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1-Benzoyl-6-methyl-3-methylsulfanyl-6,7-dihydro-1*H*-pyrano[4,3-*c*]pyrazol-4-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.033
wR factor = 0.083
Data-to-parameter ratio = 10.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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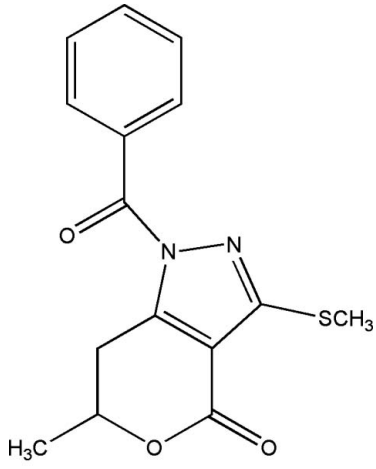
The title compound, $C_{15}H_{14}N_2O_3S$, a potent new bioactive molecule which contains pyrazole and pyrone ring systems, was synthesized by the reaction of benzohydrazide and 3-[bis(methylsulfanyl)methylene]dihydro-6-methyl-3*H*-pyran-[2,4-dione in ethanol.

Comment

In recent years, there have been a few reports of pyrone derivatives. Some patents reported pyrandione derivatives inhibiting activity for HIV proteinase (Ellsworch & Lunney, 1995; Thaisrivongs & Yang, 1994). Some bioactivities, such as antitobacco viral activity, plant-growth regulation activity, fungicidal and herbicidal bioactivities, have also been reported (Wang *et al.*, 2000; Li *et al.*, 2004). Significant activities against *Biomphalaria glabrata* egg masses have been reported (de Souza *et al.*, 2004). In view of these facts and in continuation of our interest in the chemistry of heterocycles, we have attempted to synthesize a series of pyranonopyrazole derivatives, one of which, (I), is reported here.



(I)

The molecular structure of (I) is shown in Fig. 1. The X-ray analysis reveals that there are two independent molecules which have different orientations in the asymmetric unit.

Experimental

The title compound was synthesized by adding benzohydrazide (0.136 g, 1 mmol) to an absolute ethanol solution (30 ml) containing 3-[bis(methylsulfanyl)methylene]dihydro-6-methyl-3*H*-pyran-2,4-dione (0.232 g, 1 mmol). The mixture was stirred for 5.5 h at room temperature. The product was obtained by silica-gel column chromatography using a 1:5 mixture of ethyl acetate and petroleum ether as eluant. Colourless single crystals suitable for X-ray diffraction

analysis were obtained by diffusion of *n*-hexane into a solution of the crude product in dichloromethane. ^{1}H NMR ($CDCl_3$): δ 7.49–8.16 (*m*, 5H), 4.73–4.80 (*m*, 1H), 3.15–3.69 (*m*, 2H), 2.53 (*s*, 3H), 1.58 (*d*, 3H, J = 6.3 Hz); ^{13}C NMR ($CDCl_3$): δ 166.72, 161.28, 153.22, 151.45, 133.52, 131.63, 130.97, 127.98, 110.90, 75.36, 31.25, 30.88, 20.54, 13.28; elemental analysis calculated for $C_{15}H_{16}N_2O_2S$: C 59.59, H 4.67, N 9.27%; found: C 59.56, H 4.61, N 9.26%.

Crystal data

$C_{15}H_{14}N_2O_3S$	D_x = 1.354 Mg m^{-3}
M_r = 302.35	Mo $K\alpha$ radiation
Monoclinic, *Cc*	Cell parameters from 2146 reflections
a = 24.083 (3) Å	θ = 2.3–23.6°
b = 7.393 (1) Å	μ = 0.23 mm^{-1}
c = 18.820 (3) Å	T = 293 (2) K
β = 117.765 (2)°	Block, colourless
V = 2965.2 (7) Å^3	0.38 × 0.20 × 0.10 mm
Z = 8	

Data collection

Bruker APEX-II CCD area-detector diffractometer	3972 independent reflections
	3298 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.020
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 25.0°
	$h = -28 \rightarrow 28$
T_{min} = 0.725, T_{max} = 1.000	$k = -8 \rightarrow 8$
7700 measured reflections	$l = -22 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0203P)^2 + 0.9288P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.033	
$wR(F^2)$ = 0.083	
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
3972 reflections	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
383 parameters	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
H-atom parameters constrained	Absolute structure: Flack (1983), 436 Friedel pairs
	Flack parameter: −0.01 (7)

All H atoms were placed in calculated positions [C—H = 0.93, 0.96, 0.97 or 0.98 Å for phenyl, methyl, methylene and methine H atoms, respectively] and included in the refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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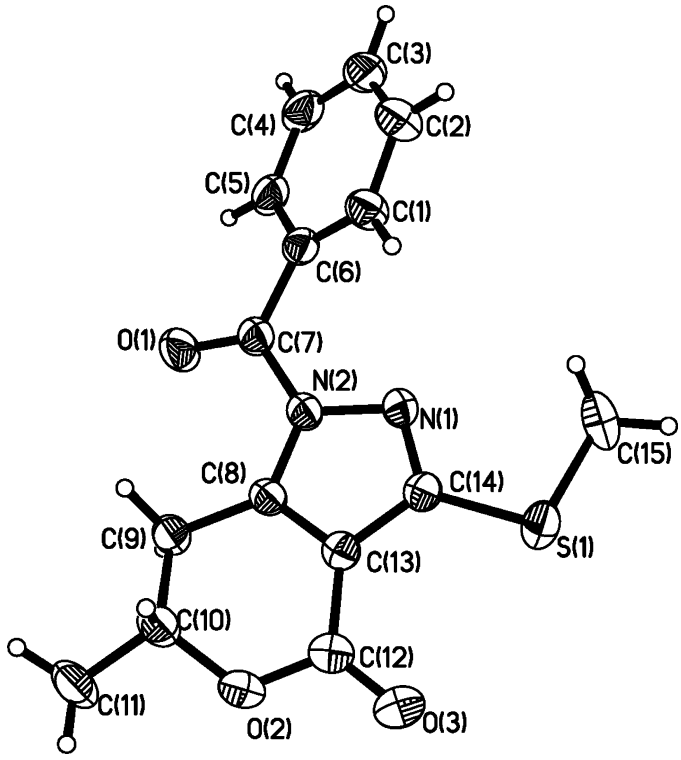

Figure 1
A view of (I), with displacement ellipsoids drawn at the 30% probability level.

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