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Structural Studies of Some Phenoxazine Derivatives

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The compound 10-3′-chlorobutyl phenoxazine (**A**), crystallizes in the triclinic space group $P\bar{1}$ with $a = 11.664(2)$ Å, $b = 12.6292(2)$ Å, $c = 10.5832(14)$ Å, $\alpha = 113.041(9)°$, $\beta = 99.543(11)°$, $\gamma = 83.340(10)°$, $V = 1412.5(3)$ Å^3 and $Z = 2$. The structure is refined to $R = 0.102$. There are two molecules in the asymmetric unit. The packing of the molecules shows stacking along all the three axes. When viewed down, *b*, the two molecules of the asymmetric unit appear almost perpendicular to each other.

The compound, 10-(3′-*N*-Pyrrolidinopropyl)-2-trifluoro methyl phenoxazine hydrochloride (**B**), crystallizes in the monoclinic space group $C2/c$ with $a = 25.046(13)$ Å, $b = 11.638(6)$ Å, $c = 14.384(28$ Å), $\beta = 107.25(8)°$, $V = 4003(2)$ Å^3 and $Z = 8$. The structure is refined to $R = 0.065$. the packing of the molecules shows stacking when viewed down *b* axis.

The compound, 10-(*N*-morpholinoacetyl)-2-trifluoromethyl phenoxazine (**C**), crystallizes in the monoclinic space group $P2_1/n$ with $a = 12.710(4)$ Å, $b = 8.5163(14)$ Å, $c = 17.157(4)$ Å, $\beta = 108.62(2)°$, $V = 1759.9(7)$ Å^3 and $Z = 4$. The structure is refined to $R = 0.041$. The packing of the molecules shows layered arrangement when viewed along *b*.

The compound, 10-(*N*-chloroacetyl)-2-trifluoromethyl phenoxazine (**D**), crystallizes in the monoclinic space group $P2_1/a$ with $a = 8.888(2)$ Å, $b = 10.870(1)$ Å, $c = 14.544(2)$ Å, $\beta = 102.48(2)°$, $V = 1372(4)$ Å^3 and $Z = 2$. The structure is refined to $R = 0.089$. Intra and intermolecular hydrogen bonds are observed in the structure.

The compound, 10-*N*-piperidino acetyl phenoxazine (**E**), crystallizes in the monoclinic space group $P2_1/a$ with $a = 12.314(4)$ Å, $b = 9.108(3)$ Å, $c = 14.586(4)$ Å, $\beta = 106.26(2)°$, $V = 1621.4(8)$ Å^3 and $Z = 4$. The structure is refined to $R = 0.08$. Packing of molecules shows stacking in two layers when viewed along *b*. One layer has the three fused rings and other layer has the cyclohexane ring.

Keywords: Crystal structure; phenoxazine derivatives

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INTRODUCTION

Phenoxazine derivatives have found extensive applications in medicine, biology and chemistry. Some earlier work on phenoxazine are found in two reviews [1, 2].

The usual synthesis of phenoxazines makes use of a suitable ortho disubstituted benzene, from which oxazine skeleton is completed by ring closure. As intermediates, during the different ring closure methods *o-o'*-disubstituted diphenyl amines [3–5] or *o-o'*-disubstituted diphenyl ethers [6–7] have been isolated. Further, substituents may be introduced into the already formed phenoxazine ring. This involves electrophillic and nucleophillic substitution, or the modification of substituents in derivatives obtained by the following ring closure. The heterocyclic oxygen atom of the phenoxazine nucleus places certain restrictions on the aromaticity of the ring system, which appears to be somewhat less aromatic. The dipole moment of phenoxazine was found to be 1.93D(benzene) [8], which indicates the non-planarity of the molecule. The proton or the substituent at the nitrogen atom may be placed either between or out of the planes of two lateral rings. Most of the phenoxazines and their derivatives have melting points below 200°C and are stable, excepting those substituted in the position para to the bridging nitrogen atom with hydroxy or amino groups which are easily oxidized.

Some phenoxazine derivatives occur naturally, mainly as derivatives of 2-amino 3H-phenoxazine 3-ones. Phenoxazine derivatives are found to be less toxic than phenothiazine [9]. For the biologically active phenoxazines the general formula is shown in Figure 1. where *X* may be hydrogen, halogen, methyl, methoxyl, acyl, cyano or trifluoromethyl and *Y* may be alkyl, acyl or dialkylamino substituted side chains. Pharmacological tests have shown that the substituents in these positions produce an exaltation of the pharmacodynamic activity. These compounds have been claimed to be nervous system depressants, in particular with sedative, anti-epileptic and tranquilising activity [10–14]. Phenoxazine derivatives are radio protective, anti-oxidative

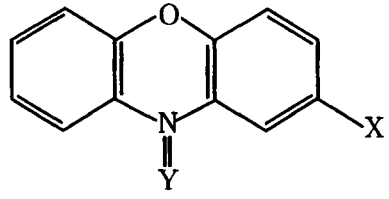

FIGURE 1 General structure of phenoxazine derivative molecule.

and also act as redox indicators [15–17]. Oxazine dyes which are oxidized phenoxazine derivatives are also used as biological stains.

In view of the importance of phenoxazine derivatives, we undertook the crystal structure studies of certain phenoxazine derivatives of biological interest. The derivatives chosen for this purpose are chloro, trifluoromethyl substituted at position two and piperidinoacetyl, chloropropyl, chlorobutyl, morpholinopropyl, morpholinoacetyl, pyrrolidinopropyl at position ten.

A. 10-3′-CHLOROBUTYL PHENOXAZINE

Experimental

The compound was synthesised following the standard protocol. The product was purified by column chromatography and the purified sample was characterized by UV-IR, ^1H and ^{13}C NMR and EIMS methods [18, 19].

Single crystals suitable for X-ray diffraction work were obtained by the method of slow evaporation of a mixture of chloroform and petroleum ether in which material was dissolved. The crystals were stable at room temperature. Table I lists the details of data collection, Wilson plot values [21], *etc*. Data were reduced by teXsan [20] data reduction program. Lorentz

TABLE I Data collection details

Details	***A***	***B***	***C***	***D***	***E***
1. Crystal colour	colourless	brown	brown	colourless	colourless
2. Diffractometer			Rigaku AFC7S		
3. Wavelength (Å)			Mo$K\alpha$, 0.71069		
4. Monochromator			Graphite		
5. Reflections for determining cell parameters (°)	22	18	12	18	24
6. 2θ range for (5)	14.70–17.61	13.73–19.62	14.01–16.10	14.20–16.20	14.56–17.95
7. $2\theta_{max}$ for data collection (°)			50		
8. Scan method			ω–2θ method		
9. Unique/Total reflections	2716/2860	2893/2980	2421/2571	2563/2852	3061/3209
10. Reflections with $I > 2\sigma(I)$	1739	1216	1871	1433	1345
11. R_{int}	0.0094	0.0542	0.0349	0.0732	0.100
12. Wilson plot Temperature factor, B Å^2	5.41	3.82	5.13	5.56	6.45
Scale factor, K	1.21	0.27	2.45	1.76	3.80

and polarisation corrections were applied and a semi-empirical absorption correction was applied based on ψ-scans [22]. The structure was solved by direct methods, SIR [23]. All the non-hydrogen atoms were revealed in the first map itself. Least-squares refinement using SHELXL-97 [24] with isotropic temperature factors for all the atoms converged the residuals to $R = 0.185$. Refinement of non-hydrogen atoms with anisotropic thermal parameters was started at this stage. After ten cycles of refinement the residuals saturated at $R = 0.102$. The hydrogen atoms were placed at calculated positions and were not refined. The largest peak and hole in the final difference map were 0.561 and -0.404 e Å^{-3} respectively.

Results and Discussions

The final positional coordinates of all the atoms, thermal parameters, bond distances and bond angles are given in Tables II–V. The bond distances and angles are in good agreement with the standard values. There are two molecules in the asymmetric unit. ORTEP [25] of the molecule at 50% probability is shown in Figure 2. The molecules appear stacked when viewed down all the three axes. Table VI gives the planarity of the molecules and the maximum deviation observed from the plane (all the planar geometry calculations were done using SHELXL-97 [24], PLATON [25] and PARST [26]. The fused rings of the phenoxazine molecule are planar in both the molecules of the asymmetric unit with a maximum deviation of 0.10 Å for N10A. All the rings of the molecules are planar. The three fused rings of each molecule are nearly planar (maximum deviation of 0.21 Å for N10A and 0.16 Å for N10B). The planes containing the three fused rings of the two molecules make a dihedral angle of 66.5° with each other. This planarity and tilt can be seen in the packing [25] of the molecules down *b* (Fig. 3).

B. 10-(3′-*N*-PYRROLIDINOPROPYL)-2-TRIFLUOROMETHYL PHENOXAZINE HYDROCHLORIDE

Experimental

The compound was synthesised following the standard protocol. The product was purified by column chromatography and the purified sample was characterized by UV-IR, ^{1}H and ^{13}C NMR and EIMS method [8, 9].

Single crystals suitable for X-ray diffraction work were obtained by the method of slow evaporation of a mixture of chloroform and ethanol in

TABLE II Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **A**

Atom	*x*	*y*	*z*	U_{eq}
C1A	0.4306(6)	0.3322(7)	1.3439(8)	0.076(2)
C2A	0.3820(5)	0.2727(6)	1.2097(8)	0.0583(18)
C3A	0.3582(5)	0.1580(6)	1.1625(8)	0.0567(17)
C4A	0.3845(6)	0.1047(7)	1.2581(9)	0.073(2)
C5A	0.4324(7)	0.1640(9)	1.3911(10)	0.089(3)
C6A	0.4541(7)	0.2779(9)	1.4360(10)	0.091(3)
O7A	0.3588(4)	0.3347(4)	1.1237(5)	0.0702(14)
C8A	0.3265(5)	0.2724(6)	0.9824(7)	0.0534(17)
C9A	0.3011(5)	0.1567(6)	0.9324(8)	0.0596(18)
N10A	0.3028(5)	0.1016(5)	1.0258(7)	0.0696(17)
C11A	0.3197(6)	0.3329(7)	0.8981(8)	0.0670(19)
C12A	0.2842(7)	0.2786(8)	0.7593(9)	0.085(2)
C13A	0.2592(7)	0.1666(8)	0.7065(9)	0.085(2)
C14A	0.2680(6)	0.1034(7)	0.7910(8)	0.076(2)
C15A	0.2637(6)	−0.0160(6)	0.9784(8)	0.073(2)
C16A	0.3600(7)	−0.1032(7)	0.9311(9)	0.087(2)
C17A	0.3275(8)	−0.2298(8)	0.8901(11)	0.100(3)
C18A	0.2606(9)	−0.2737(8)	0.7570(13)	0.111(3)
Cl1A	0.2434(2)	−0.4291(2)	0.7019(3)	0.1335(12)
C1B	−0.3174(5)	0.1418(6)	0.6408(8)	0.068(2)
C2B	−0.2382(5)	0.1934(6)	0.6079(7)	0.0553(16)
C3B	−0.2239(5)	0.3117(6)	0.6687(7)	0.0540(16)
C4B	−0.2967(6)	0.3742(6)	0.7656(8)	0.068(2)
C5B	−0.3786(6)	0.3254(7)	0.7990(8)	0.075(2)
C6B	−0.3917(6)	0.2079(7)	0.7366(9)	0.078(2)
O7B	−0.1709(4)	0.1218(4)	0.5056(6)	0.0778(16)
C8B	−0.0784(5)	0.1678(6)	0.4843(7)	0.0573(17)
C9B	−0.0592(5)	0.2854(6)	0.5445(7)	0.0541(16)
N10B	−0.1422(5)	0.3587(5)	0.6294(6)	0.0651(16)
C11B	−0.0046(6)	0.0919(6)	0.3951(7)	0.068(2)
C12B	0.0861(6)	0.1311(7)	0.3652(8)	0.073(2)
C13B	0.1052(6)	0.2454(7)	0.4242(8)	0.0685(19)
C14B	0.0348(5)	0.3225(6)	0.5154(7)	0.0605(17)
C15B	−0.1310(6)	0.4833(6)	0.6861(8)	0.0628(18)
C16B	−0.0574(6)	0.5301(6)	0.8247(7)	0.0621(18)
C17B	−0.0450(6)	0.6593(6)	0.8734(8)	0.0624(19)
C18B	0.0148(7)	0.7119(7)	1.0195(9)	0.082(2)
Cl1B	0.03224(18)	0.85984(18)	1.0690(3)	0.1001(9)

which material was dissolved. The crystals were stable at room temperature. Table I lists the details of data collection, Wilson plot values [21], *etc.* Data were reduced by teXsan [20] data reduction program. Lorentz and polarisation corrections were applied. The structure was solved by direct methods, SIR-92 [23]. All the non-hydrogen atoms were revealed in the first map itself. Least-squares refinement using SHELXL-97 [24] with isotropic temperature factors for all the non-hydrogen atom converged the residual to 0.22. The hydrogen atoms were placed at calculated positions and not refined. Hydrogen atoms for hydrochloride and for water molecule were

TABLE III Anisotropic displacement parameters (Å^2) for **A**

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1A	0.086(5)	0.076(6)	0.057(5)	0.018(4)	0.004(4)	−0.007(4)
C2A	0.062(4)	0.060(5)	0.051(5)	0.020(4)	0.015(3)	0.012(3)
C3A	0.057(3)	0.051(4)	0.061(5)	0.020(4)	0.010(3)	−0.001(3)
C4A	0.076(4)	0.077(6)	0.076(6)	0.039(5)	0.015(4)	0.006(4)
C5A	0.084(5)	0.124(8)	0.076(7)	0.062(6)	0.004(5)	0.005(5)
C6A	0.096(6)	0.119(8)	0.059(6)	0.039(6)	−0.002(4)	−0.008(5)
O7A	0.102(3)	0.050(3)	0.052(3)	0.016(3)	0.005(3)	0.005(2)
C8A	0.053(3)	0.053(4)	0.049(4)	0.014(4)	0.007(3)	0.000(3)
C9A	0.069(4)	0.051(4)	0.056(5)	0.017(4)	0.015(3)	0.004(3)
N10A	0.090(4)	0.059(4)	0.063(4)	0.026(3)	0.006(3)	−0.010(3)
C11A	0.083(4)	0.055(5)	0.067(5)	0.026(4)	0.018(4)	0.007(3)
C12A	0.115(6)	0.084(7)	0.063(6)	0.035(5)	0.014(5)	−0.005(5)
C13A	0.112(6)	0.088(7)	0.052(5)	0.023(5)	0.013(4)	0.004(5)
C14A	0.089(5)	0.072(6)	0.054(5)	0.013(4)	0.008(4)	−0.003(4)
C15A	0.073(4)	0.069(5)	0.080(6)	0.027(5)	0.019(4)	−0.014(4)
C16A	0.087(5)	0.083(6)	0.091(6)	0.026(5)	0.037(5)	0.004(4)
C17A	0.097(6)	0.105(8)	0.102(8)	0.043(7)	0.015(6)	−0.010(5)
C18A	0.108(6)	0.101(8)	0.132(10)	0.055(7)	−0.005(6)	−0.031(6)
Cl1A	0.139(2)	0.078(2)	0.147(3)	0.0082(18)	0.005(2)	−0.0084(16)
C1B	0.068(4)	0.063(5)	0.079(5)	0.032(4)	0.012(4)	0.002(3)
C2B	0.065(4)	0.047(4)	0.051(4)	0.017(3)	0.009(3)	0.007(3)
C3B	0.057(3)	0.050(4)	0.054(4)	0.020(3)	0.010(3)	0.007(3)
C4B	0.076(4)	0.062(5)	0.062(5)	0.020(4)	0.016(4)	0.005(3)
C5B	0.068(4)	0.084(6)	0.072(5)	0.025(5)	0.025(4)	0.011(4)
C6B	0.082(5)	0.075(6)	0.090(6)	0.042(5)	0.029(4)	0.011(4)
O7B	0.094(3)	0.047(3)	0.084(4)	0.006(3)	0.036(3)	−0.003(2)
C8B	0.062(4)	0.059(5)	0.049(4)	0.021(4)	0.010(3)	0.012(3)
C9B	0.066(4)	0.046(4)	0.051(4)	0.020(3)	0.011(3)	0.005(3)
N10B	0.085(4)	0.038(3)	0.066(4)	0.008(3)	0.024(3)	0.001(3)
C11B	0.089(5)	0.052(5)	0.047(4)	0.005(4)	0.010(4)	0.019(4)
C12B	0.072(4)	0.083(6)	0.062(5)	0.027(4)	0.025(4)	0.018(4)
C13B	0.077(4)	0.070(5)	0.063(5)	0.030(4)	0.015(4)	0.000(4)
C14B	0.074(4)	0.050(4)	0.056(4)	0.017(3)	0.016(3)	0.000(3)
C15B	0.075(4)	0.045(4)	0.068(5)	0.019(4)	0.018(4)	0.003(3)
C16B	0.080(4)	0.045(4)	0.056(5)	0.014(4)	0.007(4)	−0.005(3)
C17B	0.065(4)	0.050(4)	0.073(5)	0.022(4)	0.019(3)	0.002(3)
C18B	0.089(5)	0.073(6)	0.072(6)	0.018(5)	0.004(4)	−0.003(4)
Cl1B	0.0962(15)	0.0680(16)	0.110(2)	−0.0021(13)	0.0254(13)	−0.0166(11)

revealed by difference Fourier map and were refined. Refinement of the non-hydrogen atoms with anisotropic thermal parameters converged residuals to $R = 0.065$. The largest peak and hole in the final difference map was 0.491 and −0.392 e Å^{-3} respectively.

Results and Discussions

The final positional coordinates of all the atoms, thermal parameters, bond distances and bond angles are given in Tables VII–X. The thermal

TABLE IV Bond lengths for **A**

Atoms	*Length* (*Å*)	*Atoms*	*Length* (*Å*)
C1A–C6A	1.374(10)	C1B–C2B	1.350(8)
C1A–C2A	1.378(10)	C1B–C6B	1.391(10)
C2A–C3A	1.381(9)	C2B–C3B	1.394(8)
C2A–O7A	1.393(8)	C2B–O7B	1.400(7)
C3A–C4A	1.397(9)	C3B–C4B	1.376(9)
C3A–N10A	1.417(9)	C3B–N10B	1.375(8)
C4A–C5A	1.366(11)	C4B–C5B	1.356(9)
C5A–C6A	1.366(11)	C5B–C6B	1.383(10)
O7A–C8A	1.400(8)	O7B–C8B	1.371(7)
C8A–C11A	1.372(9)	C8B–C11B	1.386(8)
C8A–C9A	1.396(9)	C8B–C9B	1.397(8)
C9A–C14A	1.386(10)	C9B–C14B	1.365(8)
C9A–N10A	1.411(8)	C9B–N10B	1.424(8)
N10A–C15A	1.471(8)	N10B–C15B	1.461(7)
C11A–C12A	1.370(11)	C11B–C12B	1.350(9)
C12A–C13A	1.348(10)	C12B–C13B	1.358(9)
C13A–C14A	1.399(11)	C13B–C14B	1.377(9)
C15A–C16A	1.481(10)	C15B–C16B	1.503(9)
C16A–C17A	1.559(10)	C16B–C17B	1.524(8)
C17A–C18A	1.423(13)	C17B–C18B	1.505(10)
C18A–Cl1A	1.841(9)	C18B–Cl1B	1.759(8)

TABLE V Bond angles for **A**

Atoms	*Angle* (°)	*Atoms*	*Angle* (°)
C6A–C1A–C2A	119.8(9)	C2B–C1B–C6B	119.9(7)
C1A–C2A–C3A	122.0(8)	C1B–C2B–C3B	123.0(6)
C1A–C2A–O7A	116.2(7)	C1B–C2B–O7B	116.4(6)
C3A–C2A–O7A	121.7(7)	C3B–C2B–O7B	120.6(6)
C2A–C3A–C4A	116.8(8)	C4B–C3B–C2B	115.6(6)
C2A–C3A–N10A	120.2(7)	N10B–C3B–C2B	120.0(6)
C4A–C3A–N10A	122.9(7)	N10B–C3B–C4B	124.4(6)
C5A–C4A–C3A	121.0(9)	C5B–C4B–C3B	122.8(7)
C4A–C5A–C6A	121.2(9)	C4B–C5B–C6B	120.5(7)
C5A–C6A–C1A	119.0(9)	C5B–C6B–C1B	118.1(7)
C2A–O7A–C8A	117.2(6)	C8B–O7B–C2B	117.8(5)
C11A–C8A–C9A	122.8(8)	C11B–C8B–C9B	120.4(6)
C11A–C8A–O7A	115.7(7)	O7B–C8B–C11B	116.7(7)
C9A–C8A–O7A	121.4(7)	O7B–C8B–C9B	122.9(6)
C14A–C9A–C8A	116.9(8)	C14B–C9B–C8B	118.3(6)
C14A–C9A–N10A	123.1(7)	C14B–C9B–N10B	124.6(6)
C8A–C9A–N10A	119.8(7)	C8B–C9B–N10B	117.0(6)
C9A–N10A–C3A	116.9(6)	C3B–N10B–C9B	120.0(6)
C9A–N10A–C15A	121.2(6)	C9B–N10B–C15B	119.1(5)
C3A–N10A–C15A	121.3(6)	C3B–N10B–C15B	120.5(5)
C12A–C11A–C8A	118.8(8)	C12B–C11B–C8B	120.2(7)
C13A–C12A–C11A	120.3(9)	C11B–C12B–C13B	119.5(7)
C12A–C13A–C14A	121.4(9)	C12B–C13B–C14B	121.6(7)
C9A–C14A–C13A	119.7(9)	C9B–C14B–C13B	120.0(5)
N10A–C15A–C16A	111.2(6)	N10B–C15B–C16B	115.0(6)
C15A–C16A–C17A	114.0(7)	C15B–C16B–C17B	111.9(6)
C18A–C17A–C16A	112.3(8)	C18B–C17B–C16B	113.3(6)
C17A–C18A–Cl1A	110.7(7)	C17B–C18B–Cl1B	111.7(6)

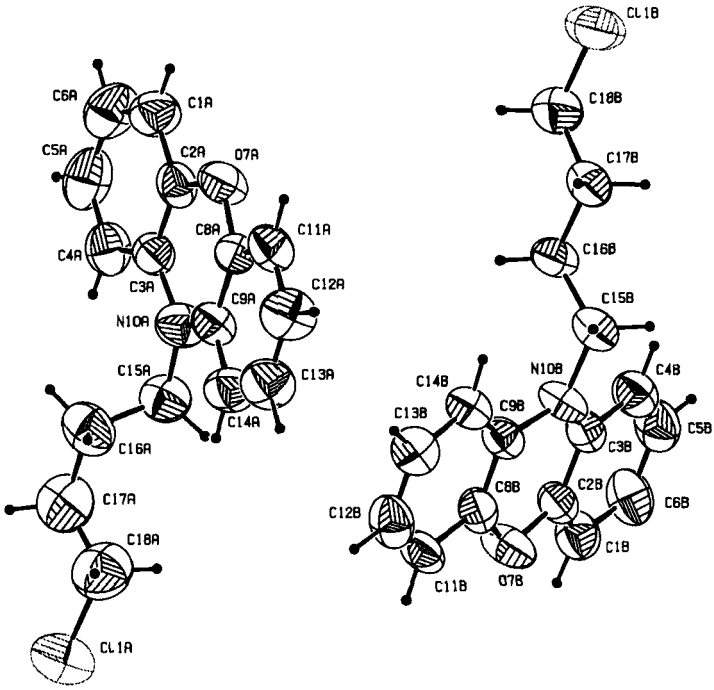

FIGURE 2 ORTEP of A at 50% probability.

parameters of the fluorine atoms are higher which is usual for the trifluoro group in a terminal position. The bond distances and angles are in agreement with the standard values. ORTEP [27] of the molecule at 50% probability is shown in Figure 4. The packing [25] of the molecules shows stacking when viewed down *b* axis (Fig. 5). Hydrogen chloride is bonded to nitrogen of the five membered ring by hydrogen bond of length 3.024(8) Å and the angle Cl1–H27···N22 is 164(11)° (dashed line in Fig. 4). In addition to this, intermolecular hydrogen bonds is also observed in the scheme O1–H28···Cl1 with a length 3.237(7) Å and angle 166(9)°. The water molecule is due to water of crystallisation. The nitrogen atom (N22) of the five membered ring is outside the plane by about 0.52 Å whereas the other atoms are in the plane. The planarity of the fused rings is given in Table VI [24–26].

TABLE VI Planarity of the rings (d is maximum deviation from the least-squares plane, (a, b) is the angle between planes a and b

Ring atoms	*Molecule A*		*Molecule B*	
	d (Å)	*Atom*	*d* (Å)	*Atom*
		A		
1. C1, C2, C3, C4, C5, C6	0.01	C6	0.01	C1
2. O7, N10, C2, C3, C8, C9	0.10	N10	0.08	N10
3. C8, C9, C11, C12, C13, C14	0.01	C11	0.01	C14
Mol. A: (1, 2) = 4.6°, (2, 3) = 3.4°, (1,3) = 7.7°				
Mol. B: (1, 2) = 3.6°, (2, 3) = 2.8°, (1,3) = 6.4°				
		B		
1. C1, C2, C3, C4, C5, C6	0.004	C1, C2, C3		
2. O7, N10, C5, C6, C8, C9	0.12	O7		
3. C8, C9, C11, C12, C13, C14	0.02	C11		
4. C23, C24, C25, C26	0.06	C25		
(1,2) = 6.2°, (2,3) = 6.0°, (1,3) = 12.2°				
		C		
1. C1, C2, C3, C4, C5, C6	0.008	C6		
2. O7, N10, C5, C6, C8, C9	0.27	N10		
3. C8, C9, C11, C12, C13, C14	0.02	C9		
4. C23, C24, C26, C27	0.002	C24, C26		
(1,2) = 19.7°, (2,3) = 19.6°, (1,3) = 38.5°				
		D		
1. C1, C2, C3, C4, C5, C6	0.04	C3		
2. O7, N10, C5, C6, C8, C9	0.26	N10		
3. C8, C9, C11, C12, C13, C14	0.01	C9		
(1,2) = 19.8°, (2,3) = 19.0°, (1,3) = 38.4°				
		E		
1. C1, C2, C3, C4, C5, C6	0.03	C3		
2. O7, N10, C5, C6, C8, C9	0.27	N10		
3. C8, C9, C11, C12, C13, C14	0.02	C12		
4. C19, C20, C22, C23	0.0002	all		
(1,2) = 21.6°, (2,3) = 18.0°, (1,3) = 39.3°				

C. 10-(*N*-MORPHOLINOACETYL)-2-TRIFLUOROMETHYL PHENOXAZINE

Experimental

The compound was synthesised following the standard protocol. The product was purified by column chromatography and the purified sample was characterized by UV-IR, ^{1}H and ^{13}C NMR and EIMS methods [18, 19].

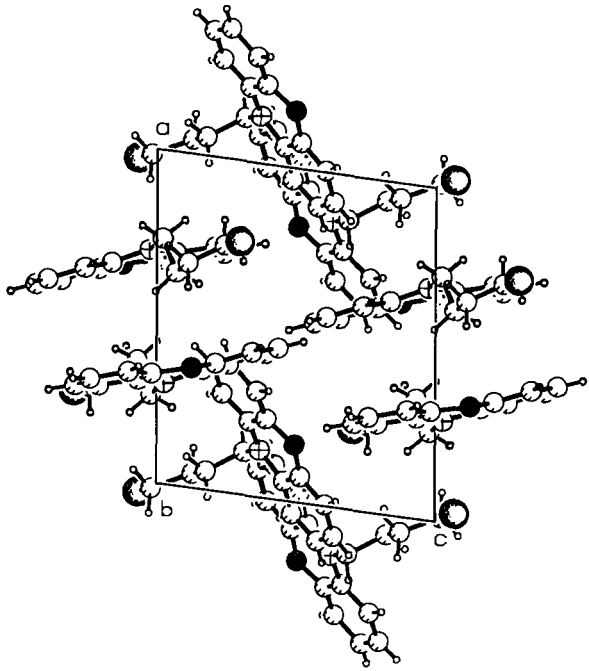

FIGURE 3 Packing down *b* of molecules of **A**.

Single crystals suitable for X-ray diffraction work were obtained by the method of slow evaporation of a mixture of chloroform and petroleum ether in which material is dissolved. The crystals were stable at room temperature. Table I lists the details of data collection, Wilson plot values [21], *etc.* Data were reduced by teXsan [20] data reduction program. Lorentz and polarisation corrections were applied. The structure was solved by direct methods, SHELXS-97 [28]. All the nonhydrogen atoms were revealed in the first map itself. Least-squares-refinement using SHELXL-97 [24] with isotropic temperature factors for all the atoms converged the residuals to $R = 0.23$. Refinement with anisotropic thermal parameters for all nonhydrogen atoms with fixing hydrogen atoms at chemically suitable positions converged residuals to $R = 0.096$. Thermal disorder occurs for all fluorine atoms. After splitting the fluorine atoms into two different sites and refining their occupancy factors the residuals converged to $R = 0.041$. Largest peak and hole in the final difference map is 0.16 and -0.15 e Å^{-3} respectively.

TABLE VII Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **B**

Atom	*x*	*y*	*z*	U_{eq}
C1	0.1920(3)	0.2169(7)	0.6669(5)	0.070(2)
C2	0.2393(4)	0.2112(8)	0.7487(6)	0.084(2)
C3	0.2835(4)	0.2812(9)	0.7570(6)	0.087(3)
C4	0.2815(3)	0.3603(9)	0.6848(7)	0.087(3)
C5	0.2352(3)	0.3670(7)	0.6039(5)	0.066(2)
C6	0.1897(3)	0.2962(6)	0.5940(5)	0.0576(18)
O7	0.2355(2)	0.4501(5)	0.5356(4)	0.0910(17)
C8	0.1960(3)	0.4404(6)	0.4465(6)	0.0648(19)
C9	0.1487(3)	0.3715(6)	0.4335(5)	0.0562(17)
N10	0.1429(2)	0.3087(5)	0.5119(4)	0.0603(15)
C11	0.2055(4)	0.5032(7)	0.3738(6)	0.078(2)
C12	0.1658(4)	0.5053(6)	0.2842(7)	0.087(3)
C13	0.1178(4)	0.4396(6)	0.2676(5)	0.074(2)
C14	0.1100(3)	0.3726(6)	0.3421(5)	0.0639(18)
C15	0.0766(5)	0.4385(11)	0.1712(9)	0.113(3)
F16	0.0889(4)	0.4856(10)	0.1040(6)	0.261(6)
F17	0.0305(5)	0.4803(14)	0.1695(6)	0.299(8)
F18	0.0583(5)	0.3406(10)	0.1357(6)	0.233(6)
C19	0.0965(3)	0.2274(6)	0.4968(5)	0.0605(18)
C20	0.1112(3)	0.1126(6)	0.4572(5)	0.0652(19)
C21	0.0708(3)	0.0193(6)	0.4581(4)	0.066(2)
N22	0.0847(2)	−0.0860(4)	0.4131(4)	0.0606(15)
C23	0.0444(4)	−0.1822(8)	0.4106(6)	0.091(3)
C24	0.0746(6)	−0.2853(8)	0.3884(8)	0.121(4)
C25	0.1349(6)	−0.2600(9)	0.4305(9)	0.137(4)
C26	0.1398(3)	−0.1381(7)	0.4584(6)	0.084(2)
Cl1	0.08308(8)	−0.01638(18)	0.20969(12)	0.0749(7)
O1	0.0000	0.1776(8)	0.2500	0.099(3)

Results and Discussions

The final positional coordinates of all the atoms, thermal parameters, bond distances and bond angles of non-hydrogen atoms are given in Tables XI–XIV. The bond distance and angles are in agreement with the standard values. ORTEP [27] of the molecule at 50% probability is shown in Figure 6. The packing [25] of molecules shows layered arrangement of the fused rings along *b* axis (Fig. 7) and the heterocyclic ring shows chair configuration. The central ring of phenoxazine is not planar whereas both the phenyl rings are planar (Tab. VI). The atoms C23, C24, C25 and C26 lie on a plane, while the nitrogen (N22) and the oxygen (O25) atoms are displaced by −0.666 Å and 0.6371 Å respectively from the plane giving a chair conformation for the ring. Intramolecular hydrogen bond, C14–H14···N22, of length 3.153 Å and angle 131.8(2)° is also observed [25]. This is shows as a dashed line in Figure 6.

TABLE VIII Anisotropic displacement parameters (Å^2) for **B**

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.075(5)	0.078(5)	0.062(5)	−0.018(4)	0.029(4)	−0.010(4)
C2	0.089(7)	0.088(6)	0.077(5)	−0.016(5)	0.027(5)	0.008(5)
C3	0.071(6)	0.119(8)	0.067(5)	−0.029(5)	0.014(4)	0.007(6)
C4	0.070(6)	0.115(7)	0.084(6)	−0.042(6)	0.034(5)	−0.015(5)
C5	0.060(5)	0.078(5)	0.070(5)	−0.028(4)	0.033(4)	−0.009(4)
C6	0.060(5)	0.056(4)	0.063(4)	−0.019(4)	0.030(4)	−0.002(4)
O7	0.083(4)	0.097(4)	0.100(4)	−0.007(4)	0.039(4)	−0.040(3)
C8	0.072(5)	0.058(4)	0.076(5)	−0.003(4)	0.040(4)	−0.008(4)
C9	0.062(4)	0.048(4)	0.070(5)	−0.003(3)	0.037(4)	0.000(4)
N10	0.058(4)	0.062(3)	0.067(4)	−0.005(3)	0.027(3)	−0.010(3)
C11	0.074(5)	0.080(6)	0.091(6)	−0.007(5)	0.040(5)	−0.010(4)
C12	0.122(7)	0.056(5)	0.106(7)	0.013(5)	0.069(6)	−0.010(5)
C13	0.100(6)	0.063(5)	0.065(5)	0.008(4)	0.034(5)	0.008(5)
C14	0.071(5)	0.056(4)	0.074(5)	0.006(4)	0.035(4)	0.001(4)
C15	0.117(9)	0.106(8)	0.111(9)	0.036(7)	0.027(8)	0.001(7)
F16	0.273(11)	0.361(13)	0.113(5)	0.100(7)	0.001(6)	−0.175(10)
F17	0.228(10)	0.50(2)	0.126(7)	−0.021(9)	−0.021(7)	0.215(13)
F18	0.292(13)	0.214(9)	0.122(6)	0.033(6)	−0.047(7)	−0.098(9)
C19	0.064(4)	0.064(4)	0.057(4)	0.007(3)	0.023(3)	−0.012(4)
C20	0.079(5)	0.076(5)	0.049(4)	−0.016(3)	0.031(4)	−0.019(4)
C21	0.081(5)	0.073(5)	0.048(4)	0.003(3)	0.026(4)	−0.007(4)
N22	0.080(4)	0.052(3)	0.051(3)	−0.002(3)	0.021(3)	0.003(3)
C23	0.092(6)	0.092(6)	0.081(6)	0.017(5)	0.017(5)	−0.024(5)
C24	0.160(11)	0.069(6)	0.123(8)	−0.006(6)	0.028(8)	−0.002(7)
C25	0.158(12)	0.082(8)	0.167(11)	−0.004(7)	0.044(9)	0.019(7)
C26	0.079(6)	0.084(6)	0.086(5)	−0.002(5)	0.018(5)	0.003(5)
Cl1	0.0797(13)	0.1050(16)	0.0448(9)	−0.0045(10)	0.0259(8)	0.0045(11)
O1	0.103(6)	0.084(6)	0.109(6)	0.000	0.029(5)	0.000

D. 10-(*N*-CHLOROACETYL)-2-TRIFLUOROMETHYL PHENOXAZINE

Experimental

The compound was synthesised following the standard protocol. The product was purified by column chromatography and the purified sample was characterized by UV-IR, ^{1}H and ^{13}C NMR and EIMS methods [18, 19].

Table I lists the data collection details. The data were reduced using teXsan data reduction program [20]. The data were collected for Lorentz and polarisation effects. The structure was solved by direct methods SIR92 [23] and expanded using Fourier techniques. Least squares refinement using teXsan [20] with isotropic temperature factors for non-hydrogen atoms converged the residual to 0.19. An empirical absorption correction using the

TABLE IX Bond lengths (Å) for **B**

Atoms	*Length (Å)*	*Atoms*	*Length (Å)*
C1–C2	1.401(11)	C12–C13	1.385(11)
C1–C6	1.385(9)	C13–C14	1.385(9)
C2–C3	1.351(11)	C13–C15	1.463(14)
C3–C4	1.378(12)	C15–F16	1.227(11)
C4–C5	1.379(11)	C15–F17	1.245(13)
C5–C6	1.379(9)	C15–F18	1.277(13)
C5–O7	1.380(9)	C19–C20	1.540(9)
C6–N10	1.405(9)	C20–C21	1.487(9)
O7–C8	1.372(9)	C21–N22	1.475(8)
C8–C11	1.354(10)	N22–C26	1.472(9)
C8–C9	1.397(9)	N22–C23	1.500(9)
C9–C14	1.383(10)	C23–C24	1.503(13)
C9–N10	1.386(8)	C24–C25	1.479(14)
N10–C19	1.463(8)	C25–C26	1.470(12)
C11–C12	1.374(11)		

TABLE X Bond angles (°) for **B**

Atoms	*Angle (°)*	*Atoms*	*Angle (°)*
C6–C1–C2	120.1(7)	C11–C12–C13	120.1(7)
C3–C2–C1	120.7(8)	C12–C13–C14	119.5(8)
C2–C3–C4	119.5(8)	C12–C13–C15	120.0(8)
C3–C4–C5	120.4(8)	C14–C13–C15	120.4(8)
C6–C5–C4	121.0(8)	C9–C14–C13	121.4(7)
C6–C5–O7	121.6(7)	F16–C15–F17	105.0(12)
C4–C5–O7	117.3(7)	F16–C15–F18	103.1(12)
C5–C6–C1	118.2(7)	F17–C15–F18	96.9(12)
C5–C6–N10	118.9(7)	F16–C15–C13	118.1(11)
C1–C6–N10	122.9(6)	F17–C15–C13	113.8(11)
C8–O7–C5	116.9(6)	F18–C15–C13	117.1(10)
C11–C8–O7	115.7(7)	N10–C19–C20	110.5(5)
C11–C8–C9	123.0(8)	C21–C20–C19	113.1(5)
O7–C8–C9	121.3(6)	N22–C21–C20	110.7(5)
C14–C9–N10	124.4(6)	C21–N22–C26	117.4(6)
C14–C9–C8	116.7(6)	C21–N22–C23	112.7(6)
N10–C9–C8	118.9(7)	C26–N22–C23	103.8(6)
C9–N10–C6	118.4(6)	C24–C23–N22	102.9(7)
C9–N10–C19	119.3(6)	C25–C24–C23	105.7(8)
C6–N10–C19	119.1(5)	C26–C25–C24	107.3(9)
C8–C11–C12	119.3(8)	C25–C26–N22	106.6(8)

program DIFABS [29] was applied which resulted in transmission factors ranging from 0.87 to 1.00. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full matrix least squares refinement was done using SHELXL-97 [24]

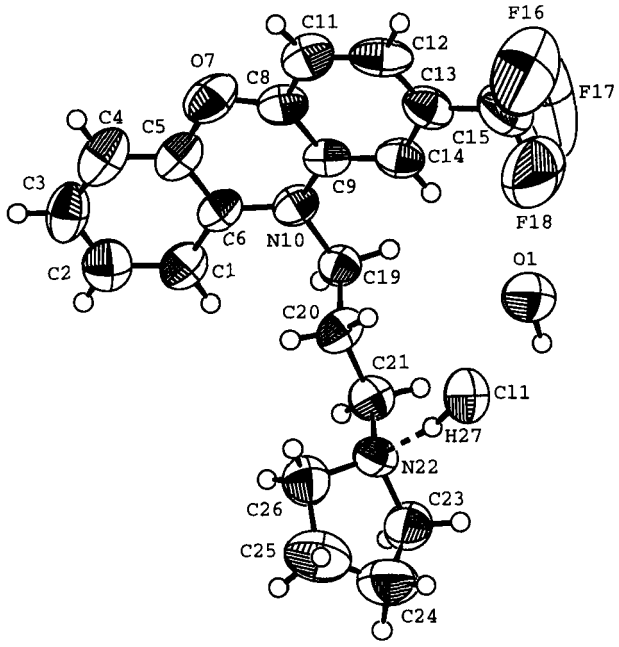

FIGURE 4 ORTEP of **B** at 50% probability.

based on 1433 reflections and 249 variable parameters which converged to $R = 0.089$ for 1162 reflections with $I > 2\sigma(I)$. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.35 and -0.46 e^{-} $Å^{-3}$ respectively.

Results and Discussions

The final positional coordinates of all the atoms, thermal parameters, bond distances and of angles non-hydrogen atoms are given in Tables XV–XVIII. The bond distances and angles are in agreement with the standard values. ORTEP [27] of the molecule at 50% probability is shown in Figure 8. The following intramolecular hydrogen bonds are observed in the structure. C12–H12···F16B with length 2.81(3) Å and 108(8)°, C12–H12···F17A with length 3.06(2) Å and 99(8)° and C12–H12···F17B with

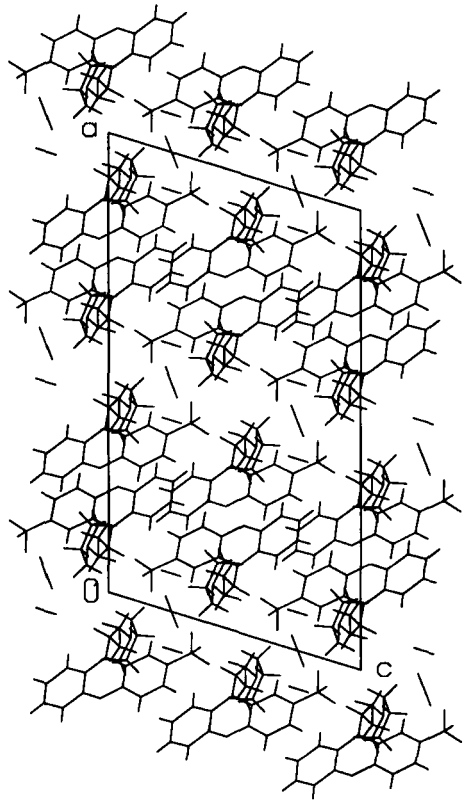

FIGURE 5 Packing down *b* of molecules of **B**.

length 2.78(3) Å and 105(8)° (shown as dashed lines in Fig. 8). The following intermolecular hydrogen bonds are observed in the structure: C1–H1···O7 with length 3.288(9) Å and C11–H11···F17B with length 3.17(4) Å and 112(7)° [26]. Table VI gives the planar details of the fused rings. The central ring is not planar, but the adjacent rings are planar. If we consider the atoms C1 to C6, O7 and N10, they lie in a plane with a maximum deviation of 0.07 Å for O7. Similarly the atoms O7, C8, C9, N10, C11 to C14 lie on a plane with a maximum deviation of 0.04 Å for O7. Thus the fused rings are bent on the line joining O7 and N10 atoms. The packing of the molecules down *a* is shown in Figure 9. The chlorine atoms all fall in a plane parallel to (010) in an alternate fashion.

TABLE XI Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **C**

Atom	*x*	*y*	*z*	U_{eq}
C1	0.7175(2)	0.4828(3)	0.40584(14)	0.0613(7)
C2	0.6313(2)	0.4523(3)	0.43694(16)	0.0684(7)
C3	0.5254(2)	0.5062(3)	0.39632(17)	0.0740(8)
C4	0.5047(2)	0.5898(3)	0.32483(16)	0.0672(7)
C5	0.59011(17)	0.6208(3)	0.29416(13)	0.0557(6)
C6	0.69732(17)	0.5692(3)	0.33467(12)	0.0521(6)
O7	0.56764(11)	0.7019(2)	0.22054(9)	0.0662(5)
C8	0.63002(18)	0.6537(3)	0.17225(13)	0.0569(6)
C9	0.73874(16)	0.6005(2)	0.20893(12)	0.0508(6)
N10	0.78031(13)	0.6057(2)	0.29694(10)	0.0512(5)
C11	0.5823(2)	0.6561(3)	0.08818(15)	0.0673(7)
C12	0.6426(2)	0.6037(3)	0.03990(15)	0.0715(8)
C13	0.7496(2)	0.5449(3)	0.07544(14)	0.0632(7)
C14	0.970(2)	0.5416(3)	0.16001(13)	0.0567(6)
C15	0.8127(3)	0.4855(4)	0.02203(17)	0.0854(9)
F16A	0.8588(17)	0.5892(10)	−0.0087(14)	0.146(6)
F17A	0.9240(6)	0.504(3)	0.0584(7)	0.149(5)
F18A	0.7417(8)	0.415(3)	−0.0462(8)	0.168(5)
F16B	0.7956(16)	0.5665(18)	−0.0441(6)	0.150(6)
F17B	0.8842(19)	0.382(2)	0.0546(7)	0.157(7)
F18B	0.8007(16)	0.3421(8)	0.0016(13)	0.152(5)
C19	0.87915(17)	0.6763(3)	0.34354(14)	0.0559(6)
O20	0.90142(12)	0.6902(3)	0.41705(10)	0.0804(6)
C21	0.95776(17)	0.7332(3)	0.29945(14)	0.0583(6)
N22	1.03182(13)	0.6095(2)	0.28938(10)	0.0528(5)
C23	1.0982(2)	0.6660(4)	0.23951(16)	0.0768(8)
C24	1.1747(2)	0.5376(4)	0.2296(2)	0.0959(10)
O25	1.24530(14)	0.4840(2)	0.30564(13)	0.0904(7)
C26	1.18171(19)	0.4290(3)	0.35388(17)	0.0729(7)
C27	1.10652(19)	0.5533(3)	0.36822(14)	0.0623(6)

E. 10-*N*-PIPERDINOACETYL PHENOXAZINE

Experimental

The compound was synthesised following the standard protocol. The product was purified by column chromatography and the purified sample was characterized by UV-IR, ^1H and ^{13}C NMR and EIMs methods [18, 19].

Table I lists the data collection details. The data were reduced using teXsan data reduction program [20]. The data were corrected for Lorentz and polarisation effects. The structure was solved by direct methods SIR92 [23] and expanded using Fourier techniques. Least squares refinement using SHELXL-97 [24] with isotropic temperature factors for non-hydrogen atoms converged the residual to 0.19. An empirical absorption correction using the program DIFABS [29] was applied which resulted in transmission

TABLE XII Anisotropic displacement parameters ($Å^2$) for **C**

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0611(14)	0.0675(16)	0.0526(14)	−0.0052(12)	0.0144(11)	0.0120(12)
C2	0.0787(18)	0.0702(16)	0.0596(14)	−0.0032(12)	0.0269(13)	0.0038(13)
C3	0.0678(17)	0.0830(18)	0.0787(18)	−0.0101(15)	0.0341(14)	−0.0035(14)
C4	0.0479(13)	0.0752(17)	0.0769(17)	−0.0063(14)	0.0175(12)	0.0046(12)
C5	0.0487(13)	0.0538(13)	0.0595(13)	−0.0008(11)	0.0101(10)	0.0046(10)
C6	0.0499(12)	0.0549(13)	0.0498(12)	−0.0075(10)	0.0133(10)	0.0041(10)
O7	0.0487(9)	0.0722(11)	0.0715(10)	0.0107(8)	0.0105(8)	0.0135(8)
C8	0.0519(13)	0.0535(13)	0.0556(13)	0.0071(10)	0.0036(11)	−0.0038(10)
C9	0.0492(12)	0.0494(13)	0.0479(12)	0.0000(10)	0.0071(10)	−0.0029(10)
N10	0.0429(10)	0.0603(12)	0.0446(10)	−0.0054(8)	0.0056(8)	0.0026(8)
C11	0.0582(14)	0.0696(16)	0.0605(15)	0.0117(12)	0.0000(13)	−0.0113(12)
C12	0.0823(19)	0.0688(17)	0.0468(13)	0.0067(13)	−0.0027(14)	−0.0229(14)
C12	0.0823(19)	0.0688(17)	0.0468(13)	0.0067(13)	−0.0027(14)	−0.0229(14)
C13	0.0825(17)	0.0544(14)	0.0499(14)	0.0024(10)	0.0172(13)	−0.0149(12)
C14	0.0603(14)	0.0551(14)	0.0516(14)	−0.0014(10)	0.0134(11)	−0.0033(11)
C15	0.127(3)	0.072(2)	0.0580(18)	−0.0026(17)	0.0307(18)	−0.009(2)
F16A	0.212(12)	0.082(4)	0.209(14)	−0.007(7)	0.158(11)	−0.023(7)
F17A	0.108(4)	0.243(15)	0.112(6)	−0.067(8)	0.058(4)	−0.004(6)
F18A	0.178(7)	0.226(15)	0.101(5)	−0.096(7)	0.044(5)	−0.033(8)
F16B	0.225(11)	0.172(12)	0.078(4)	0.049(5)	0.085(6)	0.062(9)
F17B	0.250(16)	0.156(9)	0.095(5)	0.035(6)	0.095(8)	0.117(10)
F18B	0.240(14)	0.073(4)	0.172(11)	−0.044(5)	0.107(11)	−0.022(5)
C19	0.0447(12)	0.0628(15)	0.0508(14)	−0.0083(10)	0.0012(10)	0.0079(10)
O20	0.0576(10)	0.1204(16)	0.0556(11)	−0.0247(10)	0.0073(8)	−0.0077(10)
C21	0.0474(12)	0.0544(13)	0.0645(14)	−0.0038(11)	0.0056(10)	−0.0016(10)
N22	0.0445(10)	0.0561(11)	0.0558(11)	0.0026(8)	0.0133(8)	0.0000(8)
C23	0.0659(15)	0.0879(19)	0.0811(17)	0.0110(14)	0.0300(13)	−0.0024(14)
C24	0.0817(19)	0.120(3)	0.102(2)	0.0004(19)	0.0519(19)	0.0030(18)
O25	0.0542(10)	0.1049(15)	0.1141(16)	−0.0037(12)	0.0295(11)	0.0093(10)
C26	0.0571(14)	0.0686(16)	0.0855(17)	−0.0008(14)	−0.0120(13)	−0.0071(12)
C27	0.0538(13)	0.0627(14)	0.0644(14)	0.0012(11)	0.0104(11)	0.0056(11)

factors ranging from 0.49 to 1.00. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full matrix least squares refinement was based on 2181 reflections and 233 variable parameters which converged to $R = 0.08$ for 1345 reflections with $I > 2\sigma(I)$. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.36 and −0.25 $e^- Å^{-3}$ respectively.

Results and Discussions

The final positional coordinates of all the atoms, thermal parameters, bond distances and angles non-hydrogen atoms are given in Tables XIX – XXII.

TABLE XIII Bond lengths (Å) for **C**

Atoms	*Length (Å)*	*Atoms*	*Length (Å)*
C1–C6	1.378(3)	C15–F18B	1.266(7)
C1–C2	1.388(3)	C15–F17B	1.259(7)
C2–C3	1.382(4)	C15–F16A	1.265(8)
C3–C4	1.369(4)	C15–F16B	1.286(8)
C4–C5	1.374(3)	C15–F17A	1.360(7)
C5–C6	1.388(3)	C15–F18A	1.369(7)
C5–O7	1.387(3)	F16A–F17A	1.39(3)
C6–N10	1.437(3)	F17B–F18B	1.20(3)
O7–C8	1.380(3)	C19–O20	1.207(3)
C8–C11	1.375(3)	C19–C21	1.513(3)
C8–C9	1.398(3)	C21–N22	1.459(3)
C9–C14	1.379(3)	N22–C27	1.463(3)
C9–N10	1.432(3)	N22–C23	1.461(3)
N10–C19	1.392(3)	C23–C24	1.509(4)
C11–C12	1.370(4)	C24–O25	1.403(3)
C12–C13	1.394(4)	O25–C26	1.409(3)
C13–C14	1.382(3)	C26–C27	1.498(4)
C13–C15	1.486(4)		

TABLE XIV Bond angles (°) for **C**

Atoms	*Angle (°)*	*Atoms*	*Angle (°)*
C6–C1–C2	119.6(2)	F18B–C15–F17A	104.7(13)
C1–C2–C3	120.4(2)	F17B–C15–F17A	51.7(5)
C4–C3–C2	120.2(2)	F16A–C15–F17A	63.8(14)
C3–C4–C5	119.6(2)	F16B–C15–F17A	101.5(12)
C4–C5–C6	121.1(2)	F18B–C15–F18A	49.4(4)
C4–C5–O7	119.08(19)	F17B–C15–F18A	104.8(15)
C6–C5–O7	119.80(19)	F16A–C15–F18A	102.6(6)
C5–C6–C1	119.2(2)	F16B–C15–F18A	66.6(6)
C5–C6–N10	116.78(19)	F17A–C15–F18A	138.4(6)
C1–C6–N10	124.00(19)	F18B–C15–C13	117.3(4)
C8–O7–C5	114.06(17)	F17B–C15–C13	114.8(3)
O7–C8–C11	118.7(2)	F16A–C15–C13	115.7(5)
O7–C8–C9	120.07(19)	F16B–C15–C13	112.9(6)
C11–C8–C9	121.2(2)	F17A–C15–C13	111.4(3)
C14–C9–C8	119.3(2)	F18A–C15–C13	109.8(4)
C14–C9–N10	124.32(18)	C15B–F16A–F17A	61.5(9)
C8–C9–N10	116.31(19)	C15–F17A–F16A	54.8(7)
C19–N10–C6	119.50(17)	F18B–F17B–C15	61.8(9)
C9–N10–C9	125.47(18)	F17B–F18B–C15	61.2(8)
C6–N10–C9	113.05(16)	O20–C19–N10	120.7(2)
C12–C11–C8	119.0(2)	O20–C19–C21	121.26(19)
C11–C12–C13	120.6(2)	N10–C19–C21	118.06(18)
C14–C13–C12	120.2(2)	N22–C21–C19	112.44(19)
C14–C13–C15	120.0(2)	C27–N22–C21	112.32(17)
C12–C13–C15	119.7(2)	C27–N22–C23	108.88(17)
C9–C14–C13	119.5(2)	C21–N22–C23	110.3(2)
F18B–C15–F17B	57.0(15)	N22–C23–C24	109.9(2)
F18B–C15–F16A	126.1(6)	O25–C24–C23	112.0(2)
F17B–C15–F16A	107.9(13)	C26–O25–C24	109.76(19)
F18B–C15–F16B	107.6(7)	O25–C26–C27	112.2(2)
F17B–C15–F16B	131.3(7)	N22–C27–C26	109.8(2)
F16A–C15–F16B	39.4(6)		

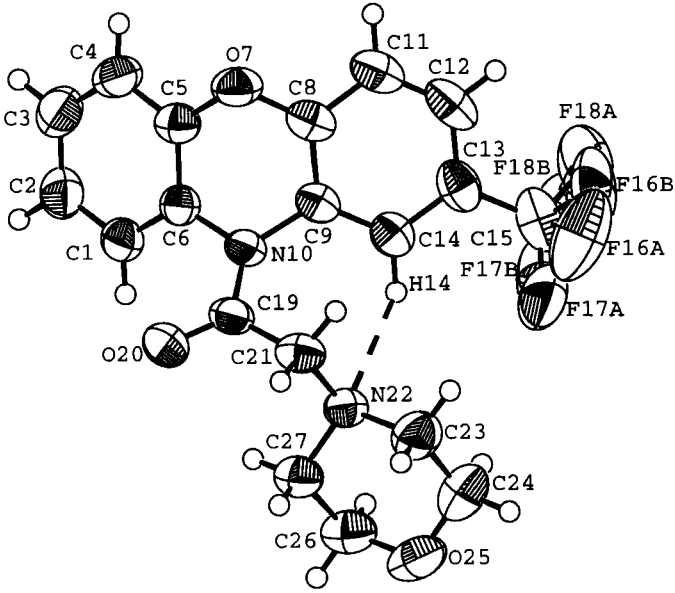

FIGURE 6 ORTEP of **C** at 50% probability.

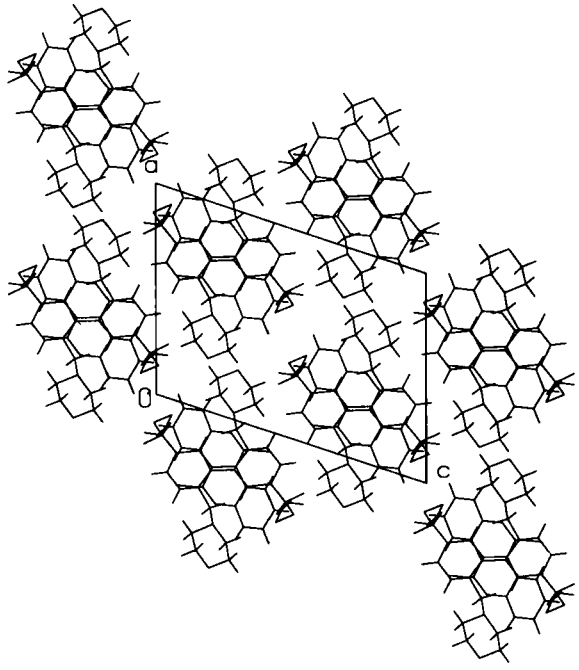

FIGURE 7 Packing down *b* of molecules of **C**.

TABLE XV Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **D**

Atom	*x*	*y*	*z*	U_{eq}
C1	0.3095(10)	0.1416(6)	0.1927(6)	0.080(2)
C2	0.1674(7)	0.1178(5)	0.2129(5)	0.0650(17)
C3	0.1566(7)	0.0690(5)	0.2981(4)	0.0640(18)
C4	0.2922(7)	0.0328(6)	0.3617(5)	0.0690(19)
C5	0.4338(9)	0.0559(6)	0.3420(5)	0.078(2)
C6	0.4409(8)	0.1141(7)	0.2583(6)	0.085(2)
O7	0.0353(5)	0.1393(4)	0.1441(3)	0.0728(14)
C8	−0.0868(7)	0.0604(5)	0.1477(4)	0.0610(17)
C9	−0.1036(7)	0.0122(5)	0.2337(4)	0.0570(16)
N10	0.0041(6)	0.0520(4)	0.3150(3)	0.0613(15)
C11	−0.1879(9)	0.0290(7)	0.0646(5)	0.074(2)
C12	−0.3042(9)	−0.0514(7)	0.0665(5)	0.079(2)
C13	−0.3199(7)	−0.1043(6)	0.1508(4)	0.0665(17)
C14	−0.2196(7)	−0.0727(5)	0.2354(4)	0.0618(17)
C15	−0.4450(9)	−0.1960(7)	0.1514(5)	0.080(2)
F16A	−0.394(3)	−0.3034(12)	0.139(2)	0.116(6)
F17A	−0.573(2)	−0.1511(12)	0.146(2)	0.129(10)
F18A	−0.416(4)	−0.270(3)	0.229(2)	0.199(16)
F16B	−0.464(3)	−0.271(3)	0.082(2)	0.150(9)
F17B	−0.571(2)	−0.173(4)	0.090(3)	0.239(17)
F18B	−0.488(3)	−0.202(2)	0.2293(13)	0.130(9)
C19	−0.0466(8)	0.1006(5)	0.3904(4)	0.0626(17)
O20	−0.1805(5)	0.0908(4)	0.3962(3)	0.0782(15)
C21	0.0733(7)	0.1660(6)	0.4615(4)	0.0665(18)
Cl22	−0.0088(2)	0.23772(17)	0.54924(11)	0.0849(10)

TABLE XVI Anisotropic displacement parameters ($Å^2$) for **D**

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.096(6)	0.064(4)	0.097(5)	−0.012(4)	0.057(5)	−0.007(4)
C2	0.074(4)	0.059(3)	0.066(4)	0.004(3)	0.023(3)	0.002(3)
C3	0.066(4)	0.053(3)	0.080(4)	−0.013(3)	0.032(4)	−0.006(3)
C4	0.070(5)	0.067(4)	0.071(4)	−0.002(3)	0.019(4)	0.008(3)
C5	0.084(5)	0.062(4)	0.094(5)	−0.005(4)	0.030(4)	0.004(3)
C6	0.066(5)	0.072(4)	0.121(6)	−0.026(4)	0.029(5)	−0.005(4)
O7	0.094(3)	0.062(3)	0.071(3)	0.0064(19)	0.038(3)	−0.002(2)
C8	0.075(4)	0.049(3)	0.065(4)	−0.004(3)	0.027(3)	0.001(3)
C9	0.071(4)	0.045(3)	0.061(4)	−0.002(3)	0.026(3)	0.004(3)
N10	0.078(3)	0.053(3)	0.056(3)	−0.002(2)	0.022(3)	−0.002(2)
C11	0.102(5)	0.074(4)	0.051(4)	0.006(3)	0.027(4)	0.007(4)
C12	0.086(6)	0.078(5)	0.073(5)	−0.009(4)	0.016(4)	−0.007(4)
C13	0.067(4)	0.068(4)	0.067(4)	−0.007(3)	0.019(3)	−0.003(3)
C14	0.070(4)	0.057(4)	0.062(4)	0.003(3)	0.022(3)	0.003(3)
C15	0.081(6)	0.067(5)	0.096(6)	−0.030(4)	0.031(5)	−0.001(5)
F16A	0.098(11)	0.081(8)	0.167(19)	−0.035(10)	0.027(12)	−0.038(7)
F17A	0.116(13)	0.080(8)	0.21(3)	0.022(10)	0.076(15)	0.006(7)
F18A	0.22(2)	0.136(17)	0.18(2)	0.089(18)	−0.092(18)	−0.115(16)
F16B	0.091(12)	0.206(18)	0.150(16)	−0.125(15)	0.023(11)	−0.074(12)
F17B	0.077(13)	0.36(3)	0.23(2)	0.20(2)	−0.073(14)	−0.098(17)
F18B	0.197(17)	0.119(13)	0.102(13)	−0.026(10)	0.094(14)	−0.090(12)
C19	0.080(4)	0.058(3)	0.057(4)	0.006(3)	0.030(3)	0.005(3)
O20	0.070(3)	0.101(3)	0.070(3)	−0.013(2)	0.029(2)	−0.007(2)
C21	0.082(4)	0.068(4)	0.051(3)	−0.008(3)	0.016(3)	−0.005(3)
Cl22	0.1010(17)	0.0954(15)	0.0609(14)	−0.0126(8)	0.0232(10)	0.0040(10)

TABLE XVII Bond lengths (Å) for **D**

Atoms	*Length*	*Atoms*	*Length*
C1–C6	1.371(11)	C12–C13	1.388(10)
C1–C2	1.381(9)	C13–C14	1.397(9)
C2–O7	1.388(8)	C13–C15	1.495(9)
C2–C3	1.371(9)	C15–F17A	1.228(18)
C3–C4	1.408(9)	C15–F16A	1.279(16)
C3–N10	1.440(8)	C15–F18B	1.274(13)
C4–C5	1.373(9)	C15–F16B	1.283(13)
C5–C6	1.386(10)	C15–F17B	1.295(19)
O7–C8	1.393(7)	C15–F18A	1.364(18)
C8–C11	1.383(10)	F16A–F18A	1.41(5)
C8–C9	1.394(8)	F16B–F17B	1.44(6)
C9–C14	1.388(8)	C19–O20	1.216(7)
C9–N10	1.419(8)	C19–C21	1.495(9)
N10–C19	1.377(7)	C21–Cl22	1.781(6)
C11–C12	1.358(10)		

TABLE XVIII Bond angles (°) for **D**

Atoms	*Angle*	*Atoms*	*Angle*
C6–C1–C2	119.4(7)	F16A–C15–F18B	105.2(19)
C1–C2–O7	119.0(6)	F17A–C15–F16B	104(2)
C1–C2–C3	120.7(7)	F16A–C15–F16B	45.3(11)
O7–C2–C3	120.2(5)	F18B–C15–F16B	130.8(15)
C2–C3–C4	119.0(6)	F17A–C15–F17B	39.8(18)
C2–C3–N10	117.2(6)	F16A–C15–F17B	110(3)
C4–C3–N10	123.7(5)	F18B–C15–F17B	104.3(18)
C5–C4–C3	120.3(6)	F16B–C15–F17B	68(3)
C4–C5–C6	119.0(7)	F17A–C15–F18A	107(2)
C1–C6–C5	121.1(6)	F16A–C15–F18A	64(3)
C2–O7–C8	114.4(4)	F18B–C15–F18A	43.6(17)
C11–C8–O7	118.9(5)	F16B–C15–F18A	104(3)
C11–C8–C9	121.1(6)	F17B–C15–F18A	131.7(13)
O7–C8–C9	120.0(5)	F17A–C15–C13	114.7(8)
C14–C9–C8	119.3(5)	F16A–C15–C13	108.8(9)
C14–C9–N10	123.9(5)	F18B–C15–C13	114.2(8)
C8–C9–N10	116.8(5)	F16B–C15–C13	113.0(9)
C19–N10–C9	120.2(5)	F17B–C15–C13	113.6(12)
C19–N10–C3	124.6(5)	F18A–C15–C13	113.0(10)
C9–N10–C3	112.7(4)	C15–F16A–F18A	60.7(16)
C8–C11–C12	119.7(6)	C15–F18A–F16A	54.9(14)
C13–C12–C11	120.4(7)	C15–F16B–F17B	56.4(17)
C12–C13–C14	120.6(6)	C15–F17B–F16B	55.7(16)
C12–C13–C15	119.9(6)	O20–C19–N10	121.0(6)
C14–C13–C15	119.5(6)	O20–C19–C21	123.7(5)
C13–C14–C9	118.9(6)	N10–C19–C21	115.2(5)
F17A–C15–F16A	134.8(14)	C19–C21–Cl22	111.3(4)
F17A–C15–F18B	67.9(13)		

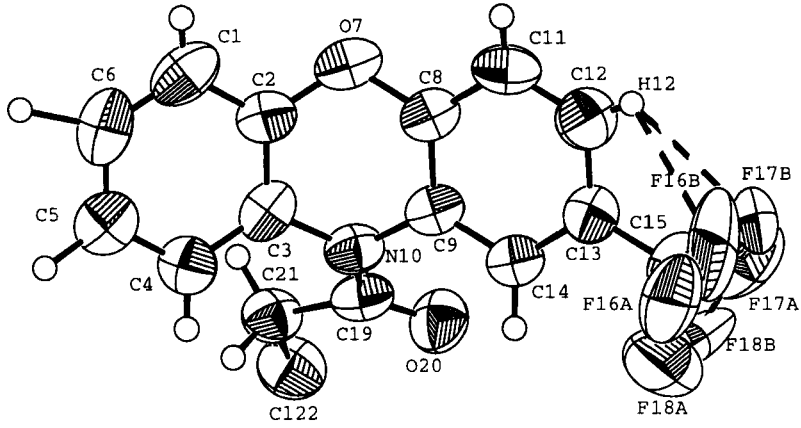

FIGURE 8 ORTEP of **D** at 50% probability.

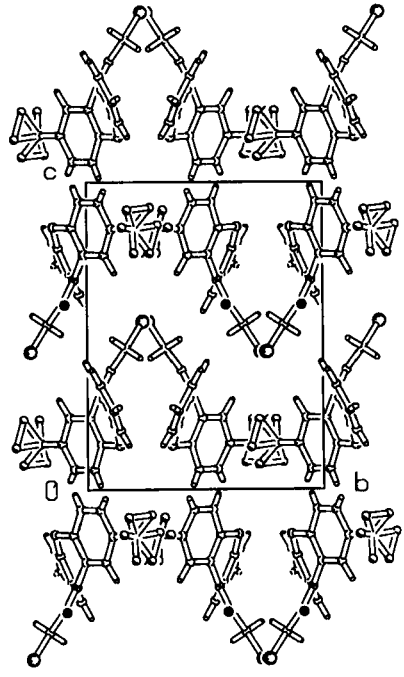

FIGURE 9 Packing down *a* of molecules of **D**.

TABLE XIX Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **E**

Atom	*x*	*y*	*z*	U_{eq}
C1	0.8761(6)	−0.1691(6)	0.2381(4)	0.0750(16)
C2	0.9139(5)	−0.0334(6)	0.2119(3)	0.0665(14)
C3	0.8431(5)	0.0832(5)	0.1922(3)	0.0694(15)
C4	0.7322(5)	0.0605(6)	0.1903(4)	0.0770(16)
C5	0.6937(6)	−0.0745(6)	0.2176(4)	0.0830(17)
C6	0.7654(6)	−0.1860(7)	0.2415(4)	0.0853(18)
O7	1.0241(3)	−0.0177(4)	0.2028(2)	0.0742(11)
C8	1.0434(4)	0.0765(6)	0.1312(3)	0.0692(15)
C9	0.9755(5)	0.1971(6)	0.1103(3)	0.0691(14)
N10	0.8902(4)	0.2194(4)	0.1663(3)	0.0688(12)
C11	1.1302(5)	0.0469(7)	0.0829(4)	0.0781(16)
C12	1.1496(6)	0.1432(8)	0.0126(4)	0.0860(19)
C13	1.0805(6)	0.2601(7)	−0.0111(4)	0.0830(17)
C14	0.9939(5)	0.2871(7)	0.0371(4)	0.0778(16)
C15	0.8771(5)	0.3523(6)	0.2071(4)	0.0762(16)
O16	0.9226(4)	0.4624(4)	0.1827(3)	0.0954(14)
C17	0.8061(5)	0.3599(6)	0.2840(4)	0.0752(16)
N18	0.8465(3)	0.2650(4)	0.3614(3)	0.0651(12)
C19	0.7612(4)	0.2340(6)	0.4189(4)	0.0787(16)
C20	0.8028(6)	0.1305(7)	0.4960(5)	0.100(2)
C21	0.9021(6)	0.1913(9)	0.5545(5)	0.107(2)
C22	0.9882(5)	0.2274(8)	0.4935(4)	0.099(2)
C23	0.9429(5)	0.3298(6)	0.4160(4)	0.0831(17)

TABLE XX Anisotropic displacement parameters ($Å^2$) for **E**

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.110(5)	0.055(3)	0.059(3)	0.003(3)	0.010(3)	0.010(3)
C2	0.082(4)	0.065(3)	0.051(3)	−0.005(2)	0.002(2)	0.003(3)
C3	0.092(4)	0.055(3)	0.058(3)	0.007(2)	0.001(3)	−0.001(3)
C4	0.089(4)	0.066(4)	0.074(3)	0.000(3)	0.006(3)	0.007(3)
C5	0.097(4)	0.066(4)	0.085(4)	0.005(3)	0.009(3)	−0.007(4)
C6	0.116(6)	0.059(3)	0.082(4)	0.004(3)	0.016(4)	−0.002(4)
O7	0.084(3)	0.071(2)	0.064(2)	0.0048(17)	−0.0014(18)	0.0058(18)
C8	0.087(4)	0.063(3)	0.055(3)	0.002(3)	−0.001(3)	−0.010(3)
C9	0.089(4)	0.064(3)	0.052(3)	−0.002(3)	0.001(3)	−0.008(3)
N10	0.089(3)	0.056(3)	0.061(3)	0.006(2)	0.008(2)	−0.004(2)
C11	0.079(4)	0.085(3)	0.069(4)	−0.020(3)	0.006(3)	−0.008(3)
C12	0.096(5)	0.098(5)	0.065(4)	−0.019(3)	0.015(3)	−0.023(4)
C13	0.106(5)	0.082(4)	0.062(3)	−0.005(3)	0.014(3)	−0.020(4)
C14	0.096(4)	0.074(4)	0.061(3)	0.005(3)	0.005(3)	−0.014(3)
C15	0.103(4)	0.055(3)	0.068(3)	0.011(3)	0.000(3)	−0.001(3)
O16	0.137(4)	0.059(2)	0.093(3)	0.004(2)	0.025(3)	−0.011(2)
C17	0.095(4)	0.059(3)	0.072(3)	0.004(3)	0.014(3)	0.012(3)
N18	0.069(3)	0.062(2)	0.064(2)	0.006(2)	0.006(2)	0.002(2)
C19	0.076(4)	0.077(4)	0.085(4)	0.003(3)	0.017(3)	−0.001(3)
C20	0.113(5)	0.096(4)	0.096(5)	0.035(4)	0.033(4)	0.002(4)
C21	0.106(5)	0.136(6)	0.078(4)	0.015(4)	0.004(4)	0.012(4)
C22	0.083(4)	0.135(6)	0.075(4)	0.011(4)	−0.001(3)	0.006(4)
C23	0.081(4)	0.086(3)	0.080(4)	−0.003(3)	0.006(3)	−0.004(3)

TABLE XXI Bond lengths (Å) for **E**

Atoms	*Length*	*Atoms*	*Length*
C1–C6	1.379(8)	N10–C15	1.369(6)
C1–C2	1.392(7)	C11–C12	1.394(8)
C2–C3	1.380(7)	C12–C13	1.379(9)
C2–O7	1.389(6)	C13–C14	1.374(8)
C3–C4	1.378(8)	C15–O16	1.224(6)
C3–N10	1.441(6)	C15–C17	1.512(8)
C4–C5	1.394(8)	C17–N18	1.457(6)
C5–C6	1.361(8)	N18–C19	1.456(6)
O7–C8	1.396(6)	N18–C23	1.463(7)
C8–C11	1.383(8)	C19–C20	1.504(8)
C8–C9	1.389(7)	C20–C21	1.502(9)
C9–C14	1.387(7)	C21–C22	1.508(9)
C9–N10	1.428(7)	C22–C23	1.514(8)

TABLE XXII Bond angles (°) for **E**

Atoms	*Angle*	*Atoms*	*Angle*
C6–C1–C2	118.5(6)	C15–N10–C3	125.0(4)
C3–C2–O7	120.0(5)	C9–N10–C3	112.3(4)
C3–C2–C1	121.0(6)	C8–C11–C12	118.1(6)
O7–C2–C1	119.0(5)	C13–C12–C11	120.7(6)
C4–C3–C2	119.1(5)	C14–C13–C12	120.3(6)
C4–C3–N10	123.8(5)	C13–C14–C9	120.3(6)
C2–C3–N10	116.8(5)	O16–C15–N10	120.9(5)
C3–C4–C5	120.0(6)	O16–C15–C17	120.7(5)
C6–C5–C4	119.8(6)	N10–C15–C17	118.4(5)
C5–C6–C1	121.2(6)	N18–C17–C15	112.1(4)
C2–O7–C8	113.6(4)	C17–N18–C19	111.3(4)
C11–C8–C9	121.7(5)	C17–N18–C23	110.6(4)
C11–C8–O7	118.7(5)	C19–N18–C23	111.2(4)
C9–C8–O7	119.6(5)	N18–C19–C20	110.6(5)
C8–C9–C14	118.8(6)	C19–C20–C21	111.4(5)
C8–C9–N10	116.9(4)	C22–C21–C20	109.1(5)
C14–C9–N10	124.3(5)	C21–C22–C23	110.9(5)
C15–N10–C9	120.8(4)	N18–C23–C22	110.4(5)

The bond distances and angles are in agreement with the standard values. ORTEP [27] of the molecule at 50% probability is shown in Figure 10. The substituted cyclohexane rings of the alternate molecules form one layer while the phenoxazine ring forms another parallel layer when viewed down *b* (Fig. 11). The phenyl rings of the phenoxazine are planar while the central ring is not so (Tab. VI). The heterocyclic ring shows chair configuration with N18 displaced by the plane (C19, C20, C22 and C23) by −0.66 Å and C21 by 0.66 Å [24–26].

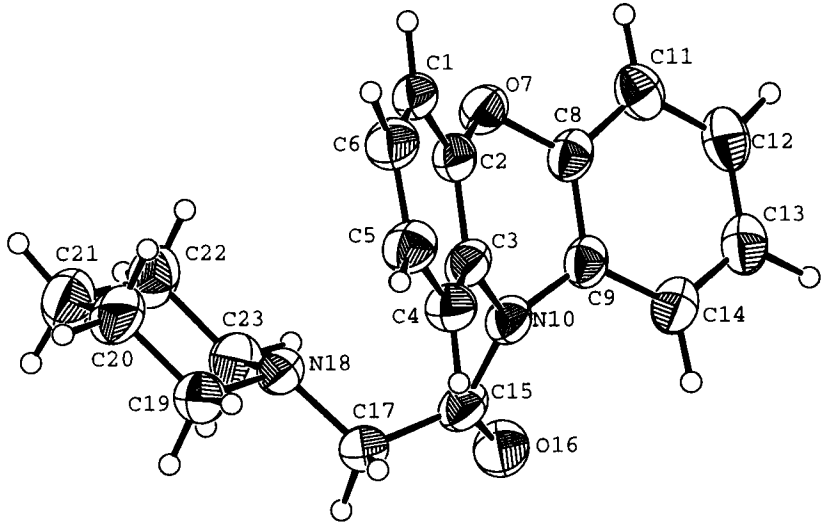

FIGURE 10 ORTEP of **E** at 50% probability.

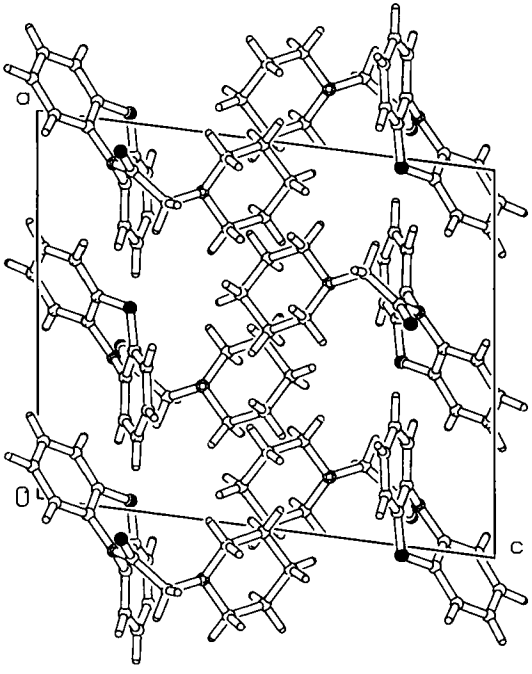

FIGURE 11 Packing down *b* of molecules of **E**.

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