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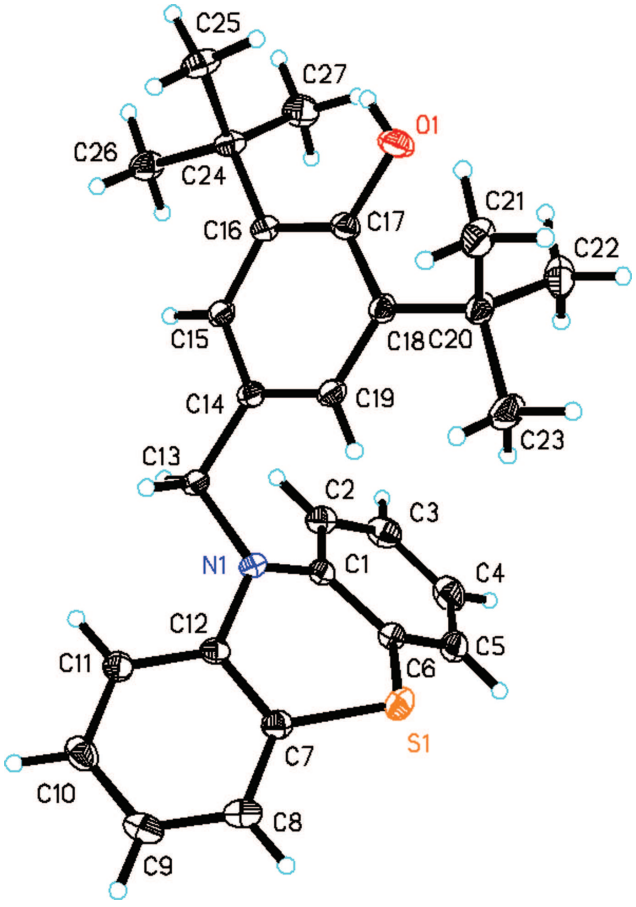
Crystal stucture of 4-((10*H*-phenothiazin-10-yl) methyl)-2,6-di-*tert*-butylphenol, $C_{27}H_{31}NOS$



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Abstract

$C_{27}H_{31}NOS$, triclinic, $P\bar{1}$ (no. 2), $a = 9.2279(8)$ Å, $b = 10.2275(9)$ Å, $c = 13.0749(12)$ Å, $\alpha = 73.910(2)°$, $\beta = 79.621(2)°$, $\gamma = 72.216(2)°$, $V = 1122.82(17)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0393$, $wR_{ref}(F^2) = 0.1072$, $T = 130$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.30 × 0.25 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.6 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	61.4°, 98.9%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11541, 6871, 0.015
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5657
$N(param)_{refined}$:	278
Programs:	SHELX [1, 2], Bruker programs [3]

Source of materials

The title compound was prepared by the reaction of 2,6-di-*tert*-butyl-4-(chloromethyl)phenol and 10*H*-phenothiazine in the presence of base. Single crystals suitable for X-ray diffraction were obtained by evaporation of a solution of the title compound in hexane at room temperature.

Experimental details

All the Friedel pairs were merged [1]. All H atoms were positioned geometrically and treated as riding, with C—H bond lengths constrained to 0.95 Å for phenyl ring H, 0.98 Å for methyl H atoms and 0.99 Å for ethyl H atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ for phenyl ring H atoms and $1.5U_{eq}(C)$ for methyl and ethyl H atoms.

Comment

As antioxidant, phenothiazine derivatives and hindered phenols have been widely used in lubricant oil. Recently, research focus on developing novel and highly effective antioxidants, especially introducing different functional groups

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

Atom	x	y	z	U_{iso}*/U_{eq}
S1	0.37889(3)	0.31619(3)	0.99383(2)	0.02274(8)
O1	0.74965(9)	0.16894(10)	0.42728(7)	0.02595(19)
H1	0.726210	0.166849	0.368695	0.039*
N1	0.24683(10)	0.39775(10)	0.78933(7)	0.01651(17)
C1	0.25542(11)	0.25588(11)	0.84362(8)	0.01648(19)
C2	0.21012(12)	0.16478(12)	0.80132(9)	0.0209(2)
H2	0.174805	0.196884	0.732398	0.025*
C3	0.21659(14)	0.02742(13)	0.85981(10)	0.0254(2)
H3	0.185728	−0.033707	0.830317	0.030*
C4	0.26775(14)	−0.02183(13)	0.96112(10)	0.0265(2)
H4	0.271101	−0.115652	1.000775	0.032*
C5	0.31379(13)	0.06758(13)	1.00354(9)	0.0233(2)
H5	0.347789	0.035206	1.072873	0.028*
C6	0.31026(12)	0.20453(12)	0.94477(8)	0.0182(2)
C7	0.23807(12)	0.47525(12)	0.95017(8)	0.0181(2)
C8	0.18419(14)	0.57530(13)	1.01172(9)	0.0231(2)
H8	0.219326	0.556245	1.079707	0.028*
C9	0.07905(14)	0.70299(13)	0.97367(10)	0.0245(2)
H9	0.044495	0.772532	1.014458	0.029*
C10	0.02487(13)	0.72814(12)	0.87554(9)	0.0232(2)
H10	−0.047596	0.814998	0.849722	0.028*
C11	0.07564(12)	0.62738(12)	0.81458(9)	0.0192(2)
H11	0.035679	0.644798	0.748544	0.023*
C12	0.18521(11)	0.50078(11)	0.85041(8)	0.01602(19)
C13	0.23364(12)	0.44079(12)	0.67425(8)	0.0181(2)
H13A	0.225469	0.543140	0.648669	0.022*
H13B	0.138737	0.425090	0.660852	0.022*
C14	0.36921(11)	0.35994(11)	0.61137(8)	0.01626(19)
C15	0.35042(12)	0.33623(11)	0.51558(8)	0.01661(19)
H15	0.250375	0.363832	0.493963	0.020*
C16	0.47385(11)	0.27307(11)	0.44980(8)	0.01575(18)
C17	0.62008(11)	0.23216(12)	0.48622(8)	0.01708(19)
C18	0.64327(11)	0.24995(11)	0.58478(8)	0.01643(19)
C19	0.51495(12)	0.31593(12)	0.64503(8)	0.01711(19)
H19	0.527430	0.331262	0.710957	0.021*
C20	0.80225(12)	0.19630(12)	0.62711(9)	0.0194(2)
C21	0.91797(13)	0.26824(14)	0.55019(10)	0.0253(2)
H21A	0.928189	0.248024	0.479826	0.038*
H21B	1.017628	0.231825	0.578673	0.038*
H21C	0.881662	0.370608	0.543182	0.038*
C22	0.86204(14)	0.03489(14)	0.64205(11)	0.0285(3)
H22A	0.790828	−0.010811	0.693814	0.043*
H22B	0.962929	0.001864	0.668581	0.043*
H22C	0.870424	0.010904	0.573279	0.043*
C23	0.79426(14)	0.22772(16)	0.73665(10)	0.0283(3)
H23A	0.760883	0.330156	0.729560	0.043*
H23B	0.895654	0.189377	0.762553	0.043*
H23C	0.721137	0.183659	0.787683	0.043*
C24	0.44832(12)	0.24846(12)	0.34388(8)	0.0177(2)
C25	0.53611(14)	0.32884(14)	0.24721(9)	0.0245(2)
H25A	0.507928	0.428918	0.248429	0.037*
H25B	0.509590	0.319917	0.180637	0.037*
H25C	0.646452	0.288632	0.251311	0.037*
C26	0.27880(13)	0.30394(14)	0.32308(9)	0.0234(2)
H26A	0.217494	0.255435	0.381867	0.035*
H26B	0.267091	0.285902	0.255620	0.035*
H26C	0.243870	0.405823	0.318501	0.035*
C27	0.49549(15)	0.08921(13)	0.34730(10)	0.0261(2)
H27A	0.602782	0.048433	0.361605	0.039*
H27B	0.483012	0.075947	0.278297	0.039*
H27C	0.430669	0.042056	0.404154	0.039*

at one molecule showing a synergistic effect [4–6]. As a promising candidate, 4-((10*H*-phenothiazi-10-yl)methyl)-2,6-di-*tert*-butylphenol was synthesized.

In the title crystal structure, the molecules are stacked in layers and held together by van der Waals forces and non-classical intermolecular C—H···O hydrogen bonds.

All bond lengths and angles are in the expected ranges.

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References

1. Sheldrick, G. M.: SHELXT-Integrated space-group and crystal-structure determination. Acta Crystallogr. **A71** (2015) 3–8.
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
3. Bruker. APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA (2012).
4. Duangkaewmanee, S.; Petsom, A.: Synergistic and antagonistic effects on oxidation stability of antioxidants in a synthetic ester based oil. Tribol. Int. **44** (2011) 266–271.
5. Ksendzova, G. A.; Sorokin, V. L.; Edimecheva, I. P.; Shadyro, O. I.: Reactions of arylamine and aminophenol derivatives, and riboflavin with organic radicals. High Energ. Chem. **38** (2004) 364–368.
6. Dyubchenko, O. I.; Nikulina, V. V.; Terakh, E. I.: Synthesis and antioxidative activity of *N*,*N*-dialkyl-ω-[4-hydroxy(methoxy)aryl]alkylamines and their *N*-Oxides. Russ. J. Appl. Chem. **78** (2005) 781–786.