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The crystal structure of 4,4'-diselanediybis(8-(hexyloxy)-3,6-dimethyl-1-(piperidin-1-yl)isoquinoline-7-carbonitrile), $C_{46}H_{60}N_6O_2Se_2$

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Abstract

$C_{46}H_{60}N_6O_2Se_2$, triclinic, $P\bar{1}$ (no. 2), $a = 10.4701(7)$ Å, $b = 10.6331(7)$ Å, $c = 20.4665(14)$ Å, $\alpha = 92.079(2)^\circ$, $\beta = 97.937(2)^\circ$, $\gamma = 95.848(2)^\circ$, $V = 2242.0(3)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0487$, $wR_{ref}(F^2) = 0.1183$, $T = 293(2)$ K.

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The molecular structure is shown in the Figure (Hydrogen atoms are omitted for clarity). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Red prismatic
Size:	0.16 × 0.13 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.69 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	22,203, 8296, 0.046
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 5396
$N(param)_{refined}$:	566
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Source of material

The title compound was prepared from the reaction of an intermediate 8-(hexyloxy)-3,6-dimethyl-1-(piperidin-1-yl)

isoquinoline-7-carbonitrile, which was obtained according to the published method [5], and selenium dioxide. The mixture of the intermediate (1.0 mmol), selenium dioxide (3.0 mmol), and xylene (15.0 mL) was stirred for 12 h at 100 °C. After the reaction solution was cooled to room temperature, the mixture was extracted with dichloromethane. After removing the solvent under reduced pressure, the crude product was purified by column chromatography using petroleum ether and ethyl acetate as the eluent to obtain the target product in 62% yield as light yellow solid. Crystals were obtained by slow evaporation from a mixture of ethyl acetate and petroleum ether.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Se1	0.05978 (4)	0.62847 (4)	0.22989 (2)	0.05821 (15)
Se2	0.14856 (4)	0.70435 (4)	0.33767 (2)	0.05218 (15)
O1	0.5998 (3)	0.7304 (3)	0.09844 (13)	0.0533 (7)
O2	0.0805 (3)	0.1629 (3)	0.48349 (15)	0.0573 (8)
N1	0.3575 (3)	0.4411 (3)	0.17419 (16)	0.0492 (8)
N2	0.5986 (5)	1.0485 (4)	0.0715 (2)	0.0953 (15)
N3	0.5664 (3)	0.4950 (3)	0.15481 (16)	0.0480 (8)
N4	0.3563 (3)	0.4107 (3)	0.40291 (17)	0.0506 (9)
N5	−0.2423 (4)	0.1623 (4)	0.5104 (2)	0.0720 (12)
N6	0.3027 (3)	0.1998 (3)	0.41680 (17)	0.0489 (9)
C1	0.2430 (4)	0.4697 (4)	0.1904 (2)	0.0504 (10)
C2	0.2146 (4)	0.5927 (4)	0.19630 (19)	0.0462 (10)
C3	0.3005 (4)	0.6916 (4)	0.17560 (18)	0.0439 (10)
C4	0.2682 (4)	0.8173 (4)	0.1709 (2)	0.0523 (11)
H4	0.1929	0.8377	0.1862	0.063*
C5	0.3428 (4)	0.9096 (4)	0.1449 (2)	0.0551 (11)
C6	0.4563 (4)	0.8772 (4)	0.1215 (2)	0.0510 (11)
C7	0.4913 (4)	0.7559 (4)	0.12499 (19)	0.0455 (10)
C8	0.4181 (4)	0.6606 (4)	0.15508 (18)	0.0410 (9)
C9	0.4469 (4)	0.5304 (4)	0.16242 (18)	0.0446 (10)
C10	0.1545 (5)	0.3564 (4)	0.2048 (3)	0.0794 (16)
H10A	0.1802	0.2813	0.1852	0.119*
H10B	0.0668	0.3665	0.1865	0.119*
H10C	0.1601	0.3494	0.2517	0.119*
C11	0.3042 (5)	1.0423 (4)	0.1383 (3)	0.0821 (16)
H11A	0.3672	1.1007	0.1656	0.123*
H11B	0.2207	1.0460	0.1522	0.123*
H11C	0.3002	1.0644	0.0931	0.123*
C12	0.5374 (5)	0.9705 (5)	0.0926 (2)	0.0641 (13)
C13	0.6837 (4)	0.5644 (4)	0.1927 (2)	0.0578 (12)
H13A	0.6683	0.6515	0.2014	0.069*
H13B	0.7548	0.5649	0.1669	0.069*
C14	0.7201 (4)	0.5046 (4)	0.2567 (2)	0.0640 (13)
H14A	0.6524	0.5118	0.2841	0.077*
H14B	0.7996	0.5499	0.2797	0.077*
C15	0.7394 (5)	0.3659 (5)	0.2466 (2)	0.0744 (14)
H15A	0.8124	0.3584	0.2228	0.089*
H15B	0.7579	0.3291	0.2890	0.089*
C16	0.6175 (5)	0.2959 (4)	0.2076 (2)	0.0678 (13)
H16A	0.6323	0.2092	0.1976	0.081*
H16B	0.5473	0.2942	0.2341	0.081*
C17	0.5796 (4)	0.3597 (4)	0.1442 (2)	0.0571 (12)
H17A	0.6447	0.3506	0.1154	0.069*
H17B	0.4979	0.3174	0.1219	0.069*
C18	0.5761 (5)	0.6874 (6)	0.0303 (2)	0.0862 (15)
H18A	0.5501	0.7556	0.0030	0.103*
H18B	0.5070	0.6182	0.0233	0.103*
C19	0.6973 (6)	0.6443 (7)	0.0122 (3)	0.1076 (15)
H19A	0.7167	0.5717	0.0376	0.129*
H19B	0.6813	0.6155	−0.0340	0.129*
C20	0.8094 (6)	0.7342 (8)	0.0217 (3)	0.1279 (17)
H20A	0.8271	0.7632	0.0679	0.153*
H20B	0.7919	0.8068	−0.0041	0.153*
C21	0.9324 (7)	0.6796 (8)	0.0015 (4)	0.1472 (19)
H21A	0.9561	0.6139	0.0311	0.177*
H21B	0.9110	0.6407	−0.0428	0.177*

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C22	1.0402 (7)	0.7711 (9)	0.0031 (4)	0.164 (2)
H22A	1.0627	0.8087	0.0476	0.197*
H22B	1.0159	0.8377	−0.0258	0.197*
C23	1.1583 (7)	0.7192 (9)	−0.0177 (4)	0.174 (3)
H23A	1.1808	0.6506	0.0093	0.261*
H23B	1.2295	0.7849	−0.0125	0.261*
H23C	1.1395	0.6891	−0.0632	0.261*
C24	0.3215 (4)	0.5257 (4)	0.3856 (2)	0.0480 (10)
C25	0.1938 (4)	0.5506 (3)	0.37558 (19)	0.0426 (9)
C26	0.0974 (4)	0.4621 (3)	0.39697 (18)	0.0387 (9)
C27	−0.0306 (3)	0.4893 (4)	0.39952 (18)	0.0405 (9)
H27	−0.0567	0.5633	0.3817	0.049*
C28	−0.1181 (4)	0.4118 (4)	0.42706 (18)	0.0409 (9)
C29	−0.0766 (4)	0.3005 (4)	0.45485 (19)	0.0429 (9)
C30	0.0484 (4)	0.2695 (4)	0.45285 (19)	0.0439 (10)
C31	0.1357 (4)	0.3449 (3)	0.42020 (19)	0.0406 (9)
C32	0.2663 (4)	0.3192 (4)	0.41250 (19)	0.0434 (9)
C33	0.4337 (4)	0.6208 (4)	0.3765 (3)	0.0770 (16)
H33A	0.5131	0.5926	0.3970	0.116*
H33B	0.4224	0.7012	0.3966	0.116*
H33C	0.4370	0.6292	0.3302	0.116*
C34	−0.2535 (4)	0.4443 (4)	0.4304 (2)	0.0537 (11)
H34A	−0.3147	0.3819	0.4048	0.081*
H34B	−0.2631	0.5260	0.4131	0.081*
H34C	−0.2692	0.4457	0.4756	0.081*
C35	−0.1655 (4)	0.2218 (4)	0.4868 (2)	0.0519 (11)
C36	0.2207 (4)	0.0904 (4)	0.3827 (2)	0.0565 (12)
H36A	0.1322	0.1110	0.3727	0.068*
H36B	0.2208	0.0196	0.4113	0.068*
C37	0.2696 (5)	0.0533 (4)	0.3193 (2)	0.0663 (13)
H37A	0.2597	0.1204	0.2888	0.080*
H37B	0.2177	−0.0226	0.2991	0.080*
C38	0.4113 (5)	0.0290 (5)	0.3316 (3)	0.0789 (15)
H38A	0.4205	−0.0439	0.3583	0.095*
H38B	0.4413	0.0109	0.2898	0.095*
C39	0.4925 (4)	0.1444 (4)	0.3669 (2)	0.0682 (13)
H39A	0.5816	0.1261	0.3778	0.082*
H39B	0.4914	0.2146	0.3380	0.082*
C40	0.4403 (4)	0.1807 (4)	0.4294 (2)	0.0569 (11)
H40A	0.4519	0.1147	0.4605	0.068*
H40B	0.4895	0.2581	0.4494	0.068*
C41 ^a	0.1771 (9)	0.1764 (8)	0.5413 (4)	0.0840 (19)
H41A ^a	0.1965	0.2656	0.5546	0.101*
H41B ^a	0.2561	0.1463	0.5300	0.101*
C42 ^a	0.1385 (9)	0.1087 (8)	0.5963 (4)	0.0886 (18)
H42A ^a	0.1131	0.0204	0.5824	0.106*
H42B ^a	0.0638	0.1432	0.6102	0.106*
C43 ^a	0.2499 (9)	0.1175 (13)	0.6558 (5)	0.100 (2)
H43A ^a	0.3297	0.0987	0.6402	0.120*
H43B ^a	0.2636	0.2029	0.6755	0.120*
C44 ^b	0.2169 (12)	0.0248 (12)	0.7074 (6)	0.117 (2)
H44A ^b	0.1820	−0.0553	0.6844	0.141*
H44B ^b	0.2975	0.0107	0.7344	0.141*
C45 ^b	0.1298 (13)	0.0560 (14)	0.7504 (7)	0.125 (3)
H45A ^b	0.0472	0.0601	0.7229	0.150*
H45B ^b	0.1593	0.1417	0.7678	0.150*
C46 ^b	0.0999 (15)	−0.0211 (14)	0.8100 (6)	0.127 (4)

Table 2: (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H46A ^b	0.1382	−0.0993	0.8086	0.190*
H46B ^b	0.0077	−0.0389	0.8083	0.190*
H46C ^b	0.1352	0.0268	0.8503	0.190*
C41 ^c	0.0908 (17)	0.157 (3)	0.5515 (7)	0.097 (4)
H41C ^c	0.0276	0.0912	0.5624	0.117*
H41D ^c	0.0713	0.2372	0.5702	0.117*
C42 ^c	0.2265 (17)	0.131 (3)	0.5819 (9)	0.104 (4)
H42C ^c	0.2919	0.1973	0.5733	0.124*
H42D ^c	0.2477	0.0501	0.5656	0.124*
C43 ^c	0.213 (2)	0.130 (3)	0.6564 (11)	0.111 (4)
H43C ^c	0.3013	0.1300	0.6781	0.133*
H43D ^c	0.1888	0.2134	0.6665	0.133*
C44 ^d	0.1350 (16)	0.0450 (19)	0.6963 (8)	0.116 (4)
H44C ^d	0.1215	−0.0395	0.6753	0.140*
H44D ^d	0.0506	0.0750	0.6964	0.140*
C45 ^d	0.2002 (16)	0.0379 (19)	0.7688 (8)	0.122 (4)
H45C ^d	0.2839	0.0052	0.7727	0.146*
H45D ^d	0.2052	0.1166	0.7949	0.146*
C46 ^d	0.088 (2)	−0.061 (2)	0.7811 (10)	0.131 (5)
H46D ^d	0.0153	−0.0184	0.7904	0.196*
H46E ^d	0.1164	−0.1094	0.8180	0.196*
H46F ^d	0.0622	−0.1168	0.7425	0.196*

^aOccupancy: 0.7, ^bOccupancy: 0.6, ^cOccupancy: 0.3, ^dOccupancy: 0.4

Experimental details

H atoms bonded to C were positioned geometrically and refined using a riding model, with $U_{iso}(H) = 1.5 U_{eq}(C)$ for methyl H atoms and $1.2 U_{eq}(C)$ for all other H atoms.

Comment

Isoquinoline is an important structural motif in the construction of various compounds with biological activities in pharmaceutical chemistry [6, 7]. In recent years, some isoquinoline derivatives have been also found to be used as solid-state fluorescence sensors for pressure, organic steam, and acid gas [8–10]. It should be pointed out that intermolecular interactions and stacking arrangements in crystalline state are found to play an important role in the solid-state stimulus-responsive fluorescent materials [11]. Thus, the synthesis and crystal structures of novel isoquinoline derivatives have received increasing attention [12, 13].

In the title compound, the pyridine ring and benzene ring in isoquinoline unit are not completely coplanar because of the existence of the piperidine and hexyloxy groups, a finding which is similar to previous reports in [8, 9].

Furthermore, two isoquinoline rings in molecule form in a “Z” shape and thus no π - π stacking is formed. The molecules are stabilized by a weak C36–H36B...N5 (2.564 Å) hydrogen bond, a C34–H34B...H41A (2.299 Å) interaction, an a weak C37–H37B...Se2 (3.078 Å) bond. The interaction between the N atom on the cyano unit and Se2 results in a distance of 3.346 Å. As a result, the molecules form a zigzag stacking mode with a reverse parallel arrangement between the upper and lower levels.

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