

## ORGANIC COMPOUNDS

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**1-*tert*-Butyl-2-(*tert*-butylamino-methylidene)-3-pyrrolidinone, C<sub>13</sub>H<sub>24</sub>N<sub>2</sub>O**

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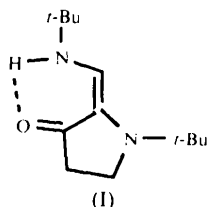
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**Abstract**

The enamine moiety on the pyrrolidinone ring is coplanar to the carbonyl function, with an *E* configuration stabilized by an intramolecular N—H···O bond.

**Comment**

The molecular structure of the title compound (I) contains a pyrrolidinone ring with the enamine fragment in the *E* configuration stabilized by an intramolecular N—H···O bond [N—H 0.92 (2), H···O 2.02 (2), N···O 2.754 (3) Å; N—H···O 135 (2)°]. The enamine system is fully conjugated with the carbonyl function, as indicated by the short 'Bu(H)N—C single bond [1.334 (3) Å] and the relatively long olefinic C=C bond [1.360 (4) Å]. The H—N—C=C—C=O moiety is planar. This confirms the NMR data as well as the assignment of the structure in solution of the isomer of the parent compound, 1-*tert*-butyl-5-(*tert*-butylimino)-2-pyrrolidinone.



The pyrrolidinone ring is slightly puckered. The Cremer & Pople (1975) puckering parameters [ $Q(2) = 0.223$  (3) Å,  $\varphi(2) = 338.1$  (8)°] indicate a half-chair configuration. The twist about an approximate twofold axis running through C=O is illustrated most clearly by

the low  $C_2(C7)$  asymmetry parameter [1.3 (3)°] (Duax, Weeks & Rohrer, 1976).

The geometry of both *tert*-butyl moieties deviates significantly from trigonal symmetry, due to intramolecular crowding [*e.g.* the *trans* bond angle N1—C1—C13, 105.8 (2)°, is significantly smaller than the two *gauche* bond angles N1—C1—C11, 109.9 (2)°, and N1—C1—C12, 110.2 (2)°]. Similar effects have been reported by de Vos, Kroon, Brouwer & Bos (1981). The structure contains no residual solvent-accessible areas.

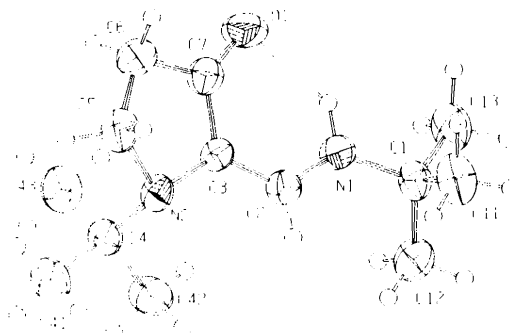


Fig. 1. Structure of 1-*tert*-butyl-2-(*tert*-butylaminomethylidene)-3-pyrrolidinone, with displacement ellipsoids at 50% probability.

**Experimental**

The preparation of the title compound was performed according to the procedure described by Wissing, Kleijn, Boersma & Van Koten (1993). The crystal was mounted on top of a glass fibre.

*Crystal data*

C<sub>13</sub>H<sub>24</sub>N<sub>2</sub>O  
*M<sub>r</sub>* = 224.35  
 Monoclinic  
*P*2<sub>1</sub>/*c*  
*a* = 19.646 (2) Å  
*b* = 6.1490 (10) Å  
*c* = 11.466 (2) Å  
 $\beta$  = 95.250 (10)°  
*V* = 1379.3 (4) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.080 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 9–20°  
 $\mu$  = 0.064 mm<sup>-1</sup>  
*T* = 295 K  
 Plate shaped  
 0.50 × 0.35 × 0.35 mm  
 Yellowish

*Data collection*

Enraf-Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans [ $\Delta\omega = (1.00 + 0.35\tan\theta)^\circ$ ]  
 Absorption correction: none  
 5152 measured reflections  
 3170 independent reflections  
 1665 observed reflections [ $I > 2.5\sigma(I)$ ]

*R*<sub>int</sub> = 0.08  
 $\theta_{\max}$  = 27.5°  
*h* = -25 → 25  
*k* = -7 → 0  
*l* = 0 → 14  
 3 standard reflections  
 frequency: 30 min  
 intensity variation: 5%

## Refinement

Refinement on  $F$  $R = 0.0555$  $wR = 0.0478$  $S = 0.62$ 

1665 reflections

221 parameters

Only coordinates of H atoms refined

 $w = 1/\sigma^2(F)$  $(\Delta/\sigma)_{\max} = 0.33$  $\Delta\rho_{\max} = 0.18 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$ Atomic scattering factors  
from Cromer & Mann  
(1968)Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	$U_{eq}$
O1	0.30252 (11)	0.1314 (3)	0.43349 (18)	0.0695 (8)
N1	0.33334 (11)	0.5126 (4)	0.55298 (19)	0.0448 (7)
N2	0.18186 (10)	0.5417 (3)	0.33521 (15)	0.0383 (7)
C1	0.38595 (12)	0.6454 (4)	0.6193 (2)	0.0440 (9)
C2	0.28109 (13)	0.5892 (5)	0.4823 (2)	0.0385 (8)
C3	0.23989 (12)	0.4619 (4)	0.40900 (19)	0.0351 (8)
C4	0.11669 (12)	0.5560 (4)	0.3914 (2)	0.0409 (8)
C5	0.17865 (16)	0.3890 (5)	0.2327 (2)	0.0510 (10)
C6	0.21343 (17)	0.1794 (5)	0.2721 (3)	0.0575 (11)
C7	0.25757 (14)	0.2448 (5)	0.3818 (2)	0.0462 (9)
C11	0.43035 (17)	0.7569 (7)	0.5357 (3)	0.0662 (13)
C12	0.35274 (19)	0.8132 (6)	0.6923 (3)	0.0617 (11)
C13	0.42781 (19)	0.4880 (6)	0.6989 (3)	0.0670 (14)
C41	0.06300 (16)	0.6632 (6)	0.3055 (3)	0.0571 (11)
C42	0.12800 (18)	0.7038 (6)	0.4984 (3)	0.0605 (11)
C43	0.09049 (17)	0.3363 (5)	0.4311 (3)	0.0592 (11)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C7	1.234 (3)	C1—C13	1.519 (4)
N1—C1	1.473 (3)	C2—C3	1.360 (4)
N1—C2	1.334 (3)	C3—C7	1.421 (4)
N2—C3	1.442 (3)	C4—C41	1.525 (4)
N2—C4	1.488 (3)	C4—C42	1.527 (4)
N2—C5	1.501 (3)	C4—C43	1.530 (4)
C1—C11	1.517 (4)	C5—C6	1.508 (4)
C1—C12	1.514 (4)	C6—C7	1.515 (4)
C1—N1—C2	125.6 (2)	C2—C3—C7	122.2 (2)
C3—N2—C4	115.56 (17)	N2—C4—C41	108.5 (2)
C3—N2—C5	102.76 (19)	N2—C4—C42	108.4 (2)
C4—N2—C5	113.57 (19)	N2—C4—C43	113.8 (2)
N1—C1—C11	109.9 (2)	C41—C4—C42	107.6 (2)
N1—C1—C12	110.2 (2)	C41—C4—C43	110.0 (2)
N1—C1—C13	105.8 (2)	C42—C4—C43	108.3 (2)
C11—C1—C12	110.1 (3)	N2—C5—C6	108.3 (2)
C11—C1—C13	110.9 (2)	C5—C6—C7	103.4 (2)
C12—C1—C13	109.8 (2)	O1—C7—C3	127.2 (2)
N1—C2—C3	123.6 (3)	O1—C7—C6	125.4 (3)
N2—C3—C2	124.1 (2)	C3—C7—C6	107.4 (2)
N2—C3—C7	112.8 (2)		

Positional parameters of the H atoms were refined with four common isotropic thermal parameters. Data collection: locally modified *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *SET4* (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1993). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1986). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *PLATON*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71668 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1111]

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## 2,6-Difluorophenylxenon(II)-tetrafluoroborat

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## Abstract

The crystal structure of the compound 2,6-difluorophenylxenon(II) tetrafluoroborate is described. The Xe atom is bonded to a C atom and weakly coordinated by one F atom of the tetrafluoroborate anion.

## Kommentar

Seit kurzem kennt man Verbindungen mit Xenon-Kohlenstoff Bindungen (Naumann, Butler, Gnann & Tyrre, 1993 und dort genannte Literatur), von denen eine bereits durch