Crystal structure of [3,5-dimethyl-4-(prop-2-ynyl)-1*H*-pyrazol-1-yl](2-furyl)methanone, $C_{13}H_{12}N_2O_2$

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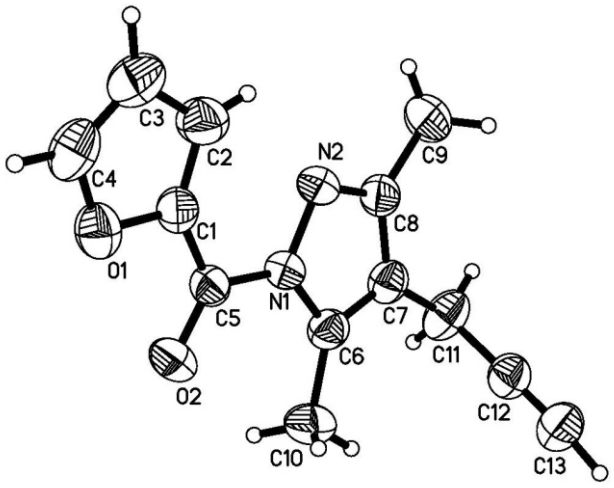
Received May 19, 2011, accepted and available on-line September 12, 2011; CCDC no. 1267/3581



Abstract

$C_{13}H_{12}N_2O_2$, monoclinic, $P12_1/c1$ (no. 14), a = 7.928(2) Å, b = 16.358(3) Å, c = 9.506(2) Å, β = 106.44(3)°, V = 1182.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.064, $wR_{ref}(F^2)$ = 0.209, T = 293 K.

Source of material

The title compound was obtained in the yield of 90 % by refluxing of 3-(prop-2-ynyl)pentane-2,4-dione with furan-2-carbohydrazide in equimolar quantity in ethanol in the presence of 5 molar % of *p*-toluenesulphonic acid (PTSA) as catalyst. Crystallization of crude substance from solution in ethanol at 350 K followed by cooling to 293 K yielded the colorless single crystals of the title compound suitable for X-ray diffraction analysis.
Elemental analysis — found: C, 68.53 %; H, 5.36 %; N, 12.40 %; calculated for $C_{13}H_{12}N_2O_2$: C, 68.41 %; H, 5.30 %; N, 12.27 %.

Experimental details

The hydrogen atoms were located from a difference Fourier map and refined isotropically, only H atoms of the $C9H_3$ methyl group were constrained to ideal geometries, with d(C—H) = 0.96 Å, and with U_{iso}(H) = 1.5 U_{eq} (C9).

Discussion

Derivatives of pyrazoles represent one of the most active classes of compounds possessing a wide spectrum of biological activities, such as antibacterial [1,2], antimicrobial [3-6], antiviral [7], antioxidant [8], anti-inflammatory [9,10], anticonvulsant [11,12], anticancer [13], anesthetic and anti-arrhythmic activities [14]. The formation of the title compound is the result of intramolecular ring closure of intermediate *N*′-(3-acetylhex-5-yn-2-ylidene)furan-2-carbohydrazide obtained as the main product in the absence of PTSA. But the *N*′-(3-acetylhex-5-yn-2-ylidene)furan-2-carbohydrazide may be also isomerized to *N*′-(3-acetylhex-2-en-5-yn-2-yl)furan-2-carbohydrazide.
In the title crystal structure, atoms of the pyrazole and furane cycles are coplanar within 0.002 Å, and the dihedral angle between the least-squares planes of both rings is equal to 9.2(1)°. In the crystal structure, molecules are associated *via* C–H···O hydrogen bonds (d(D—H) = 0.98(3) Å, d(H···A) = 2.49(3) Å, d(D···A) = 3.37(3) Å, ∠D–H···A = 150(2)°) forming an infinite chain along [010].

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.2 × 0.25 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.88 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CFD-4, $\omega/2\theta$
$2\theta_{max}$:	115.32°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9812, 4861
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1839
$N(param)_{refined}$:	190
Programs:	SHELXS-97, SHELXL-97 [15], ORTEP-3 [16], WinGX [17]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(91)	4*e*	0.2419	0.2085	–0.1106	0.100
H(92)	4*e*	0.3269	0.2201	–0.2394	0.100
H(93)	4*e*	0.3593	0.2853	–0.1137	0.100
H(2)	4*e*	0.318(3)	0.061(2)	0.188(3)	0.084(8)
H(3)	4*e*	0.191(3)	–0.034(1)	0.345(3)	0.069(7)
H(4)	4*e*	0.461(4)	–0.130(2)	0.449(3)	0.092(9)
H(101)	4*e*	1.009(5)	0.123(2)	0.174(4)	0.12(1)
H(102)	4*e*	0.994(4)	0.062(2)	0.046(3)	0.10(1)
H(103)	4*e*	1.024(5)	0.155(2)	0.023(3)	0.12(1)
H(111)	4*e*	0.659(4)	0.275(2)	–0.220(3)	0.081(8)
H(112)	4*e*	0.811(4)	0.218(2)	–0.206(3)	0.093(9)
H(13)	4*e*	1.071(4)	0.391(2)	0.068(3)	0.089(8)

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*	0.6553(2)	0.09238(8)	0.0968(2)	0.0349(7)	0.0395(7)	0.0441(7)	0.0025(5)	0.0076(5)	0.0013(6)
N(2)	4*e*	0.4871(2)	0.12331(9)	0.0408(2)	0.0357(7)	0.0447(8)	0.0496(8)	0.0047(6)	0.0056(6)	0.0043(6)
O(1)	4*e*	0.5804(2)	–0.06699(7)	0.3437(1)	0.0667(9)	0.0477(8)	0.0535(7)	0.0040(6)	0.0146(7)	0.0085(6)
O(2)	4*e*	0.8287(2)	–0.00804(8)	0.2303(2)	0.0436(7)	0.0539(8)	0.0796(9)	0.0129(6)	0.0111(6)	0.0151(7)
C(1)	4*e*	0.5400(2)	–0.0011(1)	0.2511(2)	0.047(1)	0.0382(8)	0.0423(8)	0.0002(7)	0.0088(7)	0.0001(7)
C(2)	4*e*	0.3726(3)	0.0218(1)	0.2386(2)	0.049(1)	0.062(1)	0.065(1)	0.0014(9)	0.0184(9)	0.010(1)
C(3)	4*e*	0.3045(3)	–0.0320(2)	0.3257(2)	0.061(1)	0.071(1)	0.067(1)	–0.013(1)	0.024(1)	0.004(1)
C(4)	4*e*	0.4339(3)	–0.0835(1)	0.3863(2)	0.078(2)	0.057(1)	0.054(1)	–0.017(1)	0.024(1)	0.0026(9)
C(5)	4*e*	0.6849(2)	0.0253(1)	0.1950(2)	0.0389(9)	0.0367(8)	0.0446(8)	0.0023(6)	0.0050(7)	–0.0013(6)
C(6)	4*e*	0.7755(2)	0.1325(1)	0.0402(2)	0.0407(9)	0.0434(9)	0.0448(8)	–0.0028(7)	0.0110(7)	–0.0054(7)
C(7)	4*e*	0.6804(2)	0.1898(1)	–0.0547(2)	0.049(1)	0.0418(9)	0.0401(8)	–0.0053(7)	0.0098(7)	–0.0029(7)
C(8)	4*e*	0.5038(2)	0.1816(1)	–0.0506(2)	0.0442(9)	0.0403(9)	0.0456(9)	0.0008(7)	0.0040(7)	0.0001(7)
C(9)	4*e*	0.3434(3)	0.2281(1)	–0.1364(2)	0.057(1)	0.057(1)	0.075(1)	0.012(1)	0.001(1)	0.016(1)
C(10)	4*e*	0.9680(3)	0.1152(2)	0.0789(3)	0.040(1)	0.069(2)	0.077(2)	0.003(1)	0.019(1)	0.003(1)
C(11)	4*e*	0.7516(3)	0.2483(1)	–0.1467(2)	0.069(1)	0.059(1)	0.048(1)	–0.020(1)	0.014(1)	0.0019(9)
C(12)	4*e*	0.8774(3)	0.3082(1)	–0.0605(2)	0.054(1)	0.048(1)	0.060(1)	–0.0025(8)	0.0191(9)	0.0026(8)
C(13)	4*e*	0.9794(3)	0.3553(1)	0.0081(3)	0.061(1)	0.058(1)	0.079(2)	–0.009(1)	0.019(1)	–0.011(1)

References

1. Akbas, E.; Berber, I.; Sener, A.; Hasanov, B.: Synthesis and antibacterial activity of 4-benzoyl-1-methyl-5-phenyl-1*H*-pyrazole-3-carboxylic acid and derivatives. Il Farmaco **60** (2005) 23-26.
2. Sing, A.; Rana, A. C.: Synthesis, antibacterial activity, insilico metabolism and toxicity prediction of the pyrazoline derivatives. J. Glob. Pharm. Techn. **2** (2010) 25-32.
3. Prakash, O.; Hussain, K.; Aneja, D. K.: Synthesis and antimicrobial activity of some new 2,6-bis((1-phenyl-3-aryl-1*H*-pyrazol-4-yl)methylene)cyclohexanones. Der Pharma Chemica **3** (2011) 221-226.
4. Chovatia, P. T.; Akabari, J. D.; Kachhadia, P. K.; Zalavadia, P. D.; Joshi, H. S.: Synthesis and selective antitubercular and antimicrobial inhibitory activity of 1-acetyl-3,5-diphenyl-4,5-dihydro-(1*H*)-pyrazole derivatives. J. Serb. Chem. Soc. **71** (2007) 713-720.
5. Raju, H.; Nagamani, T. S.; Chandrappa, S.; Ananda, H., Vinaya, K.; Thimmegowda N. R.; Byregowda S. M.; Rangappa, K. S.: Synthesis of 1-(4-methoxybenzyl)-3-cyclopropyl-1*H*-pyrazol-5-amine derivatives as antimicrobial agents. J. Enz. Inhib. & Med. Chem. **25** (2010) 537-543.
6. Abdel-Wahab, B. F.; Abdel-Aziz, H. A.; Ahmed, E. M.: Synthesis and antimicrobial evaluation of 1-(benzofuran-2-yl)-4-nitro-3-arylbutan-1-ones and 3-(benzofuran-2-yl)-4,5-dihydro-5-aryl-1-[4-(aryl)-1,3-thiazol-2-yl]-1*H*-pyrazoles. Eur. J. Med. Chem. **44** (2009) 2632-2635.
7. Hashem, A.; Ahmed, S.; Youssef, A.; Kamal, A. K.; Wael, S.; Elmagd, A.: Convertion of some 2(3*H*)-furanones bearing a pyrazolyl group into other heterocyclic systems with a study of their antiviral activity. Eur. J. Med. Chem. **42** (2007) 934-939.
8. Gressler, V.; Moura, S.; Flores, A. F. C.; Flores, D. C.; Colepicolo, P.; Pinto, E.: Antioxidant and antimicrobial properties of 2-(4,5-dihydro-1*H*-pyrazol-1-yl)-pyrimidine and 1-carboxamidino-1*H*-pyrazole derivatives. J. Braz. Chem. Soc. **21** (2010) 1-6.
9. Arunkumar, S.; Ilango, K.; Manikandan, R. S.; Ramalakshmi, N.: Synthesis and anti-inflammatory activity of some novel pyrazole derivatives of gallic acid. E-J. Chem. **6** (2009) S123-S128.
10. Bhaskar, V. H.; Mohite, P. B.: Synthesis analgesic, anti-inflammatory and antimicrobial activities of some 1-[5-(substituted phenyl)-4,5-dihydro-1*H*-pyrazol-3-yl]-5-phenyl-1*H*-tetrazole. J. Optoelectr. & Biomed. Mat. **2** (2010) 291-299.
11. Singh, A.; Rana A. C.: Synthesis and anticonvulsant activity of 1-[(4,5-dihydro-5-phenyl-3-(phenylamino)pyrazol-1-yl)]ethanone derivatives. J Chem. Pharm. Res. **2** (2010) 505-511.
12. Kaushik, D.; Khan, S. A.; Chawla, G.; Kumar, S.: *N*′-[(5-chloro-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)methylene] 2/4-substituted hydrazides: synthesis and anticonvulsant activity. Eur. J. Med. Chem. **45** (2010) 3943-3949.
13. Cai Z.-Y.; Yang, Y.; Liu X.-H.; Qi X.-B.: Novel 3-(1-acetyl-5-(substituted-phenyl)-4,5-dihydro-1*H*-pyrazol-3-yl)-7-fluoro-2*H*-chromen-2-one derivatives: synthesis and anticancer activity. Lett. Drug Des. & Disc. **7** (2010) 640-643.
14. Zalaru, C.; Dumitrascu, F.; Draghici, C.; Cristea, E.; Tarcomnicu, I.: Pharmacologically active 2-(1*H*-pyrazol-1-yl)acetamides. ARKIVOC (2009) 308-314.
15. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
16. Farrugia, L. J.: ORTEP-3 for Windows - a version of ORTEP-III with a Graphical User Interface (GUI) J. Appl. Crystallogr. **30** (1997) 565.
17. Farrugia, L. J.: WinGX suite for small-molecule single-crystal crystallography. J. Appl. Crystallogr. **32** (1999) 837-838.