The X-ray crystal structure of 2,4-bis(4-methoxyphenoxy) pyrimidine

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The X-ray crystal structure of 2,4-bis(4-methoxyphenoxy)pyrimidine, $C_{18}H_{16}N_2O_4$ was stabilised by the hydrogen bonding and van der Waal forces. The C–N bond in the pyrimidine moiety is shortened indicating of immonium character and good hydrogen bond donor potential. Both the phenyl rings are nearly perpendicular to the pyrimidine rings. The dihedral angles between the phenyl rings and the pyrimidine ring are 85.6(1) and 87.2(1)°. In the crystal packing, the pyrimidine rings stack with the pyrimidine rings and phenyl rings stack with the phenyl rings. The weak C–H…O interactions and π–π stacking interactions generate chains of molecules that are linked into sheets about the inversion centres.

Keywords: 2,4-bis(4-methoxyphenoxy) pyrimidine, X-ray crystal structure

A large number of 2,4-diaminopyrimidines with hydrogen, alkyl or aryl substituted in 5 and 6 positions were synthesised for their possible chemotherapeutic values.[1] 2,4-Bis(arylamino) and 2,4-bis(aryloxy) pyrimidines are potent antimicrobial and antifungal agent.[2] Various substituted compounds were tested against some gram-positive and gram-negative bacteria and a pathogenic strain of yeast and their activity compared. It was observed that, in contrast to bis-arylamino substituted pyrimidines, the biological activity of the bisarylpyrimidines does not depend on the nature of the substituent in the phenyl ring.[2] In continuation of our structural investigations on various nucleic acid components,[3] the structure of the title compound was determined.

2,4-Dichloropyrimidine (0.01 mole) and substituted phenol (0.025 mole) was mixed as a melt and subsequently cooled to room temperature. Finely powdered anhydrous K_2CO_3 (0.025 mole) was added to the reactants and mixed well. The mixture was heated on an oil bath at the optimum reaction temperature of 120°C for 30 minutes and cooled; on addition of 5 ml of toluene an oily substance separated out. The oil was extracted with hexane-ether, washed (dilute KOH, H_2O), and dried (Na_2SO_4). Crystals (m.p. 116°C lit. m.p. 116–117°C) appeared on evaporating the solvent.[1]

Experimental

$C_{18}H_{16}N_2O_4$, M_r = 324.33, crystals by slow evaporation from water/methanol mixture, crystals dimensions 0.60 × 0.55 × 0.25 mm, monoclinic, a = 8.394(3), b = 8.284(3), c = 23.083(7)Å, β = 92.60(3)°, U = 1603.4(10)Å^3, Z = 4, D_c = 1.351 g cm^{-3}, space group $P2_1/n$, MoKα radiation(λ = 0.7107)Å, μ = 0.10 mm^{-1}, F(000) = 680. Three-dimensional room temperature X-ray data were collected in the range $0 < 2\sigma < 50°$ on a AFC7 Rigaku diffractometer by the 2θ–ω scan mode (h from 0 to 9, k from 0 to 9, l from –27 to 27). Of the 2702 reflections measured, there were 2569 unique reflections, all of which were corrected for Lorentz and polarisation effects (but not for absorption), 2442 independent reflections exceeded the significance level $I > 2\sigma$ (I). The structure was solved by direct methods[4] and refined[5] by full matrix least squares on F^2. Hydrogen atoms were fixed at ideal positions and refined in riding mode with common isotropic thermal vibration parameters U_{iso} = 0.08 Å^2 and for methyl hydrogen U_{iso} = 0.10 Å^2. Three methyl hydrogen of C(20) atom were constrained with an O–H distance 0.97 and refined in riding mode. Refinement converged at a final R = 0.046 (wR_2 = 0.111, for all 2569 data, 217 parameters, maximum and mean Δ/σ (0.001, 0.000), with allowance for the thermal anisotropy of all the non-hydrogen atoms. Minimum and maximum final electron density –0.17 and 0.12 eA^{-3}. A weighting scheme $w = 1/[\sigma^2(Fo^2) + (0.0706P)^2]$ where $P = (Fo^2 + 2Fc^2)/3$ was used in the later stages of refinement. Geometric calculations were performed using PARST[6] program. Plots were produced with the ORTEP[7] programs. Complex scattering factors were taken from the program package SHELXL97.[5] See CCDC679236 for further details.

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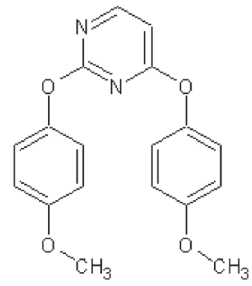

Fig. 1 The molecule.

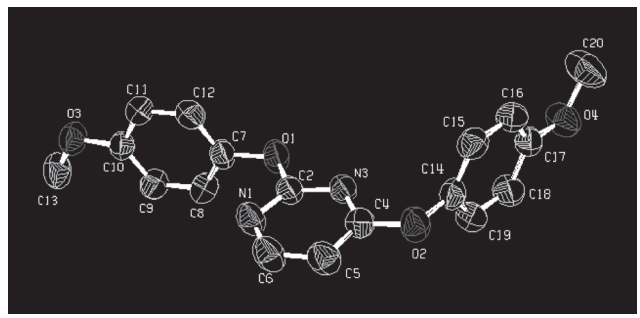

Fig. 2 Molecular structure showing 50% probability displacement ellipsoids.

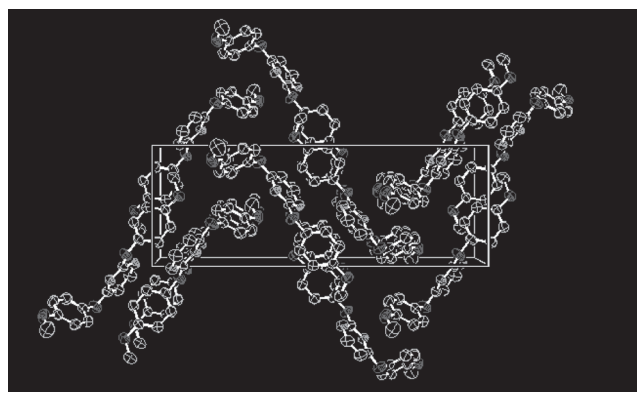

Fig. 3 Packing diagram of the molecule viewed down the *a*-axis.

The bond angles and distances are comparable to those reported for the related compounds 2,6-bis{[2-(dimethoxymethyl)phenoxy] methyl}pyridine[8] and 2,6-bis{[2-(hydroxymethyl)phenoxy]methyl} pyridine.[8] In the structure, two phenyl rings are planar and almost

perpendicular to the pyrimidine ring. The dihedral angles between the phenyl rings and the pyrimidine ring are 85.6(1) and 87.2(1)°. In the crystal packing, the pyrimidine rings stacks with the pyrimidine rings and phenyl rings stacks with the phenyl rings. The stacking distance is approximately 3.8Å.The crystal packing reveals the presence of weak intermolecular C–H ··· O and C–H ···π interactions. The C–H···O interactions generate chains of molecules that are linked into sheets about inversion centres. The molecular packing is stabilised by the hydrogen bonding and van der Waals forces. The C–N (N3 –C4 = 1.319 (2) Å) bond in the pyrimidine moiety is shortened indicating of immonium character and good hydrogen bond donor potential. The methyl hydrogen helps augment three dimensional molecular aggregation, bridging adjacent molecules by hydrogen bonding to a pyrimidine nitrogen atom N3 from one molecule and to a methyl carbon atom from an adjacent molecule. [C20–H1 = 0.97(1), C20...N3 = 3.652(5), H1...N3 = 2.768(6)Å, C20–H1...N3 = 151.9(5)°].

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