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Crystal structure of the possible sulindac impurity 2-(5-fluoro-2-methyl-1*H*-inden-3-yl)acetonitrile

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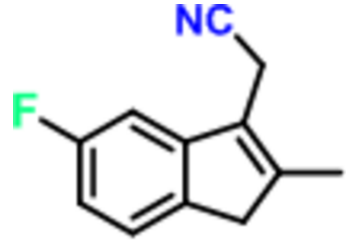
The title compound, $C_{12}H_{10}FN$, was identified as a possible critical degradation impurity of sulindac, a therapeutic COX-2 inhibitor for rheumatoid arthritis. Single-crystal X-ray analysis revealed two conformationally slightly different molecules in the asymmetric unit ($Z' = 2$), each containing an indene ring system. In the crystal structure, [100] chains formed through C—H···N interactions are connected into a tri-periodic supramolecular structure by further C—H···F and C—H···π interactions through $P2_1/n$ symmetry operations.

1. Chemical context

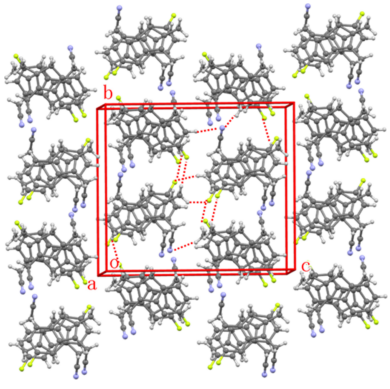
Sulindac {systematic name [(*Z*)-2-methyl-1-[(4-methylsulfinylphenyl)methylene]-5-fluoro-1*H*-inden-3-acetic acid]} is a nonsteroidal anti-inflammatory drug (NSAID) that exhibits selective cyclooxygenase-2 (COX-2) inhibitory activity, effectively suppressing COX-2 overexpression through competitive inhibition. It is applied for clinical management of rheumatoid arthritis and degenerative joint disorders (Boolbol, 1996).

While impurity profiling represents a critical component of pharmaceutical development, the present investigation focuses on the characterization of a key process-related impurity compound in sulindac synthesis, namely 2-(5-fluoro-2-methyl-1*H*-inden-3-yl)acetonitrile (**1**). Based on single-crystal X-ray diffraction analysis, we have now unambiguously determined its configuration, representing novel structural data in pharmaceutical crystallography and report the results here.



2. Structural commentary

The asymmetric unit of (**1**) comprises two molecules and is illustrated in Fig. 1*a*; an overlay plot of the two molecules (one of which is inverted relative to the other) is shown in Fig. 1*b*. The root-mean-square-deviation between the two molecules is only 0.009 Å, with d_{max} of 0.020 Å between N1 and N2. The angles C7—C10—C11 and C19—C22—C23 are 112.47 (10) and 112.65 (10)°, respectively (Fig. 2*a*), and the torsion angles between the cyanide group and its corresponding indene ring



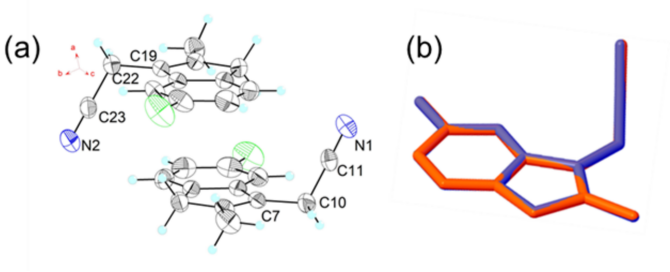


Figure 1
(*a*) The asymmetric unit of (**1**) with displacement ellipsoids drawn at the 50% probability level; (*b*) overlay plot of the two independent molecules.

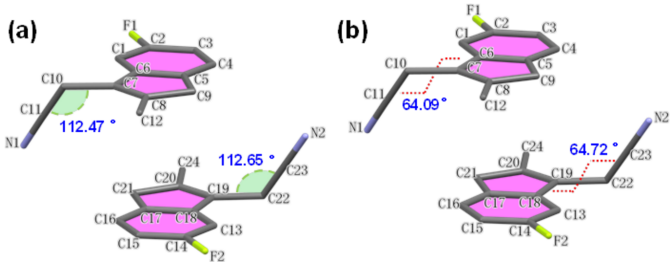


Figure 2
(*a*) The angle between the cyanide group and adjacent atoms in each of the two independent molecules; (*b*) the cyanide group and its dihedral angle with the corresponding indene ring plane in each of the two independent molecules.

plane is 64.09 (16) and 64.72 (14)° in the two independent molecules (Fig. 2*b*). The bond lengths in the two molecules are all within normal range.

3. Supramolecular features

As shown in Fig. 3, the methylene groups (C10, C22) attached to the —C≡N moiety act as hydrogen-bond donors to the cyanide N atoms (N2, N1) of adjacent molecules. These interactions (Table 1) link the molecules into an infinite supramolecular chain extending parallel to [100]. Other interactions shown in Fig. 3 include C—H···F interactions (Table 1) as well as C—H···π interactions [C22–H22*A*···π (2.780 Å) and C12—H12*B*···π (2.958 Å)], which connect the molecules into a tri-periodic supramolecular structure. A packing plot of (**1**) is shown in Fig. 4.

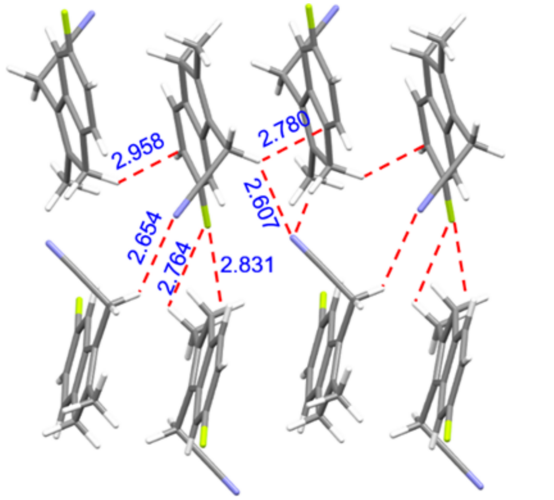


Figure 3
Intermolecular interactions (C—H···N, C—H···F and C—H···π) between adjacent molecules shown as red dashed lines. Color codes: C (gray), N (blue), F(green) and H (white).

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*B*···N2^{i}	0.99	2.65	3.3978 (18)	132
C22—H22*A*···N1^{ii}	0.99	2.61	3.4010 (16)	137
C24—H24*C*···F2^{iii}	0.98	2.76	3.227 (2)	110
C24—H24*B*···F2^{iii}	0.98	2.83	3.227 (2)	105

Symmetry codes: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

4. Database survey

A search of the Cambridge Structural Database (CSD, version 2024.1.0; Groom *et al.*, 2016) was conducted using the keyword methylindene, which retrieved eleven relevant entries: BANNUA (Tsuno *et al.*, 2003), CIRMIA (Santi *et al.*, 2007), DOBVIZ (Biali & Rappoport, 1986), FUNPAG (Xu *et al.*, 2010), HEXRAD (Bonifaci *et al.*, 1994), ICEXOD (Halterman *et al.*, 2000), SUZYER (Stenzel *et al.*, 2001), MIJKIZ (Enders *et al.*, 2002), NOYTOK (Brase *et al.*, 1998), XAWFEG (Shapiro *et al.*, 1999), and RESZEU (Herrmann *et al.*, 1997). The primary distinction among these compounds lies in the substitution patterns of the methyl group on the indene ring. Notably, an indene derivative bearing both

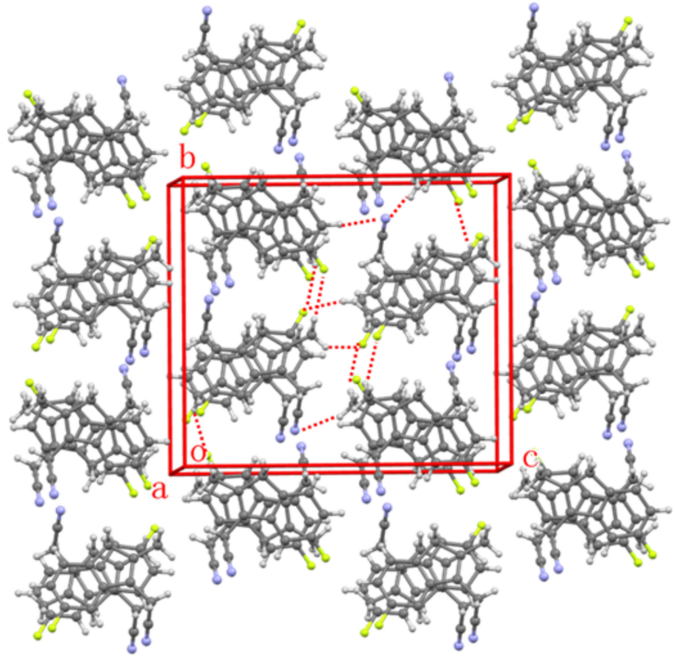


Figure 4
Packing plot of (**1**) approximately along [100]. C—H···N and C—H···F interactions are shown as red dotted lines.

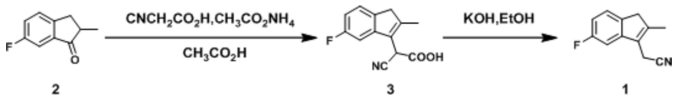


Figure 5
Synthesis scheme of the title compound (**1**).

fluorine and cyano substituents has been obtained and reported exclusively in the present work. This comparative analysis underscores the structural novelty of the title compound, particularly its unique combination of hydrogen-bonding patterns (C—H···N and C—H···F).

5. Synthesis and crystallization

Compound (**1**) was prepared according to a literature method (Xu *et al.*, 2020; Dai *et al.*, 2009). The preparation procedure is schematically shown in Fig. 5. A 100 ml round-bottomed flask equipped with a magnetic stirring bar was charged with a mixture of 6-fluoro-2-methyl-2,3-dihydro-1*H*-inden-1-one, (**2**), (20.35 g, 124 mmol), cyanoacetic acid (13.65 g, 160.4 mmol), acetic acid (7.8 g, 129.9 mmol) and ammonium acetate (3.1 g, 40.2 mmol) in toluene (50 ml). The mixture was refluxed for 24 h and then cooled to room temperature. The solvent was removed *in vacuo*, and the residue (**3**) was dissolved in ethanol without further purification. The resulting solution was added to a potassium hydroxide solution (165 g, 25%$_{wt}$, *w*/*w*, 735.2 mmol) and heated to reflux for 13 h. Then, the ethanol was removed *in vacuo*, followed by the addition of 300 ml of water. The pH value was adjusted to 8 using concentrated hydrochloric acid, and the mixture was then extracted with dichloromethane (150 ml). The aqueous layer was collected and further adjusted to a pH value of 2 with concentrated hydrochloric acid, resulting in the precipitation of a significant amount of a yellow solid of (**1**). The solid was then filtered off, washed with water, and dried in air. Yield: 17.25 g, 85.2%. ^{1}H NMR spectrum (Varian Unity Inova 500 MHz, DMSO-d_6, ppm): δ: 7.38 (*dd*, 1H, J_1 = 5.55 Hz, J_2 = 7.85 Hz), 7.23 (*dd*, 1H, J_1 = 2.2 Hz, J_2 = 9.5 Hz), 6.94 (*m*, 1H), 3.87 (*s*, 2H), 3.35 (*s*, 2H), 2.13 (*s*, 3H). Single crystals were obtained by slow evaporation of a saturated solution of (**1**) in a dichloromethane–ethanol mixture (4:1, *v*/*v*) at room temperature over 20 d.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms attached to carbon atoms were placed in calculated positions and constrained with AFIX instructions.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{10}FN$
M_r	187.21
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	170
a, *b*, *c* (Å)	7.5248 (5), 15.1897 (10), 17.1823 (9)
β (°)	99.176 (2)
V (Å^3)	1938.8 (2)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.42 × 0.29 × 0.23
Data collection	
Diffractometer	Bruker *SMART APEX* CCD area detector
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.654, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	28954, 4137, 3530
R_{int}	0.031
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.634
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.105, 1.03
No. of reflections	4137
No. of parameters	256
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.21

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

7. Authorship contribution statement

The submitted manuscript includes contributions from eight authors. Yun-Deng Wu conceptualized and designed the research framework, conducted comprehensive data analysis, and drafted the manuscript. Yun-Deng Wu, Hui Wan, Yun Xia and Jie Ni performed the synthesis, isolation, purification, and characterization of the title compound. Jian Li, Hui Zhang, Xiangyang Xu and Jun Xie contributed to the single-crystal cultivation and associated experimental evaluations. All co-authors participated in the critical revision and final approval of the manuscript for publication.

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supporting information

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Computing details

2-(5-Fluoro-2-methyl-1*H*-inden-3-yl)acetonitrile

Crystal data

$C_{12}H_{10}FN$
M_r = 187.21
Monoclinic, $P2_1/n$
a = 7.5248 (5) Å
b = 15.1897 (10) Å
c = 17.1823 (9) Å
β = 99.176 (2)°
V = 1938.8 (2) Å^3
Z = 8
$F(000)$ = 784
D_x = 1.283 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9932 reflections
θ = 2.7–26.7°
μ = 0.09 mm^{-1}
T = 170 K
Block, colourless
0.42 × 0.29 × 0.23 mm

Data collection

Bruker SMART APEX CCD area detector diffractometer
Radiation source: sealed tube
Graphite monochromator
Detector resolution: 8 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.654, T_{max} = 0.745
28954 measured reflections
4137 independent reflections
3530 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 26.8°, θ_{min} = 2.4°
h = −9→9
k = −19→19
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.03
4137 reflections
256 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.5292P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: SHELXL (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0048 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	0.08006 (14)	0.18981 (6)	0.05253 (6)	0.0683 (3)
N1	0.46276 (16)	0.13422 (7)	0.35764 (7)	0.0451 (3)
C1	0.16171 (16)	0.24962 (8)	0.17953 (8)	0.0373 (3)
H1	0.142597	0.194447	0.202895	0.045*
C2	0.13519 (18)	0.26100 (9)	0.09871 (8)	0.0446 (3)
C3	0.16163 (18)	0.33907 (10)	0.06189 (8)	0.0468 (3)
H3	0.141755	0.343036	0.006001	0.056*
C4	0.21793 (17)	0.41208 (9)	0.10788 (7)	0.0403 (3)
H4	0.236765	0.466889	0.083839	0.048*
C5	0.24625 (15)	0.40411 (8)	0.18899 (7)	0.0314 (3)
C6	0.21782 (14)	0.32325 (7)	0.22468 (7)	0.0296 (2)
C7	0.25771 (15)	0.33531 (8)	0.31024 (7)	0.0308 (3)
C8	0.30813 (16)	0.41860 (8)	0.32741 (7)	0.0343 (3)
C9	0.30565 (16)	0.47028 (8)	0.25250 (7)	0.0356 (3)
H9A	0.219788	0.519996	0.249653	0.043*
H9B	0.426803	0.493474	0.248492	0.043*
C10	0.23961 (19)	0.26298 (8)	0.36817 (8)	0.0410 (3)
H10A	0.262345	0.287413	0.422263	0.049*
H10B	0.114754	0.240222	0.358457	0.049*
C11	0.36465 (16)	0.19013 (8)	0.36265 (7)	0.0340 (3)
C12	0.3611 (2)	0.46070 (10)	0.40633 (8)	0.0503 (4)
H12A	0.282934	0.511371	0.411000	0.075*
H12B	0.486549	0.480414	0.411934	0.075*
H12C	0.348725	0.417899	0.447807	0.075*
F2	0.92640 (14)	0.55350 (6)	0.40592 (5)	0.0692 (3)
N2	0.52928 (17)	0.59111 (8)	0.10051 (8)	0.0555 (3)
C13	0.83947 (16)	0.48567 (8)	0.28300 (7)	0.0366 (3)
H13	0.857248	0.539097	0.256548	0.044*
C14	0.86929 (19)	0.47951 (9)	0.36423 (8)	0.0450 (3)
C15	0.84553 (19)	0.40399 (10)	0.40560 (8)	0.0465 (3)
H15	0.868240	0.403684	0.461595	0.056*
C16	0.78762 (17)	0.32816 (9)	0.36380 (7)	0.0394 (3)
H16	0.769749	0.275137	0.390833	0.047*
C17	0.75646 (15)	0.33098 (7)	0.28246 (7)	0.0310 (3)
C18	0.78197 (14)	0.40920 (7)	0.24219 (7)	0.0292 (2)
C19	0.73809 (14)	0.39234 (7)	0.15742 (6)	0.0280 (2)
C20	0.68786 (15)	0.30797 (7)	0.14500 (7)	0.0302 (2)
C21	0.69555 (16)	0.26085 (7)	0.22262 (7)	0.0328 (3)
H21A	0.575673	0.237782	0.229008	0.039*

H21B	0.782483	0.211482	0.227027	0.039*
C22	0.75293 (16)	0.46131 (7)	0.09589 (7)	0.0336 (3)
H22A	0.877380	0.484612	0.103829	0.040*
H22B	0.729198	0.433713	0.043056	0.040*
C23	0.62711 (17)	0.53441 (8)	0.09855 (7)	0.0370 (3)
C24	0.63087 (19)	0.26147 (8)	0.06865 (8)	0.0416 (3)
H24A	0.626613	0.303465	0.025088	0.062*
H24B	0.511239	0.235723	0.068025	0.062*
H24C	0.717319	0.214674	0.062657	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0791 (7)	0.0627 (6)	0.0581 (6)	0.0011 (5)	−0.0044 (5)	−0.0283 (5)
N1	0.0494 (6)	0.0335 (6)	0.0496 (7)	0.0066 (5)	−0.0003 (5)	0.0027 (5)
C1	0.0356 (6)	0.0331 (6)	0.0431 (7)	0.0031 (5)	0.0060 (5)	−0.0015 (5)
C2	0.0405 (7)	0.0498 (8)	0.0415 (7)	0.0044 (6)	0.0007 (5)	−0.0147 (6)
C3	0.0435 (7)	0.0669 (9)	0.0291 (6)	0.0092 (6)	0.0035 (5)	0.0015 (6)
C4	0.0367 (6)	0.0489 (7)	0.0356 (6)	0.0039 (5)	0.0066 (5)	0.0114 (5)
C5	0.0265 (5)	0.0343 (6)	0.0335 (6)	0.0033 (4)	0.0055 (4)	0.0064 (5)
C6	0.0259 (5)	0.0309 (6)	0.0323 (6)	0.0046 (4)	0.0059 (4)	0.0026 (4)
C7	0.0315 (6)	0.0314 (6)	0.0306 (6)	0.0080 (4)	0.0078 (4)	0.0047 (4)
C8	0.0336 (6)	0.0342 (6)	0.0342 (6)	0.0071 (5)	0.0025 (5)	0.0010 (5)
C9	0.0348 (6)	0.0300 (6)	0.0410 (7)	0.0001 (5)	0.0034 (5)	0.0050 (5)
C10	0.0522 (8)	0.0365 (7)	0.0376 (7)	0.0112 (6)	0.0172 (6)	0.0102 (5)
C11	0.0418 (6)	0.0298 (6)	0.0284 (6)	−0.0004 (5)	−0.0001 (5)	0.0046 (4)
C12	0.0586 (9)	0.0478 (8)	0.0411 (7)	0.0075 (7)	−0.0023 (6)	−0.0091 (6)
F2	0.0937 (7)	0.0482 (5)	0.0558 (5)	0.0070 (5)	−0.0178 (5)	−0.0238 (4)
N2	0.0527 (7)	0.0394 (6)	0.0758 (9)	0.0120 (6)	0.0146 (6)	0.0182 (6)
C13	0.0369 (6)	0.0294 (6)	0.0410 (7)	0.0045 (5)	−0.0011 (5)	−0.0025 (5)
C14	0.0485 (8)	0.0396 (7)	0.0426 (7)	0.0086 (6)	−0.0061 (6)	−0.0145 (6)
C15	0.0524 (8)	0.0559 (8)	0.0294 (6)	0.0127 (6)	0.0012 (6)	−0.0034 (6)
C16	0.0413 (7)	0.0441 (7)	0.0334 (6)	0.0068 (5)	0.0079 (5)	0.0059 (5)
C17	0.0291 (5)	0.0316 (6)	0.0330 (6)	0.0046 (4)	0.0067 (4)	0.0021 (5)
C18	0.0257 (5)	0.0289 (6)	0.0325 (6)	0.0048 (4)	0.0033 (4)	0.0001 (4)
C19	0.0267 (5)	0.0268 (5)	0.0305 (6)	0.0035 (4)	0.0051 (4)	0.0015 (4)
C20	0.0302 (5)	0.0282 (6)	0.0328 (6)	0.0022 (4)	0.0066 (4)	0.0000 (4)
C21	0.0356 (6)	0.0273 (6)	0.0363 (6)	−0.0003 (4)	0.0083 (5)	0.0032 (5)
C22	0.0369 (6)	0.0295 (6)	0.0349 (6)	0.0014 (5)	0.0069 (5)	0.0043 (5)
C23	0.0374 (6)	0.0312 (6)	0.0416 (7)	−0.0018 (5)	0.0040 (5)	0.0100 (5)
C24	0.0536 (8)	0.0328 (6)	0.0378 (7)	0.0006 (5)	0.0052 (6)	−0.0049 (5)

Geometric parameters (Å, º)

F1—C2	1.3660 (15)	F2—C14	1.3648 (15)
N1—C11	1.1375 (16)	N2—C23	1.1370 (17)
C1—H1	0.9500	C13—H13	0.9500
C1—C2	1.3820 (19)	C13—C14	1.3810 (18)

C1—C6	1.3880 (17)	C13—C18	1.3898 (16)
C2—C3	1.373 (2)	C14—C15	1.376 (2)
C3—H3	0.9500	C15—H15	0.9500
C3—C4	1.388 (2)	C15—C16	1.3903 (19)
C4—H4	0.9500	C16—H16	0.9500
C4—C5	1.3813 (17)	C16—C17	1.3804 (17)
C5—C6	1.4044 (16)	C17—C18	1.4035 (16)
C5—C9	1.4983 (17)	C17—C21	1.5006 (16)
C6—C7	1.4643 (16)	C18—C19	1.4639 (15)
C7—C8	1.3402 (17)	C19—C20	1.3434 (16)
C7—C10	1.5032 (16)	C19—C22	1.5051 (15)
C8—C9	1.5051 (16)	C20—C21	1.5065 (16)
C8—C12	1.4947 (17)	C20—C24	1.4916 (17)
C9—H9A	0.9900	C21—H21A	0.9900
C9—H9B	0.9900	C21—H21B	0.9900
C10—H10A	0.9900	C22—H22A	0.9900
C10—H10B	0.9900	C22—H22B	0.9900
C10—C11	1.4654 (17)	C22—C23	1.4646 (17)
C12—H12A	0.9800	C24—H24A	0.9800
C12—H12B	0.9800	C24—H24B	0.9800
C12—H12C	0.9800	C24—H24C	0.9800

C2—C1—H1	121.8	C14—C13—H13	121.9
C2—C1—C6	116.30 (12)	C14—C13—C18	116.15 (12)
C6—C1—H1	121.8	C18—C13—H13	121.9
F1—C2—C1	117.80 (13)	F2—C14—C13	117.48 (13)
F1—C2—C3	117.96 (12)	F2—C14—C15	118.12 (12)
C3—C2—C1	124.24 (12)	C15—C14—C13	124.40 (12)
C2—C3—H3	120.6	C14—C15—H15	120.7
C2—C3—C4	118.76 (12)	C14—C15—C16	118.66 (12)
C4—C3—H3	120.6	C16—C15—H15	120.7
C3—C4—H4	120.4	C15—C16—H16	120.5
C5—C4—C3	119.23 (12)	C17—C16—C15	119.08 (12)
C5—C4—H4	120.4	C17—C16—H16	120.5
C4—C5—C6	120.50 (12)	C16—C17—C18	120.72 (11)
C4—C5—C9	131.01 (11)	C16—C17—C21	130.98 (11)
C6—C5—C9	108.49 (10)	C18—C17—C21	108.30 (10)
C1—C6—C5	120.97 (11)	C13—C18—C17	120.99 (11)
C1—C6—C7	131.10 (11)	C13—C18—C19	130.62 (11)
C5—C6—C7	107.93 (10)	C17—C18—C19	108.40 (10)
C6—C7—C10	123.20 (11)	C18—C19—C22	123.15 (10)
C8—C7—C6	110.16 (10)	C20—C19—C18	109.75 (10)
C8—C7—C10	126.63 (11)	C20—C19—C22	127.09 (10)
C7—C8—C9	109.85 (10)	C19—C20—C21	110.04 (10)
C7—C8—C12	128.96 (12)	C19—C20—C24	128.72 (11)
C12—C8—C9	121.19 (11)	C24—C20—C21	121.24 (10)
C5—C9—C8	103.57 (9)	C17—C21—C20	103.51 (9)
C5—C9—H9A	111.0	C17—C21—H21A	111.1

C5—C9—H9B	111.0	C17—C21—H21B	111.1
C8—C9—H9A	111.0	C20—C21—H21A	111.1
C8—C9—H9B	111.0	C20—C21—H21B	111.1
H9A—C9—H9B	109.0	H21A—C21—H21B	109.0
C7—C10—H10A	109.1	C19—C22—H22A	109.1
C7—C10—H10B	109.1	C19—C22—H22B	109.1
H10A—C10—H10B	107.8	H22A—C22—H22B	107.8
C11—C10—C7	112.47 (10)	C23—C22—C19	112.65 (10)
C11—C10—H10A	109.1	C23—C22—H22A	109.1
C11—C10—H10B	109.1	C23—C22—H22B	109.1
N1—C11—C10	179.06 (14)	N2—C23—C22	179.90 (19)
C8—C12—H12A	109.5	C20—C24—H24A	109.5
C8—C12—H12B	109.5	C20—C24—H24B	109.5
C8—C12—H12C	109.5	C20—C24—H24C	109.5
H12A—C12—H12B	109.5	H24A—C24—H24B	109.5
H12A—C12—H12C	109.5	H24A—C24—H24C	109.5
H12B—C12—H12C	109.5	H24B—C24—H24C	109.5

F1—C2—C3—C4	−179.90 (11)	F2—C14—C15—C16	−179.92 (12)
C1—C2—C3—C4	−0.3 (2)	C13—C14—C15—C16	0.0 (2)
C1—C6—C7—C8	179.91 (12)	C13—C18—C19—C20	−179.93 (12)
C1—C6—C7—C10	−1.04 (19)	C13—C18—C19—C22	1.14 (18)
C2—C1—C6—C5	−0.34 (17)	C14—C13—C18—C17	0.04 (17)
C2—C1—C6—C7	179.76 (11)	C14—C13—C18—C19	179.88 (11)
C2—C3—C4—C5	0.22 (19)	C14—C15—C16—C17	0.2 (2)
C3—C4—C5—C6	−0.25 (18)	C15—C16—C17—C18	−0.24 (18)
C3—C4—C5—C9	−179.87 (12)	C15—C16—C17—C21	179.58 (12)
C4—C5—C6—C1	0.32 (17)	C16—C17—C18—C13	0.13 (17)
C4—C5—C6—C7	−179.76 (10)	C16—C17—C18—C19	−179.75 (10)
C4—C5—C9—C8	179.75 (12)	C16—C17—C21—C20	179.62 (12)
C5—C6—C7—C8	0.00 (13)	C17—C18—C19—C20	−0.07 (13)
C5—C6—C7—C10	179.05 (10)	C17—C18—C19—C22	−179.00 (10)
C6—C1—C2—F1	179.96 (11)	C18—C13—C14—F2	179.80 (11)
C6—C1—C2—C3	0.3 (2)	C18—C13—C14—C15	−0.1 (2)
C6—C5—C9—C8	0.10 (12)	C18—C17—C21—C20	−0.54 (12)
C6—C7—C8—C9	0.06 (13)	C18—C19—C20—C21	−0.29 (13)
C6—C7—C8—C12	179.99 (12)	C18—C19—C20—C24	179.74 (11)
C6—C7—C10—C11	64.09 (16)	C18—C19—C22—C23	−64.72 (14)
C7—C8—C9—C5	−0.10 (13)	C19—C20—C21—C17	0.51 (12)
C8—C7—C10—C11	−117.02 (13)	C20—C19—C22—C23	116.54 (13)
C9—C5—C6—C1	−179.98 (10)	C21—C17—C18—C13	−179.73 (10)
C9—C5—C6—C7	−0.06 (12)	C21—C17—C18—C19	0.40 (12)
C10—C7—C8—C9	−178.95 (11)	C22—C19—C20—C21	178.59 (11)
C10—C7—C8—C12	1.0 (2)	C22—C19—C20—C24	−1.4 (2)
C12—C8—C9—C5	179.97 (11)	C24—C20—C21—C17	−179.52 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*B*···N2^{i}	0.99	2.65	3.3978 (18)	132
C22—H22*A*···N1^{ii}	0.99	2.61	3.4010 (16)	137
C24—H24*C*···F2^{iii}	0.98	2.76	3.227 (2)	110
C24—H24*B*···F2^{iii}	0.98	2.83	3.227 (2)	105

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+1/2; (ii) −*x*+3/2, *y*+1/2, −*z*+1/2; (iii) −*x*+3/2, *y*−1/2, −*z*+1/2.