Clathrate Formation with Troeger Base Analogues

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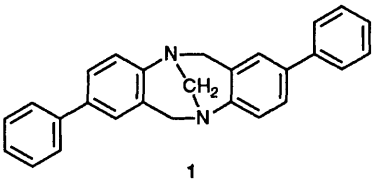
Several quaternary Troeger base analogues, **1a** and **2a–d** have been prepared and their inclusion characteristics investigated. The X-ray crystal structure of a novel Troeger base analogue **1**, 2,8-diphenyl-5,6,11,12-tetrahydro-5,11-methanodibenzo[*b,f*][1,5]diazocine, is reported.

The formation of clathrate-type† inclusion compounds by salts of Troeger's base (2,8-phenyl-5,6,11,12-tetrahydro-5,11-methanodibenzo[*b,f*][1,5]diazocine **2a**) was inadvertantly achieved in 1968 by Häring in the course of an attempt to synthesize various *N*-substituted analogues.[2] Weber and co-workers recognised that the purification difficulties encountered by Häring were in fact due to solvent enclathration. This prompted a systematic study of the inclusion properties of this salt[3] **2a** and various 2,8-substituted and *N*-quaternised analogues thereof.



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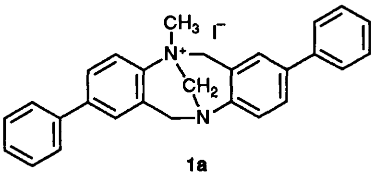
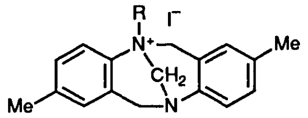


1a



2a R = Me
2b R = Et
2c R = $(CH_2)_2CH_3$
2d R = $(CH_2)_4$ (bridging butyl group)

Organic onium compounds have important functions in biological processes and Troeger base analogues may provide suitable chiral,‡ conformationally rigid skeletons for the synthesis of chelating or biomimetic systems.[4]

In order to design specific, effective Troeger-base onium compounds for use as host molecules, it is desirable to develop an understanding of the factors affecting inclusion and to determine the effect of structural modifications.

To this end the preparation of several new Troeger base analogues **1a** and **2b–d** are described and their inclusion properties investigated. The X-ray crystal structure of a racemic mixture of the 2,8-diphenyl parent system **1** is presented and the first report made of the formation of an inclusion compound by such a molecule.

Experimental

General.—Paraformaldehyde was used as supplied (BDH Chemicals Ltd.), whilst 4-aminobiphenyl (Eastman Kodak Company, Rochester NY) was recrystallised from water before use. All solvents were distilled and dried in the appropriate manner. M.p.s were determined on a CO6000/TH00 LINKAM hotstage with Nikon SM2-10 microscope and are uncorrected. Microanalyses were performed on a Haraeus combustion analyser, model CHN-RAPID. All samples were vacuum dried (2 mmHg) at 30 °C for 1 h before being used. ^1H NMR spectra were recorded on a Bruker WH90 or Varian VXR200 spectrometer in $CDCl_3$ solution using Me_4Si as an internal reference. Mass spectra were determined on a V.G. Micromass 16F mass spectrometer operating at 70 eV in a temperature range with varying lower limit to ± 220 °C. Thermal analyses were performed on a V2.1A DuPont 9900 thermal analyser or a Stanton Redcroft thermal analyser STA 780 at a uniform heating rate of 5 °C min^{-1} under a 30 cm^3 min^{-1} flow of nitrogen.

Preparation of Troeger Base **1**.—The preparation followed the method of Wagner.[5] 4-Aminobiphenyl (recrystallised) dissolved in glacial acetic acid was acidified with conc. HCl (34%) with cooling and stirring. A 1.5 molar excess of paraformaldehyde was added in portions and the solution/suspension stirred for 20 h at 15–20 °C. Solid paraformaldehyde was removed by filtration and the solvent removed under reduced pressure. The resultant residue was dissolved in ethanol and dilute NH_3 solution (20 cm^3 conc. NH_3 in 100 cm^3 H_2O) was added to pH 12. A yellow solid precipitated and the solution was stirred overnight to effect complete precipitation. The solid product was collected by filtration, dried under vacuum and then recrystallised from propan-2-ol or ethyl acetate to yield compound **1** as a fine yellow powder (36%); m.p. 200–202 °C (Found: C, 86.0; H, 5.75; N, 7.6. $C_{27}H_{22}N_2$ requires C, 86.60; H, 5.92; N, 7.48%); δ_H(200 MHz) 7.2–7.8 (16 H, br m, Ar), 4.85 (2 H, d, CH_2), 4.43 (2 H, s, CH_2) and 4.34 (2 H, d, CH_2); *m/z* 374 (M^+, 8%), 373 (30), 372 (100), 357 (15), 186 (17) and 152 (17) and fragmentations thereof.

General Method for the Alkylation of Troeger base Derivatives.—Preparation of the monomethylated Troeger base **1a**. A general alkylation method[2] was employed. To a solution of Troeger base **1** in tetrahydrofuran (THF) was added a 1.3 molar excess of methyl iodide. After being refluxed for 3 h the solution was cooled and the resultant pale yellow precipitate collected by filtration, washed and dried under vacuum. Alternatively, a 1.5 molar excess of methyl iodide was added to a solution of

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† Using the nomenclature described by Weber, ref. 1.

‡ The molecule is chiral solely due to difficulty of inversion at N.

Troeger base **1** in THF and stirred at room temperature overnight. The product **1a** was well dried (3 h at 50 °C/540 mmHg) to remove small amounts of clathrated iodomethane and then recrystallised from ethyl acetate–propan-2-ol (50:50): (79.8%), m.p. 199–202 °C (Found: C, 65.6; H, 4.9; N, 5.4. $C_{28}H_{25}N_2I$ requires C, 65.12; H, 4.88; N, 5.42%); δ_H(200 MHz) 8.28 (1 H, d, Ar), 7.2–7.9 (15 H, br m, Ar), 4.3–6.0 (6 H, br m, CH_2) and 4.15 (3 H, s, $N^+ - CH_3$); *m*/*z* 374 (M^+ − MeI, 100%), 358 (27), 345 (17), 193 (50), 187 (43), 165 (55), 142 (MeI^+, 70) and 127 (I^+, 25) and fragmentations thereof.

*Preparation of a series of monoalkylated analogues of the Troeger base, 2,8-dimethyl-5,11-methano-6*H*,12*H*-tetrahydrodibenzo*[b,f][1,5]*diazocine.* *N*-Alkyl iodide salt derivatives of Troeger's base were prepared by reaction with the appropriate alkyl iodides.

2a* (R = Me), yield 92.8%, m.p. 158–162 °C (Found: C, 55.1; H, 5.4; N, 7.1. $C_{18}H_{21}IN_2$ requires: C, 55.11; H, 5.39; N, 7.14%); δ_H(90 MHz) 7.98 (1 H, d, Ar), 7.25 (3 H, m, Ar), 6.87 (2 H, s, Ar), 4.8–5.7 (5 H, br m, CH_2), 4.19 (1 H, d, CH_2), 3.99 (3 H, s, $N^+ - CH_3$), 2.28 (3 H, s, Ar-C*H*$_3$) and 2.22 (3 H, s, Ar-C*H*$_3$); *m*/*z* 250 (M^+ − MeI, 100%), 249 (80), 235 (12) and 142 (50, MeI^+) and fragmentations thereof.

2b (R = Et), yield 51.8%, m.p. 137–142 °C (Found: C, 55.45; H, 5.75; N, 6.45. $C_{19}H_{23}IN_2$ requires C, 56.17; H, 5.71; N, 6.89%); δ_H(90 MHz) 8.0 (1 H, d, Ar), 6.8–7.4 (5 H, br m, Ar), 3.9–5.8 (11 H, br m, CH_2 and $N^+ - CH_2CH_3$), 2.2 (3 H, s, Ar-C*H*$_3$) and 1.78 (3 H, br m, Ar-C*H*$_3$); *m*/*z* 264 (M^+ − MeI, 50%), 250 (M^+ − EtI, 75), 234 (20), 142 (45) and 120 (30) and fragmentations thereof.

2c [R = $(CH_2)_2CH_3$], yield 14%, m.p. 148–151 °C (Found: C, 57.4; H, 6.05; N, 6.65. $C_{20}H_{25}IN_2$ requires C, 57.14; H, 5.99; N, 6.68%); δ_H(90 MHz) 7.95 (1 H, d, Ar), 6.8–7.4 (5 H, br m, Ar), 4.0–5.8 (13 H, br m, CH_2 and $N^+ - CH_2CH_2CH_3$) and 2.22 (6 H, s, CH_3); *m*/*z* 278 (M^+ − MeI, 33%), 250 (100), 235 (13), 142 (10) and 120 (20) and fragmentations thereof.

2d [R = $-(CH_2)_4$, bridging butyl group], yield 5.87%, m.p. 162–168 °C (Found: C, 56.05; H, 5.65; N, 6.5. $C_{38}H_{44}I_2N_4$ requires: C, 56.31; H, 5.47; N, 6.91%); δ_H(200 MHz) 7.95 (2 H, d, Ar), 6.8–7.4 (10 H, br m, Ar), 4.0–5.8(20 H, br m, N-CH_2 and bridging butyl group protons), 2.23 (6 H, s, CH_3) and 2.19 (6 H, s, CH_3); *m*/*z* 418 ($M^+ - C_{18}H_{19}N_2I$, 30%), 291 (22), 249 (46), 145 (46) and 142 (40) and fragmentations thereof.

Preparation and Characterisation of the Inclusion Compounds.—The inclusion capabilities of the 2,8-diphenyl Troeger base analogue **1** were investigated although no report of inclusion by other *N*-unsubstituted Troeger bases has appeared in the surveyed literature. The inclusion compounds were crystallised from a solution of host in potential guest solvent. The crystalline materials obtained were characterised by mass spectrometry and ^1H NMR spectroscopy.

Inclusion compounds of the *N*-alkylated iodide salts, **1a** and **2a–d**, were prepared by a suspension method as follows: a small quantity (0.15 g) of the salt was stirred, with heating, in a large excess of the potential guest solvent. In many cases the fine pale suspension changed character, forming a denser, clumpy (usually darker), solid which was filtered, washed with dry diethyl ether and dried under vacuum for 8 h. Characterisation of these inclusion compounds was achieved by: mass spectrometry, to determine the presence of a peak corresponding to the mass ion of the guest; ^1H NMR spectroscopy, to obtain host:guest ratios by comparison of integration values; microanalyses, to confirm the NMR spectroscopically derived stoicheiometries; and differential thermal analysis (DTA) and thermogravimetry (TG), to confirm that the % weight loss corresponding to loss of guest agreed with the derived host:guest stoicheiometries. The inclusion characteristics of compound **1a** with respect to a large number of potential guest solvents were tested. In addition, inclusion compounds of **2a–d** with *p*-xylene were prepared in order to study the effect of increasing the bulk of the *N*-alkyl substituent.

X-Ray Crystal Data.—Compound **1** $C_{27}H_{22}N_2$, $M_r = 374.5$, monoclinic, $a = 6.185(3)$ Å, $b = 56.474(8)$ Å, $c = 6.509(2)$ Å, $\beta = 118.35(3)°$, $U = 2001(1)$ Å^3, space group *Cc*, $Z = 4$, $D_c = 1.24$ g cm^{-3}, $\lambda = 0.710\,69$ Å, μ(Mo-Kα) = 0.38 cm^{-1}, $F(000) = 792$. Approximate crystal dimensions: 0.39 × 0.50 × 0.50 mm^3.

Crystal Data Collection and Processing.—3584 independent reflections were measured on an Enraf-Nonius CAD4 diffractometer using graphite monochromated Mo-Kα radiation, 1996 were observed with $[I_{rel} > 2\sigma(I_{rel})]$. All data were collected in the ω–2θ scan mode with variable scan and aperture widths and a maximum recording time of 40 s. Intensities of three standard reflections were checked every hour. Recentring was carried out every 200 measured reflections and Lorentz polarisation and absorption corrections were applied.

Structure Solution and Refinement.—Solution was achieved in the space group *Cc* using the direct methods program SHELXS-86[6] and refined using SHELX-76.[7] All attempts to solve or refine in the centrosymmetric space group *C*2/*c* were unsuccessful. The non-hydrogen atoms of the central, rigid part of the molecule were refined anisotropically while those of the 2,8-phenyl rings were refined isotropically since anisotropic variation resulted in unacceptably high *U*11 and *U*22 temperature factors as highly distorted thermal ellipsoids were described to incorporate other high regions of electron density in the immediate vicinities of the rings. A distinct 'shadowing' of these rings is noted indicating a relatively high degree of motion. The possibility that this was indicative of the application of too rigid symmetry constraints, required by space group *Cc*, was considered and an attempt made to solve the structure in triclinic space group *P*1. High correlation was obtained upon refinement indicating that the higher symmetry (space group *Cc*) was justified. All aromatic and methylene hydrogen atoms were generated geometrically and each type tied to a common temperature factor, which refined to 0.092(5) and 0.071(8) Å^2 respectively.

Complex neutral-atom scattering factors were employed: from Cromer and Mann[8] for non-hydrogen atoms and from Stewart, Davidson and Simpson[9] for hydrogen atoms. Molecular parameters were obtained from Parst[10] and drawings generated with PLUTO.[11] All computations were carried out on a VAX/VMS (version 4.7) computer system located at the computer centre of the University of Cape Town. The final refinement of 202 parameters using 1996 reflections converged with a maximum parameter shift of <1% to give $R = 0.077$, $R_w = 0.073$ $[w = (\sigma^2 F)^{-1}]$. Fractional atomic co-ordinates for compound **1** are given in Table 1. Bond lengths and angles involving those atoms which were refined anisotropically are listed in Tables 2 and 3, respectively. All values generally agree with those in the literature and any discrepancies can be ascribed to vibrational effects. A complete list of bond lengths and bond angles, temperature factors and torsion angles have been deposited at the Cambridge Crystallographic Data Centre (CCDC).†

Results and Discussion

Inclusion Compound of **1**.—The Troeger base analogue **1** was shown to include dioxane as a guest solvent in the host:guest

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* Formerly prepared and inclusion characteristics investigated by Weber *et al.*[3] Crystal structure determined by Wilcox.[4]

† For details see 'Instructions for Authors (1991),' *J. Chem. Soc., Perkin Trans. 2*, 1991 in the January issue.

Table 1 Fractional atomic co-ordinates (× 10^4) with esds in parentheses for compound **1**

Atom	*x/a*	*y/b*	*z/c*
C(1)	2 408(0)	1 342(1)	9 239(0)
C(2)	2 648(18)	1 517(1)	7 604(16)
C(3)	4 569(18)	1 681(1)	8 438(16)
C(4)	4 957(19)	1 834(1)	6 988(18)
C(5)	3 292(19)	1 820(1)	4 543(18)
C(6)	1 395(18)	1 665(1)	3 727(17)
C(7)	999(19)	1 513(1)	5 201(17)
C(8)	−700(18)	1 160(1)	2 958(15)
C(9)	1 164(18)	981(1)	4 594(16)
C(10)	2 247(18)	819(1)	3 748(16)
C(11)	4 061(19)	666(1)	5 218(17)
C(12)	4 842(19)	680(1)	7 623(17)
C(13)	3 768(19)	833(1)	8 460(17)
C(14)	1 897(19)	988(1)	6 985(16)
C(111)	5 242(20)	493(1)	4 301(17)
C(112)	3 756(19)	358(1)	2 308(17)
C(113)	4 874(20)	197(2)	1 470(19)
C(114)	7 273(21)	177(2)	2 504(18)
C(115)	8 808(24)	305(2)	4 458(21)
C(116)	7 753(20)	468(2)	5 380(18)
C(121)	7 049(18)	2 007(1)	7 913(16)
C(122)	7 516(20)	2 141(1)	9 898(17)
C(123)	9 501(20)	2 303(2)	10 720(19)
C(124)	10 849(21)	2 317(2)	9 627(18)
C(125)	10 439(21)	2 194(2)	7 669(20)
C(126)	8 471(19)	2 030(2)	6 827(18)
C(201)	−1 459(18)	1 251(2)	6 107(19)
N(1)	788(17)	1 141(1)	7 947(15)
N(2)	−1 055(18)	1 358(1)	4 265(15)

Table 2 Selected bond lengths/Å with esds in parentheses for compound **1**

C(1)–C(2)	1.507(11)
C(1)–N(1)	1.487(8)
C(2)–C(3)	1.397(12)
C(2)–C(7)	1.404(12)
C(3)–C(4)	1.382(15)
C(4)–C(5)	1.429(13)
C(4)–C(121)	1.501(12)
C(5)–C(6)	1.354(12)
C(6)–C(7)	1.393(15)
C(7)–N(2)	1.420(12)
C(8)–C(9)	1.523(10)
C(8)–N(2)	1.483(13)
C(9)–C(10)	1.393(15)
C(9)–C(14)	1.400(15)
C(10)–C(11)	1.379(10)
C(11)–C(12)	1.404(15)
C(11)–C(111)	1.503(16)
C(12)–C(13)	1.352(16)
C(13)–C(14)	1.404(11)
C(14)–N(1)	1.420(15)
C(201)–N(1)	1.473(12)
C(201)–N(2)	1.466(18)

Table 3 Selected bond angles/° with esds in parentheses for compound **1**

C(2)–C(1)–N(1)	111.5(4)
C(1)–C(2)–C(7)	120.9(6)
C(1)–C(2)–C(3)	120.6(7)
C(3)–C(2)–C(7)	118.5(7)
C(2)–C(3)–C(4)	122.7(8)
C(3)–C(4)–C(121)	122.0(8)
C(3)–C(4)–C(5)	117.3(7)
C(5)–C(4)–C(121)	120.7(8)
C(4)–C(5)–C(6)	120.3(8)
C(5)–C(6)–C(7)	122.1(9)
C(2)–C(7)–C(6)	119.0(8)
C(6)–C(7)–N(2)	119.9(8)
C(2)–C(7)–N(2)	121.1(8)
C(9)–C(8)–N(2)	111.7(7)
C(8)–C(9)–C(14)	119.8(6)
C(8)–C(9)–C(10)	120.4(8)
C(10)–C(9)–C(14)	119.7(7)
C(9)–C(10)–C(11)	121.5(8)
C(10)–C(11)–C(111)	121.4(8)
C(10)–C(11)–C(12)	118.3(7)
C(12)–C(11)–C(111)	120.3(7)
C(11)–C(12)–C(13)	120.7(8)
C(12)–C(13)–C(14)	121.7(9)
C(9)–C(14)–C(13)	118.0(8)
C(13)–C(14)–N(1)	119.4(8)
C(9)–C(14)–N(1)	122.6(7)
N(1)–C(201)–N(2)	112.2(10)
C(14)–N(1)–C(201)	111.4(8)
C(1)–N(1)–C(201)	105.0(6)
C(1)–N(1)–C(14)	112.3(7)
C(8)–N(2)–C(201)	106.7(6)
C(7)–N(2)–C(201)	111.6(8)
C(7)–N(2)–C(8)	111.4(9)

Table 4 Inclusion compounds formed with Troeger base analogue **1a**

Guest[a]	Stoicheiometry[b] host : guest
Toluene	1:1.25
p-Xylene	1:0.5
m-Xylene	1:0.5
p-Clorotoluene	1:0.5
m-Clorotoluene	1:0.75
Diethyl ketone	1:0.75
Cyclohexanol	1:1
Dioxane	1:0.75
THF	1:0.75
Water	1:2.25

[a] Not included: methanol, ethanol, propanol, butanol, decan-1-ol, cyclohexane and *p*-cymene. [b] In all of the above inclusion compounds, water is present, as a second guest, in varying non-stoicheiometric amounts.

ratio of 1:0.33. This is the first report of inclusion by a non-quaternised Troeger base analogue.

Inclusion Compounds of **1a**.—The 2,8-diphenyl analogue of Troeger's base, as the *N*-methylated iodide salt, includes a variety of guest solvents (Table 4). It is interesting to note that water is present and is intimately associated with many of these inclusion compounds as a second guest. Consider *e.g.* the inclusion compound formed by host **1a** and dioxane: the NMR spectroscopically derived stoicheiometry (host:guest:H_2O) is 1:0.75:1.5 and microanalysis results are in good agreement (indicating the presence of the same quantity of water). The thermogravimetric analysis (Fig. 1) indicates that this inclusion compound releases the guests between 120 and 165 °C (corrected temperatures)* as two distinct overlapping peaks, indicated as peak A and B on the DTA curve. This corresponds to a 15.5% weight loss, in good agreement with the required stoicheiometry. A weight loss of 15.3% corresponds to release of 0.75 mol dioxane and 1.5 mol H_2O per mole of host.

Interestingly, water alone may also be included as a guest by host **1a**.

A distinct propensity for aromatic guest solvents is exhibited by host **1a** although an upper size limit appears to be a

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* Only the furnace temperature can be measured directly since the platinum crucible container is located directly above the thermocouple. The temperature lag is 30 °C, estimated by accurate measurement of the m.p.s of a series of standards.

Table 5 Stoicheiometry of inclusion of *p*-xylene by the *N*-alkyl-substituted Troeger base **2a**

R	Host : guest
Me	1:0.5[a]
Et	1:1
$(CH_2)_2CH_3$	1:1.25
$(CH_2)_4$	1:0.75

[a] Stoicheiometry in agreement with that obtained by Weber *et al.* (ref. 3).

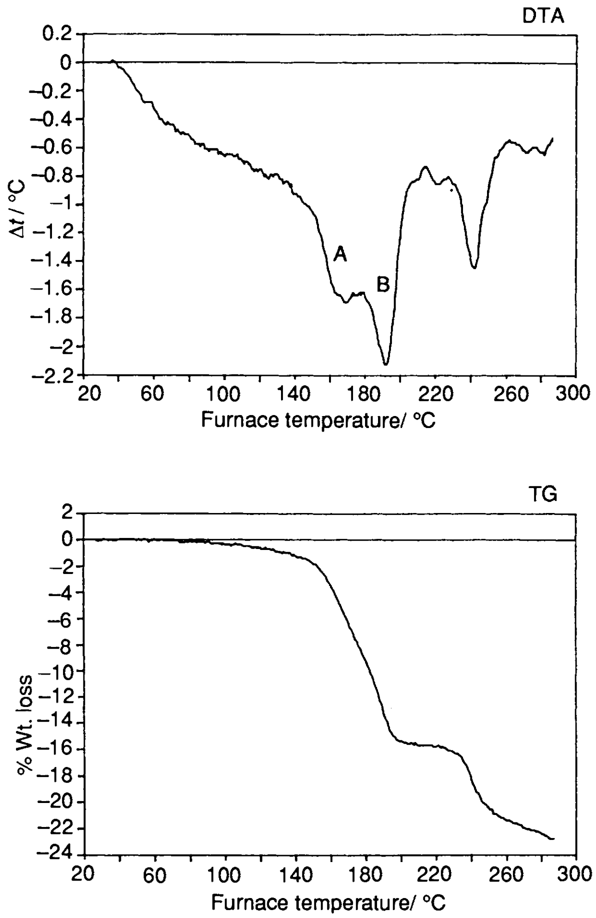

Fig. 1 DTA and TG thermograms for the inclusion compound formed with host **1a** and guests dioxane and water

constraint: *p*-cymene is not included. This inability to include *p*-cymene may be due to shape rather than size constraints as all other aromatic guests included have, or can adopt, a planar conformation while *p*-cymene cannot.

Comparison of the stoicheiometries of inclusion for common guest solvents by the 2,8-diphenyl analogue **1a** and analogous monomethylated iodide salt of Troeger's base **2a** (studied by Weber *et al.*[3]) indicates only a slight increase in the relative inclusion capacity of host **1a**, *i.e.* replacing the 2,8-methyl substituents **2a** with bulky phenyl groups **1a** has little effect on the inclusion capacity of the host.

Preliminary competition experiments to investigate the inclusion selectivity of host **1a** with respect to 1:1 mixtures of aromatic guest solvents, revealed the following: toluene is preferred over benzene and *p*-xylene is selectively included from mixtures with benzene or toluene. The 2,8-dimethyl analogue **2a** selectively includes benzene from mixtures with toluene or *p*-xylene.

Effect of Increasing Chain Length of the N-*Alkyl Substituent on the* 2,8-*Dimethyl Troeger Base Iodide Salt* **2a**.—The change in

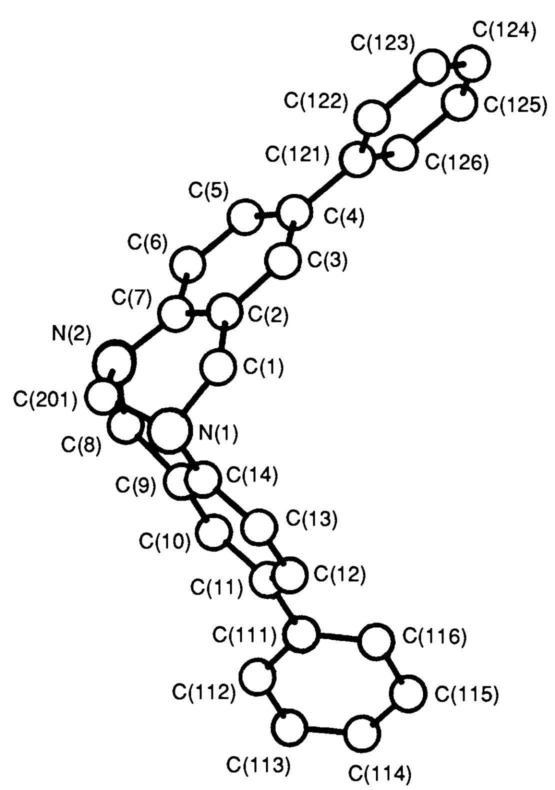

Fig. 2 A perspective view of the molecule (structure **1**) with atomic nomenclature

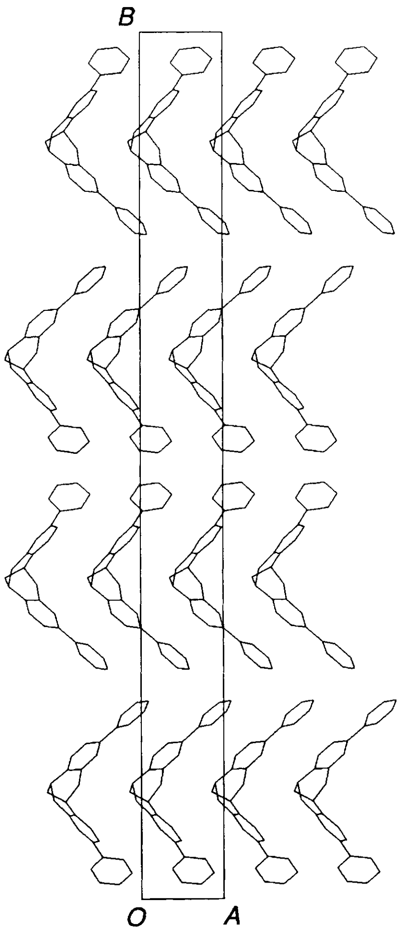

Fig. 3 A packing diagram of structure **1** viewed down [001]

stoicheiometry of inclusion with respect to *p*-xylene with increasing size of the *N*-alkyl substituent is shown in Table 5. Increasing the length of the chain by one methylene moiety has a larger effect on the inclusion capacity of the host than increasing the bulk of the 2,8-substituents. The bis-iodide Troeger base salt **2d** is shown to also include *p*-xylene.

X-Ray Crystallographic Study of Compound **1**.—A perspective view of the molecule with atomic nomenclature is shown in Fig. 2. The conformation of this rigid, chiral molecule may be described as V-shaped, 'hinged' at the N atoms. The neighbouring aromatic rings fused to the central diazocine ring are planar to within 0.02 Å and the intramolecular dihedral angle between the least-squares planes through these aromatic rings is 92.1(2)°. The terminal phenyl rings (planar to within 0.02 Å) do not lie coplanar with the respective central aromatic rings but are twisted by *ca.* 46°. The conformation is described by the torsion angles C(3)–C(4)–C(121)–C(126) = 134(1)° and C(10)–C(11)–C(111)–C(116) = 133(1)°.

A packing diagram of the structure, which comprises both enantiomers, is given in Fig. 3. Each enantiomeric molecule, being substantially 'folded', stacks as rows of spoons in the *a* direction. This arrangement precludes any cage or channel formation. Non-bonded distances are all >3.5 Å and thus unremarkable.

Acknowledgements

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