N-(4-HYDROXYPHENYL)-3,4-DIMETHOXYBENZAMIDE (165)

4-Aminophenol (1.0 g, 9.2 mmol) was refluxed in tetrahydrofuran (50 ml) and added 3,4-dimethoxybenzoyl chloride (0.8 g, 4.0 mmol) dissolved in tetrahydrofuran (10 ml) and continued refluxing for 1 h. Completion of reaction was confirmed by TLC. Removal of tetrahydrofuran under reduced pressure gave a sticky black mass, which was crystallized from methanol to afford **165** (0.9 g, 35.9%), mp. 224-227°C.

Anal.:

IR (KBr): 3540, 3500, 3320, 2930, 1640, 1510, 1265, 1225, 1020 and 820 cm^{-1}.

^{1}H NMR ($CDCl_3$-DMSO-d_6): δ 3.93 (s, 6H, 2 × -OCH_3), 6.82 (q, 2H, Ar), 6.91 (d, 1H, J = 8.63 Hz, Ar), 7.52 (m, 4H, Ar) and 9.12 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

Calcd for $C_{15}H_{15}NO_4$: C, 65.92; H, 5.53; N, 5.13. Found: C, 65.89; H, 5.42; N, 5.18.

N-(2,3-EPOXYPROPOXYPHENYL)-3,4-DIMETHOXYBENZAMIDE (166)

N-(4-Hydroxyphenyl)-3,4-dimethoxybenzamide (**165**) (2.0 g, 7.3 mmol) was refluxed in epichlorohydrin (50 ml) for 2 h, added anhydrous potassium carbonate (5.0 g) and refluxing was continued for 6 h with stirring. TLC confirmed completion of reaction. The reaction mixture was filtered and the excess of epichlorohydrin removed under reduced pressure to obtain a solid residue, which was refluxed with acetone and then with methanol to remove impurities to afford **166** (1.7 g, 70.5%), mp. 188-191°C. The product **166** was used for further reaction without crystallization.

Anal.:

IR (KBr): 3300, 1635, 1505, 1260, 1230, 1155, 1030 and 810 cm^{-1}.

^{1}H NMR ($CDCl_3$-DMSO-d_6): δ 2.77(m, 1H, -CH_2 of oxirane), 2.92 (m, 1H, -CH_2 of oxirane), 3.36 (m, 1H, -C*H* of oxirane), 3.90 (m, 1H, -OCH_2-), 3.94 (s, 3H, -OCH_3), 3.95 (s, 3H, -OC*H*$_3$), 4.29 (dd, 1H, -OC*H*$_2$-), 6.92 (m, 3H, Ar), 7.61 (m, 4H, Ar) and 9.53 ppm (s, 1H, -N*H*CO-).

Calcd for $C_{18}H_{19}NO_5$: C, 65.64; H, 5.82; N, 4.25. Found: C, 65.40; H, 5.68; N, 4.60.

N-[4-(2-HYDROXY-3-ISOPROPYLAMINOPROPOXY)PHENYL]-3,4-DIMETHOXYBENZAMIDE (167)

N-(2,3-Epoxypropoxyphenyl)-3,4-dimethoxybenzamide (**166**) (0.5 g, 1.5 mmol) was refluxed in methanol (50 ml), added isopropylamine (15 ml) and refluxing was continued for 2.5 h. Excess of reagent was removed under reduced pressure to give a solid residue. The solid residue was crystallized from acetone to afford **167** (0.36 g, 61.1%), mp. 135-138°C.

Anal.:

IR (KBr): 3300, 2920, 1640, 1510, 1265, 1240, 1025 and 815 cm^{-1}.

^{1}H NMR (CDCl$_3$-DMSO-*d$_6$*): δ 1.08 (d, 6H, -CH(C*H*$_3$)$_2$), 2.69 (m, 1H, -C*H*$_2$NH-), 2.87 (m, 2H, -C*H*$_2$NH- & -C*H*(CH$_3$)$_2$), 3.96 (m, 8H, 2 × -OC*H*$_3$ & -OC*H*$_2$-), 4.03 (m, 1H, -C*H*(OH)-), 6.91 (m, 3H, Ar), 7.61 (m, 4H, Ar) and 9.52 ppm (s, 1H, -N*H*CO-, exchanged in D$_2$O).

Calcd for $C_{21}H_{28}N_2O_5$: C, 64.93; H, 7.27; N, 7.21. Found: C, 64.86; H, 7.22; N, 7.18.

N-[4-(2-HYDROXY-3-ISOPROPYLAMINOPROPOXY)PHENYL]-3,4-DIMETHOXYBENZAMIDE (167) OXALATE [DPJ-888]

To a solution of **167** (1.25 g, 3.2 mmol) in refluxing methanol (50 ml) added oxalic acid (0.5 g, 3.9 mmol) and the refluxing continued for 0.5 h. The solvent was removed under reduced pressure and the residue crystallized from methanol-acetone mixture to afford oxalate of **167** (0.88 g, 57.2%), mp. 198-201°C.

Anal.:

IR (KBr): 3410, 3340, 2990, 1650, 1600, 1540, 1510, 1265 and 1220 cm^{-1}.

^{1}H NMR (D_2O): δ 1.24 (d, 6H, -CH(C*H*$_3$)$_2$), 3.12 (m, 2H, -C*H*$_2$NH-), 3.38 (m, 1H, -C*H*(CH$_3$)$_2$), 3.67 (s, 3H, -OC*H*$_3$), 3.69 (s, 3H, -OC*H*$_3$), 3.88 (m, 2H, -OC*H*$_2$-), 4.16 (m, 1H, -C*H*(OH)-), 6.78 (d, 3H, Ar) and 7.17 ppm (m, 4H, Ar).

Calcd for $C_{23}H_{30}N_2O_9$: C, 57.73; H, 6.32; N, 5.85. Found: C, 57.61; H, 6.33; N, 5.91.

N-[4-(3-*tert*-BUTYLAMINO-2-HYDROXYPROPOXY)PHENYL]-3,4-DIMETHOXYBENZAMIDE (168)

N-(2,3-Epoxypropoxyphenyl)-3,4-dimethoxybenzamide (**166**) (1.5 g, 4.6 mmol) was refluxed in methanol (100 ml), added *tert*-butylamine (25 ml) and refluxing was continued for 3 h. Completion of reaction was confirmed by TLC. The solvent was removed under reduced pressure and the solid residue was crystallized from acetone to afford **168** (1.26 g, 68.7%), mp. 105-108°C.

Anal.:

IR (KBr): 3310, 2960, 2925, 1665, 1505, 1260, 1235, 1210 and 1025 cm^{-1}.

^{1}H NMR (CDCl$_3$-DMSO-*d$_6$*): δ 1.14 (s, 9H, -C(C*H*$_3$)$_3$), 2.69 (m, 1H, -C*H*$_2$NH-), 2.82 (m, 1H, -C*H*$_2$NH-), 3.96 (m, 9H, 2 × -OC*H*$_3$ & -OC*H*$_2$C*H*(OH)-), 6.92 (m, 3H, Ar), 7.62 (m, 4H, Ar) and 9.64 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

Calcd for $C_{22}H_{30}N_2O_5$: C, 65.65; H, 7.51; N, 6.96. Found: C, 65.16; H, 7.65; N, 6.98.

N-[4-(3-*tert*-BUTYLAMINO-2-HYDROXYPROPOXY)PHENYL]-3,4-DIMETHOXYBENZAMIDE (168) OXALATE [DPJ-890]

To a solution of **168** (1.25 g, 3.1 mmol) in refluxing methanol (50 ml), added oxalic acid (0.4 g, 3.2 mmol) and refluxing was continued for 0.5 h. The solvent was removed under reduced pressure and the residue was crystallized from methanol-acetone mixture to afford oxalate of **168** (0.6 g, 39.2%), mp. 195-198°C.

Anal.:

IR (KBr): 3410, 3345, 3000, 1735, 1660, 1605, 1510, 1265 and 1225 cm^{-1}.

^{1}H NMR (D_2O): δ 1.27 (s, 9H, $-C(CH_3)_3$), 3.12 (m, 2H, $-CH_2NH-$), 3.65 (s, 3H, $-OCH_3$), 3.67 (s, 3H, $-OCH_3$), 3.85 (m, 2H, $-OCH_2-$), 4.11 (m, 1H, -C*H*(OH)-), 6.76 (d, 3H, Ar) and 7.11 ppm (m, 4H, Ar).

Calcd for $C_{24}H_{32}N_2O_9$: C, 58.53; H, 6.55; N, 5.69. Found: C, 58.67; H, 6.57; N, 5.75.

N-{4-[2-HYDROXY-3-(4-METHYLPIPERAZIN-1-YL)PROPOXY]PHENYL}-3,4-DIMETHOXYBENZAMIDE (169)

A mixture of *N*-(2,3-epoxypropoxyphenyl)-3,4-dimethoxybenzamide (**166**) (1.5 g, 4.6 mmol) and *N*-methylpiperazine (1 ml, 9.0 mmol) in methanol (75 ml) was refluxed for 4 h. Completion of reaction was confirmed by TLC. The solvent was removed under reduced pressure and the solid residue was crystallized from ethyl acetate to afford **169** (1.23 g, 62.6%), mp. 140-143°C.

Anal.:

IR (KBr): 3460, 3285, 2935, 2795, 1640, 1510, 1270, 1235, 1140, 1020 and 815 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ 2.30 (s, 3H, $>NCH_3$), 2.40-2.72 (bm, 10H, $-CH_2N(CH_2CH_2)_2N-$), 3.94 (s, 6H, 2 × $-OCH_3$), 3.98 (d, 2H, $-OCH_2-$), 4.10 (m, 1H, -C*H*(OH)-), 6.91 (m, 3H, Ar), 7.38 (dd, 1H, Ar), 7.51 (m, 3H, Ar) and 7.78 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

Calcd for $C_{23}H_{31}N_3O_5$: C, 64.32; H, 7.27; N, 9.78. Found: C, 64.75; H, 7.32; N, 10.18.

N-{4-[2-HYDROXY-3-(4-METHYLPIPERAZIN-1-YL)PROPOXY]PHENYL}-3,4-DIMETHOXYBENZAMIDE (169) OXALATE [DPJ-893]

Oxalic acid (0.7 g, 5.5 mmol) was added to a solution of **169** (1.0 g, 2.3 mmol) in refluxing methanol (50 ml) and refluxing was continued for 15 min. Filtered the precipitates and crystallized from methanol to afford oxalate of **169** (0.65 g, 45.8%), mp. 209-212°C.

Anal.:

IR (KBr): 3470, 3435, 3380, 2930, 2330, 1720, 1640, 1505, 1410, 1265, 1230, 1080 and 1020 cm^{-1}.

^{1}H NMR (D_2O): δ 2.90 (s, 3H, >NCH_3), 3.37-3.58 (bm, 10H, -$CH_2N(CH_2CH_2)_2N$-), 3.74 (s, 6H, 2 × -OCH_3), 3.95 (m, 2H, -OCH_2-), 4.35 (m, 1H, -CH(OH)-), 6.88 (dd, 3H, Ar), 7.23 (d, 3H, Ar) and 7.33 ppm (d, 1H, Ar).

Calcd for $C_{27}H_{35}N_3O_{13}$: C, 53.20; H, 5.79; N, 6.89. Found: C, 53.18; H, 5.72; N, 6.80.

N-(4-HYDROXYPHENYL)-3,4,5-TRIMETHOXYBENZAMIDE (170)

4-Aminophenol (5.0 g, 45.8 mmol) was refluxed in tetrahydrofuran (100 ml) and then added 3,4,5-trimethoxybenzoyl chloride (4.0 g, 17.3 mmol) dissolved in tetrahydrofuran (10 ml) in small portions and refluxing was continued for 7 h. Completion of reaction was confirmed by TLC. Removal of tetrahydrofuran under reduce pressure gave a solid residue, which was crystallized from methanol to afford **170** (2.5 g, 18%), mp. 225-228°C.

Anal.:

IR (KBr): 3335, 1655, 1585, 1545, 1505, 1460, 1340, 1245, 1210 and 1020 cm^{-1}.

^{1}H NMR ($CDCl_3$-DMSO-d_6): δ 3.89 (s, 3H, -OCH_3), 3.93 (s, 6H, 2 × -OCH_3), 6.84 (d, 2H, J = 8.8 Hz, Ar), 7.21 (s, 2H, Ar), 7.49 (d, 2H, J = 8.7 Hz, Ar), 8.57 (s, 1H, -N*H*CO-, exchanged in D_2O) and 8.92 ppm (s, 1H, -O*H*, exchanged in D_2O).

Calcd for $C_{16}H_{17}NO_5$: C, 63.36; H, 5.65; N, 4.62. Found: C, 63.53; H, 5.42; N, 4.75.

N-(2,3-EPOXYPROPOXYPHENYL)-3,4,5-TRIMETHOXYBENZAMIDE (171)

N-(4-Hydroxyphenyl)-3,4,5-trimethoxybenzamide (**170)** (5.0 g, 16.5 mmol) was refluxed in epichlorohydrin (60 ml) for 2 h, added anhydrous potassium carbonate (6.0 g) and refluxing was continued for 7 h. Completion of reaction was determined by TLC. The reaction mixture was filtered and excess of epichlorohydrin removed under reduced pressure to obtain a solid residue, which was crystallized from acetone to afford **171** (3.0 g, 50.6%), mp. 140-143°C.

Anal.:

IR (KBr): 3300, 2830, 1640, 1580, 1520, 1500, 1420, 1340, 1230 and 1120 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ 2.78 (q, 1H, -CH_2 of oxirane), 2.93 (t, 1H, -CH_2 of oxirane), 3.73 (m, 1H, -C*H* of oxirane), 3.95 (m, 10H, -OCH_2- & 3 × -OCH_3), 4.25 (dd, 1H, -OCH_2-), 6.94 (d, 2H, J = 8.9 Hz, Ar), 7.08 (s, 2H, Ar), 7.54 (d, 2H, J = 8.8 Hz, Ar) and 7.74 ppm (s, 1H, -N*H*CO-).

Calcd for $C_{19}H_{21}NO_6$: C, 63.50; H, 5.89; N, 3.90. Found: C, 63.67; H, 5.83; N, 4.32.

N-[4-(2-HYDROXY-3-ISOPROPYLAMINOPROPOXY)PHENYL]-3,4,5-TRIMETHOXYBENZAMIDE (172)

N-(2,3-Epoxypropoxyphenyl)-3,4,5-trimethoxybenzamide (**171**) (2.50 g, 7.0 mmol) was refluxed in methanol (50 ml), added isopropylamine (5 ml) and refluxing was continued for 2 h. Completion of reaction was determined by TLC. The solvent was removed under reduced pressure and the residue was crystallized from ether-acetone mixture to afford **172** (2.5 g, 85.9%), mp. 125-128°C.

Anal.:

IR (KBr): 3280, 2920, 1640, 1585, 1515, 1455, 1405, 1335, 1235, 1125 and 810 cm^{-1}.

^1H NMR ($CDCl_3$): δ 1.10 (d, 6H, -CH(CH_3)$_2$), 2.72-2.89 (m, 3H, -CH_2NHC*H*<), 3.91 (s, 3H, -OCH_3), 3.94 (s, 6H, 2 × -OCH_3), 4.01 (m, 3H, -OCH_2C*H*(OH)-), 6.94 (d, 2H, *J* = 8.9 Hz, Ar), 7.07 (s, 2H, Ar), 7.52 (d, 2H, *J* = 8.9 Hz, Ar) and 7.63 ppm (s, 1H, -N*H*CO-).

Calcd for $C_{22}H_{30}N_2O_6$: C, 63.14; H, 7.23; N, 6.69. Found: C, 63.43; H, 7.22; N, 6.39.

N-[4-(2-HYDROXY-3-ISOPROPYLAMINOPROPOXY)PHENYL]-3,4,5-TRIMETHOXYBENZAMIDE (172) OXALATE [DPJ-782]

To a refluxing solution of **172** (1.5 g, 3.6 mmol) in methanol (50 ml), added oxalic acid (0.5 g, 3.9 mmol) and refluxing was continued for 0.5 h. The solvent was removed under reduced pressure and the residue crystallized from acetone to afford oxalate of **172** (1.5 g, 82.3%), mp. 228-231°C.

Anal.:

IR (KBr): 3320, 1640, 1620, 1580, 1510, 1230 and 1125 cm^{-1}.

^1H NMR (D_2O): δ 1.22 (d, 6H, -CH(CH_3)$_2$), 3.11 (m, 2H, -CH_2NH-), 3.36 (s, 1H, -C*H*(CH_3)$_2$), 3.64 (s, 3H, -OCH_3), 3.70 (s, 6H, 2 × -OCH_3), 3.87 (b, 2H, -OCH_2-), 4.13 (b, 1H, -C*H*(OH)-), 6.77 (d, 2H, *J* = 7.7 Hz, Ar), 6.89 (s, 2H, Ar) and 7.19 ppm (d, 2H, *J* = 7.7 Hz, Ar).

Calcd for $C_{24}H_{32}N_2O_{10}$: C, 56.69; H, 6.34; N, 5.51. Found: C, 56.77; H, 6.39; N, 5.60.

N-[4-(3-*tert*-BUTYLAMINO-2-HYDROXYPROPOXY)PHENYL]-3,4,5-TRIMETHOXYBENZAMIDE (173)

N-(2,3-Epoxypropoxyphenyl)-3,4,5-trimethoxybenzamide (**171**) (3.0 g, 8.4 mmol) was refluxed in (50 ml), added *tert*-butylamine (5 ml) and refluxing was continued for 4 h. Completion of reaction was confirmed by TLC. The solvent was removed under reduced pressure to give an oily residue **173** (3.5 g, 96.9%).

Anal.:

^{1}H NMR ($CDCl_3$): δ 1.13 (s, 9H, -C(C*H*$_3$)$_3$), 2.50 (b, 2H, -N*H*- & -O*H*, exchanged in D_2O), 2.69 (q, 1H, -C*H*$_2$NH-), 2.85 (t, 1H, -C*H*$_2$NH-), 3.86 (s, 6H, 2 × -OC*H*$_3$), 3.88 (s, 3H, -OC*H*$_3$), 3.96 (m, 3H, -OC*H*$_2$C*H*<), 6.87 (d, 2H, *J* = 8.9 Hz, Ar), 7.07 (s, 2H, Ar), 7.51 (d, 2H, *J* = 8.9 Hz, Ar) and 8.13 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

N-[4-(3-*tert*-BUTYLAMINO-2-HYDROXYPROPOXY)PHENYL]-3,4,5-TRIMETHOXYBENZAMIDE (173) OXALATE [DPJ-784]

Oxalic acid (0.75 g, 6.0 mmol) was added to a solution of **173** (1.5 g, 3.5 mmol) in refluxing methanol (50 ml) and continued refluxing for 0.5 h. Concentrated the reaction mixture and left overnight for crystallization to afford oxalate of **173** (1.1 g, 57.7%), mp. 231-232°C.

Anal.:

IR (KBr): 3380, 3160, 2980, 2780, 1660, 1620, 1590, 1510, 1235 and 1125 cm^{-1}.

^{1}H NMR ($CDCl_3$-DMSO-*d*$_6$): δ 1.35 (s, 9H, -C(C*H*$_3$)$_3$), 2.95 (t, 1H, -C*H*$_2$NH-), 3.14 (d, 1H, -C*H*$_2$NH-), 3.60-4.15 (bm, 13H, -OC*H*$_2$CH(O*H*)CH$_2$N*H*- & 3 × -OC*H*$_3$), 4.34 (bs, 1H, -C*H*(OH)-), 6.88 (d, 2H, *J* = 8.8 Hz, Ar), 7.31 (s, 2H, Ar), 7.64 (d, 2H, *J* = 8.7 Hz, Ar) and 9.94 ppm (s, 1H, -N*H*CO-).

Calcd for $C_{25}H_{34}N_2O_{10}$: C, 57.46; H, 6.56; N, 5.36. Found: C, 57.32; H, 6.55; N, 5.44.

N-{4-[2-HYDROXY-3-(4-METHYLPIPERAZIN-1-YL)PROPOXY]PHENYL}-3,4,5-TRIMETHOXYBENZAMIDE (174)

N-(2,3-Epoxypropoxyphenyl)-3,4,5-trimethoxybenzamide (**171**) (1.5 g, 4.2 mmol) and *N*-methylpiperazine (1.0 ml, 9.0 mmol) were refluxed in methanol (75 ml) for 8 h. Completion of reaction was confirmed by TLC. The solvent was removed under reduced pressure and the oily residue was dissolved in dry acetone, any insoluble portion was filtered out. The acetone portion was evaporated to give an oily residue of **174** (1.5 g, 78.2%).

Anal.:

^{1}H NMR ($CDCl_3$): δ 2.30 (s, 3H, >NCH_3), 2.60 (bm, 10H,-$CH_2N(CH_2CH_2)_2N$-), 3.88 (s, 9H, 3 × -OCH_3), 3.97 (t, 2H, -OCH_2-), 4.08 (m, 1H, -C*H*(OH)-), 6.90 (d, 2H, J = 9.0 Hz, Ar), 7.06 (s, 2H, Ar), 7.52 (d, 2H, J = 8.9 Hz, Ar) and 7.95 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

N-{4-[2-HYDROXY-3-(4-METHYLPIPERAZIN-1-YL)PROPOXY]PHENYL}-3,4,5-TRIMETHOXYBENZAMIDE (174) OXALATE [DPJ-786]

To a solution of **174** (1.0 g, 2.2 mmol) in refluxing methanol (50 ml), added oxalic acid (0.8 g, 6.3 mmol) and refluxing was continued for 0.5 h. Concentrated and left for crystallization to afford oxalate of **174** (0.43 g, 31.1%), mp. 202-205°C.

Anal.:

IR (KBr): 3470, 3425, 3380, 2930, 2525, 1630, 1590, 1225 and 1120 cm^{-1}.

^{1}H NMR (D_2O): δ 2.89 (s, 3H, >NCH_3), 3.33 (m, 2H, -CH_2N<), 3.50-3.70 (bd, 17H, -$N(CH_2CH_2)_2N$- & 3 × -OCH_3), 3.81 (b, 2H, -OCH_2-), 4.31 (b, 1H, -C*H*(OH)-), 6.69 (d, 2H, J = 7.5 Hz, Ar), 6.80 (s, 2H, Ar) and 7.14 ppm (d, 2H, J = 7.2 Hz, Ar).

Calcd for $C_{28}H_{37}N_3O_{14}$: C, 52.58; H, 5.83; N, 6.57. Found: C, 52.34; H, 5.89; N, 6.47.

N-(5-HYDROXYNAPHTHALEN-1-YL)ACETAMIDE (175)

To a suspension of 5-amino-1-naphthol (3.0 g, 18.8 mmol) in water (30 ml) was added acetic anhydride (6 ml), the reaction mixture was manually stirred and warmed on a water bath (60°C) for 5 min to get a clear solution. The reaction mixture was allowed to stand overnight and filtered to afford **175** (3.0 g, 79.1%), mp. 155-158°C. The product was used for next step without crystallization.

Anal.:

IR (KBr): 3280, 1620, 1580, 1535, 1410, 1370, 1270 and 770 cm^{-1}.

^1H NMR ($CDCl_3$-DMSO-d_6): δ 2.28 (s, 3H, -NHCOCH_3), 6.91 (d, 1H, Ar), 7.30 (t, 1H, Ar), 7.42 (m, 2H, Ar), 7.76 (d, 1H, Ar), 8.12 (d, 1H, Ar) and 8.83 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

Calcd for $C_{12}H_{11}NO_2$: C, 71.63; H, 5.51; N, 6.96. Found: C, 71.77; H, 5.22; N, 6.51.

N-[5-(2,3-EPOXYPROPOXY)NAPHTHALEN-1-YL]ACETAMIDE (176)

N-(5-Hydroxynaphthalen-1-yl)acetamide (**175**) (3.0 g, 14.9 mmol) was refluxed in epichlorohydrin (75 ml) for 0.5 h, added anhydrous potassium carbonate (5.0 g) and refluxing was continued for 5 h. Completion of reaction was confirmed by TLC. The reaction mixture was filtered and excess of epichlorohydrin removed under reduced pressure to obtain a solid residue, which was crystallized from acetone to obtain **176** (1.0 g, 26.1%), mp. 150-153°C.

Anal.:

IR (KBr): 3240, 3040, 1650, 1540, 1405, 1270, 1240, 1040 and 770 cm^{-1}.

^1H NMR ($CDCl_3$-DMSO-d_6): δ 2.27 (s, 3H, -NHCOCH_3), 2.86 (q, 1H, -CH_2 of oxirane), 2.96 (t, 1H, -CH_2 of oxirane), 3.50 (m, 1H, -C*H* of oxirane), 4.09 (dd, 1H, -OCH_2-), 4.47 (dd, 1H, -OC*H*$_2$-), 6.86 (d, 1H, Ar), 7.42 (m, 2H, Ar), 7.64 (d, 1H, Ar), 7.74 (d, 1H, Ar), 8.14 (d, 1H, Ar) and 9.46 ppm (s, 1H, -N*H*CO-).

Calcd for $C_{15}H_{15}NO_3$: C, 70.02; H, 5.88; N, 5.44. Found: C, 69.62; H, 5.61; N, 5.52.

N-[5-(2-HYDROXY-3-ISOPROPYLAMINOPROPOXY)NAPHTHALEN-1-YL]ACETAMIDE (177)

N-[5-(2,3-Epoxypropoxy)naphthalen-1-yl]acetamide (**176**) (1.0 g, 3.9 mmol) was refluxed in methanol (50 ml), added isopropylamine (10 ml) and refluxing was continued for 3 h. Completion of reaction was confirmed by TLC. The excess reagent was removed under reduced pressure. The solid residue was crystallized from acetone to afford **177** (0.8 g, 65.1%), mp. 155-158°C.

Anal.:

IR (KBr): 3415, 3270, 2925, 1655, 1545, 1415, 1270, 1040 and 770 cm^{-1}.

^{1}H NMR (CDCl$_3$-DMSO-*d*$_6$): δ 1.10 (d, 6H, -CH(C*H*$_3$)$_2$), 2.28 (s, 3H, -NHCOC*H*$_3$), 2.83 (m, 2H, -C*H*$_2$NHC*H*<), 2.98 (t, 1H, -C*H*$_2$NH-), 4.16 (m, 3H, -OC*H*$_2$C*H*<), 6.86 (d, 1H, Ar), 7.42 (m, 2H, Ar), 7.61 (d, 1H, Ar), 7.76 (d, 1H, Ar), 8.16 (d, 1H, Ar) and 9.33 ppm (s, 1H, -N*H*CO-, exchanged in D$_2$O).

Calcd for $C_{18}H_{24}N_2O_3$: C, 68.33; H, 7.65; N, 8.85. Found: C, 68.58; H, 7.40; N, 8.58.

N-[5-(2-HYDROXY-3-ISOPROPYLAMINOPROPOXY)NAPHTHALEN-1-YL]ACETAMIDE (177) OXALATE [DPJ-953]

To a solution of **177** (0.8 g, 2.5 mmol) in refluxing methanol (50 ml), added oxalic acid (0.4 g, 3.2 mmol) and refluxing was continued for 0.5 h. The solvent was removed under reduced pressure and the residue crystallized from methanol-acetone mixture to afford oxalate of **177** (0.73 g, 70.6%), mp. 177-180°C.

Anal.:

IR (KBr): 3395, 3245, 3040, 2980, 2930, 2855, 2780, 1655, 1535, 1405, 1260, 1230, 1085, 1030, 770 and 710 cm^{-1}.

^{1}H NMR (D_2O): δ 1.29 (d, 6H, -CH(C*H*$_3$)$_2$), 2.23 (s, 3H, -NHCOC*H*$_3$), 3.28 (m, 2H, -C*H*$_2$NH-), 3.44 (m, 1H, -C*H*(CH$_3$)$_2$), 4.20 (m, 2H, -OC*H*$_2$-), 4.38 (m, 1H, -C*H*(OH)-), 6.95 (t, 1H, Ar), 7.47 (m, 4H, Ar) and 8.19 ppm (d, 1H, Ar).

Calcd for $C_{20}H_{26}N_2O_7$: C, 59.10; H, 6.45; N, 6.89. Found: C, 59.22; H, 6.43; N, 6.93.

***N*-[5-(3-*tert*-BUTYLAMINO-2-HYDROXYPROPOXY)NAPHTHALEN-1-YL]ACETAMIDE (178)**

N-[5-(2,3-Epoxypropoxy)naphthalen-1-yl]acetamide (**176**) (0.94 g, 3.6 mmol) was refluxed in methanol (50 ml), added *tert*-butylamine (15 ml) and refluxing was continued for 2 h. Completion of reaction was confirmed by TLC. The excess reagent was removed under reduced pressure and the solid residue was crystallized from acetone to afford **178** (0.93 g, 77.1%), mp. 82-85°C.

Anal.:

IR (KBr): 3400, 2965, 2920, 2880, 1660, 1540, 1410, 1265, 1210, 1030 and 780 cm^{-1}.

^{1}H NMR (CDCl$_3$-DMSO-*d$_6$*): δ 1.14 (s, 9H, -C(C*H*$_3$)$_3$), 2.26 (s, 3H, -NHCOC*H*$_3$), 2.79 (dd, 1H, -C*H*$_2$NH-), 2.94 (dd, 1H, -C*H*$_2$NH-), 4.14 (m, 3H, -OC*H*$_2$C*H*<), 6.83 (d, 1H, Ar), 7.38 (m, 2H, Ar), 7.54 (d, 1H, Ar), 7.77 (d, 1H, Ar), 8.11 (d, 1H, Ar) and 8.33 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

Calcd for $C_{19}H_{26}N_2O_3$: C, 69.06; H, 7.93; N, 8.48. Found: C, 68.98; H, 8.02; N, 8.36.

N-[5-(3-*tert*-BUTYLAMINO-2-HYDROXYPROPOXY)NAPHTHALEN-1-YL]ACETAMIDE (178) OXALATE [DPJ-955]

Added oxalic acid (0.4 g, 3.2 mmol) to a solution of **178** (0.92 g, 2.8 mmol) in refluxing methanol (50 ml) and refluxing was continued for 0.5 h. The solvent was removed under reduced pressure and the residue crystallized from methanol-acetone mixture to afford oxalate of **178** (0.91 g, 77.3%), mp. 202-205°C.

Anal.:

IR (KBr): 3350, 3300, 3270, 3070, 3030, 2980, 2920, 2850, 1650, 1510, 1405, 1295, 1260, 1230 and 730 cm^{-1}.

^{1}H NMR (D_2O): δ 1.37 (s, 9H, -C(C*H*$_3$)$_3$), 2.27 (s, 3H, -NHCOC*H*$_3$), 3.29 (m, 2H, -C*H*$_2$NH-), 4.24 (m, 2H, -OC*H*$_2$-), 4.39 (m, 1H, -C*H*(OH)-), 6.99 (t, 1H, Ar), 7.50 (m, 4H, Ar) and 8.22 ppm (d, 1H, Ar).

Calcd for $C_{21}H_{28}N_2O_7$: C, 59.99; H, 6.71; N, 6.66. Found: C, 60.03; H, 6.80; N, 6.69.

N-(5-HYDROXYNAPHTHALEN-1-YL)-3,4-DIMETHOXYBENZAMIDE (179)

5-Amino-1-naphthol (1.0 g, 6.3 mmol) was refluxed in tetrahydrofuran (50 ml), and added 3,4-dimethoxybenzoyl chloride (1.0 g, 5.0 mmol) dissolved in tetrahydrofuran (10 ml) and continued refluxing for 1 h. Completion of reaction was confirmed by TLC. Removal of tetrahydrofuran under reduced pressure gave a solid residue, which was crystallized from methanol to yield **179** (1.0 g, 49.2%), mp. 242-244°C.

IR (KBr): 3515, 3255, 1635, 1580, 1495, 1350, 1260, 1210, 1125, 1015 and 770 cm^{-1}.

^{1}H NMR ($CDCl_3$-DMSO-d_6): δ 3.94 (s, 6H, 2 × -OC*H*$_3$), 6.91 (d, 1H, Ar), 6.98 (d, 1H, Ar), 7.29 (t, 1H, Ar), 7.45 (q, 2H, Ar), 7.71 (m, 3H, Ar), 8.17 (d, 1H, Ar) and 9.78 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

Calcd for $C_{19}H_{17}NO_4$: C, 70.58; H, 5.30; N, 4.33. Found: C, 70.59; H, 5.56; N, 4.66.

N-[5-(2,3-EPOXYPROPOXY)NAPHTHALEN-1-YL]-3,4-DIMETHOXYBENZAMIDE (180)

N-(5-Hydroxynaphthalen-1-yl)-3,4-dimethoxybenzamide (**179**) (1.0 g, 3.1 mmol) was refluxed in epichlorohydrin (50 ml) for 2 h, added anhydrous potassium carbonate (2.0 g), continued refluxing for 4 h. Completion of reaction was confirmed by TLC. The reaction mixture was filtered while hot to remove solid materials. The excess of epichlorohydrin was removed under vacuum to give a solid residue, which was crystallized from methanol to afford **180** (0.75 g, 63.9%), mp. 167-170°C.

Anal.:

IR (KBr): 3225, 2995, 2915, 1625, 1500, 1395, 1260, 1025 and 765 cm^{-1}.

^{1}H NMR ($CDCl_3$-DMSO-d_6): δ 2.87 (q, 1H, -CH_2 of oxirane), 2.98 (q, 1H, -CH_2 of oxirane), 3.51 (m, 1H, -C*H* of oxirane), 3.96 (s, 6H, 2 × -OCH_3), 4.12 (dd, 1H, -OCH_2-), 4.47 (dd, 1H, -OCH_2-), 6.86 (d, 1H, Ar), 6.97 (d, 1H, Ar), 7.40 (m, 1H, Ar), 7.51 (t, 1H, Ar), 7.61 (d, 1H, Ar), 7.73 (m, 3H, Ar), 8.23 (d, 1H, Ar) and 9.41 ppm (s, 1H, -N*H*CO-).

Calcd for $C_{22}H_{21}NO_5$: C, 69.64; H, 5.58; N, 3.69. Found: C, 69.75; H, 5.49; N, 3.77.

N-[5-(2-HYDROXY-3-ISOPROPYLAMINOPROPOXY)NAPHTHALEN-1-YL]-3,4-DIMETHOXYBENZAMIDE (181)

N-[5-(2,3-Epoxypropoxy)naphthalen-1-yl]-3,4-dimethoxybenzamide (**180**) (0.5 g, 1.3 mmol) was refluxed in methanol (50 ml), added isopropylamine (5 ml) and refluxing was continued for 2 h. Completion of reaction was confirmed by TLC. The excess reagent was removed under reduced pressure and the residue was crystallized from acetone-ether mixture to afford **181** (0.5 g, 86.5%), mp. 162-165°C.

Anal.:

IR (KBr): 3310, 2965, 2900, 1650, 1585, 1490, 1260, 1225, 1155, 1020 and 770 cm^{-1}.

^{1}H NMR ($CDCl_3$-DMSO-d_6): δ 1.11 (d, 6H, -C(C*H*$_3$)$_2$), 2.78-3.01 (bm, 3H, -C*H*$_2$NHC*H*<), 3.95 (s, 6H, 2 × -OC*H*$_3$), 4.18 (m, 3H, -OC*H*$_2$C*H*<), 6.88 (d, 1H, Ar), 6.98 (d, 1H, Ar), 7.39 (t, 1H, Ar), 7.48 (t, 1H, Ar), 7.69 (m, 4H, Ar), 8.24 (d, 1H, Ar) and 9.79 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

Calcd for $C_{25}H_{30}N_2O_5$: C, 68.47; H, 6.90; N, 6.39. Found: C, 68.32; H, 6.43; N, 6.83.

N-[5-(2-HYDROXY-3-ISOPROPYLAMINOPROPOXY)NAPHTHALEN-1-YL]-3,4-DIMETHOXYBENZAMIDE (181) OXALATE [DPJ-983]

Oxalic acid (0.3 g, 2.4 mmol) was added to a solution of **181** (0.75 g, 1.7 mmol) in refluxing methanol (50 ml) and refluxing was continued for 10 min. Concentrated the reaction mixture and left for crystallization to afford oxalate of **181** (0.7 g, 77.4%), mp. 251-254°C.

Anal.:

IR (KBr): 3380, 3245, 2930, 2840, 2700, 1620, 1580, 1520, 1500, 1400, 1285, 1160, 1010 and 770 cm^{-1}.

^{1}H NMR ($CDCl_3$-CF_3COOH): δ 1.49 (d, 6H, -C(C*H*$_3$)$_2$), 3.63 (bd, 2H, -C*H*$_2$NH-), 3.86 (m, 1H, -C*H*(CH$_3$)$_2$), 4.01 (s, 6H, 2 × -OC*H*$_3$-), 4.35 (q, 2H, -OC*H*$_2$-), 4.76 (bs, 1H, -C*H*(OH)-), 6.89 (d, 1H, Ar), 7.11 (d, 1H, Ar), 7.44 (m, 6H, Ar), 8.19 (d, 1H, Ar) and 8.72 ppm (s, 1H, -N*H*CO-).

Calcd for $C_{27}H_{32}N_2O_9$: C, 61.36; N, 6.10; N, 5.30. Found: C, 61.84; H, 6.20; N, 5.41.

N-[5-(3-*tert*-BUTYLAMINO-2-HYDROXYPROPOXY)NAPHTHALEN-1-YL]-3,4-DIMETHOXYBENZAMIDE (182)

N-[5-(2,3-Epoxypropoxy)naphthalen-1-yl)-3,4-dimethoxybenzamide (**180**) (1.0 g, 2.6 mmol) was refluxed in methanol (50 ml), added *tert*-butylamine (10 ml) and refluxing was continued for 2 h. Completion of reaction was confirmed by TLC.

Removed the excess reagent under reduced pressure and the residue was crystallized from acetone-ether mixture to afford **182** (1.1 g, 90.6%), mp. 148-151°C.

Anal.:

IR (KBr): 3260, 2940, 1640, 1590, 1510, 1410, 1330, 1265, 1215, 1125, 1025 and 770 cm^{-1}.

^{1}H NMR ($CDCl_3$-DMSO-d_6): δ 1.15 (s, 9H, -C(C*H*$_3$)$_3$), 2.79 (q, 1H, -C*H*$_2$NH-), 2.95 (t, 1H, -C*H*$_2$NH-), 3.03 (b, 2H, -N*H*- & -O*H*, exchanged in D_2O), 3.94 (s, 6H, 2 × -OCH_3), 4.16 (m, 3H, -OC*H*$_2$C*H*<), 6.86 (d, 1H, Ar), 6.94 (d, 1H, Ar), 7.38 (t, 1H, Ar), 7.47 (t, 1H, Ar), 7.58 (d, 1H, Ar), 7.71 (m, 3H, Ar), 8.22 (d, 1H, Ar) and 9.59 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

Calcd for $C_{26}H_{32}N_2O_5$: C, 69.00; H, 7.13; N, 6.19. Found: C, 68.81; H, 6.98; N, 6.05.

N-[5-(3-*tert*-BUTYLAMINO-2-HYDROXYPROPOXY)NAPHTHALEN-1-YL]-3,4-DIMETHOXYBENZAMIDE (182) OXALATE [DPJ-985]

To a solution of **182** (0.75 g, 1.7 mmol) in refluxing methanol (50 ml), added oxalic acid (0.3 g, 2.4 mmol) and refluxing was continued for 10 min. Concentrated the reaction mixture and left for crystallization to afford oxalate of **182** (0.45 g, 50%), mp. 257-260°C.

Anal.:

IR (KBr): 3475, 3380, 3230, 2960, 2800, 1615, 1585, 1540, 1500, 1400, 1260, 1220, 1020 and 760 cm^{-1}.

^{1}H NMR ($CDCl_3$-CF_3COOH): δ 1.52 (s, 9H, -C(C*H*$_3$)$_3$), 3.52 (bs, 2H, -C*H*$_2$NH-), 4.00 (s 3H, -OC*H*$_3$), 4.02 (s 3H, -OC*H*$_3$), 4.32 (bs, 2H, -OC*H*$_2$-), 4.72 (b, 1H, -C*H*(OH)-), 6.88 (d, 1H, Ar), 7.08 (d, 1H, Ar), 7.52 (d, 5H, Ar), 7.65 (s, 1H, Ar), 8.16 (d, 1H, Ar) and 8.58 ppm (s, 1H, -N*H*CO-).

Calcd for $C_{28}H_{34}N_2O_9$: C, 61.98; H, 6.32; N, 5.16. Found: C, 62.10; H, 6.48; N, 5.06.

METHYL 2-(4-ALLYL-2-METHOXYPHENOXY)ACETATE (186)

A mixture of 4-allyl-2-methoxyphenol (eugenol, **183**) (2.0 g, 12.2 mmol) and anhydrous potassium carbonate (2.0 g) were refluxed in anhydrous ethyl methyl ketone (50 ml) while stirring magnetically for 2 h. Methyl chloroacetate (2 ml, 22.8 mmol), catalytic amounts of potassium iodide and triethylamine were added to the reaction mixture and continued refluxing for 10 h. The solid materials were filtered and the solvent was removed under reduced pressure. The viscous oily product obtained was column chromatographed over silica gel (100-200 mesh) using chloroform as eluent to give **186** (2.0 g, 69.5%)

Anal.:

^{1}H NMR ($CDCl_3$): δ 3.33 (d, 2H, -$CH_2CH{=}CH_2$) 3.78 (s, 3H, -$COOCH_3$), 3.85 (s, 3H, ArOCH_3), 4.66 (s, 2H, -OCH_2-), 5.07 (m, 2H, -$CH_2CH{=}CH_2$), 5.94 (m, 1H, -$CH_2CH{=}CH_2$) and 6.76 ppm (m, 3H, Ar).

2-(4-ALLYL-2-METHOXYPHENOXY)-*N*-(2-DIETHYLAMINOETHYL)-ACETAMIDE (187)

Methyl 2-(4-allyl-2-methoxyphenoxy)acetate (**186**) (2.0 g, 8.5 mmol) and 2-diethylaminoethylamine (2.0 ml, 14.2 mmol) were mixed together and heated at 80°C for 8 h. TLC determined the completion of reaction. The product obtained was left on sodium carbonate solution (10%) overnight, and then extracted with chloroform (3 × 30 ml). Combined the chloroform layers, washed with water and distilled to give an oily residue **187** (1.5 g, 55.3%), which could not be crystallized and was used as such for the preparation of oxalate.

Anal.:

^{1}H NMR ($CDCl_3$): δ 0.98 (t, 6H, -N(CH_2CH_3)$_2$), 2.53 (m, 6H, -$CH_2N(CH_2CH_3)_2$), 3.37 (m, 4H, -CONHCH_2- & -$CH_2CH=CH_2$), 3.86 (s, 3H, ArOCH_3), 4.51 (s, 2H, -OCH_2-), 5.10 (m, 2H, -$CH_2CH=CH_2$), 5.93 (m, 1H, -$CH_2CH=CH_2$), 6.76 (m, 3H, Ar) and 7.42 ppm (b, 1H, -CON*H*-, exchanged in D_2O).

2-(4-ALLYL-2-METHOXYPHENOXY)-*N*-(2-DIETHYLAMINOETHYL)-ACETAMIDE (187) OXALATE [DPJ-632]

Added oxalic acid (1.0 g, 7.9 mmol) to a solution of **187** (1.0 g, 3.1 mmol) in methanol (50 ml) and refluxed for 0.5 h. The solvent was removed under reduced pressure and the product obtained was crystallized from dry acetone to afford oxalate of **187** (1.0 g, 76.5%), mp 100-103°C.

Anal.:

IR (KBr): 3511, 3412, 2652, 1679, 1516, 1226, 1143, 1033, 813 and 761 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ 1.24 (t, 6H, -N(CH_2CH_3)$_2$), 3.15 (m, 6H, -$CH_2N(CH_2CH_3)_2$), 3.27 (d, 2H, -$CH_2CH=CH_2$), 3.67 (q, 2H, -CONHCH_2-), 3.81(s, 3H, ArOCH_3), 4.45 (s, 2H, -OCH_2-), 5.05 (m, 2H, -$CH_2CH=CH_2$), 5.88 (m, 1H, -$CH_2CH=CH_2$), 6.65 (m, 2H, Ar), 6.75 (d, 1H, Ar) and 7.94 ppm (t, 1H, -CON*H*-, exchanged in D_2O).

Calcd for $C_{20}H_{30}N_2O_7$: C, 58.52; H, 7.37; N, 6.82. Found: C, 57.95; H, 7.25; N, 6.96.

METHYL 2-(2-ISOPROPYL-5-METHYLPHENOXY)ACETATE (188)

A mixture of 2-isopropyl-5-methylphenol (thymol, **160**) (2.0 g, 13.3 mmol) and anhydrous potassium carbonate (2.0 g) in anhydrous ethyl methyl ketone (50 ml) were refluxed while stirring magnetically for 2 h. Methyl chloroacetate (4 ml, 45.6 mmol), catalytic amounts of potassium iodide and triethylamine were added to the reaction mixture and continued refluxing for 10 h. The solid material was removed by filtering the reaction mixture. The solvent was removed under reduced pressure to give a viscous oily product, which was column chromatographed over silica gel (100-200 mesh) using chloroform as eluent to give **188** (2.0 g, 67.6%).

Anal.:

^{1}H NMR ($CDCl_3$): δ 1.22 (d, 2H, -CH(CH_3)$_2$), 2.29 (s, 3H, ArCH_3), 3.37 (m, 1H, -CH(CH$_3$)$_2$), 3.77 (s, 3H, -OCH_3), 4.62 (s, 2H, -OCH_2-), 6.52 (s, 1H, Ar), 6.77 (d, 1H, J = 7.7 Hz, Ar) and 7.12 ppm (d, 1H, J = 7.8 Hz, Ar).

N-(2-DIETHYLAMINOETHYL)-2-(2-ISOPROPYL-5-METHYLPHENOXY)-ACETAMIDE (189)

Methyl 2-(2-isopropyl-5-methylphenoxy)acetate (**188**) (2.0 g, 9.0 mmol) and 2-diethylaminoethylamine (2.0 ml, 14.2 mmol) were mixed together and heated at 80°C for 8 h. The completion of reaction was confirmed by TLC. The product obtained was left on sodium carbonate solution (10%) overnight, and then extracted with chloroform (3 × 30 ml). The chloroform layers were combined, washed with water and distilled to give an oily residue **189** (0.7 g, 23.6%), which could not be crystallized and was used as such for the preparation of oxalate.

Anal.:

^{1}H NMR ($CDCl_3$): δ 0.98 (t, 6H, -N(CH$_2$CH_3)$_2$), 1.23 (d, 6H, -CH(CH_3)$_2$), 2.31(s, 3H, ArCH_3), 2.55 (m, 6H, -CH_2N(CH_2CH$_3$)$_2$), 3.39 (m, 3H, -CONHCH_2- & -CH(CH$_3$)$_2$), 4.49 (s, 2H, -OCH_2-), 6.61 (s, 1H, Ar), 6.81 (d, 1H, J = 7.6 Hz, Ar), 7.13 (d, 1H, J = 7.7 Hz, Ar) and 7.36 ppm (b, 1H, -CONH-).

N-(2-DIETHYLAMINOETHYL)-2-(2-ISOPROPYL-5-METHYLPHENOXY)-ACETAMIDE (189) OXALATE [DPJ-629]

To a solution of **189** (1.0 g, 3.3 mmol) in methanol (50 ml) added oxalic acid (1.0 g, 7.9 mmol) and refluxed for 0.5 h. The solvent was removed under reduced pressure and the product obtained was crystallized from dry acetone to afford oxalate of **189** (0.7 g, 54.1%), mp 116-119°C.

Anal.:

IR (KBr): 3405, 2962, 2483, 2366, 2309, 1653, 1406, 1113, 814 and 719 cm^{-1}.

^1H NMR ($CDCl_3$): δ 1.21 (d, 6H, $-CH(CH_3)_2$) 1.30 (t, 6H, $-N(CH_2CH_3)_2$), 2.20 (s, 3H, $ArCH_3$), 3.25 (m, 7H, $-CH_2N(CH_2CH_3)_2$ & $-CH(CH_3)_2$), 3.77 (q, 2H, $-CONHCH_2-$), 4.50 (s, 2H, $-OCH_2-$), 6.58 (s, 1H, Ar), 6.81 (d, 1H, *J* = 7.7 Hz, Ar), 7.12 (d, 1H, *J* = 7.7 Hz, Ar) and 7.62 ppm (t, 1H, -CON*H*-, exchanged in D_2O).

Calcd for $C_{20}H_{32}N_2O_6$: C, 60.59; H, 8.13; N, 7.07. Found: C, 60.22; H, 8.23; N, 7.20.

METHYL 2-(NAPHTHALENE-1-YLOXY)ACETATE (190)

A mixture of 1-naphthol (5.0 g, 34.7 mmol) and anhydrous potassium carbonate (5.0 g) were refluxed in anhydrous ethyl methyl ketone (100 ml) while stirring magnetically for 2 h. Methyl chloroacetate (4 ml, 45.6 mmol), catalytic amounts of potassium iodide and triethylamine were added to the reaction mixture and continued refluxing for 12 h. The solid materials were removed by filtration. The solvent was removed under reduced pressure to give a viscous oily product which was column chromatographed over silica gel (100-200 mesh) using chloroform as eluent to give **190** (6.0 g, 80%)

Anal.:

^{1}H NMR ($CDCl_3$): δ 3.82 (s, 3H, -COOC*H*$_3$), 4.82 (s, 2H, -OC*H*$_2$-), 6.70 (d, 1H, Ar), 7.34 (t, 1H, Ar), 7.49 (m, 3H, Ar), 7.80 (m, 1H, Ar) and 8.36 ppm (q, 1H, Ar).

METHYL 2-(NAPHTHALENE-2-YLOXY)ACETATE (191)

A mixture of 2-naphthol (5.0 g, 34.7 mmol) and anhydrous potassium carbonate (5.0 g) were refluxed in anhydrous ethyl methyl ketone (100 ml) while stirring magnetically for 2 h. Methyl chloroacetate (4 ml, 45.6 mmol), catalytic amounts of potassium iodide and triethylamine were added to the reaction mixture and continued refluxing for 12 h. The reaction mixture was filtered and the solvent was removed under reduced pressure. The residue crystallized from acetone to afford **191** (5.5 g, 73.3%), mp. 70-72°C.

Anal.:

IR (KBr): 3070, 2960, 1760, 1440, 1210, 1180, 1080, 850 and 755 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ 3.83 (s, 3H, -COOC*H*$_3$), 4.76 (s, 2H, -OC*H*$_2$-), 7.07 (d, 1H, Ar), 7.24 (dd, 1H, Ar), 7.36 (m, 1H, Ar), 7.45 (m, 1H, Ar), 7.72 (d, 1H, Ar) and 7.77 ppm (d, 2H, Ar).

Calcd for $C_{13}H_{12}O_3$: C, 72.21; H, 5.59. Found: C, 72.05; H, 5.26.

METHYL 2-(5-ACETYLAMINONAPHTHALEN-1-YLOXY)ACETATE (192)

A mixture of *N*-(5-hydroxynaphthalen-1-yloxy)acetamide (**175**) (4.5 g, 22.4 mmol) and anhydrous potassium carbonate (10.0 g) were refluxed in anhydrous ethyl methyl ketone (100 ml) while stirring magnetically for 2 h. Methyl chloroacetate (5 ml, 57.0 mmol) was added to the reaction mixture and continued refluxing for 4 h. The reaction mixture was filtered and the solvent was removed under reduced pressure. The residue obtained was crystallized from a mixture of ethyl methyl ketone and acetone to afford **192** (4.0 g, 65.5%), mp. 181-184°C.

Anal.:

IR (KBr): 3265, 1740, 1650, 1530, 1410, 1230, 1085 and 775 cm^{-1}.

^1H NMR ($CDCl_3$-DMSO-d_6): δ 2.26 (s, 3H, -NHCOCH_3), 3.82 (s, 3H, -COOCH_3), 4.83 (s, 2H, -OCH_2-), 6.73 (d, 1H, Ar), 7.36 (t, 1H, Ar), 7.45 (t, 1H, Ar), 7.63 (d, 1H, Ar), 7.76 (d, 1H, Ar), 8.19 (d, 1H, Ar) and 9.16 ppm (s, 1H, -N*H*CO-, exchanged in D_2O).

Calcd for $C_{15}H_{15}NO_4$: C, 65.92; H, 5.53; N, 5.13. Found: C, 65.68; H, 5.30; N, 5.30.

N-(2-DIETHYLAMINOETHYL)-2-(NAPHTHALENE-1-YLOXY)ACETAMIDE (193)

Methyl 2-(naphthalene-1-yloxy)acetate (**190**) (2.0 g, 9.3 mmol) and 2-diethylaminoethylamine (2.0 ml, 14.2 mmol) were mixed together and heated at 80°C for 10 h. The completion of reaction was confirmed by TLC. The product obtained was left on sodium carbonate solution (10%) overnight, and then extracted with chloroform (3 × 30 ml). Combined the chloroform extracts, washed with water and distilled to give an oily residue **193** (1.5 g, 55.3%), which could not be crystallized and was used as such for the preparation of oxalate.

Anal.:

^1H NMR ($CDCl_3$): δ 0.95 (t, 6H, -N(CH_2CH_3)$_2$), 2.51 (m, 6H, -CH_2N(CH_2CH$_3$)$_2$), 3.40 (q, 2H, -CONHCH_2-), 4.63 (s, 2H, -OCH_2-), 6.73 (d, 1H, Ar), 7.31 (t, 1H, Ar), 7.47 (m, 3H, Ar), 7.61 (b, 1H, -CON*H*-), 7.78 (m, 1H, Ar) and 8.28 ppm (q, 1H, Ar).

N-(2-DIETHYLAMINOETHYL)-2-(NAPHTHALENE-1-YLOXY)ACETAMIDE (193) OXALATE [DPJ-574]

To a solution of **193** (2.0 g, 6.7 mmol) in methanol (50 ml) added oxalic acid (1.0 g, 7.9 mmol) and refluxed for 0.5 h. The solvent was removed under reduced pressure and the product obtained was crystallized from dry acetone to afford oxalate of **193** (1.2 g, 45.2%), mp 93-96°C.

IR (KBr): 3443, 3055, 2985, 2650, 1674, 1399, 1106, 794 and 716 cm^{-1}

^{1}H NMR ($CDCl_3$): δ 1.27 (t, 6H, -N(CH_2CH_3)$_2$), 3.15 (q, 4H, -N(CH_2CH_3)$_2$), 3.24 (t, 2H, -CH_2CH_2N<) 3.73 (d, 2H, -CONHC*H*$_2$-), 4.70 (s, 2H, -OC*H*$_2$-), 6.79 (d, 1H, Ar), 7.35 (t, 1H, Ar), 7.50 (m, 3H, Ar), 7.80 (q, 1H, Ar), 8.40 (q, 1H, Ar) and 8.55 ppm (s, 1H, -CON*H*- , exchanged in D_2O).

Calcd for $C_{20}H_{26}N_2O_6$: C, 61.53; H, 6.71; N, 7.17. Found: C, 61.23; H, 6.51; N, 7.47.

N-(2-DIETHYLAMINOETHYL)-2-(NAPHTHALENE-2-YLOXY)ACETAMIDE (194)

Methyl 2-(naphthalene-2-yloxy)acetate (**191**) (2.0 g, 9.3 mmol) and 2-diethylaminoethylamine (2.0 ml, 14.2 mmol) were mixed together and heated at 80°C for 8 h. The completion of reaction was confirmed by TLC. The product obtained was left on sodium carbonate solution (10%) overnight, and then extracted with chloroform (3 × 30 ml). The chloroform layers were combined, washed with water and distilled to give an oily residue **194** (0.9 g, 32.4%), which could not be crystallized and was used as such for the preparation of oxalate.

Anal.:

^{1}H NMR ($CDCl_3$): δ 0.95 (t, 6H, -N(CH_2CH_3)$_2$), 2.48 (q, 4H, -N(CH_2CH_3)$_2$), 2.55 (t, 2H, -NHCH_2CH_2N<), 3.38 (q, 2H, -CONHC*H*$_2$-), 4.61 (s, 2H -OC*H*$_2$-), 7.16 (m, 2H, Ar), 7.36 (bm, 2H, Ar, -CON*H*-), 7.45 (m, 1H, Ar) and 7.74 ppm (m, 3H, Ar).

N-(2-DIETHYLAMINOETHYL)-2-(NAPHTHALENE-2-YLOXY)ACETAMIDE (194) OXALATE [DPJ-595]

Oxalic acid (1.0 g, 7.9 mmol) was added to a solution of **194** (2.0 g, 5.1 mmol) in methanol (50 ml) and refluxed for 0.5 h. The solvent was removed under reduced pressure and the product obtained was crystallized from dry acetone to afford oxalate of **194** (1.1 g, 42.3%), mp. 114-117°C.

Anal.:

IR (KBr): 3303, 3057, 2975, 2649, 1669, 1395, 1219, 854 and 717 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ 1.24 (t, 6H, $-N(CH_2CH_3)_2$), 3.17 (m, 6H, $-CH_2N(CH_2CH_3)_2$), 3.64 (d, 2H, $-CONHCH_2-$), 4.63 (s, 2H, $-OCH_2-$), 7.20 (d, 1H, Ar), 7.27 (dd, 1H, Ar), 7.35 (t, 1H, Ar), 7.44 (t, 1H, Ar), 7.77 (t, 3H, Ar) and 8.64 ppm (s, 1H, $-CONH-$).

Calcd for $C_{20}H_{26}N_2O_6$: C, 61.53; H, 6.71; N, 7.17. Found: C, 61.20; H, 6.96; N, 7.57.

2-(5-ACETYLAMINONAPHTHALEN-1-YLOXY)-*N*-(2-DIETHYLAMINOETHYL)-ACETAMIDE (195)

Methyl 2-(5-acetylaminonaphthalen-1-yloxy)acetate (**192**) (1.0 g, 3.7 mmol) and 2-diethylaminoethylamine (1.0 ml, 7.1 mmol) were mixed together and heated at 100°C for 6 h. The completion of reaction was confirmed by TLC. Dissolved the solid residue in acetone, filtered of any insoluble material and crystallized from acetone-ether mixture to afford **195** (0.5 g, 38.2%), mp. 134-137°C.

Anal.:

IR (KBr): 3280, 2970, 2930, 1655, 1505, 1410, 1265, 1055 and 780 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ 0.98 (t, 6H, $-N(CH_2CH_3)_2$), 2.32 (s, 2H, $-NHCOCH_3$), 2.57 (m, 6H, $-CH_2N(CH_2CH_3)_2$), 3.44 (q, 2H, $-CONHCH_2-$), 4.67 (s, 2H, $-OCH_2-$), 6.82

(d, 1H, Ar), 7.49 (m, 5H, Ar, -CON*H*-, one proton exchanged in D_2O), 7.96 (d, 1H, Ar) and 8.19 ppm (d, 1H, Ar).

Calcd for $C_{20}H_{27}N_3O_3$: C, 67.20; H, 7.61; N, 11.76. Found: C, 67.37; H, 8.12; N, 11.66.

2-(5-ACETYLAMINONAPHTHALEN-1-YLOXY)-*N*-(2-DIETHYLAMINOETHYL)-ACETAMIDE (195) OXALATE [DPJ-973]

To a solution of **195** (0.5 g, 1.4 mmol) in methanol (50 ml), added oxalic acid (0.2 g, 1.6 mmol) and refluxed for 10 min. The solvent was removed under reduced pressure and the product obtained was crystallized from methanol-acetone mixture to afford oxalate of **195** (0.4 g, 63.9%), mp. 148-151°C.

Anal.:

IR (KBr): 3380, 3285, 1660, 1525, 1405, 1365, 1225 and 790 cm^{-1}.

^1H NMR (D_2O): δ 1.18 (t, 6H, -N(CH_2CH_3)$_2$), 2.27 (s, 3H, -NHCOC*H*$_3$), 3.15 (q, 4H, - N(C*H*$_2$CH$_3$)$_2$), 3.25 (t, 2H, -NHCH$_2$C*H*$_2$N<), 3.62 (t, 2H, -CONHC*H*$_2$-), 4.77 (s, 2H, -OC*H*$_2$-), 6.88 (d, 1H, Ar), 7.49 (m, 4H, Ar) and 8.26 ppm (d, 1H, Ar),

Calcd for $C_{22}H_{29}N_3O_7$: C, 59.05; H, 6.53; N, 9.39. Found: C, 58.95; H, 6.12; N, 9.23.

2-(4-ALLYL-2-METHOXYPHENOXY)-*N*-[2-(3,4-DIMETHOXYPHENYL)ETHYL]-ACETAMIDE (196) [DPJ-844]

Methyl 2-(4-allyl-2-methoxyphenoxy)acetate (**186**) (1.8 g, 7.6 mmol) and homoveratrylamine (2 ml, 11.9 mmol) were mixed thoroughly and left at room temperature for 24 h to give a solid product. The solid residue was given washings several times with hexane to remove unreacted homoveratrylamine. The solid residue was column chromatographed over silica gel (100-200 mesh) using chloroform as eluent. The product was then crystallized from acetone-ether mixture to give **196** (1.1 g, 37.5%), mp. 94-96°C.

Anal.:

IR (KBr): 3330, 2960, 2930, 1655, 1510, 1265, 1230, 1140, 1020 and 805 cm^{-1}.

^1H NMR ($CDCl_3$): δ 2.79 (t, 2H, $-NHCH_2CH_2-$), 3.34 (d, 2H, $-CH_2CH=CH_2$), 3.58 (q, 2H, $-NHCH_2CH_2-$), 3.77 (s, 3H, $-OCH_3$), 3.82 (s, 3H, $-OCH_3$), 3.85 (s, 3H, $-OCH_3$), 4.49 (s, 2H, $-OCH_2-$), 5.09 (m, 2H, $-CH_2CH=CH_2$), 5.95 (m, 1H, $-CH_2CH=CH_2$), 6.71 (m, 4H, Ar), 6.78 (dd, 2H, Ar) and 7.07 ppm (b, 1H, -CON*H*-).

Calcd for $C_{22}H_{27}NO_5$: C, 68.55; H, 7.06; N, 3.64. Found: C, 68.46; H, 7.00; N, 3.88.

N-[2-(3,4-DIMETHOXYPHENYL)ETHYL]-2-(NAPHTHALENE-1-YLOXY)-ACETAMIDE (197) [DPJ-835]

Methyl 2-(naphthalene-1-yloxy)acetate (**190**) (1.0 g, 4.6 mmol) and homoveratrylamine (1 ml, 5.9 mmol) were mixed thoroughly and left at room temperature for 24 h. The oily residue was given washings several times with hexane till a solid residue was obtained to remove unreacted excess of homoveratrylamine. The solid residue was column chromatographed over silica gel (100-200 mesh) using chloroform as eluent. The product was then crystallized from methanol to afford **197** (0.5 g, 29.6%), mp. 106-109°C.

Anal.:

IR (KBr): 3300, 1660, 1550, 1510, 1260, 1230, 1020 and 765 cm^{-1}.

^1H NMR ($CDCl_3$): δ 2.79 (t, 2H, $-NHCH_2CH_2-$), 3.65 (q, 2H, $-NHCH_2CH_2-$), 3.80 (s, 3H, $-OCH_3$), 3.81 (s, 3H, $-OCH_3$), 4.66 (s, 2H, $-OCH_2-$), 6.67 (m, 5H, Ar, -CON*H*-), 7.35 (t, 1H, Ar), 7.49 (m, 3H, Ar), 7.82 (d, 1H, Ar) and 7.91 ppm (d, 1H, Ar).

Calcd for $C_{22}H_{23}NO_4$: C, 72.31; H, 6.34; N, 3.84. Found: C, 72.17; H, 6.25; N, 3.63.

N-[2-(3,4-DIMETHOXYPHENYL)ETHYL]-2-(NAPHTHALENE-2-YLOXY)-ACETAMIDE (198) [DPJ-843]

Methyl 2-(naphthalene-2-yloxy)acetate (**191**) (1.0 g, 4.6 mmol) and homoveratrylamine (2 ml, 11.9 mmol) were mixed thoroughly and left at room temperature for 24 h. The oily residue was given washings several times with hexane till all of homoveratrylamine is removed. The solid residue was crystallized from methanol to give **198** (1.1 g, 62.1%), mp. 132-135°C.

Anal.:

IR (KBr): 3370, 2940, 1655, 1535, 1510, 1260, 1230, 1140 and 835 cm^{-1}

^{1}H NMR ($CDCl_3$): δ 2.79 (t, 2H, $-NHCH_2CH_2-$), 3.60 (q, 2H, $-NHCH_2CH_2-$), 3.81 (s, 3H, $-OCH_3$), 3.82 (s, 3H, $-OCH_3$), 4.59 (s, 2H, $-OCH_2-$), 6.67 (m, 4H, Ar), 7.08 (m, 2H, Ar), 7.37 (m, 1H, Ar), 7.47 (m, 1H, Ar) and 7.75 ppm (m, 3H, Ar).

Calcd for $C_{22}H_{23}NO_4$: C, 72.31; H, 6.34; N, 3.84. Found: C, 72.81; H, 6.35; N, 4.09.

METHYL 2-[4-(3,4,5-TRIMETHOXYBENZAMIDO)PHENOXY]ACETATE (199)

A mixture of *N*-(4-hydroxyphenyl)-3,4,5-trimethoxybenzamide (**170**) (2.0 g, 6.6 mmol) and anhydrous potassium carbonate (4.0 g) were refluxed in anhydrous ethyl methyl ketone (100 ml) while stirring for 2 h. Methyl chloroacetate (2 ml, 22.8 mmol), catalytic amounts of potassium iodide and triethylamine were added to the reaction mixture and continued refluxing with stirring for 6 h. Completion of reaction was confirmed by TLC. The reaction mixture was filtered and the solvent was removed under reduced pressure to obtain a solid residue, which was crystallized from acetone-ether mixture to afford **199** (1.8 g, 72.7%), mp. 140-141ºC.

Anal.:

IR (KBr): 3440, 3270, 2940, 1755, 1640, 1590, 1505, 1230, 1130, 1070 and 810 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ 3.82 (s, 3H, -COOC*H*$_3$), 3.90 (s, 3H, -OC*H*$_3$), 3.92 (s, 6H, 2 × -OC*H*$_3$), 4.65 (s, 2H, -OC*H*$_2$-), 7.07 (s, 2H, Ar), 6.92 (d, 2H, Ar), 7.55 (d, 2H, Ar) and 7.79 ppm (s, 1H, -N*H*CO-).

Calcd for $C_{19}H_{21}NO_7$: C, 60.79; H, 5.64; N, 3.73. Found: C, 60.89; H, 5.51; N, 3.81.

N-[2-(3,4-DIMETHOXYPHENYL)ETHYL]-2-[4-(3,4,5-TRIMETHOXYBENZAMIDO)PHENOXY]ACETAMIDE (200) [DPJ-848]

Methyl 2-[4-(3,4,5-trimethoxybenzamido)phenoxy]acetate (**199**) (1.0 g, 2.7 mmol) and homoveratrylamine (1.5 ml, 8.9 mmol) were mixed thoroughly by warming on a water bath, and left at room temperature for 24 h. The solid material was given washings several times with hexane to remove excess of homoveratrylamine. The solid residue was crystallized from methanol to afford **200** (1.2 g, 85.9%), mp. 144-146°C.

Anal.:

IR (KBr): 3440, 3280, 1645,1560, 1500, 1230, 1120, 815 and 785 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ 2.78 (t, 2H, -NHCH$_2$C*H*$_2$-), 3.56 (q, 2H, -NHC*H*$_2$CH$_2$-), 3.82 (s, 3H, -OC*H*$_3$), 3.85 (s, 3H, -OC*H*$_3$), 3.90 (s, 9H, 3 × -OC*H*$_3$), 4.44 (s, 2H, -OC*H*$_2$-), 6.61 (t, 1H, -CON*H*-), 6.69 (m, 2H, Ar), 6.80 (m, 3H, Ar), 7.12 (s, 2H, Ar), 7.53 (dd, 2H, Ar) and 8.09 ppm (s, 1H, ArN*H*CO-, exchanged in D_2O).

Calcd for $C_{28}H_{32}N_2O_8$: C, 64.11; H, 6.15; N, 5.34. Found: C, 63.89; H, 6.25; N, 5.22.