Crystal structures of *p*-methyl-*N*-(2-phenyl-1*H*-indol-7-yl)-benzene-sulfonamide, $C_{21}H_{18}N_2O_2S$, and *p*-methoxy-*N*-(2-phenyl-1*H*-indol-7-yl)-benzene-sulfonamide, $C_{21}H_{18}N_2O_3S$

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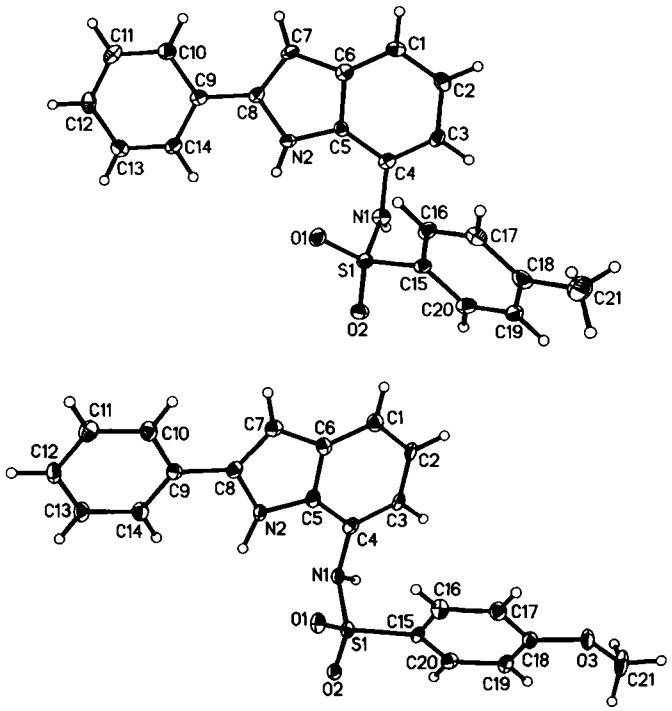

Abstract

$C_{21}H_{18}N_2O_2S$, orthorhombic, *Pbca* (no. 61), $a = 5.030(1)$ Å, $b = 18.894(4)$ Å, $c = 36.178(7)$ Å, $V = 3438.2$ Å^3, $Z = 8$, $R_{gt}(F) = 0.052$, $wR_{ref}(F^2) = 0.120$, $T = 100$ K.

$C_{21}H_{18}N_2O_3S$, orthorhombic, *Pbca* (no. 61), $a = 5.018(2)$ Å, $b = 20.010(7)$ Å, $c = 35.34(1)$ Å, $V = 3548.5$ Å^3, $Z = 8$, $R_{gt}(F) = 0.056$, $wR_{ref}(F^2) = 0.137$, $T = 100$ K.

Source of material

Basic 2-phenyl-indole group was prepared from selectively protected 2-amino-3-nitrophenol and phenylacetylene, according to Sonogashira coupling reaction [1]. The resulting 7-nitro-2-phenylindole was reduced into 7-amino-2-phenyindole with hydrogen by Pd/C and then converted into *p*-substituted *N*-(2-phenyl-1*H*-indol-7-yl)-benzene-sulfonamides with the corresponding *p*-substituted benzene-sulfonyl chloride in pyridine and tetrahydrofuran [2]. Colorless single crystals for X-ray analysis were grown by slow evaporation of the solutions of the compounds in ethyl acetate at 4 °C.

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Discussion

Individual syntheses of new heterocycles from indole have attached the attention of chemists in recent years because of their extensive application in biological activity [3]. An active intermediate *N*-(1*H*-indol-7-yl)methanesulfonamide is reacted with the appropriate carbinol to give the indole-derived modulators for the steroid hormone nuclear receptors [4]; the typical 2-arylidene-3-indolinone compounds obtained from direct hydrolysis of the a primary or secondary aryl amines are effective components for oxidative hair dyes [5]; the *N*-(7-indolyl)benzene-sulfonamides as a potential antitumor agent have been used for the discovery of potent cell cycle inhibitors [6]. During the design of high-affinity antipsychotic drugs from indole derivatives with amine, the structure-activity relationships of the substituent patterns on the pharmacophore template have been also attended [6]. We determined a series of crystal structures of *p*-substituted *N*-(2-phenyl-1*H*-indol-7-yl)-benzene-sulfonamides by X-ray single-crystal diffraction to gain better insight into the structures of these molecules with hydrogen-bonding patterns.

In the five-membered rings, the angles at N2 atoms are 110.1(3)° for *p*-methyl-*N*-(2-phenyl-1*H*-indol-7-yl)-benzene-sulfonamide (**I**), 109.5(3)° for *p*-methyl-*N*-(2-phenyl-1*H*-indol-7-yl)-benzene-sulfonamide (**II**), and 108.7(2)° for *p*-bromo-*N*-(2-phenyl-1*H*-indol-7-yl)-benzene-sulfonamide (**III**) [7]; the N2—C5 and N2—C8 bond lengths of these compounds [average 1.377(4) Å] are well within the range of the values normally considered standard for single C—N (1.47 Å) and double C=N (1.28 Å) bonds, which indicates that the environment around N2 atom is a normal sp^2-coordination as expected for π-conjugation of the indole ring [7]. The indole groups of three compounds are nearly coplanar with the average deviation of 0.0015 Å from the least squares plane. The rings of C15→C20 benzene and the planes of the indole groups make dihedral angles of 44.7(3)°, 39.5(3)°, and 40.0(3)° for **I**, **II**, and **III**, respectively; while the dihedral angles of the C9→C14 phenyl rings and the indole planes [4.5(2)° for **I**, 14.5(2)° for **II** and 34.9(2)° for **III**] among the three compounds have large differences, suggestive of steric repulsions between the phenyl rings and the indole planes being obviously influenced by different substitute groups. The average N1—C4 and N1—S1 bond lengths of the compounds are 1.440(2) Å and 1.628(2) Å, respectively, indicative of the standard single bond of the sp^3-hybridized amine-N1 atom. The angles at N1 in the structures of the three compounds are in the range of 117.9(2)° – 120.4(2)°. The environment around S1 is a highly distorted tetrahedron, indicated by the bond angles of O1–S1–O2 [average 120.3(2)°] being largely deviated from the standard planar angle of 109.2°. The average double S=O bond length is 1.428(2) Å.

In the crystal structure of **I**, two molecules are connected parallel to each other by N–H···O hydrogen bonds, with an N···O distance of 2.963(3) Å and an N–H···O angle of 169.1(3)°, to form a one-dimensional linear structure. The crystal packing in **II** is governed by two kinds of hydrogen bonds: the relatively strong N–H···O interaction [d(N···O) = 2.969(3) Å, ∠N–H···O = 161.4(3)°] arranging the molecules in chains running along [100] and the relatively weak C–H···O interaction [d(C···O) = 3.251(3) Å, ∠C–H···O = 167.4(2)°] connecting the chains into layers parallel to the (001) plane. There is no evidence of any aromatic π-π stacking of the indole rings in the present compounds. In the structure of **III**, a pair of head-to-tail intermolecular N–H···O bonds link adjacent molecules to form a dimer which is further linked via the N–H···O bonds [d(N···O) = 2.873(3) Å, ∠N–H···O = 166.6(3)°] to form a one-dimensional column [8].

1. *p*-Methyl-*N*-(2-phenyl-1*H*-indol-7-yl)-benzene-sulfonamide, $C_{21}H_{18}N_2O_2S$

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.08 × 0.10 × 0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.07 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	17110, 3369
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1924
$N(param)_{refined}$:	236
Program:	SHELXTL [9]

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

Atom	Site	x	y	z	U_{iso}
H(1B)	8c	0.2410	0.0761	0.1042	0.022
H(2)	8c	0.7951	0.0157	0.1321	0.020
H(1)	8c	0.6816	0.2009	0.2332	0.025
H(2A)	8c	0.3434	0.2561	0.2010	0.024
H(3)	8c	0.2073	0.2132	0.1436	0.022
H(7)	8c	1.0458	0.0787	0.2296	0.022
H(10)	8c	1.3478	–0.0220	0.2278	0.025
H(11)	8c	1.6488	–0.1137	0.2226	0.029
H(12)	8c	1.6687	–0.1809	0.1690	0.026
H(13)	8c	1.3756	–0.1568	0.1212	0.027
H(14)	8c	1.0767	–0.0654	0.1261	0.023
H(16)	8c	0.8107	0.2500	0.0923	0.023
H(17)	8c	0.7614	0.3689	0.0797	0.025
H(19)	8c	0.1268	0.3252	0.0167	0.024
H(20)	8c	0.1752	0.2068	0.0292	0.024
H(21A)	8c	0.3640	0.4430	0.0119	0.055
H(21B)	8c	0.5797	0.4609	0.0418	0.055
H(21C)	8c	0.2785	0.4584	0.0528	0.055

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}
S(1)	8c	0.5480(2)	0.12484(4)	0.06891(2)	0.0146(4)	0.0217(5)	0.0149(4)	0.0008(4)	0.0001(4)	0.0006(4)
O(1)	8c	0.8240(4)	0.1140(1)	0.07818(6)	0.015(1)	0.029(2)	0.020(1)	0.005(1)	0.001(1)	0.002(1)
O(2)	8c	0.4319(5)	0.0876(1)	0.03871(6)	0.022(1)	0.026(1)	0.017(1)	0.001(1)	–0.001(1)	–0.003(1)
N(1)	8c	0.3771(5)	0.1035(1)	0.10584(7)	0.015(2)	0.020(2)	0.019(1)	–0.007(1)	0.002(1)	0.002(1)
N(2)	8c	0.8099(5)	0.0400(1)	0.15211(7)	0.017(2)	0.021(2)	0.013(1)	0.001(1)	–0.002(1)	–0.002(1)
C(1)	8c	0.6271(7)	0.1832(2)	0.21041(9)	0.025(2)	0.023(2)	0.015(2)	–0.002(2)	–0.001(2)	0.000(2)
C(2)	8c	0.4254(7)	0.2163(2)	0.19106(8)	0.019(2)	0.020(2)	0.022(2)	0.000(2)	0.007(2)	–0.000(2)
C(3)	8c	0.3428(7)	0.1902(2)	0.15631(8)	0.011(2)	0.024(2)	0.019(2)	0.003(2)	0.000(2)	0.005(2)
C(4)	8c	0.4593(7)	0.1315(2)	0.14108(8)	0.013(2)	0.022(2)	0.016(2)	–0.003(2)	0.001(2)	0.004(2)
C(5)	8c	0.6611(7)	0.0981(2)	0.16069(8)	0.016(2)	0.015(2)	0.015(2)	0.001(2)	0.003(2)	0.001(1)
C(6)	8c	0.7489(7)	0.1230(2)	0.19555(8)	0.014(2)	0.021(2)	0.017(2)	–0.004(2)	0.003(1)	0.004(2)
C(7)	8c	0.9529(7)	0.0759(2)	0.20737(8)	0.016(2)	0.026(2)	0.013(2)	–0.001(2)	–0.003(2)	0.003(1)
C(8)	8c	0.9891(6)	0.0257(2)	0.18042(8)	0.013(2)	0.018(2)	0.016(2)	–0.001(1)	–0.001(1)	0.004(1)
C(9)	8c	1.1769(6)	–0.0332(2)	0.17749(8)	0.014(2)	0.022(2)	0.015(2)	–0.004(2)	0.003(1)	0.003(1)
C(10)	8c	1.3525(7)	–0.0488(2)	0.20628(9)	0.021(2)	0.022(2)	0.019(2)	–0.002(2)	0.000(2)	0.000(2)
C(11)	8c	1.5340(7)	–0.1039(2)	0.20311(9)	0.019(2)	0.026(2)	0.028(2)	–0.000(2)	–0.008(2)	0.006(2)
C(12)	8c	1.5456(7)	–0.1444(2)	0.17115(9)	0.016(2)	0.017(2)	0.033(2)	0.001(2)	0.005(2)	0.002(2)
C(13)	8c	1.3714(7)	–0.1296(2)	0.14264(9)	0.025(2)	0.021(2)	0.021(2)	0.000(2)	0.002(2)	–0.002(2)
C(14)	8c	1.1910(7)	–0.0748(2)	0.14565(9)	0.016(2)	0.024(2)	0.018(2)	–0.001(2)	–0.003(2)	0.003(2)
C(15)	8c	0.5033(6)	0.2159(2)	0.06133(8)	0.013(2)	0.022(2)	0.012(2)	–0.002(1)	0.001(1)	0.002(1)
C(16)	8c	0.6744(7)	0.2651(2)	0.07679(8)	0.014(2)	0.026(2)	0.017(2)	0.002(2)	–0.000(2)	0.004(2)
C(17)	8c	0.6441(7)	0.3364(2)	0.06934(9)	0.020(2)	0.023(2)	0.020(2)	–0.002(2)	0.002(2)	–0.004(2)
C(18)	8c	0.4391(8)	0.3602(2)	0.04643(8)	0.030(2)	0.021(2)	0.016(2)	0.003(2)	0.009(2)	0.001(1)
C(19)	8c	0.2656(7)	0.3102(2)	0.03176(8)	0.019(2)	0.024(2)	0.016(2)	0.004(2)	–0.000(2)	0.001(2)
C(20)	8c	0.2946(7)	0.2393(2)	0.03907(8)	0.016(2)	0.028(2)	0.015(2)	–0.003(2)	0.001(2)	0.001(2)
C(21)	8c	0.4130(9)	0.4377(2)	0.0374(1)	0.051(3)	0.028(2)	0.031(2)	0.005(2)	–0.002(2)	0.008(2)

2. *p*-Methoxy-*N*-(2-phenyl-1*H*-indol-7-yl)-benzene-sulfonamide, $C_{21}H_{18}N_2O_3S$

Table 4. Data collection and handling.

Crystal:	colorless bar, size 0.08 × 0.08 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.08 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	56.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	20153, 4218
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1682
$N(param)_{refined}$:	246
Program:	SHELXTL [9]

Table 5. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*c*	0.7759	0.6196	0.4036	0.061
H(2)	8*c*	0.1537	0.5241	0.3761	0.041
H(1A)	8*c*	0.2796	0.6983	0.2662	0.032
H(2A)	8*c*	0.6228	0.7507	0.2991	0.031
H(3A)	8*c*	0.7544	0.7133	0.3587	0.028
H(7A)	8*c*	−0.0816	0.5826	0.2704	0.027
H(10A)	8*c*	−0.4273	0.4987	0.2776	0.031
H(11A)	8*c*	−0.6892	0.4036	0.2778	0.036
H(12A)	8*c*	−0.6081	0.3176	0.3217	0.033
H(13A)	8*c*	−0.2712	0.3315	0.3664	0.033
H(14A)	8*c*	−0.0129	0.4284	0.3671	0.032
H(16A)	8*c*	0.1123	0.7430	0.4040	0.030
H(17A)	8*c*	0.1502	0.8596	0.4067	0.032
H(19A)	8*c*	0.7819	0.8419	0.4771	0.032
H(20A)	8*c*	0.7392	0.7241	0.4742	0.029
H(21A)	8*c*	0.8543	0.9483	0.4503	0.061
H(21B)	8*c*	0.6790	0.9498	0.4881	0.061
H(21C)	8*c*	0.6624	1.0105	0.4587	0.061

Table 6. 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}
S(1)	8*c*	0.4025(2)	0.63567(5)	0.43556(3)	0.0239(5)	0.0139(5)	0.0252(5)	0.0007(5)	−0.0008(5)	−0.0013(5)
O(1)	8*c*	0.1304(5)	0.6198(1)	0.42746(7)	0.024(1)	0.017(1)	0.036(2)	−0.000(1)	0.001(1)	−0.002(1)
O(2)	8*c*	0.5312(5)	0.6074(1)	0.46791(6)	0.030(2)	0.018(2)	0.024(2)	0.000(1)	−0.003(1)	0.001(1)
O(3)	8*c*	0.4700(5)	0.9288(1)	0.44175(7)	0.036(2)	0.013(1)	0.037(2)	−0.003(1)	−0.004(1)	−0.001(1)
N(1)	8*c*	0.5747(6)	0.6112(1)	0.39901(8)	0.023(2)	0.014(2)	0.023(2)	−0.000(2)	−0.002(1)	−0.000(1)
N(2)	8*c*	0.1493(6)	0.5476(2)	0.35099(9)	0.025(2)	0.014(2)	0.024(2)	−0.003(2)	−0.001(1)	−0.001(2)
C(1)	8*c*	0.3345(7)	0.6819(2)	0.2902(1)	0.028(2)	0.023(2)	0.028(2)	0.000(2)	0.006(2)	−0.003(2)
C(2)	8*c*	0.5361(8)	0.7131(2)	0.3100(1)	0.034(2)	0.014(2)	0.030(2)	−0.007(2)	0.002(2)	−0.005(2)
C(3)	8*c*	0.6158(7)	0.6905(2)	0.3457(1)	0.028(2)	0.014(2)	0.028(2)	0.001(2)	0.000(2)	−0.008(2)
C(4)	8*c*	0.4978(7)	0.6360(2)	0.3624(1)	0.019(2)	0.020(2)	0.025(2)	0.005(2)	0.002(2)	−0.006(2)
C(5)	8*c*	0.2942(7)	0.6037(2)	0.3422(1)	0.025(2)	0.014(2)	0.028(2)	0.003(2)	0.002(2)	−0.002(2)
C(6)	8*c*	0.2121(7)	0.6258(2)	0.3061(1)	0.018(2)	0.018(2)	0.026(2)	0.003(2)	0.003(2)	0.000(2)
C(7)	8*c*	0.0119(7)	0.5805(2)	0.2938(1)	0.024(2)	0.021(2)	0.022(2)	0.007(2)	0.002(2)	−0.004(2)
C(8)	8*c*	−0.0229(7)	0.5330(2)	0.3215(1)	0.025(2)	0.016(2)	0.024(2)	0.005(2)	−0.000(2)	−0.005(2)
C(9)	8*c*	−0.1922(7)	0.4735(2)	0.3222(1)	0.023(2)	0.017(2)	0.021(2)	0.004(2)	0.001(2)	−0.001(2)
C(10)	8*c*	−0.3951(8)	0.4649(2)	0.2958(1)	0.027(2)	0.019(2)	0.031(2)	0.002(2)	−0.004(2)	0.002(2)
C(11)	8*c*	−0.5498(8)	0.4081(2)	0.2958(1)	0.029(2)	0.027(2)	0.034(3)	0.002(2)	−0.003(2)	−0.005(2)
C(12)	8*c*	−0.5037(8)	0.3572(2)	0.3220(1)	0.029(2)	0.018(2)	0.036(2)	−0.006(2)	0.007(2)	−0.003(2)
C(13)	8*c*	−0.3040(7)	0.3656(2)	0.3482(1)	0.035(2)	0.016(2)	0.033(2)	0.000(2)	−0.003(2)	0.000(2)
C(14)	8*c*	−0.1491(7)	0.4233(2)	0.3486(1)	0.027(2)	0.023(2)	0.030(2)	−0.003(2)	−0.004(2)	0.002(2)
C(15)	8*c*	0.4228(7)	0.7241(2)	0.4390(1)	0.022(2)	0.014(2)	0.023(2)	0.003(2)	0.001(2)	−0.005(2)
C(16)	8*c*	0.2478(8)	0.7632(2)	0.4189(1)	0.019(2)	0.024(2)	0.034(3)	0.001(2)	−0.004(2)	−0.000(2)
C(17)	8*c*	0.2696(8)	0.8320(2)	0.4205(1)	0.030(2)	0.021(2)	0.029(2)	0.003(2)	−0.003(2)	0.001(2)
C(18)	8*c*	0.4683(7)	0.8600(2)	0.4425(1)	0.028(2)	0.018(2)	0.025(2)	0.000(2)	0.005(2)	−0.003(2)
C(19)	8*c*	0.6453(8)	0.8218(2)	0.4624(1)	0.028(2)	0.021(2)	0.031(2)	−0.002(2)	0.001(2)	−0.005(2)
C(20)	8*c*	0.6198(8)	0.7519(2)	0.4605(1)	0.024(2)	0.024(2)	0.023(2)	0.006(2)	−0.001(2)	−0.001(2)
C(21)	8*c*	0.6834(8)	0.9620(2)	0.4613(1)	0.045(3)	0.016(2)	0.061(3)	−0.006(2)	−0.015(2)	−0.006(2)

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