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# *N*-(4a-Morpholino-2,3,4,4a,9,9a-hexahydro-1*H*-xanthen-9-yl)phenylamine

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.068
*wR* factor = 0.179
Data-to-parameter ratio = 17.3

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



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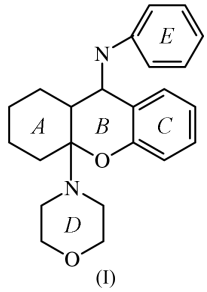
In the title compound, $C_{23}H_{28}N_2O_2$, the pyran ring in the xanthene moiety adopts a half-chair conformation. The molecular structure is influenced by intramolecular N—H···N and C—H···O hydrogen bonds and the crystal structure is stabilized by C—H···O and C—H···π interactions.

## Comment

An X-ray study of the title compound, (I), was of interest because xanthene derivatives have been well documented as biogenic precursors (Vinod & Gupta, 1979) and neuroleptic (Lassen *et al.*, 1980) and antiallergic (Pfister, 1980) agents. They also possess antiviral activity (Carr *et al.*, 1976). These derivatives act as potent human CCRI receptor antagonists (Naya *et al.*, 2003).




(I)

In the title molecule (Fig. 1), bond lengths in the aromatic rings (*C* and *E*), the cyclohexane ring (*A*) and the morpholine ring (*D*) have normal values (Allen *et al.*, 1987). The geometry of the pyran ring in the xanthene moiety (Table 1) is comparable to that observed in other xanthene derivatives (Jeyakanthan *et al.*, 1999; Miao *et al.*, 1996). The sum of the bond angles around N15 of 337.2 (2)° is indicative of the $sp^3$ character of the atom. The aromatic rings *E* and *C* are almost perpendicular to each other, with a dihedral angle of 85.5 (1)°. The pyran ring in the xanthene moiety adopts a half-chair conformation, with asymmetry parameter $\Delta C_2$(C7—C2) = 0.003 (1) (Nardelli, 1983). The cyclohexane ring (*A*) and the morpholine ring (*D*) both adopt chair conformations. The molecular structure is influenced by weak N—H···N and C—H···O hydrogen bonds. The crystal structure is stabilized by C—H···O interactions and C—H···π interactions involving the symmetry-related aromatic ring *C* (Table 2 and Fig. 2).

## Experimental

A solution of 0.3 g of cyclohexanone, 0.27 g of morpholin and a catalytic amount of $InCl_3$ in 20 ml of acetonitrile was refluxed for 3 h.

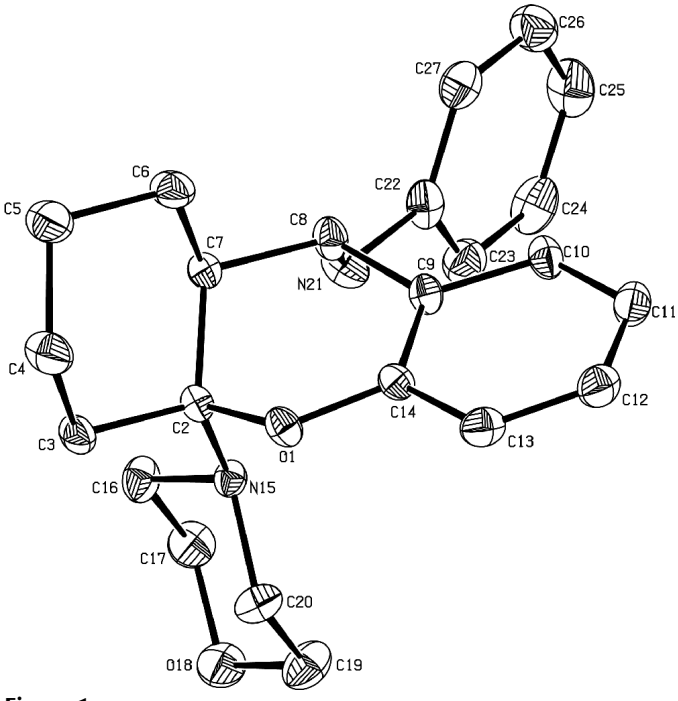



**Figure 1**
The molecular structure of the title compound, showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

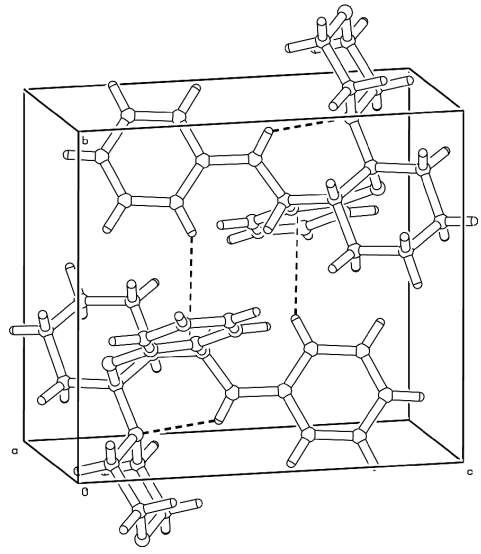



**Figure 2**
The packing of the molecules, viewed approximately down the *a* axis.

The reaction mixture was cooled to room temperature under a nitrogen atmosphere and 0.6 g of *o*-hydroxybenzylideneaniline was added. The reaction mixture was stirred at ambient temperature for 20 min. After completion of the reaction, the reaction mixture was quenched by addition of water and extracted with ethyl acetate. The combined organic layer was dried over anhydrous $Na_2SO_4$, filtered and concentrated under vacuum. The crude crystalline product was recrystallized using ethyl acetate to yield the title compound. The melting point of the title compound is 429–431 K.

*Crystal data*

| | |
|---|---|
| $C_{23}H_{28}N_2O_2$ | $Z = 2$ |
| $M_r = 364.47$ | $D_x = 1.254$ Mg m$^{-3}$ |
| Triclinic, $P\overline{1}$ | Mo $K\alpha$ radiation |
| $a = 9.6452$ (7) Å | Cell parameters from 2317 reflections |
| $b = 10.1247$ (8) Å | $\theta = 2.3$–$27.2^\circ$ |
| $c = 11.1168$ (9) Å | $\mu = 0.08$ mm$^{-1}$ |
| $\alpha = 81.359$ (1)° | $T = 293$ (2) K |
| $\beta = 80.165$ (1)° | Block, colourless |
| $\gamma = 64.920$ (1)° | 0.26 × 0.20 × 0.16 mm |
| $V = 965.11$ (18) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 3415 reflections with $I > 2\sigma(I)$ |
| | $R_{int} = 0.014$ |
| $\omega$ scans | $\theta_{max} = 28.0^\circ$ |
| Absorption correction: none | $h = -12 \rightarrow 12$ |
| 6035 measured reflections | $k = -11 \rightarrow 13$ |
| 4223 independent reflections | $l = -14 \rightarrow 14$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.088P)^2 + 0.492P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.068$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $wR(F^2) = 0.179$ | $\Delta\rho_{max} = 0.97$ e Å$^{-3}$ |
| $S = 1.02$ | $\Delta\rho_{min} = -0.35$ e Å$^{-3}$ |
| 4223 reflections | |
| 244 parameters | |
| H-atom parameters constrained | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1—C14 | 1.369 (2) | C8—C9 | 1.521 (3) |
| O1—C2 | 1.440 (2) | C9—C14 | 1.393 (3) |
| C2—N15 | 1.479 (2) | N15—C20 | 1.465 (3) |
| C2—C7 | 1.540 (3) | N15—C16 | 1.468 (2) |
| C7—C8 | 1.535 (3) | C17—O18 | 1.414 (3) |
| C8—N21 | 1.439 (3) | O18—C19 | 1.420 (3) |
| | | | |
| C20—N15—C16 | 107.9 (2) | C16—N15—C2 | 114.3 (1) |
| C20—N15—C2 | 115.0 (2) | | |
| O1—C2—N15—C16 | 171.42 (15) | C7—C8—N21—C22 | 151.6 (2) |
| C3—C2—N15—C16 | 57.0 (2) | C8—N21—C22—C27 | −26.6 (3) |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N21—H21···N15 | 0.86 | 2.44 | 2.949 (3) | 118 |
| C20—H20*B*···O1 | 0.97 | 2.35 | 2.737 (2) | 103 |
| C11—H11···O18$^{i}$ | 0.93 | 2.82 | 3.458 (3) | 126 |
| C12—H12···O18$^{ii}$ | 0.93 | 2.83 | 3.524 (3) | 132 |
| C27—H27···*CgC*$^{iii}$ | 0.93 | 2.87 | 3.704 (3) | 150 |

Symmetry codes: (i) $1-x, -y, 1-z$; (ii) $x-1, 1+y, z$; (iii) $1-x, 1-y, 1-z$. *CgC* denotes the centroid of aromatic ring *C*.

H atoms were positioned geometrically, with C—H distances in the range 0.93–0.98 Å and N—H 0.86 Å. They were allowed to ride on their parent atoms with the isotropic displacement parameter $U_{iso}$(H) set at $1.2U_{eq}$(C or N). The final cycles of refinement showed the highest difference peak of 0.97 e Å$^{-3}$, much larger than the absolute value of the deepest hole (−0.35 e Å$^{-3}$). This peak was located at a distance of 1.27 Å from both C8 and N21. The possibility of a disordered aminophenyl substituent (N21/C22–C27) was examined but we were unable to find a suitable disorder model. Reflections were measured to $\theta_{max}$ of 28.0° with 91% completeness, but the data are 97% complete to 25°.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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