Crystal structure of 2-acetyl-2-(4-methylbenzenesulfonamido)-*N*-phenyl-pent-4-ynamide, $C_{20}H_{20}N_2O_4S$

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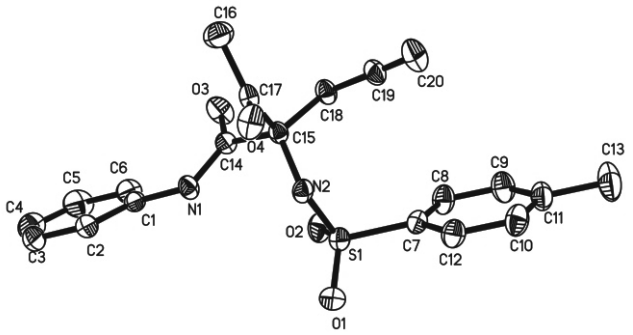

Abstract

$C_{20}H_{20}N_2O_4S$, monoclinic, $P12_1/c1$ (no. 14), a = 10.703(5) Å, b = 7.757(5) Å, c = 23.238(5) Å, β = 91.298(5)°, V = 1928.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.048, $wR_{ref}(F^2)$ = 0.140, T = 293 K.

Source of material

To a solution of 2-acetyl-*N*-phenylpent-4-ynamide (215 mg, 1.0 mmol) and 4-methylbenzenesulfonamide (0.342 mg, 2.0 mmol) in CH_2Cl_2 (3.0 mL), $FeCl_2$ (13 mg, 0.1 mmol) was added. The reaction mixture was stirred at room temperature for 24 hours. When the starting material was consumed (monitored by TLC), the reaction mixture was quenched with 1M NH_4Cl (10 mL). The organic and aqueous layers were separated, and the aqueous layer was extracted with dichloromethane (3 times 10 mL). The combined organic layers were dried over $MgSO_4$ and filtered. The filtrate was concentrated *in vacuo*, and then the residue was purified by silica gel column chromatography (petroleum ether : diethyl ether = 10 : 3), to afford the product 2-acetyl-2-(4-methylbenzenesulfonamido)-*N*-phenylpent-4-ynamide (251 mg, 65 % yield). It was redissolved in a mixture of diethyl ether with ethyl acetate (10 mL, v/v = 3 : 1). This solution was sealed and kept at room temperature for three days. Colorless crystals of the title compound were separated and analyzed.

Experimental details

All H atoms on C atoms were generated geometrically and refined as riding atoms with d(C—H) = 0.96 Å (CH_3), 0.97 Å (CH_2) or 0.93 Å (CH), and U_{iso}(H) = 1.2 or 1.5 U_{eq}(C). The H atoms bonded to nitrogen atoms were located from difference Fourier map and refined with U_{iso}(H) = 1.5 U_{eq}(N).

Discussion

It is well-known that the carbon-nitrogen linkage can be formed directly from two simple carbon-hydrogen bonds (C—H) through metal-catalyzed reactions [1]. Compared with precious metal catalysts such as Pt, Rh, Ru, Pd, Au, Ag, etc., iron is cheaper [2]. Therefore, the study of iron-catalyzed reactions involved in the carbon-nitrogen linkage has received great attention. In this work, under $FeCl_2$ catalyst condition, the reaction of 2-acetyl-*N*-phenylpent-4-ynamide and 4-methylbenzenesulfonamide gave the title compound 2-acetyl-2-(4-methylbenzenesulfonamido)-*N*-phenyl-pent-4-ynamide.

The title compound crystallizes with one molecule in the asymmetric unit. Geometric parameters are in the usual ranges. d(C14—O3) = 1.213(2) Å and d(C17—O4) = 1.195(2) Å are consistent with the values of the C=O double bond in carbonyl compounds. The bond length of 1.452(2) Å indicates the formation of C15—N2 single bond. There are two benzene rings in the structure, and the dihedral angles between them are 5.7°. The adjacent molecules are linked into a chain along b axis by intermolecular N–H···O and C–H···O hydrogen bonding interactions.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.13 × 0.18 × 0.21 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.96 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{max}$:	58.42°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12905, 4556
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2979
$N(param)_{refined}$:	250
Programs:	SHELXS-97, SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.1098	0.0330	0.6473	0.068
H(3)	4*e*	–0.0773	–0.0845	0.7893	0.098
H(4)	4*e*	0.0850	0.0232	0.8455	0.079
H(5)	4*e*	0.2588	0.1385	0.8032	0.059
H(6)	4*e*	–0.0648	–0.0790	0.6907	0.092
H(8)	4*e*	0.6055	0.0660	0.5077	0.067
H(9)	4*e*	0.7578	0.1370	0.4432	0.076
H(10)	4*e*	0.9889	0.2666	0.5731	0.071
H(12)	4*e*	0.8385	0.1964	0.6379	0.061
H(13A)	4*e*	0.9579	0.2388	0.4205	0.139
H(13B)	4*e*	1.0615	0.1868	0.4660	0.139
H(13C)	4*e*	1.0110	0.3764	0.4640	0.139
H(16A)	4*e*	0.3556	0.7394	0.7000	0.097
H(16B)	4*e*	0.2628	0.5990	0.6760	0.097
H(16C)	4*e*	0.3503	0.6975	0.6340	0.097
H(18A)	4*e*	0.4582	0.3190	0.5522	0.053
H(18B)	4*e*	0.3795	0.4835	0.5665	0.053
H(20)	4*e*	0.7212	0.6856	0.5631	0.086
H(1A)	4*e*	0.372(2)	0.170(3)	0.7179(9)	0.063
H(2A)	4*e*	0.588(2)	0.292(3)	0.6859(8)	0.058

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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}
C(1)	4*e*	0.2007(2)	0.0993(2)	0.72107(8)	0.033(1)	0.035(1)	0.056(1)	–0.0004(8)	0.0069(8)	0.0069(8)
C(2)	4*e*	0.1046(2)	0.0325(3)	0.6872(1)	0.048(1)	0.049(1)	0.074(1)	–0.010(1)	0.001(1)	0.002(1)
C(3)	4*e*	–0.0070(3)	–0.0387(3)	0.7721(2)	0.056(2)	0.054(2)	0.137(3)	–0.010(1)	0.037(2)	0.021(2)
C(4)	4*e*	0.0898(2)	0.0261(3)	0.8056(1)	0.068(2)	0.052(1)	0.079(2)	0.004(1)	0.031(1)	0.018(1)
C(5)	4*e*	0.1936(2)	0.0950(3)	0.78039(9)	0.048(1)	0.047(1)	0.054(1)	0.0031(9)	0.0105(9)	0.0085(9)
C(6)	4*e*	0.0007(2)	–0.0353(3)	0.7133(1)	0.052(2)	0.057(2)	0.120(2)	–0.025(1)	0.002(2)	0.013(1)
C(7)	4*e*	0.7057(2)	0.1253(2)	0.57912(8)	0.035(1)	0.044(1)	0.045(1)	0.0016(8)	0.0050(8)	–0.0034(8)
C(8)	4*e*	0.6826(2)	0.1069(3)	0.52104(9)	0.043(1)	0.076(2)	0.049(1)	–0.008(1)	0.0022(9)	–0.009(1)
C(9)	4*e*	0.7742(2)	0.1494(3)	0.48249(9)	0.067(2)	0.081(2)	0.044(1)	–0.009(1)	0.011(1)	–0.006(1)
C(10)	4*e*	0.9117(2)	0.2265(3)	0.5598(1)	0.041(1)	0.066(1)	0.070(1)	–0.010(1)	0.008(1)	–0.012(1)
C(11)	4*e*	0.8896(2)	0.2098(3)	0.5010(1)	0.057(1)	0.056(1)	0.063(1)	–0.005(1)	0.023(1)	–0.004(1)
C(12)	4*e*	0.8219(2)	0.1849(3)	0.59859(9)	0.042(1)	0.060(1)	0.050(1)	–0.004(1)	0.0008(9)	–0.0067(9)
C(13)	4*e*	0.9890(3)	0.2573(4)	0.4591(1)	0.086(2)	0.100(2)	0.095(2)	–0.021(2)	0.048(2)	–0.008(2)
C(14)	4*e*	0.3144(2)	0.2650(2)	0.64813(7)	0.0323(9)	0.045(1)	0.0372(9)	–0.0030(8)	–0.0033(8)	0.0024(7)
C(15)	4*e*	0.4424(2)	0.3562(2)	0.64009(6)	0.0299(9)	0.045(1)	0.0304(8)	–0.0022(8)	–0.0003(7)	0.0050(7)
C(16)	4*e*	0.3439(2)	0.6497(3)	0.6719(1)	0.065(2)	0.052(1)	0.076(2)	0.008(1)	0.003(1)	–0.001(1)
C(17)	4*e*	0.4417(2)	0.5153(3)	0.68075(7)	0.037(1)	0.049(1)	0.0340(9)	–0.0092(8)	0.0081(8)	0.0012(8)
C(18)	4*e*	0.4535(2)	0.4182(3)	0.57749(7)	0.044(1)	0.059(1)	0.0302(9)	–0.0039(9)	0.0005(8)	0.0062(8)
C(19)	4*e*	0.5639(2)	0.5257(3)	0.57031(8)	0.054(1)	0.059(1)	0.0345(9)	0.000(1)	0.0080(9)	0.0097(9)
C(20)	4*e*	0.6516(3)	0.6148(4)	0.5663(1)	0.068(2)	0.080(2)	0.068(2)	–0.018(1)	0.015(1)	0.013(1)
N(1)	4*e*	0.3082(2)	0.1763(2)	0.69723(6)	0.0283(8)	0.057(1)	0.0406(8)	–0.0067(7)	–0.0007(6)	0.0101(7)
N(2)	4*e*	0.5457(1)	0.2489(2)	0.65999(6)	0.0297(8)	0.051(1)	0.0349(7)	–0.0004(7)	–0.0008(6)	0.0001(6)
O(1)	4*e*	0.6546(2)	–0.0248(2)	0.67373(6)	0.064(1)	0.066(1)	0.0674(9)	0.0169(8)	0.0115(8)	0.0227(8)
O(2)	4*e*	0.4857(1)	0.0028(2)	0.59932(6)	0.0459(9)	0.0598(9)	0.0704(9)	–0.0165(7)	0.0098(7)	–0.0109(7)
O(3)	4*e*	0.2287(1)	0.2875(2)	0.61393(6)	0.0409(8)	0.087(1)	0.0575(9)	–0.0157(8)	–0.0181(7)	0.0229(8)
O(4)	4*e*	0.5197(1)	0.5259(2)	0.71820(6)	0.0477(9)	0.080(1)	0.0472(8)	–0.0060(8)	–0.0026(7)	–0.0198(7)
S(1)	4*e*	0.59217(5)	0.07169(6)	0.62972(2)	0.0388(3)	0.0463(3)	0.0495(3)	–0.0003(2)	0.0075(2)	0.0032(2)

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