazine-2-carboxylate **14** in yields of 27 and 21%, respectively. Furthermore, (*RS*)-2,4,4-trimethyl-3-oxopentanamide **15** was isolated in a small amount (10%) from

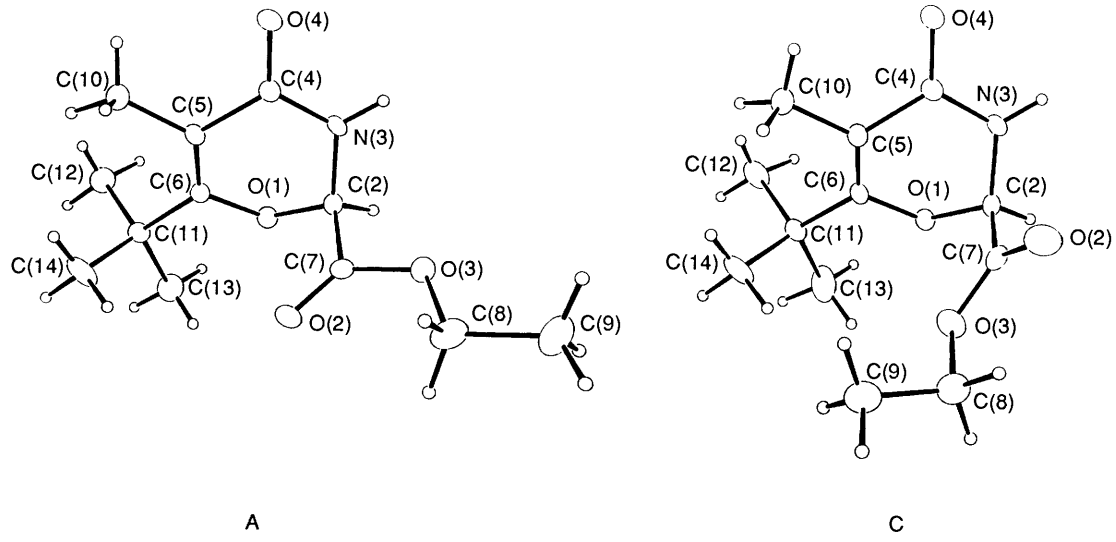
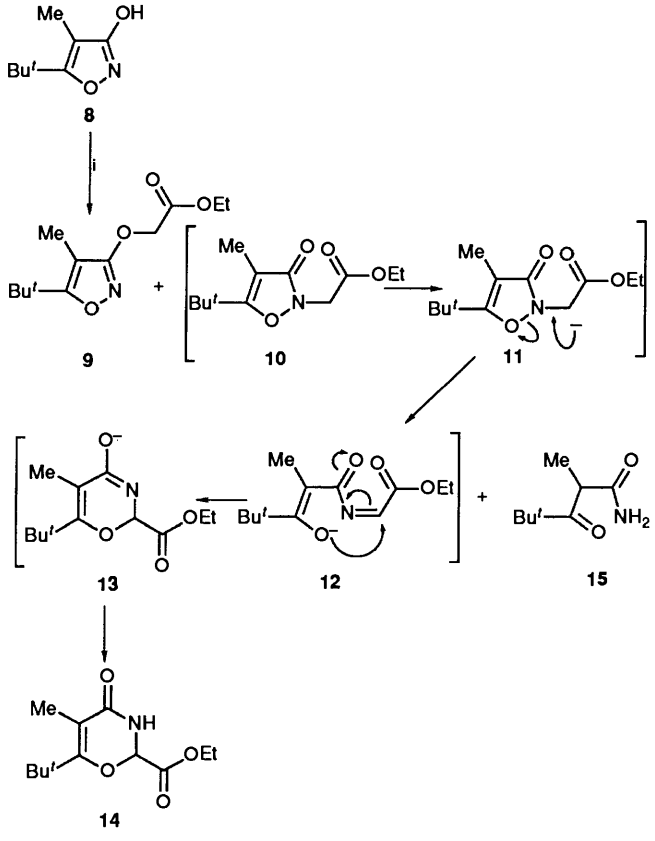


Fig. 1 Perspective drawings with numbering system of two (A and C) of the three (A, B and C) independent molecules in the asymmetric unit of compound 14.

The overall shape of the two molecules A and C are almost the same, except for the conformations of the ester groups. The overall shape of molecule B (not shown) is almost identical with that of molecule A, except for the *tert*-butyl group, which is rotated *ca.* 30° in molecule B compared to molecule A. In all of the three molecules the six membered rings adopt distorted half-boat conformations (Table 1).

The thermal ellipsoids for the non-hydrogen atoms correspond to 50% probability; hydrogen atoms are represented as spheres of arbitrary radius.



Scheme 2 *Reagents:* i, K_2CO_3, $ClCH_2COOC_2H_5$

the reaction mixture (Scheme 2). The structure of **14** has been established by an X-ray analysis. Selected bond lengths, valency and torsion angles of the three (A, B and C) independent molecules in the asymmetric unit, and dimensions of intermolecular hydrogen bonds are given in Table 1. Perspective drawings of molecule A and molecule C are shown in Fig. 1.

We believe that compound **14** is generated *via* the initially formed *N*-alkylated product **10**. The postulated rearrangement process may be initiated by the formation of the α-carbanion **11**, followed by a ring opening to give the dioxo imine anion **12**. An analogous anionic intermediate has been proposed to be generated during the rearrangement of 3-benzyloxy-5-methylisoxazole under strongly basic conditions.[13] An intramolecular nucleophilic attack of the enolate ion on the imine carbon atom of **12** can explain the formation of the 4-oxo-2,3-dihydro-4*H*-1,3-oxazine derivative **14** *via* the corresponding anion **13**.

Conversion of **9** into **14**, under the reaction conditions used, does not take place, as prolonged treatment of **9** with anhydrous potassium carbonate in acetone (50 °C) did not cause significant decomposition or transformation of **9**. Under similar conditions compound **14** did not undergo any detectable transformation into **15**. We assume that compound **15** is formed by hydrolytic cleavage of the proposed intermediate **12** due to the presence of small amounts of water in the reaction mixture.

In light of these observations, we have re-examined the reaction between **5** and ethyl chloroacetate (Scheme 1). In agreement with earlier findings,[9] only compound **6** could be isolated in a pure form from a complex reaction mixture. Attempts to detect in, or isolate from, this reaction mixture the 4-oxo-2,3-dihydro-4*H*-1,3-oxazine and/or β-oxocarboxamide equivalents of **14** and **15**, respectively, as potential rearrangement products of **7** were, however, unsuccessful. We tentatively conclude that these acetamidomalonates have escaped detection and isolation due to instability.

Experimental

M.p.s were determined in capillary tubes and are corrected. Elemental analyses were performed by Mr. G. Cornali, Microanalytical Laboratory, Leo Pharmaceutical Products, DK-2750 Ballerup, Denmark. IR spectra, obtained on a Perkin-Elmer 781 Infrared Spectrophotometer, were recorded in KBr pellets. ^1H NMR spectra were recorded on a Varian EM360L spectrometer using TMS as an internal standard, *J* values are given in Hz. TLC and gravity column chromatography were performed on silica F_{254} plates (Merck) and silica gel (Woelm, 0.063–0.200 mm), respectively. Evaporations were performed at temperatures below 50 °C, using a vacuum rotatory evaporator

Table 1 Selected bond lengths, valency and torsion angles for the three independent molecules A, B and C in the asymmetric unit of compound **14**, and dimensions of intermolecular hydrogen bonds. Estimated standard deviations are given in parentheses

(a) Bond lengths (Å)

	Length (Å)		Length (Å)
O(1)–C(2)	A 1.410(2)	C(7)–O(3)	A 1.336(2)
	B 1.409(2)		B 1.329(2)
	C 1.424(2)		C 1.329(2)
C(2)–N(3)	A 1.438(2)	O(3)–C(8)	A 1.453(2)
	B 1.430(2)		B 1.462(2)
	C 1.430(2)		C 1.466(2)
N(3)–C(4)	A 1.351(2)	C(8)–C(9)	A 1.503(3)
	B 1.354(2)		B 1.501(3)
	C 1.347(2)		C 1.490(3)
C(4)–C(5)	A 1.481(2)	C(4)–O(4)	A 1.240(2)
	B 1.481(2)		B 1.239(2)
	C 1.483(2)		C 1.240(2)
C(5)–C(6)	A 1.351(2)	C(5)–C(10)	A 1.501(2)
	B 1.353(2)		B 1.504(2)
	C 1.355(2)		C 1.505(2)
C(6)–O(1)	A 1.374(2)	C(6)–C(11)	A 1.529(2)
	B 1.384(2)		B 1.527(2)
	C 1.375(2)		C 1.527(2)
C(2)–C(7)	A 1.539(2)	C(11)–C(12)	A 1.536(2)
	B 1.545(2)		B 1.530(3)
	C 1.547(2)		C 1.537(3)
C(7)–O(2)	A 1.206(2)	C(11)–C(13)	A 1.529(3)
	B 1.207(2)		B 1.529(3)
	C 1.198(2)		C 1.527(3)
C(11)–C(14)	A 1.534(3)		
	B 1.532(2)		
	C 1.537(3)		

(b) Valency angles (°)

	Angle (°)		Angle (°)
C(6)–O(1)–C(2)	A 116.1(1)	O(1)–C(6)–C(11)	A 110.4(1)
	B 115.0(1)		B 110.2(1)
	C 113.5(1)		C 111.5(1)
O(1)–C(2)–N(3)	A 112.1(1)	C(2)–C(7)–O(2)	A 126.1(2)
	B 111.6(1)		B 125.2(2)
	C 110.5(1)		C 120.8(2)
O(1)–C(2)–C(7)	A 108.6(1)	C(2)–C(7)–O(3)	A 108.3(1)
	B 110.0(1)		B 109.0(1)
	C 111.5(1)		C 113.6(1)
N(3)–C(2)–C(7)	A 112.6(1)	O(2)–C(7)–O(3)	A 125.6(2)
	B 112.9(1)		B 125.7(2)
	C 110.8(1)		C 125.4(2)
C(2)–N(3)–C(4)	A 119.8(1)	C(7)–O(3)–C(8)	A 115.7(1)
	B 118.8(1)		B 116.4(1)
	C 117.2(1)		C 116.9(1)
N(3)–C(4)–C(5)	A 116.3(1)	O(3)–C(8)–C(9)	A 106.5(2)
	B 115.8(1)		B 107.4(2)
	C 115.6(1)		C 109.9(2)
N(3)–C(4)–O(4)	A 121.7(1)	C(6)–C(11)–C(12)	A 109.6(1)
	B 121.8(1)		B 112.4(1)
	C 122.4(1)		C 110.0(1)
C(5)–C(4)–O(4)	A 121.9(1)	C(6)–C(11)–C(13)	A 110.9(1)
	B 122.2(1)		B 110.5(1)
	C 121.8(2)		C 111.0(2)
C(4)–C(5)–C(6)	A 118.6(1)	C(6)–C(11)–C(14)	A 110.4(1)
	B 118.1(1)		B 108.7(1)
	C 118.2(1)		C 110.2(1)
C(4)–C(5)–C(10)	A 114.7(1)	C(12)–C(11)–C(13)	A 107.5(2)
	B 115.2(1)		B 106.2(2)
	C 115.4(1)		C 108.5(2)
C(6)–C(5)–C(10)	A 126.4(2)	C(12)–C(11)–C(14)	A 111.5(2)
	B 126.3(1)		B 110.0(2)
	C 126.0(2)		C 110.2(2)
C(5)–C(6)–O(1)	A 121.2(1)	C(13)–C(11)–C(14)	A 106.9(2)
	B 120.3(1)		B 109.1(2)
	C 120.4(1)		C 107.1(2)
C(5)–C(6)–C(11)	A 128.4(1)		
	B 129.6(2)		
	C 128.0(1)		

Table 1 (*continued*)

(c) Torsion angles (°)

	Angle (°)		Angle (°)
O(6)–O(1)–C(2)–N(3)	A −44.9(2)	N(3)–C(2)–C(7)–O(3)	A −69.2(2)
	B −48.8(2)		B −65.1(2)
	C −52.6(2)		C 146.6(2)
O(1)–C(2)–N(3)–C(4)	A 40.2(2)	O(2)–C(7)–O(3)–C(8)	A −4.8(3)
	B 44.2(2)		B −5.5(3)
	C 51.0(2)		C 1.3(3)
C(2)–N(3)–C(4)–C(5)	A −12.3(3)	C(7)–O(3)–C(8)–C(9)	A 169.9(2)
	B −10.7(3)		B 166.5(2)
	C −16.4(3)		C 112.9(2)
N(3)–C(4)–C(5)–C(6)	A −11.6(3)	O(4)–C(4)–C(5)–	A −8.9(3)
	B −19.1(3)	C(10)	B −17.4(3)
	C −16.9(3)		C −13.6(3)
C(4)–C(5)–C(6)–O(1)	A 6.2(3)	C(10)–C(5)–C(6)–	A 2.4(3)
	B 14.0(3)	C(11)	B 8.3(3)
	C 14.4(3)		C 10.1(3)
C(5)–C(6)–O(1)–C(2)	A 23.1(2)	C(5)–C(6)–C(11)–	A 64.4(3)
	B 20.6(2)	C(12)	B 35.6(3)
	C 21.0(2)		C 60.4(3)
O(1)–C(2)–C(7)–O(2)	A −11.3(3)	C(5)–C(6)–C(11)–	A −58.8(3)
	B −8.4(3)	C(14)	B −86.5(3)
	C −160.5(2)		C −61.2(3)
N(3)–C(2)–C(7)–O(2)	A 113.4(2)	O(1)–C(6)–C(11)–	A 6.5(2)
	B 116.9(2)	C(13)	B −24.7(2)
	C −37.0(3)		C 4.2(3)
O(1)–C(2)–C(7)–O(3)	A 166.1(2)		
	B 169.6(2)		
	C 23.1(2)		

Intermolecular hydrogen bond distances (Å) and angles (°)[a]

$X–H_{Molecule}$ $Y_{Molecule}$	X–H (Å)	H···Y (Å)	X···Y (Å)	XHY (°)
$N(3)–H_A\cdots O_A(4)^{i}$	0.86(2)	1.97(2)	2.830(2)	173(2)
$N(3)–H_B\cdots O_C(4)^{ii}$	0.84(2)	1.97(2)	2.810(2)	173(2)
$N(3)–H_C\cdots O_B(4)^{ii}$	0.89(2)	2.02(2)	2.893(2)	174(2)

[a] Symmetry code: (i) $-x, 1-y, 1-z$; (ii) $-x, 1-y, -z$.

connected to a water aspirator. Light petroleum was the fraction boiling < 50 °C.

Ethyl (5-tert-*Butyl*-4-*methylisoxazol*-3-*yloxy*)*acetate* **9**, *Ethyl* (RS)-6-tert-*Butyl*-5-*methyl*-4-*oxo*-2,3-*dihydro*-4H-1,3-*oxazine*-2-*carboxylate* **14** *and* (RS)-2,4,4-*Trimethyl*-3-*oxopentanamide* **15**.—To a solution of **8**[14] (5.65 g, 36 mmol) in dry acetone (100 cm^3) was added anhydrous potassium carbonate (12.6 g, 91 mmol). After stirring of this suspension at 50 °C for 1 h, ethyl chloroacetate (11.5 cm^3, 109 mmol) was added and stirring at 50 °C was continued for 20 h. The filtered and evaporated reaction mixture was left at −20 °C for 3 d, and then diluted with toluene (15 cm^3) and the suspension of partially crystallized product, crude **15**, was filtered off. Recrystallization (ethyl acetate–light petroleum) gave *compound* **15** (0.57 g, 10%), m.p. 136–138 °C; ν_{max}/cm^{-1} 3415s, 3185m, 2985–2800mw, 1710s, 1685s, 1635m, 1485–1455m, 1385s and 1360m; $\delta_H(CDCl_3)$ 6.4 (1 H, br s), 5.7 (1 H, br s), 3.94 (1 H, q, *J* 7), 1.40 (3 H, d, *J* 7) and 1.18 (9 H, s) (Found: C, 61.0; H, 9.65; N, 8.9. $C_8H_{15}NO_2$ requires C, 61.10; H, 9.61; N, 8.94%).

The evaporated filtrate, obtained after the isolation of crude **15**, was subjected to gravity column chromatography (150 g of silica gel). Elution with toluene containing ethyl acetate (10–30%) gave *compound* **9** (2.37 g, 27%), b.p. 121–122 °C (0.3 mmHg), m.p. 55–57 °C; ν_{max}/cm^{-1} 2980–2875mw, 1755s, 1635m, 1515s, 1485s, 1475–1435m and 1415m; $\delta_H(CDCl_3)$ 4.78 (2 H, s), 4.26 (2 H, q, *J* 7), 1.98 (3 H, s), 1.34 (9 H, s) and 1.29 (3 H, t, *J* 7) (Found: C, 59.3; H, 7.85; N, 5.55. $C_{12}H_{19}NO_4$ requires C, 59.72; H, 7.94; N, 5.83%), and *compound* **14** (1.85 g, 21%), m.p. 90–92 °C (ethyl acetate–light petroleum); ν_{max}/cm^{-1}

Table 2 Positional parameters for the three independent molecules (A, B and C) in the asymmetric unit of compound **14**. Estimated standard deviations are given in parentheses

Atom	Molecule	*x*	*y*	*z*
O(1)	A	0.264 5(1)	0.494 8(1)	0.746 75(9)
	B	0.219 7(1)	0.159 3(1)	0.451 8(1)
	C	0.330 2(1)	0.873 1(1)	0.022 20(9)
C(2)	A	0.260 2(2)	0.540 7(2)	0.659 3(1)
	B	0.220 6(2)	0.220 3(2)	0.373 8(1)
	C	0.302 1(2)	0.904 2(2)	−0.072 3(1)
N(3)	A	0.133 6(2)	0.520 2(1)	0.603 5(1)
	B	0.095 4(2)	0.212 9(1)	0.321 3(1)
	C	0.178 7(2)	0.846 4(1)	−0.122 6(1)
C(4)	A	0.035 9(2)	0.525 1(2)	0.650 3(1)
	B	0.002 7(2)	0.220 9(1)	0.373 0(1)
	C	0.082 3(2)	0.846 5(2)	−0.073 9(1)
C(5)	A	0.063 1(2)	0.529 3(2)	0.758 4(1)
	B	0.035 1(2)	0.217 1(2)	0.479 7(1)
	C	0.119 3(2)	0.879 8(2)	0.034 0(1)
C(6)	A	0.173 5(2)	0.509 0(1)	0.800 8(1)
	B	0.134 7(2)	0.175 3(2)	0.512 3(1)
	C	0.238 6(2)	0.879 9(2)	0.077 8(1)
C(7)	A	0.322 4(2)	0.661 6(2)	0.685 6(1)
	B	0.293 7(2)	0.337 4(2)	0.412 5(1)
	C	0.313 7(2)	1.027 0(2)	−0.065 3(1)
C(8)	A	0.398 6(2)	0.814 2(2)	0.613 7(2)
	B	0.373 8(2)	0.503 1(2)	0.359 8(2)
	C	0.408 4(2)	1.200 2(2)	0.026 6(2)
C(9)	A	0.436 8(2)	0.835 3(2)	0.517 5(2)
	B	0.413 6(2)	0.538 5(2)	0.268 1(2)
	C	0.348 8(2)	1.239 7(2)	0.108 0(2)
C(10)	A	−0.044 6(2)	0.543 8(2)	0.808 5(2)
	B	−0.056 8(2)	0.249 6(2)	0.539 7(1)
	C	0.013 4(2)	0.896 8(2)	0.085 9(1)
C(11)	A	0.214 5(2)	0.491 8(2)	0.906 9(1)
	B	0.171 7(2)	0.137 7(2)	0.610 8(1)
	C	0.288 8(2)	0.879 8(2)	0.186 5(1)
C(12)	A	0.128 4(2)	0.389 5(2)	0.927 4(2)
	B	0.056 5(2)	0.085 0(2)	0.651 4(2)
	C	0.217 5(2)	0.778 7(2)	0.220 6(2)
C(13)	A	0.350 2(2)	0.476 8(2)	0.924 7(2)
	B	0.249 3(2)	0.052 6(2)	0.599 5(2)
	C	0.429 7(2)	0.880 6(3)	0.204 4(2)
C(14)	A	0.212 2(2)	0.590 6(2)	0.978 7(2)
	B	0.251 7(3)	0.234 4(2)	0.684 2(2)
	C	0.273 8(3)	0.981 4(2)	0.248 3(2)
O(2)	A	0.351 0(1)	0.712 6(1)	0.767 0(1)
	B	0.331 0(1)	0.374 9(1)	0.498 0(1)
	C	0.263 5(2)	1.064 5(1)	−0.131 6(1)
O(3)	A	0.344 2(1)	0.699 6(1)	0.603 0(1)
	B	0.312 1(1)	0.388 9(1)	0.336 4(1)
	C	0.388 3(2)	1.082 9(1)	0.016 5(1)
O(4)	A	−0.072 1(1)	0.519 8(1)	0.604 4(1)
	B	−0.105 1(1)	0.223 9(1)	0.332 3(1)
	C	−0.030 1(1)	0.814 5(1)	−0.115 3(1)

3175m, 3060m, 2980–2865sm, 1750s, 1675s, 1610w, 1480m and 1410m; $\delta_H(CDCl_3)$ 6.9 (1 H, br s), 5.36 (1 H, d, *J* 3), 4.29 (2 H, q, *J* 7), 1.92 (3 H, s), 1.32 (3 H, t, *J* 7) and 1.28 (9 H, s) (Found: C, 59.85; H, 7.95; N, 5.65. $C_{12}H_{19}NO_4$ requires C, 59.72; H, 7.94; N, 5.83%).

X-Ray Crystallographic Analysis of Ethyl (RS)-6-tert-*Butyl*-5-*methyl*-4-*oxo*-2,3-*dihydro*-4H-1,3-*oxazine*-2-*carboxylate* **14**.—The colourless needle shaped crystals used for the X-ray examination were crystallized from ethyl acetate–light petroleum.

Crystal data. $C_{12}H_{19}NO_4$, M = 241.29. Triclinic, a = 10.991(2), b = 12.857(1), c = 13.931(1) Å, α = 96.305(7), β = 99.22(1), γ = 101.93(1)°, U = 1880.3(8) Å^3, space group $P\bar{1}$ (no. 2), Z = 6, D_c = 1.278 g cm^{-3}, μ(Mo-Kα) = 0.89 cm^{-1}.

Data collection and processing. A single crystal of the size 0.13 × 0.20 × 0.50 mm was used for the determination of the unit cell parameters, and for the collection of intensity data. The measurements were performed at 110(±5) K on an Enraf-Nonius CAD-4 diffractometer. The crystal was cooled in a stream of nitrogen gas provided by an Enraf-Nonius low-temperature device. The temperature was kept constant within 1 K during the experiment. Graphite monochromated Mo-Kα radiation (λ = 0.710 73 Å) was used. Intensities of a hemisphere (θ < 28°, h 0 ⟶ 14, k − 16 ⟶ 16, l − 18 ⟶ 18) were measured using the $\omega - 2\theta$ scan mode. Intensities of three reflections measured every 10^4 s showed no unusual variation over the course of the experiment. Of the 9054 unique reflections, 5974 were classified as observed, *i.e.* had $|F_o|^2 \geq 3\sigma(|F_o|^2)$ where $\sigma(|F_o|^2) = [\sigma_c^2(|F_o|^2) + (0.07|F_o|^2)^2]^{\frac{1}{2}}$. The $\sigma(|F_o|^2)$ was calculated from counting statistics. No absorption corrections were made.

Structure Solution and Refinement. The positions of the non-hydrogen atoms were obtained by application of 'direct methods' (SHELX86).[15,16] The hydrogen atoms were subsequently located on a series of difference Fourier maps. Final full-matrix least-squares calculations[17] included an overall scale factor, atomic coordinates for all atoms, anisotropic thermal parameters for the non-hydrogen atoms, and isotropic thermal parameters for the hydrogen atoms. Final R and R_w values are 0.047 and 0.065, respectively. The quantity minimized was $\Sigma w(|F_o| - k|F_c|)^2$ with $w^{-1} = \sigma^2(|F_o|)$, $\sigma(|F_o|) = \sigma(|F_o|^2)/2|F_o|$. Residual electron density in final difference Fourier maps is within +0.39 and −0.28 e Å^{-3}.

All calculations, except the structure solution, were carried out by using the Enraf-Nonius Structure Determination Package.[17] The scattering factors were taken from Cromer and Mann,[18] except for hydrogen, which was taken from Stewart *et al.*[19]

Table 2 lists the final positional parameters of the non-hydrogen atoms.

Final positional parameters for hydrogen atoms, anisotropic and isotropic thermal parameters for non-hydrogen atoms and hydrogen atoms, respectively, and bond lengths and angles involving hydrogen atoms are available on request from the Cambridge Crystallographic Data Centre.*

* For details see para. 5.6.3 Instructions for Authors (1991), *J. Chem. Soc., Perkin Trans. 1*, 1991, Issue 1.

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