was chosen on the basis of statistical tests and confirmed by subsequent results. In the β form, the observed systematic absences $0kl$, $k = 2n + 1$, $h0l$, $l = 2n + 1$, and $hk0$, $h + k = 2n + 1$, indicated space group $Pbcn$. Computations were performed using $CRYSTALS$ (Watkin, Carruthers & Betteridge, 1988) adapted on a MicroVAXII. Scattering factors for all atoms were as incorporated in $CRYSTALS$. The structures were solved by direct methods. All remaining non-H atoms were found by successive electron-density map calculations. For the β (orthorhombic) form, the density map showed two prominent but unequal peaks, one on either side of the plane. The only satisfactory explanation of this was the assumption of a disordered model. Fractional oxygen, referred to as O(1) and O(2), was assigned to these two positions, with occupancy factors summing to unity and with the same isotropic displacement parameters. The refinement of occupancy led to 0.69 for O(1) and 0.31 for O(2). Atomic coordinates were refined together with anisotropic displacement parameters [isotropic for O(2)]. At this stage, the H atoms were located on a difference electron-density map, their coordinates refined with an overall isotropic displacement parameter and then included as fixed contributors for further structure-factor calculations. Least-squares refinements were carried out by minimizing the function $\Sigma w(|F_o| - |F_c|)^2$. The models reached convergence with R and wR having the values listed. The criteria for a satisfactory complete analysis were the ratios of the r.m.s. shift to standard deviation being less than 0.1 and there being no significant features in the final difference maps.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1162). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Second Triclinic Polymorph of Coumarin 153

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Abstract

Coumarin 153, 2,3,6,7-tetrahydro-9-(trifluoromethyl)-$1H,5H,11H$-[1]benzopyrano[6,7,8-ij]quinolizin-11-one, $C_{16}H_{14}F_3NO_2$, was found to exist in a new polymorphic form in the triclinic system. There is an earlier report of a crystal structure determination of this compound, also in the triclinic system, but with a different cell volume and packing mode [Gridunova *et al.* (1992). *Kristallografiya*, **37**, 366–372]. There are three molecules in the asymmetric unit of the title polymorph compared with two in the latter case; all three molecules show disorder in the piperidine ring system. The molecules are planar and are stacked parallel to the (011) plane. The crystal structure is stabilized by C—H$\cdots$O hydrogen bonds.

Comment

The title compound, (I), a rigidized aminocoumarin, has attracted extensive study since it has been used as a laser dye (Maeda, 1984; Priyadarsini, Naik & Moorthy, 1989; Kunjappu, 1993; McCarthy & Blanchard, 1993). The crystal structure determination of this compound was undertaken as part of our structural studies on laser dyes. In the present study, we found coumarin 153 to crystallize in a new triclinic unit cell (space group $P\bar{1}$), with three molecules, A, B and C, in the asymmetric unit (Fig. 1). This is different from the structure reported earlier by Gridunova *et al.* (1992), which has a smaller triclinic unit cell (also $P\bar{1}$) and only two molecules in the asymmetric unit. This compound has also been found to crystallize in the orthorhombic system (Sivakumar & Chinnakali, 1991). Recently,

(I)

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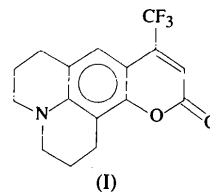
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polymorphism has also been observed in other amino-coumarin derivatives (Jasinski & Paight, 1994; Jasinski & Woudenberg, 1995).

Bond lengths, angles and selected torsion angles of the three molecules of the title compound are listed in Table 2. The numbering scheme has been kept the same for all three molecules, the second positions of the same atom being represented by primes (*e.g.* C17 and C17′). Bond lengths and angles in the coumarin rings of these three molecules are normal and are in agreement with those observed in other rigidized coumarins, *e.g.* coumarin 480 (Chinnakali, Sivakumar & Natarajan, 1990*a*) and coumarin 314 (Yip, Fun, Sivakumar, Zhou, Shawkataly & Teoh, 1995), and with the Gridunova polymorph. The coumarin rings are planar, with maximum deviations of 0.087 (2), 0.058 (2) and 0.009 (2) Å for atom O11 in molecules *A*, *B* and *C*, respectively. The angles between the least-squares planes of the pyrone and benzene rings of the coumarin moieties are 2.88 (6), 2.24 (5) and 1.09 (7)° for molecules *A*, *B* and *C*, respectively. This apparently shows that for molecules with disorder of both piperidine rings, there is a greater deviation from planarity of their coumarin moieties.
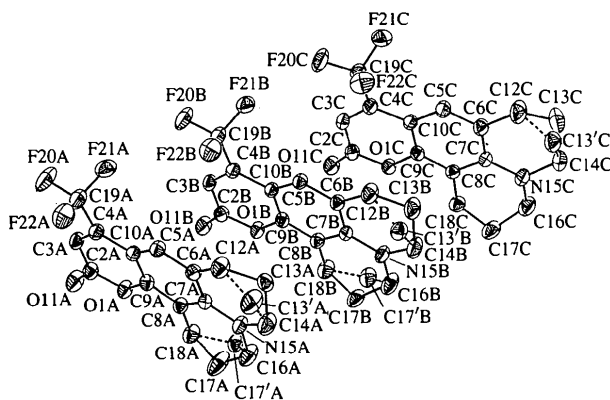

Fig. 1. The structure of coumarin 153 showing the atom-numbering scheme and 30% probability displacement ellipsoids.

The molecules were found to have disorder in the piperidine rings; for molecules *A* and *B*, both the piperidine rings are disordered at atoms C13 and C17, whereas molecule *C* has disorder only at atom C13. In the Gridunova polymorph, one molecule has disorder for both atoms C13 and C17, while the other has disorder only at atom C13. The same kind of disorder observed for molecule *C* was found to be present in coumarin 337 (Chinnakali, Selladurai, Sivakumar, Subramanian & Natarajan, 1990). So, atom C13 apparently has a greater chance of being disordered than C17. In other words, the piperidine ring comprising atom C13 has more tendency towards a flip-flop between the up/down sofa conformation than the other ring containing atom C17. The conformations of the piperidine rings in

the present structure are defined by the asymmetry parameters (Nardelli, 1983*a*) given in Table 3.

The average C—F bond length of the trifluoro-methyl group in molecules *A*, *B* and *C* is 1.334 (6) Å; the F—C—F and F—C—C angles have average values of 106.3 (5) and 112.5 (6)°, respectively (1.335 Å, 106.6 and 112.2° for the Gridunova polymorph). These values also agree with the corresponding values in the structures of coumarin 485 (7-dimethyl-amino-4-trifluoromethylcoumarin; Chinnakali, Sivakumar & Natarajan, 1990*b*) and coumarin 503 (7-ethyl-amino-6-methyl-4-trifluoromethylcoumarin; Chinnakali, Sivakumar & Natarajan, 1992). A considerable difference has been observed in the positioning of the F atoms of the trifluoromethyl groups in our structure compared with that of the Gridunova polymorph, where one of the F atoms lies in the plane of the coumarin moiety and the other two are separated by 120°. In our case, the trifluoromethyl group has been rotated about the C4—C19 bond by different magnitudes for the three molecules *A*, *B* and *C* (see torsion angles C3—C4—C19—F20/F21/F22 in Table 2). As a result of this, the F···F short contact distance between the molecules is longer in our case [F21*A*···F20*B* 3.029 (2) and F21*B*···F20*C* 2.961 (2) Å] compared with the value of 2.854 (4) Å in the Gridunova polymorph.

Molecules *A* and *C* are linked by two weak C—H···O hydrogen bonds [C3*A*—H3*A* 0.87 (3), H3*A*···O11*C*^{i} 2.50 (3), C3*A*···O11*C*^{i} 3.355 (3) Å, C3*A*—H3*A*···O11*C*^{i} 170 (2)°; C3*C*—H3*C* 0.87 (2), H3*C*···O11*A*^{i} 2.46 (2), C3*C*···O11*A*^{i} 3.313 (3) Å, C3*C*—H3*C*···O11*A*^{i} 170 (2)°; symmetry code: (i) $-x$, $-y$, $-z+1$] and form layers parallel to the (011) plane. Molecule *B* is also linked to an adjacent molecule *B*^{i} by hydrogen bonds of the same type [C3*B*—H3*B* 0.93 (3), H3*B*···O11*B*^{i} 2.42 (3), C3*B*···O11*B*^{i} 3.346 (3) Å, C3*B*—H3*B*···O11*B*^{i} 174 (2)°].

A comparison of packing modes in the two triclinic polymorphs is shown in Fig. 2. It is evident that in our structure, the stacking of the molecules is 3 × 3, whereas in the Gridunova polymorph it is 2 × 2, according to the number of molecules present in an asymmetric unit. In the crystal lattice, the molecules are stacked in two-dimensional layers parallel to the (011) planes (Fig. 3). We have discussed in our earlier paper (Yip, Fun, Sivakumar, Zhou, Shawkataly & Teoh, 1995) the fact that the rigidized aminocoumarin molecules are always stacked in parallel planes, with an interlayer distance of around 3.6 Å (coumarin 480 3.673, coumarin 337 3.612 and coumarin 314 3.624 Å). The present structure also falls into the same category, with an interlayer distance of 3.768 Å. The slight increase in the distance is justified by the presence of the trifluoromethyl group at C4. In the Gridunova polymorph, the layers are found to be parallel to the (111) planes, with a separation of 3.779 Å, and are cross linked by F···F (2.854 Å) short contacts between the F atoms of molecules *A* and *B*.
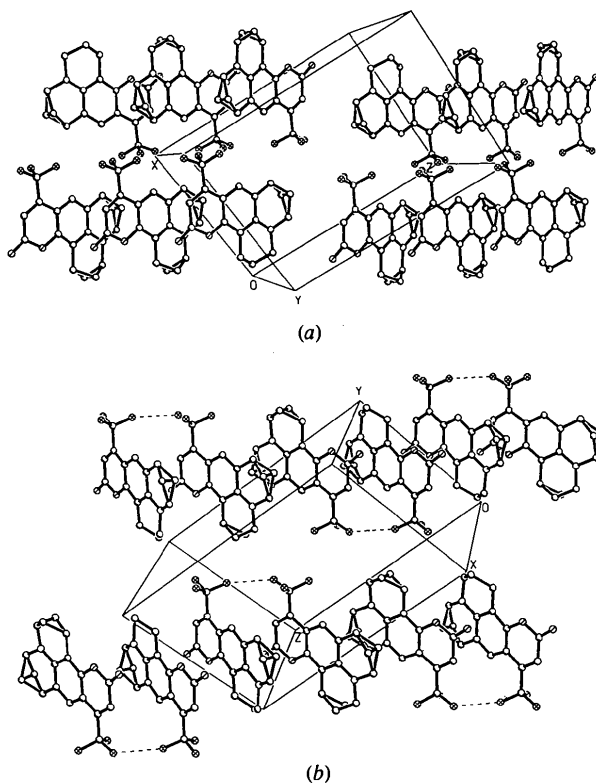
Fig. 2. The packing of coumarin $15\overline{3}$ molecules (*a*) in the present study and (*b*) in the study of Gridunova *et al.* (1992).
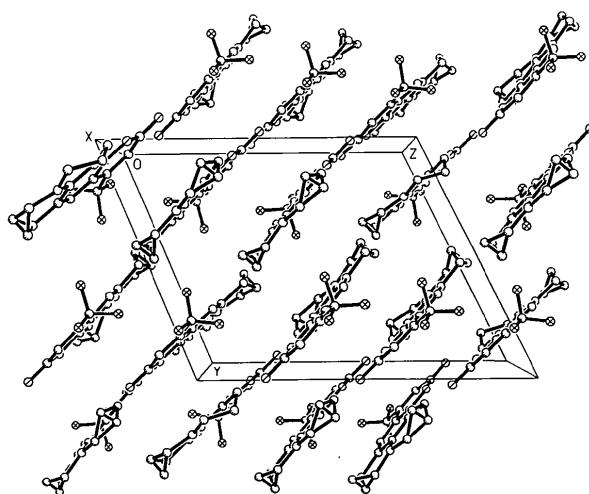
Fig. 3. The packing of the molecules viewed down the *a* axis.

Experimental

The compound was purchased from the Sigma Chemical Company and recrystallized from a mixture of methanol and dichloromethane by slow evaporation.

Crystal data

$C_{16}H_{14}F_3NO_2$	Mo $K\alpha$ radiation
$M_r = 309.28$	$\lambda = 0.71073$ Å

Triclinic
$P\overline{1}$
$a = 12.068\,(1)$ Å
$b = 13.137\,(1)$ Å
$c = 15.374\,(1)$ Å
$\alpha = 66.76\,(1)°$
$\beta = 89.16\,(1)°$
$\gamma = 68.82\,(1)°$
$V = 2064.9\,(3)$ Å^3
$Z = 6$
$D_x = 1.492$ Mg m^{-3}

Cell parameters from 40 reflections
$\theta = 5-12.5°$
$\mu = 0.125$ mm^{-1}
$T = 293\,(2)$ K
Transparent block
$0.70 \times 0.38 \times 0.32$ mm
Light green

Data collection

Siemens *P*4 four-circle diffractometer
$\theta-2\theta$ scans
Absorption correction: none
7958 measured reflections
6863 independent reflections
4184 observed reflections
$[I > 2\sigma(I)]$

$R_{int} = 0.0124$
$\theta_{max} = 25.0°$
$h = -1 \rightarrow 13$
$k = -13 \rightarrow 14$
$l = -18 \rightarrow 18$
3 standard reflections
monitored every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0419$
$wR(F^2) = 0.1332$
$S = 0.934$
6863 reflections
690 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0818P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.001$

$\Delta\rho_{max} = 0.241$ e Å^{-3}
$\Delta\rho_{min} = -0.146$ e Å^{-3}
Extinction correction:
*SHELXL*93 (Sheldrick, 1993)
Extinction coefficient:
0.0041 (7)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
O1*A*	0.03097 (12)	0.12158 (12)	0.68120 (9)	0.0531 (4)
C2*A*	−0.0621 (2)	0.0872 (2)	0.71270 (14)	0.0521 (5)
C3*A*	−0.1716 (2)	0.1463 (2)	0.64651 (15)	0.0515 (5)
C4*A*	−0.1846 (2)	0.2355 (2)	0.56038 (13)	0.0452 (5)
C5*A*	−0.0915 (2)	0.3695 (2)	0.44411 (13)	0.0445 (5)
C6*A*	0.0044 (2)	0.4028 (2)	0.42201 (13)	0.0432 (4)
C7*A*	0.1137 (2)	0.3370 (2)	0.48768 (13)	0.0424 (4)
C8*A*	0.1203 (2)	0.2406 (2)	0.57457 (13)	0.0448 (5)
C9*A*	0.0203 (2)	0.2134 (2)	0.59304 (12)	0.0420 (4)
C10*A*	−0.0882 (2)	0.2743 (2)	0.52998 (12)	0.0409 (4)
O11*A*	−0.04429 (14)	0.01096 (15)	0.79283 (10)	0.0764 (5)
C12*A*	−0.0046 (2)	0.5076 (2)	0.33026 (14)	0.0611 (6)
C13*A*	0.1152 (5)	0.5016 (5)	0.3008 (4)	0.0502 (13)
C13′*A*	0.0904 (6)	0.5477 (6)	0.3273 (5)	0.083 (2)
C14*A*	0.2055 (2)	0.4698 (2)	0.3809 (2)	0.0739 (7)
N15*A*	0.21032 (14)	0.36851 (15)	0.46654 (12)	0.0525 (4)
C16*A*	0.3177 (2)	0.3098 (3)	0.5370 (2)	0.0838 (8)
C17*A*	0.3143 (6)	0.2297 (7)	0.6326 (5)	0.102 (2)
C17′*A*	0.3417 (5)	0.1857 (5)	0.5954 (4)	0.0580 (14)

$C_{16}H_{14}F_3NO_2$

C18A	0.2339 (2)	0.1695 (2)	0.64682 (15)	0.0635 (6)
C19A	−0.3035 (2)	0.2988 (2)	0.4974 (2)	0.0608 (6)
F20A	−0.38075 (12)	0.24713 (14)	0.53138 (10)	0.0953 (5)
F21A	−0.29585 (12)	0.30496 (13)	0.40902 (9)	0.0782 (4)
F22A	−0.35623 (12)	0.41287 (13)	0.48687 (10)	0.0869 (4)
O1B	0.24348 (11)	0.09188 (11)	0.40690 (8)	0.0497 (3)
C2B	0.1490 (2)	0.0613 (2)	0.44185 (13)	0.0481 (5)
C3B	0.0369 (2)	0.1219 (2)	0.37748 (14)	0.0477 (5)
C4B	0.0246 (2)	0.2069 (2)	0.28909 (13)	0.0436 (5)
C5B	0.1203 (2)	0.3327 (2)	0.16646 (13)	0.0417 (4)
C6B	0.2180 (2)	0.3611 (2)	0.14067 (12)	0.0404 (4)
C7B	0.3286 (2)	0.2952 (2)	0.20517 (12)	0.0396 (4)
C8B	0.3346 (2)	0.2028 (2)	0.29436 (12)	0.0418 (4)
C9B	0.2324 (2)	0.1801 (2)	0.31722 (12)	0.0396 (4)
C10B	0.1228 (2)	0.2422 (2)	0.25489 (12)	0.0387 (4)
O11B	0.16790 (14)	−0.01381 (13)	0.52228 (10)	0.0667 (4)
C12B	0.2104 (2)	0.4615 (2)	0.04600 (14)	0.0588 (6)
C13B	0.3260 (5)	0.4582 (5)	0.0160 (3)	0.0680 (14)
C13′B	0.3003 (4)	0.5116 (4)	0.0483 (3)	0.0515 (11)
C14B	0.4236 (2)	0.4173 (2)	0.0900 (2)	0.0689 (7)
N15B	0.42667 (14)	0.3235 (2)	0.18063 (11)	0.0517 (4)
C16B	0.5389 (2)	0.2595 (2)	0.2454 (2)	0.0789 (8)
C17B	0.5311 (5)	0.1946 (6)	0.3450 (4)	0.069 (2)
C17′B	0.5557 (4)	0.1413 (5)	0.3141 (4)	0.0668 (15)
C18B	0.4505 (2)	0.1300 (2)	0.36340 (15)	0.0634 (6)
C19B	−0.0949 (2)	0.2697 (2)	0.22602 (14)	0.0534 (5)
F20B	−0.17262 (11)	0.21968 (14)	0.26321 (9)	0.0865 (5)
F21B	−0.08698 (11)	0.26885 (11)	0.13970 (8)	0.0649 (4)
F22B	−0.14567 (11)	0.38549 (12)	0.21086 (10)	0.0781 (4)
O1C	0.46182 (11)	0.04380 (11)	0.12833 (8)	0.0478 (3)
C2C	0.3652 (2)	0.0185 (2)	0.16454 (14)	0.0501 (5)
C3C	0.2518 (2)	0.0835 (2)	0.10177 (15)	0.0503 (5)
C4C	0.2415 (2)	0.1638 (2)	0.01182 (13)	0.0453 (5)
C5C	0.3440 (2)	0.2698 (2)	−0.11797 (13)	0.0518 (5)
C6C	0.4450 (2)	0.2898 (2)	−0.14686 (13)	0.0531 (5)
C7C	0.5548 (2)	0.2256 (2)	−0.08209 (13)	0.0439 (5)
C8C	0.5575 (2)	0.1427 (2)	0.01128 (12)	0.0411 (4)
C9C	0.4530 (2)	0.1261 (2)	0.03594 (12)	0.0398 (4)
C10C	0.3434 (2)	0.1883 (2)	−0.02603 (13)	0.0415 (4)
O11C	0.38396 (14)	−0.05589 (14)	0.24557 (10)	0.0703 (5)
C12C	0.4422 (2)	0.3781 (3)	−0.2463 (2)	0.0889 (9)
C13C	0.5548 (4)	0.3553 (6)	−0.2791 (4)	0.080 (2)
C13′C	0.5409 (5)	0.4159 (5)	−0.2569 (4)	0.0659 (15)
C14C	0.6598 (2)	0.3170 (2)	−0.2093 (2)	0.0746 (7)
N15C	0.65571 (15)	0.2460 (2)	−0.11011 (12)	0.0560 (5)
C16C	0.7700 (2)	0.1755 (3)	−0.0472 (2)	0.0741 (8)
C17C	0.7555 (2)	0.1408 (3)	0.0556 (2)	0.0777 (8)
C18C	0.6718 (2)	0.0757 (2)	0.0821 (2)	0.0544 (5)
C19C	0.1207 (2)	0.2298 (2)	−0.0495 (2)	0.0558 (5)
F20C	0.03677 (11)	0.19524 (15)	−0.00621 (10)	0.0938 (5)
F21C	0.12125 (11)	0.21639 (12)	−0.13120 (9)	0.0717 (4)
F22C	0.08171 (11)	0.34863 (12)	−0.07577 (10)	0.0804 (4)

Table 2. *Geometric parameters (Å, °) for the three molecules of (I)*

	Molecule A	Molecule B	Molecule C
O1—C2	1.370 (2)	1.376 (2)	1.377 (2)
O1—C9	1.383 (2)	1.376 (2)	1.381 (2)
C2—O11	1.202 (2)	1.199 (2)	1.202 (2)
C2—C3	1.435 (3)	1.443 (3)	1.441 (3)
C3—C4	1.341 (3)	1.343 (3)	1.342 (3)
C4—C10	1.434 (3)	1.443 (2)	1.439 (3)
C4—C19	1.494 (3)	1.501 (3)	1.499 (3)
C5—C6	1.371 (3)	1.368 (3)	1.369 (3)
C5—C10	1.405 (2)	1.401 (2)	1.399 (3)
C6—C7	1.425 (3)	1.429 (3)	1.424 (3)
C6—C12	1.504 (3)	1.506 (2)	1.503 (3)
C7—N15	1.368 (2)	1.370 (2)	1.368 (2)
C7—C8	1.410 (2)	1.408 (2)	1.410 (2)
C8—C9	1.373 (3)	1.381 (2)	1.379 (2)
C8—C18	1.512 (3)	1.507 (2)	1.507 (3)
C9—C10	1.401 (2)	1.405 (2)	1.402 (2)
C12—C13	1.492 (6)	1.454 (5)	1.413 (4)
C12—C13′	1.417 (7)	1.466 (5)	1.432 (6)
C13—C14	1.478 (6)	1.439 (5)	1.471 (6)
C13′—C14	1.414 (7)	1.493 (5)	1.488 (6)
C14—N15	1.439 (3)	1.441 (2)	1.451 (3)
N15—C16	1.452 (3)	1.445 (3)	1.451 (3)
C16—C17	1.441 (7)	1.450 (6)	1.488 (4)
C16—C17′	1.435 (7)	1.433 (6)	
C17—C18	1.424 (7)	1.465 (6)	1.503 (3)
C17′—C18	1.550 (6)	1.492 (6)	
C19—F20	1.329 (2)	1.330 (2)	1.322 (2)
C19—F21	1.332 (2)	1.333 (2)	1.336 (2)
C19—F22	1.341 (3)	1.340 (2)	1.341 (2)
C2—O1—C9	123.0 (2)	122.8 (2)	122.5 (2)
O11—C2—O1	117.1 (2)	117.1 (2)	116.6 (2)
O11—C2—C3	126.4 (2)	126.3 (2)	126.5 (2)
O1—C2—C3	116.5 (2)	116.7 (2)	116.9 (2)
C4—C3—C2	121.7 (2)	121.5 (2)	121.4 (2)
C3—C4—C10	121.1 (2)	121.3 (2)	121.3 (2)
C3—C4—C19	119.1 (2)	119.3 (2)	118.8 (2)
C10—C4—C19	119.7 (2)	119.3 (2)	119.8 (2)
C6—C5—C10	123.0 (2)	122.9 (2)	122.6 (2)
C5—C6—C7	119.1 (2)	119.4 (2)	119.7 (2)
C5—C6—C12	121.0 (2)	121.1 (2)	121.1 (2)
C7—C6—C12	119.8 (2)	119.5 (2)	119.3 (2)
N15—C7—C8	120.7 (2)	120.7 (2)	120.7 (2)
N15—C7—C6	119.9 (2)	120.0 (2)	120.0 (2)
C8—C7—C6	119.4 (2)	119.3 (2)	119.3 (2)
C9—C8—C7	118.5 (2)	118.6 (2)	118.2 (2)
C9—C8—C18	120.4 (2)	120.6 (2)	121.3 (2)
C7—C8—C18	121.1 (2)	120.7 (2)	120.4 (2)
C8—C9—O1	115.6 (2)	115.6 (2)	115.3 (2)
C8—C9—C10	124.1 (2)	123.4 (2)	123.8 (2)
O1—C9—C10	120.3 (2)	121.1 (2)	120.9 (2)
C9—C10—C5	115.9 (2)	116.4 (2)	116.4 (2)
C9—C10—C4	117.2 (2)	116.6 (2)	117.0 (2)
C5—C10—C4	126.9 (2)	127.0 (2)	126.7 (2)
C13—C12—C6	112.7 (3)	114.6 (2)	114.2 (3)
C13′—C12—C6	114.2 (3)	112.1 (2)	114.1 (3)
C14—C13—C12	112.9 (3)	116.9 (4)	116.6 (4)
C14—C13′—C12	121.9 (5)	112.8 (3)	114.3 (4)
C13—C14—N15	114.0 (2)	116.8 (3)	116.6 (3)
C13′—C14—N15	116.3 (3)	112.8 (3)	111.1 (3)
C7—N15—C14	122.4 (2)	122.2 (2)	121.4 (2)
C7—N15—C16	120.2 (2)	121.0 (2)	120.5 (2)
C14—N15—C16	116.9 (2)	116.9 (2)	116.5 (2)
C17—C16—N15	118.5 (3)	115.8 (3)	112.3 (2)
C17′—C16—N15	113.0 (3)	114.9 (3)	
C18—C17—C16	119.3 (5)	116.5 (4)	110.9 (2)
C18—C17′—C16	111.8 (4)	115.8 (4)	
C17—C18—C8	113.3 (3)	111.6 (3)	111.2 (2)
C17′—C18—C8	109.0 (3)	111.3 (3)	
F20—C19—F21	106.4 (2)	106.4 (2)	106.5 (2)
F20—C19—F22	106.5 (2)	107.2 (2)	106.8 (2)
F21—C19—F22	105.7 (2)	106.1 (2)	105.3 (2)
F20—C19—C4	112.9 (2)	112.3 (2)	113.0 (2)
F21—C19—C4	113.3 (2)	112.7 (2)	112.9 (2)
F22—C19—C4	111.5 (2)	111.7 (2)	111.8 (2)
C12—C6—C7—N15	−0.8 (3)	−0.1 (3)	1.1 (3)
C7—C6—C12—C13	22.7 (3)	18.4 (4)	24.5 (5)
C7—C6—C12—C13′	−13.2 (4)	−24.1 (3)	−18.3 (5)
C6—C7—N15—C14	2.3 (3)	−1.2 (3)	−10.3 (3)
C6—C12—C13—C14	−45.4 (4)	−35.8 (6)	−40.5 (7)
C6—C12—C13′—C14	27.2 (7)	48.6 (4)	43.6 (6)
C12—C13—C14—N15	47.5 (4)	35.4 (6)	32.3 (7)
C12—C13′—C14—N15	−26.3 (8)	−50.3 (4)	−51.4 (5)
C13—C14—N15—C7	−26.1 (4)	−16.4 (4)	−5.9 (4)
C13′—C14—N15—C7	10.5 (5)	26.2 (4)	34.6 (4)
N15—C7—C8—C18	0.1 (3)	−2.3 (3)	0.4 (3)
C8—C7—N15—C16	−5.4 (4)	0.0 (3)	5.3 (3)
C7—N15—C16—C17	−7.8 (6)	−19.1 (4)	−33.6 (4)
C7—N15—C16—C17′	34.5 (4)	23.9 (4)	
N15—C16—C17—C18	27.1 (9)	40.9 (6)	55.3 (4)
N15—C16—C17′—C18	−56.3 (4)	−45.5 (5)	
C16—C17—C18—C8	−30.8 (8)	−41.4 (6)	−48.9 (3)
C16—C17′—C18—C8	48.9 (4)	41.7 (5)	
C7—C8—C18—C17	17.8 (5)	22.6 (4)	22.1 (3)
C7—C8—C18—C17′	−21.4 (3)	−18.0 (4)	

Table 3. *Asymmetry parameters (Å) of the three molecules of (I)*

Ring	Molecule	Asymmetry Parameter	Conformation
C7,C6,C12,C13,-C14,N15	A	Δ_s(C7) 0.010 (2)	Sofa
	B	Δ_s(C7) 0.011 (1)	Sofa
	C	Δ_2(C7–N15) 0.027 (2)	Half-chair
C7,C6,C12,C13',-C14,N15	A	Δ_s(C7) 0.012 (2)	Sofa
	B	Δ_s(C7) 0.008 (1)	Sofa
	C	Δ_2(C7–C6) 0.033 (2), Δ_s(C7) 0.068 (2)	Half-chair/Sofa
C8,C7,N15,C16,-C17,C18	A	Δ_2(N15–C7) 0.028 (2), Δ_s(C7) 0.041 (2)	Half-chair/Sofa
	B	Δ_s(C7) 0.018 (2)	Sofa
	C	Δ_s(C7) 0.043 (1), Δ_2(C7–C8) 0.069 (1)	Sofa/Half-chair
C8,C7,N15,C16,-C17',C18	A	Δ_s(C7) 0.042 (2), Δ_2(C7–C8) 0.070 (1)	Sofa/Half-chair
	B	Δ_s(C7) 0.018 (2)	Sofa

The structure was solved by direct methods using the 'brute force' option (TREF 1000 with 500 reflections), since the structure solution was not possible with default options (*SHELXS*86; Sheldrick, 1990*a*). All the disordered atoms were located from the difference Fourier maps. The site occupancy factors of these atoms were refined with isotropic displacement parameters and values ranging from 0.4 to 0.6 were obtained in the early stages and later fixed at 0.5 during anisotropic refinements due to their large correlation with displacement parameters. The H atoms attached to all the normal atoms were located from difference Fourier maps and refined isotropically, while the H atoms attached to the disordered atoms were fixed geometrically and allowed to ride on their respective C atoms with an isotropic displacement parameter U of 0.08 $Å^2$.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93. Geometrical calculations: *PARST* (Nardelli, 1983*b*).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: KH1030). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,3,6,6-Tetramethyl-10-(4-methylphenyl)-3,4,6,7,9,10-hexahydro-1,8(2H,5H)-acridinedione

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Abstract

The title molecule, $C_{24}H_{29}NO_2$, consists of a partially hydrogenated acridine ring system with one phenyl substituent on the dihydropyridine ring. The compound crystallizes with a half molecule per asymmetric unit, which has twofold symmetry. The dihydropyridine ring is almost planar, whereas both the outer rings adopt half-chair conformations.

Comment

Compounds of the acridine type have long been known to be potent frameshift mutagens in viruses and bacteria (Acheson, 1956). Acridines bind to DNA by intercalation (Lerman, 1961; Karle, Cysyk & Karle, 1980;