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2-*tert*-Butyloxymethyl-6-cyano-2,3-dihydro-5*H*-oxazolo[3,2-*a*]pyrimidin-5-one

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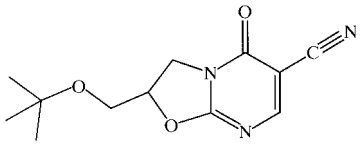
The condensation reaction of 2-amino-5-*tert*-butyloxymethyl-2-oxazoline with ethyl cyano(ethoxymethylene)acetate led to the title cycloadduct. The structure indicates a delocalization in the pyrimidine ring.

Comment

2–Amino-2-oxazolines are found useful synthons for the preparation of various pyrimidine derivatives (Kampe, 1974; Forfar *et al.*, 1990; Chaimbault *et al.*, 1999). As an example, 5-*tert*-butyloxymethyl-2-amino-2-oxazoline reacted with ethyl cyano(ethoxymethylene)acetate (EMCA) to yield 2-*tert*-butyloxymethyl-6-cyano-2,3-dihydro-5*H*-oxazolo[3,2-*a*]pyrimidin-5-one, (I). Its structure was established by X-ray crystallography. The analysis of bond lengths and angles indicated a π delocalization on the pyrimidine cycle. The fused rings are found almost coplanar. The orientation of the *tert*-butyloxymethyl chain is defined by the torsion angle C6—O7—C8—C9 = −140.8 (3)°. The crystalline cohesion is ensured by intermolecular van der Waals contacts.



(I)

Experimental

Ethyl cyano(ethoxymethylene)acetate (50 mmol) is added dropwise to a solution of 2-amino-5-*tert*-butyloxymethyl-1,3-oxazoline (50 mmol) in ethanol (150 ml). The solution is stirred magnetically at room temperature overnighis any delat. The solvent is evaporated under reduced pressure and the product is recrystallized from tetrachloroethylene (77%). Colourless crystals were grown in ethanol and had m.p. = 375 K.

Crystal data

$C_{12}H_{15}N_3O_3$	D_x = 1.312 Mg m^{-3}
M_r = 249.27	Cu $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
a = 11.903 (1) Å	θ = 3.95–64.93°
b = 10.895 (2) Å	μ = 0.798 mm^{-1}
c = 10.343 (1) Å	T = 296 (2) K
β = 109.76 (1)°	Plate, colourless
V = 1262.3 (3) Å^3	0.25 × 0.25 × 0.02 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	1243 reflections with $I > 2\sigma(I)$
ω–2θ scans	θ_{max} = 64.93°
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 13$
T_{min} = 0.854, T_{max} = 0.999	$k = 0 \rightarrow 12$
2060 measured reflections	$l = -12 \rightarrow 11$
2060 independent reflections	2 standard reflections every 90 reflections
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0738P)^2 + 0.5706P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.057	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.1312	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
S = 1.120	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
2059 reflections	Extinction correction: *SHELXL*93
164 parameters	Extinction coefficient: 0.0023 (5)
H-atom parameters not refined	

Table 1
Selected geometric parameters (Å, °).

C3—C6	1.500 (5)	C11—N12	1.340 (4)
C4—C6	1.498 (5)	N12—C17	1.379 (4)
C5—C6	1.494 (5)	N12—C13	1.456 (4)
C6—O7	1.450 (4)	N14—C15	1.356 (4)
O7—C8	1.416 (4)	C15—C16	1.362 (4)
C8—C9	1.506 (5)	C16—C18	1.429 (5)
C9—O10	1.469 (4)	C16—C17	1.431 (4)
C9—C13	1.525 (5)	C17—O20	1.224 (4)
O10—C11	1.326 (4)	C18—N19	1.147 (4)
C11—N14	1.302 (4)		
O7—C6—C5	111.0 (3)	O10—C11—N12	112.0 (3)
O7—C6—C3	103.4 (3)	C11—N12—C17	122.6 (3)
C5—C6—C3	111.0 (4)	C11—N12—C13	111.0 (3)
O7—C6—C4	110.3 (3)	C17—N12—C13	126.3 (3)
C5—C6—C4	110.2 (4)	N12—C13—C9	101.3 (3)
C3—C6—C4	110.8 (4)	C11—N14—C15	113.1 (3)
C8—O7—C6	117.4 (3)	N14—C15—C16	124.8 (3)
O7—C8—C9	108.4 (3)	C15—C16—C18	121.2 (3)
O10—C9—C8	107.2 (3)	C15—C16—C17	120.6 (3)
O10—C9—C13	104.1 (3)	C18—C16—C17	118.2 (3)
C8—C9—C13	115.2 (3)	O20—C17—N12	120.7 (3)
C11—O10—C9	108.1 (2)	O20—C17—C16	127.3 (3)
N14—C11—O10	121.1 (3)	N12—C17—C16	112.0 (3)
N14—C11—N12	126.9 (3)	N19—C18—C16	178.8 (4)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: Nonius (unpublished); program(s) used to solve structure:

*SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

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