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4-Benzyl-3-(ethenylidene)azetidin-2-one: the First α-Vinylidene-β-lactam

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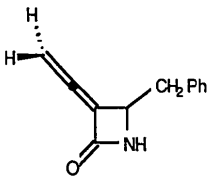
Abstract. $C_{12}H_{11}NO$, $M_r = 185{\cdot}23$, monoclinic, $P2_1/n$, $a = 7{\cdot}239$ (2), $b = 6{\cdot}984$ (2), $c = 19{\cdot}996$ (5) Å, $\beta = 93{\cdot}84$ (2)°, $V = 1008{\cdot}67$ (45) Å^3, $Z = 4$, $D_x = 1{\cdot}22$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·73 cm^{-1}, $F(000) = 392$, $T = 295$ K. Final $R = 0{\cdot}037$ for 1030 observed reflections. The atoms in the β-lactam ring and the allene moiety are coplanar. Each molecule is linked by two intermolecular hydrogen bonds [2·901 (3) Å] between O and N(4). This is the first example of an allene fused to a β-lactam ring.

Experimental. Crystals of the title compound (I) are colorless prisms. Crystal of dimensions 0·56 × 0·41 × 0·23 mm used. Accurate unit-cell parameters obtained by least-squares fit of 15 reflections in the range $10 < 2\theta < 25°$, space group $P2_1/n$ from systematic absences ($0k0$, k odd, $h0l$, $h+l$ odd); automatic Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$



(I)

radiation, $\theta/2\theta$ scan mode, variable scan rate (3·0–14·9° min^{-1} depending on intensity), 1660 measured reflections, 1326 independent reflections in the range $3 < 2\theta < 45°$, $R_{int} = 0{\cdot}009$, hkl range $h-7\rightarrow7$, $k0\rightarrow7$, $l0\rightarrow21$, 1030 observed reflectins with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change (~ 1·0%) in intensity during data collection; Lorentz–polarization correction, no absorption or extinction corrections. Direct-methods *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares using *SHELX*76 (Sheldrick, 1976), all non-hydrogen atoms anisotropic; H atoms located in difference Fourier maps included independently in the refinement with isotropic temperature factors; $w = 1/[\sigma^2(F) + 0{\cdot}000132F^2]$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0{\cdot}037$, $wR = 0{\cdot}039$; $(\Delta/\sigma)_{max} = 0{\cdot}04$, $\Delta\rho_{(max,\ min)} = 0{\cdot}19$, $-0{\cdot}11$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, N and O used were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters of the non-H atoms are given in Table 1.† The identification of the atoms and the configuration of the title compound

† Lists of structure factors, anisotropic temperature factors, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44082 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters for the non-hydrogen atoms with e.s.d.'s in parentheses*

	x	y	z	U_{eq}(Å^2)*
O	0·2125 (2)	0·8407 (2)	0·0213 (1)	0·0709 (4)
C(1)	0·1356 (3)	0·7761 (3)	−0·0297 (1)	0·0560 (6)
C(2)	0·1494 (3)	0·5976 (3)	−0·0705 (1)	0·0519 (5)
C(3)	−0·0087 (3)	0·6748 (3)	−0·1180 (1)	0·0582 (6)
N(4)	−0·0011 (3)	0·8458 (3)	−0·0730 (1)	0·0633 (5)
C(5)	0·2502 (3)	0·4459 (3)	−0·0668 (1)	0·0528 (5)
C(6)	0·3556 (3)	0·2950 (4)	−0·0618 (1)	0·0680 (6)
C(7)	−0·1876 (3)	0·5651 (3)	−0·1209 (1)	0·0606 (5)
C(8)	−0·3378 (3)	0·6589 (3)	−0·1659 (1)	0·0515 (6)
C(9)	−0·3733 (3)	0·6022 (3)	−0·2317 (1)	0·0576 (5)
C(10)	−0·5106 (3)	0·6873 (3)	−0·2724 (1)	0·0600 (6)
C(11)	−0·6146 (3)	0·8314 (4)	−0·2480 (1)	0·0611 (6)
C(12)	−0·5799 (3)	0·8914 (4)	−0·1833 (1)	0·0682 (6)
C(13)	−0·4423 (3)	0·8058 (4)	−0·1427 (1)	0·0650 (6)

* The standard deviations of the U_{eq}'s were calculated according to Schomaker & Marsh (1983).

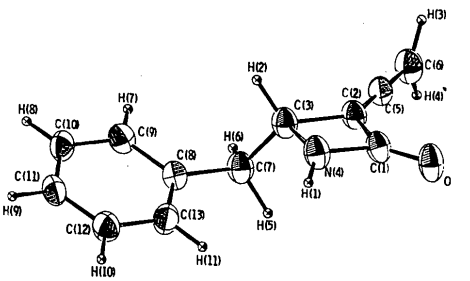


Fig. 1. *ORTEP* drawing (Johnson, 1965) of the molecule. Thermal ellipsoids scaled to enclose 30% probability. Hydrogen atoms are represented as spheres of arbitrary radii.

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Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

O–C(1)	1·215 (3)	C(7)–C(8)	1·513 (3)
C(1)–C(2)	1·497 (3)	C(8)–C(9)	1·382 (3)
C(1)–N(4)	1·361 (3)	C(8)–C(13)	1·374 (3)
C(2)–C(3)	1·535 (3)	C(9)–C(10)	1·377 (3)
C(2)–C(5)	1·286 (3)	C(10)–C(11)	1·366 (3)
C(3)–N(4)	1·494 (3)	C(11)–C(12)	1·367 (3)
C(3)–C(7)	1·502 (3)	C(12)–C(13)	1·379 (3)
C(5)–C(6)	1·301 (3)		

O–C(1)–C(2)	136·3 (2)	C(2)–C(5)–C(6)	178·3 (2)
O–C(1)–N(4)	132·4 (2)	C(3)–C(7)–C(8)	112·7 (2)
C(2)–C(1)–N(4)	91·3 (2)	C(7)–C(8)–C(9)	121·5 (2)
C(1)–C(2)–C(3)	88·3 (2)	C(7)–C(8)–C(13)	120·8 (2)
C(1)–C(2)–C(5)	135·5 (2)	C(9)–C(8)–C(13)	117·7 (2)
C(3)–C(2)–C(5)	136·2 (2)	C(8)–C(9)–C(10)	121·3 (2)
C(2)–C(3)–N(4)	85·0 (2)	C(9)–C(10)–C(11)	120·0 (2)
C(2)–C(3)–C(7)	116·7 (2)	C(10)–C(11)–C(12)	119·5 (2)
N(4)–C(3)–C(7)	115·3 (2)	C(11)–C(12)–C(13)	120·3 (2)
C(1)–N(4)–C(3)	95·3 (2)	C(8)–C(13)–C(12)	121·2 (2)

C(2)–C(3)–C(7)–C(8)	176·6 (2)	C(3)–C(7)–C(8)–C(9)	96·3 (2)
C(3)–C(7)–C(8)–C(13)	−82·8 (2)	N(4)–C(3)–C(7)–C(8)	79·1 (2)

are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1. Bond lengths, selected bond angles and torsion angles with their standard deviations are given in Table 2. The packing of the molecules in the unit cell is shown in the stereoscopic drawing in Fig. 2.

Related literature. The title compound was prepared (Buynak, Mathew, Rao, Haley, George & Siriwardane, 1987) by sequential treatment of the α-(α′-trimethylsilyl)ethylidene-β-lactam (Buynak, Rao, Chandrasekaran & Haley, 1985) with hypochlorous acid, potassium fluoride and ceric ammonium nitrate. The related structural studies include 2-azetidinones (De Meester, Buynak & Chu, 1986), allenedicarboxylic acid (Berkovitch-Yellin, Leiserowitz & Nader, 1977) and diphenylvinylidene cyclobutane (Berkovitch-Yellin, Lahav & Leiserowitz, 1974).

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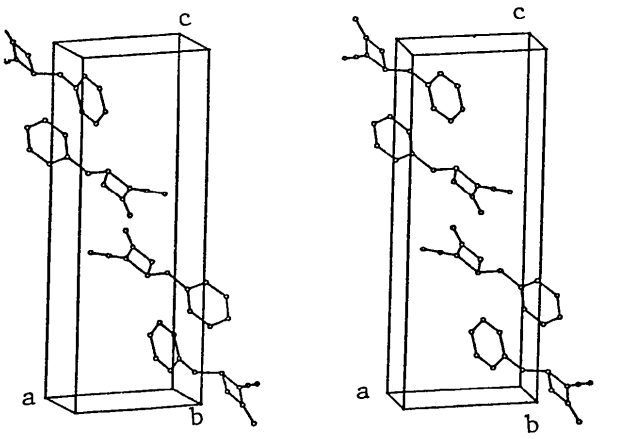

Fig. 2. Stereoscopic drawing of the molecular packing in the unit cell.

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Structure of *o*-Anisic Acid

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Abstract. *o*-Methoxybenzoic acid. $C_8H_8O_3$, M_r = 152·15, monoclinic, $P2_1/c$, a = 7·719 (2), b = 14·911 (3), c = 6·994 (2) Å, β = 115·52 (2)°, V = 726·5 (7) Å^3, Z = 4, D_x = 1·39 Mg m^{-3}, Mo $K\alpha$ (graphite monochromator), λ = 0·71073 Å, μ = 0·10 mm^{-1}, $F(000)$ = 320, T = 293 (1) K, R = 0·039

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