

6-Hexyl-7-hydroxy-3-(3-phenyl-1*H*-1,2,4-triazol-5-yl)-2*H*-chromen-2-one monohydrateVyacheslav N. Baumer,^{a*}
Sergiy M. Kovalenko,^b
Kostyantyn M. Sytnyk^b and
Valentyn P. Chernykh^c^aInstitute for Single Crystals, National Academy of Sciences of Ukraine, 60 Lenin ave., Kharkiv 61001, Ukraine, ^bDepartment of Organic Chemistry, Ukrainian National University of Pharmacy, 4 Blyukher str., Kharkiv 61002, Ukraine, and ^cUkrainian National University of Pharmacy, 4 Blyukher str., Kharkiv 61002, UkraineCorrespondence e-mail:
baumer@xray.isc.kharkov.com

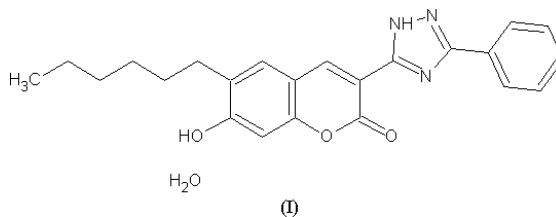
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ (C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.099
Data-to-parameter ratio = 14.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

On heating *N'*-benzoyl-7-hydroxy-6-hexyl-2-oxo-2*H*-chromene-3-carbohydrazonamide in dimethylformamide, the process of cyclization leads to the formation of a triazole derivative of a 3-substituted chromenone, C₂₃H₂₃N₃O₃·H₂O, as confirmed by this crystal structure investigation. The three ring systems are almost coplanar and, in the crystal structure, a three-dimensional network of hydrogen bonds is formed; these are of the types O–H···O, N–H···N, O–H···N and C–H···O.

Comment

In a continuation of our previous investigations on new approaches to the synthesis of 3-substituted chromenone derivatives (Kovalenko *et al.*, 1996), the title compound, (I), was synthesized by heating *N'*-benzoyl-7-hydroxy-6-hexyl-2-oxo-2*H*-chromene-3-carbohydrazonamide in dimethylformamide (DMF). Since a path of cyclization was not known *a priori*, an X-ray crystallographic investigation was carried out to determine the molecular structure of the product. The results of the present study show that, under the above reaction conditions, a substituted 3-(1,2,4-triazol-5-yl)-2*H*-chromen-2-one is formed.



The three ring systems are almost coplanar. The chromenone moiety (O1, C2–C10) is planar to within 0.025 Å, the maximum deviations being 0.035 (1) and –0.038 (1) Å for atoms O1 and C8, respectively. Atoms O2 and O3 are displaced from this plane by 0.073 (2) and –0.103 (2) Å, respectively. The triazole ring is planar to within 0.004 Å, and is rotated by 3.06 (9)° with respect to the chromenone plane. The phenyl ring makes a dihedral angle of 12.93 (11)° with the triazole ring. These dihedral angles, together with the torsion angles C4–C3–C11–N1, C2–C3–C11–N2, N1–C12–C13–C18 and N3–C12–C13–C14 (Table 1) show that the three rings are slightly rotated with respect to each other. This arrangement may be influenced by the intramolecular N2–H2···O2 hydrogen bond (Table 2) and short intramolecular contacts N1···H4 (2.63 Å), N1···H18 (2.64 Å) and N2···H14 (2.59 Å); the van der Waals radii for N and H atoms are 1.55 and 1.20 Å, respectively (Bondi, 1964).

Received 19 July 2004

Accepted 22 July 2004

Online 31 July 2004

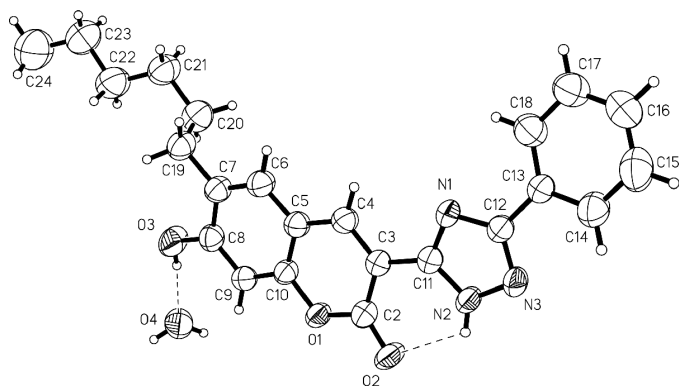


Figure 1
A view of (I), showing displacement ellipsoids drawn at the 50% probability level and the atom-numbering scheme. Hydrogen bonds are indicated by dashed lines.

The first atom of the hexyl chain, C19, deviates from the chromenone plane by 0.060 (2) Å; the hexyl C atoms are coplanar to within 0.024 Å, and this plane makes a dihedral angle of 20.3 (2)° with the chromenone plane.

The three-dimensional system of hydrogen bonds observed in the crystal structure involves both inter- and intramolecular hydrogen bonds (Table 2).

Experimental

6-Hexyl-7-hydroxy-3-(3-phenyl-1*H*-1,2,4-triazol-5-yl)-2*H*-chromen-2-one was prepared by a known literature procedure (Kovalenko *et al.*, 1996). A solution of *N*'-benzoyl-6-hexyl-7-hydroxy-2-oxo-2*H*-chromene-3-carbohydrazonamide (1 mmol) in DMF (10 ml) was refluxed for 30 min. On completion of the reaction, the mixture was cooled and the resulting precipitate was filtered off, washed with water, cold propan-2-ol (2 × 5 ml) and recrystallized from ethanol–water (1:1) to give 6-hexyl-7-hydroxy-3-(3-phenyl-1*H*-1,2,4-triazol-5-yl)-2*H*-chromen-2-one (yield 68%). Crystals of the title compound were grown by evaporation of an ethanol–water solution of the product.

Crystal data

C₂₃H₂₃N₃O₃·H₂O
M_r = 407.46
 Triclinic, *P* $\bar{1}$
a = 9.465 (2) Å
b = 10.316 (2) Å
c = 12.518 (3) Å
 α = 79.521 (17)°
 β = 88.450 (17)°
 γ = 64.259 (17)°
V = 1080.7 (4) Å³

Z = 2
D_x = 1.252 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 24 reflections
 θ = 11.0–12.0°
 μ = 0.09 mm⁻¹
T = 293 (2) K
 Block, light yellow
 0.45 × 0.20 × 0.15 mm

Data collection

Siemens *P3/PC* diffractometer
 2θ/θ scans
 Absorption correction: none
 4168 measured reflections
 3991 independent reflections
 2430 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.073

θ _{max} = 25.5°
h = -10 → 11
k = -10 → 12
l = 0 → 14
 2 standard reflections every 98 reflections
 intensity decay: 1%

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.045
wR(*F*²) = 0.099
S = 1.01
 3991 reflections
 272 parameters

H-atom parameters constrained
w = 1/[σ²(*F_o*²) + (0.0345*P*)²]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.13 e Å⁻³
 Δρ_{min} = -0.13 e Å⁻³

Table 1

Selected torsion angles (°).

| | | | |
|--------------|--------------|----------------|-------------|
| C4–C3–C11–N2 | −176.68 (14) | N3–C12–C13–C18 | 165.86 (14) |
| C2–C3–C11–N2 | 4.1 (2) | N1–C12–C13–C18 | −11.3 (2) |
| C4–C3–C11–N1 | 2.1 (2) | N3–C12–C13–C14 | −12.4 (2) |
| C2–C3–C11–N1 | −177.19 (13) | N1–C12–C13–C14 | 170.45 (14) |

Table 2

Hydrogen-bonding geometry (Å, °).

| <i>D</i> –H... <i>A</i> | <i>D</i> –H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> –H... <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| O3–H3...O4 | 0.92 | 1.81 | 2.701 (2) | 162 |
| N2–H2...O2 | 0.86 | 2.23 | 2.779 (2) | 121 |
| N2–H2...N3 ⁱ | 0.86 | 2.35 | 2.960 (2) | 129 |
| O4–H4A...N1 ⁱⁱ | 0.89 | 2.10 | 2.981 (2) | 168 |
| O4–H4B...O2 ⁱⁱⁱ | 0.89 | 2.34 | 3.206 (2) | 167 |
| C14–H14...O2 ⁱ | 0.93 | 2.46 | 3.304 (2) | 151 |

Symmetry codes: (i) −1 − *x*, 1 − *y*, 1 − *z*; (ii) *x*, 1 + *y*, *z*; (iii) −*x*, 2 − *y*, 1 − *z*.

All H atoms were located in a difference map and treated as riding, with N–H = 0.86 Å, O–H in the range 0.89–0.92 Å and C–H in the range 0.93–0.97 Å. *U*_{iso}(H) values were set equal to 1.2*U*_{eq} of the carrier atom.

Data collection: *P3* (Siemens, 1989); cell refinement: *P3*; data reduction: *XDISK* (Siemens, 1991) and *XPREP* (Siemens, 1991); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1991); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported in part by the Ministry of Education of Ukraine. VB acknowledges the ICDD for financial support (grant #03-02).

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
 Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–446.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Kovalenko, S. N., Zubkov, V. A., Chernykh, V. P., Turov, A. V. & Ivkov, S. M. (1996). *Khim. Get. Soedin. SSSR*, pp. 186–192. (In Russian.)
 Sheldrick, G. M. (1997). *SHELXL97*. Release 97-2. University of Göttingen, Germany.
 Siemens (1989). *P3*. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.
 Siemens (1991). *XDISK*, *XPREP* and *XP*. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.