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Crystal Structure and Thermal Chemical Properties of 2-Anilino-3-Methyl-6-Dibutylaminofluorane

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The crystal structure of 2-anilino-3-methyl-6-dibutylaminofluorane has been determined by single crystal X-ray diffraction method. The molecules are linked by N1-H1A·····O3 intermolecular interactions into zig-zag chains. The packing is stabilized by dipole-dipole and van der Waals' forces. The title compound starts to decompose in the range of 300–480°C.

INTRODUCTION

Xanthones are a class of natural products that have been shown to display a wide range of pharmacological properties[1, 2]. ODB compounds have previously been synthesized but only partially characterized[3, 4]. To our knowledge, recently a few crystal structures of 4,5-disubstituted fluorane derivatives have been described[5]. This paper reports the X-ray molecular structure of ODB-2‡.

All chemicals were of analytical reagent grade and used directly without further purification. Elemental analysis was performed by Perkin-Elmer 240. Calcd. for ODB-2: C, 78.95; H, 6.77; N, 5.26%. Found: C, 78.98; H, 6.72; N, 5.32%. The synthetic route to the title compound is shown in **Scheme-1.**

The bond lengths and angles in (I) are within normal ranges[7]. All the C—C bond distances in the benzene rings have typical C_{sp^2}-C_{sp^2} values. The 9H-xanthene moiety is almost planar, with the pyran ring distorted towards a boat conformation. Atoms C_{10} and O_1 deviate by 0.080(5) and 0.143(1) Å from the $C_9/C_{11}/C_{16}/C_{17}$

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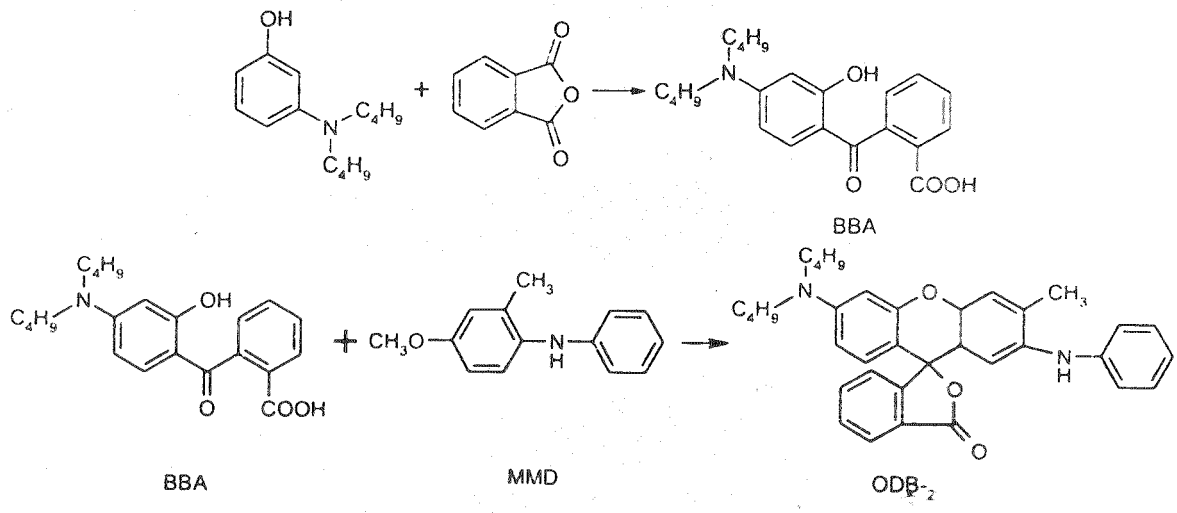
‡The data were collected by Bruker Smart 1000 CCD diffractometer. The structure was solved by direct methods and expanded by using Fourier differential techniques with SHELXL-97[6]. All non-hydrogen atoms were located with successive difference Fourier syntheses. The structure was refined by full-matrix least-squares method on F^2 with anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms were added according to the theoretical models. Full matrix least-squares refinement.

Crystal data for ODB-2: $C_{35}H_{36}N_2O_3$, m.w. = 532.66, 0.10 × 0.12 × 0.29 mm, monoclinic, space group $P2_1/c_1$, a = 8.611(2) Å, b = 22.261(6) Å, c = 16.676(4) Å, β = 111.233(11)°, z = 4, d_{calc} = 1.187 g cm^{-3}, $2\theta_{max}$ = 26.1°, MoK_α (λ = 0.71073 Å), μ = 0.075 mm^{-1}, ω scan, T = 298 K, 5844 independent reflections, 3244 observed reflections [I > 2σ(I)], R = 0.0749, wR = 0.1872, $\Delta\rho_{max}$ = 0.50 eÅ^{-3}, $\Delta\rho_{min}$ = 0.27eÅ^{-3}.

Atomic coordinates, bond lengths, bond angles and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre (CCDC). These data can be obtained free of charge *via* www.ccdc.cam.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (44)(1223)336033; or deposit@ccdc.cam.ac.uk). Any request to the CCDC for data should quote the full literature citation and CCDC reference number 266405. For details, see 'Notice to Authors', *Mendeleev Commun.*, Issue 1, 2003.



Scheme-1

plane, respectively. The two benzene rings in the xanthene moiety make a dihedral angle of 13.5(2)° with each other. The 2-benzofuran-1(3H)-one system is perfectly planar, with the dihedral angle of 1.7(2)° between the furane ring and its fused benzene ring. The xanthene moiety and the benzofurane system are nearly orthogonal, with a dihedral angle of 86.4(1)° between their planes. The relative orientation of these two moieties is described by the sp^3 hybridization state of atom C_{10}. The sums of the angles around N_1 and N_2 are almost 360° [359.9(3)° for N_1 and 359.7(3)° for N_2], suggesting the planar conformation of these two N atoms. The C_6—N_1—C_7 angle is 123.1(3)°, implying that the C_1—C_6 benzene ring is pushed away from the xanthene moiety, with the dihedral angle being 72.4(2)°. Meanwhile, the C_1—C_6 benzene ring is deviating 70.1(2)° from the benzofurane system. The two butyl groups attached to N_2 are each planar, with the dihedral angle of 70.3(1)° between these two groups. Fig. 1 shows the molecular structure of the compound.

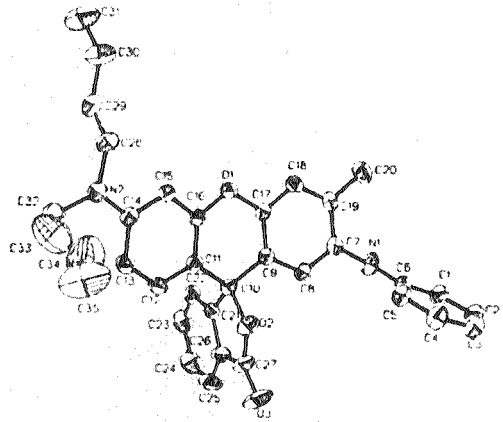


Fig. 1. The molecular structure of the title compound with the atomic numbering scheme. All the H atoms were omitted for clarity

In the crystal structure, the molecules are linked by N_1—H_1·····AO_3 intermolecular interactions into zig-zag chains. The packing is stabilized by dipole-dipole and van der Waals' forces.

Thermogravimetric (TG) analysis and differential thermogravimetric (DTG) analysis show that the title compound starts to decompose in the range of 300~480°C.

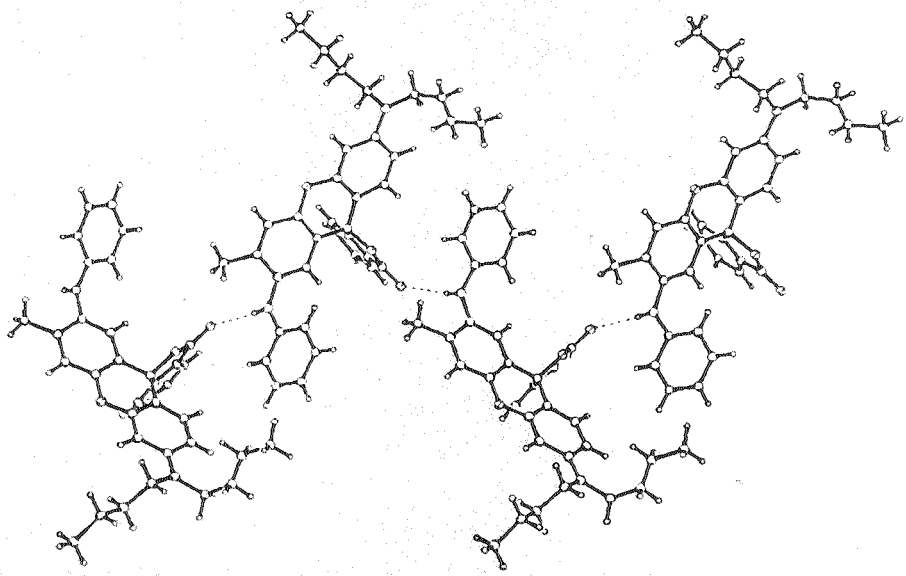


Fig. 2. A view of the crystal packing for the title compound showing the zig-zag chains

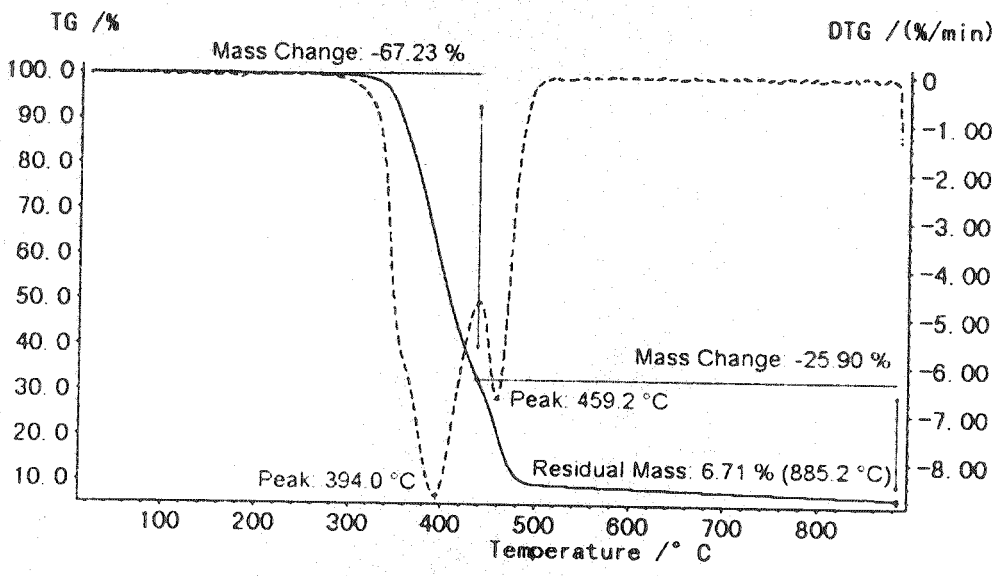


Fig. 3. Thermal analysis curves of the title compound

ACCKNOWLEDGEMENTS

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