

Notes

Synthesis of functionalized diaryl alkanes from azines

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Substituted diaryl alkanes are synthesized from benzalazines and acetophenone/propionophenone azines via Friedel-Craft's reaction with substituted mono- and poly-nuclear aromatic hydrocarbons. Diaryl methanes/ethanes and propanes are obtained by reaction with benzalazine, *N,N'*-bis(1-phenyl) azine and *N,N'*-bis(1-propyl) azine, respectively.

Keywords: Azines, *N,N'*-bis(1-phenyl)azine, *N,N'*-bis(1-propyl)azine, diarylethane, diarylpropane

Diarylmethanes are important structural intermediates in several biologically active compounds¹ and drugs² such as trimethoprim, papaverin and piritrexim. The usual method for the synthesis of this class of compounds include (i) condensation between one mole of formaldehyde and two moles of benzene in presence of concentrated sulfuric acid³; (ii) Wolf-Kishner reduction of aromatic ketones⁴ or by Clemmenson reduction⁵; (iii) Catalytic condensation of Grignard reagent with hydrocarbons⁶ besides other methods where diaryl methanes are obtained as side products. A recent method for the preparation of functionalized diarylmethanes involved the Suzuki-Miyaura-coupling of benzylic phosphates with arylboronic acids⁷. Other methods for the synthesis of diaryl methanes and their derivatives are also reported in the literature⁸. A new method for the preparation of diarylalkanes starting from azines which can be easily prepared in the laboratory by reaction of carbonyl compounds with hydrazines or hydrazones⁹ (**Scheme I**) is reported.

Azines were prepared by following standard methods⁸⁻¹⁰. For this investigation the azines prepared from benzaldehyde, acetophenone and propionophenone (**Table I**) are chosen.

Results and Discussion

As part of ongoing research and in continuation of earlier work¹⁰ here in the synthesis of substituted

diaryl ethanes **4** and diaryl propanes **5** starting from the azines obtained by condensation of aldehydes and ketones with hydrazine by Friedel-Craft reaction with aromatic hydrocarbon is reported. The products obtained were all solids and the yields vary from moderate to good, **Scheme II**.

The reactions were carried out using various Lewis acids (AlCl_3 , $\text{BF}_3 \cdot \text{OEt}_2$) in aprotic solvents (CH_2Cl_2 , CS_2 and chlorobenzene). However appreciable yields of the products were obtained only when the reactions were carried using AlCl_3 as the acid **Table II**. A typical ^1H NMR spectrum of the product **5** showed the presence of a triplet at δ 4.11 (-CH-), a quintet (broad) at 1.63 (-CH₂-) and a triplet at 1.05 (-CH₃-) besides the aromatic peaks which appear as multiplets between 7.02-8.12.

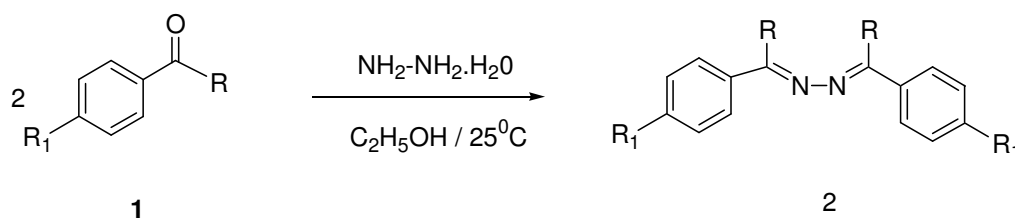
Experimental Section

^1H and ^{13}C NMR spectra were recorded on a Bruker AMX 400 instrument using CDCl_3 as the solvent. Chemical shifts are reported in ppm using tetramethylsilane as internal standard. *J* values are given in Hz. IR spectra were recorded on a Perkin-Elmer BX spectrophotometer. ESI mass spectra were measured on an ion trap analyzer Esquire 3000 (Bruker Daltonics). CHN analyses were recorded on a VarioEL analyser. Column chromatography was performed using silica gel (60-120 mesh, Merck).

General procedure for the preparation of substituted diaryl alkanes. Synthesis diaryl methane **3**

Step of 1: A mixture of benzaldehyde (15 mL, 0.10 mole) and hydrazine hydrate (5 mL, 0.1 mole) was stirred at RT in ethanol (15 mL) for 8-9 hr. After the reaction was completed (monitored by TLC), ethanol was removed from the mixture under reduced pressure. The product on recrystallization from diethylether gave yellow crystals of *N,N'*-bis(1-methyl)azine **2a**.

Step 2: To a 250 mL round bottom flask, a mixture of 1.33 g (0.05 mole) of *N,N'*-bis(1-methyl)azine **2a** and benzene or toluene (1.57 g, 0.02 mole) was added. Anhydrous aluminium chloride (1.32 g, 0.01 mole) was then added and the mixture was refluxed for 8-12 hr. When the reaction was completed (monitored by TLC), enough water is



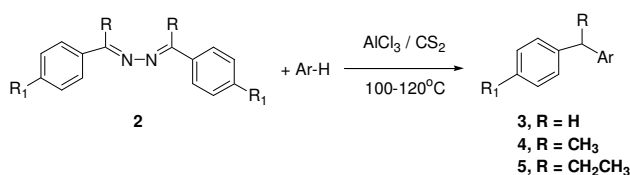
R = H, -CH₃, -CH₂CH₃

R₁ = H, -Cl, -Br, -NO₂, -OH, -OMe

Scheme I

Table I — Preparation of substituted *N,N'*-bis(1-alkyl)azine

Entry	Products	R	Time/hr	Intermediate
1	2a	H	14	<i>N,N'</i> -bis(1-methyl)azine
2	2b	-CH ₃	17	<i>N,N'</i> -bis(1-ethyl)azine
3	2c	-CH ₂ CH ₃	18	<i>N,N'</i> -bis(1-propyl)azine



Scheme II

added in multiple (50 mL) and extracted with diethyl ether. The organic layer was dried with anhydrous Na₂SO₄ and filtered. The solvent was evaporated off and the crude product purified by column chromatography using silica gel as the solid phase and hexane as eluent to give **3a**.

Other compounds **3b-g** were synthesized in similar manner using compound **2a** and various selected aromatic hydrocarbons. Characterization data are presented as shown below.

Diphenyl methane 3a: Yellow crystals, m.p. 90-91°C; IR (KBr): 2936, 2863, 3067, 3032 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.02-7.42 (m, 10H, Ar-H), 4.03 (s, 2H); ¹³C NMR: δ 40.1, 126.1, 127.9, 128.4, 128.7, 129.6, 143.2; Anal. Calcd. for C₁₃H₁₂: C, 87.6; H, 7.30. Found: C, 87.68; H, 7.36%.

4-Tolyl-1-phenylmethane 3b: Yellow crystals, m.p. 97-98°C; IR (KBr): 2930, 2867, 3042 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.02-7.43 (m, 9H, Ar-H), 3.87 (s, 2H), 2.35 (s, 3H); ¹³C NMR: δ

20.01, 41.4, 126.23, 127.3, 128.02, 129.8, 135.22, 140.3, 143.7; Anal. Calcd. For C₁₄H₁₄: C, 92.30; H, 7.69. Found: C, 92.36; H, 7.42 %.

Phenyl-1-(2,5-xylyl) methane 3c: Yellow crystals, m.p. 119-20°C; IR (KBr): 2902, 3044 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 6.42-7.37 (m, 8H, Ar-H); 3.83 (s, 2H), 2.32 (s, 3H); ¹³C NMR: δ 15.20, 21.7, 35.42, 126, 128.6, 129.6, 135.2, 143.3; Anal. Calcd. for C₁₅H₁₆: C, 91.87; H, 8.16. Found: C, 91.36; H, 8.42%.



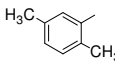
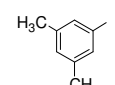
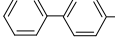
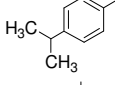



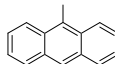
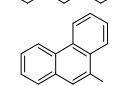
4-Chlorophenyl-1-(3,5-xylyl)-methane 3d: Yellow crystals, m.p. 109-10°C; IR (KBr): 2902, 3044 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 6.71-7.76 (m, 7H, Ar-H), 4.02 (s, 2H), 2.37 (s, 3H); ¹³C NMR: δ 21.23, 42.23, 126.23; 127.45, 128.22, 129.1, 131.3, 138, 142.5; Anal. Calcd. for C₁₅H₁₅Cl: C, 78.08; H, 6.55. Found: C, 78.12; H, 6.42%.

4-[1-(4-Chlorophenyl)-methyl] biphenyl 3e: Pale Yellow crystals, m.p. 92-93°C; IR (KBr): 2875, 3038 cm⁻¹, ¹H NMR (400 MHz, CDCl₃): δ 7.01-7.47 (m, 13H, Ar-H), 3.82 (s, H); ¹³C NMR: δ 41.3, 126.23, 127.45, 128.22, 129.1, 131.02, 134.21, 136.15, 141.5; Anal. Calcd. for C₁₉H₁₅Cl: C, 81.86; H, 5.42. Found: C, 81.36; H, 5.40 %.

4-[1-(4-Bromophenyl)-methyl]-isopropyl benzene 3f: Yellow crystals, m.p. 103-04°C; IR (KBr): 2925, 2860 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 6.82-7.37 (m, 8H, Ar-H), 4.02 (s, 2H), 3.73 (m, 1H), 1.20 (dd, *J* = 7 Hz, 3H); ¹³C NMR: δ 24.23, 41.23, 120.6, 126.23, 128.22, 130.1, 132.34, 140, 142.45, 146.5; Anal. Calcd. for C₁₆H₁₇Br: C, 66.78 ; H, 5.64. Found: C, 66.37; H, 5.56%.

2-(4-Bromobenzyl)-naphthalene 3g: Yellow crystals, m.p. 97-98°C; IR (KBr): 3040, 2923,

Table II — Preparation of substituted diaryl methanes **3a-k**

Entry	Product	R ¹	Ar	Time/hr	Yield (%) ^a
1	3a	H		20	62
2	3b	H		22	56
3	3c	H		24	72
4	3d	Cl		22	76
5	3e	Cl		20	62
6	3f	Br		20	63
7	3g	Br		24	58
8	3h	OH		22	54
9	3i	OMe		24	67
10	3j	NO ₂		22	63
11	3k	NO ₂		22	70

^a Isolated yields are determined after work-up and chromatography purification

2884 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 6.10-7.57 (m, 11H, Ar-H), 4.28 (s, 2H); ¹³C NMR: δ 38.23, 120.5, 125.2, 126.23, 128.22, 130.1, 132.5, 134, 142.4; Anal. Calcd. for C₁₇H₁₃Br: C, 68.70; H, 4.42. Found: C, 68.36; H, 4.44%.

4-(4-Methylbenzyl) phenol 3h: Yellow crystals, m.p. 94-95°C; IR (KBr): 2930, 2865, 3047 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 6.15-7.14 (m, 8H, Ar-H), 5.34 (s, Ar-OH), 3.86 (s, 2H), 2.31 (s, 3H); ¹³C NMR: δ 20.3, 40.4, 117.8, 128.02, 129.6, 135.24, 140.2, 154.82; Anal. Calcd. for C₁₄H₁₄O: C, 84.81; H, 7.12. Found: C, 84.36; H, 7.14%.


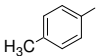
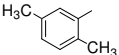
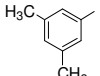
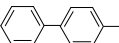
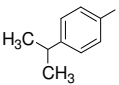

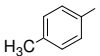

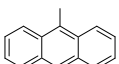

1-(4-Methoxy-benzyl)-naphthalene 3i: Yellow crystals, m.p. 124-25°C; IR (KBr): 2930, 2837, 3040 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.10-8.17 (m, 11H, Ar-H), 4.28 (s, 2H), 3.21 (s, 3H); ¹³C NMR: δ 38.23, 56.5, 115.2, 126.23, 128.22, 129.1, 134.5, 135, 159.05; Anal. Calcd. for C₁₈H₁₆O: C, 87.07; H, 6.49. Found: C, 87.36; H, 6.44%.

9-(4-Nitrobenzyl)-anthracene 3j: Colourless crystals, m.p. 93-94°C; IR (KBr): 2978, 3041, 2965, 2938, 2918 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.02-8.14 (m, 13H, Ar-H), 4.23 (s, 2H); ¹³C NMR: δ 37.25, 124.9, 125.32, 126.21, 127.40, 128.26, 129.21, 131.5, 132.0, 145.25, 149.12; Anal. Calcd. for C₂₁H₁₅NO₂: C, 80.49; H, 4.82, N 4.47. Found: C, 80.36; H, 4.74; N 4.42 %.

9-(4-Nitrobenzyl)-phenanthrene 3k: Yellow crystals, m.p. 114-15°C; IR (KBr): 2978, 3041, 2965, 2938, 2918, 2875 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.11-8.15 (m, 13H, Ar-H), 4.14 (s, 2H); ¹³C NMR: δ 40.03, 122, 125.31, 126.22, 128.28, 129.31, 130, 141.2, 145.27, 149.14; Anal. Calcd. for C₂₁H₁₅NO₂: C, 80.49; H, 4.82; N, 4.47. Found: C, 80.06; H, 4.56; N, 4.49%.

Synthesis of diaryl ethane 4: Other compounds **4a-g** were synthesized in similar manner using acetophenone (step 1) to gave **2b** (Table II) and then

Table III — Preparation of substituted diaryl ethanes **4a-k**

Entry	Product	R ¹	Ar	Time/hr	Yield (%) ^a
1	4a	H		18	73
2	4b	H		18	87
3	4c	H		16	74
4	4d	Cl		20	78
5	4e	Cl		20	76
6	4f	Br		20	66
7	4g	Br		24	77
8	4h	OH		25	58
9	4i	OMe		25	56
10	4j	NO ₂		20	86
11	4k	NO ₂		20	82

^a Isolated yields are determined after work-up and chromatography purification

(step 2) with various selected aromatic hydrocarbons. Work-up of the reaction-mixture followed by column chromatography yielded yellow to dark brown crystalline solids **4a-k** (Table III). Characterization data are presented as shown below.

Diphenyl ethane 4a: Pale yellow crystals m.p. 112-13°C; IR (KBr): 2872, 2892, 3070, 3030 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 1.55 (d, 3H), 4.23 (q, 1H), 7.02-7.24 (m, 10H, Ar-H); ¹³C NMR: δ 24.3, 38.40, 127.2, 128.8, 129.6, 143.2; Anal. Calcd. for C₁₄H₁₄: C, 92.3; H, 7.69. Found: C, 92.32, H, 7.56%.



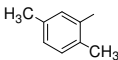

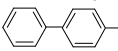
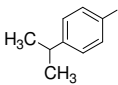



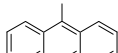

4-Tolyl-2-phenylethane 4b: Pale yellow crystals m.p. 119-20°C; IR (KBr): 2877, 2895, 3068, 3033 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 1.62 (d, 3H), 2.38 (s, 3H), 4.21 (q, 1H), 7.02-7.44 (m, 9H, Ar-H); ¹³C NMR: δ 20.12, 24.13, 38.34, 127.2, 128.8, 129.6, 135.12, 140.06, 143.2; Anal. Calcd. for C₁₅H₁₆: C, 91.8; H, 8.16. Found: C, 91.73; H, 8.17 %.

Phenyl-1-methyl-(2,5-xylyl) methane 4c: Yellow crystals m.p. 102-03°C; IR (KBr): 2877, 2885, 3063, 3037 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 1.48 (d, 3H), 2.37 (s, 3H), 4.24 (q, 1H), 6.18-7.47 (m, 8H, Ar-H); ¹³C NMR: δ 21.5, 24.42, 40.23, 126.12, 127.32, 128.4, 129.6, 143.12; Anal. Calcd. for C₁₆H₁₈: C, 91.42; H, 8.57. Found: C, 91.46; H, 8.62%.

4-Chlorophenyl-1-methyl-(3,5-xylyl) methane 4d: Brown crystals m.p. 171-72°C; IR (KBr): 2875, 3010 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 1.42 (d, 3H), 2.38 (s, 3H), 4.24 (q, 1H), 6.14-7.57 (m, 7H, Ar-H); ¹³C NMR: δ 21.76, 24.12, 38.23, 126.04, 127.12, 128.8, 129.6, 131.12, 138.1, 143.12; Anal. Calcd. for C₁₆H₁₇Cl: C, 78.50; H, 7.07. Found: C, 78.02; H, 7.92%.

1-[1-(4-Bromophenyl)-ethyl] naphthalene 4g: Dark brown crystals m.p. 110-12°C; IR (KBr): 2910, 3042 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 1.28 (d, 3H), 4.24 (q, 1H), 7.12-7.27 (m, 11H, Ar-H);

Table IV — Preparation of substituted diaryl propanes **5a-k**

Entry	Product	R ¹	Ar	Time/hr	Yield (% ^a)
1	5a	H		17	72
2	5b	H		18	86
3	5c	H		18	82
4	5d	Cl		20	66
5	5e	Cl		22	68
6	5f	Br		22	70
7	5g	Br		24	77
8	5h	OH		24	54
9	5i	OMe		18	57
10	5j	NO ₂		22	73
11	5k	NO ₂		22	80

^a Isolated yields are determined after work-up and chromatography purification

¹³C NMR: δ 24.12, 37.23, 123.12, 125.3, 126.10, 128.8, 129.6, 143.12; Anal. Calcd. for C₁₈H₁₅Br: C, 69.31; H, 6.39. Found: C, 69.12; H, 6.46%.

9-[1-(4-Nitrophenyl)-ethyl]anthracene 4j: Brown crystals m.p. 81-83°C; IR (KBr): 2975, 3040, 2965, 2938, 2918, 2875, 835-806 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 1.27 (d, 3H), 4.22 (q, 1H), 7.12-8.27 (m, 13H, Ar-H); ¹³C NMR: δ 24.12, 36.23, 124, 125.12, 126.05, 128.8, 129.6, 131.12, 132.01, 143.12, 149.04; Anal. Calcd. for C₂₂H₁₈: C, 80.71; H, 5.39; N, 4.28. Found: C, 80.52; H, 5.41; N, 4.36%.

9-[1-(4-Nitrophenyl)-ethyl]phenanthrene 4k: Brown crystals m.p. 120-21°C; IR (KBr): 2945, 3048, 2968 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 1.44 (d, 3H), 4.28 (q, 1H), 7.01-8.06 (m, 13H, Ar-H); ¹³C NMR: δ 24.22, 36.33, 124.03, 126, 127.22, 128.8, 129.8, 131.10, 143.22; Anal. Calcd. for C₂₂H₁₇NO₂: C, 80.71; H, 5.23; N, 4.28. Found: C, 80.42; H, 5.22; N, 4.33%.

Synthesis of diaryl propane 5: Other compounds **5a-g** were synthesized in similar manner using propiophenone (step 1) to gave **2c** (Table II) and then (step 2) with various selected aromatic hydrocarbons. Work-up of the reaction-mixture followed by column chromatography yielded yellow to dark brown crystalline solids **5a-k** (Table IV). Characterization data are presented as shown below.

Diphenyl propane 5a: Light brown crystals m.p. 117-18°C; IR (KBr): 2970, 2890, 3071, 3032 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 1.05 (t, 3H, *J* 7.2, -CH₂CH₃), 1.98 (q, 2H, *J* 7.3, -CH₂CH₃), 4.11 (t, 1H), 7.02-7.4 (m, 10H, Ar-H); ¹³C NMR: δ 12.4, 32.91, 44.2, 126.11, 127.2, 128.4, 129.2, 131.3, 143.61, 144.5; Anal. Calcd. for C₁₅H₁₆: C, 91.78; H, 8.16. Found: C, 91.72; H, 8.18%.

4-(1-Phenyl propyl) toluene 5b: Pale yellow liquid b.p. 104-105 °C; IR (KBr): 2920 3042 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 0.95 (t, 3H, *J* 7.2, -

CH_2CH_3), 1.96 (q, 2H, J 7.3, $-\text{CH}_2\text{CH}_3$), 4.21 (t, 1H), 7.04-7.48 (m, 9H, Ar-H); ^{13}C NMR: δ 11.12, 32.01, 44.32, 126.13, 127.2, 128.14, 129.21, 131.33, 143.62, 144.45; Anal. Calcd. for $\text{C}_{16}\text{H}_{18}$: C, 91.54; H, 8.57. Found: C, 91.62; H, 8.21%.

2-(1-Phenyl propyl)-xylene 5c: Colourless needles m.p. 87-88°C; IR (KBr): 2902, 3040 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 1.05 (t, 3H, J 7.2 $-\text{CH}_2\text{CH}_3$), 1.98 (q, 2H, J 7.3, $-\text{CH}_2\text{CH}_3$), 4.11 (t, 1H), 6.72-7.40 (m, 8H, Ar-H); ^{13}C NMR: δ 12.4, 32.91, 44.2, 126.11, 127.2, 128.4, 129.2, 131.3, 143.61, 144.5; Anal. Calcd. for $\text{C}_{17}\text{H}_{20}$: C, 91.04; H, 8.92. Found: C, 91.46; H, 8.62%.

4-Chlorophenyl-1-ethyl-(3,5-xylyl) methane 5d: Pale yellow needles m.p. 128-29°C; IR (KBr): 2922, 3042 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 1.07 (t, 3H, J 7.2 $-\text{CH}_2\text{CH}_3$), 1.92 (q, 2H, J 7.3, $-\text{CH}_2\text{CH}_3$), 4.19 (t, 1H), 7.01-7.34 (m, 7H, Ar-H); ^{13}C NMR: δ 12.6, 21.25, 32.94, 44.12, 126.14, 127.12, 128.4, 129.2, 138.3, 142.11, 143.61, 144.5; Anal. Calcd. for $\text{C}_{17}\text{H}_{19}\text{Cl}$: C, 78.94; H, 7.42. Found: C, 78.46; H, 7.32%.

4-[1-(4-Chlorophenyl)-propyl] biphenyl 5e: Brown crystals m.p. 123-24°C; IR (KBr): 2875, 3010 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 1.12 (t, 3H, J 7.2 $-\text{CH}_2\text{CH}_3$), 1.94 (q, 2H, J 7.3 $-\text{CH}_2\text{CH}_3$), 4.14 (t, 1H), 7.12-7.64 (m, 13H, Ar-H); ^{13}C NMR: δ 12.4, 21.57, 32.91, 44.2, 126.13, 127.12, 128.14, 129.22, 134.13, 136.12, 143.53, 144.15; Anal. Calcd. for $\text{C}_{21}\text{H}_{19}\text{Cl}$: C, 82.20; H, 6.24. Found: C, 82.12; H, 6.32%.

1-[1-(4-Bromophenyl) propyl] naphthalene 5g: Pale yellow crystals m.p. 92-93°C; IR(KBr): 2910, 3040 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 0.97 (t, 3H, J 7.2, CH_2CH_3), 2.18 (q, 2H, J 7.3, $-\text{CH}_2\text{CH}_3$), 4.12 (t, 1H), 7.01-7.76 (m, 11H, Ar-H); ^{13}C NMR: δ 11.4, 32.71, 42.12, 120.02, 123.23, 125.21, 127.12, 128.24, 130.4, 131.12, 132.2, 133.32, 142.11, 143.31; Anal. Calcd. for $\text{C}_{19}\text{H}_{17}\text{Br}$: C, 70.17; H, 5.31. Found: C, 70.12; H, 5.36%.

9-[1-(4-Nitrophenyl)-ethyl]anthracene 5j: Light brown crystals m.p. 141-42°C; IR (KBr): 2975, 3040, 2965, 2875; ^1H NMR (400 MHz, CDCl_3): δ 0.98 (t, 3H, J 7.2 $-\text{CH}_2\text{CH}_3$), 1.88 (q, 2H, J 7.3, $-\text{CH}_2\text{CH}_3$), 4.17 (t, 1H), 7.11-8.21 (m, 13H, Ar-H); ^{13}C NMR:

δ 11.4, 32.71, 40.12, 124.12, 125.43, 126.14, 127.12, 128.24, 129.12, 132.12, 144.11, 145.9; Anal. Calcd. for $\text{C}_{24}\text{H}_{17}\text{NO}_2$: C, 80.17; H, 5.23; N, 4.28. Found: C, 80.12; H, 5.41; N, 4.23 %.

9-[1-(4-Nitrophenyl)ethyl]phenanthrene 5k: Pale yellow crystals m.p. 122-23°C; IR (KBr): 2942, 3042, 2948 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 1.26 (t, 3H, J 7.3, CH_2CH_3), 1.94 (t, 1H, J 7.2, CHCH_2-), 4.72 (t, 1H), 7.54-8.18 (m, 13H, Ar-H); ^{13}C NMR: 12.07, 32.24, 42.81, 122.12, 124.11, 125.14, 126.21, 128.3, 129.23, 130.21, 131.41, 132.21, 141.32, 144.14, 149.24; Anal. Calcd. for $\text{C}_{24}\text{H}_{17}\text{NO}_2$: C, 80.24; H, 5.75. Found: C, 80.42; H, 5.52%.

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