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4-(2,4-Difluorophenyl)-2-(*N*-methylcarbamoyl)phenyl acetate

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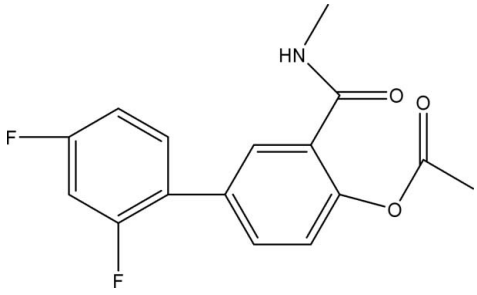
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.130; data-to-parameter ratio = 17.0.

In the title compound, $C_{16}H_{13}F_2NO_3$, the two benzene rings and all attached non-H atoms are indivdually planar. The two planes, *viz*. the benzene ring and the two substituent F atoms (*A*), and the benzene ring and the attached carbonyl C atom, oxy O atom and aromatic C atom (*B*), have largest deviations of −0.0278 (10) and 0.0272 (9) Å, respectively, and a dihedral angle between the planes of 37.78 (3)°. The dihedral angles between plane *B* and the methylaminocarbonyl and acetoxy groups are 61.83 (5) and 48.82 (8)°, respectively.

Related literature

For related literature, see: Hannah & Ruyle (1978); Rao & Hu (2005).



Experimental

Crystal data

$C_{16}H_{13}F_2NO_3$
M_r = 305.27
Monoclinic, $P2_1/c$
a = 9.8972 (5) Å
b = 19.9625 (11) Å
c = 7.4737 (4) Å
β = 107.180 (2)°
V = 1410.72 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
0.25 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.971, T_{max} = 0.976
18835 measured reflections
3441 independent reflections
2741 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
S = 1.05
3441 reflections
202 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2190).

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supplementary materials

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4-(2,4-Difluorophenyl)-2-(*N*-methylcarbamoyl)phenyl acetate

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Comment

Diflunisal is a analgesic and non-steroidal anti-inflammatory drug. (Hannah & Ruyle, 1978). In a continuation of our work on the structure–activity relationship of diflunisal derivatives, we have obtained a colorless crystalline compound that was the product of the reaction of acetyl chloride, monomethylamine and diflunisal. The structural identity of our product, (I), was resolved using single-crystal X-ray diffraction.

The molecular structure of (I) is illustrated in Fig. 1. Atoms F1, F2, C5, C7, C8, C9, C10, C11 and C12 are coplanar, the largest deviation being −0.0278 (10) Å for C5. Atoms O1, C1, C2, C3, C4, C5, C6, C7 and C15 are coplanar, the largest deviation being 0.0272 (9) Å for C7. The dihedral angles between the two planes is 37.78 (3)°. The dihedral angles between the O1/C1–C7/C15 plane and the O3/C15/N1/C16 and O1/O2/C13/C14 planes are 61.83 (5) and 48.82 (8)°, respectively.

Experimental

The title compound was prepared from acetyl chloride, monomethylamine and diflunisal, according to the procedure of Rao & Hu (2005).

Refinement

H atoms were added at calculated positions and refined using a riding model. H atoms were given isotropic displacement parameters equal to 1.2(or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameters of their parent atoms and C—H distances were restrained to 0.93 Å for those bonded to phenyl ring and 0.96 Å for those bonded to methyl. N—H distance was restrained to 0.86 Å.

Figures

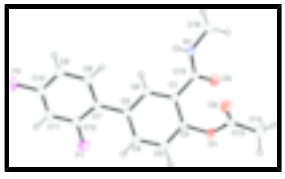


Fig. 1. The structure of (I), shown with 30% probability displacement ellipsoids.

4-(2,4-Difluorophenyl)-2-(*N*-methylcarbamoyl)phenyl acetate

Crystal data

$C_{16}H_{13}F_2NO_3$	F_{000} = 632
M_r = 305.27	D_x = 1.437 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation

	λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 9.8972 (5) Å	θ = 9.9–13.9º
b = 19.9625 (11) Å	μ = 0.12 mm^{-1}
c = 7.4737 (4) Å	T = 298 (2) K
β = 107.180 (2)º	Prismatic, colourless
V = 1410.72 (13) $Å^3$	0.25 × 0.20 × 0.20 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.024
Radiation source: fine-focus sealed tube	θ_{max} = 28.3º
Monochromator: graphite	θ_{min} = 2.0º
T = 298(2) K	h = −13→12
ω/2θ scans	k = −25→25
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	l = −9→9
T_{min} = 0.971, T_{max} = 0.976	3 standard reflections
18835 measured reflections	every 60 min
3441 independent reflections	intensity decay: 0.3%
2741 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2 + 0.396P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.130	$(\Delta/\sigma)_{max} < 0.001$
S = 1.05	$\Delta\rho_{max}$ = 0.30 e $Å^{-3}$
3441 reflections	$\Delta\rho_{min}$ = −0.22 e $Å^{-3}$
202 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.010 (2)
Secondary atom site location: difference Fourier map	

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2sigma(F^2)$ is used only for calculat-

ing *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	0.32311 (11)	0.74045 (5)	0.49721 (19)	0.0707 (4)
F2	0.76499 (10)	0.83535 (6)	0.5573 (2)	0.0824 (4)
O1	−0.26535 (10)	0.87205 (5)	0.07940 (16)	0.0442 (3)
O2	−0.25482 (12)	0.94857 (6)	−0.13686 (17)	0.0528 (3)
O3	−0.21960 (11)	0.99622 (6)	0.27918 (18)	0.0510 (3)
N1	−0.03370 (13)	1.04551 (6)	0.21903 (19)	0.0418 (3)
H1	0.0471	1.0401	0.1998	0.050*
C1	−0.03767 (13)	0.92526 (7)	0.23466 (19)	0.0339 (3)
C2	−0.11773 (14)	0.86970 (7)	0.1527 (2)	0.0382 (3)
C3	−0.05561 (16)	0.80854 (8)	0.1458 (2)	0.0468 (4)
H3	−0.1109	0.7722	0.0903	0.056*
C4	0.08875 (15)	0.80110 (7)	0.2211 (2)	0.0431 (3)
H4	0.1300	0.7595	0.2167	0.052*
C5	0.17344 (14)	0.85508 (7)	0.30349 (19)	0.0341 (3)
C6	0.10768 (13)	0.91656 (7)	0.30923 (18)	0.0331 (3)
H6	0.1629	0.9530	0.3648	0.040*
C7	0.32962 (14)	0.84948 (7)	0.37554 (19)	0.0346 (3)
C8	0.41649 (15)	0.90169 (8)	0.3515 (2)	0.0406 (3)
H8	0.3745	0.9407	0.2925	0.049*
C9	0.56228 (16)	0.89772 (9)	0.4118 (2)	0.0482 (4)
H9	0.6180	0.9332	0.3943	0.058*
C10	0.62193 (16)	0.84022 (9)	0.4978 (3)	0.0511 (4)
C11	0.54416 (17)	0.78679 (9)	0.5286 (3)	0.0524 (4)
H11	0.5873	0.7482	0.5889	0.063*
C12	0.39927 (16)	0.79306 (8)	0.4659 (2)	0.0432 (3)
C13	−0.32361 (15)	0.91518 (7)	−0.0630 (2)	0.0406 (3)
C14	−0.48084 (16)	0.91338 (10)	−0.1116 (3)	0.0536 (4)
H14A	−0.5095	0.9279	−0.0059	0.080*
H14B	−0.5135	0.8685	−0.1445	0.080*
H14C	−0.5208	0.9426	−0.2158	0.080*
C15	−0.10582 (14)	0.99183 (7)	0.2462 (2)	0.0353 (3)
C16	−0.08854 (18)	1.11262 (8)	0.2203 (3)	0.0502 (4)
H16A	−0.0248	1.1442	0.1914	0.075*
H16B	−0.0978	1.1223	0.3420	0.075*
H16C	−0.1795	1.1159	0.1283	0.075*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0535 (6)	0.0495 (6)	0.1028 (9)	−0.0014 (5)	0.0133 (6)	0.0329 (6)
F2	0.0277 (5)	0.0760 (8)	0.1328 (11)	0.0125 (5)	0.0070 (6)	−0.0009 (8)
O1	0.0253 (5)	0.0412 (6)	0.0621 (7)	−0.0064 (4)	0.0067 (4)	0.0045 (5)

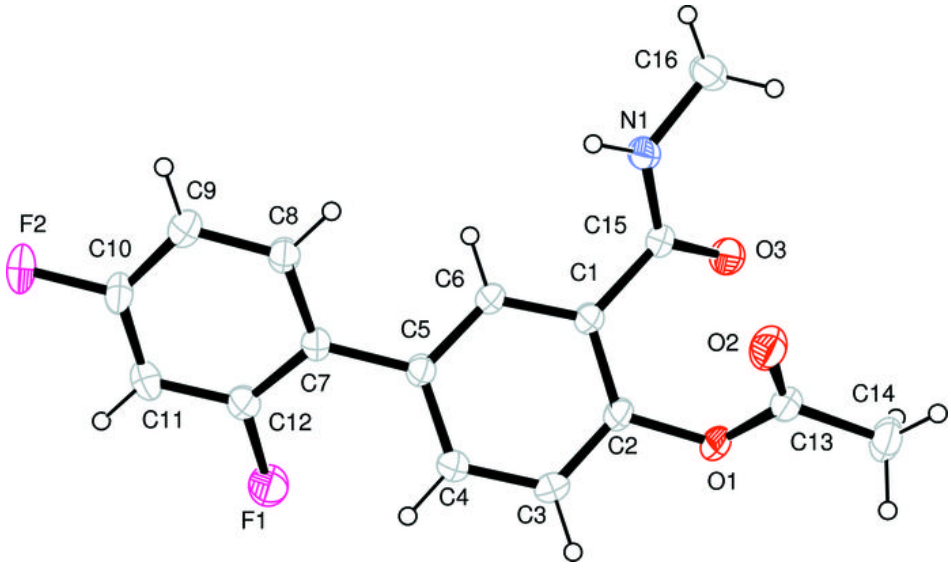
O2	0.0378 (6)	0.0616 (7)	0.0559 (7)	−0.0040 (5)	0.0091 (5)	0.0097 (5)
O3	0.0343 (6)	0.0470 (6)	0.0782 (8)	−0.0002 (4)	0.0267 (5)	−0.0036 (6)
N1	0.0324 (6)	0.0342 (6)	0.0618 (8)	0.0017 (5)	0.0186 (5)	0.0021 (5)
C1	0.0278 (6)	0.0332 (7)	0.0409 (7)	−0.0013 (5)	0.0108 (5)	0.0023 (5)
C2	0.0250 (6)	0.0373 (7)	0.0507 (8)	−0.0041 (5)	0.0089 (6)	0.0048 (6)
C3	0.0356 (8)	0.0331 (7)	0.0679 (10)	−0.0085 (6)	0.0093 (7)	−0.0020 (7)
C4	0.0352 (7)	0.0308 (7)	0.0618 (9)	0.0001 (6)	0.0121 (7)	0.0022 (6)
C5	0.0284 (6)	0.0345 (7)	0.0393 (7)	0.0003 (5)	0.0096 (5)	0.0047 (5)
C6	0.0274 (6)	0.0331 (7)	0.0383 (6)	−0.0030 (5)	0.0090 (5)	0.0006 (5)
C7	0.0293 (7)	0.0355 (7)	0.0384 (7)	0.0023 (5)	0.0092 (5)	0.0011 (5)
C8	0.0319 (7)	0.0390 (8)	0.0500 (8)	0.0016 (6)	0.0108 (6)	0.0076 (6)
C9	0.0328 (7)	0.0477 (9)	0.0644 (10)	−0.0034 (6)	0.0145 (7)	0.0022 (7)
C10	0.0266 (7)	0.0557 (10)	0.0673 (10)	0.0074 (6)	0.0082 (7)	−0.0046 (8)
C11	0.0409 (8)	0.0442 (9)	0.0653 (10)	0.0139 (7)	0.0053 (7)	0.0051 (8)
C12	0.0384 (8)	0.0369 (8)	0.0524 (8)	0.0018 (6)	0.0107 (6)	0.0070 (6)
C13	0.0311 (7)	0.0405 (8)	0.0470 (7)	−0.0013 (6)	0.0064 (6)	−0.0051 (6)
C14	0.0299 (8)	0.0669 (11)	0.0591 (10)	0.0022 (7)	0.0057 (7)	−0.0045 (8)
C15	0.0271 (6)	0.0366 (7)	0.0406 (7)	−0.0003 (5)	0.0076 (5)	−0.0001 (5)
C16	0.0493 (9)	0.0363 (8)	0.0674 (10)	0.0049 (7)	0.0208 (8)	0.0040 (7)

Geometric parameters (Å, °)

F1—C12	1.3529 (18)	C5—C7	1.4833 (18)
F2—C10	1.3565 (17)	C6—H6	0.9300
O1—C13	1.3579 (19)	C7—C12	1.387 (2)
O1—C2	1.4014 (16)	C7—C8	1.3961 (19)
O2—C13	1.1978 (18)	C8—C9	1.381 (2)
O3—C15	1.2248 (17)	C8—H8	0.9300
N1—C15	1.3361 (18)	C9—C10	1.362 (2)
N1—C16	1.4464 (19)	C9—H9	0.9300
N1—H1	0.8599	C10—C11	1.374 (3)
C1—C6	1.3916 (18)	C11—C12	1.376 (2)
C1—C2	1.3951 (19)	C11—H11	0.9300
C1—C15	1.5042 (19)	C13—C14	1.490 (2)
C2—C3	1.375 (2)	C14—H14A	0.9600
C3—C4	1.380 (2)	C14—H14B	0.9600
C3—H3	0.9300	C14—H14C	0.9600
C4—C5	1.392 (2)	C16—H16A	0.9600
C4—H4	0.9300	C16—H16B	0.9600
C5—C6	1.3959 (19)	C16—H16C	0.9600
C13—O1—C2	118.69 (11)	C10—C9—H9	121.0
C15—N1—C16	121.57 (12)	C8—C9—H9	121.0
C15—N1—H1	119.2	F2—C10—C9	118.69 (15)
C16—N1—H1	119.2	F2—C10—C11	118.15 (15)
C6—C1—C2	117.55 (12)	C9—C10—C11	123.16 (14)
C6—C1—C15	121.01 (12)	C10—C11—C12	116.82 (15)
C2—C1—C15	121.42 (12)	C10—C11—H11	121.6
C3—C2—C1	121.34 (13)	C12—C11—H11	121.6
C3—C2—O1	116.18 (12)	F1—C12—C11	116.62 (13)

C1—C2—O1	122.44 (12)	F1—C12—C7	119.49 (13)
C2—C3—C4	120.06 (13)	C11—C12—C7	123.89 (14)
C2—C3—H3	120.0	O2—C13—O1	123.12 (13)
C4—C3—H3	120.0	O2—C13—C14	126.56 (15)
C3—C4—C5	120.82 (13)	O1—C13—C14	110.31 (13)
C3—C4—H4	119.6	C13—C14—H14A	109.5
C5—C4—H4	119.6	C13—C14—H14B	109.5
C4—C5—C6	117.97 (12)	H14A—C14—H14B	109.5
C4—C5—C7	121.94 (12)	C13—C14—H14C	109.5
C6—C5—C7	120.03 (12)	H14A—C14—H14C	109.5
C1—C6—C5	122.25 (12)	H14B—C14—H14C	109.5
C1—C6—H6	118.9	O3—C15—N1	122.50 (13)
C5—C6—H6	118.9	O3—C15—C1	121.96 (12)
C12—C7—C8	115.62 (13)	N1—C15—C1	115.53 (11)
C12—C7—C5	123.54 (12)	N1—C16—H16A	109.5
C8—C7—C5	120.83 (12)	N1—C16—H16B	109.5
C9—C8—C7	122.60 (14)	H16A—C16—H16B	109.5
C9—C8—H8	118.7	N1—C16—H16C	109.5
C7—C8—H8	118.7	H16A—C16—H16C	109.5
C10—C9—C8	117.90 (15)	H16B—C16—H16C	109.5

Fig. 1


C16
N1
C9
C8
F2
C10
C15
O3
C6
C1
C5
O2
C7
C14
C11
C12
C2
C13
O1
C4
C3
F1