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2-[(1*S*,3*S*)-3-Acetyl-2,2-dimethylcyclobutyl]-*N*-(2,6-difluorophenyl)acetamide

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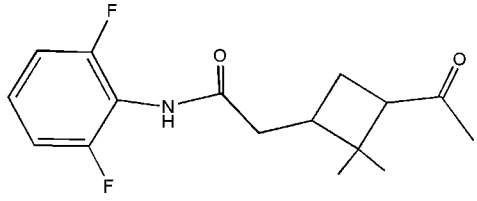
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.069; wR factor = 0.207; data-to-parameter ratio = 15.9.

The title compound, $C_{16}H_{19}F_2NO_2$, was synthesized from 2,6-difluorobenzenamine and 2-(3-acetyl-2,2-dimethylcyclobutyl)-acetyl chloride, which was obtained through the reaction of 2-(3-acetyl-2,2-dimethylcyclobutyl)acetic acid (pinonic acid) and thionyl chloride. The crystal structure involves N—H···O hydrogen bonds.

Related literature

For related literature, see: Moglioni *et al.* (2000); Ribas *et al.* (1980); Wolk & Goldschmidt (1986).



Experimental

Crystal data

$C_{16}H_{19}F_2NO_2$
M_r = 295.32
Monoclinic, $P2_1/n$
a = 8.7840 (18) Å
b = 12.914 (3) Å
c = 13.747 (3) Å
β = 99.20 (3)°
V = 1539.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.40 × 0.20 × 0.10 mm

Data collection

Nonius CAD4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.931, T_{max} = 0.960
3222 measured reflections
3022 independent reflections
1682 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.207
S = 1.01
3022 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H0A···O1^{i}	0.86	2.12	2.949 (5)	161

Symmetry code: (i) $-x+1, -y+1, -z+2$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; 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: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2381).

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supplementary materials

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2-[(1*S*,3*S*)-3-Acetyl-2,2-dimethylcyclobutyl]-*N*-(2,6-difluorophenyl)acetamide

Y. Yin, C. Han, Z. Song and Z. Wang

Comment

The cyclobutane moiety is a structural feature present in several natural or designed products with interesting biological properties (Moglioni *et al.*, 2000). Some researchers have synthesized many bioactive substances, such as sex pheromones (Wolk *et al.*, 1986), juvenile hormones (Ribas *et al.*, 1980) *etc*. Thus, using pinonic acid as a starting material, we may synthesize new cyclobutane derivatives with biological properties. We have synthesized the title compound and report here its crystal structure.

The molecular structure is shown in Fig. 1 and the crystal packing in Fig. 2.

Experimental

The title compound was synthesized from 2,6-difluorobenzenamine and 2-(3-acetyl-2,2-dimethylcyclobutyl) acetyl chloride at room temperature·The acetyl chloride was obtained using -(3-acetyl-2,2-dimethylcyclobutyl)acetic acid (pinonic acid),thionyl chloride as raw materials and dichloromethane as solvent. Pinonic acid(27 mmol) and thionyl chloride(32 mmol) were dissolved in dichloromethane(50 ml). The resulting mixture was refluxed for 8 h. After refluxing the solvent was distilled away under vacuum and the remainder was 2-(3-acetyl-2,2-dimethylcyclobutyl)acetyl chloride. The acetyl chloride reacted with 2,6-difluorobenzenamine(27 mmol) for 24 h using dichloromethane as solvent. After the reaction was complete the solvent was distilled away and the crude title compound was gained. The pure compound was obtained by crystallizing from a mixture of ethanol (40 ml) and water (40 ml). Crystals of the title compound suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Refinement

All H atoms bonded to the C atoms were placed geometrically at the distances of 0.93–0.97 Å and included in the refinement in riding motion approximation with U_{iso}(H) = 1.2 or 1.5U_{eq} of the carrier atom.

Figures

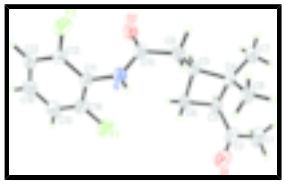
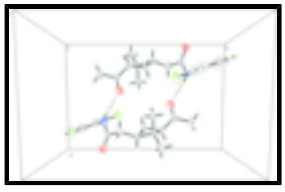


Fig. 1. A view of the molecular structure of (I), showing displacement ellipsoids at the 30% probability level. Dash lines indicate N—H···O hydrogen bonds and O—H···N hydrogen bonds.



2-[(1*S*,3*S*)-3-acetyl-2,2-dimethylcyclobutyl]-*N*-(2,6-difluorophenyl)acetamide

Crystal data

$C_{16}H_{19}F_2NO_2$	F_{000} = 624
M_r = 295.32	D_x = 1.274 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 392 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.7840 (18) Å	Cell parameters from 25 reflections
b = 12.914 (3) Å	θ = 9–14º
c = 13.747 (3) Å	μ = 0.10 mm^{-1}
β = 99.20 (3)º	T = 293 (2) K
V = 1539.4 (5) Å^3	Block, colourless
Z = 4	0.40 × 0.20 × 0.10 mm

Data collection

Nonius CAD4 diffractometer	R_{int} = 0.032
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 2.2º
T = 293(2) K	h = −10→10
ω/2θ scans	k = 0→15
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→16
T_{min} = 0.931, T_{max} = 0.960	3 standard reflections
3222 measured reflections	every 200 reflections
3022 independent reflections	intensity decay: none
1682 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.069	H-atom parameters constrained
$wR(F^2)$ = 0.207	$w = 1/[\sigma^2(F_o^2) + (0.060P)^2 + 2.3P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max} < 0.001$
3022 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}

190 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *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	0.3196 (4)	0.6850 (2)	1.17341 (19)	0.0972 (10)
F2	0.1731 (4)	0.97005 (19)	0.9730 (2)	0.0912 (9)
O1	0.4326 (4)	0.3369 (3)	1.0109 (3)	0.0866 (10)
O2	0.0165 (3)	0.7648 (2)	0.9196 (2)	0.0720 (8)
N	0.2668 (4)	0.7654 (2)	0.9872 (2)	0.0567 (8)
H0A	0.3592	0.7486	0.9802	0.068*
C1	0.2701 (7)	0.2067 (3)	0.9316 (4)	0.0961 (17)
H1A	0.3562	0.1623	0.9541	0.144*
H1B	0.2472	0.2041	0.8609	0.144*
H1C	0.1819	0.1838	0.9589	0.144*
C2	0.3089 (6)	0.3152 (3)	0.9638 (3)	0.0621 (11)
C3	0.1890 (5)	0.3958 (3)	0.9335 (3)	0.0561 (10)
H3A	0.0876	0.3686	0.9416	0.067*
C4	0.1781 (4)	0.4476 (3)	0.8287 (3)	0.0551 (9)
C5	0.1363 (5)	0.5490 (3)	0.8790 (3)	0.0538 (9)
H5A	0.0242	0.5511	0.8766	0.065*
C6	0.2092 (5)	0.5046 (3)	0.9783 (3)	0.0552 (10)
H6A	0.1494	0.5167	1.0307	0.066*
H6B	0.3160	0.5247	0.9984	0.066*
C7	0.0567 (6)	0.4049 (4)	0.7473 (4)	0.0863 (15)
H7A	−0.0410	0.4021	0.7700	0.129*
H7B	0.0859	0.3366	0.7299	0.129*
H7C	0.0487	0.4492	0.6906	0.129*
C8	0.3368 (5)	0.4528 (3)	0.7956 (3)	0.0697 (12)
H8A	0.3563	0.3889	0.7640	0.105*
H8B	0.4150	0.4637	0.8519	0.105*
H8C	0.3382	0.5090	0.7500	0.105*
C9	0.1893 (5)	0.6545 (3)	0.8476 (3)	0.0604 (10)
H9A	0.2997	0.6538	0.8474	0.072*
H9B	0.1379	0.6710	0.7816	0.072*

C10	0.1500 (5)	0.7342 (3)	0.9197 (3)	0.0521 (9)
C11	0.2446 (4)	0.8255 (3)	1.0702 (3)	0.0518 (9)
C12	0.1926 (5)	0.9262 (3)	1.0629 (3)	0.0602 (10)
C13	0.1628 (5)	0.9823 (3)	1.1429 (4)	0.0743 (13)
H13A	0.1261	1.0498	1.1354	0.089*
C14	0.1885 (5)	0.9364 (4)	1.2332 (4)	0.0809 (14)
H14A	0.1694	0.9735	1.2880	0.097*
C15	0.2414 (6)	0.8377 (4)	1.2453 (3)	0.0798 (14)
H15A	0.2602	0.8076	1.3075	0.096*
C16	0.2665 (5)	0.7839 (3)	1.1642 (3)	0.0661 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.157 (3)	0.0625 (17)	0.0727 (17)	0.0316 (17)	0.0194 (17)	0.0134 (13)
F2	0.141 (2)	0.0525 (15)	0.0784 (18)	0.0138 (15)	0.0126 (16)	0.0088 (13)
O1	0.084 (2)	0.076 (2)	0.101 (3)	0.0196 (19)	0.018 (2)	0.0029 (19)
O2	0.0753 (19)	0.070 (2)	0.0693 (19)	0.0084 (16)	0.0069 (15)	−0.0178 (15)
N	0.067 (2)	0.0504 (19)	0.0536 (19)	0.0059 (16)	0.0134 (16)	−0.0069 (15)
C1	0.139 (5)	0.044 (3)	0.113 (4)	0.009 (3)	0.048 (4)	−0.006 (3)
C2	0.080 (3)	0.047 (2)	0.068 (3)	0.005 (2)	0.037 (2)	0.008 (2)
C3	0.070 (2)	0.0351 (19)	0.070 (3)	−0.0024 (17)	0.033 (2)	0.0002 (18)
C4	0.064 (2)	0.049 (2)	0.056 (2)	0.0020 (18)	0.0200 (18)	−0.0100 (18)
C5	0.073 (2)	0.043 (2)	0.050 (2)	0.0064 (18)	0.0218 (18)	−0.0021 (17)
C6	0.080 (3)	0.046 (2)	0.046 (2)	0.0025 (19)	0.0292 (19)	−0.0013 (17)
C7	0.090 (3)	0.079 (3)	0.083 (3)	0.008 (3)	−0.008 (3)	−0.027 (3)
C8	0.099 (3)	0.058 (3)	0.061 (3)	0.011 (2)	0.042 (2)	−0.003 (2)
C9	0.089 (3)	0.051 (2)	0.045 (2)	0.003 (2)	0.019 (2)	−0.0009 (18)
C10	0.071 (2)	0.040 (2)	0.049 (2)	0.0011 (19)	0.0207 (19)	0.0050 (17)
C11	0.062 (2)	0.042 (2)	0.052 (2)	0.0051 (17)	0.0099 (17)	−0.0065 (17)
C12	0.071 (3)	0.051 (2)	0.057 (2)	0.0063 (19)	0.005 (2)	−0.003 (2)
C13	0.078 (3)	0.058 (3)	0.084 (3)	0.015 (2)	0.004 (2)	−0.023 (2)
C14	0.086 (3)	0.088 (4)	0.066 (3)	0.017 (3)	0.005 (2)	−0.028 (3)
C15	0.096 (3)	0.092 (4)	0.050 (2)	0.008 (3)	0.009 (2)	−0.011 (3)
C16	0.090 (3)	0.051 (2)	0.059 (3)	0.009 (2)	0.016 (2)	0.002 (2)

Geometric parameters (Å, °)

F1—C16	1.359 (5)	C6—H6A	0.9700
F2—C12	1.345 (5)	C6—H6B	0.9700
O1—C2	1.206 (5)	C7—H7A	0.9600
O2—C10	1.237 (4)	C7—H7B	0.9600
N—C10	1.332 (5)	C7—H7C	0.9600
N—C11	1.419 (4)	C8—H8A	0.9600
N—H0A	0.8600	C8—H8B	0.9600
C1—C2	1.492 (6)	C8—H8C	0.9600
C1—H1A	0.9600	C9—C10	1.507 (5)
C1—H1B	0.9600	C9—H9A	0.9700
C1—H1C	0.9600	C9—H9B	0.9700

C2—C3	1.492 (6)	C11—C12	1.376 (5)
C3—C6	1.533 (5)	C11—C16	1.383 (5)
C3—C4	1.577 (5)	C12—C13	1.377 (6)
C3—H3A	0.9800	C13—C14	1.363 (6)
C4—C7	1.520 (6)	C13—H13A	0.9300
C4—C8	1.536 (5)	C14—C15	1.357 (7)
C4—C5	1.551 (5)	C14—H14A	0.9300
C5—C6	1.524 (5)	C15—C16	1.361 (6)
C5—C9	1.525 (5)	C15—H15A	0.9300
C5—H5A	0.9800		
C10—N—C11	122.4 (3)	H7A—C7—H7B	109.5
C10—N—H0A	118.8	C4—C7—H7C	109.5
C11—N—H0A	118.8	H7A—C7—H7C	109.5
C2—C1—H1A	109.5	H7B—C7—H7C	109.5
C2—C1—H1B	109.5	C4—C8—H8A	109.5
H1A—C1—H1B	109.5	C4—C8—H8B	109.5
C2—C1—H1C	109.5	H8A—C8—H8B	109.5
H1A—C1—H1C	109.5	C4—C8—H8C	109.5
H1B—C1—H1C	109.5	H8A—C8—H8C	109.5
O1—C2—C1	121.5 (4)	H8B—C8—H8C	109.5
O1—C2—C3	121.5 (4)	C10—C9—C5	108.2 (3)
C1—C2—C3	117.0 (4)	C10—C9—H9A	110.1
C2—C3—C6	119.7 (4)	C5—C9—H9A	110.1
C2—C3—C4	119.2 (3)	C10—C9—H9B	110.1
C6—C3—C4	87.9 (3)	C5—C9—H9B	110.1
C2—C3—H3A	109.4	H9A—C9—H9B	108.4
C6—C3—H3A	109.4	O2—C10—N	121.9 (3)
C4—C3—H3A	109.4	O2—C10—C9	122.3 (4)
C7—C4—C8	111.1 (3)	N—C10—C9	115.7 (4)
C7—C4—C5	116.5 (3)	C12—C11—C16	115.5 (4)
C8—C4—C5	112.7 (3)	C12—C11—N	122.9 (4)
C7—C4—C3	116.8 (4)	C16—C11—N	121.5 (3)
C8—C4—C3	111.4 (3)	F2—C12—C11	117.3 (4)
C5—C4—C3	86.3 (3)	F2—C12—C13	120.0 (4)
C6—C5—C9	119.1 (3)	C11—C12—C13	122.8 (4)
C6—C5—C4	89.2 (3)	C14—C13—C12	118.3 (4)
C9—C5—C4	121.6 (3)	C14—C13—H13A	120.8
C6—C5—H5A	108.5	C12—C13—H13A	120.8
C9—C5—H5A	108.5	C15—C14—C13	121.5 (4)
C4—C5—H5A	108.5	C15—C14—H14A	119.2
C5—C6—C3	88.8 (3)	C13—C14—H14A	119.2
C5—C6—H6A	113.8	C14—C15—C16	118.5 (5)
C3—C6—H6A	113.8	C14—C15—H15A	120.8
C5—C6—H6B	113.8	C16—C15—H15A	120.8
C3—C6—H6B	113.8	F1—C16—C15	120.1 (4)
H6A—C6—H6B	111.1	F1—C16—C11	116.6 (4)
C4—C7—H7A	109.5	C15—C16—C11	123.3 (4)
C4—C7—H7B	109.5		

O1—C2—C3—C6	−11.5 (6)	C4—C5—C9—C10	−171.9 (3)
C1—C2—C3—C6	169.5 (4)	C11—N—C10—O2	7.8 (6)
O1—C2—C3—C4	94.2 (5)	C11—N—C10—C9	−168.6 (3)
C1—C2—C3—C4	−84.7 (5)	C5—C9—C10—O2	−74.6 (5)
C2—C3—C4—C7	98.3 (5)	C5—C9—C10—N	101.8 (4)
C6—C3—C4—C7	−138.4 (4)	C10—N—C11—C12	−67.9 (5)
C2—C3—C4—C8	−30.8 (5)	C10—N—C11—C16	108.6 (4)
C6—C3—C4—C8	92.5 (3)	C16—C11—C12—F2	178.6 (4)
C2—C3—C4—C5	−143.8 (4)	N—C11—C12—F2	−4.7 (6)
C6—C3—C4—C5	−20.5 (3)	C16—C11—C12—C13	−0.5 (6)
C7—C4—C5—C6	138.9 (4)	N—C11—C12—C13	176.2 (4)
C8—C4—C5—C6	−91.0 (4)	F2—C12—C13—C14	−178.0 (4)
C3—C4—C5—C6	20.6 (3)	C11—C12—C13—C14	1.1 (7)
C7—C4—C5—C9	−97.0 (5)	C12—C13—C14—C15	−0.3 (8)
C8—C4—C5—C9	33.2 (5)	C13—C14—C15—C16	−1.1 (8)
C3—C4—C5—C9	144.8 (4)	C14—C15—C16—F1	179.9 (4)
C9—C5—C6—C3	−147.4 (3)	C14—C15—C16—C11	1.7 (8)
C4—C5—C6—C3	−21.2 (3)	C12—C11—C16—F1	−179.2 (4)
C2—C3—C6—C5	143.6 (3)	N—C11—C16—F1	4.0 (6)
C4—C3—C6—C5	20.9 (3)	C12—C11—C16—C15	−1.0 (7)
C6—C5—C9—C10	−63.1 (5)	N—C11—C16—C15	−177.7 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0A···O1^{i}	0.86	2.12	2.949 (5)	161

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2.

Fig. 1

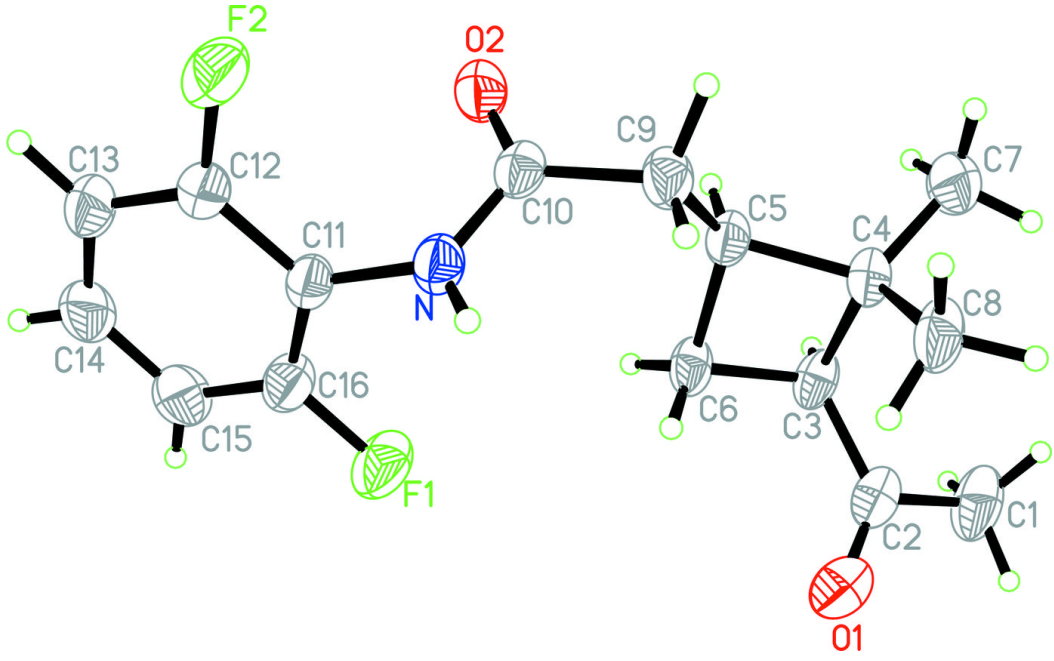


Fig. 2