Crystal structure of spiro-[2.3′′]oxindole-spiro[3.3′]-5′-(4-methylsulfanylphenylmethylidene)-1′-benzyl-4′(1*H*)-pyridinone-4-(4-methylsulfanylphenyl)hexahydro-1*H*-pyrrolizine — dichloromethane, $C_{40}H_{39}N_3O_2S_2 \cdot CH_2Cl_2$

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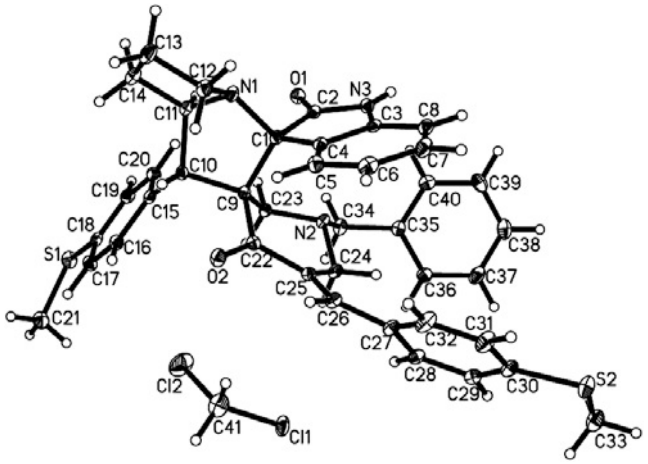

Abstract

$C_{41}H_{41}Cl_2N_3O_2S_2$, triclinic, $P\bar{1}$ (no. 2), a = 9.1853(9) Å, b = 12.846(1) Å, c = 17.559(2) Å, α = 70.299(9)°, β = 77.36(1)°, γ = 71.996(9)°, V = 1839.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.044, $wR_{ref}(F^2)$ = 0.097, T = 113 K.

Source of material

A mixture of 1-benzyl-3,5-bis(4-(methylthio)benzylidene)-piperidin-4-one (1 mmol), isatin (1 mmol) and proline (1 mmol) were refluxed in methanol (60 ml) until the complete dissolution of the starting material as evidenced by the TLC. After the reaction was over, the solvent was removed in vacuo and the residue was separated by column chromatography (silica gel, petroleum ether/ethylacetate = 5:1) to give the title compound. 20 mg of the title compound was dissolved in 15 ml dichloromethane/hexane mixed solvent; the solution was kept at room temperature for 15 d by natural evaporation to give colorless single crystals suitable for X-ray diffraction analysis.

Experimental details

The H atoms bound to C were included in the riding model approximation with d(C—H) = 0.95 - 0.99 Å and U_{iso}(H) = 1.2 U_{eq}(C) thermal parameters related to the atoms to which they were bonded. The H3 atom bound to N3 was found in difference Fourier map and refined freely.

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Discussion

In the title compound, the two spiro junctions link a 2-oxindole ring, a hexahydro-1*H*-pyrrolizine ring and a piperidine ring in an chair conformation. Two molecules are connected into a dimer by two N–H···O hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	colorless chip, size 0.10 × 0.20 × 0.24 mm
Wavelength:	Mo K_α radiation (0.71070 Å)
μ:	3.31 cm^{-1}
Diffractometer, scan mode:	Rigaku Saturn CCD, φ/ω
$2\theta_{max}$:	55.82°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	18478, 8642
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5217
$N(param)_{refined}$:	480
Programs:	SHELXS-97 [1], SHELXL-97 [2], SHELXTL [3]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(3)	2*i*		1.022(3)	0.586(2)	0.930(1)	0.045(7)
H(5)	2*i*		1.2692	0.5402	0.6508	0.027
H(6)	2*i*		1.2851	0.7305	0.6023	0.031
H(7)	2*i*		1.1814	0.8517	0.6843	0.033
H(8)	2*i*		1.0695	0.7831	0.8195	0.028
H(10)	2*i*		1.1921	0.2560	0.7320	0.021
H(11)	2*i*		1.1900	0.2092	0.9045	0.025
H(12A)	2*i*		1.4674	0.3965	0.7581	0.031
H(12B)	2*i*		1.3773	0.3539	0.7098	0.031
H(13A)	2*i*		1.5811	0.2130	0.8294	0.040
H(13B)	2*i*		1.5750	0.1919	0.7449	0.040
H(14A)	2*i*		1.3892	0.0978	0.8028	0.036
H(14B)	2*i*		1.4265	0.0932	0.8895	0.036
H(16)	2*i*		1.0447	0.1665	0.6896	0.025
H(17)	2*i*		0.9042	0.0321	0.7183	0.029
H(19)	2*i*		0.8663	–0.0016	0.9584	0.027
H(20)	2*i*		1.0071	0.1309	0.9299	0.029
H(21A)	2*i*		0.6639	–0.0255	0.7501	0.048
H(21B)	2*i*		0.6381	–0.1506	0.7950	0.048
H(21C)	2*i*		0.8050	–0.1360	0.7498	0.048
H(23A)	2*i*		0.8093	0.3513	0.8061	0.020
H(23B)	2*i*		0.8818	0.3293	0.8871	0.020
H(24A)	2*i*		0.7023	0.6472	0.7544	0.023
H(24B)	2*i*		0.6590	0.5426	0.7427	0.023
H(26)	2*i*		0.9762	0.6301	0.5792	0.025
H(28)	2*i*		0.5834	0.7424	0.6460	0.026
H(29)	2*i*		0.4519	0.9346	0.6221	0.027

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(31)	2*i*		0.8546	1.0222	0.5231	0.038
H(32)	2*i*		0.9826	0.8319	0.5427	0.035
H(33A)	2*i*		0.3178	1.1233	0.5612	0.052
H(33B)	2*i*		0.3004	1.2337	0.5889	0.052
H(33C)	2*i*		0.3429	1.1087	0.6520	0.052
H(34A)	2*i*		0.6784	0.4314	0.9456	0.026
H(34B)	2*i*		0.5753	0.4675	0.8739	0.026
H(36)	2*i*		0.3980	0.6548	0.8414	0.031

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(37)	2*i*		0.2700	0.8224	0.8766	0.040
H(38)	2*i*		0.3621	0.8711	0.9705	0.040
H(39)	2*i*		0.5838	0.7517	1.0282	0.035
H(40)	2*i*		0.7136	0.5866	0.9907	0.028
H(41A)	2*i*	0.507	0.7357	0.4486	0.4889	0.046
H(41B)	2*i*	0.507	0.8380	0.4885	0.5313	0.046
H(41C)	2*i*	0.493	0.8055	0.4866	0.4782	0.046
H(41D)	2*i*	0.493	0.8469	0.4737	0.5654	0.046

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*i*		0.75883(6)	–0.09942(4)	0.87441(3)	0.0262(3)	0.0275(3)	0.0352(3)	–0.0121(2)	0.0015(2)	–0.0102(2)
S(2)	2*i*		0.55864(6)	1.14480(4)	0.56164(3)	0.0329(3)	0.0196(3)	0.0403(3)	–0.0043(2)	–0.0019(2)	–0.0043(2)
O(1)	2*i*		1.0313(1)	0.3784(1)	0.96002(7)	0.0255(7)	0.0217(7)	0.0161(7)	–0.0065(5)	–0.0005(6)	–0.0037(5)
O(2)	2*i*		1.0803(2)	0.4250(1)	0.63371(7)	0.0273(8)	0.0224(7)	0.0186(7)	–0.0031(6)	0.0021(6)	–0.0060(6)
N(1)	2*i*		1.2805(2)	0.3407(1)	0.82881(9)	0.0150(8)	0.0173(8)	0.0258(9)	–0.0017(6)	–0.0001(7)	–0.0076(7)
N(2)	2*i*		0.7880(2)	0.4955(1)	0.83381(8)	0.0141(8)	0.0150(7)	0.0141(8)	–0.0016(6)	0.0015(6)	–0.0008(6)
N(3)	2*i*		1.0525(2)	0.5611(1)	0.8915(1)	0.0191(9)	0.0203(8)	0.0190(9)	–0.0038(6)	0.0001(7)	–0.0087(7)
C(1)	2*i*		1.1352(2)	0.4304(1)	0.8130(1)	0.0146(9)	0.0178(9)	0.021(1)	–0.0017(7)	–0.0017(8)	–0.0069(8)
C(2)	2*i*		1.0632(2)	0.4515(2)	0.8970(1)	0.0136(9)	0.0208(9)	0.021(1)	–0.0015(7)	–0.0018(8)	–0.0087(8)
C(3)	2*i*		1.1062(2)	0.6206(1)	0.8129(1)	0.0116(9)	0.0196(9)	0.022(1)	–0.0022(7)	–0.0038(7)	–0.0048(8)
C(4)	2*i*		1.1610(2)	0.5477(1)	0.7639(1)	0.0120(9)	0.0174(9)	0.021(1)	–0.0043(7)	–0.0016(7)	–0.0048(8)
C(5)	2*i*		1.2288(2)	0.5889(2)	0.6850(1)	0.017(1)	0.024(1)	0.025(1)	–0.0057(8)	0.0005(8)	–0.0085(8)
C(6)	2*i*		1.2375(2)	0.7023(2)	0.6562(1)	0.019(1)	0.028(1)	0.027(1)	–0.0099(8)	–0.0004(8)	–0.0015(9)
C(7)	2*i*		1.1772(2)	0.7741(2)	0.7054(1)	0.022(1)	0.018(1)	0.038(1)	–0.0070(8)	–0.0031(9)	–0.0010(9)
C(8)	2*i*		1.1104(2)	0.7343(2)	0.7853(1)	0.016(1)	0.021(1)	0.034(1)	–0.0012(8)	–0.0050(9)	–0.0098(9)
C(9)	2*i*		1.0298(2)	0.3787(1)	0.7807(1)	0.0144(9)	0.0153(9)	0.017(1)	–0.0029(7)	–0.0002(7)	–0.0054(7)
C(10)	2*i*		1.1290(2)	0.2544(1)	0.7864(1)	0.018(1)	0.0166(9)	0.017(1)	–0.0025(7)	–0.0007(7)	–0.0071(7)
C(11)	2*i*		1.2413(2)	0.2316(1)	0.8471(1)	0.019(1)	0.0173(9)	0.025(1)	0.0001(7)	–0.0052(8)	–0.0074(8)
C(12)	2*i*		1.4117(2)	0.3393(2)	0.7629(1)	0.015(1)	0.029(1)	0.032(1)	–0.0040(8)	0.0024(8)	–0.0126(9)
C(13)	2*i*		1.5117(2)	0.2178(2)	0.7918(1)	0.021(1)	0.032(1)	0.040(1)	0.0032(9)	–0.0015(9)	–0.014(1)
C(14)	2*i*		1.3957(2)	0.1446(2)	0.8361(1)	0.024(1)	0.025(1)	0.039(1)	0.0035(9)	–0.0083(9)	–0.0103(9)
C(15)	2*i*		1.0409(2)	0.1645(1)	0.8058(1)	0.020(1)	0.0135(9)	0.020(1)	–0.0010(7)	–0.0034(8)	–0.0034(8)
C(16)	2*i*		1.0084(2)	0.1325(2)	0.7447(1)	0.020(1)	0.023(1)	0.019(1)	–0.0039(8)	0.0010(8)	–0.0072(8)
C(17)	2*i*		0.9241(2)	0.0523(2)	0.7616(1)	0.022(1)	0.027(1)	0.026(1)	–0.0062(8)	–0.0007(8)	–0.0128(9)
C(18)	2*i*		0.8692(2)	0.0018(1)	0.8417(1)	0.017(1)	0.0157(9)	0.029(1)	–0.0009(7)	–0.0033(8)	–0.0065(8)
C(19)	2*i*		0.9023(2)	0.0327(1)	0.9033(1)	0.027(1)	0.0181(9)	0.019(1)	–0.0047(8)	–0.0042(8)	–0.0003(8)
C(20)	2*i*		0.9861(2)	0.1118(1)	0.8864(1)	0.030(1)	0.021(1)	0.020(1)	–0.0052(8)	–0.0056(9)	–0.0049(8)
C(21)	2*i*		0.7110(2)	–0.1033(2)	0.7818(1)	0.025(1)	0.034(1)	0.043(1)	–0.0119(9)	–0.002(1)	–0.017(1)
C(22)	2*i*		1.0013(2)	0.4509(1)	0.6937(1)	0.0160(9)	0.0190(9)	0.019(1)	–0.0065(7)	–0.0004(7)	–0.0061(8)
C(23)	2*i*		0.8698(2)	0.3803(1)	0.8310(1)	0.0167(9)	0.0146(9)	0.016(1)	–0.0034(7)	–0.0006(7)	–0.0032(7)
C(24)	2*i*		0.7430(2)	0.5666(1)	0.7537(1)	0.0154(9)	0.0187(9)	0.019(1)	–0.0025(7)	–0.0005(8)	–0.0027(8)
C(25)	2*i*		0.8747(2)	0.5591(1)	0.6856(1)	0.0171(9)	0.0199(9)	0.016(1)	–0.0052(7)	–0.0013(7)	–0.0045(8)
C(26)	2*i*		0.8920(2)	0.6471(1)	0.6193(1)	0.018(1)	0.022(1)	0.018(1)	–0.0039(8)	–0.0012(8)	–0.0025(8)
C(27)	2*i*		0.7987(2)	0.7655(2)	0.6005(1)	0.019(1)	0.021(1)	0.019(1)	–0.0035(8)	–0.0022(8)	–0.0016(8)
C(28)	2*i*		0.6393(2)	0.7991(2)	0.6217(1)	0.021(1)	0.021(1)	0.018(1)	–0.0066(8)	–0.0026(8)	0.0003(8)
C(29)	2*i*		0.5607(2)	0.9138(2)	0.6081(1)	0.019(1)	0.026(1)	0.019(1)	–0.0050(8)	–0.0015(8)	–0.0040(8)
C(30)	2*i*		0.6404(2)	0.9983(2)	0.5742(1)	0.025(1)	0.020(1)	0.021(1)	–0.0035(8)	–0.0044(8)	0.0006(8)
C(31)	2*i*		0.7992(2)	0.9654(2)	0.5490(1)	0.024(1)	0.024(1)	0.038(1)	–0.0096(9)	–0.0013(9)	0.0031(9)
C(32)	2*i*		0.8753(2)	0.8522(2)	0.5613(1)	0.020(1)	0.027(1)	0.029(1)	–0.0045(8)	0.0024(9)	0.0037(9)
C(33)	2*i*		0.3571(2)	1.1536(2)	0.5947(1)	0.036(1)	0.028(1)	0.029(1)	0.0022(9)	0.0055(9)	–0.0089(9)
C(34)	2*i*		0.6488(2)	0.4908(1)	0.8944(1)	0.020(1)	0.0201(9)	0.021(1)	–0.0067(8)	0.0045(8)	–0.0042(8)
C(35)	2*i*		0.5691(2)	0.6024(1)	0.9130(1)	0.0143(9)	0.0204(9)	0.019(1)	–0.0064(7)	0.0051(7)	–0.0050(8)
C(36)	2*i*		0.4365(2)	0.6743(2)	0.8793(1)	0.018(1)	0.030(1)	0.030(1)	–0.0042(8)	–0.0032(9)	–0.0116(9)
C(37)	2*i*		0.3601(2)	0.7739(2)	0.9003(1)	0.020(1)	0.029(1)	0.045(1)	0.0026(9)	–0.004(1)	–0.011(1)
C(38)	2*i*		0.4147(2)	0.8030(2)	0.9559(1)	0.032(1)	0.027(1)	0.039(1)	–0.0082(9)	0.006(1)	–0.015(1)
C(39)	2*i*		0.5461(2)	0.7325(2)	0.9898(1)	0.034(1)	0.032(1)	0.029(1)	–0.018(1)	0.0048(9)	–0.0130(9)
C(40)	2*i*		0.6223(2)	0.6340(2)	0.9678(1)	0.019(1)	0.027(1)	0.022(1)	–0.0089(8)	0.0007(8)	–0.0034(9)
C(41)	2*i*	0.507(9)	0.744(1)	0.4616(6)	0.5401(8)	0.025(3)	0.041(1)	0.040(2)	–0.008(2)	0.012(2)	–0.011(1)
Cl(1)	2*i*	0.507	0.5841(4)	0.5669(3)	0.5617(2)	0.020(1)	0.026(1)	0.059(2)	0.0011(8)	–0.0054(9)	–0.0228(9)
Cl(2)	2*i*	0.507	0.7616(3)	0.3348(2)	0.6167(3)	0.063(1)	0.0311(8)	0.064(2)	–0.0157(7)	–0.022(1)	0.009(1)
C(41')	2*i*	0.493	0.770(1)	0.4653(6)	0.5375(9)	0.025(3)	0.041(1)	0.040(2)	–0.008(2)	0.012(2)	–0.011(1)
Cl(1')	2*i*	0.493	0.5932(6)	0.5572(4)	0.5568(3)	0.049(2)	0.071(2)	0.160(4)	0.006(2)	–0.010(2)	–0.070(2)
Cl(2')	2*i*	0.493	0.7617(3)	0.3253(2)	0.5702(4)	0.071(1)	0.0341(8)	0.071(3)	–0.0132(7)	0.005(1)	–0.0109(9)

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