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PARTIALLY FLUORINATED HETEROCYCLIC COMPOUNDS. PART 19 [1]. THE FORMATION OF FISCHER INDOLE PRODUCTS FROM A SERIES OF HYDRAZONES DERIVED FROM PENTAFLUOROPHENYLHYDRAZINE AND 1,3,4,5,6,7,8-HEPTAFLUORO-2-NAPHTHYLHYDRAZINE THE SURPRISING LOSS OF o-FLUORINE

FRANK D. BENKE and GERALD M. BROOKE

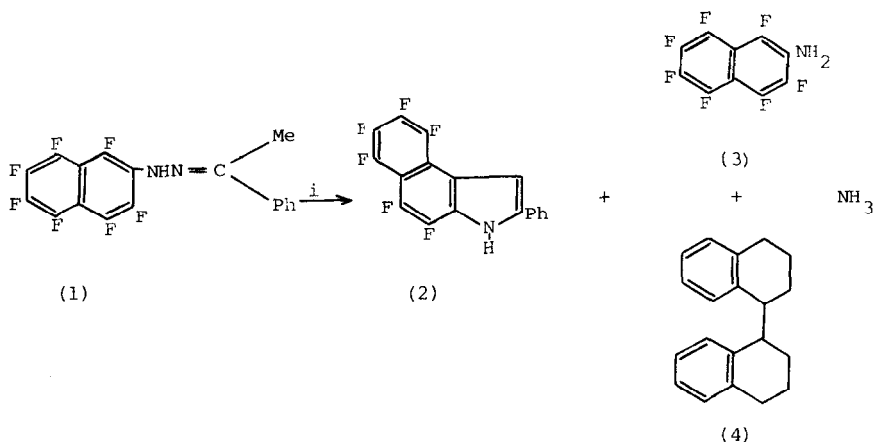
Chemistry Department, Science Laboratories, South Road, Durham, DH1 3LE (UK)

SUMMARY

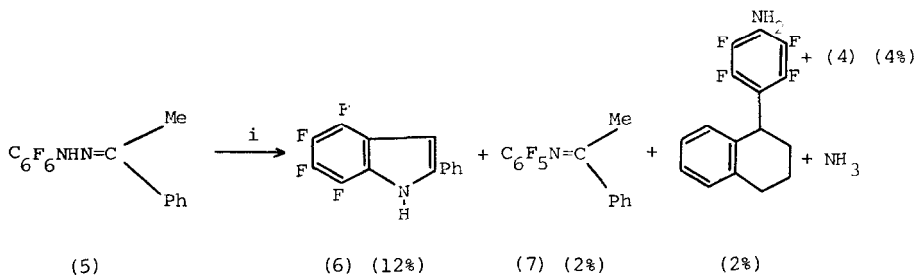
The pentafluorophenyl and 1,3,4,5,6,7,8-heptafluoro-2-naphthylhydrazones of a variety of aldehydes and ketones give 4,5,6,7-tetrafluoroindole and 4,5,6,7,8,9-hexafluorobenz[e]indole derivatives when heated under reflux in tetralin. These products are typical Fischer indole products - yet the substrates all have fluorine atoms in positions ortho to the nitrogen rather than hydrogen which is a pre-requisite in the conventional reaction. Acetaldehyde (10) and cyclohexanone (11) pentafluorophenylhydrazones give 4,5,6,7-tetrafluoroindole (8) (1.5%) and 5,6,7,8-tetrafluoro-1,2,3,4-tetrahydrocarbazole (9) (18%) respectively. 4-Methylacetophenone (12), propiophenone (13), 1,2-diphenylethanone (14), acetone (15) and phenylacetaldehyde-1,3,4,5,6,7,8-heptafluoro-2-naphthylhydrazones give 4,5,6,7,8,9-hexafluoro-2-(4-methylphenyl)- (16) (41%), -2-phenyl-3-methyl- (17) (34%), -2,3-diphenyl- (18) (42%), -2-methyl- (19) (20%), and -3-phenyl- (20) (24%) -benz[e]indoles respectively.

RESULTS AND DISCUSSION

In the previous paper in this series [1], acetophenone 1,3,4,5,6,7,8-heptafluoro-2-naphthylhydrazone (1) in refluxing tetralin was shown to give a complex mixture of products from which were isolated 4,5,6,7,8,9-hexafluoro-2-phenylbenz[e]indole (2) (37%), 1,3,4,5,6,7,8-heptafluoro-2-naphthylamine (3) (21%) and 1,1',2,2',3,3',4,4'-octahydro-1,1'-bisnaphthyl (4) (6%) (Scheme 1); ammonia is also evolved during the course of this



Scheme 1. Reagents: i, tetralin, reflux

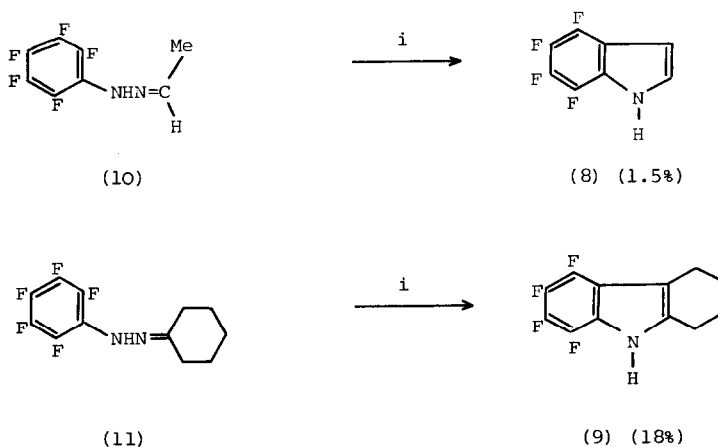


Scheme 2. Reagents: i, tetralin, reflux

reaction. When the experiment was repeated with acetophenone pentafluorophenylhydrazine (5), from among the four products isolated were 4,5,6,7-tetrafluoro-2-phenylindole (6) (12%) and pentafluoro-N-( $\alpha$ -methylbenzylidene)aniline (7) (2%) (Scheme 2). With the exception of the indole derivatives, the formation of all the other products in these two reactions could be rationalised in terms of an initial homolytic cleavage of the N-N bond in the parent hydrazones. Of particular significance, however, was the demonstration that the imine (7) was not the precursor to the indole (6) (via tautomerism to an enamine followed by nucleophilic displacement of ortho-fluorine); and the corresponding imine in the 2-naphthalene series was not the precursor to the benz[e]indole derivative (2). The formation of the Fischer indole products (2) and (6) from the hydrazones (1) and (5) respectively by the loss of ortho fluorine is

a surprising process which remains undefined at the present time. In this paper we report an investigation of reactions with the hydrazones of a variety of aldehydes and ketones derived from pentafluorophenyl hydrazine [2] and 1,3,4,5,6,7,8-heptafluoro-2-naphthylhydrazine [3] in refluxing tetralin. We have only looked for indole derivatives in the complex reaction products, though the amine (3) was also isolated in most cases from the naphthylhydrazones.

4,5,6,7-Tetrafluoroindole (8) and 5,6,7,8-tetrafluoro-1,2,3,4-tetrahydrocarbazole (9) were chosen as target molecules initially, as these had been prepared by other methods previously: (8) via cyclisation reactions involving nucleophilic displacement of ortho fluorine [4]; and (9) via a conventional Fischer indole reaction using cyclohexanone 2,3,4,5-tetrafluorophenylhydrazone [5]. Acetaldehyde pentafluorophenylhydrazone (10) on heating under reflux in dry tetralin for 24 h. gave a very complex product from which compound (8) was isolated in very low yield (1.5%). It is noteworthy that no arylhydrazones of acetaldehyde have been reported to undergo the Fischer indole reaction [6]. Cyclohexanone pentafluorophenylhydrazone (11) in refluxing tetralin gave compound (9) in 18% yield. This synthesis compares favourably in economic terms with the process utilising the conventional Fischer indole reaction which gave 50% of (9), but which required the much less accessible 2,3,4,5-tetrafluorophenylhydrazine for reaction with cyclohexanone [5]. The results of the two new cyclisation reactions are shown in Scheme 3.



Scheme 3. Reagents: i, tetralin, reflux

We next turned our attention to hydrazones derived from 1,3,4,5,6,7,8-heptafluorophenylhydrazine since in the one experiment before [1], the acetophenone derivative (1) had been shown to give the cyclised indole product more efficiently than the reaction with acetophenone pentafluorophenylhydrazone (5) - presumably because of the higher double bond character in the 1,2-position of the naphthalene ring. The new hydrazones which were prepared, and the products which were isolated from their reaction in refluxing tetralin, are shown in the Table. The mode of cyclisation (to C-1 rather than C-3) was clearly revealed by the presence of only one *peri* coupling  $J_{F-F}$  (61-71 Hz) in the  $^{19}F$  n.m.r. spectra.

Three conclusions can be drawn from the data in the Table:

- (a) the formation of the 4,5,6,7,8,9-hexafluorobenz[e]indole derivatives, and the 4,5,6,7-tetrafluoroindole derivatives described earlier in this paper, do not demand a hydrogen ortho to the nitrogen in the polyfluoroarylhydrazone precursors as in the conventional Fischer indole reaction. Surprising as it is, fluorine atoms ortho to the nitrogen do not prevent the formation of Fischer indole products;
  - (b) the cyclisation product is formed more efficiently from the hydrazones derived from aryl ketones than from acetone or from phenylacetaldehyde;
- and (c) the 4,5,6,7,8,9-hexafluorobenz[e]indole derivatives described in this paper are not available by conventional Fischer indole syntheses since at present there are no known routes to 3,4,5,6,7,8-hexafluoro-2-naphthylhydrazine from which hydrazones having a hydrogen ortho to the nitrogen could be prepared.

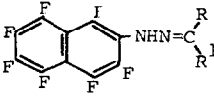
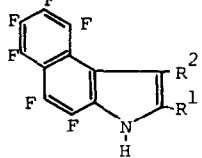
## EXPERIMENTAL

$^1H$  (60 MHz) and  $^{19}F$  n.m.r. (56.4 MHz) spectra were obtained with a Varian EM 360L spectrometer. Chemical shifts are upfield from internal  $CFCl_3$  ( $\delta_F$ ), or downfield from internal T.M.S. ( $\delta_H$ ).

### Flash Chromatography [7]

Flash chromatography separations of the products from the indole derivative forming reactions were all carried out on Merck silica gel 60 (230-400 mesh) (160 mm x 50 mm) using  $CCl_4/CHCl_3$  1:1 V/V except where stated.

TABLE

2-naphthyl- <sup>a</sup> hydrazone	Yield (%)	Products from reaction in refluxing tetralin	
		Benz[e]indole	Yield (%)    Compound(3) (%)
			
R = CH <sub>3</sub> R <sup>1</sup> = C <sub>6</sub> H <sub>5</sub> (1)	61 [1]	R <sup>2</sup> = H R <sup>1</sup> = C <sub>6</sub> H <sub>5</sub> (2)	37 [1]    21 [1]
R = CH <sub>3</sub> R <sup>1</sup> = 4-MeC <sub>6</sub> H <sub>4</sub> (12)	81	R <sup>2</sup> = H R <sup>1</sup> = 4-MeC <sub>6</sub> H <sub>4</sub> (16)	41    12
R = CH <sub>2</sub> CH <sub>3</sub> R <sup>1</sup> = C <sub>6</sub> H <sub>5</sub> (13)	84	R <sup>2</sup> = CH <sub>3</sub> R <sup>1</sup> = C <sub>6</sub> H <sub>5</sub> (17)	34    19
R = CH <sub>2</sub> C <sub>6</sub> H <sub>5</sub> R <sup>1</sup> = C <sub>6</sub> H <sub>5</sub> (14)	48	R <sup>2</sup> = C <sub>6</sub> H <sub>5</sub> R <sup>1</sup> = C <sub>6</sub> H <sub>5</sub> (18)	42    0
R = CH <sub>3</sub> R <sup>1</sup> = CH <sub>3</sub> (15)	76	R <sup>2</sup> = H R <sup>1</sup> = CH <sub>3</sub> (19)	20    17
R = CH <sub>2</sub> C <sub>6</sub> H <sub>5</sub> R <sup>1</sup> = H	<sup>b</sup>	R <sup>2</sup> = C <sub>6</sub> H <sub>5</sub> R <sup>1</sup> = H (20)	24 <sup>c</sup> 8

<sup>a</sup> From 1,3,4,5,6,7,8-heptafluoro-2-naphthylhydrazone and the corresponding carbonyl compound.

<sup>b</sup> Not isolated.

<sup>c</sup> Based on the hydrazone derivative used.

Acetaldehyde Pentafluorophenylhydrazone (10)

Pentafluorophenylhydrazine [2] (10.1 g) in ethanol (50 ml) was treated with acetaldehyde (5.0 g) and the mixture was heated reflux for 5 min. The volatile components of the mixture were removed by distillation in vacuo, and the solid residue was crystallised from light petroleum (bp 60-80°) to give acetaldehyde pentafluorophenylhydrazone (10) nc (7.82 g, 68%), m.p. 52-53° (Found: C, 42.58; H, 2.03; N, 12.54%.  $C_8H_5F_5N_2$  requires C, 42.87; H, 2.25; N, 12.50%);  $\nu_{\max}$  3230  $cm^{-1}$  (N-H).

Cyclohexanone Pentafluorophenylhydrazone (11)

Pentafluorophenylhydrazine [2], and cyclohexanone were heated under reflux in ethanol for 2 h. The crude product was crystallised from ethanol/water to give cyclohexanone pentafluorophenylhydrazone (11) nc (75%), m.p. 68-69° (Found: C, 52.00; H, 4.11; N, 9.70%;  $M^+$ , 278.  $C_{12}H_{11}F_5N_2$  requires C, 51.80; H, 3.99; N, 10.07%;  $M$ , 278)  $\nu_{\max}$  3230  $cm^{-1}$  (N-H)

4-Methylacetophenone 1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazone (12)

1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazine [3] and 4-methylacetophenone were heated under reflux in ethanol for 2 h. The crude product was crystallised from toluene-light petroleum (bp 100-120°) to give 4-methylacetophenone 1,3,4,5,6,7,8-heptafluoro-2-naphthylhydrazone (12) nc (8 m.p. 192-192.5°. (Found: C, 56.87; H, 2.62; N, 7.08%.  $C_{19}H_{11}F_7N_2$  requires C, 57.00; H, 2.77; N, 7.00%);  $\nu_{\max}$  3370  $cm^{-1}$  (N-H).

Propiophenone 1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazone (13)

1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazine [3] and propiophenone were heated under reflux in ethanol for 2 h. The crude product was crystallised from light petroleum (bp 100-120°) to give propiophenone 1,3,4,5,6,7,8-heptafluoro-2-naphthylhydrazone (13) nc (84%), m.p. 138-139° (Found: C, 57.03; H, 2.49; N, 6.65%.  $C_{19}H_{11}F_7N_2$  requires C, 57.00; H, 2.77; N, 7.00%);  $\nu_{\max}$  3370  $cm^{-1}$  (N-H).

1,2-Diphenylethanone 1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazone (14)

1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazine [3] and 1,2-diphenylethanone (deoxybenzoin) were heated under reflux in ethanol for 2 h. The

crude product was crystallised from toluene-light petroleum (bp 100-120°) to give 1,2-diphenylethanone 1,3,4,5,6,7,8-heptafluoro-2-naphthylhydrazone (14) nc (48%), m.p. 173.5-175° (Found: C, 62.64 H, 2.45; N, 5.73.  $C_{24}H_{13}F_7N_2$  requires C, 62.35; H, 2.82; N, 6.06%);  $\nu_{max}$  3380  $cm^{-1}$  (N-H).

Acetone 1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazone (15)

1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazine [3] and acetone were heated under reflux in ethanol for 1 h. The crude product was crystallised from light petroleum (bp 60-80°) to give acetone 1,3,4,5,6,7,8-heptafluoro-2-naphthylhydrazone (15) nc (76%) m.p. 99.5-100° (Found: C, 48.10; H, 1.78; N, 8.24%.  $C_{13}H_7F_7N_2$  requires C, 48.16; H, 2.18; N, 8.64%);  $\nu_{max}$  3370  $cm^{-1}$  (N-H).

Reaction of Acetaldehyde Pentafluorophenylhydrazone (10) in Tetralin

The phenylhydrazone (10) (5.42 g) was heated under reflux in dry tetralin (50 ml) for 24 h. The solvent was distilled under reduced pressure (0.05 mm) using an external water bath at ca. 50°. The black residue was distilled in vacuo and the fraction collected at  $\leq 140^\circ$  at 0.05 mm Hg was subjected to flash chromatography (see above). The sixth of a series of 125 ml fractions was shown to contain 4,5,6,7-tetrafluoroindole by analytical t.l.c. (silica) using the same solvent system and using the authentic material as standard. Further purification of this fraction by preparative t.l.c. (silica) gave 4,5,6,7-tetrafluoroindole (8) [4c] (0.067 g. 1.5%), identified by i.r.

Reaction of Cyclohexanone Pentafluorophenylhydrazone (11) in Tetralin

The phenylhydrazone (11) (2.51 g) was heated under reflux in dry tetralin (100 ml) for 24 h. during which time ammonia was evolved. The solvent was distilled under reduced pressure (0.001 mm Hg) using an external water bath at ca. 60° and the complex mixture of products in the residue was partially separated by flash chromatography (see above). The mixture of mid-range moving components (0.86 g) was mainly indole material (0.386 g, 18%) and was separated by fractional sublimation at 50°/0.001 mm Hg. Recrystallisation of the sublimate from light petroleum (bp 60-80°) gave 5,6,7,8-tetrafluoro-1,2,3,4-tetrahydrocarbazole (?), m.p.

136.5-137° (lit., [5] 140-143°) (Found: C, 59.65; H, 3.30; N, 6.15%; M<sup>+</sup>, 243. C<sub>12</sub>H<sub>9</sub>F<sub>4</sub>N requires C, 59.26; H, 3.73; N, 5.76%; M, 243); δ<sub>F</sub> (CDCl<sub>3</sub>) 155.3 (t), 163.7 (t), 168.7 (t) and 171.9 ppm (t) with intensities in the ratio 1:1:1:1; δ<sub>H</sub>(CDCl<sub>3</sub>) 7.8 (N-H), 2.75 and 1.85 (two CH<sub>2</sub>); ν<sub>max</sub> 3475 cm<sup>-1</sup> (N-H).

Reaction of 4-Methylacetophenone 1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazone(12) in Tetralin

The naphthylhydrazone(12) (1.00 g) was heated under reflux in dry tetralin (50 ml) for 24 h., during which time ammonia was evolved. The solvent was removed as before and the complex product was partially separated by flash chromatography (see above). The mixture of mid-range moving components was shown by <sup>19</sup>F n.m.r. to contain materials with six fluorine atoms and after one crystallisation from toluene-light petroleum (bp 100-120°), fractional sublimation of the two component mixture (by t.l.c.) at 40°/0.05 mm Hg gave 1,3,4,5,6,7,8-heptafluoro-2-naphthylamine(3) (0.079 g, 12%), while sublimation at 140-150°/0.005 mm Hg gave 4,5,6,7,8,9-hexafluoro-2-(4-methylphenyl)benz[e]indole(16) nc (0.378 g, 41%) m.p. 216.5-217.5° [from light petroleum (bp 100-120°)] (Found: C, 62.36; H, 2.54; N, 3.94%; M<sup>+</sup>, 365. C<sub>19</sub>H<sub>9</sub>F<sub>6</sub>N requires C, 62.47; H, 2.48; N, 3.83%, M, 365); δ<sub>F</sub>[(CD<sub>3</sub>)<sub>2</sub>CO] 145.4 (t), 147.9 (d of m, peri J<sub>F-F</sub> 61 Hz), 155.3 (m), 157.5 (d of d peri J<sub>F-F</sub> 61 Hz), 160.9 (t of m) and 162.9 ppm (t) with intensities in the ratio 1:1:1:1:1:1; δ<sub>H</sub>(C<sub>6</sub>D<sub>6</sub>) 2.15 (CH<sub>3</sub>), 6.99 (C<sub>6</sub>H<sub>4</sub>), 7.32 (3H) and 8.04 (N-H) with intensities in the ratio ca. 3:4:1:1; ν<sub>max</sub> 3455 cm<sup>-1</sup>.

Reaction of Propiophenone 1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazone(13) in Tetralin

The naphthylhydrazone(13) (2.00 g) was heated under reflux with dry tetralin (80 ml) for 24 h. during which time ammonia was evolved. The solvent was removed as before and the complex product was partially separated by flash chromatography (see above). From the mixture of mid-range moving components (1.237 g) two components were separated by fractional sublimation: at 40-45/0.001 mm Hg, 1,3,4,5,6,7,8-heptafluoro-2-naphthylamine(3) (0.258 g, 19%) was obtained, while sublimation at 120-140°/0.001 mm Hg gave 4,5,6,7,8,9-hexafluoro-2-phenyl-3-methylbenz[e]indole(17) nc (0.628 g, 34%) m.p. 169-169.5° [from light petroleum



(bp. 100-120°). (Found: C, 62.63; H, 2.21, N, 4.01%,  $M^+$  365.  
 $C_{19}H_9F_6N$  requires C, 62.47; H, 2.48; N, 3.83%;  $M$ , 365);  $\delta_F$  [( $CD_3$ )<sub>2</sub>CO] 137.3 (m), 148.3 (d of m, peri  $J_{F-F}$  71 Hz), 156.4 (d of m), 157.8 (d of d, peri  $J_{F-F}$  71 Hz), 161.1 (t) and 163.4 ppm (t) with intensities in the ratio 1:1:1:1:1:1;  $\delta_H$  [( $CD_3$ )<sub>2</sub>CO] 2.45 ( $CH_3$ , d, J, 14 Hz), 7.55 ( $C_6H_5$ ) and 11.50 (N-H);  $\nu_{max}$  3395  $cm^{-1}$  (N-H).

Reaction of 1,2-Diphenylethanone 1,3,4,5,6,7,8-heptafluoro-2-naphthylhydrazone (14) in Tetralin

The naphthylhydrazone (14) (2.212 g) was heated under reflux with dry tetralin (50 ml) for 24 h. during which time ammonia was evolved. The solvent was removed as before and the complex product was partially separated by flash chromatography (see above) using  $CHCl_3/CHCl_4$  (70:30 V/V) as eluant. No 1,3,4,5,6,7,8-heptafluoro-2-naphthylamine was detected in the enriched mid-range moving component and sublimation at 160-170°/0.001 mm Hg gave 4,5,6,7,8,9-hexafluoro-2,3-diphenylbenz[e]indole (18) nc (0.868 g, 42%), m.p. 223.5-224° [from toluene/light petroleum (bp 100-120°)], (Found: C, 67.58; H, 3.00; N, 2.95%;  $M^+$  427.  $C_{24}H_{11}F_6N$  requires C, 67.45; H, 2.59; n, 3.28%;  $M$ , 427);  $\delta_F$  [( $CD_3$ )<sub>2</sub>CO] 131.9 (t), 148.0 (d of m, peri  $J_{F-F}$  68 Hz), 155.6 (m), 156.7 (d of d, peri  $J_{F-F}$  68 Hz), 160.6 (t);  $\nu$  162.6 ppm (t), with intensities in the ratio 1:1:1:1:1:1;  $\delta_H$  [( $CD_3$ )<sub>2</sub>CO] 7.33 (2 x  $C_6H_5$ ) and 11.8 (N-H);  $\nu_{max}$  3465  $cm^{-1}$  (N-H).

Reaction of Acetone 1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazone (15) in Tetralin

The naphthylhydrazone (15) (0.50 g) was heated under reflux with dry tetralin (10 ml) for 4 h., during which time ammonia was evolved. The solvent was removed as before and the complex product was partially separated by flash chromatography (see above). From the mixture of mid-range moving components two components were separated by fractional sublimation: at  $\leq 50^\circ/0.001$  mm Hg, 1,3,4,5,6,7,8-heptafluoro-2-naphthylamine (3) (0.07 g, 17%) was obtained, while sublimation at  $70^\circ/0.001$  mm Hg gave 4,5,6,7,8,9-hexafluoro-2-methyl-benz[e]indole (19) nc (0.09 g, 20%) m.p. 193-193.5° [from light petroleum (bp 80-100°)]. (Found: C, 53.68; H, 1.32; N, 4.74%;  $M^+$ , 289.  $C_{13}H_5F_6N$  requires C, 53.99; H, 1.74; N, 4.84%;  $M$ , 289);  $\delta_F$  [( $CD_3$ )<sub>2</sub>CO] 145.9 (t), 148.3 (d of m, peri  $J_{F-F}$

62 Hz), 156.1 (d), 159.4 (d of d, peri  $J_{F-F}$  62 Hz), 161.5 (t of m) and 163.6 ppm (t) with intensities in the ratio 1:1:1:1:1;  $\delta_H(\text{CDCl}_3)$  2.55 (s,  $\text{CH}_3$ ), 6.86 (s, 3-H) and 8.6 (N-H) in the ratio 3:1:1,  $\nu_{\text{max}}$  3465  $\text{cm}^{-1}$ .

Reaction of 1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazine with Phenyl-acetaldehyde in Tetralin

1,3,4,5,6,7,8-Heptafluoro-2-naphthylhydrazine [3] (1.107 g) phenyl-acetaldehyde (0.483 g) and dry tetralin (40 ml) were heated together under reflux. The water produced in the initial condensation reaction was azeotropically distilled off (ca. 10 ml distillate collected) and the heating under reflux was continued for a total of 24 h. during which time ammonia was evolved. The solvent was removed as before and the complex product was partially separated by flash chromatography (see above). From the mixture of mid-range moving components two compounds were separated by fractional sublimation: at 40-50 $^{\circ}$ /0.001 mm Hg. 1,3,4,5,6,7,8-heptafluoro-2-naphthylamine(3) (0.084 g, 8%), while sublimation at 160-165 $^{\circ}$ /0.001 mm Hg gave 4,5,6,7,8,9-hexafluoro-3-phenylbenz[e]indole(20) nc (0.322 g, 24%) m.p. 253-253.5 $^{\circ}$  [from toluene/light petroleum (bp 100-120 $^{\circ}$ )]. (Found: C, 61.59; H, 1.99; N, 3.83%;  $M^+$ , 351.  $\text{C}_{18}\text{H}_7\text{F}_6\text{N}$  requires C, 61.54; H, 2.01, N, 3.99;  $M$ , 351);  $\delta_F[(\text{CD}_3)_2\text{CO}]$  132.0 (t), 148.3 (d of t, peri  $J_{F-F}$  68 Hz), 156.5 (m), 157.6 (d of d, peri  $J_{F-F}$  68 Hz), 160.9 (t of m) and 163.0 ppm (t) with intensities in the ratio 1:1:1:1:1:1;  $\delta_H[(\text{CD}_3)_2\text{CO}]$  7.3 ( $\text{C}_6\text{H}_5$ ), 7.4 (2-H) and 11.7 (N-H);  $\nu_{\text{max}}$  3400  $\text{cm}^{-1}$ .

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