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4-Alkyloxy-4'-stilbazoles New heterocyclic mesogens

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The synthesis of a new series of heterocyclic mesogens, the trans-4-alkyloxy-4'-stilbazoles ($C_nH_{2n+1}OC_6H_4CH=CHC_5H_4N$), is described and the phase behaviour is reported for all homologues between methyloxy (C_1) and dodecyloxy (C_{12}). For $n < 3$, the compounds are non-mesomorphic; monotropic smectic phases are observed for $n = 3$ and 4, whilst homologues with $n = 5$ to 12 show enantiotropic smectic phases. For comparison the synthesis and properties of two trans-4-alkyloxy-stilbenes are also reported. The phases are assigned on the basis of either X-ray data or optical textures.

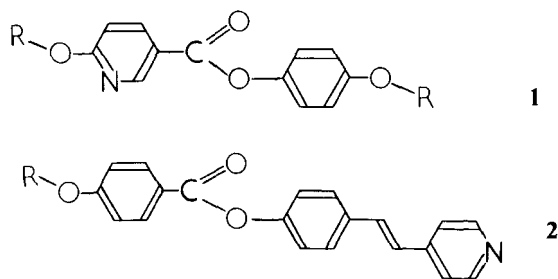
1. Introduction

Recent publications from these laboratories [1-3] have described the synthesis and properties of several series of novel transition metal containing liquid crystals, formed by the coordination of neutral, mesogenic ligands to metal centres. The ligands have come mainly from two families, the commercially available cyanobiphenyls and cyanobicyclohexyls and the trans-4-alkyloxy-4'-stilbazoles, the synthesis and properties of which are now described.

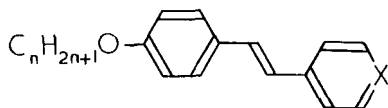
Heterocycle containing mesogens have been reported by many authors [4-8] and examples are known where the heterocycle is found either as a terminating function (2) or as a constituent of the rigid central core (1). The benzoate esters synthesised by Young *et al.* [9] (2) were found to show strong nematic behaviour. For example, the 4-stilbazole ester of *p-n*-octyloxybenzoic acid shows a nematic phase between 117 and 214°C as well as a lower temperature (95°C) crystal to crystal transition. However, the nematic thermal stability of the 2-substituted compounds (relative to the phenyl analogue) decreased. This was attributed to a molecular twist, induced by the interaction of the non-bonded electrons of the pyridine nitrogen interacting with the π -electrons of the olefinic double bond; this reduces the π -interactions in the molecule and lowers the molecular polarizability.

Work by Nash and Gray has shown that the inclusion of a pyridine or pyrimidine ring into a compound favours the formation of smectic phases; they also reduce the melting temperature (T_{cs}) relative to other heterocycles [10].

In this paper we report the synthesis and characterization of a new series of heterocyclic mesogens: the trans-4-alkyloxy-4'-stilbazoles (3) of general formula $C_nH_{2n+1}OC_6H_4CH=CHC_5H_4N$ (abbreviated to *n*-OPhVPy where *n* denotes the number of carbon atoms in the terminal alkyl chain). A series of 12 stilbazole ethers ($n = 1$ to 12) has been prepared and characterized by polarized optical microscopy and differential scanning calorimetry (D.S.C.). As a comparison, two members of the



analogous series of stilbene ethers have also been prepared: the *trans*-4-alkyloxy-stilbenes (**4**) of general formula $C_nH_{2n+1}OC_6H_4CH=CHC_6H_5$ (*n*-OPhVPh). The only mesogenic stilbene compounds which have previously been reported [9, 11–14] are the 4,4'-disubstituted stilbenes.



3 X = N

4 X = C-H

2. Results and discussion

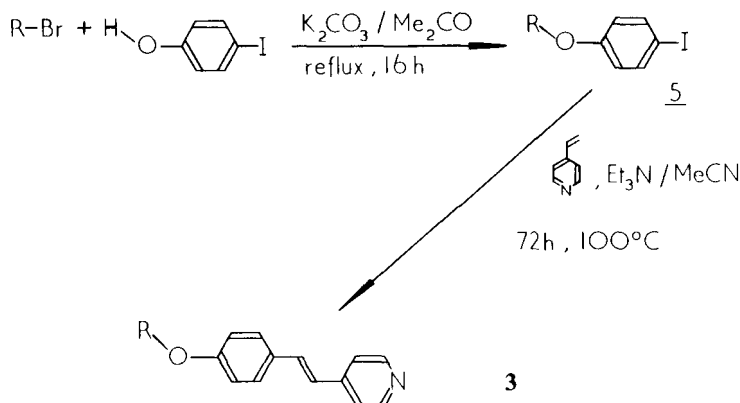
2.1. Synthesis

The *trans*-4-alkyloxy-4'-stilbazoles were first prepared using the method described by Shaw and Wagstaff [15] in which 4-picoline is condensed with an appropriate 4-alkyloxybenzaldehyde. The latter is formed in a Williamson ether synthesis using 4-hydroxybenzaldehyde and an appropriate 1-bromoalkane. The condensation reaction of the substituted benzaldehyde with 4-picoline was found to proceed in low yield, typically 1–20 per cent with respect to the starting alkyloxybenzaldehyde. The product isolated invariably contained acetic anhydride as solvent of crystallisation which proved difficult to remove. Therefore an alternative synthetic route was developed to improve both yield and purity. A Williamson ether synthesis was employed to prepare the intermediate compound 4-alkyloxyiodobenzene (**5**). The subsequent reaction of **5** with 4-vinylpyridine in a palladium acetate catalysed Heck reaction [16] (see the scheme) gave the relevant stilbazole in good yield (up to 80 per cent) and as an analytically pure white crystalline solid. The *trans*-4-alkyloxy-stilbenes (**4**) were prepared by a Williamson ether synthesis between an appropriate 1-bromoalkane and *trans*-4-hydroxy-stilbene, to give the appropriate stilbene ether as a white crystalline solid in good yield (50–60 per cent) and of analytical purity.

2.2. Mesomorphic properties

(a) *Trans*-4-alkyloxy-4'-stilbazoles

Optical studies by polarized microscopy with a heated stage yielded data on phase types and accurate transition temperatures. Typical heating and cooling



The synthetic route to trans-4-alkoxy-4'-stilbazoles (*n*-OPhVPy).

rates of 0.1 to 1.0 K min⁻¹ were employed about each transition with rates of 10.0 K min⁻¹ between widely separated transitions. These observations were then confirmed by differential scanning calorimetry.

For the series of stilbazole ethers, *n*-OPhVPy, it was found that the compounds with *n* = 1, 2 showed no mesomorphic behaviour, melting to an isotropic liquid at temperatures above 100°C and then supercooling back to the crystalline form. On extending the chain length to *n* = 3, the compound was found to melt to an isotropic liquid but to supercool to form a transient, monotropic smectic B phase. For *n* = 4, a monotropic S_B phase was observed on supercooling from the isotropic liquid which then underwent a further transition to the more highly ordered smectic E phase. For the higher homologues, *n* = 5 to 12, two enantiotropic smectic phases were observed, S_E and the less viscous S_B phase. The optical transition temperatures together with their associated transition enthalpies and entropies are presented in table 1.

The smectic E phase of the *n* = 8 and *n* = 6 homologues was identified by X-ray diffraction [17]. The layer spacing (for *n* = 8) was found to be 25.8 (0.5) Å and this compared well with the calculated value for the length of the molecule (using molecular models) of about 23 Å. The S_E phase of the other homologues was assigned on the basis of similar optical textures. The narrow temperature range of the S_B phase prevented an X-ray identification and it was therefore identified by its natural texture obtained on cooling the isotropic fluid; this showed spherulitic structures [18] around air bubbles and characteristic [19] lancets (see figure 1). Unfortunately it was not possible to distinguish between a crystal and a hexatic smectic B as the texture was in each case obtained from the isotropic state and therefore the paramorphic texture which might have distinguished the two was not accessible.

Figure 2 shows mesophase transition temperatures as a function of *n* for those compounds (*n* = 3 to 12) showing mesomorphic properties. As can be seen, the range of stability of the smectic E phase is greatest (13.3°C) when *n* = 6, whilst that of the smectic B phase is greatest (3.7°C) when *n* = 8. The C–S_E and to a lesser extent the S_E–S_B transition curves show the odd–even effect with the transition temperatures being higher when the terminal alkyl chain contains an odd number of carbon atoms. For *n* > 8 the stability of the S_E phase decreases as the three transition curves begin

Table 1. Thermal data for 4-alkyloxy-4'-stilbazoles.

Derivative	Transition	$T/^\circ\text{C}$	$\Delta H/\text{J g}^{-1}$	$\Delta S_m/R$
1-OPhVPy	C-C'	123.8	1.7	0.1
	C'-I	135.7	115.6	7.2
	I-C	105.6	-106.8	-7.2
2-OPhVPy	C-I	153.5	122.7	7.8
	I-C	135.4	-118.0	-7.8
3-OPhVPy	C-I	112.0	82.4	6.2
	I-S _B	94.9	-88.2†	-6.9†
	S _B -C	94.8		
4-OPhVPy	C-I	95.5	106.5	8.8
	I-S _B	90.5	-71.7†	-6.0†
	S _B -S _E	89.4		
5-OPhVPy	C-S _E	85.2	127.1†	11.4†
	S _E -S _B	85.7		
	S _B -I	86.8		
	I-S _B	86.1		
	S _B -S _E	85.3		
6-OPhVPy	C-S _E	73.2	60.9	6.0
	S _E -S _B	87.1	40.1†	3.8†
	S _B -I	88.5		
	I-S _B	88.4	-41.8†	-3.9†
	S _B -S _E	86.6		
7-OPhVPy	C-S _E	85.6	92.7	9.2
	S _E -S _B	88.4	42.4†	4.2†
	S _B -I	89.5		
	I-S _B	88.8	-41.3†	-4.1†
	S _B -S _E	88.2		
8-OPhVPy	C-S _E	75.2	72.0	7.7
	S _E -S _B	85.2	42.6†	4.4†
	S _B -I	88.9		
	I-S _B	88.2	-42.4†	-4.4†
	S _B -S _E	84.3		
9-OPhVPy	C-S _E	86.1	149.3†	16.1†
	S _E -S _B	87.0		
	S _B -I	87.9		
	I-S _B	87.7		
	S _B -S _E	86.4		
10-OPhVPy	C-S _E	84.5	78.3	8.9
	S _E -S _B	86.2	40.4†	4.6†
	S _B -I	87.5		
	I-S _B	86.9	-40.3†	-4.5†
	S _B -S _E	86.1		
11-OPhVPy	C-S _E	86.3	156.7†	18.3†
	S _E -S _B	87.0		
	S _B -I	89.8		
	I-S _B	87.3		
	S _B -S _E	83.6		
12-OPhVPy	C-C'	84.4	150.0†	18.3†
	C'-S _E	85.4		
	S _E -S _B	86.4		
	S _B -I	87.9		
	I-S _B	85.4		
	S _B -S _E	83.7		

† Combined enthalpies or entropies of transition.

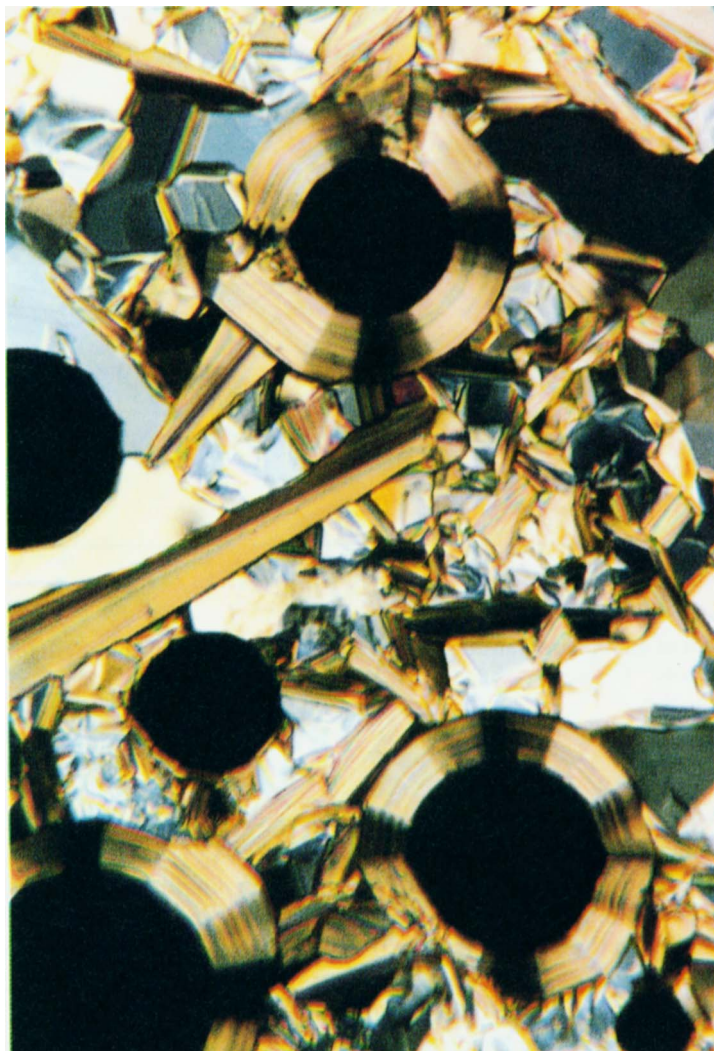


Figure 1. Photomicrograph of the smectic B phase of 8-OPhVPy at 87.4°C, obtained by cooling from the isotropic phase (magnification $\times 100$).

to converge. This is due to the fact that the increasing molecular weight causes an increase in the temperature of the C–S_E transition.

Many of the enthalpies (see table 1) determined from D.S.C. studies are given as combined enthalpies, since several of the mesophase transitions are close together in temperature and as because the observed enthalpies are large the D.S.C. peaks are broad. Resolution of such peaks proved difficult and even scan rates of 0.5 K min⁻¹ failed to give baseline separation, although the separate peaks could often be resolved. The crystallization (melting) enthalpy could be obtained because of supercooling of the S_E phase. The value appears to be dependent on the length of the terminal alkyl chain from 60.9 J g⁻¹ for 6-OPhVPy to 114.4 J g⁻¹ for the higher homologues. The S_E–S_B and S_B–I transitions could not be resolved sufficiently to obtain independent enthalpies so are always quoted as combined values (i.e. S_E–S_B–I).

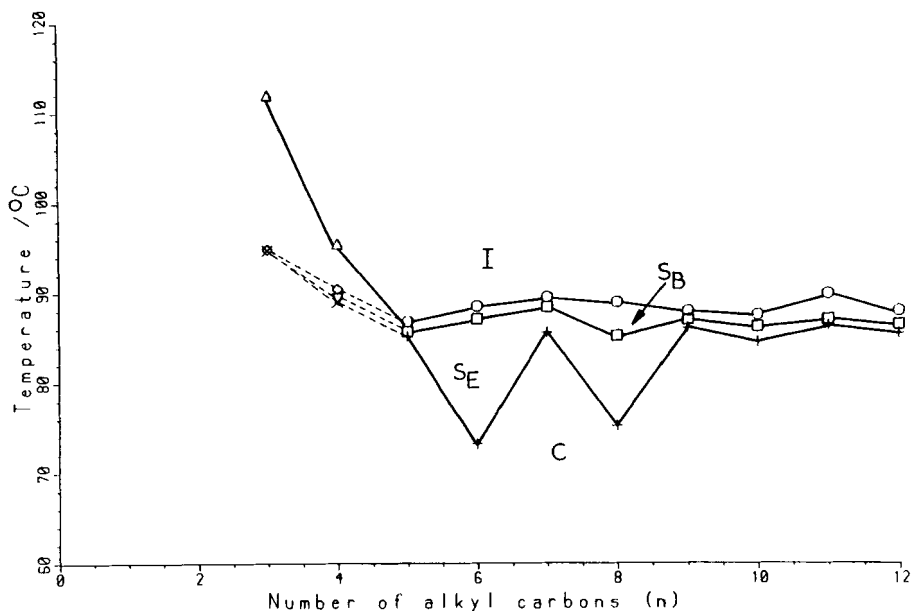


Figure 2. Plot of mesophase transition temperature against alkyl chain length for 4-alkyloxy-4'-stilbazole. C-S_E, +; S_E-S_B, □; S_B-I, ○; C-I, △; (I-S_B), ◇; (S_B-S_E), ▽; (S_B-C/S_E-C), ×.

(b) *Trans*-4-alkyloxy-stilbenes

In the stilbazole ethers, the octyl derivative (8-OPhVPy) showed the widest smectic B range. By contrast, optical studies showed the analogous 4-octyloxystilbene (8-OPhVPh) to be non-mesomorphic, melting to an isotropic liquid at 106.1°C and supercooling back to the crystalline form. However, when the alkyl chain was extended to decyl (10-OPhVPh) a monotropic smectic B phase was observed (identified by its optical texture) over a range of 0.6°C. These observations were confirmed by D.S.C., with baseline peak separation being achieved for the S_B-I and C-S_B transitions in the case of 10-OPhVPy. The properties observed are in agreement with other observations [10] which show that the crystal thermal stabilities are lower for heterocyclic compounds than in the analogous phenyl derivatives. Thermodynamic data for the two homologues determined from D.S.C. studies is presented in table 2 together with accurate transition temperatures.

Table 2. Thermal data for 4-alkyloxy-4'-stilbenes.

Derivative	Transition	$T/^\circ\text{C}$	$\Delta H/\text{J g}^{-1}$	$\Delta S_m/R$
8-OPhVPh	C-I	106.1	129.4	12.7
	I-C	96.2	-127.3	-12.8
10-OPhVPh	C-I	106.8	140.6	15.0
	I-S _B	94.0	-13.9	-1.5
	S _B -I	93.6	-116.3	-12.9

3. Experimental

1-Bromoalkanes, 4-iodophenol and 4-vinylpyridine (Aldrich) and trans-4-hydroxystilbene (Lancaster Synthesis) were used as supplied. All solvents were dried and distilled prior to use. Microanalyses were determined by the University of Sheffield Microanalytical Service. ^1H N.M.R. spectra were recorded on Perkin-Elmer R12B and Bruker AM250 spectrometers whilst ^{13}C N.M.R. spectra were recorded on the Bruker AM250 using the JMOD pulse sequence [20].

Optical characterisation was performed using a Swift polarizing microscope equipped with a Linkam hot-stage and temperature controller. Differential scanning calorimetry (D.S.C.) measurements were made on the Perkin-Elmer DSC7 system and a Perkin-Elmer DSC2 with a Perkin-Elmer 3600 data station using TADS software. Heating and cooling rates of between 0.5 and 10.0 K min^{-1} were employed with the samples encapsulated in standard aluminium pans.

Trans-4-alkyloxy-4'-stilbazole: route 1

4-Octyloxybenzaldehyde

A mixture of 4-hydroxybenzaldehyde (13.00 g, 0.11 mol), 1-bromooctane (20.56 g, 0.11 mol) and potassium hydroxide (8.00 g, 0.14 mol) was heated under reflux in ethanol (40 cm^3) for 16 hours. On cooling, the potassium bromide was removed by filtration and washed thoroughly with ethanol (100 cm^3). The ethanol was removed under vacuum to leave an orange residue which was washed with aqueous sodium hydroxide (25 cm^3 , 10 per cent). The separated organic layer was dried over sodium sulphate and distilled under vacuum to give the product as a colourless oil (b.p. = 180°C/1 mm Hg). The oil was characterized by its proton N.M.R. spectrum. Yield = 14.62 g (59 per cent).

Trans-4-octyloxy-4'-stilbazole [15]

A solution of 4-octyloxybenzaldehyde (11.00 g, 46.9 mmol) and 4-picoline (4.37 g, 46.9 mmol) in acetic anhydride (29 cm^3) was heated under reflux for 40 hours. The resultant dark oil was refrigerated for 16 hours to give a brown solid which was filtered and air dried. The filtrate was concentrated and recrystallized to yield additional solid. The dry solid was recrystallized, first from hot *n*-hexane (*c.* 500 cm^3) and secondly from cold acetone, to give the product as white crystals which were filtered and dried *in vacuo*. Yield = 1.35 g (9 per cent). Microanalysis for $\text{C}_{21}\text{H}_{27}\text{NO}$: calculated (found): C 81.5 (81.8), H 8.8 (8.7), N 4.5 (4.5).

Trans-4-alkyloxy-4'-stilbazole: route 2

4-Octyloxyiodobenzene

A mixture of 4-iodophenol (19.42 g, 88.3 mmol), 1-bromooctane (17.04 g, 88.3 mmol) and potassium carbonate (12.2 g, 88.3 mmol) in acetone (90 cm^3) was heated under reflux for 16 hours. On cooling, water was added to dissolve the potassium bromide produced and the resulting mixture extracted into ether (3 \times 50 cm^3). The combined ether extracts were washed with aqueous solutions of sodium chloride (50 cm^3 , saturated) and sodium hydroxide (50 cm^3 , 10 per cent) and the separated ethereal layer dried over sodium sulphate. After filtration and evaporation of the solvent, the resultant pale yellow oil was purified by distillation under vacuum. The pure

Table 3. Experimental data for 4-alkoxyiodobenzenes ($C_nH_{2n+1}OC_6H_4I$).

<i>n</i> -	Formula	Microanalysis: calc (found)			<i>T</i> †/°C	Percentage yield
		C	H	I		
1-	C ₇ H ₇ IO	Used as supplied			48–50‡	—
2-	C ₈ H ₉ IO	38.7 (38.2)	3.7 (3.8)	51.2 (51.3)	145/0.3 mm	76
3-	C ₉ H ₁₁ IO	41.2 (42.0)	4.2 (4.2)	48.4 (47.5)	149/0.3 mm	68
4-	C ₁₀ H ₁₃ IO	43.5 (44.0)	4.7 (4.7)	46.0 (45.6)	141/0.1 mm	68
5-	C ₁₁ H ₁₅ IO	45.5 (45.4)	5.2 (5.3)	43.7 (43.4)	159/1.3 mm	67
6-	C ₁₂ H ₁₇ IO	47.4 (47.5)	5.6 (5.7)	41.7 (42.3)	138/1.7 mm	63
7-	C ₁₃ H ₁₉ IO	49.1 (49.3)	6.0 (5.8)	39.9 (39.2)	205/3.5 mm	62
8-	C ₁₄ H ₂₁ IO	50.6 (50.4)	6.4 (6.1)	38.2 (37.7)	169/0.3 mm	61
9-	C ₁₅ H ₂₃ IO	52.0 (52.3)	6.7 (6.5)	36.7 (35.7)	19‡	59
10-	C ₁₆ H ₂₅ IO	53.3 (53.6)	7.0 (6.9)	35.2 (35.3)	27‡	51
11-	C ₁₇ H ₂₇ IO	54.6 (54.9)	7.3 (7.2)	33.9 (33.4)	32‡	58
12-	C ₁₈ H ₂₉ IO	55.7 (55.8)	7.5 (7.4)	32.7 (33.0)	37‡	50

† Boiling point or melting point; boiling points are given as $T^\circ\text{C}/\text{mm Hg}$.

‡ Melting points are given in $^\circ\text{C}$.

product was isolated as a colourless oil (b.p. = $169^\circ\text{C}/0.3$ mm Hg). Yield = 17.89 g (61 per cent).

For the higher homologues, nonyloxy and above, the crude products were isolated as solids which were purified by recrystallization from cold acetone rather than by distillation. The product was filtered quickly and air dried to yield the desired material as white crystalline flakes. Yields, melting/boiling points and microanalytical data are presented in table 3.

Trans-4-octyloxy-4'-stilbazole [16]

A solution of 4-octyloxyiodobenzene (15.00 g, 45.1 mmol), 4-vinylpyridine (5.29 g, 50.0 mmol) and triethylamine (4.57 g, 45.1 mmol) in acetonitrile (18.5 cm^3) was placed in a thick-walled Pyrex tube of the Fischer–Porter type under an atmosphere of argon. Palladium acetate (102.8 mg, 0.45 mmol) was added and the tube flushed thoroughly with argon. The solution was heated for 72 hours at 100°C under conditions of constant volume; the pressure rose to between 3.0 and 4.5 atm. PTFE seals were used as the acetonitrile vapour was found to attack the normal rubber seals fitted to the apparatus. On cooling, the solid formed was taken into methylene chloride (100 cm^3 , neutralized) and the solution washed with water (50 cm^3). The layers were separated and the aqueous solution washed with a further 100 cm^3 of methylene chloride. The combined organic layers were reduced to about half their original volume and dried over sodium sulphate. After filtration, the solvent was removed by rotary evaporation to yield the crude product as a yellow–orange solid which was extracted repeatedly (Soxhlet apparatus) with *n*-hexane. The resultant pale yellow solid was purified by repeated recrystallizations from cold acetone to give the product as white crystals of analytical purity. Yield = 11.16 g (80 per cent). Yields and microanalytical data for the complete series are presented in table 4.

Trans-4-octyloxy-stilbene

A mixture of *trans*-4-hydroxy-stilbene (3.80 g, 19.4 mmol), 1-bromooctane (3.80 g, 19.8 mmol) and potassium carbonate (2.68 g, 19.4 mmol) in acetone (25 cm^3) was

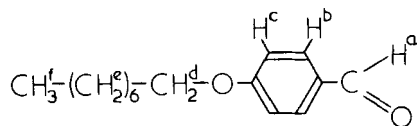
Table 4. Experimental data for 4-alkyloxy-4'-stilbazoles (*n*-OPhVPy).

<i>n</i> -	Formula	Microanalysis: calc (found)			Percentage yield
		C	H	N	
1-	C ₁₄ H ₁₃ NO	79.6 (78.9)	6.2 (6.4)	6.6 (6.3)	81
2-	C ₁₅ H ₁₅ NO	80.0 (80.1)	6.7 (7.0)	6.2 (6.0)	58
3-	C ₁₆ H ₁₇ NO	80.3 (80.1)	7.2 (7.4)	5.9 (5.6)	58
4-	C ₁₇ H ₁₉ NO	80.6 (80.3)	7.6 (7.4)	5.5 (5.2)	66
5-	C ₁₈ H ₂₁ NO	80.9 (80.5)	7.9 (8.2)	5.2 (4.8)	48
6-	C ₁₉ H ₂₃ NO	81.2 (81.2)	8.3 (8.3)	5.0 (5.0)	57
7-	C ₂₀ H ₂₅ NO	81.3 (81.4)	8.5 (8.6)	4.7 (4.9)	72
8-	C ₂₁ H ₂₇ NO	81.5 (82.2)	8.8 (8.8)	4.5 (4.5)	80
9-	C ₂₂ H ₂₉ NO	81.7 (81.5)	9.0 (9.0)	4.3 (4.4)	49
10-	C ₂₃ H ₃₁ NO	81.9 (81.8)	9.3 (9.0)	4.2 (4.4)	52
11-	C ₂₄ H ₃₃ NO	82.0 (82.3)	9.5 (9.4)	4.0 (3.7)	54
12-	C ₂₅ H ₃₅ NO	82.1 (82.4)	9.7 (9.6)	3.8 (3.6)	49

heated under reflux for 16 hours. Water (50 cm³) was then added to the cooled reaction mixture to dissolve the potassium bromide produced and the product extracted into ether (2 × 50 cm³). The combined ethereal extracts were washed with aqueous solutions of sodium chloride (50 cm³, saturated) and sodium hydroxide (50 cm³, 10 per cent) and the separated organic layer dried over sodium sulphate. Filtration and rotary evaporation of the ether yielded the crude product as an off-white solid. Recrystallization from cold acetone gave the product as white crystalline flakes which were filtered and oven dried. Yield = 3.13 g (52 per cent). Microanalysis for C₂₂H₂₈O: calculated (found): C 85.7 (86.0), H 9.1 (9.4). Trans-4-decyloxy-4'-stilbene: yield = 64 per cent. Microanalysis for C₂₄H₃₂O: calculated (found): C 85.7 (85.5), H 9.6 (9.4).

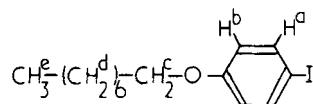
¹H N.M.R. data

4-Octyloxybenzaldehyde: 60 MHz, CDCl₃



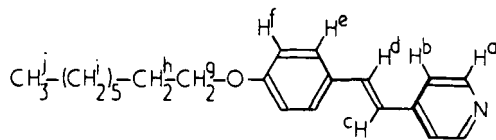
δ 9.9 (*s*, 1H, H^a), 7.8 (*d*, 2H, H^b), 7.0 (*d*, 2H, H^c), 4.1 (*t*, 2H, H^d), 1.3 (*m*, 12H, H^e), 0.9 (*t*, 3H, H^f).

4-Octyloxyiodobenzene: 60 MHz, CDCl₃



δ 7.6 (*d*, 2H, H^b, *J* = 8.7 Hz), 6.7 (*d*, 2H, H^a, *J* = 8.8 Hz), 4.0 (*t*, 2H, H^c, *J* = 6.1 Hz), 1.3 (*m*, 12H, H^d), 0.9 (*t*, 3H, H^e).

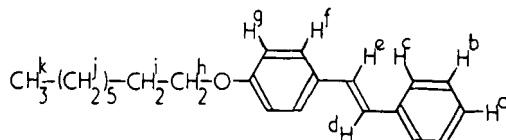
4-Octyloxy-4'-stilbazole: 250 MHz, CDCl₃



The aromatic protons appear as *AA'XX'* spin systems and are analysed as such. The olefinic protons appear as an *AB* spin system so modulated coupling constants are cited.

δ 8.49 (*AA'XX'*, 2H, H^a, $J = 6.0$ Hz), 7.57 (*AA'XX'*, 2H, H^f, $J = 8.7, 0.2$ Hz), 7.46 (*AA'XX'*, 2H, H^b, $J = 6.1$ Hz), 7.45 (*AB*, 1H, H^d, $|J| = 16.3$ Hz), 7.06 (*AB*, 1H, H^c, $|J| = 16.3$ Hz), 6.94 (*AA'XX'*, 2H, H^e, $J = 8.7, 0.2$ Hz), 4.2 (*t*, 2H, H^s, $J = 6.7$ Hz), 1.76 (*qn*, 2H, H^h, $J = 6.8$ Hz), 1.29 (*m*, 10H, Hⁱ), 0.88 (*t*, 3H, H^j, $J = 6.8$ Hz).

Trans-4-octyloxy-stilbene: 250 MHz, CDCl₃



The monosubstituted phenyl ring appears as a 5-proton spin system and as such coupling constants are unassignable. The disubstituted phenyl ring appears as an *AA'XX'* spin system and is assigned as such. The olefinic protons appear as an *AB* spin system so modulated coupling constants are cited.

δ 7.50 (2H, H^c), 7.45 (*AA'XX'*, 2H, H^g), 7.35 (2H, H^b), 7.24 (1H, H^a), 7.08 (*AB*, 1H, H^c, $|J| = 16.0$ Hz), 6.98 (*AB*, 1H, H^d, $|J| = 16.3$ Hz), 6.90 (*AA'XX'*, 2H, H^f, $J = 8.9, 0.1$ Hz), 3.97 (*t*, 2H, H^h, $J = 6.7$ Hz), 1.79 (*qn*, 2H, Hⁱ, $J = 6.9$ Hz), 1.32 (*m*, 10H, H^j), 0.91 (*t*, 3H, H^k, $J = 6.7$ Hz).

¹³C N.M.R. data

Trans-4-octyloxy-4'-stilbazole: 250 MHz, CDCl₃

δ 13.93 (CH₃-); 22.52, 25.89, 29.09, 31.68 (-CH₂-); 67.91 (-CH₂-O-); 114.60, 120.4 (-CH=CH-); 123.24, 128.18, 132.60, 149.75 (=CH- arom.); 128.41, 144.84, 159.58 (=C < arom.).

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