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The crystal structure of *N'*-(*tert*-butyl)-*N'*-(3,5-dimethylbenzoyl)-3-methoxy-*N*,2-dimethylbenzohydrazide, C₂₃H₃₀N₂O₃

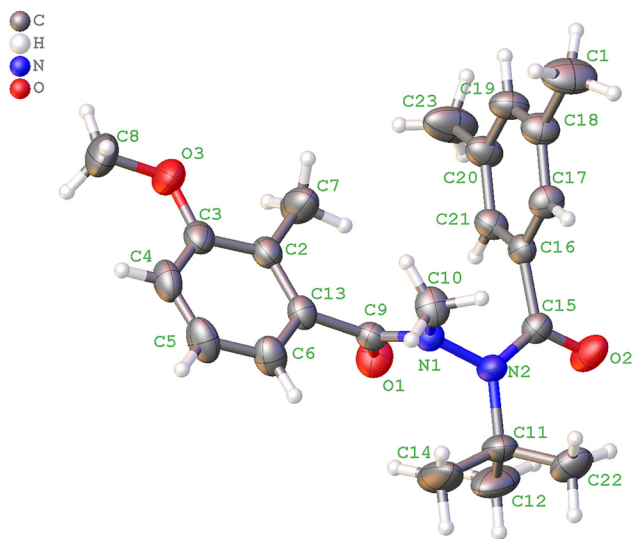


Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10820, 3889, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2930
$N(\text{param})_{\text{refined}}$:	261
Programs:	Bruker, ¹ Olex2, ² SHELX ^{3,4}

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Abstract

C₂₃H₃₀N₂O₃, monoclinic, $P2_1/c$ (no. 14), $a = 15.4357(14)$ Å, $b = 7.2340(7)$ Å, $c = 21.8274(17)$ Å, $\beta = 114.881(5)^\circ$, $V = 2211.1(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0492$, $wR_{\text{ref}}(F^2) = 0.1284$, $T = 293$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

In a 100 mL round-bottom flask *N'*-(*tert*-butyl)-*N'*-(3,5-dimethylbenzoyl)-3-methoxy-2-methylbenzohydrazide

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(500 mg; 1.36 mmol, CH₃I (193 mg; 1.36 mmol), K₂CO₃ (123 mg; 0.89 mmol) and DMF (50 mL) were thoroughly dissolved and mixed, stirred and refluxed at 150 °C in nitrogen atmosphere for 2.5 h, 200 mL of saline was added to the reaction solution cooled to room temperature, and the aqueous layer was extracted with ethyl acetate (50 ml × 3), dry the organic phase with anhydrous sodium sulfate, filtered, and concentrated under reduced-pressure. Methanol is used to recrystallize to obtain the desired compound (white solid, 364 mg, 70 %). Single crystals were obtained by crystallization from a mixture of dichloromethane (1 mL) and n-hexane(2 mL).

2 Experimental details

Using Olex2,² the structure was solved with the SHELXT³ program and refined with the SHELXL⁴ package. All hydrogen atoms were placed in the calculated positions.

3 Comment

Hydrazide compounds have been widely used and studied as insect growth regulators. One of the most representative is methoxyfenozide.^{5–9} Based on the original compound, methoxyfenozide, we synthesized the new compound described in this paper with moderate yields. The introduction of the N1 methyl group has caused some significant changes in the properties of methoxyfenozide itself. The reaction route

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H4A	−0.020602	0.672294	0.698823	0.062*
H5A	0.122340	0.692779	0.793352	0.068*
H6A	0.234369	0.461617	0.817913	0.059*
H7A	0.098923	0.026114	0.654459	0.096*
H7B	0.042991	0.152634	0.591854	0.096*
H7C	−0.010442	0.062275	0.631512	0.096*
H8A	−0.165543	0.573920	0.623102	0.118*
H8B	−0.196097	0.520175	0.547252	0.118*
H8C	−0.112998	0.661931	0.582423	0.118*
H10A	0.290686	0.365806	0.692414	0.073*
H10B	0.397548	0.301601	0.726583	0.073*
H10C	0.356701	0.429954	0.766025	0.073*
H12A	0.342639	−0.129446	0.885253	0.124*
H12B	0.444174	−0.132758	0.945697	0.124*
H12C	0.424651	−0.247160	0.880189	0.124*
H13A	0.560851	−0.092759	0.870214	0.089*
H13B	0.582105	0.041672	0.931195	0.089*
H13C	0.560599	0.121546	0.859473	0.089*
H14A	0.433251	0.313880	0.873584	0.154*
H14B	0.455673	0.216087	0.942713	0.154*
H14C	0.351454	0.214179	0.885480	0.154*
H17A	0.409504	0.050303	0.651001	0.051*
H19A	0.181957	−0.148430	0.495130	0.058*
H21A	0.222310	−0.272749	0.683392	0.048*
H22A	0.303257	−0.001109	0.472552	0.117*
H22B	0.401533	0.045330	0.532556	0.117*
H22C	0.316675	0.186060	0.511706	0.117*
H23A	0.105250	−0.448821	0.585853	0.118*
H23B	0.082274	−0.380513	0.512469	0.118*
H23C	0.044800	−0.268872	0.557988	0.118*
C1	0.15737 (12)	0.2968 (2)	0.73825 (9)	0.0377 (4)
C2	0.07154 (13)	0.2812 (3)	0.68104 (9)	0.0401 (4)
C3	0.00594 (14)	0.4270 (3)	0.66702 (9)	0.0448 (5)
C4	0.02441 (15)	0.5784 (3)	0.70871 (11)	0.0519 (5)
C5	0.10978 (15)	0.5904 (3)	0.76515 (12)	0.0563 (6)
C6	0.17649 (14)	0.4520 (3)	0.78004 (11)	0.0494 (5)
C7	0.04870 (17)	0.1157 (3)	0.63564 (11)	0.0643 (6)
C8	−0.14312 (18)	0.5521 (4)	0.58884 (13)	0.0784 (8)
C9	0.22626 (12)	0.1386 (2)	0.76037 (8)	0.0350 (4)
C10	0.34218 (14)	0.3301 (3)	0.73426 (10)	0.0484 (5)
C11	0.43968 (14)	0.0313 (3)	0.86724 (9)	0.0519 (5)
C12	0.41002 (18)	−0.1350 (4)	0.89741 (11)	0.0828 (9)
C13	0.54556 (14)	0.0248 (3)	0.88353 (10)	0.0596 (6)
C14	0.4180 (2)	0.2104 (5)	0.89480 (12)	0.1027 (12)
C15	0.38713 (12)	−0.1133 (2)	0.75285 (8)	0.0359 (4)
C16	0.32404 (12)	−0.1077 (2)	0.67837 (8)	0.0347 (4)
C17	0.35367 (13)	−0.0193 (3)	0.63419 (9)	0.0426 (4)
C18	0.30033 (14)	−0.0342 (3)	0.56485 (9)	0.0469 (5)
C19	0.21797 (14)	−0.1382 (3)	0.54148 (9)	0.0487 (5)
C20	0.18685 (13)	−0.2284 (3)	0.58463 (9)	0.0460 (5)
C21	0.24143 (13)	−0.2124 (3)	0.65352 (9)	0.0397 (4)
C22	0.3335 (2)	0.0574 (4)	0.51592 (11)	0.0782 (8)
C23	0.09660 (17)	−0.3420 (4)	0.55780 (11)	0.0784 (8)
N1	0.31417 (10)	0.16738 (19)	0.76127 (7)	0.0347 (3)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
N2	0.38365 (10)	0.0310 (2)	0.79140 (7)	0.0376 (4)
O1	0.20613 (9)	−0.00672 (18)	0.77963 (7)	0.0503 (4)
O2	0.44035 (10)	−0.24500 (18)	0.77644 (7)	0.0562 (4)
O3	−0.07596 (10)	0.4043 (2)	0.60924 (7)	0.0637 (4)

adopted in this paper has good substrate applicability and functional group compatibility, which provides a convenient and efficient reaction route for the introduction of different substitutes at the N1 position of methoxyfenozide.

The molecule in the title compound adopts a non-planar conformation, with two aromatic rings located in different planes, separated by a dihedral angle of 92.9°. In the crystal structure, there are two carbonyl groups and one nitrogen-nitrogen single bond, and the two carbonyl groups are connected by a nitrogen-nitrogen single bond, and the length of the nitrogen-nitrogen single bond of N(1)–N(2) is 1.401(19) Å.¹⁰ The similar structures that have been published remain structurally stable by O–H···O hydrogen bond, the molecules are assembled by the intermolecular interactions among the benzene rings and dimethylbenzene units and CH···O hydrogen bonds between the acyl oxygen atom and the methoxy group into two-dimensional (2D) supramolecular layers.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Competing interests: The authors declare no conflicts of interest regarding this article.

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