Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

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

Elise J. C. de Vries,* Martin W. Bredenkamp, Tia Jacobs and Gareth O. Lloyd

Department of Chemistry, University of Stellenbosch, Private Bag X1, Matieland 7602, South Africa

Correspondence e-mail: ejcdv@sun.ac.za

Received 5 July 2005
Accepted 3 August 2005
Online 12 August 2005

Key indicators

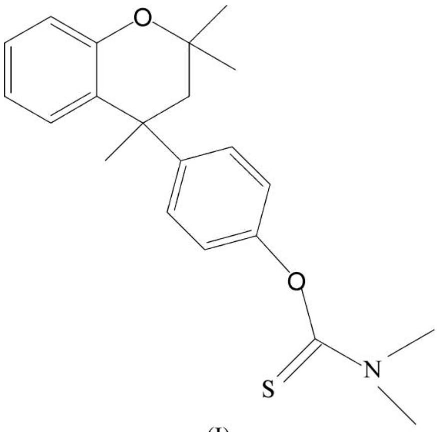
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.071
wR factor = 0.161
Data-to-parameter ratio = 19.0

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

The title compound, $C_{21}H_{26}NO_2S$, was obtained during the synthesis of the thiol derivative of Dianin's compound. Unlike Dianin's compound and the thiol derivatives, the title compound revealed no strong hydrogen-bonding interactions.

Comment

4-*p*-Hydroxyphenyl-2,2,4-trimethylthiochroman, commonly known as Dianin's compound, is one of the focus areas of our research (Esterhuysen *et al.*, 2005; Lloyd & Bredenkamp, 2005; Lloyd *et al.*, 2005). Dianin's compound and its thiol derivatives are particularly interesting because of the role of hydrogen bonding in clathrate formation. In Dianin's compound, six hydroxyl molecules are linked to each other through a network of O—H···O hydrogen bonds such that the O atoms form a near-planar hexagon. These hexametric units stack directly above each other to form long chains (with hourglass-shaped repeat units) parallel to the *c* axis. The thiol analogue behaves analogously.



(I)

During the synthesis of 4-*p*-mercaptophenyl-2,2,4-trimethylthiochroman (the thiol derivative) from Dianin's compound, one of the intermediate products, (I), crystallized. X-ray diffraction data were collected on the dimethylthiocarbamate intermediate compound at low temperature (Fig. 1).

Compound (I) crystallized in the monoclinic space group *C*2/*c* with a relatively short *b* axis of 6.6341 (13) Å. A thorough investigation revealed that no strong or weak hydrogen-bonding interactions occur within the crystal structure. The molecules pack in layers parallel to the short *b* axis. The most probable intermolecular interactions present are the electrostatic interactions (dipole–dipole) between the dimethylthiocarbamate groups of neighbouring molecules (Fig. 2). This is

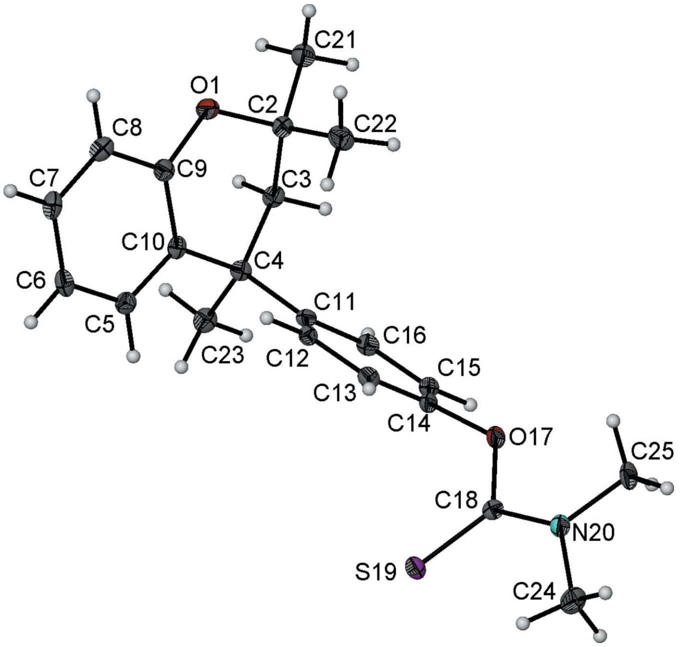


Figure 1
The molecular structure of (I), showing atom labels and 50% probability ellipsoids for non-H atoms.

best visualized when the dimethylthiocarbamate groups are orientated towards one another and the more 'hydrophobic' Dianin's compound groups are oriented together showing an amphiphilic type of packing.

Experimental

Compound (I) was prepared by adding Dianin's compound (11.33 g, 0.04 mol) to a solution of sodium metal (1.07 g, 0.05 mol) in absolute ethanol (40 ml) under argon. Complete dissolution occurred on heating. Removal of the solvent under reduced pressure gave the sodium salt of Dianin's compound. Dimethylthiocarbamoyl chloride (8.41 g, 0.07 mol) in dimethylformamide (56 ml) was added dropwise to the salt at 283 K under argon. Thereafter the reaction mixture was stirred for 1.5 h at 313–328 K. Upon cooling, the mixture was added to water (120 ml) and extracted with benzene/petroleum-ether (4:1) in 4 × 100 ml portions. The combined organic extracts were washed with water, 5% NaOH solution and NaCl solution, 3 × 100 ml portions in each case. The dried solution ($MgSO_4$) was evaporated under reduced pressure to give a yellow oil (Hardy *et al.*, 1979). Upon recrystallization from methanol, colourless crystals formed (yield 64.3%).

Crystal data

$C_{21}H_{25}NO_2S$
$M_r = 355.48$
Monoclinic, $C2/c$
$a = 41.017$ (8) Å
$b = 6.6341$ (13) Å
$c = 14.048$ (3) Å
$\beta = 99.733$ (3)°
$V = 3767.6$ (13) Å^3
$Z = 8$

$D_x = 1.253$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 4336 reflections
$\theta = 2.0$–$28.3°$
$\mu = 0.19$ mm^{-1}
$T = 100$ (2) K
Rectangular block, colourless
0.30 × 0.20 × 0.10 mm

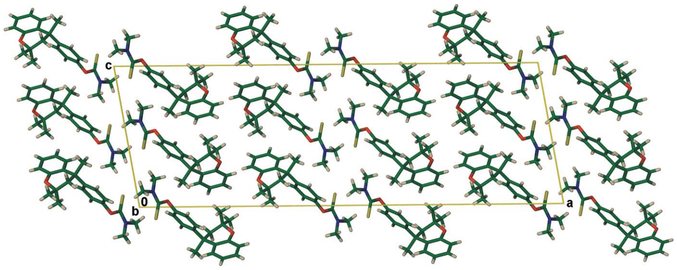


Figure 2
The crystal structure packing diagram for (I), showing the amphiphilic character, viewed along [010].

Data collection

Bruker APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.884$, $T_{\max} = 0.982$
10853 measured reflections

4336 independent reflections
3699 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 28.3°$
$h = -53 \rightarrow 53$
$k = -8 \rightarrow 8$
$l = -13 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.161$
$S = 1.18$
4336 reflections
228 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 8.1707P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.75$ e Å^{-3}
$\Delta\rho_{\min} = -0.35$ e Å^{-3}

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

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* (Atwood & Barbour, 2003; Barbour, 2001); software used to prepare material for publication: *X-SEED*.

The authors thank the National Research Fund of South Africa for its financial support.

References

Atwood, J. L. & Barbour, L. J. (2003). *Cryst. Growth Des.* **3**, 3–8.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Bruker (2001). *SMART*. Version 5.625. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2002). *SAINT*. Version 6.36a. Bruker AXS Inc., Madison, Wisconsin, USA.
Esterhuysen, C., Bredenkamp, M. W. & Lloyd, G. O. (2005). *Acta Cryst.* C**61**, o32–o34.
Hardy, A. D. U., McKendrick, J. J., MacNicol, D. D. & Wilson, D. R. (1979). *J. Chem. Soc. Perkin Trans. II*, pp. 729–734.
Lloyd, G. O. & Bredenkamp, M. W. (2005). *Acta Cryst.* E**61**, o1512–o1514.
Lloyd, G. O., Bredenkamp, M. W. & Barbour, L. J. (2005). *Chem. Commun.* **32**, 4053.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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