Crystal structure of 2-(*N*-acetyl)-5-phenylpent-4-ynamide, C₁₃H₁₄N₂O₂

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Table 1. Parameters used for the X-ray data collection

Crystal:	colorless plate, size 0.13 x 0.24 x 0.40 mm
Wavelength:	Mo K α radiation (0.7107 Å)
μ :	0.84 cm ⁻¹
Diffractometer:	Rigaku AFC6R
Scan mode:	$\omega/2\theta$
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{unique}}$:	2542
Criterion for I_o :	$I_o > 3 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	307
Program:	TEXSAN

Source of material: Prepared from the palladium catalysed coupling of 2-(*N*-acetyl)pent-4-ynamide with iodobenzene in piperidine (see ref. 1). Recrystallised from an acetonitrile solution of the compound, mp 445 K – 446 K.

Two independent molecules comprise the asymmetric unit, labelled a and b, with only minor conformational differences between them; identical numbering schemes were employed. Significant intermolecular H-bonding is evident, involving all of the acidic H atoms. Thus, N1a–H1a...O22a [$d(\text{A} \cdots \text{H})$ 1.05 Å, $d(\text{H} \cdots \text{B})$ 1.86 Å, $d(\text{A} \cdots \text{B})$ 2.894(4) Å and $\text{A} \cdots \text{H} \cdots \text{B}$ 169°], N1a–H1b...O1b [0.89 Å, 2.11 Å, 2.961(4) Å and 160°] and N21a–H21a...O1b [0.94 Å, 2.07 Å, 2.946(4) Å and 155°] are found for molecule a and N1b–H1c...O22b [0.87 Å, 2.06 Å, 2.879(4) Å and 156°], N1b–H1d...O1a [1.05 Å, 1.95 Å, 2.965(4) Å and 160°] and N21b–H21b...O1a [1.03 Å, 1.88 Å, 2.907(4) Å and 170°] are found for molecule b. These combine to give rise to a complex 3-D network.

C₁₃H₁₄N₂O₂, triclinic, $P\bar{1}$ (No. 2), $a = 13.192(6)$ Å, $b = 13.849(5)$ Å, $c = 6.955(3)$ Å, $\alpha = 92.23(3)^\circ$, $\beta = 92.37(5)^\circ$, $\gamma = 80.39(4)^\circ$, $V = 1251.1$ Å³, $Z = 4$, $R(F) = 0.062$, $R_w(F) = 0.069$.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1a)	2i	0.1541	−0.0504	0.2535	0.081
H(1b)	2i	0.2263	−0.1652	0.3130	0.059
H(1c)	2i	0.3273	−0.4586	0.7481	0.085
H(1d)	2i	0.2558	−0.3567	0.8273	0.088
H(2a)	2i	0.0617	−0.0678	−0.0308	0.051
H(2b)	2i	0.4164	−0.4663	0.4432	0.060
H(3a)	2i	0.1804	0.0415	−0.0286	0.116
H(3b)	2i	0.1429	0.0299	−0.2305	0.079
H(3c)	2i	0.2838	−0.5561	0.4287	0.110
H(3d)	2i	0.3151	−0.5406	0.1815	0.106
H(21a)	2i	0.1518	−0.1978	−0.3016	0.065
H(21b)	2i	0.3163	−0.3264	0.1641	0.053
H(23a)	2i	−0.0710	−0.2188	−0.5297	0.134
H(23b)	2i	−0.0255	−0.1651	−0.6115	0.133
H(23c)	2i	0.0521	−0.2389	−0.5555	0.105
H(23d)	2i	0.5279	−0.2766	0.0881	0.272
H(23e)	2i	0.5235	−0.3248	−0.0391	0.165
H(23f)	2i	0.4212	−0.2726	−0.0521	0.123
H(52a)	2i	0.4307	−0.2148	−0.4908	0.075
H(52b)	2i	0.0318	−0.3117	−0.0712	0.101
H(53a)	2i	0.5975	−0.2820	−0.5648	0.166
H(53b)	2i	−0.1211	−0.2336	−0.1685	0.178
H(54a)	2i	0.7335	−0.1890	−0.5097	0.116
H(54b)	2i	−0.2708	−0.2542	0.0265	0.063
H(55a)	2i	0.6970	−0.0372	−0.2914	0.106
H(55b)	2i	−0.2537	−0.3742	0.2346	0.104
H(56a)	2i	0.5329	0.0106	−0.2066	0.119
H(56b)	2i	−0.0857	−0.4618	0.3099	0.082

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(1a)	2i	0.2247(2)	−0.2435(2)	0.0035(4)	0.052(2)	0.051(2)	0.054(2)	0.019(1)	−0.002(1)	0.000(1)
O(1b)	2i	0.2499(2)	−0.2807(2)	0.5071(4)	0.058(2)	0.050(2)	0.053(2)	0.022(1)	−0.005(1)	0.002(1)
O(22a)	2i	−0.0602(2)	−0.0629(2)	−0.2992(4)	0.046(2)	0.072(2)	0.085(2)	0.024(2)	−0.004(2)	−0.010(2)
O(22b)	2i	0.5429(2)	−0.4354(2)	0.2437(5)	0.040(2)	0.085(2)	0.108(3)	0.020(2)	−0.001(2)	0.029(2)
N(1a)	2i	0.1945(3)	−0.1217(2)	0.2301(5)	0.067(2)	0.057(2)	0.048(2)	0.015(2)	−0.005(2)	−0.003(2)

Table 3. (Continued)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
N(1b)	2i	0.2903(3)	−0.4030(3)	0.7158(5)	0.072(3)	0.062(2)	0.041(2)	0.018(2)	0.000(2)	0.005(2)
N(21a)	2i	0.1015(2)	−0.1450(2)	−0.2646(5)	0.042(2)	0.055(2)	0.045(2)	0.020(2)	−0.004(2)	−0.005(2)
N(21b)	2i	0.3732(2)	−0.3757(2)	0.2304(4)	0.035(2)	0.055(2)	0.048(2)	0.018(2)	0.001(2)	0.007(2)
C(1a)	2i	0.1853(3)	−0.1592(3)	0.0507(6)	0.036(2)	0.048(3)	0.044(2)	0.008(2)	0.002(2)	0.001(2)
C(1b)	2i	0.2923(3)	−0.3641(3)	0.5425(6)	0.031(2)	0.049(3)	0.052(3)	0.005(2)	−0.007(2)	−0.002(2)
C(2a)	2i	0.1247(3)	−0.0906(3)	−0.0922(6)	0.041(2)	0.046(3)	0.056(3)	0.019(2)	0.000(2)	0.000(2)
C(2b)	2i	0.3481(3)	−0.4325(3)	0.3873(5)	0.037(2)	0.053(3)	0.040(2)	0.014(2)	−0.002(2)	−0.002(2)
C(3a)	2i	0.1817(4)	−0.0061(3)	−0.1320(7)	0.088(4)	0.046(3)	0.074(3)	0.008(3)	−0.003(3)	0.007(2)
C(3b)	2i	0.2826(3)	−0.5105(3)	0.3198(6)	0.065(3)	0.051(3)	0.069(3)	0.004(2)	−0.001(3)	−0.010(2)
C(4a)	2i	0.2856(4)	−0.0386(4)	−0.2008(7)	0.084(4)	0.069(3)	0.060(3)	−0.015(3)	0.003(3)	0.004(3)
C(4b)	2i	0.1783(4)	−0.4693(3)	0.2545(7)	0.063(3)	0.061(3)	0.079(3)	−0.014(3)	0.003(3)	−0.014(3)
C(5a)	2i	0.3705(4)	−0.0673(4)	−0.2507(7)	0.083(4)	0.081(4)	0.063(3)	−0.024(3)	−0.001(3)	0.012(3)
C(5b)	2i	0.0923(4)	−0.4334(3)	0.2035(7)	0.059(3)	0.066(3)	0.076(3)	−0.010(3)	0.002(3)	−0.015(3)
C(22a)	2i	0.0089(3)	−0.1279(3)	−0.3541(6)	0.041(3)	0.056(3)	0.055(3)	0.011(2)	−0.002(2)	0.007(2)
C(22b)	2i	0.4688(3)	−0.3798(3)	0.1725(6)	0.042(3)	0.056(3)	0.058(3)	0.011(2)	−0.003(2)	0.009(2)
C(23a)	2i	−0.0065(3)	−0.1954(4)	−0.5230(7)	0.052(3)	0.095(4)	0.073(3)	0.010(3)	−0.015(2)	−0.020(3)
C(23b)	2i	0.4827(3)	−0.3133(4)	0.0153(8)	0.049(3)	0.103(4)	0.096(4)	0.008(3)	0.007(3)	0.036(3)
C(51a)	2i	0.4731(4)	−0.1019(4)	−0.3147(7)	0.067(3)	0.085(4)	0.055(3)	−0.025(3)	−0.003(3)	0.014(3)
C(51b)	2i	−0.0100(3)	−0.3888(3)	0.1465(7)	0.053(3)	0.058(3)	0.069(3)	−0.014(2)	0.002(3)	−0.012(3)
C(52a)	2i	0.4941(4)	−0.1856(4)	−0.4300(9)	0.061(4)	0.094(4)	0.115(5)	−0.020(3)	−0.009(3)	−0.009(4)
C(52b)	2i	−0.0238(4)	−0.3206(4)	0.0054(7)	0.058(3)	0.082(4)	0.081(4)	−0.018(3)	0.009(3)	−0.004(3)
C(53a)	2i	0.5915(5)	−0.2185(5)	−0.4980(9)	0.068(4)	0.114(5)	0.126(5)	−0.017(4)	−0.005(4)	−0.013(4)
C(53b)	2i	−0.1216(4)	−0.2734(4)	−0.0488(7)	0.058(3)	0.087(4)	0.084(4)	−0.012(3)	0.001(3)	0.006(3)
C(54a)	2i	0.6692(4)	−0.1683(6)	−0.446(1)	0.061(4)	0.159(7)	0.113(5)	−0.021(4)	−0.006(4)	0.009(5)
C(54b)	2i	−0.2047(4)	−0.2958(4)	0.0355(9)	0.056(3)	0.078(4)	0.107(4)	−0.003(3)	0.006(3)	0.001(3)
C(55a)	2i	0.6510(5)	−0.0854(6)	−0.3265(9)	0.080(5)	0.169(7)	0.084(5)	−0.066(5)	−0.013(4)	0.012(4)
C(55b)	2i	−0.1927(4)	−0.3637(4)	0.1761(9)	0.053(3)	0.098(4)	0.109(5)	−0.021(3)	0.016(3)	−0.007(4)
C(56a)	2i	0.5540(5)	−0.0518(5)	−0.2635(8)	0.102(5)	0.126(5)	0.071(4)	−0.055(4)	−0.005(4)	0.005(3)
C(56b)	2i	−0.0959(4)	−0.4106(4)	0.2332(7)	0.074(4)	0.077(4)	0.065(3)	−0.010(3)	0.005(3)	0.000(3)

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References

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