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Crystal structure of 3-iodo-*N*²-(2-methyl-1-(methylsulfonyl)propan-2-yl)-*N*¹-(2-methyl-4-(perfluoropropan-2-yl)phenyl)phthalamide, C₂₃H₂₂F₇I₁N₂O₄S₁

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Abstract

8[C₂₃H₂₂F₇I₁N₂O₄S₁], monoclinic, *Cc* (no. 9), *a* = 19.762(3) Å, *b* = 24.690(4) Å, *c* = 12.106(2) Å, β = 113.696(2)°, *V* = 5408.8(15) Å³, *Z* = 1, *R*_{gt}(*F*) = 0.0488, *wR*_{ref}(*F*²) = 0.1411, *T* = 296(2) K.

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The asymmetric unit of the title crystal structure 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.

Source of material

Twenty percent flubendiamide water dispersible granules (10 g) were dissolved in 30 mL acetone, and the filtrate was concentrated and crystallized after suction filtration to obtain an off-white solid product (1.58 g) with a yield of 79%. The off-white solid was recrystallized from methanol, and colorless crystals were obtained at room temperature after a few days.

Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.90–0.97 Å with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2 U_{\text{eq}}(\text{C})$ for all other H atoms.

Comment

Flubendiamide, 3-iodo-*N*-(2-methanesulfonyl-1,1-dimethylethyl)-*N'*-(2-methyl-4-(1,2,2,2-tetrafluoro-1-trifluoromethylethyl)phenyl)phthalamide, is a new modern insecticide of phthalic acid diamides commercialized by Japan's Shinko Co., Ltd. and Bayer Crop Science Company of Germany [5–7]. In 1993, Nihon Nohyaku discovered the parent compound of flubendiamide during the research and development of the herbicide of pyrazine dicarboxamide. Through the discovery of effective substituents, he synthesized flubendiamide in 1998. Flubendiamide is a systemic foliar insecticide with extremely high activity against a broad spectrum of lepidopteran insects [8–10]. And it works fast and lasts for a long time. However, the toxicity to mammals is extremely low, and non-target organisms have almost no impact. It is less harmful to the biological

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3214 (4)	0.2403 (3)	−0.0290 (6)	0.0404 (16)
C2	0.2995 (4)	0.1950 (3)	0.0318 (6)	0.0391 (15)
C3	0.3091 (5)	0.1407 (4)	0.0062 (7)	0.0491 (18)
C4	0.2835 (6)	0.0987 (4)	0.0526 (10)	0.067 (2)
H4	0.289174	0.063165	0.032527	0.080*
C5	0.2488 (6)	0.1097 (4)	0.1296 (10)	0.065 (2)
H5	0.233742	0.081380	0.165102	0.078*
C6	0.2370 (5)	0.1617 (4)	0.1531 (8)	0.059 (2)
H6	0.210986	0.168330	0.200951	0.071*
C7	0.2622 (4)	0.2055 (3)	0.1085 (7)	0.0402 (15)
C8	0.2535 (4)	0.2620 (3)	0.1429 (6)	0.0422 (16)
C9	0.1665 (4)	0.3244 (3)	0.1698 (7)	0.0466 (17)
C10	0.1207 (5)	0.3592 (4)	0.0817 (8)	0.055 (2)
H10	0.105407	0.350772	0.000383	0.066*
C11	0.0976 (6)	0.4077 (4)	0.1173 (12)	0.074 (3)
H11	0.067233	0.431490	0.058865	0.089*
C12	0.1195 (7)	0.4204 (4)	0.2380 (12)	0.073 (3)
C13	0.1662 (7)	0.3853 (5)	0.3230 (10)	0.073 (3)
H13	0.183007	0.394640	0.404142	0.087*
C14	0.1889 (5)	0.3367 (4)	0.2922 (8)	0.058 (2)
C15	0.2371 (10)	0.2995 (7)	0.3871 (10)	0.103 (5)
H15A	0.242812	0.312921	0.464859	0.155*
H15B	0.284629	0.297427	0.383374	0.155*
H15C	0.215099	0.264180	0.374767	0.155*
C16	0.0909 (10)	0.4696 (6)	0.277 (2)	0.113 (6)
C17	0.1001 (10)	0.5218 (7)	0.224 (2)	0.120 (5)
C18	0.0252 (13)	0.4609 (9)	0.297 (2)	0.128 (6)
C19	0.2691 (4)	0.3053 (3)	−0.2012 (6)	0.0439 (16)
C20	0.3163 (6)	0.2824 (5)	−0.2652 (9)	0.068 (3)
H20A	0.365207	0.274896	−0.206647	0.102*
H20B	0.318756	0.308466	−0.322329	0.102*
H20C	0.294229	0.249601	−0.306609	0.102*
C21	0.1916 (6)	0.3156 (5)	−0.2925 (10)	0.071 (3)
H21A	0.172299	0.283242	−0.338216	0.106*
H21B	0.192322	0.344162	−0.345978	0.106*
H21C	0.160951	0.326091	−0.251616	0.106*
C22	0.2998 (5)	0.3572 (4)	−0.1269 (8)	0.056 (2)
H22A	0.340168	0.346644	−0.052364	0.067*
H22B	0.261246	0.372025	−0.105472	0.067*
C23	0.4266 (7)	0.4010 (5)	−0.1450 (13)	0.084 (3)
H23A	0.445594	0.426902	−0.184493	0.126*
H23B	0.435927	0.365041	−0.165484	0.126*
H23C	0.450587	0.405917	−0.059227	0.126*
C24	0.0051 (4)	0.2088 (3)	0.9579 (7)	0.0419 (16)
C25	−0.0119 (4)	0.2631 (3)	0.8960 (7)	0.0414 (16)
C26	−0.0494 (5)	0.3024 (4)	0.9309 (7)	0.0507 (18)
C27	−0.0677 (5)	0.3521 (4)	0.8729 (8)	0.057 (2)
H27	−0.093314	0.377724	0.897322	0.069*
C28	−0.0475 (5)	0.3631 (3)	0.7793 (8)	0.057 (2)
H28	−0.058964	0.396514	0.740719	0.069*
C29	−0.0098 (5)	0.3245 (3)	0.7414 (7)	0.0514 (19)
H29	0.002981	0.331985	0.677020	0.062*
C30	0.0085 (4)	0.2748 (3)	0.8000 (6)	0.0384 (15)
C31	0.0559 (4)	0.2360 (3)	0.7680 (7)	0.0439 (16)
C32	0.0698 (4)	0.1870 (4)	0.6033 (7)	0.0514 (19)

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.21 × 0.16 × 0.13 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.34 mm ^{−1}
Diffractionmeter, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	20,707, 9580, 0.027
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	8203
$N(\text{param})_{\text{refined}}$:	693
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
C33	0.1013 (6)	0.1396 (4)	0.6603 (9)	0.063 (2)
H33	0.099568	0.130356	0.733587	0.075*
C34	0.1362 (7)	0.1052 (5)	0.6069 (11)	0.076 (3)
H34	0.156911	0.072722	0.644300	0.091*
C35	0.1398 (6)	0.1191 (5)	0.5008 (11)	0.072 (3)
C36	0.1081 (6)	0.1673 (5)	0.4445 (9)	0.069 (3)
H36	0.110865	0.176621	0.372031	0.083*
C37	0.0733 (5)	0.2012 (4)	0.4927 (8)	0.057 (2)
C38	0.0397 (6)	0.2524 (5)	0.4290 (9)	0.076 (3)
H38A	0.062589	0.262305	0.375394	0.114*
H38B	0.047265	0.280719	0.487136	0.114*
H38C	−0.012304	0.247171	0.383565	0.114*
C39	0.1807 (7)	0.0840 (6)	0.4446 (14)	0.095 (4)
C42	−0.0434 (5)	0.1146 (3)	0.9414 (8)	0.0490 (18)
C43	−0.0555 (7)	0.1167 (5)	1.0589 (10)	0.074 (3)
H43A	−0.024012	0.143797	1.111258	0.111*
H43B	−0.043995	0.082015	1.098040	0.111*
H43C	−0.106206	0.125457	1.040756	0.111*
C44	0.0283 (6)	0.0855 (4)	0.9605 (10)	0.068 (3)
H44A	0.036337	0.086870	0.887414	0.102*
H44B	0.025116	0.048464	0.981798	0.102*
H44C	0.068662	0.102932	1.024212	0.102*
C45	−0.1111 (5)	0.0864 (3)	0.8475 (8)	0.0506 (18)
H45A	−0.153290	0.110144	0.830186	0.061*
H45B	−0.120412	0.053934	0.884307	0.061*
C46	−0.2017 (6)	0.0531 (5)	0.6206 (10)	0.070 (3)
H46A	−0.207546	0.041174	0.541826	0.105*
H46B	−0.230727	0.085097	0.613474	0.105*
H46C	−0.218001	0.025029	0.659227	0.105*
F1	0.1488 (9)	0.4850 (6)	0.4041 (12)	0.180 (5)
F2	0.1548 (8)	0.5255 (5)	0.1951 (15)	0.162 (4)
F3	0.0377 (9)	0.5198 (6)	0.1150 (16)	0.183 (5)
F4	0.0930 (7)	0.5658 (4)	0.2815 (14)	0.152 (4)
F5	−0.0260 (8)	0.4434 (7)	0.1993 (18)	0.189 (6)
F6	−0.0056 (8)	0.5021 (5)	0.3272 (15)	0.165 (5)
F7	0.0309 (11)	0.4240 (8)	0.3690 (19)	0.203 (6)
I1	0.36428 (5)	0.12208 (4)	−0.10412 (7)	0.0916 (3)
I2	−0.07813 (4)	0.28808 (3)	1.07728 (6)	0.0813 (3)
N1	0.2632 (3)	0.2637 (3)	−0.1176 (5)	0.0417 (14)
H1	0.219654	0.253770	−0.126091	0.050*
N2	0.1853 (3)	0.2745 (3)	0.1336 (6)	0.0462 (15)
H2	0.151071	0.250602	0.103721	0.055*
N3	0.0315 (4)	0.2220 (3)	0.6520 (6)	0.0480 (15)
H3	−0.010131	0.235105	0.603317	0.058*
N4	−0.0431 (4)	0.1700 (3)	0.8969 (6)	0.0441 (14)
H4A	−0.076099	0.178089	0.826944	0.053*
O1	0.3856 (3)	0.2531 (3)	−0.0024 (5)	0.0543 (14)
O2	0.3051 (3)	0.2934 (2)	0.1810 (6)	0.0546 (14)
O3	0.2969 (5)	0.4088 (3)	−0.3209 (6)	0.080 (2)
O4	0.3236 (5)	0.4594 (3)	−0.1341 (8)	0.079 (2)
O5	0.0578 (3)	0.2023 (2)	1.0562 (5)	0.0555 (14)
O6	0.1154 (3)	0.2203 (3)	0.8463 (5)	0.0574 (15)
O7	−0.0862 (4)	0.1134 (3)	0.6563 (6)	0.0622 (15)
O8	−0.0678 (4)	0.0185 (3)	0.7230 (7)	0.0705 (17)
S3	−0.10845 (12)	0.06747 (8)	0.70715 (19)	0.0500 (4)
S4	0.33232 (13)	0.41046 (9)	−0.1915 (2)	0.0571 (5)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
F13	0.1693 (6)	0.0073 (4)	0.3237 (10)	0.127 (3)
F9	0.2957 (5)	0.0537 (5)	0.4547 (11)	0.141 (4)
F12	0.0712 (6)	0.0408 (6)	0.3216 (12)	0.152 (4)
F10	0.2978 (6)	0.1098 (6)	0.5918 (13)	0.155 (4)
F8	0.1672 (10)	0.1079 (7)	0.3186 (15)	0.194 (6)
C41	0.1393 (8)	0.0392 (7)	0.3720 (17)	0.108 (5)
C40	0.2584 (9)	0.0800 (8)	0.5047 (18)	0.108 (5)
F11	0.2628 (10)	0.0404 (7)	0.6038 (15)	0.191 (6)
F14	0.1408 (11)	0.0060 (7)	0.4750 (17)	0.197 (6)

enrichment and biomagnification that may cause animals in the body, reflecting excellent environmental performance [11]. Currently, there are more than 200 crops registered for flubendiamide [12]. The mechanism of action of bisamide insecticides is relatively novel compared to traditional nerve agents. They are a type of muscle toxins, mainly contact and stomach poisons, and insecticides with antifeedant activity [13, 14]. The mechanism of action of flubendiamide is to activate the calcium ion release channel in the ryanodine receptor cells, leading to the uncontrolled release of stored calcium ions, and ultimately causing insects to stop eating, lethargy, contractile paralysis and until death [15].

There are two crystallographic independent molecules in the asymmetric unit (molecule A and B in the figure). In the molecule of the title compound bond lengths and angles are very similar to those given in the literature [16, 17]. The torsion angles of C3–C2–C1–N1, C2–C1–N1–C19, C1–N1–C19–C22, N1–C19–C22–S4 and C19–C22–S4–C23 of the molecule A are 103.3(8)°, 173.0(7)°, 68.7(9)°, 166.7(6)° and 93.9(9)°, respectively. Those torsion angles of the molecule B are 98.6(9)°, 174.5(7)°, 175.4(7)°, 73.1(8)° and 166.4(7)°, respectively.

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