3D View of 2-(3-nitro-phenyl)-1,3-bis(1-naphthylmethyl)-imidazolidine(3h)

3D View of 2-(2-chloro-phenyl)-1,3-bis(1-naphthylmethyl)-imidazolidine(3n)
3.4. EXPERIMENTAL

Synthesis of 2-substituted-1,3-bis(1-naphthylmethyl)-imidazolidine derivatives

3.4.1. Reaction Scheme

Scheme 3. Reagents and conditions: synthetic route to target compounds 3a-n. (i) dry benzene; (ii) refluxed azeotropically using Dean-Stark apparatus; (iii) refluxed for further 8 h; (iv) CH$_3$OH, DCM, NaBH$_4$, stirred for 7 h, 10˚C - 18˚C; (v) absolute C$_2$H$_5$OH, R-CHO, stirred at room temp.

**Where** a, R= C$_6$H$_5$; b, R= C$_{10}$H$_{14}$; c, R= 2-OH-C$_6$H$_4$; d, R= 4-OCH$_3$-C$_6$H$_4$; e, R= 4-N(CH$_3$)$_2$-C$_6$H$_4$; f, R= 4-Cl-C$_6$H$_4$; g, R= 3,4-OCH$_3$-C$_6$H$_3$; h, R= 3-NO$_2$-C$_6$H$_4$; i, R= 4-NO$_2$-C$_6$H$_4$; j, R= 4-OH- C$_6$H$_4$; k, R= C$_4$H$_9$O; l, R= 4-OH-3-OCH$_3$-C$_6$H$_3$; m, R= 4-F-C$_6$H$_4$; n, R= 2-Cl-C$_6$H$_4$. 
3.4.2. CHEMISTRY

The chemicals & solvents used for the experimental work were commercially procured from the different chemical suppliers such as E. Merck India Ltd, S.D.Fine Chem.Ltd, CDH Pvt Ltd, and Qualigens. The solvents and reagents were of LR grade.

Various solvents used during the course of present investigations were properly purified by conventional methods. Some liquid reagents were distilled off, before use while solid chemicals were used without further purification.

The physicochemical techniques employed for the characterization of all the synthesized compounds include melting point determination, elemental analysis, thin layer chromatography (TLC), infrared (IR), proton nuclear magnetic resonance ($^1$H NMR), carbon nuclear magnetic resonance ($^{13}$C NMR), and mass spectroscopy.

All the synthesized compounds were dried in vacuum desiccator over silica gel.

The melting point of the compounds was determined in open capillary tubes in the Hicon digital instrument.

Elemental analysis was performed on Vario EL III CHNOS analyzer using sulphanilic acid as a standard.

Completion of the reaction was monitored using thin layer chromatography (TLC). TLC was accomplished with silica gel-G chromatoplates of 15 cm length and 0.2 cm thickness. The spots were visualized either by exposing the developed and dried plates to iodine vapours or in UV chamber.

The infrared (IR) spectras of the synthesized compounds were recorded in the region 4000-400 cm\(^{-1}\) range using potassium bromide (KBr) discs on FT-IR Shimadzu 8400S spectrometer with resolution of 8 cm\(^{-1}\) and total of 20 number of scan.

The proton nuclear magnetic resonance ($^1$H NMR) spectra of the compounds were recorded on Brooker model DPX-300 NMR spectrometer at 300 MHz DMSO-$d_6$ as solvent. Some spectras were also recorded on 400 MHz, using DMSO-$d_6$ as solvent. Tetramethylsilane (TMS) was used as an internal standard and values of the chemical shifts are given in $\delta$ scale. Chemical shift are recorded in parts per million (ppm, $\delta$) and the signals are described as a s (singlet), d (doublet), dd (double doublet), and m (multiplet).
The carbon nuclear magnetic resonance ($^{13}$C NMR) spectra of compounds were recorded on Brooker model DPX-300 NMR spectrometer using DMSO-$d_6$ as solvent. Tetramethyldisilane (TMS) was used as an internal standard and values of the chemical shift are given in δ scale. Mass spectra of the compounds were recorded using Perkin Elmer 500 spectrometer.
To achieve this goal, 1-naphthaldehyde was selected as starting material to obtain some new 2-substituted-1,3-bis(1-naphthylmethyl)-imidazolidine derivatives (3a-n).

3.4.3. Synthesis of $N,N'$-bis(1-naphthylmethylene)ethane-1,2-diamine (1)

A mixture of 1-naphthaldehyde (5.4 mL, 0.02 mol) and ethylenediamine (1.5 mL) in dry benzene (30 mL) was refluxed in a R.B. flask after fitting the Dean-Stark apparatus in order to remove the water azeotropically. After removal of water the reaction mixture was refluxed for another 8 h and then left at room temperature overnight. A brownish white compound crystallized out which was filtered and recrystallized from methanol to give TLC pure, yellow crystals (1), m.p. 116-118 °C, yield (4.78 g, 71%).

$^1$H NMR (DMSO-$d_6$) δ (ppm): 4.04 (s, 4H, 2×CH$_2$), 7.36-7.64 (m, 8H, naphthalene), 7.79-8.14 (m, 6H, naphthalene), 8.98 (s, 2H, aldimine protons).

Reaction 3.1: Synthetic route of $N,N'$-bis(1-naphthylmethylene)ethane-1,2-diamine.

3.4.4. Synthesis of $N,N'$-bis(1-naphthylmethyl)ethane-1,2-diamine (2)

The above diSchiff’s base (1) (3.36 g, 0.01 mol) was dissolved in a mixture of methanol and dichloromethane (30 mL + 10 mL). A solution of sodium borohydride (1.0 g) in 2N NaOH solution (2.0 mL) was diluted with water (12.0 mL). This solution was added to the solution of (1) while stirring magnetically under ice cold conditions at such a rate that the reaction temperature did not rise above 18 °C. The contents were then stirred for 7 h. After completion of the reaction, methanol was distilled off and the residue was diluted with water (25.0 mL). It was then extracted with diethyl ether (3x20mL). The ethereal layer was dried over sodium sulphate. After filtering off the inorganic salt, ether was distilled off. A yellowish white solid mass was obtained. Purity of tetrahydrodiSchiff’s base was checked by TLC using benzene: acetone (8:2) as mobile phase, m.p. 68-70 °C, yield (2.21 g, 65%).
$^1$H NMR (DMSO-$d_6$) $\delta$ (ppm): 2.13 (s, 2H, NH) 2.50 (s, 4H, 2×CH$_2$), 4.11 (s, 4H, 2×CH$_2$, naphthylmethylene), 7.38-7.65 (m, 8H, naphthalene), 7.76-8.12 (m, 6H, naphthalene).

**Reaction 3.2:** Synthetic route of $N,N'$-bis(1-naphthylmethyl)ethane-1,2-diamine.

### 3.4.5. Synthesis of 2-substituted-1,3-bis(1-naphthylmethyl)imidazolidines (3a-n)

**Reaction 3.3:** Synthetic route of 2-substituted-1,3-bis(1-naphthylmethyl)imidazolidine (3a-n).

#### 3.4.5.1. 2-(Phenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3a)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution benzaldehyde (0.212 g, 0.2 mL, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 5 h. It was then left in a refrigerator overnight. A solid mass in the form of light yellowish crystals separated out which was filtered, washed with ethanol and dried, m. p. 144-146 °C, yield (0.270 g, 63%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

**FTIR (KBr pellet) cm$^{-1}$:** 3186 (aromatic C-H stretch), 2869 (aliphatic C-H stretch), 1596, 1560, 1524 (C=C ring stretch), 1307 (C-N ring stretch), 748 (C-N bending).

$^1$H NMR (DMSO-$d_6$) $\delta$ (ppm): 2.50, 2.63 (m, 4H, 2×CH$_2$, tetrahydroimidazole), 3.58, 3.62 (d, 4H, $J = 3.9$ Hz, 2×CH$_2$, naphthylmethylene) 4.12 (s, CH, tetrahydroimidazole), 7.32 (m, 2H, naphthalene), 7.40-7.55 (m, 5H, ArH), 7.60-7.66 (m, 6H, naphthalene), 7.73-7.86 (m, 6H, naphthalene).
\(^{13}\)C NMR (DMSO-\(d_6\)) \(\delta\) (ppm): 138.4 (1C), 137.5 (2C), 137.2 (2C), 129.8 (2C), 128.3 (2C), 128.1 (2C), 127.7 (1C), 126.9 (2C), 126.1 (2C), 122.4 (2C), 122.1 (2C), 120.3 (2C), 118.6 (2C), 116.9 (2C), 88.1 (1C), 52.2 (2C), 43.7 (2C).

**MS:** \(m/z\) 428 (M+), 351, 300, 287, 141, 91, 65.

**Anal. Calcd for C\(_{31}\)H\(_{28}\)N\(_2\):** C, 86.88; H, 6.59; N, 6.54; Found C, 86.40; H, 6.61; N, 6.48%.

### 3.4.5.2. 2-(1-Naphthyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3b)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution 1-naphthaldehyde (0.312 g, 0.3 mL, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 6 h. It was then left in a refrigerator overnight. A solid mass in the form of brownish crystals separated out which was filtered, washed with ethanol and dried, m. p. 80-82 °C, yield (0.310 g, 65%). On TLC examination in benzene: ethanol (8:2) it was found to be pure.

**FTIR (KBr pellet) cm\(^{-1}\):** 3016 (aromatic C-H stretch), 2885 (aliphatic C-H stretch), 1623, 1492, 1469 (C=C ring stretch), 1272 (C-N ring stretch), 806 (C-N bending).

\(^1\)H NMR (DMSO-\(d_6\)) \(\delta\) (ppm): 2.48, 2.90 (m, 4H, 2×CH\(_2\), tetrahydroimidazole), 3.75, 4.09 (d, 4H, 2×CH\(_2\), naphthylmethylene) 5.03 (s, CH, tetrahydroimidazole), 7.27-8.47 (m, 21H, naphthalene).

\(^{13}\)C NMR (DMSO-\(d_6\)) \(\delta\) (ppm): 139.7 (1C), 137.3 (2C), 129.6 (3C), 128.5 (3C), 127.6 (3C), 126.5 (1C), 126.1 (2C), 121.4 (3C), 121.0 (3C), 120.4 (3C), 118.6 (3C), 117.3 (3C), 89.3 (1C), 52.1 (2C), 43.8 (2C).

**Anal. Calcd for C\(_{35}\)H\(_{30}\)N\(_2\):** C, 87.83; H, 6.32; N, 5.85; Found C, 88.20; H, 6.11; N, 5.98%.

### 3.4.5.3. 2-(2-Hydroxyphenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3c)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution 2-hydroxybenzaldehyde (0.244 g, 0.23 mL, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 6 h. It was then left in a refrigerator overnight. A solid mass in the form of yellowish crystals separated out which was filtered, washed with ethanol and dried, m. p. 124-126 °C, yield (0.244 g, 55%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.
FTIR (KBr pellet) cm$^{-1}$: 3382 (OH), 3089 (aromatic C-H stretch), 2923 (aliphatic C-H stretch), 1623, 1496, 1454 (C=C ring stretch), 1272 (C-N ring stretch), 1188 (C-O), 748 (C-N bending).

$^1$H NMR (DMSO-$d_6$) $\delta$ (ppm): 2.49, 2.71 (m, 4H, 2×CH$_2$, tetrahydroimidazole), 4.47, 5.25 (d, 4H, 2×CH$_2$ naphthylmethylen), 5.65 (s, CH, tetrahydroimidazole), 5.98 (s, 1H, OH), 7.30 (m, 2H, naphthalene), 7.38-7.48 (m, 4H, ArH), 7.50-8.60 (m, 12H, naphthalene).

$^{13}$C NMR (DMSO-$d_6$) $\delta$ (ppm): 142.7 (1C), 138.5 (2C), 137.2 (2C), 129.8 (1C), 128.3 (2C), 128.1 (2C), 127.9 (2C), 126.8 (2C), 126.1 (2C), 123.4 (2C), 122.3 (1C), 121.0 (2C) 120.5 (2C), 117.6 (2C), 114.3 (1C), 88.1 (1C), 52.2 (2C), 43.7 (2C).

Anal. Calcd for C$_{31}$H$_{28}$N$_2$O: C, 83.75; H, 6.35; N, 6.30; Found C, 83.40; H, 6.52; N, 6.15%.

3.4.5.4. 2-(4-Methoxyphenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3d)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution 4-methoxybenzaldehyde (0.272 g, 0.26 mL, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 7 h. It was then left in a refrigerator overnight. A solid mass in the form of yellowish crystals separated out which was filtered, washed with ethanol and dried, m. p. 102-106 °C, yield (0.261 g, 57%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

FTIR (KBr pellet) cm$^{-1}$: 3039 (aromatic C-H stretch), 2862 (aliphatic C-H stretch), 1623, 1550, 1527 (C=C ring stretch), 1307 (C-N ring stretch), 1207 (C-O), 744 (C-N bending).

$^1$H NMR (DMSO-$d_6$) $\delta$ (ppm): 2.48, 2.74 (m, 4H, 2×CH$_2$, tetrahydroimidazole), 3.56, 3.80 (d, 4H, 2×CH$_2$ naphthylmethylen), 4.09 (s, 3H, OCH$_3$), 4.38 (s, CH, tetrahydroimidazole), 7.07 (dd, 2H, $J = 6.9$ Hz, $J = 2.1$ Hz, ArH), 7.12 (dd, 2H, $J = 5.7$ Hz, $J = 1.8$ Hz, ArH), 7.33-7.98 (m, 14H, naphthalene).

$^{13}$C NMR (DMSO-$d_6$) $\delta$ (ppm): 143.8 (1C), 137.4 (2C), 136.7 (2C), 131.5 (1C), 130.6 (2C), 128.6 (2C), 128.3 (2C), 127.2 (2C), 126.3 (2C), 122.5 (2C), 122.0 (2C), 121.3 (2C), 119.7 (2C), 118.4 (2C), 86.4 (1C), 54.7 (1C), 53.2 (2C), 44.3 (2C).

MS: $m/z$ 458 (M+), 351, 330, 317, 141, 121.

Anal. Calcd for C$_{32}$H$_{30}$N$_2$O: C, 83.81; H, 6.59; N, 6.11; Found C, 84.13; H, 6.48; N, 6.00%.
3.4.5.5. 2-(4-Dimethylaminophenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3e)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution 4-dimethylaminobenzaldehyde (0.298 g, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 8 h. It was then left in a refrigerator overnight. A solid mass in the form of white crystals separated out which was filtered, washed with ethanol and dried, m. p. 126-130 °C, yield (0.273 g, 58%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

FTIR (KBr pellet) cm⁻¹: 3043 (aromatic C-H stretch), 2854 (aliphatic C-H stretch), 1623, 1550, 1527 (C=C ring stretch), 1305 (C-N ring stretch), 748 (C-N bending).

¹H NMR (DMSO-d₆) δ (ppm): 2.48, 2.98 (m, 4H, 2×CH₂, tetrahydroimidazole), 2.73 (s, 6H, 2×CH₃), 3.71, 3.82 (d, 4H, 2×CH₂, naphthylmethylene), 4.95 (s, CH, tetrahydroimidazole), 7.27 (dd, 2H, J = 7.2 Hz, J = 2.4 Hz, ArH), 7.40 (dd, 2H, J = 5.4 Hz, J = 1.8 Hz, ArH), 7.77-8.47 (m, 14H, naphthalene).

¹³C NMR (DMSO-d₆) δ (ppm): 140.1 (1C), 134.2 (2C), 132.3 (2C), 129.3 (2C), 128.4 (2C), 127.7 (2C), 127.2 (1C), 126.8 (2C), 126.1 (2C), 122.9 (2C), 122.4 (2C), 120.6 (2C), 118.3 (2C), 117.4 (2C), 87.8 (1C), 51.6 (2C), 42.9 (2C), 38.1 (2C).

Anal. Calcd for C₃₃H₃₃N₃: C, 84.04; H, 7.05; N, 8.91; Found C, 83.60; H, 6.75; N, 9.00%.

3.4.5.6. 2-(4-Chlorophenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3f)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution 4-chlorobenzaldehyde (0.281 g, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 5 h. It was then left in a refrigerator overnight. A solid mass in the form of light yellowish crystals separated out which was filtered, washed with ethanol and dried, m. p. 110-114 °C, yield (0.333 g, 72%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

FTIR (KBr pellet) cm⁻¹: 3047 (aromatic C-H stretch), 2854 (aliphatic C-H stretch), 1623, 1527, 1477 (C=C ring stretch), 1292 (C-N ring stretch), 802 (C-N bending), 705 (C-Cl).

¹H NMR (DMSO-d₆) δ (ppm): 2.50, 2.63 (m, 4H, 2×CH₂, tetrahydroimidazole), 3.88, 4.20, (d, 4H, 2×CH₂, naphthylmethylene) 4.47 (s, CH, tetrahydroimidazole), 7.16 (dd, 2H, J = 5.1
Hz, \( J = 1.8 \) Hz, ArH), 7.33 (dd, 2H, \( J = 6.1 \) Hz, \( J = 2.4 \) Hz, ArH), 7.42-8.19 (m, 14H, naphthalene).

\(^{13}\)C NMR (DMSO-\(d_6\) \(\delta\) (ppm)): 136.1 (1C), 133.7 (2C), 132.1 (2C), 127.9 (2C), 127.6 (2C), 126.5 (2C), 126.0 (1C), 125.2 (2C), 123.7 (2C), 122.6 (2C), 121.1 (2C), 120.6 (2C), 118.8 (2C), 117.2 (2C), 88.2 (1C), 52.4 (2C), 43.7 (2C).

**MS:** m/z 465 (M+2), 463 (M+), 351, 334, 321, 141, 125.

**Anal. Calcd for C_{31}H_{27}ClN_{2}:** C, 80.42; H, 5.88; N, 6.05; Found C, 79.99; H, 5.68; N, 6.10%.

3.4.5.7. 2-(3,4-Dimethoxyphenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3g)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution, 3,4-dimethoxybenzaldehyde (0.332 g, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 7 h. It was then left in a refrigerator overnight. A solid mass in the form of yellowish crystals separated out which was filtered, washed with ethanol and dried, m.p. 106-108 °C, yield (0.200 g, 41%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

**FTIR (KBr pellet) cm\(^{-1}\):** 3085 (aromatic C-H stretch), 2850 (aliphatic C-H stretch), 1623, 1492, 1469 (C=C ring stretch), 1272 (C=N ring stretch), 752 (C-N bending).

\(^1\)H NMR (DMSO-\(d_6\) \(\delta\) (ppm)): 2.53, 2.74 (m, 4H, 2\(\times\)CH\(_2\), tetrahydroimidazole), 3.29, 3.38 (d, 4H, 2\(\times\)CH\(_2\) naphthylmethylene), 3.94 (s, 6H, 2\(\times\)OCH\(_3\)), 4.38 (s, CH, tetrahydroimidazole), 7.09-7.33 (m, 3H, ArH), 7.38-7.98 (m, 14H, naphthalene).

\(^{13}\)C NMR (DMSO-\(d_6\) \(\delta\) (ppm)): 137.6 (1C), 137.4 (1C), 134.8 (2C), 131.6 (2C), 131.2 (2C), 129.2 (2C), 128.0 (1C), 127.5 (1C), 126.2 (2C), 126.0 (2C), 123.6 (2C), 122.4 (2C), 121.1 (2C), 118.7 (2C), 116.5 (2C), 87.3 (1C), 53.1 (2C), 51.6 (2C), 41.4 (2C).

**MS:** m/z 488 (M+), 360, 351, 347, 151, 141.

**Anal. Calcd for C_{33}H_{32}N_{2}O_{2}:** C, 81.12; H, 6.60; N, 5.73; Found C, 80.92; H, 6.51; N, 5.64%.

3.4.5.8. 2-(3-Nitrophenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3h)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution, 3-nitrobenzaldehyde (0.302 g, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 9 h. It was then left in a refrigerator overnight. A solid mass
in the form of creamy white crystals separated out which was filtered, washed with ethanol and dried, m. p. 112-114 °C, yield (0.293 g, 62%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

**FTIR (KBr pellet) cm⁻¹:** 3213 (aromatic C-H stretch), 2854 (aliphatic C-H stretch), 1604, 1577, 1539 (C=C ring stretch), 1261 (C-N ring stretch), 756 (C-N bending).

**¹H NMR (DMSO-d₆) δ (ppm):** 2.56, 2.71 (m, 4H, 2×CH₂, tetrahydroimidazole), 3.58, 3.62 (d, 4H, 2×CH₂, naphthylmethylene) 4.32 (s, CH, tetrahydroimidazole), 7.34-7.66 (m, 14H, naphthalene), 7.73-7.79 (m, 4H, ArH).

**¹³C NMR (DMSO-d₆) δ (ppm):** 146.1 (1C), 141.9 (1C), 140.7 (1C), 140.2 (1C), 132.7 (2C), 129.6 (2C), 128.5 (2C), 127.7 (2C), 126.8 (2C), 126.4 (2C), 124.6 (1C), 123.2 (1C), 122.8 (2C), 120.2 (2C), 117.6 (2C), 117.1 (2C), 88.1 (1C), 52.2 (2C), 43.9 (2C).

**Anal. Calcd for C₃₁H₂₇N₃O₂:** C, 78.62; H, 5.75; N, 8.87; Found C, 78.94; H, 5.60; N, 8.78%.

### 3.4.5.9. 2-(4-Nitrophenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3i)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution 4-nitrobenzaldehyde (0.302 g, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 8 h. It was then left in a refrigerator overnight. A solid mass in the form of yellowish crystals separated out which was filtered, washed with ethanol and dried, m. p. 94-98 °C, yield (0.312 g, 66%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

**FTIR (KBr pellet) cm⁻¹:** 3066 (aromatic C-H stretch), 2927 (aliphatic C-H stretch), 1654, 1639 (C=C ring stretch), 1319 (C-N ring stretch), 748 (C-N bending).

**¹H NMR (DMSO-d₆) δ (ppm):** 2.57, 2.78 (m, 4H, 2×CH₂, tetrahydroimidazole), 3.52, 3.62 (d, 4H, 2×CH₂, naphthylmethylene) 4.34 (s, CH, tetrahydroimidazole), 7.32-7.63 (m, 14H, naphthalene), 7.73 (dd, 2H, J = 5.7 Hz, J = 2.1 Hz, ArH), 7.84 (dd, 2H, J = 6.1 Hz, J = 1.8 Hz, ArH).

**¹³C NMR (DMSO-d₆) δ (ppm):** 136.4 (1C), 135.2 (1C), 134.1 (2C), 129.8 (2C), 128.9 (2C), 128.5 (2C), 127.8 (2C), 124.6 (2C), 123.5 (2C), 121.6 (2C), 119.8 (2C), 119.3 (2C), 118.2 (2C), 115.9 (2C), 86.2 (1C), 53.6 (2C), 42.7 (2C).

**Anal. Calcd for C₃₁H₂₇N₃O₂:** C, 78.62; H, 5.75 N, 8.87; Found C, 78.41; H, 5.60; N, 8.98%.
3.4.5.10. 2-(4-Hydroxyphenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3j)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution 4-hydroxybenzaldehyde (0.244 g, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 6 h. It was then left in a refrigerator overnight. A solid mass in the form of light yellowish crystals separated out which was filtered, washed with ethanol and dried, m. p. 102-104 °C, yield (0.168 g, 38%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

FTIR (KBr pellet) cm⁻¹: 3344 (O-H), 3047 (aromatic C-H stretch), 2941 (aliphatic C-H stretch), 1596, 1508, 1434 (C=C ring stretch), 1249 (C-N ring stretch), 1215 (C-O), 779 (C-N bending).

¹H NMR (DMSO-d₆) δ (ppm): 2.43, 2.76 (m, 4H, 2×CH₂, tetrahydroimidazole), 4.41, 5.21 (d, 4H, 2×CH₂, naphthylmethylene) 5.65 (s, CH, tetrahydroimidazole), 5.97 (s, 1H, OH), 7.26 (m, 2H, naphthalene), 7.32 (dd, 2H, J = 6.1 Hz, J = 2.1 Hz, ArH), 7.45 (dd, 2H, J = 5.4 Hz, J = 1.8 Hz, ArH), 7.54-8.42 (m, 12H, naphthalene).

¹³C NMR (DMSO-d₆) δ (ppm): 143.7 (1C), 132.0 (2C), 131.5 (2C), 128.6 (2C), 128.3 (2C), 128.1 (1C), 127.0 (2C), 126.6 (2C), 124.4 (2C), 122.3 (2C), 121.4 (2C), 120.5 (2C), 119.7 (2C), 118.3 (2C), 86.2 (1C), 54.6 (2C), 45.3 (2C).

MS: m/z 444 (M+), 351, 316, 303, 141, 107.

Anal. Calcd for C₃₁H₂₈N₂O: C, 83.75; H, 6.35; N, 6.30; Found C, 83.50; H, 6.71; N, 6.42%.

3.4.5.11. 2-(2-Furanyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3k)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution furfuraldehyde (0.193 g, 0.19 mL, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 9 h. It was then left in a refrigerator overnight. A solid mass in the form of dusty white crystals separated out which was filtered, washed with ethanol and dried, m. p. 108-112 °C, yield (0.284 g, 68%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

FTIR (KBr pellet) cm⁻¹: 3109 (aromatic C-H stretch), 2885 (aliphatic C-H stretch), 1608, 1577, 1524 (C=C ring stretch), 1384 (C-N imide ring stretch), 1265 (asymmetric C-O-C), 752 (C-N bending).
\(^1\)H NMR (DMSO-\(d_6\)) \(\delta\) (ppm): 2.49, 2.62 (m, 4H, 2×CH\(_2\), tetrahydroimidazole), 3.54, 3.71 (d, 4H, 2×CH\(_2\), naphthylmethylene) 4.76 (s, CH, tetrahydroimidazole), 6.97-7.10 (m, 3H, furan), 7.28-7.76 (m, 14H, naphthalene).

\(^{13}\)C NMR (DMSO-\(d_6\)) \(\delta\) (ppm): 146.5 (1C), 141.8 (1C), 129.4 (2C), 128.1 (2C), 126.3 (2C), 125.2 (2C), 124.6 (2C), 123.5 (2C), 121.6 (2C), 121.4 (2C), 120.7 (2C), 116.2 (2C), 112.6 (1C), 110.3 (1C), 88.4 (1C), 51.2 (2C), 46.2 (2C).

MS: \(m/z\) 418 (M\(^+\)), 351, 290, 277, 141, 81.

**Anal. Calcd for C\(_{29}\)H\(_{26}\)N\(_2\)O:** C, 83.22; H, 6.26; N, 6.69; Found C, 82.95; H, 6.20; N, 6.55%.

3.4.5.12. 2-(4-Hydroxy-3-methoxyphenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3l)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution 4-hydroxy-3-methoxybenzaldehyde (0.304 g, 0.002 mol) was added and the mixture stirred on a magnetic stirrer for 7 h. It was then left in a refrigerator overnight. A solid mass in the form of yellowish crystals separated out which was filtered, washed with ethanol and dried, m. p. 115-117 °C, yield (0.166 g, 35%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

**FTIR (KBr pellet) cm\(^{-1}\):** 3398 (O-H), 3043 (aromatic C-H stretch), 2854 (aliphatic C-H stretch), 1704 (C=O), 1523, 1527, 1481 (C=C ring stretch), 1292 (C-N ring stretch), 1207 (C-O stretch), 748 (C-N bending).

\(^1\)H NMR (DMSO-\(d_6\)) \(\delta\) (ppm): 2.53, 2.74 (m, 4H, 2×CH\(_2\), tetrahydroimidazole), 3.38, 3.52 (d, 4H, 2×CH\(_2\), naphthylmethylene), 3.94 (s, 3H, OCH\(_3\)), 4.38 (s, CH, tetrahydroimidazole), 7.07-7.12 (m, 3H, ArH), 7.36-7.87 (m, 14H, naphthalene), 9.85 (s, 1H, OH).

\(^{13}\)C NMR (DMSO-\(d_6\)) \(\delta\) (ppm): 145.2 (1C), 141.4 (1C), 141.1 (1C), 133.2 (2C), 129.2 (2C), 128.6 (2C), 127.8 (2C), 127.4 (2C), 126.6 (2C), 126.2 (2C), 123.1 (1C), 122.7 (2C), 121.6 (2C), 119.1 (1C), 118.7 (1C), 117.2 (2C), 84.3 (1C), 50.6 (2C), 46.3 (1C), 43.7 (2C).

**Anal. Calcd for C\(_{32}\)H\(_{30}\)N\(_2\)O\(_2\):** C, 80.98; H, 6.37; N, 5.90; Found C, 81.33; H, 6.48; N, 5.81%.

3.4.5.13. 2-(4-Fluorophenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3m)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution 4-fluorobenzaldehyde (0.248 g, 0.24 mL, 0.002 mol) and the mixture
stirred on a magnetic stirrer for 7 h. It was then left in a refrigerator overnight. A solid mass in the form of light yellowish crystals separated out which was filtered, washed with ethanol and dried, m. p. 136-138 °C, yield (0.330 g, 74%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

**FTIR (KBr pellet) cm⁻¹:** 3228 (aromatic C-H stretch), 2842 (aliphatic C-H stretch), 1539, 1515, 1428 (C=C ring stretch), 1384 (C-F), 1269 (C-N ring stretch), 748 (C-N bending).

**¹H NMR (DMSO-d₆) δ (ppm):** 2.51, 2.63 (m, 4H, 2×CH₂, tetrahydroimidazole), 3.67, 3.82 (d, 4H, 2×CH₂, naphthylmethylene) 4.78 (s, CH, tetrahydroimidazole), 7.04 (dd, 2H, J = 6.9 Hz, J = 2.1 Hz, ArH), 7.45 (dd, 2H, J = 5.7 Hz, J = 1.8 Hz, ArH), 7.60-7.94 (m, 14H, naphthalene).

**¹³C NMR (DMSO-d₆) δ (ppm):** 148.6 (1C), 136.8 (2C), 136.0 (1C), 131.2 (2C), 127.5 (2C), 127.1 (2C), 126.2 (2C), 124.8 (2C), 124.6 (2C), 123.4 (2C), 122.8 (2C), 119.6 (2C), 118.9 (2C), 116.6 (2C), 88.1 (1C), 51.8 (2C), 46.3 (2C).

**Anal. Calcd for C₃₁H₂₇FN₂:** C, 83.38; H, 6.09; N, 6.27; Found C, 82.89; H, 6.21; N, 6.57%.

### 3.4.5.14. 2-(2-Chlorophenyl)-1,3-bis(1-naphthylmethyl)imidazolidine (3n)

Compound 2 (0.680 g, 0.002 mol) was dissolved in absolute alcohol (12 mL) in a flat bottom flask. To this solution was added 2-chlorobenzaldehyde (0.281 g, 0.27 mL, 0.002 mol) and the mixture stirred on a magnetic stirrer for 6 h. It was then left in a refrigerator overnight. A solid mass in the form of creamy white crystals separated out which was filtered, washed with ethanol and dried, m. p. 124-126 °C, yield (0.319 g, 69%). On TLC examination in benzene: ethanol (9:1) it was found to be pure.

**FTIR (KBr pellet) cm⁻¹:** 3082 (aromatic C-H stretch), 2858 (aliphatic C-H stretch), 1596, 1527, 1473 (C=C ring stretch), 1307 (C-N ring stretch), 848 (C-Cl stretch), 750 (C-N bending).

**¹H NMR (DMSO-d₆) δ (ppm):** 2.48, 2.62 (m, 4H, 2×CH₂, tetrahydroimidazole), 3.54, 3.71 (d, 4H, 2×CH₂, naphthylmethylene) 4.72 (s, CH, tetrahydroimidazole), 7.06-7.15 (m, 4H, ArH), 7.32-7.83 (m, 14H, naphthalene).
$^{13}$C NMR (DMSO-$d_6$) $\delta$ (ppm): 134.1 (1C), 133.9 (2C), 133.6 (2C), 129.6 (2C), 128.6 (2C), 127.3 (1C), 126.1 (2C), 124.7 (2C), 124.5 (2C), 122.1 (2C), 120.6 (2C), 119.3 (2C), 118.6 (2C), 117.2 (2C), 86.4 (1C), 54.2 (2C), 46.7 (2C).

Anal. Calcd for C$_{31}$H$_{27}$ClN$_2$: C, 80.42; H, 5.88; N, 6.05; Found C, 80.65; H, 6.04; N, 6.13%.

Physical constants of the synthesized compounds of 2-substituted-1,3-bis(1-naphthylmethyl)-imidazolidine (3a-n) derivatives are given in Table 3.3.

Table 3.3: Physical constants of the synthesized compounds 3a-n.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Ar</th>
<th>Yield (%)</th>
<th>M.P. (°C)</th>
<th>$R_f$</th>
<th>Molecular Formula</th>
<th>Molecular Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>3a</td>
<td>C$_6$H$_5$</td>
<td>63</td>
<td>144-146</td>
<td>0.43</td>
<td>C$<em>{31}$H$</em>{28}$N$_2$</td>
<td>428.57</td>
</tr>
<tr>
<td>3b</td>
<td>C$_{10}$H$_7$</td>
<td>65</td>
<td>80-82</td>
<td>0.45</td>
<td>C$<em>{35}$H$</em>{30}$N$_2$</td>
<td>478.63</td>
</tr>
<tr>
<td>3c</td>
<td>2-OH-C$_6$H$_4$</td>
<td>55</td>
<td>124-126</td>
<td>0.37</td>
<td>C$<em>{31}$H$</em>{28}$N$_2$O</td>
<td>444.57</td>
</tr>
<tr>
<td>3d</td>
<td>4-OCH$_3$-C$_6$H$_4$</td>
<td>57</td>
<td>102-106</td>
<td>0.36</td>
<td>C$<em>{32}$H$</em>{30}$N$_2$O</td>
<td>458.59</td>
</tr>
<tr>
<td>3e</td>
<td>4-N(CH$_3$)$_2$-C$_6$H$_4$</td>
<td>58</td>
<td>126-130</td>
<td>0.33</td>
<td>C$<em>{33}$H$</em>{33}$N$_3$</td>
<td>471.64</td>
</tr>
<tr>
<td>3f</td>
<td>4-Cl-C$_6$H$_4$</td>
<td>72</td>
<td>110-114</td>
<td>0.44</td>
<td>C$<em>{31}$H$</em>{29}$ClN$_2$</td>
<td>463.01</td>
</tr>
<tr>
<td>3g</td>
<td>3,4-OCH$_3$-C$_6$H$_3$</td>
<td>41</td>
<td>106-108</td>
<td>0.30</td>
<td>C$<em>{33}$H$</em>{32}$N$_2$O$_2$</td>
<td>488.62</td>
</tr>
<tr>
<td>3h</td>
<td>3-NO$_2$-C$_6$H$_4$</td>
<td>62</td>
<td>112-114</td>
<td>0.41</td>
<td>C$<em>{31}$H$</em>{29}$N$_2$O$_2$</td>
<td>473.57</td>
</tr>
<tr>
<td>3i</td>
<td>4-NO$_2$-C$_6$H$_4$</td>
<td>66</td>
<td>94-98</td>
<td>0.35</td>
<td>C$<em>{31}$H$</em>{29}$N$_3$O$_2$</td>
<td>473.57</td>
</tr>
<tr>
<td>3j</td>
<td>4-OH- C$_6$H$_4$</td>
<td>38</td>
<td>102-104</td>
<td>0.33</td>
<td>C$<em>{31}$H$</em>{28}$N$_2$O$_2$</td>
<td>444.57</td>
</tr>
<tr>
<td>3k</td>
<td>C$_6$H$_2$O</td>
<td>68</td>
<td>108-112</td>
<td>0.44</td>
<td>C$<em>{30}$H$</em>{26}$N$_2$O</td>
<td>418.53</td>
</tr>
<tr>
<td>3l</td>
<td>4-OH-3-OCH$_3$-C$_6$H$_3$</td>
<td>35</td>
<td>115-117</td>
<td>0.37</td>
<td>C$<em>{32}$H$</em>{30}$N$_2$O$_2$</td>
<td>474.59</td>
</tr>
<tr>
<td>3m</td>
<td>4-F-C$_6$H$_4$</td>
<td>74</td>
<td>136-138</td>
<td>0.38</td>
<td>C$<em>{31}$H$</em>{29}$FN$_2$</td>
<td>446.56</td>
</tr>
<tr>
<td>3n</td>
<td>2-Cl-C$_6$H$_4$</td>
<td>69</td>
<td>124-126</td>
<td>0.46</td>
<td>C$<em>{31}$H$</em>{29}$ClN$_2$</td>
<td>463.01</td>
</tr>
</tbody>
</table>