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S-[4-(2,2,4-Trimethylchroman-4-yl)phenyl] *N*,*N*-dimethylthiocarbamate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.076
wR factor = 0.184
Data-to-parameter ratio = 17.4

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

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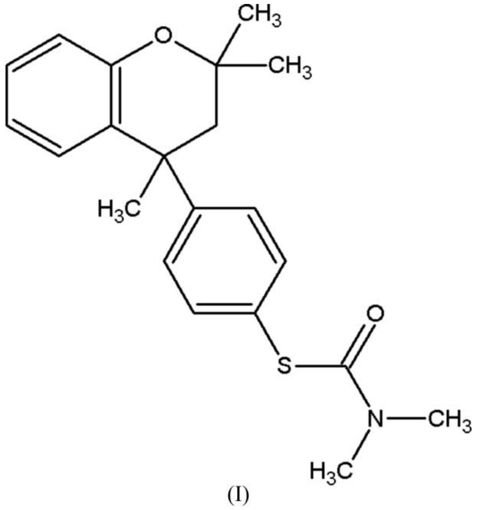
Unlike the previously reported structure of the isomer *O*-[4-(2,2,4-trimethylchroman-4-yl)phenyl] *N*,*N*-dimethylthiocarbamate [de Vries, Bredenkamp, Jacobs & Lloyd (2005). *Acta Cryst.* E**61**, o2871–o2872], which packs in amphiphilic layers, the title compound, $C_{21}H_{25}NO_2S$, (I), packs in an up–down alternating layer fashion.

Comment

Dianin's compound, 4-*p*-hydroxyphenyl-2,2,4-trimethylchroman, is part of our research focus in the field of crystal engineering (Esterhuysen *et al.*, 2005; Lloyd & Bredenkamp, 2005; Lloyd *et al.*, 2005). During the conversion of Dianin's compound to its thiol deriviative, the racemic mixture of *O*-[4-(2,2,4-trimethylchroman-4-yl)phenyl] *N*,*N*-dimethylthiocarbamate, (II), was revealed to have an interesting crystal structure that was reported by us (de Vries *et al.*, 2005). In furthering our investigation of the packing motifs employed by these derivatives of Dianin's compound, a racemic mixture of the title compound, (I), was crystallized and the structure is reported here (Fig. 1).



(I)

Unlike compound (II), compound (I) crystallizes in the triclinic space group $P\overline{1}$. As for compound (II), the unit-cell parameters include a relatively short axis of 6.8812 (13) Å. In the absence of any appreciable interactions, the packing of (I) is expected to be similar to that in (II). The electrostatic interactions of the dimethylthiocarbamate groups, recognized as the determining packing force in (II), also appear to be a structurally important factor in (I) (Fig. 2). However, whereas (II) packs in two-dimensional amphiphilic type layers, (I) forms alternating up–down layers of one-dimensional amphi-

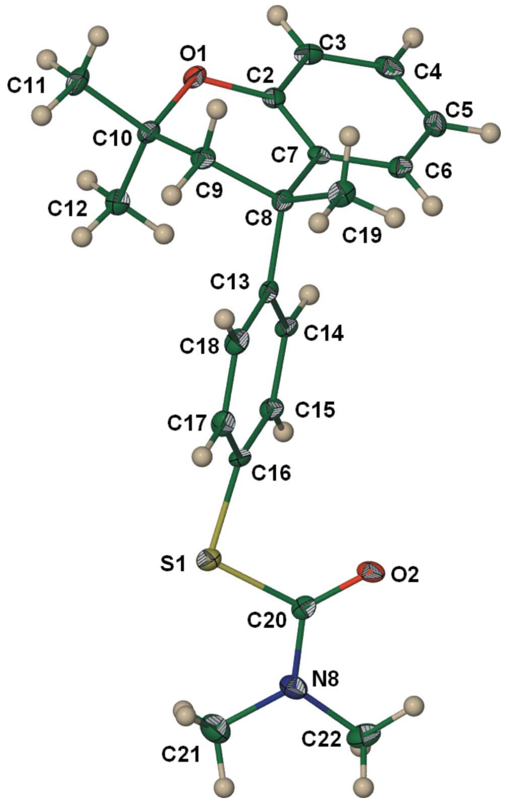


Figure 1
The molecular structure of (I), showing the atom labels and 50% probability displacement ellipsoids for non-H atoms.

philic strands, seen clearly when viewed along [100] (Fig. 3). The variation in packing can be explained in terms of close-packing effects. In compound (II), the exposed S atom is considerably larger than the exposed O atom of (I) (Fig. 4).

Experimental

Compound (I) was prepared from compound (II) according to the literature method of thermal rearrangement (Hardy *et al.*, 1979; de Vries *et al.*, 2005)). Crystals were grown by slow evaporation of a concentrated methanolic solution of (I).

Crystal data

$C_{21}H_{25}NO_2S$
$M_r = 355.48$
Triclinic, $P\bar{1}$
$a = 6.8812\ (13)$ Å
$b = 11.542\ (2)$ Å
$c = 12.747\ (2)$ Å
$\alpha = 107.797\ (3)^\circ$
$\beta = 98.907\ (3)^\circ$
$\gamma = 100.972\ (3)^\circ$
$V = 921.5\ (3)$ Å^3
$Z = 2$
$D_x = 1.281$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1899 reflections
$\theta = 3.1$–27.8°
$\mu = 0.19$ mm^{-1}
$T = 100\ (2)$ K
Block, colourless
$0.27 \times 0.18 \times 0.17$ mm

Data collection

Bruker APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
5615 measured reflections
3972 independent reflections
3094 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$
$\theta_{\max} = 28.1^\circ$
$h = -6 \rightarrow 9$
$k = -15 \rightarrow 14$
$l = -16 \rightarrow 16$

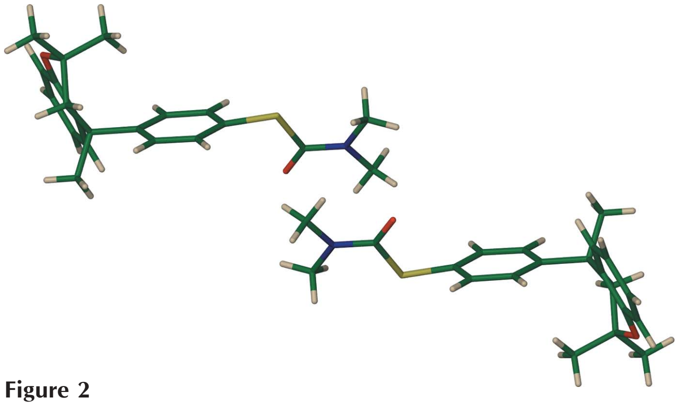


Figure 2
The packing of two molecules of compound (I), showing the electrostatic interactions between proximal dimethylthiocarbamate groups. Molecules are shown in capped-stick representation.

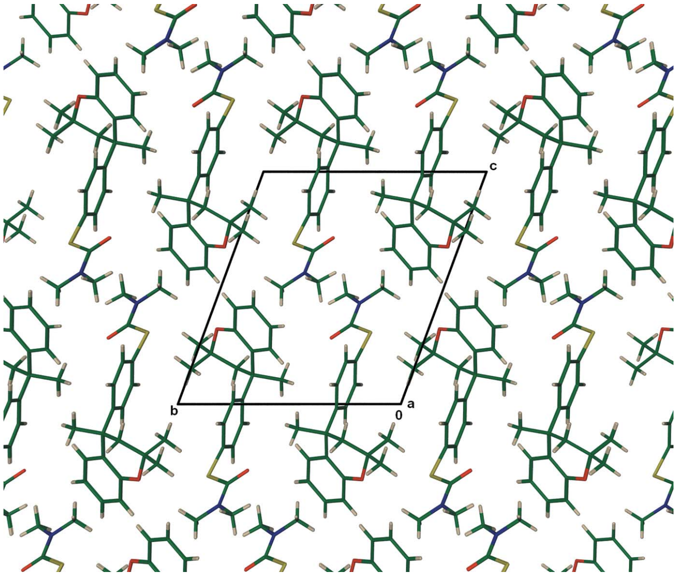
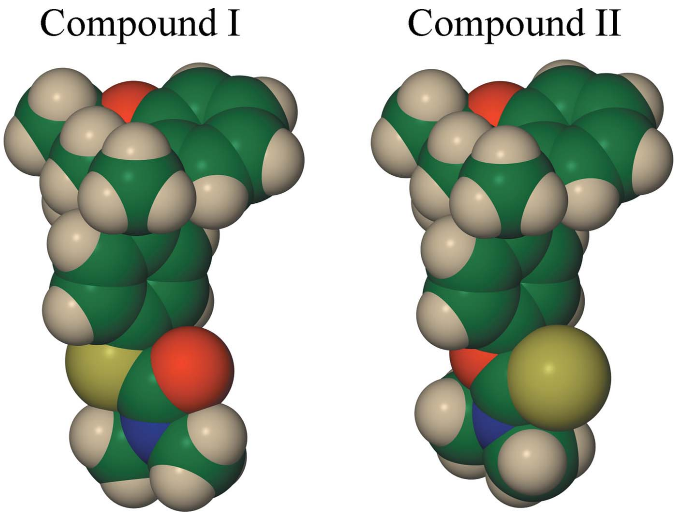


Figure 3
The alternating layer of one-dimensional amphiphilic strands in the structure of (I). Molecules are shown in capped-stick representation.



Compound I

Compound II

Figure 4
Size comparison between compounds (I) and (II), both shown in space-filling representation.

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Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.076$
$wR(F^2) = 0.184$
$S = 1.08$
3972 reflections
228 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0887P)^2 + 0.6231P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.71$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

All H atoms were positioned geometrically (C—H = 0.95, 0.98 and 0.99 Å for aromatic, methyl and CH_2 groups, respectively) and constrained to ride on their parent atoms; $U_{iso}(H)$ values were set at $1.2U_{eq}(C)$ for CH_2 and aromatic groups, and at $1.5U_{eq}(C)$ for CH_3 groups.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001; Atwood & Barbour, 2003); software used to prepare material for publication: *X-SEED*.

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