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N-[3-(Prop-1-yn-1-yl)phenyl]benzenesulfonamide

Leslie W. Pineda[a,b] and Jorge A. Cabezas[a]*

[a]Escuela de Química, Universidad de Costa Rica, 11501-2060, San José, Costa Rica, and [b]Centro de Electroquímica y Energía Química (CELEQ), Universidad de Costa Rica, 11501-2060, San José, Costa Rica. *Correspondence e-mail: jorge.cabezas@ucr.ac.cr

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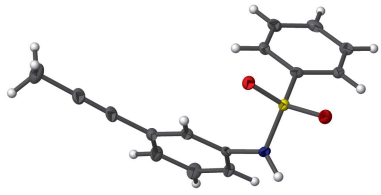
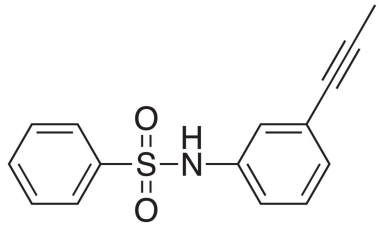
Structural data: full structural data are available from iucrdata.iucr.org

In the title sulfanilamide derivative, $C_{15}H_{13}NO_2S$, which shows significant activity against *Staphylococcus aureus* and *Escherichia coli*, the dihedral angle between the planes of the aromatic rings is 62.15 (19)° and the four-coordinate S atom adopts an almost ideal tetrahedral geometry. In the crystal, N—H···O and C—H···O hydrogen bonds link the molecules into a three-dimensional network.

3D view



Chemical scheme



Structure description

In 1932, a drug called Prontosil was discovered by the pharmaceutical division of IG Farbenindustrie, an industrial conglomerate of German companies, including Bayer Company. It was found to be very successful treating several diseases in humans, provoked by *Staphylococcus* and *Streptococcus*. Prontosil was the first antibacterial drug, with life-saving capability, to be used systematically for the treatment of bacterial infections in the body. It belongs to a family of compounds called sulfa drugs or sulfonamides. In the 1940s and 1950s, most of the sulfa drugs were replaced by penicillin and other drugs, which proved to be more effective against more types of bacteria. However, nowadays, some sulfa drugs such as sulfamethoxazole, in combination with trimethoprim (co-trimoxazole), are still used extensively to inhibit the growth of bacteria that produce opportunistic infections in patients with AIDS, and bacterial infections such as pneumonia, bronchitis and infections of the urinary tract, ears and intestines (Brumfitt & Hamilton-Miller, 1993).

As part of our studies in this area we now report the synthesis of of the title sulfanilamide derivative, **1**, and its crystal structure. This compound, has been found to be very effective against *Staphylococcus aureus* and *Escherichia coli*, and minimal inhibitory concentrations (MIC) of 12.5 μg ml^{-1} and 25.0 μg ml^{-1} have been obtained respectively (Cabezas & Arias, 2019).

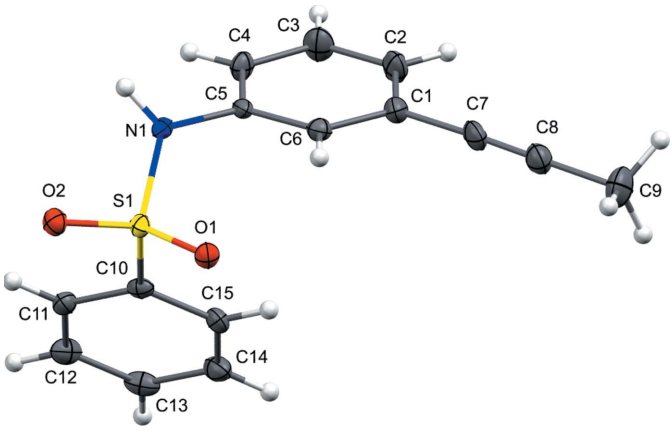


Figure 1
The title molecule with 50% probability ellipsoids.

The crystal structure of **1** has monoclinic symmetry with one molecule in the asymmetric unit: the molecular structure consists of a benzenesulfonamide fragment bound to a benzene ring bearing in its 3-position a propyne substituent (Fig. 1): the dihedral angle between the C1–C6 and C10–C15 benzene rings is 62.15 (19)°. The length of the carbon–carbon triple bond (C7≡C8) is 1.181 (5) Å, with the C7—C8—C9 and C8—C7—C1 angles being 178.8 (4) and 178.1 (4)°, respectively, which are slightly distorted from the expected linear geometry. The calculation of the angular structural index (τ_4 = 0.94; and τ_4' = 0.90) for the four-coordinate S1 atom, which binds to O1, O2, N1 and C10 from the benzene ring (Yang *et al.*, 2007; Okuniewski *et al.*, 2015; Rosiak *et al.*, 2018) indicates that it adopts an almost ideal tetrahedral geometry (τ_4 = 0 for an ideal square and 1 for an ideal tetrahedron). In the extended structure of **1**, weak N1—H1···O2, C4—H4···O1 and C6—H6···O1 hydrogen bonds are observed (Table 1, Fig. 2), leading to the formation of a three-dimensional network.

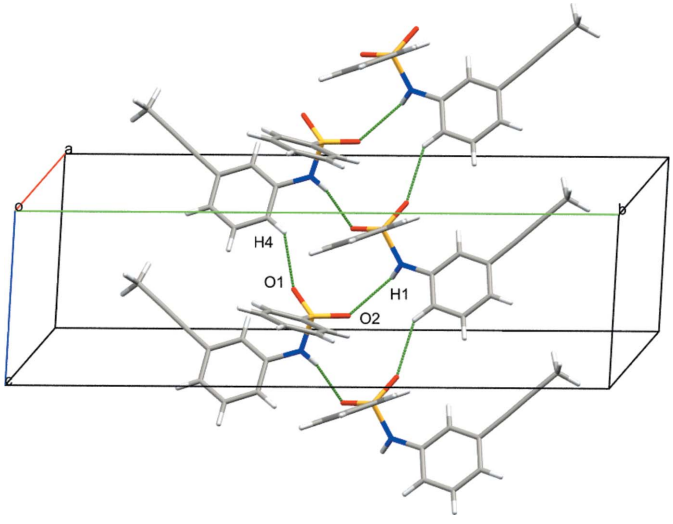


Figure 2
Part of an [001] hydrogen-bonded chain with N—H···O2, C—H···O1 hydrogen bonds shown as green lines.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.88	2.55	2.984 (4)	111
C4—H4···O1^{ii}	0.95	2.45	3.256 (4)	143
C6—H6···O1	0.95	2.39	2.955 (4)	118

Symmetry codes: (i) $x, -y+1, z+\frac{1}{2}$; (ii) $x, y, z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{13}NO_2S$
M_r	271.32
Crystal system, space group	Monoclinic, *Cc*
Temperature (K)	100
a, *b*, *c* (Å)	8.4596 (4), 24.9769 (13), 7.1310 (4)
β (°)	117.557 (2)
V (Å^3)	1335.80 (12)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.24
Crystal size (mm)	0.35 × 0.20 × 0.15
Data collection	
Diffractometer	Bruker D8 Venture CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.704, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9630, 3028, 2716
R_{int}	0.037
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.089, 1.03
No. of reflections	3028
No. of parameters	173
No. of restraints	2
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.39, −0.47
Absolute structure	Flack x determined using 1163 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter	0.02 (3)

Computer programs: *APEX3* and *SAINT* (Bruker, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2006) and *publCIF* (Westrip, 2010).

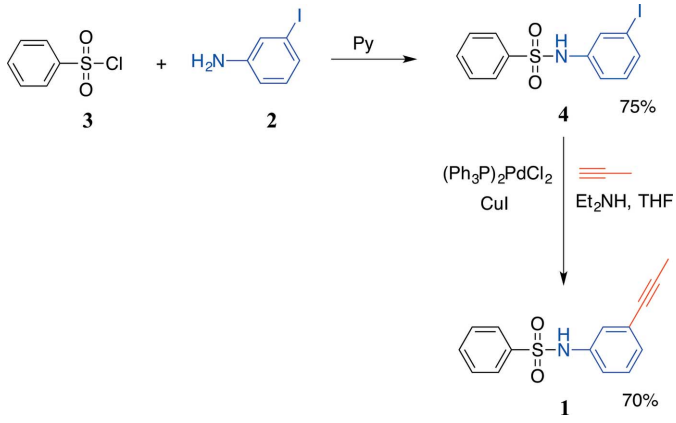


Figure 3
A synthetic scheme for the preparation of the title compound.

Synthesis and crystallization

The title compound, **1**, was synthesized by treatment of 3-iodoaniline, **2**, with benzenesulfonyl chloride, **3**, in the presence of pyridine, at room temperature to obtain, after purification by column chromatography (ether:hexane, 40:60), iodosulfonamide, **4**, in 75% yield. This aromatic iodide **4**, was treated with propyne, under Sonogashira's reaction conditions (Sonogashira *et al.*, 1975), using CuI and $(Ph_3P)_2PdCl_2$ as catalysts, (Fig. 3). After purification by column chromatography, using a solvent mixture of hexane:ethyl acetate (75:25), compound **1** was isolated in 70% yield, and with an overall yield of 53%. The product was recrystallized from ethyl acetate solution at room temperature to result in light-yellow blocks of the title compound.

Refinement

Crystal data, data collection and structure refinement are summarized in Table 2.

Acknowledgements

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full crystallographic data

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Crystal data

$C_{15}H_{13}NO_2S$
M_r = 271.32
Monoclinic, *Cc*
a = 8.4596 (4) Å
b = 24.9769 (13) Å
c = 7.1310 (4) Å
β = 117.557 (2)°
V = 1335.80 (12) Å^3
Z = 4
F(000) = 568
D_x = 1.349 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4995 reflections
θ = 2.8–27.5°
μ = 0.24 mm^{-1}
T = 100 K
Block, clear light yellow
0.35 × 0.20 × 0.15 mm

Data collection

Bruker D8 Venture CCD diffractometer
Radiation source: Incoatec Microsource
Mirrors monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2015)
T_{min} = 0.704, T_{max} = 0.746
9630 measured reflections
3028 independent reflections
2716 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.5°, θ_{min} = 2.8°
h = −10→10
k = −32→32
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.089
S = 1.03
3028 reflections
173 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 1.2876P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}
Absolute structure: Flack *x* determined using 1163 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter: 0.02 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All hydrogen atoms were placed geometrically and refined using a riding-atom model approximation, with C—H = 0.95–1.00 Å, with U_{iso}(H) = 1.2U_{eq}(C). A rotating group model was used for the methyl groups.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.73168 (11)	0.55417 (3)	0.33500 (12)	0.01205 (19)
O1	0.7499 (3)	0.58926 (10)	0.1883 (4)	0.0159 (5)
O2	0.7908 (3)	0.49971 (10)	0.3513 (4)	0.0171 (6)
N1	0.8464 (4)	0.57903 (11)	0.5727 (4)	0.0138 (6)
H1	0.9279	0.5593	0.6732	0.017*
C1	0.7946 (5)	0.72772 (15)	0.5484 (6)	0.0188 (8)
C2	0.7594 (6)	0.73618 (15)	0.7189 (6)	0.0243 (9)
H2	0.7403	0.7715	0.7534	0.029*
C3	0.7522 (6)	0.69340 (15)	0.8383 (8)	0.0267 (9)
H3	0.7297	0.6994	0.9553	0.032*
C4	0.7781 (5)	0.64153 (15)	0.7865 (6)	0.0176 (8)
H4	0.7705	0.612	0.8661	0.021*
C5	0.8149 (4)	0.63297 (13)	0.6184 (5)	0.0134 (7)
C6	0.8256 (5)	0.67587 (14)	0.5006 (5)	0.0156 (7)
H6	0.8538	0.6699	0.388	0.019*
C7	0.7989 (5)	0.77163 (15)	0.4189 (6)	0.0216 (9)
C8	0.7988 (5)	0.80679 (16)	0.3081 (6)	0.0232 (9)
C9	0.7969 (7)	0.85150 (17)	0.1717 (8)	0.0345 (11)
H9A	0.8463	0.8393	0.0788	0.052*
H9B	0.8692	0.8811	0.26	0.052*
H9C	0.6741	0.8637	0.0855	0.052*
C10	0.5068 (4)	0.55427 (14)	0.2802 (5)	0.0138 (7)
C11	0.4331 (5)	0.50847 (14)	0.3182 (5)	0.0159 (7)
H11	0.5029	0.477	0.3718	0.019*
C12	0.2569 (5)	0.50936 (17)	0.2771 (6)	0.0207 (8)
H12	0.205	0.4782	0.3023	0.025*
C13	0.1549 (5)	0.55524 (16)	0.1993 (6)	0.0202 (8)
H13	0.0343	0.5557	0.1737	0.024*
C14	0.2302 (5)	0.60067 (15)	0.1587 (6)	0.0215 (8)
H14	0.1597	0.6319	0.1025	0.026*
C15	0.4069 (5)	0.60055 (14)	0.1997 (6)	0.0168 (7)
H15	0.4588	0.6315	0.1733	0.02*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0130 (4)	0.0114 (4)	0.0127 (4)	0.0013 (4)	0.0067 (3)	−0.0001 (4)

O1	0.0207 (14)	0.0150 (12)	0.0146 (12)	0.0001 (10)	0.0103 (11)	0.0000 (10)
O2	0.0190 (13)	0.0147 (12)	0.0183 (13)	0.0030 (10)	0.0094 (11)	−0.0010 (10)
N1	0.0139 (15)	0.0131 (14)	0.0117 (14)	0.0025 (12)	0.0035 (12)	0.0011 (11)
C1	0.0196 (19)	0.0142 (17)	0.0189 (19)	−0.0017 (15)	0.0057 (15)	0.0006 (15)
C2	0.037 (2)	0.0141 (19)	0.022 (2)	0.0016 (17)	0.0139 (19)	−0.0035 (16)
C3	0.040 (3)	0.0230 (19)	0.0225 (19)	0.001 (2)	0.0188 (19)	−0.002 (2)
C4	0.0222 (19)	0.0162 (18)	0.0174 (18)	−0.0003 (15)	0.0119 (16)	0.0039 (15)
C5	0.0119 (17)	0.0106 (17)	0.0132 (17)	−0.0002 (13)	0.0019 (14)	−0.0016 (13)
C6	0.0148 (17)	0.0169 (18)	0.0140 (17)	−0.0019 (14)	0.0058 (14)	−0.0005 (14)
C7	0.026 (2)	0.0125 (19)	0.025 (2)	−0.0011 (16)	0.0105 (17)	−0.0060 (16)
C8	0.028 (2)	0.017 (2)	0.025 (2)	−0.0021 (17)	0.0128 (19)	−0.0025 (18)
C9	0.043 (3)	0.024 (2)	0.039 (3)	0.001 (2)	0.021 (2)	0.011 (2)
C10	0.0132 (15)	0.0171 (17)	0.0110 (16)	0.0015 (15)	0.0054 (14)	−0.0030 (14)
C11	0.0180 (18)	0.0158 (18)	0.0129 (16)	−0.0009 (15)	0.0062 (14)	0.0010 (14)
C12	0.0190 (19)	0.028 (2)	0.0158 (18)	−0.0072 (16)	0.0088 (15)	0.0017 (16)
C13	0.0142 (18)	0.030 (2)	0.0172 (18)	−0.0017 (17)	0.0078 (15)	−0.0075 (17)
C14	0.0182 (19)	0.019 (2)	0.022 (2)	0.0015 (16)	0.0046 (16)	−0.0051 (16)
C15	0.0194 (19)	0.0125 (17)	0.0168 (17)	−0.0014 (14)	0.0069 (15)	−0.0034 (14)

Geometric parameters (Å, º)

S1—O1	1.427 (3)	C7—C8	1.181 (5)
S1—O2	1.435 (3)	C8—C9	1.477 (5)
S1—N1	1.636 (3)	C9—H9A	0.98
S1—C10	1.756 (3)	C9—H9B	0.98
N1—C5	1.440 (4)	C9—H9C	0.98
N1—H1	0.88	C10—C11	1.388 (5)
C1—C6	1.394 (5)	C10—C15	1.390 (5)
C1—C2	1.396 (6)	C11—C12	1.380 (5)
C1—C7	1.445 (5)	C11—H11	0.95
C2—C3	1.385 (6)	C12—C13	1.387 (6)
C2—H2	0.95	C12—H12	0.95
C3—C4	1.392 (5)	C13—C14	1.395 (5)
C3—H3	0.95	C13—H13	0.95
C4—C5	1.387 (5)	C14—C15	1.384 (5)
C4—H4	0.95	C14—H14	0.95
C5—C6	1.390 (5)	C15—H15	0.95
C6—H6	0.95		
O1—S1—O2	119.34 (15)	C8—C7—C1	178.1 (4)
O1—S1—N1	108.24 (15)	C7—C8—C9	178.8 (4)
O2—S1—N1	104.96 (15)	C8—C9—H9A	109.5
O1—S1—C10	108.01 (16)	C8—C9—H9B	109.5
O2—S1—C10	108.67 (16)	H9A—C9—H9B	109.5
N1—S1—C10	107.00 (15)	C8—C9—H9C	109.5
C5—N1—S1	120.4 (2)	H9A—C9—H9C	109.5
C5—N1—H1	119.8	H9B—C9—H9C	109.5
S1—N1—H1	119.8	C11—C10—C15	121.6 (3)

C6—C1—C2	119.5 (3)	C11—C10—S1	119.6 (3)
C6—C1—C7	119.1 (3)	C15—C10—S1	118.8 (3)
C2—C1—C7	121.4 (3)	C12—C11—C10	118.9 (3)
C3—C2—C1	120.4 (4)	C12—C11—H11	120.5
C3—C2—H2	119.8	C10—C11—H11	120.5
C1—C2—H2	119.8	C11—C12—C13	120.6 (4)
C2—C3—C4	119.9 (4)	C11—C12—H12	119.7
C2—C3—H3	120.1	C13—C12—H12	119.7
C4—C3—H3	120.1	C12—C13—C14	119.7 (3)
C5—C4—C3	119.9 (4)	C12—C13—H13	120.1
C5—C4—H4	120.1	C14—C13—H13	120.1
C3—C4—H4	120.1	C15—C14—C13	120.4 (4)
C4—C5—C6	120.4 (3)	C15—C14—H14	119.8
C4—C5—N1	118.6 (3)	C13—C14—H14	119.8
C6—C5—N1	120.9 (3)	C14—C15—C10	118.7 (3)
C5—C6—C1	119.8 (3)	C14—C15—H15	120.7
C5—C6—H6	120.1	C10—C15—H15	120.7
C1—C6—H6	120.1		

O1—S1—N1—C5	54.7 (3)	O1—S1—C10—C11	148.8 (3)
O2—S1—N1—C5	−176.8 (3)	O2—S1—C10—C11	18.0 (3)
C10—S1—N1—C5	−61.5 (3)	N1—S1—C10—C11	−94.9 (3)
C6—C1—C2—C3	−1.1 (6)	O1—S1—C10—C15	−31.3 (3)
C7—C1—C2—C3	178.6 (4)	O2—S1—C10—C15	−162.1 (3)
C1—C2—C3—C4	−0.8 (6)	N1—S1—C10—C15	85.0 (3)
C2—C3—C4—C5	1.4 (6)	C15—C10—C11—C12	−0.7 (5)
C3—C4—C5—C6	−0.2 (5)	S1—C10—C11—C12	179.1 (3)
C3—C4—C5—N1	178.1 (4)	C10—C11—C12—C13	−0.1 (5)
S1—N1—C5—C4	126.3 (3)	C11—C12—C13—C14	1.1 (6)
S1—N1—C5—C6	−55.4 (4)	C12—C13—C14—C15	−1.3 (6)
C4—C5—C6—C1	−1.6 (5)	C13—C14—C15—C10	0.5 (5)
N1—C5—C6—C1	−179.9 (3)	C11—C10—C15—C14	0.5 (5)
C2—C1—C6—C5	2.3 (5)	S1—C10—C15—C14	−179.3 (3)
C7—C1—C6—C5	−177.5 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.88	2.55	2.984 (4)	111
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Symmetry codes: (i) *x*, −*y*+1, *z*+1/2; (ii) *x*, *y*, *z*+1.