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Crystal structure of 1-*tert*-butyl-3-(2,6-diisopropyl-4-phenoxyphenyl)-2-methylisothiourea, C₂₄H₃₄N₂OS

$V = 2357.51(11) \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.0334$, $wR_{\text{ref}}(F^2) = 0.0780$, $T = 170 \text{ K}$.

CCDC no.: 2062596

The molecular 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

The title compound was prepared according to the known procedure in 1 step with commercial available

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.32 \times 0.28 \times 0.12 \text{ mm}$
Wavelength:	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
μ :	0.15 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ_{max} , completeness:	26.4° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	27,534, 4811, 0.042
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 4318
$N(\text{param})_{\text{refined}}$:	265
Programs:	CrysAlis ^{PRO} [2], SHELX [3], OLEX2 [4]

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Abstract

C₂₄H₃₄N₂OS, $M = 398.59$, orthorhombic, $P2_12_1$ (no. 19), $a = 11.0576(3) \text{ \AA}$, $b = 11.8406(3) \text{ \AA}$, $c = 18.0061(5) \text{ \AA}$,

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6023 (2)	0.3534 (2)	0.50133 (12)	0.0349 (5)
H1	0.520670	0.354149	0.484953	0.042*
C2	0.6429 (3)	0.2696 (2)	0.54925 (14)	0.0478 (6)
H2A	0.588462	0.212782	0.565644	0.057*
C3	0.7611 (3)	0.2681 (2)	0.57326 (14)	0.0564 (8)
H3	0.788590	0.209817	0.605347	0.068*
C4	0.8393 (3)	0.3520 (3)	0.55029 (14)	0.0524 (7)
H4	0.920469	0.351955	0.567568	0.063*
C5	0.8011 (2)	0.4362 (2)	0.50247 (13)	0.0389 (6)
H5	0.855328	0.493668	0.486743	0.047*
C6	0.68212 (19)	0.43535 (19)	0.47784 (12)	0.0297 (5)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C7	0.53744 (19)	0.51286 (19)	0.39532 (12)	0.0315 (5)
C8	0.4423 (2)	0.57215 (19)	0.42493 (13)	0.0327 (5)
H8	0.453688	0.615599	0.468726	0.039*
C9	0.32932 (19)	0.56885 (19)	0.39104 (12)	0.0306 (5)
C10	0.31512 (19)	0.50297 (18)	0.32625 (11)	0.0280 (5)
C11	0.41377 (18)	0.44387 (18)	0.29545 (12)	0.0284 (4)
C12	0.5250 (2)	0.44957 (19)	0.33157 (12)	0.0317 (5)
H12	0.592554	0.409689	0.312182	0.038*
C13	0.2223 (2)	0.6322 (2)	0.42389 (13)	0.0378 (5)
H13	0.172093	0.659816	0.381396	0.045*
C14	0.2579 (3)	0.7353 (2)	0.46961 (16)	0.0562 (7)
H14A	0.300009	0.710738	0.514718	0.084*
H14B	0.185143	0.777653	0.483300	0.084*
H14C	0.311577	0.783674	0.440231	0.084*
C15	0.1433 (3)	0.5543 (3)	0.46962 (17)	0.0586 (8)
H15A	0.121841	0.487767	0.440008	0.088*
H15B	0.069412	0.594217	0.484308	0.088*
H15C	0.187382	0.530388	0.514127	0.088*
C16	0.4009 (2)	0.3746 (2)	0.22466 (12)	0.0357 (5)
H16	0.326073	0.400792	0.198642	0.043*
C17	0.3859 (3)	0.2494 (2)	0.24095 (16)	0.0644 (9)
H17A	0.456844	0.221874	0.268043	0.097*
H17B	0.377859	0.207888	0.194139	0.097*
H17C	0.313222	0.237847	0.271181	0.097*
C18	0.5063 (3)	0.3910 (3)	0.17186 (16)	0.0709 (10)
H18A	0.517131	0.471783	0.161908	0.106*
H18B	0.489875	0.351376	0.125168	0.106*
H18C	0.580129	0.360575	0.194382	0.106*
C19	0.16496 (18)	0.54265 (18)	0.23830 (11)	0.0274 (5)
C20	0.1889 (2)	0.6841 (2)	0.11308 (14)	0.0453 (6)
H20A	0.176004	0.615714	0.083470	0.068*
H20B	0.240017	0.736900	0.085288	0.068*
H20C	0.110812	0.719720	0.123705	0.068*
C21	−0.03438 (19)	0.4402 (2)	0.22528 (13)	0.0369 (5)
C22	−0.0863 (2)	0.4689 (3)	0.30155 (16)	0.0533 (7)
H22A	−0.021501	0.467161	0.338650	0.080*
H22B	−0.148518	0.413505	0.314774	0.080*
H22C	−0.122257	0.544549	0.300167	0.080*
C23	−0.1330 (3)	0.4485 (3)	0.16654 (18)	0.0615 (8)
H23A	−0.167491	0.524783	0.166931	0.092*
H23B	−0.196697	0.393359	0.177354	0.092*
H23C	−0.098382	0.432828	0.117519	0.092*
C24	0.0199 (2)	0.3225 (2)	0.22425 (17)	0.0506 (7)
H24A	0.055387	0.307758	0.175335	0.076*
H24B	−0.043529	0.266776	0.234352	0.076*
H24C	0.082830	0.317024	0.262387	0.076*
N1	0.19756 (15)	0.48856 (15)	0.29648 (10)	0.0297 (4)
N2	0.05803 (16)	0.52464 (16)	0.20359 (11)	0.0330 (4)
H2	0.039 (2)	0.570 (2)	0.1678 (14)	0.038 (7)*
O1	0.65132 (14)	0.52063 (14)	0.42933 (9)	0.0372 (4)
S1	0.26203 (5)	0.64746 (5)	0.19872 (3)	0.03594 (15)

diafenthion as starting material affording the product as a white solid [1]. The crude product was recrystallized from 1,2-dichloroethane.

Experimental details

All H atoms were positioned geometrically and treated as riding, with C–H bond lengths constrained to 0.98 Å for methyl H atoms, 1.00 Å for *tert*-butyl H atoms, 0.95 Å for alkenyl H atoms, and 0.86 Å for nitric H atoms, and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ for alkenyl H atoms, and 1.5 $U_{\text{eq}}(\text{C})$ for methyl H atoms, ethyl H atoms, and *tert*-butyl H atoms.

Comment

Diafenthion is an important insecticide and acaricide with no cross resistance problem, and is efficient on high resistant insects [5, 6]. The structure of the corresponding *S*-methylisothiurea of diafenthion was chemically named as 1-*tert*-butyl-3-(2,6-diisopropyl-4-phenoxyphenyl)-2-methylisothiurea, which was also reported with good insecticidal activity against mites [7]. Crystallization is known to affect the potency and stability of drugs. The crystal structure of diafenthion has been reported [8], but here we report the crystal structure of the title compound. In the crystal structure, the crystal packing is held together by van der Waals forces. All geometric parameters are in the expected ranges.

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References

- Ge X., Li J. *Method for Preparing Methylmercaptodiafenthion*. Patent CN105693581, 2016.
- Agilent Technologies. *CrysAlis^{PRO}, Version 1.171.37.35(release 13-08-2014 CrysAlis171.NET)*; Agilent Technologies: Santa Clara, CA, USA, 2017.
- Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
- Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
- Pascual A., Rindlisbacher A. *N*-(pyrid-3-yl)thioureas and derivatives as acaricides. I. Synthesis and biological properties. *Pestic. Sci.* 1994, *42*, 253–263.

6. Kayser H., Eilinger P. Metabolism of diafenthiuron by microsomal oxidation: procide activation and inactivation as mechanisms contributing to selectivity. *Pest Manag. Sci.* 2001, 57, 975–980.
7. Draebek J., Böger M. *Phenoxyphenylisothiourees, Process for their Preparation and their Use in Pest Control and* *Phenoxyphenylthiourees as Intermediate Products for the Preparation of Phenoxyphenylisothiourees and their Application in Pest Control.* Patent FR2465720A1, 1981.
8. Jeon Y., Kang G., Cho S., Kim T. H. Diafenthiuron: 1-*tert*-butyl-3-(2,6-diisopropyl-4-phenoxyphenyl)thiourea. *Acta Crystallogr.* 2014, E70, o807.