Electronic Supplementary Material (ESI) for Inorganic Chemistry Frontiers

Supplementary Information

Synthesis and Characterization of Nano-ZnS particles:

Nano-ZnS particles were prepared by simple, economical and efficient chemical bath deposition method. In a typical experiment, zinc acetate (8 mmol) and thiourea (8 mmol) were dissolved in ethylene glycol (100 mL) under ultrasonic and added NaOH solution slowly to fix the pH value about ~ 10. Then, PVP (5 g) is added to the above solution to stop the further growth of nano particles and protect the nano-ZnS particles from oxidation due to atmospheric air, and then the transparent solution was heated to 120 °C and maintained at this temperature for 1 h under Continuous stirring. The product obtained was milky white in color, which was separated from the reaction mixture by centrifugation and washed with ethanol several times. Then the product was treated with 0.1 M HNO_3 for 5 minutes and was dried in vacuum oven for characterization. The morphological, Structural and chemical composition of ZnS nano particles were analyzed with SEM –EDX (JEOL JSM-6610-LV- with OXFORD EDS), and XRD (PANalytical: XPERT-PRO) equipment.

Experimental Section:

The study was initiated with an investigation of nano - ZnS particles as catalyst in the synthesis of xanthenes via reaction of aromatic aldehyde with dimidone (scheme 1) and aromatic aldehyde with β-naphathol (Scheme 2) at 90 ^{o}C without using any hazard organic solvents. To a solution of aromatic aldehyde (1 m mol) and dimidone (2 m mol) in ethanol (10 mL) ,nano-ZnS particles catalyst (0.05 mmol) was added. The mixture was refluxed for 20 min. After completion of the reaction (the progress of the reaction was monitored by TLC using *n*-hexane: ethyl acetate as eluent), the mixture was cooled and the solid residue was separated and dissolved in dichloromethane. The solution was filtered and solid nano-ZnS particles catalyst was isolated

and could be reused. The organic phase was evaporated and the reaction mixture was recrystallized in ethanol to give pure product. All the products are known compounds.

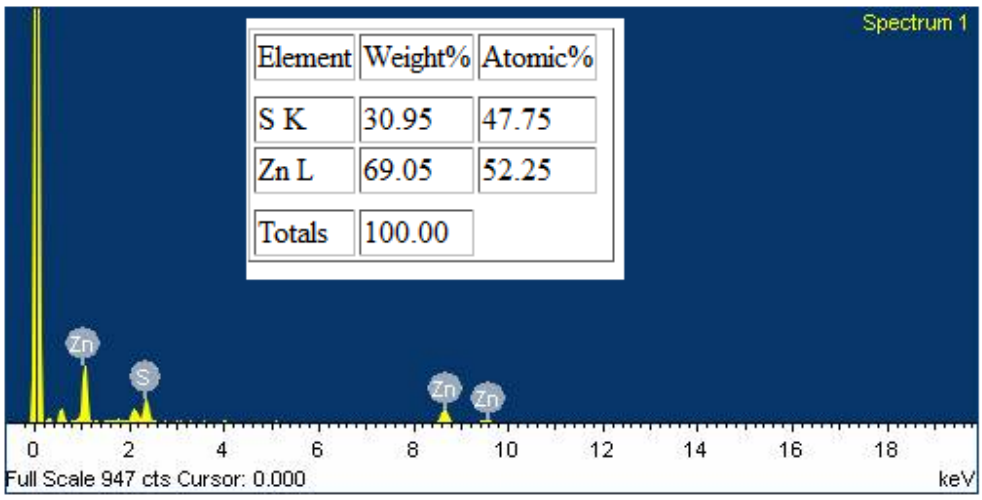
General procedure for the synthesis of 1, 8-dioxo-octahydroxanthenes:

A mixture of 1 mmol of aldehyde, 2 mmol of dimidone and ZnS (10-20mol %) was heated at 90 ^{0}C. The mixture was refluxed for an appropriate time**.** The progress of the reaction was monitored by TLC. After completion of the reaction, the catalyst was separated by simple filtration. Then solvent was evaporated and the solid was crystallized by ethanol.

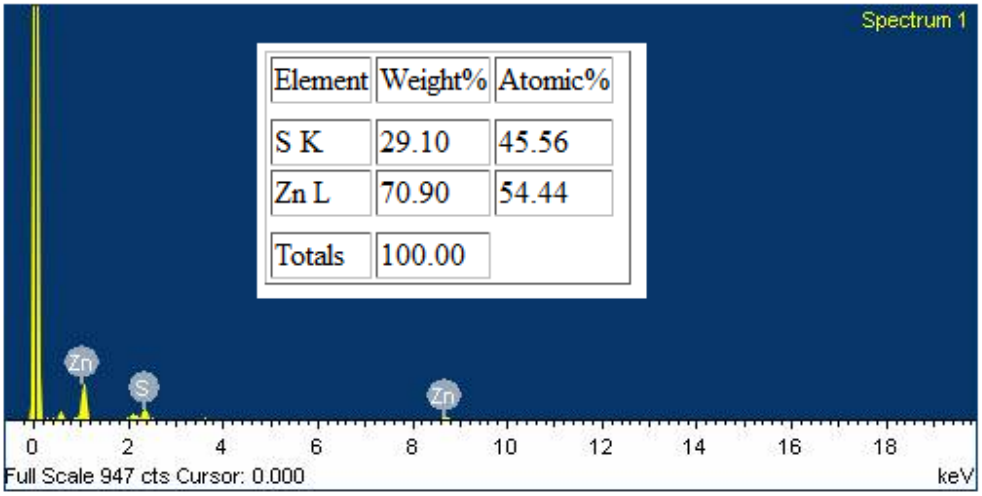
General Procedure for the Synthesis of Aryl -14H-dibenzo [a, j] xanthenes:

A mixture of 2-naphthol (2 mmol), aldehyde (1 mmol) and nano-ZnS (10-20 mol %) was heated at 90 °C. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was washed with EtOAc and filtered to recover the catalyst. The filtrate was evaporated and the crude products were purified by recrystallization with ethyl alcohol. To afford the pure 14-aryl -14Hdibenzo [a, j] xanthenes derivatives in 80-96% yields.

EDS analysis of nano-ZnS catalyst before and after 0.1M HNO_3 treatment:



1 **Before treatment**

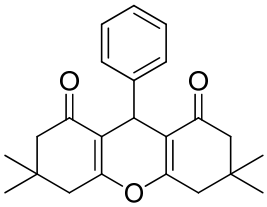


2 **After treatment**

Representative Spectral data and corresponding NMR, IR and Mass Spectroscopy:

Table-3; Entry-1

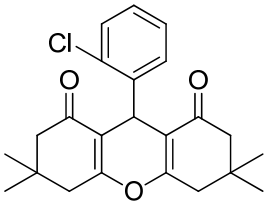
3,3,6,6-tetramethyl-9-phenyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



White solid, m.p.: 203-204 ^{0}C; IR (KBr) (υ_{max},cm^{-1}): 3350, 2980, 1795, 1725, 1699, 1640, 1520, 1360, 1345, 1260, 1233, 1201, 1195, 850, 843; ^{1}H NMR (500MHz, $CDCl_3$),: 1.02 (s, 6H), 1.13 (s, 6H), 2.19 (d,2H, J = 16.2 Hz), 2.26 (d, 2H, J = 16.2 Hz), 2.50 (s, 4H),4.78 (s, 1H), 7.12 (t, 1H, J = 7.2 Hz), 7.24 (t, 2H, J = 7.5Hz), 7.32 (d, 2H, J = 7.6Hz); ^{13}C NMR ($CDCl_3$, 125MHz): 27.75, 29.69, 32.26, 32.61, 41.29, 51.18, 116.07, 126.76, 128.45, 128.80, 144.54, 162.70, 196.76; MS (*m/z*): 350(M^+), 273, 217, 77; Elemental analysis: Found (%): C,78.63; H, 7.32; Calcd. for $C_{23}H_{26}O_3$ (350.54): C, 78.83, H,7.48.

Table-3; Entry-2

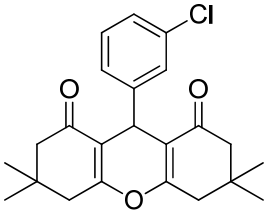
9-(2-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



White solid, m.p:25-227 ^{0}C; IR (K Br) : ($\upsilon_{max,}$cm^{-1})3392, 3064, 2960, 2929, 1719, 1664, 1595, 1380, 1166; ^{1}H NMR(500 MHz, $CDCl_3$) : 1.05 (s, 6H), 1.13 (s, 6H), 2.19 (d,2H, *J* = 16.2 Hz), 2.26 (d, 2H, *J* = 16.2 Hz), 2.48 (s, 4H),5.03 (s, 1H), 7.09 (dt, 1H, *J*1 = 7.6Hz, *J*2 = 1.6 Hz), 7.19 (dt, 1H, *J*1 = 7.6Hz, *J*2 = 1.1 Hz), 7.26 (dd, 1H, *J*1 = 7.9Hz, *J*2 =1.0 Hz),), 7.46 (d, 1H, *J* = 7.3 Hz); ^{13}C NMR (125MHz, $CDCl_3$): 27.79, 29.69, 32.28, 32.43, 41.25, 51.14, 114.13, 126.74, 128.20, 130.56, 133.34, 133.88, 140.32, 163.37, 196.84. LC-MS: 406 [M+23]; Elemental analysis: Found (%): C, 71.66; H, 6.41; Calcd. for $C_{23}H_{25}ClO_3$(384.15): C, 71.77; H, 6.55.

Table-3; Entry-3

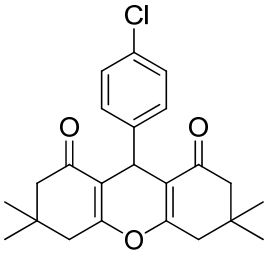
9-(3-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



m.p.:184-186 ^{0}C;^{1}H NMR (500 MHz, $CDCl_3$): 0.99 (s, 6H), 1.09 (s, 6H), 2.17 (d,2H, *J* = 16.2 Hz), 2.22 (d, 2H, *J* = 16.2 Hz), 2.46 (s, 4H),4.71 (s, 1H), 7.06 (dt, 1H, *J*1 = 9.1Hz, *J*2 = 1.5 Hz), 7.13 (t,1H, *J* = 7.9 Hz), 7.21 (d, 1H, *J* = 1.2 Hz), 7.23 (t, 1H, *J* = 1.3Hz); ^{13}C NMR (125 MHz, $CDCl_3$): 27.79, 29.62, 32.16,32.63, 41.27, 51.13, 115.51, 127.05, 127.40, 128.75,129.65, 134.28, 146.54, 162.99, 196.67.

Table-3; Entry-4

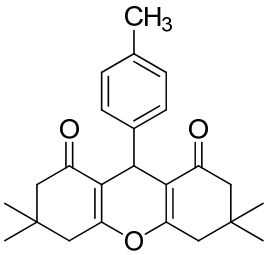
9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



White solid, m.p.: 230-232 ^{0}C; ^{1}H NMR(500 MHz, $CDCl_3$) : 1.03 (s, 6H), 1.14 (s, 6H), 2.20 (d,2H, *J* = 16.3 Hz), 2.27 (d, 2H, *J* = 16.3 Hz), 2.50 (s, 4H),4.75 (s, 1H), 7.22 (d, 2H, *J* = 8.5 Hz), 7.27 (d, 2H, *J* = 8.5Hz); ^{13}C NMR (125 MHz, $CDCl_3$),: 27.72, 29.68, 31.89,32.61, 41.28, 51.13, 115.69, 128.63, 130.19, 132.45,143.13, 162.83, 196.71.

Table-3; Entry-5

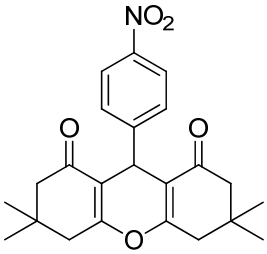
3,3,6,6-tetramethyl-9-(p-tolyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



White solid , m.p. : 217-218°C; IR (K Br) ($\upsilon_{max,}cm^{-1}$): 3143, 2954, 1720, 1587, 1375, 1199; ^{1}H NMR (500 MHz, $CDCl_3$): δH 1.00 (6H, s, CMe_2); 1.09 (6H, s, CMe_2);2.20 (4H, d, J = 3.43 Hz, 2 × CH_2); 2.24 (3H, s, CH_3); 2.45(4H, s, 2 × CH_2); 4.70 (1H, s, CH); 7.00 (2H, s, J = 8.1 Hz,ArH); 7.15 (2H, d, J = 8.1 Hz);LC-MS: 387 [M^{+}23]; Elemental analysis: Found (%): C, 79.23; H, 7.262; Calcd. for $C_{24}H_{28}O_3$ (364.48): C, 79.09; H, 7.74.

Table-3; Entry-6

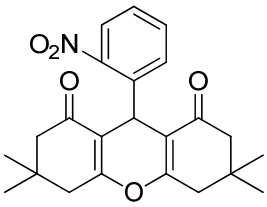
3,3,6,6-tetramethyl-9-(4-nitrophenyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



m.p. : 222-223 ^{0}C, ^{1}H NMR(500 MHz, $CDCl_3$): 1.02 (s, 6H), 1.15 (s, 6H), 2.20 (d,2H, *J* = 16.3 Hz), 2.29 (d, 2H, *J* = 16.3 Hz), 2.53 (s, 4H),4.86 (s, 1H), 7.51 (dd, 2H, *J*1 = 7.0 Hz, *J*2 = 1.7 Hz), 8.12(dd, 2H, *J*1= 7.0 Hz, *J*2 = 1.7 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) : 27.70, 29.64, 32.64, 32.79, 41.27, 51.03, 114.96,123.83, 129.78, 146.92, 151.94, 163.36, 196.63

Table-3; Entry-7

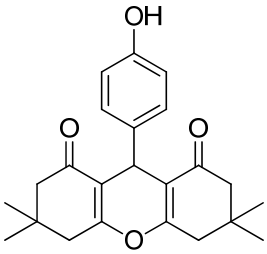
3,3,6,6-tetramethyl-9-(2-nitrophenyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



White solid, m.p.: 258-262 °C; IR (K Br) ($\upsilon_{max,}cm^{-1}$) :3095, 2932, 2920, 1707, 1643, 1556, 1366. ^{1}H NMR ($CDCl_3$): δH 1.00 (6H, s,CMe_2); 1.11 (6H, s, CMe_2); 2.05-2.25 (4H, q, J = 16.2 Hz, 2 ×CH_2); 2.47 (4H, s, 2 × CH_2); 5.48 (1H, s, CH); 7.27-7.78 (4H,m, ArH); ^{13}C NMR(125 MHz, $CDCl_3$),: 28.00, 29.16, 29.36, 32.48, 41.26, 51.04, 114.60, 125.03, 127.59,131.46, 132.40, 138.46, 150.27, 163.44, 196.73; LC-MS: 418 [M^{+}23]; Elemental analysis: Found(%): C, 69.67; H, 6.23; Calcd. for $C_{23}H_{25}NO_5$ (395.45): C,69.86; H, 6.37.

Table-3; Entry-8

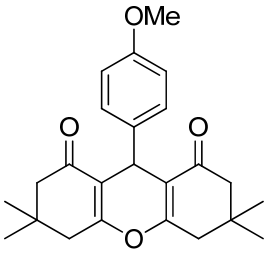
9-(4-hydroxyphenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



White solid, m.p.: 250-251 °C, IR (KBr) ($\upsilon_{max,}cm^{-1}$) : 3498, 3078, 2933,2929, 1719, 1645, 1577, 1384, 1166.^{1}H NMR (500 MHz, $CDCl_3$): δH1.00 (6H, s, CMe_2); 1.11 (6H, s, CMe_2); 2.12-2.24 (4H, q, J =16.6 Hz, 2 × CH_2); 2.45 (4H, s, 2 × CH_2); 4.61 (1H, s, CH);6.77 (2H, m, ArH.); 6.93 (2H, m,ArH); LC-MS: 389 [M^{+}23]; Elemental analysis: Found (%): C, 75.21; H, 7.20; Calcd. for$C_{23}H_{26}O_4$ (366.18): C, 75.38; H, 7.15.

Table-3; Entry-9

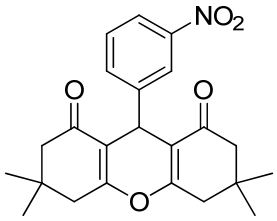
9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



Colorless solid; m.p.: 241–243 ^{0}C, IR(K Br) ($\upsilon_{max,}$cm^{-1}): 3014, 2958, 2873,1891, 1667, 1624, 1510, 1360, 1215; ^{1}H NMR: (200 MHz, $CHCl_3$): d 0.99 (s, 6H), 1.10 (s, 6H), 2.19–2.21 (d,J = 3.43 Hz, 4H), 2.45 (s, 4H), 3.73 (s, 3H), 4.70 (s, 1H),6.73 (d, J = 8.71 Hz, 2H), 7.18 (d, J = 8.71 Hz, 2H);); ^{13}C NMR(125 MHz, $CDCl_3$),: 27.76, 29.69, 31.38, 32.61, 41.29,51.20, 55.52, 113.89, 116.21, 129.73, 136.98, 158.38,162.48, 196.86. LC–MS: 403 [M^{+}23].

Table-3; Entry-10

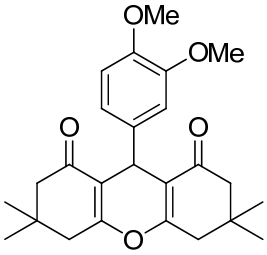
3,3,6,6-tetramethyl-9-(3-nitrophenyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



White solid, m.p.: 170-172 ^{o}C; IR(KBr) ($\upsilon_{max,}$cm^{-1}): 2960, 2873, 1680, 1675, 1593, 1529, 1377, 1311,1251, 1157, 1042, 842, 763, 732, 665; ^{1}H NMR(500 MHz, $CDCl_3$,) : 0.98 (s, 6H), 1.10 (s, 6H), 2.15 (d,2H, *J* = 16.3 Hz), 2.24 (d, 2H, *J* = 16.3 Hz), 2.49 (s, 4H),4.82 (s, 1H), 7.38 (t, 1H, *J* = 7.9 Hz), 7.79 (d, 1H, *J* = 7.7Hz), 7.96 (dd, 1H, *J*1 = 8.0Hz, *J*2 = 1.9 Hz), 8.02 (t, 1H, *J* =1.9 Hz); ^{13}C NMR (125 MHz,$CDCl_3$,): 27.72, 29.61,32.52, 32.66, 41.23, 51.06, 114.96, 122.06, 123.02,129.21, 136.07, 146.74, 148.73, 163.46, 196.76 LC–MS: 417 [M^{+}23].

Table-3; Entry-11

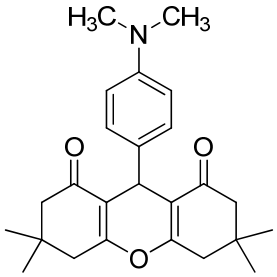
9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



m.p.: 179-181 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.00 (s, 6H, 2 x CH_3), 1.10 (s, 6H, 2 x CH_3), 2.20 (q, J=7.6Hz, 4H, 2 x CH_2), 2.46 (s, 4H, 2 x CH_2), 3.79 (s, 3H, OCH_3), 3.85 (s,3H, OCH_3), 4.70 (s, 1H, CH), 6.70–6.77 (m, 2H, Ar), 6.90 (s, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 27.26, 29.34, 31.22, 32.19, 40.89, 50.75, 55.75, 55.88, 110.83, 112.29, 115.76, 120.11, 136.98, 147.46, 148.45, 162.11, 196.5.

Table-3; Entry-12

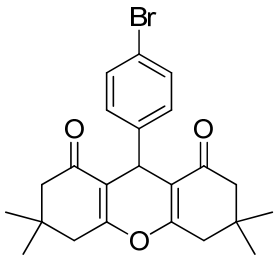
9-(4-(dimethylamino)phenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione



Yellow solid, m.p.: 222-225 °C; IR (K Br) ($\upsilon_{max,}cm^{-1}$): 3087, 3010,2923, 2901, 1709, 1643, 1573, 1376, 1133; ^{1}H NMR ($CDCl_3$) : δH 1.01 (6H, s, CMe_2); 1.11 (6H, s, CMe_2); 2.11-2.22 (4H, q, J = 16.05 Hz, 2 × CH_2); 2.42 (4H, s, 2 × CH_2); 4.61 (1H, s,CH); 7.13 (2H, m, ArH.); 7.25 (2H, m, ArH);LC-MS: 418[M^{+}23]; Elemental analysis: Found (%): C, 76.21; H, 7.90;Calcd. for $C_{23}H_{31}NO_3$ (393.52): C, 76.30; H, 7.94.

Table-3; Entry-13

9-(4-bromophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione

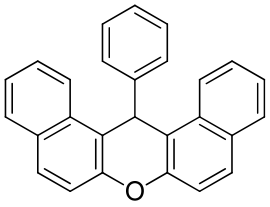


White solid, m.p.: 240-241 °C; IR (KBr) ($\upsilon_{max,}cm^{-1}$): 3078, 2946, 2910, 1701, 1623, 1545, 1376; ^{1}H NMR (500 MHz, $CDCl_3$,): 1.03 (s, 6H), 1.14 (s, 6H), 2.20 (d, 2H, *J* = 16.3 Hz), 2.27 (d, 2H, *J* = 16.3 Hz), 2.50 (s, 4H), 4.74 (s, 1H), 7.21 (d, 2H, *J* = 8.4 Hz), 7.37 (d, 2H, *J* = 8.4Hz); ^{13}C NMR (125 MHz, $CDCl_3$),: 27.73, 29.69, 31.98,32.62, 41.28, 51.12, 115.63, 120.66, 130.60,

131.57,143.64, 162.82, 196.69 .MS (ESI) m/z 429($[M^+H]$)$^+$. Anal. Calcd for C_{23} H_{25} Br O_3: C, 64.34; H, 5.87; Found: C, 64.33, H, 5.87.

Table-4; Entry-1

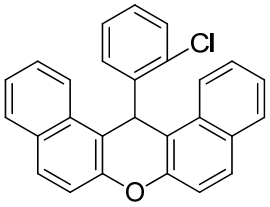
14-phenyl-14H-dibenzo[a,j]xanthene



Pale yellow solid, m.p. : 182-183 ^{O}C; IR (K Br) ($\upsilon_{max,}cm^{-1}$) : 3068, 3020, 2885, 1620,1590, 1512, 1488, 1457, 1402, 1252, 1080, 1025, 965, 825,745, 700; ^{1}H NMR($CDCl_3$, 400 MHz): 8.40 (d, *J* = 8.4 Hz, 2H), 7.82 (d, *J* = 7.9 Hz,2H), 7.79 (d, *J* = 8.8 Hz, 2H), 7.58 (t, *J* = 7.7 Hz, 2H), 7.53(d, *J* = 7.5 Hz, 2H), 7.49 (d, *J* = 8.8 Hz, 2H), 7.41 (t, *J* = 7.5Hz, 2H), 7.15 (t, *J* = 7.5 Hz, 2H), 7.00 (t, *J* = 7.5 Hz, 1H),6.49 (s, 1H); ^{13}C NMR: 148.7, 145.0, 131.3, 131.0,128.8, 128.5, 128.1, 126.8, 126.5, 126.3, 124.2, 122.6,117.9, 117.4, 38.2; ESI-MS (*m*/*z*): 358 $[M^+H]$ $^+$; Anal. Calcd for $C_{27}H_{18}O$: C, 90.47; H, 5.06.Found: C, 90.42; H, 5.08

Table-4; Entry-2

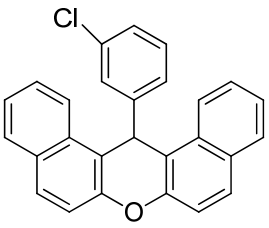
14-(2-chlorophenyl)-14H-dibenzo[a,j]xanthenes



White crystals, m.p. : 216-218^{O}C; IR (K Br) ($\upsilon_{max,}cm^{-1}$): 3056, 1620, 1592, 1514, 1459, 1429, 1402, 1254, 1032, 964, 828, 810, 749, 739 ; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.85 (s, 1 H), 6.93-6.99 (m, 2 H), 7.30 (d, *J*= 7.1 Hz, 1 H), 7.44 (d,*J*= 7.6 Hz, 1 H), 7.48 (t, *J*= 7.5 Hz, 2 H), 7.54 (d, *J*= 8.8 Hz, 2 H), 7.67 (t, *J*= 7.5 Hz, 2 H), 7.84 (d, *J*= 8.9 Hz, 2 H), 7.87 (d, *J*= 8.0 Hz, 2 H), 8.79 (d, *J*= 8.5 Hz, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$):,116.9, 118.2, 123.3, 124.9, 127.4, 128.5, 128.8, 129.2, 129.8, 130.2, 130.3, 130.9, 131.4,132.0, 143.2, 148.7.ESI-MS (*m*/*z*): 392 $[M^+H]^+$; Anal. Calcd for $C_{27}H_{17}ClO$: C, 82.54; H, 4.36.Found: C, 82-51; H, 4.32

Table-4; Entry-3

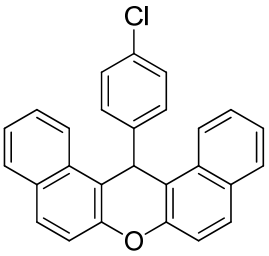
14-(3-chlorophenyl)-14H-dibenzo[a,j]xanthenes



Brown solid, m.p.: 172-173 ^{0}C, IR (K Br) ($\upsilon_{max,}$cm^{-1}): 3067, 1621,1590, 1572, 1514, 1455, 1430, 1397, 1245, 1065, 959, 811, 755,746.; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.51 (s, 1 H), 7.02 (d, *J*= 8.4 Hz, 1 H), 7.12 (t, *J*= 7.6 Hz, 1 H), 7.46-7.50 (m, 4 H), 7.53 (d, *J*= 8.9 Hz, 2 H), 7.65 (t, *J*= 7.1 Hz, 2 H), 7.85 (d, *J*= 8.9 Hz, 2 H), 7.89 (d, *J*= 8.0 Hz, 2 H), 8.37 (d, *J*= 8.5 Hz, 2 H). ^{13}C NMR: 148.5, 146.8, 134.5, 131.2, 131.0, 129.7, 129.1, 128.8, 128.2, 127.1, 126.8, 126.4, 124.5, 122.4, 118.1, 116.4, 37.8;EI-MS:*m/z* (%) = 392 (M^{+}); Anal. Calcd for $C_{27}H_{17}ClO$: C, 82.54; H, 4.36. Found: C, 82.48; H, 4.42.

Table-4; Entry-4

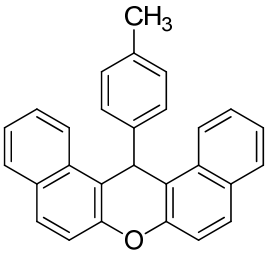
14-(4-chlorophenyl)-14H-dibenzo[a,j]xanthenes



Brown solid,m.p.: 287–288 ^{0}C; IR (K Br) ($\upsilon_{max,}$cm^{-1}):3025, 1620, 1590, 1483, 1457, 1400, 1239, 1082, 1012, 958, 831, 806, 740.; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.51 (s, 1 H), 7.15 (d, *J*= 8.5 Hz, 2 H), 7.45-7.49 (m, 4 H), 7.52 (d, *J*= 8.9 Hz, 2 H), 7.61 (t, *J*= 7.3 Hz, 2 H), 7.85 (d, *J*= 8.9 Hz, 2 H), 7.88 (d, *J*= 8.0 Hz, 2 H), 8.36 (d, *J*= 8.5 Hz, 2 H); ^{13}C NMR ($CDCl_3$):37.37, 116.73, 118.02, 122.40, 124.37, 126.92, 128.64, 128.91,129.09, 129.49, 131.04, 131.24, 132.07, 143.46, 148.66; MS (m/z, %): 392 (25), 281 (100), 252 (45), 75 (34).

Table-4; Entry-5

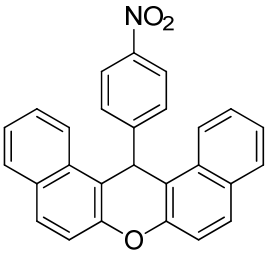
14-(p-tolyl)-14H-dibenzo[a,j]xanthenes



Yellow solid, m.p.: 227-228 ^{0}C; IR (K Br) ($\upsilon_{max,}$cm^{-1}): 3019, 2901, 1620, 1590, 1508, 1457, 1429, 1401, 1247, 961, 808, 739, 609.; ^{1}H NMR (400 MHz, CDCl3) δ = 2.18 (s, 3 H), 6.50 (s, 1 H), 7.00 (d, *J*= 8.0 Hz, 2 H), 7.44-7.48 (m, 4 H), 7.53 (d, *J*= 8.9 Hz, 2 H), 7.63 (t, *J*= 7.0 Hz, 2 H), 7.83 (d, *J*= 8.9 Hz, 2 H), 7.87 (d, *J*= 8.0 Hz, 2 H), 8.44 (d, *J*= 8.5 Hz, 2 H);^{13}C NMR: 147.9, 147.7,142.6, 135.8, 130.7, 128.8, 128.5, 127.6, 126.7, 124.4,123.3, 117.5,117.3, 117.1, 37.2, 20.3;EI-MS: *m/z* (%) = 372(M^{+}); Anal. Calcd for $C_{28}H_{20}O$: C, 90.29; H, 5.41. Found:C, 90.37,H 5.48.

Table-4; Entry-6

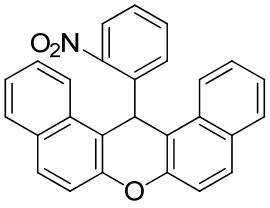
14-(4-nitrophenyl)-14H-dibenzo[a,j]xanthenes



Yellow solid: m.p. : 311-313 ^{o}C; IR (KBr) ($\upsilon_{max,}$cm^{-1}): 3065, 2930, 1622, 1590, 1520, 1458, 1401, 1342, 1200,1141, 1108, 1015, 965, 850, 828, 810, 743;^{1}HNMR: 8.30 (d, *J* = 8.5 Hz, 2H), 7.98 (d, *J* = 8.7 Hz, 2H),7.85 (d, *J* = 4.0 Hz, 2H), 7.82 (d, *J* = 5.5 Hz, 2H), 7.66 (d, *J*= 8.7 Hz, 2H), 7.61 (t, *J* = 5.5 Hz, 2H), 7.50 (d, *J* = 8.8 Hz, 2H), 7.45 (t, *J* = 7.9 Hz, 2H), 6.60 (s, 1H); 13C NMR: 152.5, 148.0, 145.8,135.1,130.9,130.7,129.7, 128.6,127.3, 124.7, 123.5, 123.0, 117.8, 116.2, 36.5;EI-MS:*m/z* (%) = 403 (M^{+}); Anal. Calcd for$C_{27}H_{17}NO_{3}$: C, 80.38;H, 4.25; N, 3.47. Found: C, 80.30; H, 4.35; N, 3.55.

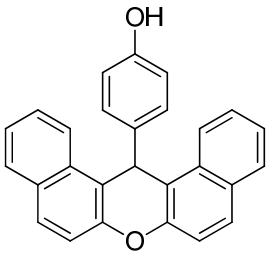
Table-4; Entry-7

14-(2-nitrophenyl)-14H-dibenzo[a,j]xanthenes



Yellow solid, m.p. : 292-293 ^{0}C.IR (KBr) ($\upsilon_{max,}$cm^{-1}): 3402, 3056, 2928, 1615,1594, 1524, 1352, 1243, 1140, 815, 750;^{1}H NMR ($CDCl_3$, 300 MHz): δ6.94 (s, 1H), 7.41-7.46 (m, 3H), 7.56-7.65 (m, 4H), 7.79 (d, *J* = 2.1, 1H), 7.89-7.94 (m,4H), 8.13 (d, *J* = 7.8, 1H), 8.45 (s, 1H), 8.70 (d, *J* = 6, 2H), ^{13}C NMR ($CDCl_3$, 75 MHz):δ 36.4, 116.9, 118.1, 122.0, 122.5, 123.6, 125.2, 127.7, 129.1, 130.0, 130.4, 131.1, 134.7,147.9, 148.3, 148.6.EI-MS: *m/z*(%) = 403 (M^+); Anal. Calcd for $C_{27}H_{17}NO_3$: C, 80.38; H,4.25; N, 3.47. Found: C, 80.26; H, 4.32; N,3.57

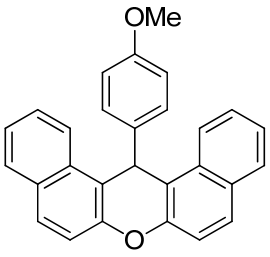
Table-4; Entry-8
14-(4-Hydroxyphenyl)-14*H*-dibenzo[*a,j*]xanthenes,



Pink solid, m.p. : 140-141 ^{0}C.IR (KBr) ($\upsilon_{max,}$cm^{-1}) : 3403, 3058, 2920, 1620,1593,1512, 1457, 1432, 1401, 1245, 1175, 1064, 1038, 962,817, 746, 609.^{1}H NMR ($CDCl_3$, 400 MHz): _ 8.17 (d, *J* =8.4 Hz, 2H), 7.63 (d, *J* = 8.0 Hz, 2H), 7.58 (d, *J* = 8.8 Hz, 2H),7.38 (t, *J* = 7.5 Hz, 2H), 7.27 (d, *J* = 8.8 Hz, 2H), 7.19 (t, *J* =7.6 Hz, 2H), 7.17(d, *J* = 8.4 Hz, 2H), 6.40 (d, *J* = 8.0 Hz, 2H),6.23 (s, 1H) ppm;^{13}C NMR: 154.1, 149.0, 137.7, 131.7, 131.5, 129.7,129.2, 129.0, 127.3, 124.7, 123.2, 118.5, 117.8, 115.8,37.6;EI-MS: *m/z* (%) = 374 (M^+); Anal.Calcd for $C_{27}H_{18}O_2$: C, 86.61; H, 4.85. Found: C, 86.55; H,4.80.

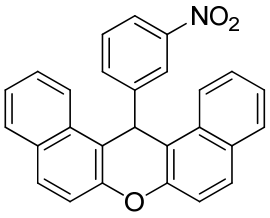
Table-4; Entry-9

14-(4-methoxyphenyl)-14H-dibenzo[a,j]xanthenes



Yellow solid, m.p. : 204-206.IR (KBr) ($\upsilon_{max,}cm^{-1}$) : 3072, 2833, 1591, 1457, 1430, 1399, 1248, 1177, 1027, 961, 830, 808, 741.^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.66 (s, 3 H), 6.49 (s, 1 H), 6.71 (d, *J*= 8.8 Hz, 2 H), 7.43 7.47 (m, 4 H), 7.52 (d, *J*= 8.9 Hz, 2 H), 7.63 (t, *J*= 8.1 Hz, 2 H), 7.82 (d, *J*= 8.9 Hz, 2 H), 7.87 (d, *J*= 8.0 Hz, 2 H), 8.43 (d, *J*= 8.5 Hz, 2 H).^{13}C NMR ($CDCl_3$, 75 MHz): δ 36.0, 55.3, 114.1, 118.1, 123.9,124.9, 127.9, 124.9, 127.3, 129.2, 131.1, 135.7, 138.1, 147.0, 148.3, 149.4, 156.8.EI-MS: *m/z* (%) = 388 (M^+);Anal. Calcd for $C_{28}H_{20}O_2$: C, 86.57; H, 5.19. Found: C,86.45; H, 5.27.

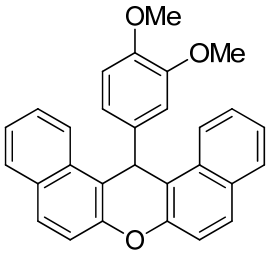
Table-4; Entry-10



m.p. : 211-213 ^{0}C,IR (KBr) ($\upsilon_{max,}cm^{-1}$) : 3080, 1621, 1592, 1529, 1458, 1430, 1401, 1347, 1251, 1140, 1081, 964, 825, 808, 744. ^{1}H NMR (400 MHz, $CDCl_3$) : δ = 6.60 (s, 1 H), 7.29 (t, *J*= 7.7 Hz, 1 H), 7.48 (t, *J*= 7.5 Hz, 2 H), 7.55 (d, *J*= 8.9 Hz, 2 H), 7.66 (t, *J*= 7.7 Hz, 2 H), 7.84-7.88 (m, 6 H), 8.33 (d, *J*= 8.5 Hz, 2 H), 8.47 (s, 1 H).

Table-4; Entry-11

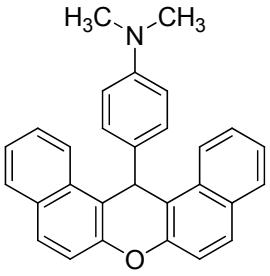
14-(3,4-dimethoxyphenyl)-14H-dibenzo[a,j]xanthenes



IR (KBr) ($\upsilon_{max,}cm^{-1}$) : 3064, 2932, 2832, 1622, 1592,1514, 1457, 1433,1401, 1239, 1140, 1072, 1020, 961, 859,819, 748. ^{1}H NMR ($CDCl_3$, 400 MHz): 8.21 (d, *J* =6.0 Hz, 2H), 7.22-7.70 (m, 12H), 6.93 (d, *J* = 8.0 Hz, 1H),6.72 (s, 1H), 6.46 (d, *J* = 8.0 Hz, 1H), 6.26 (s, 1H), 3.49 (s,6H) ppm;

Table-4; Entry-12

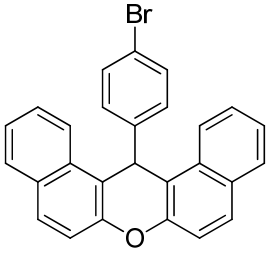
4-(14H-dibenzo[a,j]xanthen-14-yl)-N,N-dimethylaniline



^{1}H NMR (DMSO, 300MHz): δ 2.60 (s, 6H), 6.42 (d, *J* = 7.2 Hz, 2H), 6.56 (s, 1H), 7.37-7.92 (m, 12H), 8.64 (d,*J* = 8, 2H),^{13}C NMR ($CDCl_3$, 75 MHz): δ 36.0, 40.8, 112.6, 118.1, 118.4, 123.9, 124.8,127.2, 128.8, 128.9, 129.0, 131.1, 131.4, 133.8, 148.3, 149.0. MS: *m/z* = 402 (M^{++1}), 401(M^{+}), 357 (M^{+}-N(CH3)2), 281 (M^{+}- N,N-dimethylaniline.

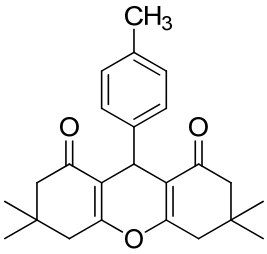
Table-4; Entry-13

14-(4-bromophenyl)-14H-dibenzo [a,j]xanthenes

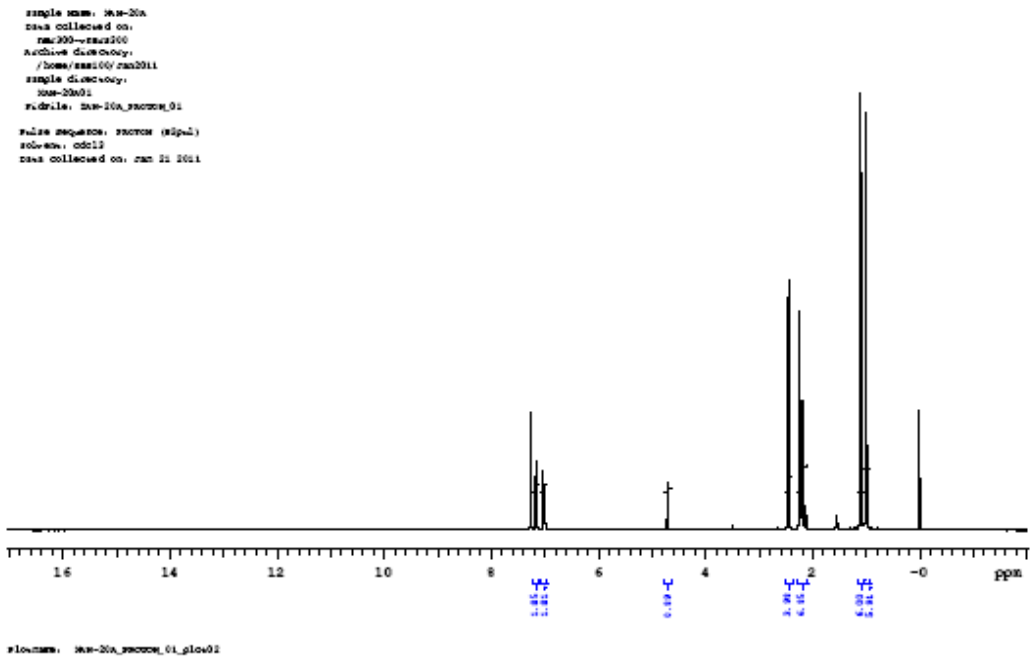


m.p. : 297–299 ^{0}C.IR (KBr) ($\upsilon_{max,}cm^{-1}$) : 3068, 2905, 1620, 1590, 1481, 1456, 1430, 1400, 1238, 1072, 1008, 961, 826,808, 739. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.50 (s, 1 H), 7.30 (d, *J*= 8.5 Hz, 2 H), 7.42-7.48 (m, 4 H), 7.52 (d, *J*= 8.9 Hz, 2 H), 7.63 (t, *J*= 7.2 Hz, 2 H), 7.85 (d, *J*= 8.9 Hz, 2 H), 7.88 (d, *J*= 8.0 Hz, 2 H), 8.36 (d, *J*= 8.5 Hz, 2 H).^{13}C NMR ($CDCl_3$): 37.46, 116.64,118.02, 120.21, 122.39, 124.38, 126.93, 128.91,129.12,129.88, 131.03, 131.23, 131.58, 143.98, 148.65;MS (m/z,%): 437 (20), 281 (100), 252 (40),75(15).

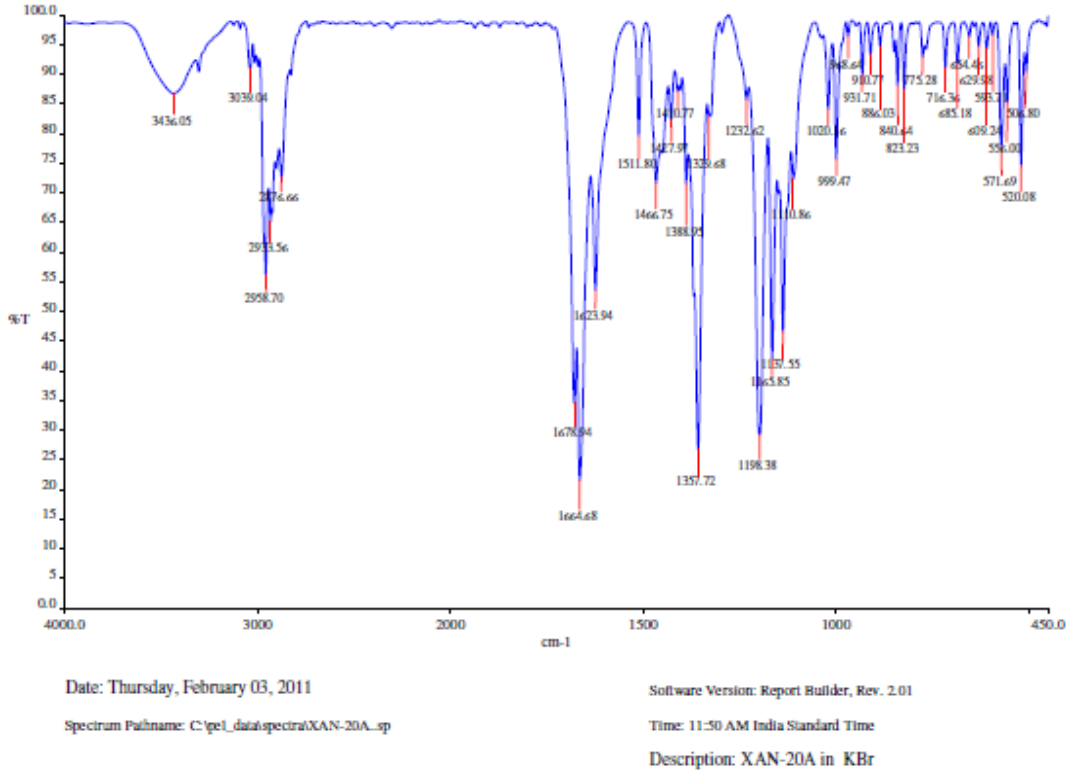
1. 3,3,6,6-Tetramethyl-9-(4-methylphenyl)-1,8-dioxooctahydroxanthene (table-3; entry 5)



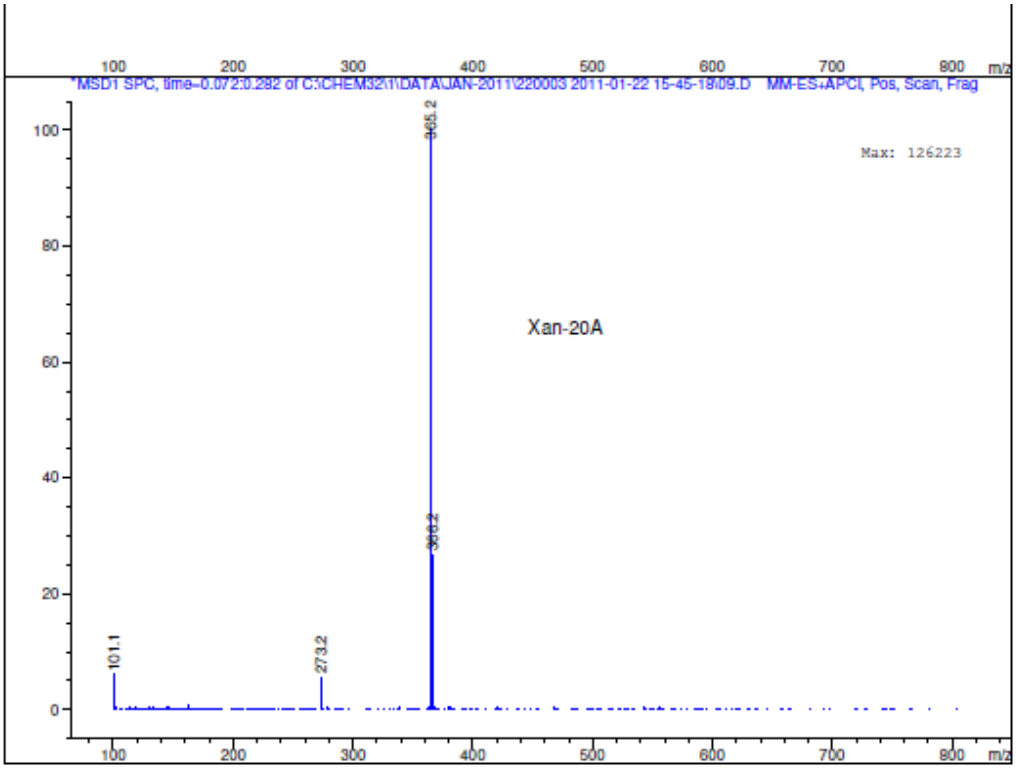
Yellow crystals, m.p.:215-217^{o}C, (Lit.:24217-218 C); IR (KBr), ($\upsilon_{max,}$cm^{-1}): 3039, 2958,1664,1466,1198,1165,840. ^{1}H NMR ($CDCl_3$, 300 MHz): δ = 0.99 (s, 6H), 1.09 (s, 6H), 2.15 (d, 2H, J = 16.5 Hz), 2.23(d,2H ,J=16.5Hz), 2.45 (s, 4H), 4.70 (s, 1H), 7.01 (d, 2H, J = 7.5 Hz), 7.17 (d, 2H, J =7.5Hz). ESI-MS (m/z): 364 [M^{+}H] $^{+}$; Anal. Calcd for $C_{24}H_{28}O_3$: C, 79.09; H, 7.74.Found: C, 79.02; H, 7.79.



^{1}HNMR- table-3; entry 5

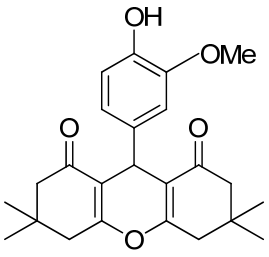


IR- table-3; entry 5



Mass spectroscopy - table-3; entry 5

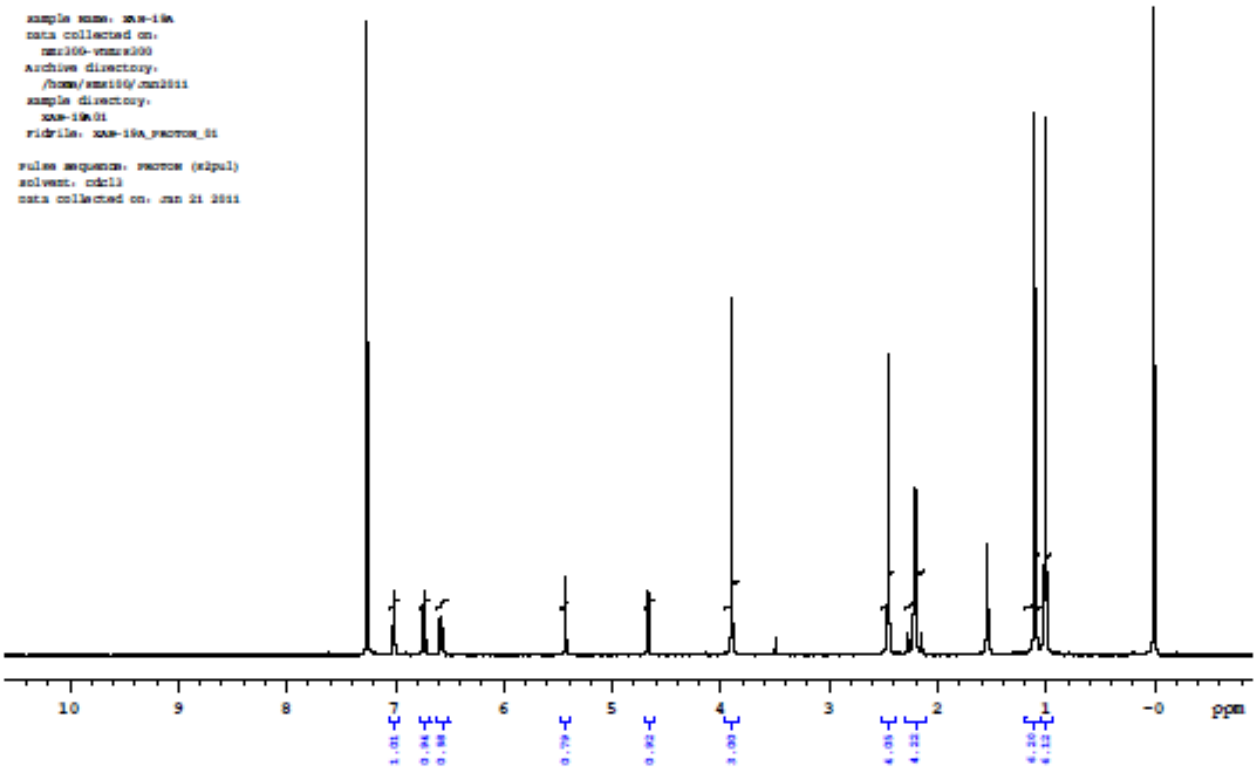
2. 3,3,6,6-Tetramethyl-9-(3,methoxy4-hydroxyphenyl)-1,8-dioxooctahydroxanthene (table-3; entry-14)



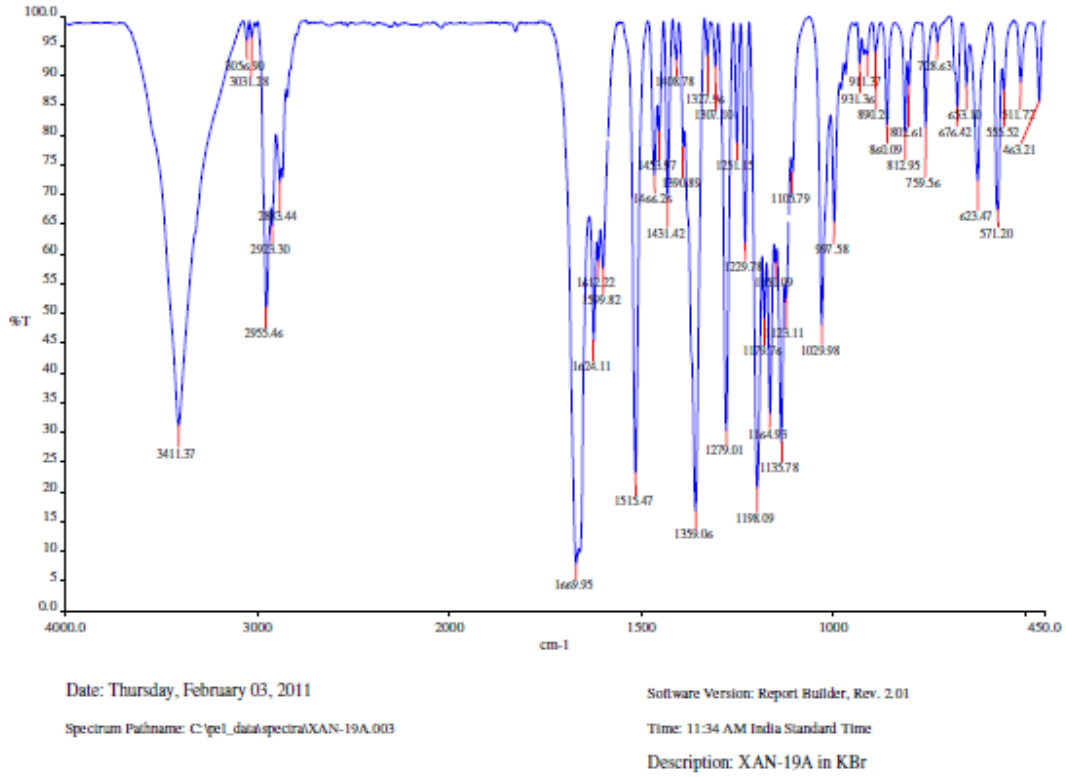
White solid, m.p.: -210^{O}, IR(KBr) ($\upsilon_{max,}cm^{-1}$) : 3401, 2954,1669,1624, 1515,1466,1359,1251, 1165,1135,1029,997,860,812,759,676, ^{1}HNMR (300 MHz, CDC1) δ = 7.01 (1H, d, J = 2.1Hz),

6.73 (1H, d, J = 8.4 Hz), 6.57 (dd, J= 8.4 2.1 Hz,1H), 5.42 (s,1H), 4.66(s,IH), 3.89 (s,3H), 2.45(s,4H), 2.24 (2H, d, J = 16.2 Hz), 2.18(d,2H,J=16.2Hz), 1.01 (s,6H), 1.00 (s,6H).

ESI- MS (*m*/*z*): 396 $[M+H]^+$; Anal. Calcd for $C_{24}H_{28}O_5$: C, 72.70; H, 7.12.Found: C, 72.67; H, 7.09

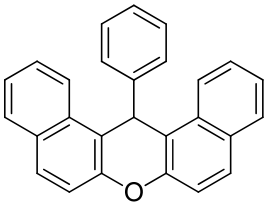


^{1}HNMR - table-3; entry-14

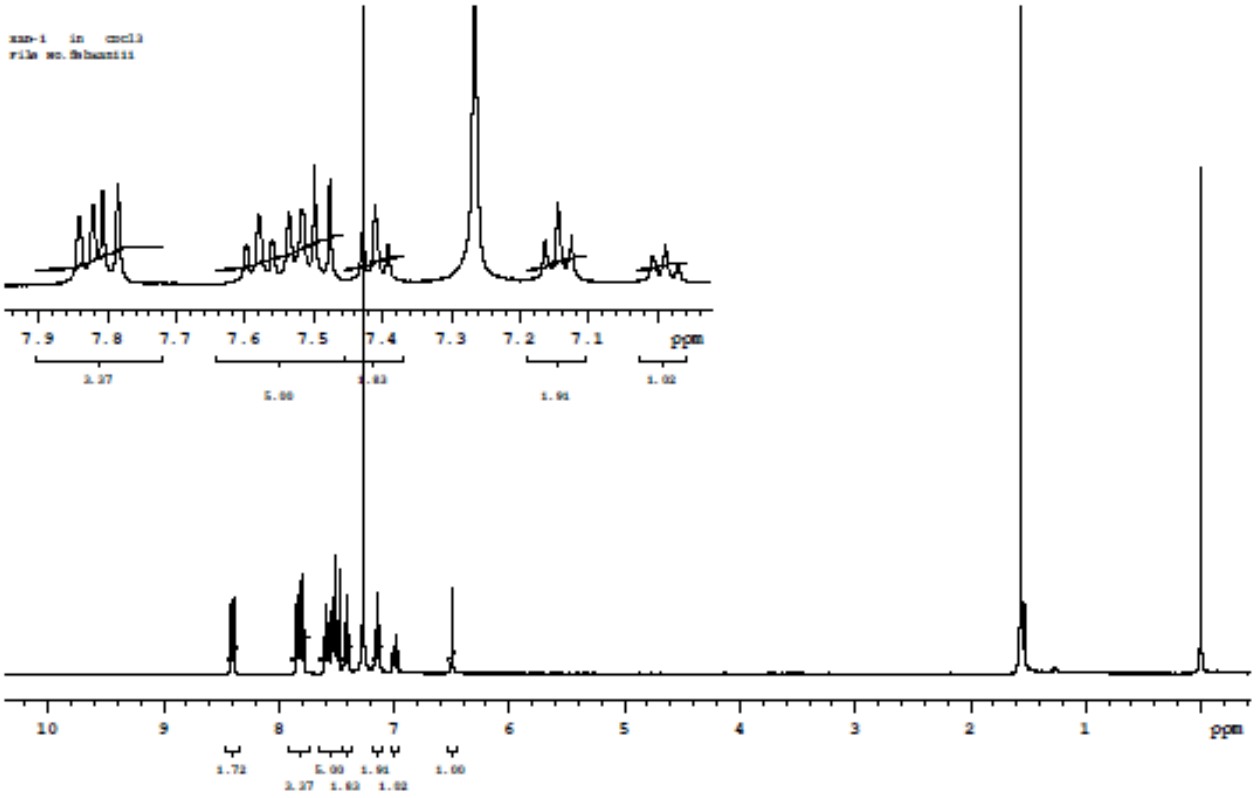


IR - table-3; entry-14

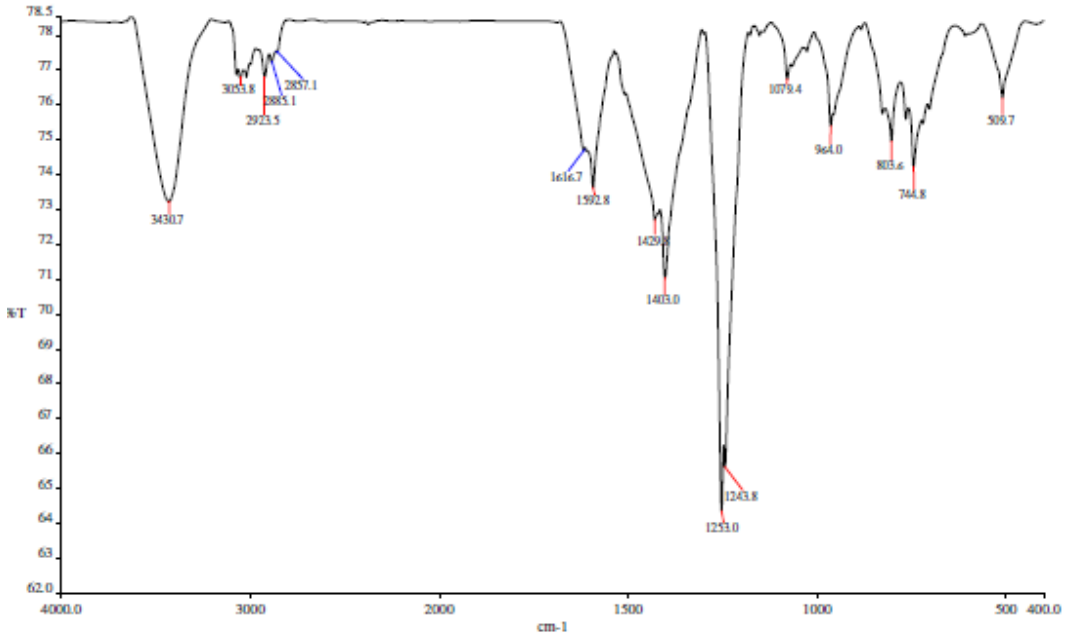
3. 14-Phenyl-14H-dibenzo [a, j] xanthenes (table-4; entry1)



Colorless crystals, m.p.: 186 ^{O}C. IR (KBr) ($\upsilon_{max,}cm^{-1}$) : 3053, 2923, 2885, 1622, 1592, 1429, 1403, 1253, 1152, 1079, 964, 744. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.46 (s, 1H, CH), 6.96 (t, *J* = 7.2 Hz, 1H, ArH), 7.12 (t, *J* = 7.2 Hz, 2H, ArH), 7.36–7.58 (m, 8H, ArH), 7.74–7.81 (m, 4H, ArH), 8.37 (d, *J* = 8.4 Hz, 2H, ArH); ESI MS (*m*/*z*): 358 $[M^{+}H]^{+}$; Anal. Calcd for $C_{27}H_{18}O$: C, 90.47; H, 5.06.Found: C, 90.42; H, 5.08



^{1}HNMR- table-4; entry1

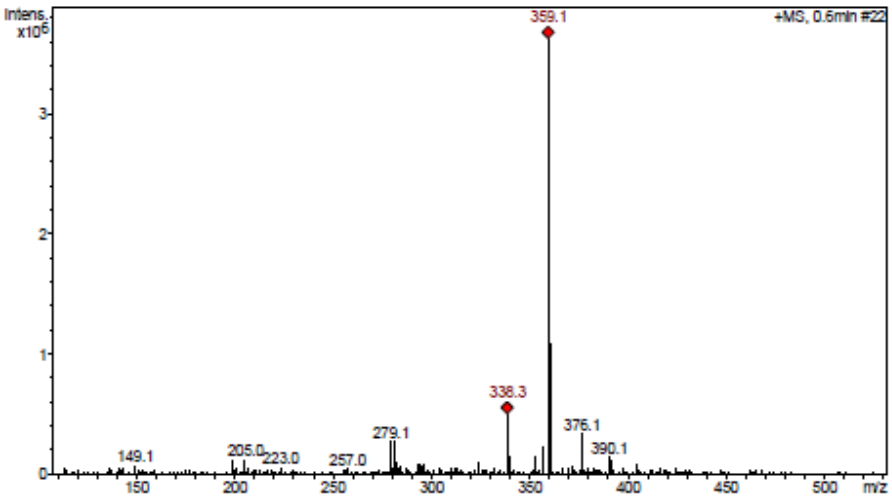


Sample Code : Xan-1

Comments: KBr Pellet

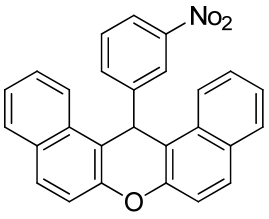
Date Created: Fri Feb 11 16:13:20 2011 India Standard Time (GMT+5:30)

IR- table-4; entry1

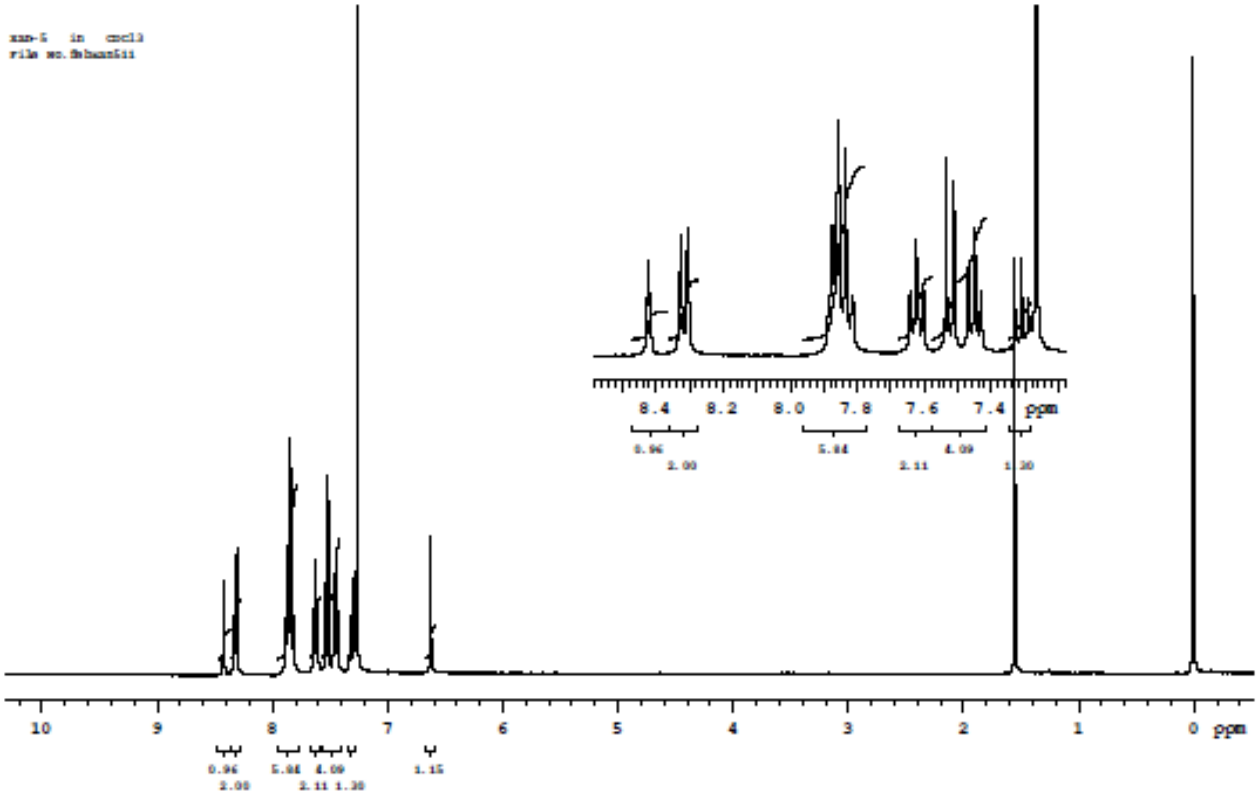


Mass spectroscopy- table-4; entry-1

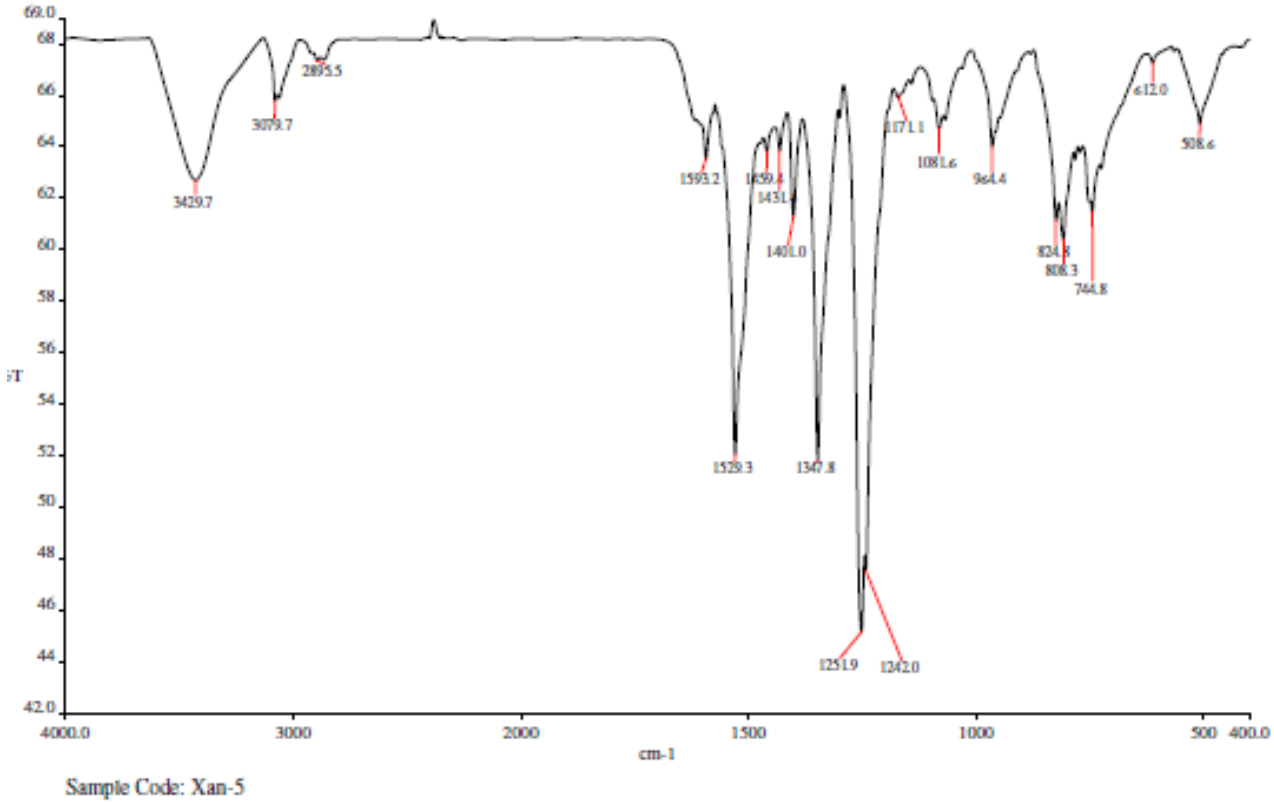
4. 14-(3-Nitrophenyl)-14H-dibenzo [a,j]xanthenes (table-4;entry 10)



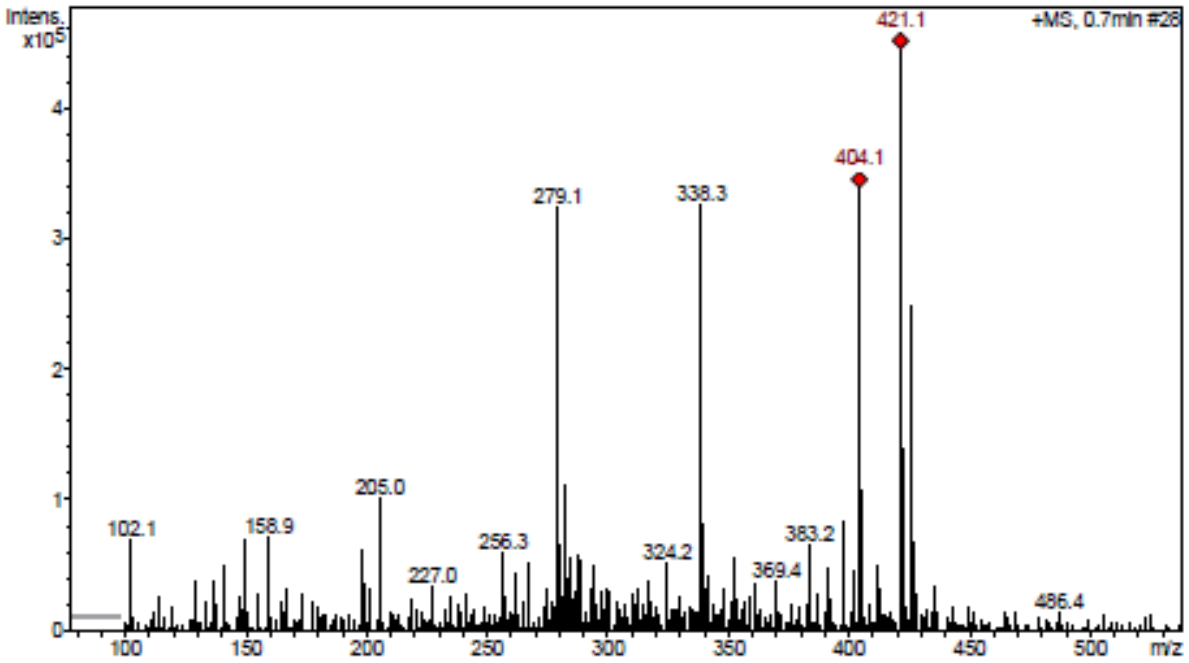
White solid, m.p.: 305-308^{O}C, IR (KBr) ($\upsilon_{max,}$cm^{-1}) : 3429,3079,1593,1529,1347,1242,1171,808,744. ^{1}H-NMR (200 MHz, $CDCl_3$) δ = 8.40 (s, 1 H); 8.25(d, *J*= 8.0, 2 H); 7.90 – 7.7 0 (m, 6 H); 7.63 – 7.21 (m, 7H); 6.50 (s, 1 H). ESI MS (m/z): 403 $[M^{+}H]^{+}$; Anal. Calcd for $C_{27}H_{17}NO_3$: C, 82.54; H, 4.36.Found: C, 82-51; H, 4.32



^{1}HNMR- table-4; entry 10



IR- table-4; entry 10



Mass Spectroscopy- table-4; entry 10