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N′-[(1*E*)-(4–Fluorophenyl)methylidene] adamantane-1-carbohydrazide, $C_{18}H_{21}FN_2O$

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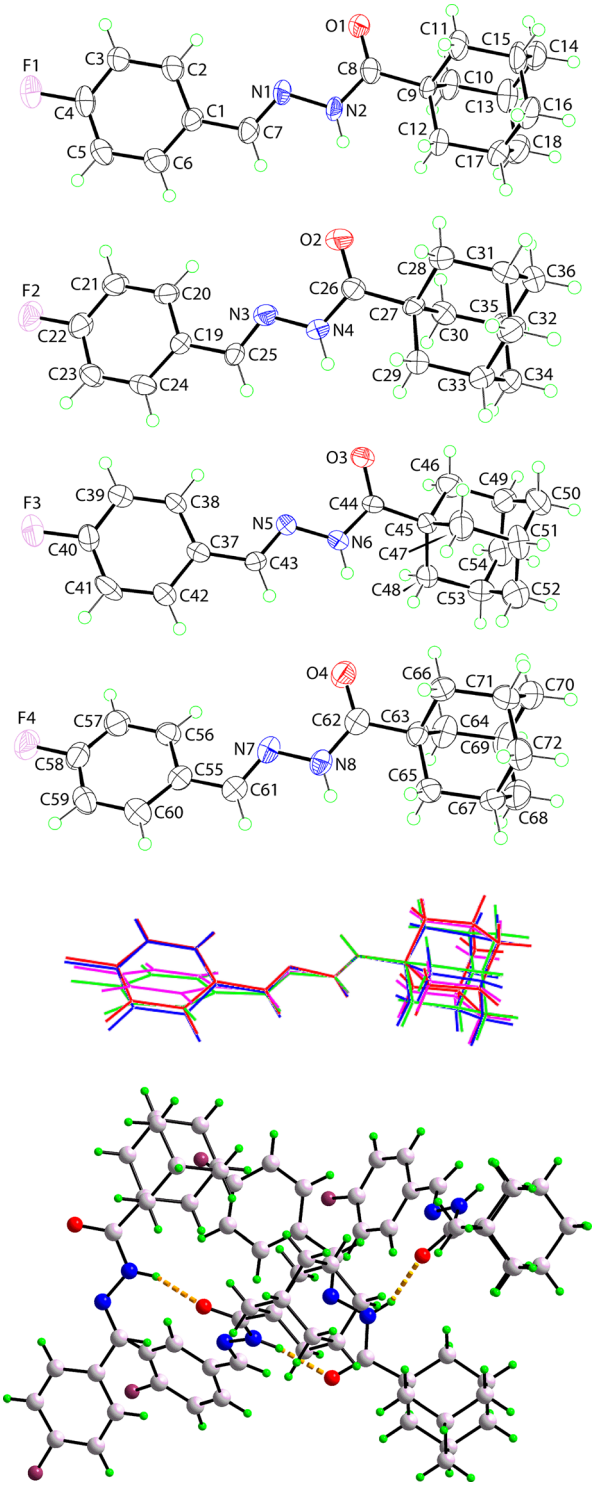
Abstract

$C_{18}H_{21}FN_2O$, triclinic, $P\bar{1}$ (no. 2), a = 10.6377(5) Å, b = 14.4869(7) Å, c = 21.0557(13) Å, α = 98.583(5)°, β = 91.979(4)°, γ = 90.101(4)°, V = 3206.5(3) Å^3, Z = 8, $R_{gt}(F)$ = 0.0815, w$R_{ref}(F^2)$ = 0.1930, T = 172 K.

CCDC no.: 2269765.

The crystallographic images is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.



1 Source of material

To a solution of adamantane-1-carbohydrazide (1.94 g, 0.01 mol), in ethanol (10 mL), 4-fluorobenzaldehyde (1.24 g, 0.01 mol) was added, after which the mixture was heated under reflux for 1 h. On cooling, the separated crude product was filtered, washed with cold ethanol, dried and recrystallised from ethanol to yield 2.76 g (92 %) of the title compound as colourless prisms [5]. **M.pt** 476–478 K. **^{1}H NMR (DMSO-d_6, 500.13 MHz)**: δ 1.66–1.69 (m, 6H, adamantane–H), 1.88 (s, 6H, adamantane–H), 2.01 (s, 3H, adamantane–H), 5.67 (s, 1H, NH), 7.27–7.29 (m, 2H, Ar–H), 7.85–7.87 (m, 2H, Ar–H),

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Table 1: Data collection and handling.

Crystal:	Prism, colourless
Size:	0.34 × 0.10 × 0.08 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	0.69 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω-scans
θ_{max}, completeness:	67.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	30,239, 11417, 0.110
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4950
$N(param)_{refined}$:	794
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], *Win*GX and ORTEP [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
F1	−0.1538 (3)	0.4631 (2)	0.55040 (16)	0.0597 (9)
O1	0.5599 (3)	0.6578 (2)	0.71639 (17)	0.0415 (8)
N1	0.3204 (4)	0.6887 (3)	0.68507 (18)	0.0338 (9)
N2	0.3985 (3)	0.7607 (3)	0.71546 (18)	0.0336 (9)
H2N	0.369822	0.817346	0.727422	0.040*
C1	0.1120 (4)	0.6462 (3)	0.6448 (2)	0.0350 (11)
C2	0.1443 (5)	0.5555 (4)	0.6194 (3)	0.0444 (13)
H2	0.228494	0.535208	0.624113	0.053*
C3	0.0548 (5)	0.4944 (4)	0.5871 (3)	0.0443 (13)
H3	0.076772	0.432475	0.569632	0.053*
C4	−0.0653 (5)	0.5250 (4)	0.5812 (3)	0.0456 (14)
C5	−0.1020 (5)	0.6127 (4)	0.6045 (3)	0.0575 (16)
H5	−0.186767	0.631560	0.599507	0.069*
C6	−0.0118 (5)	0.6740 (4)	0.6358 (3)	0.0548 (15)
H6	−0.035062	0.736249	0.651573	0.066*
C7	0.2038 (4)	0.7130 (4)	0.6777 (2)	0.0384 (12)
H7	0.178392	0.774609	0.693797	0.046*
C8	0.5212 (4)	0.7388 (4)	0.7256 (2)	0.0361 (11)
C9	0.6111 (4)	0.8197 (3)	0.7512 (2)	0.0314 (10)
C10	0.6512 (5)	0.8112 (4)	0.8213 (2)	0.0453 (14)
H10A	0.576701	0.819112	0.848521	0.054*
H10B	0.685875	0.748318	0.823221	0.054*
C11	0.7307 (5)	0.8067 (4)	0.7115 (2)	0.0458 (14)
H11A	0.766350	0.744110	0.713743	0.055*
H11B	0.708961	0.810751	0.665882	0.055*
C12	0.5585 (5)	0.9170 (3)	0.7501 (3)	0.0466 (14)
H12A	0.483706	0.925778	0.777056	0.056*
H12B	0.532086	0.924228	0.705622	0.056*
C13	0.7508 (5)	0.8857 (4)	0.8474 (3)	0.0525 (16)
H13	0.775708	0.878971	0.892762	0.063*
C14	0.8651 (5)	0.8715 (4)	0.8059 (3)	0.0545 (15)
H14A	0.930832	0.918032	0.822764	0.065*
H14B	0.899862	0.808439	0.807347	0.065*
C15	0.8286 (5)	0.8819 (4)	0.7374 (3)	0.0523 (16)
H15	0.904661	0.873521	0.710560	0.063*
C16	0.7749 (5)	0.9777 (4)	0.7351 (3)	0.0521 (15)
H16A	0.751900	0.984757	0.690169	0.063*
H16B	0.838849	1.025882	0.751673	0.063*
C17	0.6590 (5)	0.9911 (4)	0.7758 (3)	0.0526 (15)
H17	0.623854	1.054664	0.774022	0.063*
C18	0.6963 (6)	0.9824 (4)	0.8455 (3)	0.0577 (17)
H18A	0.759537	1.030861	0.862512	0.069*
H18B	0.621631	0.991431	0.872523	0.069*
F2	0.4861 (3)	0.2190 (2)	0.40972 (13)	0.0562 (9)
O2	0.2780 (3)	0.4090 (2)	0.79129 (16)	0.0453 (9)
N3	0.3941 (4)	0.4404 (3)	0.68572 (18)	0.0363 (10)
N4	0.3993 (4)	0.5034 (3)	0.74254 (18)	0.0352 (9)
H4N	0.443069	0.555589	0.745206	0.042*
C19	0.4600 (4)	0.4001 (3)	0.5785 (2)	0.0327 (10)
C20	0.3698 (5)	0.3302 (3)	0.5614 (2)	0.0394 (12)
H20	0.301924	0.324673	0.588767	0.047*
C21	0.3786 (5)	0.2684 (4)	0.5043 (2)	0.0451 (13)
H21	0.319606	0.218929	0.492938	0.054*
C22	0.4750 (5)	0.2815 (4)	0.4654 (2)	0.0442 (13)
C23	0.5626 (5)	0.3507 (4)	0.4782 (2)	0.0413 (12)
H23	0.626912	0.357340	0.448995	0.050*
C24	0.5550 (4)	0.4112 (4)	0.5356 (2)	0.0395 (12)
H24	0.614572	0.460531	0.545947	0.047*
C25	0.4598 (4)	0.4620 (3)	0.6406 (2)	0.0314 (10)
H25	0.508376	0.517895	0.646951	0.038*
C26	0.3365 (4)	0.4835 (3)	0.7935 (2)	0.0344 (11)
C27	0.3371 (4)	0.5577 (3)	0.8539 (2)	0.0288 (10)
C28	0.3065 (5)	0.5090 (3)	0.9113 (2)	0.0427 (13)
H28A	0.371876	0.462166	0.917227	0.051*
H28B	0.224562	0.476048	0.903018	0.051*
C29	0.4640 (4)	0.6093 (3)	0.8669 (2)	0.0377 (12)
H29A	0.483979	0.640871	0.829884	0.045*
H29B	0.531617	0.564059	0.872605	0.045*
C30	0.2350 (5)	0.6292 (4)	0.8445 (2)	0.0420 (12)
H30A	0.152904	0.597040	0.834757	0.050*
H30B	0.255068	0.661473	0.807772	0.050*
C31	0.3009 (5)	0.5813 (4)	0.9722 (2)	0.0453 (13)
H31	0.282260	0.548928	1.009629	0.054*
C32	0.4269 (5)	0.6327 (4)	0.9849 (2)	0.0464 (14)
H32A	0.423166	0.678900	1.024537	0.056*
H32B	0.494267	0.587651	0.991403	0.056*
C33	0.4569 (5)	0.6824 (3)	0.9284 (2)	0.0381 (12)
H33	0.539374	0.715926	0.937068	0.046*
C34	0.3535 (5)	0.7519 (3)	0.9191 (2)	0.0417 (12)
H34A	0.372730	0.784598	0.882437	0.050*
H34B	0.349223	0.799138	0.958085	0.050*
C35	0.2271 (5)	0.7011 (4)	0.9060 (2)	0.0445 (13)
H35	0.159639	0.747312	0.899909	0.053*
C36	0.1964 (5)	0.6510 (4)	0.9632 (3)	0.0509 (15)
H36A	0.190143	0.697098	1.002666	0.061*
H36B	0.114669	0.617823	0.954745	0.061*
F3	0.6689 (3)	−0.0236 (2)	0.55387 (16)	0.0611 (9)
O3	−0.0344 (3)	0.1633 (2)	0.71402 (16)	0.0394 (8)
N5	0.2065 (4)	0.1920 (3)	0.68900 (18)	0.0347 (9)
N6	0.1310 (3)	0.2625 (3)	0.71977 (18)	0.0346 (9)
H6N	0.162725	0.318169	0.734047	0.041*
C37	0.4112 (4)	0.1494 (3)	0.6539 (2)	0.0357 (11)
C38	0.3746 (4)	0.0629 (3)	0.6197 (2)	0.0375 (12)
H38	0.289247	0.043395	0.619680	0.045*
C39	0.4607 (5)	0.0049 (4)	0.5858 (3)	0.0439 (13)

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H39	0.435489	−0.054108	0.562931	0.053*
C40	0.5817 (5)	0.0345 (4)	0.5862 (2)	0.0422 (12)
C41	0.6240 (5)	0.1189 (4)	0.6192 (3)	0.0545 (16)
H41	0.709782	0.137259	0.618955	0.065*
C42	0.5366 (5)	0.1759 (4)	0.6525 (3)	0.0464 (13)
H42	0.563029	0.234767	0.675167	0.056*
C43	0.3224 (4)	0.2131 (3)	0.6878 (2)	0.0340 (11)
H43	0.351481	0.271870	0.709599	0.041*
C44	0.0088 (4)	0.2439 (3)	0.7272 (2)	0.0313 (10)
C45	−0.0728 (4)	0.3251 (3)	0.7566 (2)	0.0353 (11)
C46	−0.2089 (5)	0.3056 (4)	0.7343 (3)	0.0538 (15)
H46A	−0.234899	0.243994	0.744633	0.065*
H46B	−0.216850	0.303299	0.687116	0.065*
C47	−0.0616 (6)	0.3288 (4)	0.8302 (3)	0.0538 (15)
H47A	0.026748	0.341641	0.845297	0.065*
H47B	−0.086967	0.267935	0.841884	0.065*
C48	−0.0328 (5)	0.4207 (4)	0.7412 (3)	0.0464 (13)
H48A	−0.035179	0.419665	0.694089	0.056*
H48B	0.054587	0.434740	0.757567	0.056*
C49	−0.2971 (5)	0.3826 (4)	0.7675 (3)	0.0556 (16)
H49	−0.386386	0.369073	0.752636	0.067*
C50	−0.2827 (6)	0.3834 (4)	0.8404 (3)	0.0632 (18)
H50A	−0.340666	0.429812	0.862788	0.076*
H50B	−0.304900	0.321250	0.851020	0.076*
C51	−0.1490 (5)	0.4079 (4)	0.8629 (3)	0.0566 (16)
H51	−0.141069	0.411402	0.910627	0.068*
C52	−0.1104 (6)	0.4999 (4)	0.8442 (3)	0.0612 (17)
H52A	−0.022352	0.514413	0.859248	0.073*
H52B	−0.164547	0.549990	0.865460	0.073*
C53	−0.1215 (5)	0.4973 (4)	0.7725 (3)	0.0542 (15)
H53	−0.095974	0.559159	0.761252	0.065*
C54	−0.2543 (5)	0.4752 (4)	0.7493 (3)	0.0545 (15)
H54A	−0.260463	0.472072	0.702019	0.065*
H54B	−0.310227	0.525645	0.768450	0.065*
F4	−0.0334 (3)	0.2765 (2)	0.59721 (14)	0.0620 (10)
O4	−0.2531 (3)	0.0961 (2)	0.21522 (16)	0.0445 (9)
N7	−0.1316 (4)	0.0564 (3)	0.31933 (17)	0.0338 (9)
N8	−0.1292 (4)	−0.0034 (3)	0.26168 (17)	0.0353 (9)
H8N	−0.086685	−0.055885	0.258265	0.042*
C55	−0.0573 (4)	0.0947 (3)	0.4269 (2)	0.0338 (11)
C56	−0.1545 (5)	0.1577 (4)	0.4445 (2)	0.0392 (12)
H56	−0.226360	0.158710	0.416539	0.047*
C57	−0.1471 (5)	0.2185 (4)	0.5020 (2)	0.0472 (14)
H57	−0.212765	0.261185	0.514017	0.057*
C58	−0.0425 (6)	0.2148 (4)	0.5406 (2)	0.0467 (14)
C59	0.0536 (5)	0.1543 (4)	0.5278 (2)	0.0438 (13)
H59	0.123491	0.153642	0.557042	0.053*
C60	0.0457 (5)	0.0926 (4)	0.4695 (2)	0.0418 (13)
H60	0.111185	0.048986	0.459014	0.050*
C61	−0.0590 (4)	0.0340 (3)	0.3650 (2)	0.0366 (11)
H61	−0.008234	−0.020246	0.358522	0.044*
C62	−0.1945 (4)	0.0213 (4)	0.2109 (2)	0.0365 (11)
C63	−0.2025 (5)	−0.0462 (3)	0.1476 (2)	0.0350 (11)
C64	−0.3339 (5)	−0.0928 (4)	0.1427 (3)	0.0501 (15)
H64A	−0.399754	−0.044305	0.146757	0.060*
H64B	−0.342415	−0.130242	0.178002	0.060*

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C65	−0.1029 (5)	−0.1222 (4)	0.1413 (2)	0.0470 (13)
H65A	−0.110106	−0.159662	0.176705	0.056*
H65B	−0.018200	−0.093131	0.144877	0.056*
C66	−0.1897 (5)	0.0107 (3)	0.0924 (2)	0.0418 (12)
H66A	−0.105297	0.040275	0.095110	0.050*
H66B	−0.253397	0.060732	0.095813	0.050*
C67	−0.1193 (5)	−0.1866 (4)	0.0761 (2)	0.0478 (14)
H67	−0.053373	−0.236015	0.072460	0.057*
C68	−0.2499 (6)	−0.2316 (4)	0.0720 (3)	0.0626 (18)
H68A	−0.261531	−0.273771	0.030629	0.075*
H68B	−0.257917	−0.269200	0.107228	0.075*
C69	−0.3506 (5)	−0.1566 (4)	0.0770 (3)	0.0540 (16)
H69	−0.435700	−0.186759	0.073771	0.065*
C70	−0.3386 (5)	−0.0978 (4)	0.0231 (3)	0.0545 (15)
H70A	−0.351713	−0.137758	−0.019077	0.065*
H70B	−0.403482	−0.048600	0.026890	0.065*
C71	−0.2081 (5)	−0.0534 (4)	0.0276 (2)	0.0462 (13)
H71	−0.200366	−0.015371	−0.008019	0.055*
C72	−0.1084 (5)	−0.1282 (4)	0.0213 (2)	0.0502 (14)
H72A	−0.023922	−0.098968	0.023304	0.060*
H72B	−0.119693	−0.168947	−0.020677	0.060*

[a]Occupancy: 0.854 (7).

8.25 (s, 1H, CH=N). **^{13}C NMR (DMSO-d_6, 125.76 MHz)**: δ 27.45, 34.57, 36.01, 39.95 (adamantane–C), 114.25, 128.85, 131.0, 166.53 (Ar–C), 144.67 (CH=N), 177.06 (C=O).

2 Experimental details

The C-bound H atoms were geometrically placed (C–H = 0.95–1.00 Å) and refined as riding with U_{iso}(H) = 1.2 U_{eq}(C). The N-bound H atoms were located in a difference map and refined with N–H = 0.88 Å and with U_{iso}(H) = 1.2 U_{eq}(N). Owing to poor agreement, one reflection, i.e. (2 13 5), was omitted from the final cycles of refinement.

Due to the weak diffracting crystals only about 43 % of the measured reflections are observed (see Table 2). This fact is expressed by an enlarged R factor for all reflections (0.1715). Nevertheless the refinement was convergent and all geometric parameters as well as displacement ellipsoids are all in the plausible ranges. Also the completeness in the choosen 2θ range is near 99 %. Thus we don't have any doubts concerning the chemical and crystallographic plausability of the title structure.

3 Discussion

Hydrazide and hydrazone derivatives have proved to possess desirable chemotherapeutic activities [6, 7]. In the

same way, the adamantane cage was identified some time ago as being an essential building block in various chemotherapeutic agents [8, 9]. In the present study, details of the X-ray structure of the title adamantane-1-carohydrazide-hydrazone analogue, hereafter (I), which was originally prepared as a crucial intermediate [5] for the synthesis of potential chemotherapeutic agents, are described.

There are four independent molecules comprising the asymmetric-unit of (I) and the molecular structures of these are shown in the upper four images of the figure (50 % probability ellipsoids). The molecules comprise an amide residue, with an anti-conformation, connected, via the carbon atom to an adamantanyl residue and via an imine bond to a 4-fluorophenyl ring. The overlay diagram shown in the figure indicates two distinct conformations; the molecules were overlaid so the CNO atoms of the amide residue are coincident; the red, green, blue and pink images represent the O1- to O4-containing molecules, respectively. With respect to the overlay of the fluorophenyl rings, it is evident the O1- and O3-containing molecules adopt similar conformations but distinct to those for the inverted–O2- and O4-molecules. However, there other twists in the molecule indicating there is no clear correlation between the dihedral angles between the least-squares planes through the CNO and C_6 atoms, i.e. 12.4(4), 19.8(6), 16.6(5) and 27.0(5)° for the O1- to O4-molecules, respectively. While the C7–N1–N2–C8 torsion angle of 178.8(4)° [the equivalent angles for the O2-, O3- and O4-molecules are –175.3(4), –175.3(4) and –174.1(4)°, respectively] and N2–N1–C7–C1 torsion angle of 177.9(4)° [–179.7(4), –175.9(4) and 179.9(3)°, respectively] are consistent with more or less planar, all-trans conformations, significant conformational differences arise from twists about the imine–C–C(fluorophenyl) bonds, in the O2- and O4-molecules [15.4(8) and 19.3(7)°] compared with effectively co-planar arrangements for the O1- and O3-molecules [–1.0(7) and 1.4(8)°]. The solution NMR results (see Experimental) confirm these differences are due to solid-state effects and are not chemically significant.

There are two closely related derivatives in the literature which differ in their substitution patterns in the phenyl ring, namely, the 2–PPh_2 [10] and 2,6-dichloro [11] derivatives, hereafter (II) and (III), respectively. These present the same anti-conformation for the amide residue. The dihedral angles between the least-squares planes through the CNO and C_6 atoms of 17.20(13) and 48.97(15)° for (II) and (III), respectively, highlight the rather different conformation adopted in the solid-state by (III) compared with (I) and (II).

The anti-conformation adopted by each of the four independent molecules of (I) facilitates the formation of conventional amide–N–H···O(amide) hydrogen bonds [N2–H2n···O4^{i}: H2N···O4^{i} = 2.06 Å, N2···O4^{i} = 2.844(5) Å with angle at H2n = 148°; N4–H4n···O1: H4n···O1 = 2.10 Å, N4···O1 = 2.940(5) Å with angle at H4n = 159°; N6–H6n···O2: H6n···O2 = 2.03 Å, N6···O2 = 2.844(5) Å with angle at H6n = 154°; N8–H8n···O3^{ii}: H8n···O3^{ii} = 2.16 Å, N8···O3^{ii} = 2.994(5) Å with angle at H8n = 159° for symmetry operations (i): $-x, 1-y, 1-z$ and (ii): $-x, -y, 1-z$] between all four independent molecules leading to a twisted supramolecular chain along the *b*-axis as illustrated in the lower image of the figure (hydrogen bonds are represented by orange dashed lines). A myriad of other directional contacts are evident, e.g. fluorophenyl–C–H···F, imine- and methylene–C–H···O(amide) and imine–C–H···N(imine). These serve to connect the supramolecular chains into double-layers with the adamantanyl residues projecting to either side. The layers stack along the c-axis with the adamantanyl residues inter-digitating along this axis.

Previous work has proven systematic analyses of the calculated Hirshfeld surfaces for the individual molecules of crystals with multiple molecules in the asymmetric-unit can provide further evidence for the assigned crystallographic symmetry [12]. Thus, the calculation of the Hirshfeld surfaces and fingerprint plots was undertaken by employing established procedures [13] and CrystalExplorer [14]. Clear distinctions are noted in the percentage contributions to the calculated Hirshfeld surfaces, commencing with H···H surface contacts which are computed to be 54.5, 61.6, 55.1 and 61.9 % for the O1–O4-molecules, respectively, compared to 64.7 % calculated for the entire asymmetric-unit. The overall contacts involving H atoms are 94.9, 97.2, 96.2 and 96.7 %, respectively, with the major differences involving C···H/H···C [15.3, 12.9, 14.8 and 12.7 %], F···H/H···F [9.9, 7.7, 10.7 and 7.8 %] and O···H/H···O [8.7, 8.3, 9.1 and 7.9 %] contacts. Differences are also noted in F···C/C···F surface contacts, i.e. 2.4, 0.4, 1.5 and 0.7 %, respectively. All other contacts, i.e. F···N/N···F, F···O/O···F, F···F and N···N, contribute less than 1.0 % to the individual surfaces.

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