Synthesis, Characterization and Single Crystal X-ray Diffraction Analysis of Fused Triazolo/Thiadiazole Clubbed with Indole Scaffold

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X-Ray structure determinations protocol

The crystal of **9** was immersed in cryo-oil, mounted in a loop, and measured at a temperature of 120 K. The X-ray diffraction data was collected on a Rigaku Oxford Diffraction Supernova diffractometer using Mo Kα radiation. The *CrysAlisPro* [1] software package was used for cell refinement and data reduction. An analytical absorption correction (*CrysAlisPro* [1]) was applied to the intensities before structure solution. Structure was solved by intrinsic phasing (*SHELXT* [2]) method. Structural refinement was carried out using *SHELXL* [3] software with *SHELXLE* [4] graphical user interface. The NH hydrogen atom was located from the difference Fourier map and refined isotropically. All other hydrogen atoms were positioned geometrically and constrained to ride on their parent atoms, with C-H = 0.95 – 0.98 Å and U_{iso} = 1.2-1.5·U_{eq}(parent atom).

References

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33 Sheldrick, G.M. Crystal structure refinement with SHELXL. *Acta Cryst. C.* **2015**, *71*, 3–8.
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Table S1. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **9**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	8793(1)	6693(1)	3808(1)	23(1)
N(1)	4046(2)	3998(1)	1161(1)	19(1)

N(2)	5966(2)	5593(1)	1249(1)	22(1)
N(3)	7007(2)	6291(1)	1597(1)	25(1)
N(4)	6826(2)	5487(1)	3074(1)	19(1)
N(5)	7259(2)	5288(1)	4205(1)	19(1)
C(1)	4883(2)	3809(1)	3041(2)	20(1)
C(2)	3823(2)	3118(1)	2600(2)	20(1)
C(3)	3232(2)	2400(1)	3088(2)	23(1)
C(4)	2153(2)	1855(1)	2410(2)	25(1)
C(5)	1660(2)	2003(1)	1244(2)	24(1)
C(6)	2235(2)	2697(1)	726(2)	21(1)
C(7)	3319(2)	3255(1)	1423(1)	19(1)
C(8)	4984(2)	4328(1)	2147(1)	19(1)
C(9)	5880(2)	5111(1)	2137(1)	19(1)
C(10)	7489(2)	6196(1)	2691(2)	21(1)
C(11)	8296(2)	5870(1)	4691(1)	19(1)
C(12)	9065(2)	5859(1)	5891(1)	19(1)
C(13)	10147(2)	6507(1)	6366(2)	23(1)
C(14)	10863(2)	6492(1)	7509(2)	25(1)
C(15)	10541(2)	5840(1)	8203(2)	23(1)
C(16)	11281(3)	5843(1)	9450(2)	31(1)
C(17)	9478(2)	5185(1)	7717(2)	23(1)
C(18)	8744(2)	5193(1)	6577(1)	21(1)

Table S2. Selected bond lengths [Å] and angles [°] for **9**

S(1)-C(10)	1.7275(18)
S(1)-C(11)	1.7734(17)
N(1)-C(7)	1.376(2)
N(1)-C(8)	1.381(2)
N(2)-C(9)	1.324(2)
N(2)-N(3)	1.402(2)
N(3)-C(10)	1.306(2)
N(4)-C(10)	1.360(2)
N(4)-C(9)	1.370(2)
N(4)-N(5)	1.3747(19)
N(5)-C(11)	1.304(2)

Symmetry transformations used to generate equivalent atoms:

Table S3. Bond lengths [Å] and angles [°] for **9**.

S(1)-C(10)	1.7275(18)
S(1)-C(11)	1.7734(17)
N(1)-C(7)	1.376(2)
N(1)-C(8)	1.381(2)
N(1)-H(1)	0.85(2)
N(2)-C(9)	1.324(2)
N(2)-N(3)	1.402(2)
N(3)-C(10)	1.306(2)
N(4)-C(10)	1.360(2)
N(4)-C(9)	1.370(2)
N(4)-N(5)	1.3747(19)
N(5)-C(11)	1.304(2)
C(1)-C(8)	1.368(2)
C(1)-C(2)	1.427(2)
C(1)-H(1A)	0.9500
C(2)-C(3)	1.403(2)
C(2)-C(7)	1.412(2)
C(3)-C(4)	1.375(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.401(3)
C(4)-H(4)	0.9500
C(5)-C(6)	1.386(2)
C(5)-H(5)	0.9500
C(6)-C(7)	1.402(2)
C(6)-H(6)	0.9500
C(8)-C(9)	1.437(2)
C(11)-C(12)	1.456(2)
C(12)-C(18)	1.395(2)
C(12)-C(13)	1.395(2)
C(13)-C(14)	1.385(2)
C(13)-H(13)	0.9500
C(14)-C(15)	1.383(3)
C(14)-H(14)	0.9500
C(15)-C(17)	1.399(2)
C(15)-C(16)	1.504(2)
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
C(17)-C(18)	1.385(2)
C(17)-H(17)	0.9500
C(18)-H(18)	0.9500
C(10)-S(1)-C(11)	87.61(8)
C(7)-N(1)-C(8)	108.31(14)
C(7)-N(1)-H(1)	124.8(16)
C(8)-N(1)-H(1)	126.4(16)
C(9)-N(2)-N(3)	109.54(14)
C(10)-N(3)-N(2)	104.79(14)
C(10)-N(4)-C(9)	105.87(14)
C(10)-N(4)-N(5)	118.88(14)
C(9)-N(4)-N(5)	135.17(14)

C(11)-N(5)-N(4)	107.48(14)
C(8)-C(1)-C(2)	106.89(15)
C(8)-C(1)-H(1A)	126.6
C(2)-C(1)-H(1A)	126.6
C(3)-C(2)-C(7)	119.24(16)
C(3)-C(2)-C(1)	133.89(16)
C(7)-C(2)-C(1)	106.87(15)
C(4)-C(3)-C(2)	119.10(17)
C(4)-C(3)-H(3)	120.4
C(2)-C(3)-H(3)	120.4
C(3)-C(4)-C(5)	120.97(17)
C(3)-C(4)-H(4)	119.5
C(5)-C(4)-H(4)	119.5
C(6)-C(5)-C(4)	121.80(17)
C(6)-C(5)-H(5)	119.1
C(4)-C(5)-H(5)	119.1
C(5)-C(6)-C(7)	117.00(16)
C(5)-C(6)-H(6)	121.5
C(7)-C(6)-H(6)	121.5
N(1)-C(7)-C(6)	130.16(16)
N(1)-C(7)-C(2)	107.94(15)
C(6)-C(7)-C(2)	121.88(16)
C(1)-C(8)-N(1)	109.98(15)
C(1)-C(8)-C(9)	129.12(16)
N(1)-C(8)-C(9)	120.90(15)
N(2)-C(9)-N(4)	107.75(14)
N(2)-C(9)-C(8)	127.46(16)
N(4)-C(9)-C(8)	124.79(15)
N(3)-C(10)-N(4)	112.04(15)
N(3)-C(10)-S(1)	138.55(14)
N(4)-C(10)-S(1)	109.37(12)
N(5)-C(11)-C(12)	123.27(15)
N(5)-C(11)-S(1)	116.67(13)
C(12)-C(11)-S(1)	120.05(12)
C(18)-C(12)-C(13)	119.08(16)
C(18)-C(12)-C(11)	120.27(15)
C(13)-C(12)-C(11)	120.65(16)
C(14)-C(13)-C(12)	120.12(17)
C(14)-C(13)-H(13)	119.9
C(12)-C(13)-H(13)	119.9
C(15)-C(14)-C(13)	121.42(16)
C(15)-C(14)-H(14)	119.3
C(13)-C(14)-H(14)	119.3
C(14)-C(15)-C(17)	118.20(16)
C(14)-C(15)-C(16)	120.93(17)
C(17)-C(15)-C(16)	120.86(17)
C(15)-C(16)-H(16A)	109.5
C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(15)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(18)-C(17)-C(15)	121.11(17)

C(18)-C(17)-H(17)	119.4
C(15)-C(17)-H(17)	119.4
C(17)-C(18)-C(12)	120.07(16)
C(17)-C(18)-H(18)	120.0
C(12)-C(18)-H(18)	120.0

Symmetry transformations used to generate equivalent atoms:

Table S4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **9.** The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	28(1)	19(1)	22(1)	1(1)	1(1)	-5(1)
N(1)	21(1)	20(1)	16(1)	1(1)	1(1)	-2(1)
N(2)	27(1)	21(1)	19(1)	1(1)	3(1)	-1(1)
N(3)	30(1)	22(1)	21(1)	1(1)	2(1)	-3(1)
N(4)	21(1)	18(1)	16(1)	0(1)	2(1)	0(1)
N(5)	20(1)	21(1)	15(1)	0(1)	1(1)	0(1)
C(1)	21(1)	22(1)	17(1)	1(1)	2(1)	0(1)
C(2)	19(1)	20(1)	20(1)	2(1)	4(1)	1(1)
C(3)	25(1)	22(1)	23(1)	4(1)	4(1)	1(1)
C(4)	26(1)	20(1)	30(1)	4(1)	8(1)	-1(1)
C(5)	23(1)	21(1)	29(1)	-4(1)	3(1)	-1(1)
C(6)	22(1)	20(1)	21(1)	-2(1)	2(1)	2(1)
C(7)	18(1)	17(1)	22(1)	1(1)	4(1)	2(1)
C(8)	18(1)	22(1)	17(1)	-1(1)	2(1)	1(1)
C(9)	21(1)	20(1)	16(1)	-2(1)	2(1)	2(1)
C(10)	24(1)	18(1)	22(1)	0(1)	4(1)	-1(1)
C(11)	21(1)	17(1)	20(1)	0(1)	5(1)	1(1)
C(12)	18(1)	19(1)	19(1)	-3(1)	2(1)	2(1)
C(13)	24(1)	21(1)	24(1)	-2(1)	2(1)	-2(1)
C(14)	22(1)	25(1)	25(1)	-8(1)	0(1)	-2(1)
C(15)	21(1)	26(1)	21(1)	-6(1)	1(1)	6(1)
C(16)	32(1)	36(1)	22(1)	-6(1)	-2(1)	6(1)
C(17)	26(1)	24(1)	20(1)	0(1)	2(1)	2(1)
C(18)	22(1)	20(1)	21(1)	-2(1)	2(1)	-1(1)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **9.**

	x	y	z	U(eq)
H(1A)	5418	3892	3809	25
H(3)	3574	2291	3876	28
H(4)	1735	1372	2737	30
H(5)	911	1616	797	29
H(6)	1908	2791	-67	26
H(13)	10393	6959	5905	27
H(14)	11590	6939	7824	30
H(16A)	12277	6221	9595	46
H(16B)	11648	5276	9694	46
H(16C)	10399	6035	9875	46
H(17)	9256	4728	8177	28
H(18)	8020	4745	6263	25
H(1)	4020(30)	4182(15)	490(20)	37(6)

Table S6. Torsion angles [°] for **9**.

C(9)-N(2)-N(3)-C(10)	-0.54(19)
C(10)-N(4)-N(5)-C(11)	-0.2(2)
C(9)-N(4)-N(5)-C(11)	176.04(18)
C(8)-C(1)-C(2)-C(3)	-179.03(19)
C(8)-C(1)-C(2)-C(7)	0.11(19)
C(7)-C(2)-C(3)-C(4)	-1.1(3)
C(1)-C(2)-C(3)-C(4)	177.92(18)
C(2)-C(3)-C(4)-C(5)	0.9(3)
C(3)-C(4)-C(5)-C(6)	0.1(3)
C(4)-C(5)-C(6)-C(7)	-0.8(3)
C(8)-N(1)-C(7)-C(6)	178.59(17)
C(8)-N(1)-C(7)-C(2)	-0.26(19)
C(5)-C(6)-C(7)-N(1)	-178.16(17)
C(5)-C(6)-C(7)-C(2)	0.5(2)
C(3)-C(2)-C(7)-N(1)	179.38(15)
C(1)-C(2)-C(7)-N(1)	0.09(19)
C(3)-C(2)-C(7)-C(6)	0.4(3)
C(1)-C(2)-C(7)-C(6)	-178.87(15)
C(2)-C(1)-C(8)-N(1)	-0.27(19)
C(2)-C(1)-C(8)-C(9)	179.45(16)
C(7)-N(1)-C(8)-C(1)	0.34(19)
C(7)-N(1)-C(8)-C(9)	-179.42(15)
N(3)-N(2)-C(9)-N(4)	1.10(19)
N(3)-N(2)-C(9)-C(8)	-178.42(16)
C(10)-N(4)-C(9)-N(2)	-1.20(19)
N(5)-N(4)-C(9)-N(2)	-177.79(17)
C(10)-N(4)-C(9)-C(8)	178.33(16)
N(5)-N(4)-C(9)-C(8)	1.7(3)
C(1)-C(8)-C(9)-N(2)	179.14(18)
N(1)-C(8)-C(9)-N(2)	-1.2(3)
C(1)-C(8)-C(9)-N(4)	-0.3(3)
N(1)-C(8)-C(9)-N(4)	179.39(15)
N(2)-N(3)-C(10)-N(4)	-0.2(2)
N(2)-N(3)-C(10)-S(1)	177.12(17)
C(9)-N(4)-C(10)-N(3)	0.9(2)
N(5)-N(4)-C(10)-N(3)	178.15(15)
C(9)-N(4)-C(10)-S(1)	-177.25(11)
N(5)-N(4)-C(10)-S(1)	0.01(19)
C(11)-S(1)-C(10)-N(3)	-177.3(2)
C(11)-S(1)-C(10)-N(4)	0.14(13)
N(4)-N(5)-C(11)-C(12)	-178.31(15)
N(4)-N(5)-C(11)-S(1)	0.33(17)
C(10)-S(1)-C(11)-N(5)	-0.29(14)
C(10)-S(1)-C(11)-C(12)	178.39(14)
N(5)-C(11)-C(12)-C(18)	1.7(3)
S(1)-C(11)-C(12)-C(18)	-176.90(13)
N(5)-C(11)-C(12)-C(13)	-179.11(16)
S(1)-C(11)-C(12)-C(13)	2.3(2)
C(18)-C(12)-C(13)-C(14)	-1.1(3)
C(11)-C(12)-C(13)-C(14)	179.66(16)
C(12)-C(13)-C(14)-C(15)	0.5(3)

C(13)-C(14)-C(15)-C(17)	0.5(3)
C(13)-C(14)-C(15)-C(16)	-178.04(17)
C(14)-C(15)-C(17)-C(18)	-0.9(3)
C(16)-C(15)-C(17)-C(18)	177.65(16)
C(15)-C(17)-C(18)-C(12)	0.3(3)
C(13)-C(12)-C(18)-C(17)	0.7(3)
C(11)-C(12)-C(18)-C(17)	179.96(16)

Symmetry transformations used to generate equivalent atoms: