Electronic Supplementary Material (ESI) for RSC Advances.


# One-pot four component domino strategy for the synthesis of novel spirooxindole pyrrolizine linked 1,2,3-triazoles *via* stereo- and regioselective [3+2] cycloaddition reaction in acidic Medium.

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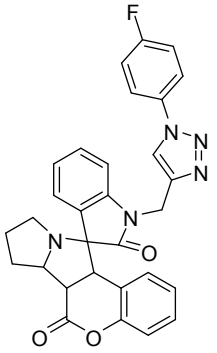
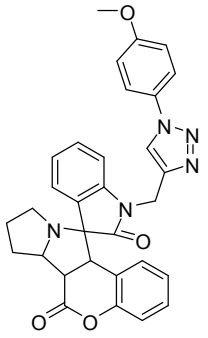
**Supplementary data**

**1'-((1-(4-fluorophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H* spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5a)**



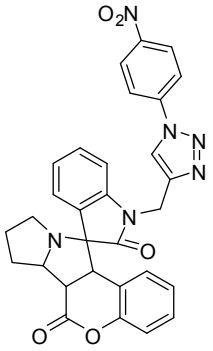
White solid; Yield = 89%; M.p.= 190-192; IR ($CHCl_3$) ν/$cm^{-1}$ = 2965, 1756, 1516, 1461, 1227, 752; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 7.76 (d, 1H, *J* = 6.87 Hz, Ar-H), 7.73-7.69 (m, 2H, Ar-H), 7.48 (s, 1H, Triazole), 7.42-7.34 (m, 3H, Ar-H), 7.17-7.13 (t, 1H, Ar-H), 7.05 (d, 1H, *J* = 7.63 Hz, Ar-H), 6.78 (d, 1H, *J* = 8.39, Ar-H), 6.62-6.59 (t, 1H, Ar-H), 6.47-6.44 (t, 1H, Ar-H), 6.05 (d, 1H, *J* =7.63 Hz, Ar-H), 4.67-4.55 (q, 2H, -$CH_2$), 4.40-4.38 (dt, 1H, J = 3.41, 8.01 Hz, -CH), 4.14-4.11 (d, 1H, *J* = 11.45 Hz, -CH), 3.38-3.36 (dd, 1H, *J* = 11.45, 3.21, Hz, -CH), 3.17-3.13 (m, 1H, -$CH_2$), 2.58-2.55 (m, 1H, -$CH_2$), 2.34-2.28 (m, 1H, -$CH_2$), 1.86-1.77 (m, 2H, -$CH_2$), 1.63-1.57 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.3, 167.5, 161.7, 150.7, 143.2, 142.8, 132.9, 130.0, 128.2, 127.2, 126.6, 125.3, 123.0, 122.8, 122.6, 120.9, 117.4, 116.7, 116.2, 109.5, 76.0, 67.4, 50.3, 46.8, 42.8, 33.9, 32.7, 26.0; LCMS (ESI) *m/z* calcd for $C_{30}H_{24}FN_5O_3$ : 521.1865 [$M^+$]; found: 522.1937 [$M^+$ +H].

**1'-((1-(4-methoxyphenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5b)**



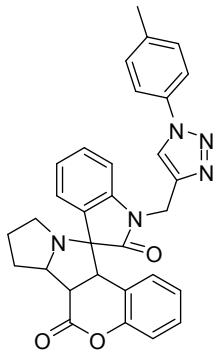
White solid; Yield = 85%; M.p.= 173-175; IR ($CHCl_3$) ν/$cm^{-1}$ = 2964, 1757, 1517, 1225, 1041, 754; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 7.76-7.75 (m, 1H, Ar-H), 7.54-7.53 (m, 2H, Ar-H), 7.37-7.33 (m, 1H, Ar-H), 7.28 (s, 1H, triazole), 7.14-7.12 (m, 2H, Ar-H), 7.08-7.06 (m, 1H, Ar-H), 7.03-7.01 (m, 1H, Ar-H), 6.80-6.78 (m, 1H, Ar-H), 6.63-6.59 (m, 1H, Ar-H), 6.48-6.44 (m, 1H, Ar-H), 6.07-6.05 (m, 1H, Ar-H), 4.66-4.53 (q, 2H, -$CH_2$), 4.37 (brs, 1H, -CH), 4.13-4.10 (m, 1H, -$CH_2$), 3.77 (s, 3H, -$OCH_3$), 3.43-3.31 (m, 1H, -$CH_2$), 3.14-3.11 (m, 1H, -$CH_2$), 2.55-2.54 (m, 1H, -CH2), 2.33-2.27 (m, 1H, -$CH_2$), 1.90-1.75 (m, 2H, -$CH_2$), 1.62-1.57 (m, 1H, -$CH_2$); 13C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.3, 167.5, 159.3, 150.7, 143.2, 142.6, 130.0, 129.7, 128.3, 127.2, 126.6, 125.3, 123.0, 122.5, 122.1, 120.6, 117.5, 116.2, 114.8, 109.5, 76.0, 67.4, 55.5, 50.4, 46.8, 42.8, 34.0, 32.7, 26.1; LCMS (ESI) *m/z* calcd for $C_{31}H_{27}N_5O4$ : 533.2065 [$M^+$]; found: 534.2146 [$M^+$ +H].

**1'-((1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5c)**



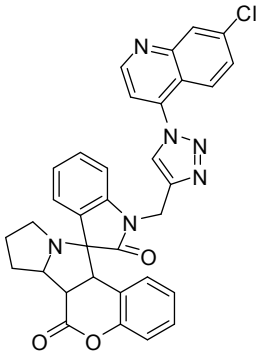
Pale Yellow soli; Yield = 87%; M.p. = 212-214; IR ($CHCl_3$) ν/$cm^{-1}$ = 2926, 1755, 1342, 1171, 752; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 8.40 (d, 2H, *J* = 8.39 Hz, Ar-H), 8.01 (d, 2H, *J* = 9.16, Ar-H), 7.90 (s, 1H, triazole), 7.75 (d, 1H, *J* = 6.87, Ar-H), 7.39-7.36 (t, 1H, Ar-H), 7.17-7.10 (m, 2H, Ar-H), 6.77 (d, 1H, *J* = 7.63, Ar-H), 6.62-6.58 (t, 1H, Ar-H), 6.44-6.41 (t, 1H, Ar-H), 6.01 (d, 1H, *J* = 7.63, Ar-H), 4.68-4.58 (q, 2H, -$CH_2$), 4.38 (brs, 1H, -CH), 4.11-4.08 (m, 1H, -$CH_2$), 3.43-3.30 (m, 1H, -$CH_2$), 3.15-3.11 (m, 1H, -$CH_2$), 2.59-2.55 (m, 1H, -$CH_2$), 2.33-2.27 (m, 1H, -$CH_2$), 1.84-1.73 (m, 2H, -$CH_2$), 1.62-1.56 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.2, 167.5, 150.6, 146.7, 143.3, 143.1, 140.5, 130.0, 128.2, 127.1, 126.5, 125.5, 125.3, 122.9, 122.5, 121.2, 120.7, 117.3, 116.1, 109.6, 76.03, 67.5, 50.3, 46.8, 43.0, 33.7, 32.7, 26.0; LCMS (ESI) *m/z* calcd for $C_{30}H_{24}N_6O_5$ : 548.1811 [$M^+$]; found: 549.1888 [$M^+$ +H].

**1'-((1-p-tolyl-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6H-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5d)**



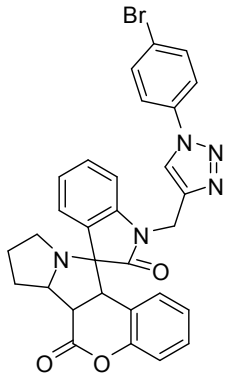
White solid; Yield = 80%; M.p. = 170-173; IR ($CHCl_3$) ν/$cm^{-1}$= 2962, 1754, 1492, 1224, 1170, 751; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 7.78-7.75 (m, 3H, Ar-H), 7.67-7.65 (m, 2H, Ar-H), 7.62 (s, 1H, triazole), 7.40-7.36 (t, 1H, Ar-H), 7.18-7.15 (t, 1H, Ar-H), 7.08 (d, 1H, *J* = 7.63 Hz, Ar-H), 6.80-6.79 (d, 1H, *J* = 7.63, Ar-H), 6.65-6.61 (t, 1H, Ar-H), 6.49-6.45 (t, 1H, Ar-H), 6.05 (d, 1H, *J* =7.63 Hz, Ar-H), 4.68-4.57 (q, 2H, -$CH_2$), 4.39 (brs, 1H, -CH), 4.15-4.12 (m, 1H, -$CH_2$), 3.50-3.38 (m, 1H, -$CH_2$), 3.17-3.16 (m, 1H, -$CH_2$), 2.60-2.59 (m, 1H, -$CH_2$), 2.35-2.29 (m, 1H, -$CH_2$), 2.03 (s, 3H, -$CH_3$), 1.86-1.80 (m, 2H, -$CH_2$), 1.64-1.58 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.2, 167.5, 150.6, 143.2, 142.6, 138.4, 134.0, 130.1, 130.0, 128.2, 127.2, 126.5, 125.3, 123.0, 122.5, 123.0, 122.5, 120.4, 120.2, 117.4, 116.2, 109.5, 76.0, 67.4, 50.3, 46.8, 42.8, 33.9, 32.7, 26.0, 20.5; LCMS (ESI) *m/z* calcd for $C_{31}H_{27}N_5O_3$ : 517.2106 [$M^+$]; found: 518.2180 [$M^+$ +H].

**1'-((1-(7-chloroquinolin-4-yl)-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5e)**



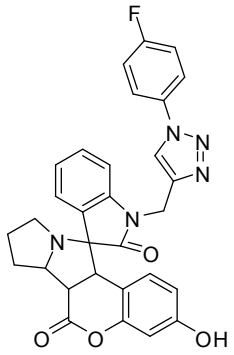

White solid; Yield = 84%; M.p. = 204-206; IR ($CHCl_3$) ν/$cm^{-1}$= 2963, 1758, 1609, 1225, 1175, 755; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 9.16-9.15 (m, 1H, Ar-H), 8.29 (s, 1H, Ar-H), 7.83 (d, 1H, $J$ = 7.63 Hz, Ar-H), 7.79 (s, 1H, triazole), 7.67-7.66 (m, 1H, Ar-H), 7.46-7.42 (t, 1H, Ar-H) 7.36 (s, 1H, Ar-H), 7.24-7.20 (t, 1H, Ar-H), 7.12 (d, 1H, $J$ =7.63 Hz, Ar-H), 6.69 (d, 1H, $J$ = 8.39, Ar-H), 6.39-6.30 (m, 2H, Ar-H), 6.12 (d, 1H, $J$ = 7.63 Hz, Ar-H), 4.84-4.72 (q, 2H, -$CH_2$), 4.45-4.41 (m, 1H, -CH), 4.19-4.16 (m, 1H, -CH), 3.41-3.38 (m, 1H, -$CH_2$), 3.24-3.17 (m, 1H, -$CH_2$), 2.64-2.61 (m, 1H, -$CH_2$), 2.39- 2.32 (m, 1H, -$CH_2$), 1.91-1.78 (m, 1H, -$CH_2$), 1.68-1.62 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.3, 167.4, 152.3, 150.3, 150.8, 149.4, 143.1, 142.7, 139.9, 135.4, 130.1, 129.0, 128.2, 128.0, 127.2, 126.7, 125.2, 124.2, 122.9, 122.5, 120.1, 117.5, 117.0, 116.2, 109.4, 75.9, 67.4, 50.4, 46.7, 42.6, 33.9, 32.8, 30.5, 26.0; LCMS (ESI) *m/z* calcd for $C_{33}H_{25}ClN_6O_3$ : 588.1671 [$M^+$]; found: 589.1746 [$M^+$ +H].

**1'-((1-(4-bromophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5f)**



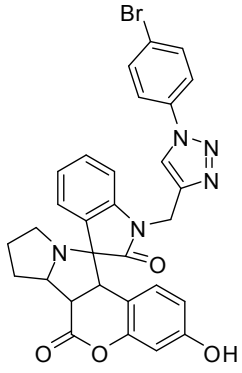

White solid; Yield = 88%; M.p. = 174-176; IR ($CHCl_3$) ν/$cm^{-1}$= 2923, 1753, 1493, 1225, 1172, 753; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 7.80 (d, 3H, $J$ = 8.39, Ar-H), 7.69 (d, 2H, $J$ = 9.16, Ar-H), 7.64 (s, 1H, triazole), 7.43-7.39 (t, 1H, Ar-H), 7.22-7.18 (t, 1H, Ar-H), 7.11 (d, 1H, $J$ = 8.39 Hz, Ar-H), 6.81-6.79 (m, 1H, Ar-H), 6.69-6.65 (t, 1H, Ar-H), 6.52-6.49 (t, 1H, Ar-H), 6.09 (d, 1H, Ar-H), 4.71-4.60 (q, 2H, -$CH_2$), 4.45-4.42 (m, 1H, -CH), 4.16 (d, 1H, $J$ = 11.4 Hz, -$CH_2$), 3.42-3.38 (m, 1H, Ar-H), 3.23- 3.16 (m, 1H, -$CH_2$), 2.63-2.60 (m, 1H, -$CH_2$), 2.39-2.32 (m, 1H, -$CH_2$), 1.90-1.78 (m, 2H, -$CH_2$), 1.70-1.61 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.2, 167.4, 150.6, 143.3, 142.8, 135.5, 132.7, 130.0, 128.1, 127.2, 126.5, 123.0, 122.5, 122.2, 121.4, 120.7, 117.3, 116.1, 109.5, 76.0, 67.4, 50.3, 46.7, 42.9, 33.8, 32.7, 26.0; LCMS (ESI) *m/z* calcd for $C_{30}H_{24}BrN_5O_3$ : 581.1064 [$M^+$]; found: 582.1138 [$M^+$+H], 584.1123 [$M^+$ +(H+2)].

**1'-((1-(4-fluorophenyl)-1H-1,2,3-triazol-4-yl)methyl)-3-hydroxy-6b,7,8,9-tetrahydro-6H-spiro[chromeno[3,4-a]pyrrolizine-11,3'-indoline]-2',6(6aH,11aH)-dione (5g)**



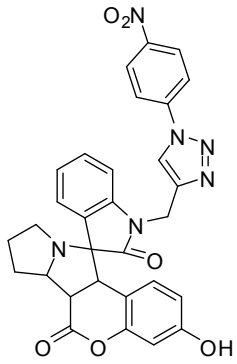

White solid; Yield = 85%; M.p. = 172-174; IR ($CHCl_3$) ν/$cm^{-1}$= 2924, 1715, 1514, 1231, 1163, 755; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 9.48 (s, 1H, -OH), 7.82 (s, 1H, triazole), 7.78-7.73 (m, 3H, Ar-H), 7.41-7.37 (m, 3H, Ar-H), 7.19-7.16 (t, 1H, Ar-H), 7.06 (d, 1H, *J* = 7.63 Hz, Ar-H), 6.29 (s, 1H, Ar-H), 6.08 (d, 1H, *J* = 7.63 Hz, Ar-H), 6.00 (d, 1H, *J* = 8.39 Hz, Ar-H), 4.68 (s, 2H, -$CH_2$), 4.39 (m, 1H, -CH), 4.08-4.05 (m, 1H, -$CH_2$), 3.49-3.36 (m, 1H, -$CH_2$), 3.17 (brs, 1H, -$CH_2$), 2.59-2.56 (m, 1H, -$CH_2$), 2.34-2.30 (m, 1H, -$CH_2$), 1.86-1.76 (m, 2H,- $CH_2$), 1.65-1.61 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.5, 167.8, 162.9, 160.4, 157.6, 151.5, 143.4, 142.9, 132.8, 129.9, 128.0, 126.5, 125.5, 122.6, 122.5, 120.9, 116.8, 116.6, 110.9, 109.4, 107.3, 102.8, 75.6, 67.0, 50.3, 47.0, 42.2, 34.0, 32.8, 26.1; LCMS (ESI) *m/z* calcd for $C_{30}H_{24}FN_5O_4$ : 537.1809 [$M^+$]; found: 538.1884 [$M^+$ +H].

**1'-((1-(4-bromophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-3-hydroxy-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5h)**



White solid; Yield = 83%; M.p. = 174-177; IR ($CHCl_3$) ν/$cm^{-1}$ = 2924, 1720, 1514, 1231, 1163, 758; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 9.49 (s, 1H, OH), 7.87 (s, 1H, triazole), 7.76-7.74 (m, 3H, Ar-H), 7.69-7.67 (m, 2H, Ar-H), 7.40-7.36 (t, 1H, Ar-H), 7.19-7.15 (t, 1H, Ar-H), 7.05 (d, 1H, $J$ = 8.39 Hz, Ar-H), 6.29 (s, 1H, Ar-H), 6.08 (d, 1H, $J$ = 6.10 Hz, Ar-H), 5.99 (d, 1H, $J$ = 8.01 Hz, Ar-H), 4.68 (s, 2H, -$CH_2$), 4.38 (brs, 1H, -CH), 4.07-4.04 (m, 1H, -CH), 3.46-3.43 (m, 1H, -CH), 3.16-3.13 (m, 1H, -$CH_2$), 2.58-2.55 (m, 1H, -$CH_2$), 2.36-2.30 (m, 1H, -$CH_2$), 1.92-1.75 (m, 2H,-$CH_2$), 1.67-1.55 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.4, 167.7, 157.5, 151.4, 143.3, 143.1, 135.6, 132.7, 129.9, 127.9, 126.5, 125.5, 122.4, 122.0, 121.3, 120.6, 110.8, 109.5, 107.4, 102.8, 75.67, 67.0, 50.3, 46.9, 42.3, 34.02, 32.8, 26.1; LCMS (ESI) *m/z* calcd for $C_{30}H_{24}BrN_5O_4$ : 597.1003 [$M^+$]; found: 598.1076 [$M^+$ +H]; 600.1063 [$M^+$+(H+2)].

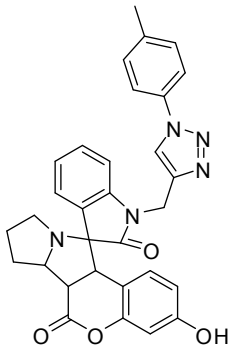
**3-hydroxy-1'-((1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5i)**




Pale yellow solid; Yield = 86%; M.p. = 182-184; IR ($CHCl_3$) ν/$cm^{-1}$= 2920, 1711, 1521, 1346, 1170, 752; 1H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 9.38 (s, 1H, -OH), 8.40 (d,1H, $J$ =8.39 Hz, Ar-H), 8.06-8.05 (m, 1H, Ar-H), 8.02 (s, 1H, triazole), 7.77 (d, 1H, $J$ =7.35, Ar-H), 7.42-7.38 (t, 1H, Ar-H), 7.20-7.16 (t, 1H, Ar-H), 7.10 (d, 1H, $J$ = 7.63 Hz, Ar-H), 6.25 (s, 1H, Ar-H), 6.04-

6.02 (m, 1H, Ar-H), 5.95-5.93 (m, 1H, Ar-H), 4.71 (s, 2H, -$CH_2$), 4.39 (brs, 1H, -CH), 4.06-4.03 (m, 1H, -CH), 3.49-3.40 (m, 1H, -$CH_2$), 3.17-3.18 (m, 1H, -$CH_2$), 2.58-2.52 (m, 1H, -$CH_2$), 2.34-2.32 (m, 1H, -$CH_2$), 1.87-1.78 (m, 2H, -$CH_2$), 1.65-1.61 (m, 1H,-$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.5, 167.7, 157.5, 151.4, 146.6, 143.5, 143.2, 140.5, 129.9, 127.9, 126.5, 125.5, 122.5, 121.1, 120.7, 110.7, 109.4, 107.3, 102.7, 75.6, 67.1, 50.3, 46.9, 42.3, 33.9, 32.8, 30.7, 26.1; LCMS (ESI) *m/z* calcd for $C_{30}H_{24}N_6O_6$ : 564.1752 [$M^+$]; found: 565.1825 [$M^+$ +H].

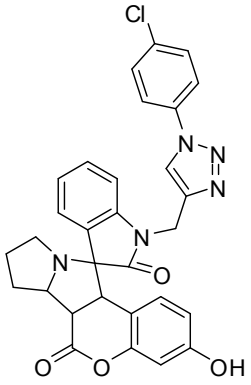
**3-hydroxy-1'-((1-p-tolyl-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5j)**




White solid; Yield = 81%; M.p. = 173-175; IR ($CHCl_3$) ν/$cm^{-1}$= 2924, 1713, 1515, 1166, 755; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 9.57 (s, 1H, -OH), 7.82 (s, 1H, triazole), 7.78 (d, 1H, *J* = 6.87 Hz, Ar-H), 7.58 (d, 1H, *J* = 8.39 Hz, Ar-H), 7.39-7.34 (m, 3H, Ar-H), 7.19-7.15 (t, 1H, Ar-H), 7.04 (d, 1H, *J* = 7.63 Hz, Ar-H), 6.33-6.31 (m, 1H, Ar-H), 6.14-6.11 (m, 1H, Ar-H), 6.04-6.02 (m, 1H, Ar-H), 4.71-4.63 (m, 2H,-$CH_2$), 4.41-4.36 (m, 1H, -CH), 4.09-4.06 (m, 1H, -CH), 3.33-3.32 (m, 1H, Ar-H), 3.19-3.13 (m, 1H, -$CH_2$), 2.57-2.54 (m, 1H, -$CH_2$), 2.35 (s, 3H, -$CH_3$), 2.32-2.30 (m, 1H, -$CH_2$), 1.87-1.75 (m, 2H, -$CH_2$), 1.67-1.56 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.5, 167.7, 157.6, 151.5, 143.4, 142.8, 138.3, 134.0, 130.2.

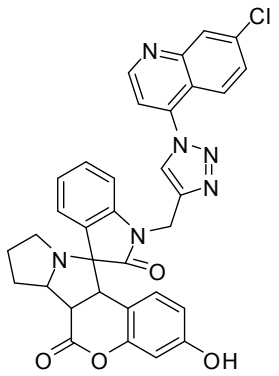
129.9, 128.0, 126.5, 125.5, 122.4, 120.5, 119.9, 110.9, 109.4, 107.4, 102.9, 75.6, 67.0, 50.3, 47.0, 42.1, 34.1, 32.8, 30.7, 26.1, 20.6; LCMS (ESI) *m/z* calcd for $C_{31}H_{27}N_5O_4$ : 533.2053 [$M^+$]; found: 534.2127 [$M^+$ +H].

**1'-((1-(4-chlorophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-3-hydroxy-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5k)**



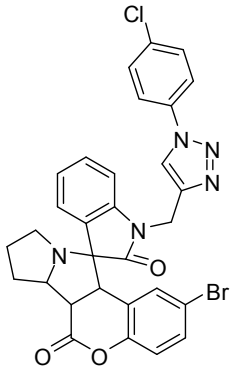

White solid; Yield = 87%; M.p. = 175-177; IR ($CHCl_3$) ν/$cm^{-1}$= 2923, 1712, 1498, 1169, 752; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 9.48 (s, 1H, -OH), 7.87 (s, 1H, triazole), 7.78-7.74 (m, 3H, Ar-H), 7.62 (d, 1H, *J* = 8.77 Hz, Ar-H), 7.40-7.36 (t, 1H, Ar-H), 7.19-7.16 (t, 1H, Ar-H), 7.06 (d, 1H, *J* = 7.63 Hz, Ar-H), 6.32-6.26 (m, 1H, Ar-H), 6.09-6.07 (m, 1H, Ar-H), 5.99 (d, 1H, *J* = 8.39 Hz, Ar-H), 4.68 (s, 2H, -$CH_2$, 4.41-4.37 (m, 1H, -CH), 4.07-4.05 (d, 1H, *J* = 11.4 Hz, -CH), 3.30-3.29 (m, 1H, -$CH_2$), 3.19-3.13 (m, 1H, -$CH_2$), 2.58-2.55 (m, 1H, -$CH_2$), 2.36-2.30 (m, 1H, -$CH_2$), 1.88-1.76 (m, 2H, -$CH_2$), 1.68-1.59 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.5, 167.7, 157.5, 151.4, 143.3, 143.0, 135.0, 132.9, 129.8, 127.9, 126.5, 125.5, 122.4, 121.8, 120.7, 110.8, 109.4, 107.3, 102.8, 75.6, 67.0, 50.3, 46.9, 42.2, 34.0, 32.7, 26.1; LCMS (ESI) *m/z* calcd for $C_{30}H_{24}ClN_5O_4$ : 553.1514 [$M^+$]; found: 554.1587 [$M^+$ +H].

**1'-((1-(7-chloroquinolin-4-yl)-1*H*-1,2,3-triazol-4-yl)methyl)-3-hydroxy-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5l)**



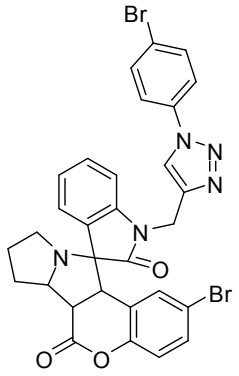

White solid; Yield = 84%; M.p. = 185-187; IR ($CHCl_3$) ν/$cm^{-1}$= 2931, 1712, 1609, 1462, 1160, 753; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 9.19 (s, 1H, -OH), 9.10-9.09 (m, 1H, Ar-H), 8.25 (s, 1H, triazole), 7.80-7.78 (m, 2H, Ar-H), 7.74-7.71 (m, 1H, Ar-H), 7.62-7.61 (m, 1H, Ar-H), 7.53 (s, 1H, Ar-H), 7.43-7.39 (t, 1H, Ar-H), 7.22-7.18 (t,1H, Ar-H), 7.09 (d, 1H, *J* = 7.63 Hz, Ar-H), 6.17 (s, 1H, Ar-H), 5.97-5.88 (m, 2H, Ar-H), 4.86-4.75 (q, 2H, -$CH_2$), 4.47-4.33 (m, 1H, -CH), 4.06 (d, 1H, *J* = 11.4 Hz, -CH), 3.53-3.43 (m, 1H, -$CH_2$), 3.18-3.15 (m, 1H, -$CH_2$), 2.61-2.57 (m, 1H, -$CH_2$), 2.37-2.30 (m, 1H, -$CH_2$), 1.87-1.76 (m, 2H, -$CH_2$), 1.65-1.57 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.6, 167.8, 157.4, 152.4, 151.4, 149.3, 143.3, 142.8, 139.9, 135.2, 129.9, 128.8, 128.1, 127.9, 126.5, 125.4, 125.2, 124.4, 122.5, 120.3, 116.9, 110.9, 109.4, 107.3, 102.6, 75.6, 67.0, 50.4, 46.8, 42.3, 34.2, 32.8, 30.7, 26.1; LCMS (ESI) *m/z* calcd for $C_{33}H_{25}ClN_6O_4$ : 604.1626 [$M^+$]; found: 605.1700 [$M^+$ +H].

**2-bromo-1'-((1-(4-chlorophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5m)**



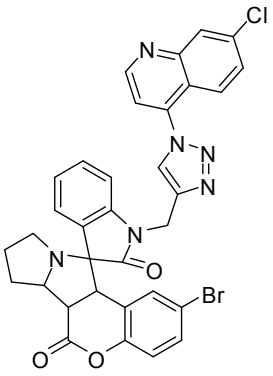

White solid; Yield = 88%; M.p. = 215-216; IR ($CHCl_3$) ν/$cm^{-1}$= 2931, 1760, 1475, 1167, 748; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 7.83 (s, 1H, triazole), 7.80-7.77 (m, 2H, Ar-H), 7.65 (d, 1H, *J* = 8.39 Hz, Ar-H), 7.47-7.43 (t, 1H, Ar-H), 7.23-7.18 (m, 2H, Ar-H), 6.83-6.81 (m, 1H, Ar-H), 6.78-6.76 (m, 1H, Ar-H), 6.60 (s, 1H, Ar-H), 4.75-4.59 (q, 2H, -$CH_2$), 4.43 (brs, 1H, -CH), 4.14 (d, 1H, *J* = 11.4 Hz, -CH), 3.41-3.38 (m, 1H, -$CH_2$), 3.17-3.13 (m, 1H, -$CH_2$), 2.66-2.62 (m, 1H, -$CH_2$), 2.39-2.32 (m, 1H, -$CH_2$), 1.88-1.77 (m, 2H, -$CH_2$), 1.67-1.63 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.0, 167.0, 149.8, 143.1, 142.6, 135.1, 132.9, 130.7, 130.1, 129.7, 129.4, 126.5, 124.9, 122.6, 121.8, 120.8, 119.9, 118.3, 114.4, 109.7, 76.1, 67.6, 50.3, 46.3, 42.8, 33.7, 32.6, 26.0; LCMS (ESI) *m/z* calcd for $C_{30}H_{23}BrClN_5O_3$ : 615.0680 [$M^+$]; found: 616.0754 [$M^+$ +H]; 618.0737 [$M^+$+(H+2)].

**2-bromo-1'-((1-(4-bromophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5n)**



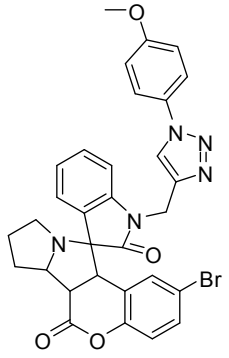

White solid; Yield = 85%; M.p. = 177-179; IR ($CHCl_3$) ν/$cm^{-1}$= 2928, 1758, 1610, 1489, 1223, 748; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 7.85 (s, 1H, triazole), 7.81-7.74 (m, 6H, Ar-H), 7.45-7.43 (t, 1H, ArH), 7.24-7.18 (m, 2H, Ar-H),6.84-6.79 (m, 2H, Ar-H), 6.06 (s, 1H, Ar-H), 4.75-4.59 (q, 2H, -$CH_2$), 4.45-4.41 (m, 1H, -CH), 4.15 (d, 1H, *J* = 11.4 Hz, -CH), 3.42-3.41 (m, 1H, -$CH_2$), 3.19-3.14 (m, 1H, -$CH_2$), 2.66-2.62 (m, 1H, -$CH_2$), 2.39-2.34 (m, 1H, -$CH_2$), 1.89-1.78 (m, 2H, -$CH_2$), 1.70-1.62 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.0, 167.0, 149.9, 143.1, 142.6, 135.5, 132.7, 130.7, 130.1, 129.4, 126.5, 124.9, 122.6, 122.0, 121.3, 120.8, 119.9, 118.3, 114.5, 109.7, 76.1, 67.6, 50.3, 46.3, 42.9, 33.8, 32.6, 26.0; LCMS (ESI) *m/z* calcd for $C_{30}H_{23}Br_2N_5O_3$ : 659.0167 [$M^+$]; found: 660.0246 [$M^+$ +H]; 662.0232 [$M^+$+(H+2)].

**2-bromo-1'-((1-(7-chloroquinolin-4-yl)-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5o)**



White solid; Yield = 86%; M.p. = 174-176; IR ($CHCl_3$) ν/$cm^{-1}$= 2969, 1761, 1609, 1476, 1224, 1174, 754; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 9.15-9.14 (m, 1H, Ar-H), 8.30-8.28(m, 1H, Ar-H), &.90 (d, 1H, *J* = 9.54 Hz, Ar-H), 7.83 (d, 1H, *J* = 7.63 Hz, Ar-H), 7.80-7.77 (dd, 1H, Ar-H), 7.72-7.71 (m, 1H, Ar-H), 7.59 (s, 1H, triazole), 7.49-7.45 (t, 1H, Ar-H), 7.26-7.22 (t, 1H, Ar-H), 7.16 (d, 1H, *J* = 7.63 Hz, Ar-H), 6.76 (d, 1H, *J* = 8.77 Hz, Ar-H), 6.64-6.62 (dd, 1H, Ar-H), 6.17-6.16 (m, 1H, Ar-H), 4.88-4.73 (q, 2H, -$CH_2$), 4.45-4.41 (m, 1H, -CH), 4.20-4.17 (m, 1H, -CH), 3.42-3.40 (m, 1H, -$CH_2$), 3.21-3.15 (m, 1H, -$CH_2$), 2.66-2.63 (m, 1H, -$CH_2$), 2.38-2.33 (m, 1H, -$CH_2$), 1.91-1.78 (m, 2H, -$CH_2$), 1.71-1.61 (m, 1H, -$CH_2$); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.1, 166.9, 152.3, 149.9, 149.3, 143.1, 142.6, 139.9, 135.4, 130.8, 130.2, 129.6, 129.0, 128.1, 126.7, 125.4, 124.8, 124.2, 122.7, 120.1, 120.0, 118.5, 116.8, 114.3, 109.5, 76.0, 67.5, 50.2, 46.3, 42.6, 33.9, 32.7, 26.0; LCMS (ESI) *m/z* calcd for $C_{33}H_{24}BrClN_6O_3$ : 666.0775 [$M^+$]; found: 667.0840 [$M^+$ +H]; 669.0842 [$M^+$+(H+2)].

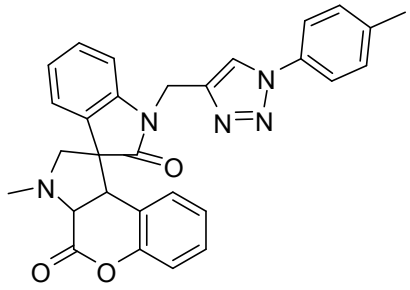
**2-bromo-1'-((1-(4-methoxyphenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-6b,7,8,9-tetrahydro-6*H*-spiro[chromeno[3,4-*a*]pyrrolizine-11,3'-indoline]-2',6(6*aH*,11*aH*)-dione (5p)**




White solid; Yield = 82%; M.p. = 185-187; IR ($CHCl_3$) ν/$cm^{-1}$= 2966, 1710, 1610, 1516, 1170, 750; $^1$H NMR (400MHz, DMSO-$d_6$) $\delta_H$ = 7.79 (d, 1H, *J* = 7.63 Hz,Ar-H), 7.65 (d, 2H, *J* = 8.39 Hz, Ar-H), 7.55 (s, 1H, triazole,) 7.46-7.42 (t, 1H, Ar-H), 7.24-7.20 (t, 1H, Ar-H), 7.16 (d, 1H,

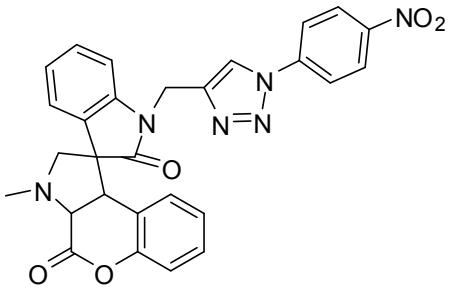
*J* = 8.39 Hz, Ar-H), 7.12 (d, 1H, *J* = 8.39 Hz, Ar-H), 6.84 (d, 1H, *J* = 8.39 Hz, Ar-H), 6.80-6.77 (dd, 1H, Ar-H), 6.11-6.10 (m, 1H, Ar-H), 4.75-4.59 (q, 2H, -$CH_2$), 4.45-4.41 (m, 1H, -CH), 4.17-4.15 (m, 1H, -CH), 3.82 (s, 3H, -$OCH_3$), 3.42-3.38 (dd, 1H, -$CH_2$), 3.20-3.14 (m, 1H, -$CH_2$), 2.66-2.62 (t, 1H, -$CH_2$), 2.39-2.33 (m, 1H, -$CH_2$), 1.90-1.78 (m, 2H, -$CH_2$), 1.71-1.62 (m, 1H, -CH) $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta_C$ = 175.0, 166.9, 159.2, 149.9, 143.2, 142.3, 130.8, 130.1, 129.7, 129.5, 126.5, 124.8, 122.6, 121.9, 120.5, 119.8, 118.3, 114.7, 114.4, 109.6, 76.1, 67.5, 55.5, 50.2, 46.3, 42.7, 33.9, 32.5, 30.6, 25.9; LCMS (ESI) *m/z* calcd for $C_{33}H_{24}BrClN_6O_3$ : 611.1161 [$M^+$]; found: 612.1236 [$M^+$ +H]; 614.1219 [$M^+$+(H+2)].

**2-methyl-1'-((1-(4-methylphenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-3,3*a*-dihydro-2*H*-spiro[chromeno[3,4-*c*]pyrrole-1,3'-indoline]-2',4(9*bH*)-dione (5q)**



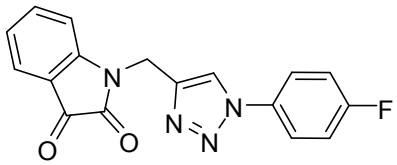

Off-White solid; M.p.: 193-196 $^oC$; Yield 71%; IR ($CHCl_3$, $cm^{-1}$) ν/max: 3017, 1713, 1213, 739; $^1$H NMR (400 MHz, DMSO, $d_6$) $\delta_H$: 8.47 (s, 1H, ArH), 7.69-7.67 (m, 2H, ArH), 7.42-7.41(m, 1H, ArH), 7.37-7.32 (m, 3H, ArH), 7.27-7.25 (m, 2H, ArH),7.19-7.18 (m, 1H, ArH), 7.18-7.04 (m, 2H, ArH), 7.0-7.08 (m, 1H, ArH), 4.99-4.88 (m, 2H, N-$CH_2$), 4.20-4.17 (m, 1H, CH), 4.09- 4.04 (m, 1H, CH), 3.69-3.65 (m, 1H, CH), 3.26-3.22 (m, 1H, CH), 2.34 (s, 3H, $CH_3$), 1.91 (s, 3H, $CH_3$); $^{13}$C NMR (100 MHz, $CDCl_3$) $^{13}$C: 175.2, 166.3, 149.1, 143.1, 138.4, 134.2, 130.2, 128.4, 128.3, 128.0, 124.7, 124.0, 123.0, 122.0, 121.2, 119.8, 116.3, 109.2, 74.5, 72.3, 60.4, 60.1, 49.2, 33.8, 20.5; LCMS (ESI) m/z calcd. for $C_{29}H_{25}N_5O_3$: 491.1957, LCMS (ESI) found: 492.2036 [M+H]$^+$.

**2-methyl-1'-((1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)-3,3*a*-dihydro-2*H*-spiro[chromeno[3,4-*c*]pyrrole-1,3'-indoline]-2',4(9*bH*)-dione (5r).**



Pale yellow solid; M.p.: 208-210 $^{o}$C; Yield 78%; IR ($CHCl_3$, $cm^{-1}$) ν/max: 2925, 1718, 1347, 754; $^{1}$H NMR (400 MHz, DMSO, $d_6$) $\delta_H$: 8.79 (s, 1H, ArH), 8.43-8.41 (m, 2H, ArH), 8.15-8.13 (m, 2H, ArH), 7.41-7.27(m, 4H, ArH), 7.17-7.09(m, 4H, ArH), 5.04-4.90(m, 2H, N-$CH_2$), 4.20-4.06(m, 2H, $CH_2$), 3.75-3.59 (m, 1H, CH), 3.26-3.24 (m, 1H, CH), 1.91 (s, 3H, $CH_3$); $^{13}$C NMR (100 MHz, DMS, $d_6$) $^{13}$C: 175.2, 166.3, 149.1, 146.7, 143.8, 143.0, 140.0, 129.5, 128.4, 128.3, 128.0, 125.6, 124.7, 121.9, 120.5, 109.2, 74.5, 72.3, 60.4, 60.1, 49.2, 33.9; LCMS (ESI) m/z calcd. for $C_{28}H_{22}N_6O_5$: 352.1652 [$M^+$]; found: 523.1734 [M+H]$^+$.

**1-((1-(4-fluorophenyl)-1*H*-1,2,3-triazol-4-yl)methyl)indoline-2,3-dione (6a)**



Yellow solid; M.p.: 174-177 $^{o}$C; Yield 82%; IR ($CHCl_3$ , $cm^{-1}$) ν/max: 2923, 1737, 1611, 1231; $^{1}$H NMR (400 MHz, $CDCl_3$) $\delta_H$: 8.01 (s, 1H, ArH), 7.67-7.56 (m, 4H, ArH), 7.35-7.33 (m, 2H, ArH), 7.18-7.08 (m, 3H, ArH), 5.08 (s, 2H, $OCH_2$); $^{13}$C NMR (100 MHz, $CDCl_3$) $^{13}$C: 182.9, 150.0, 138.6, 125.4, 124.1, 122.5, 122.4, 117.5, 116.9, 116.7, 111.4, 35.3; LCMS (ESI) m/z calcd. for $C_{17}H_{11}FN_4O_2$: 322.08, LCMS (ESI) found: 323.0939 [M+H]$^+$.

**Figure 1.** $^{1}$H NMR spectrum of compound **5a** (400 MHz, DMSO).

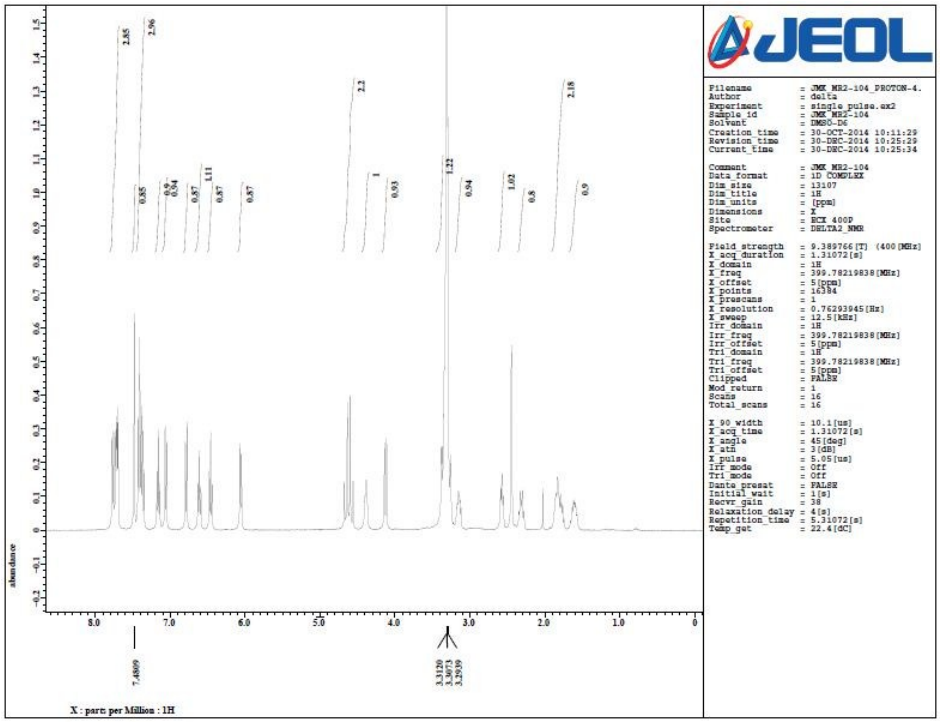


**Figure 2.** $^{13}$C NMR spectrum of compound **5a** (100 MHz, DMSO).

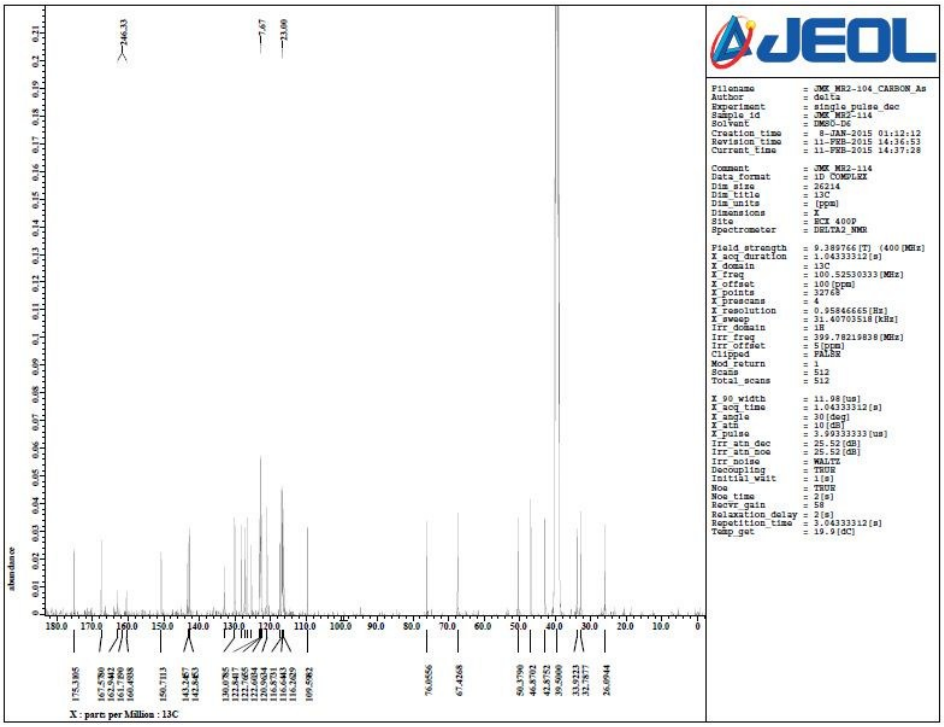
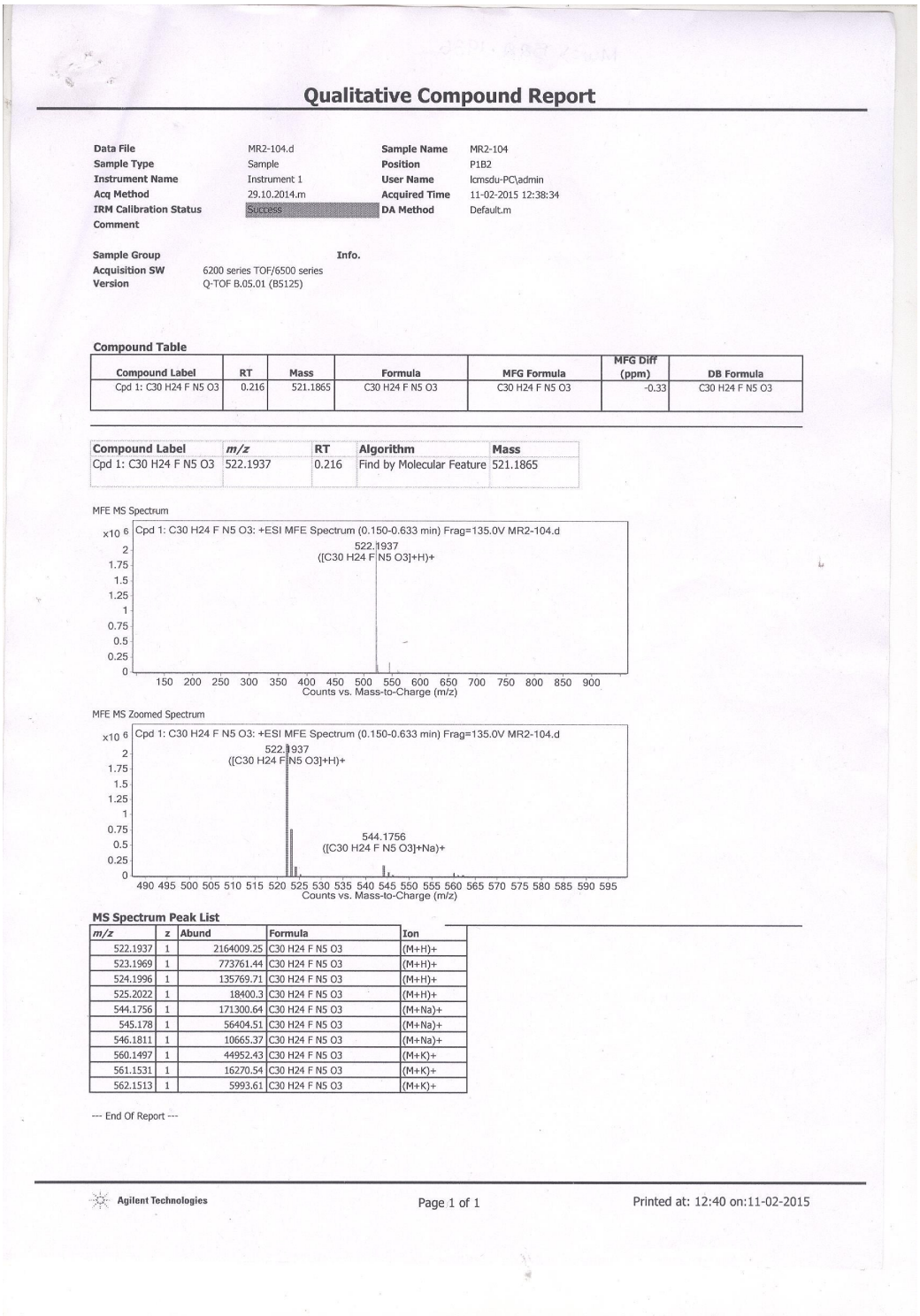


**Figure 3.** Mass spectrum of compound **5a**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2-104.d | **Sample Name** | MR2-104 |
| **Sample Type** | Sample | **Position** | P1B2 |
| **Instrument Name** | Instrument 1 | **User Name** | lcmsdu-PC\admin |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 11-02-2015 12:38:34 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Sample Group** **Info.**
**Acquisition SW** 6200 series TOF/6500 series
**Version** Q-TOF B.05.01 (B5125)

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C30 H24 F N5 O3 | 0.216 | 521.1865 | C30 H24 F N5 O3 | C30 H24 F N5 O3 | -0.33 | C30 H24 F N5 O3 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C30 H24 F N5 O3 | 522.1937 | 0.216 | Find by Molecular Feature | 521.1865 |

MFE MS Spectrum



MFE MS Zoomed Spectrum



**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 522.1937 | 1 | 2164009.25 | C30 H24 F N5 O3 | (M+H)+ |
| 523.1969 | 1 | 773761.44 | C30 H24 F N5 O3 | (M+H)+ |
| 524.1996 | 1 | 135769.71 | C30 H24 F N5 O3 | (M+H)+ |
| 525.2022 | 1 | 18400.3 | C30 H24 F N5 O3 | (M+H)+ |
| 544.1756 | 1 | 171300.64 | C30 H24 F N5 O3 | (M+Na)+ |
| 545.178 | 1 | 56404.51 | C30 H24 F N5 O3 | (M+Na)+ |
| 546.1811 | 1 | 10665.37 | C30 H24 F N5 O3 | (M+Na)+ |
| 560.1497 | 1 | 44952.43 | C30 H24 F N5 O3 | (M+K)+ |
| 561.1531 | 1 | 16270.54 | C30 H24 F N5 O3 | (M+K)+ |
| 562.1513 | 1 | 5993.61 | C30 H24 F N5 O3 | (M+K)+ |

--- End Of Report ---

**Figure 4.** $^{1}$H NMR spectrum of compound **5b** (400 MHz, DMSO).

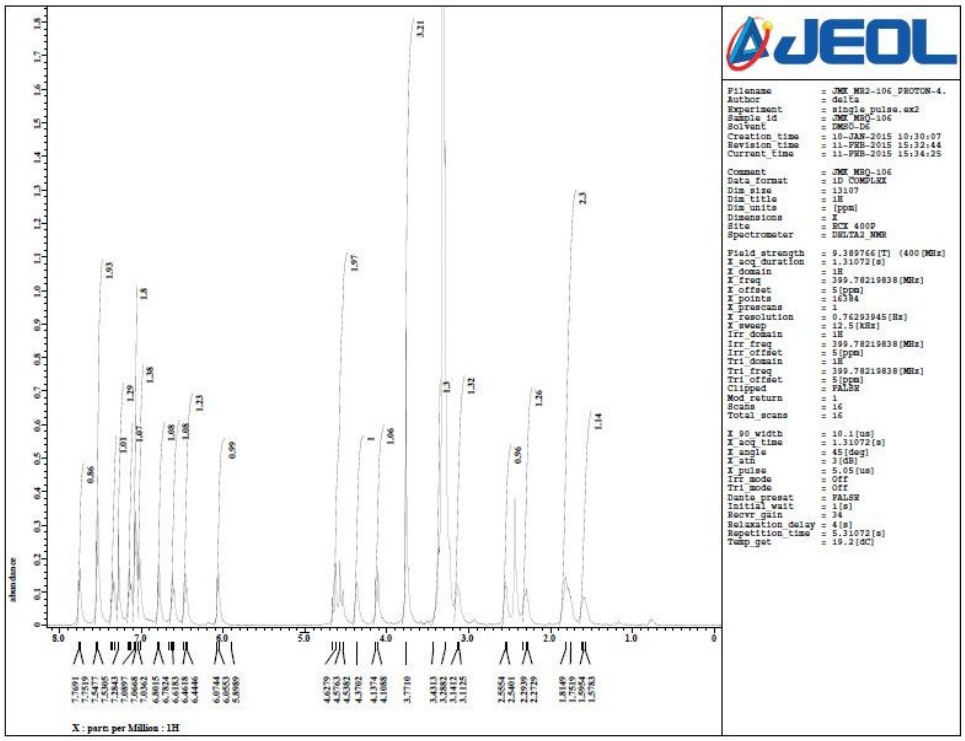



**Figure 5.** $^{13}$C NMR spectrum of compound **5b** (100 MHz, DMSO).

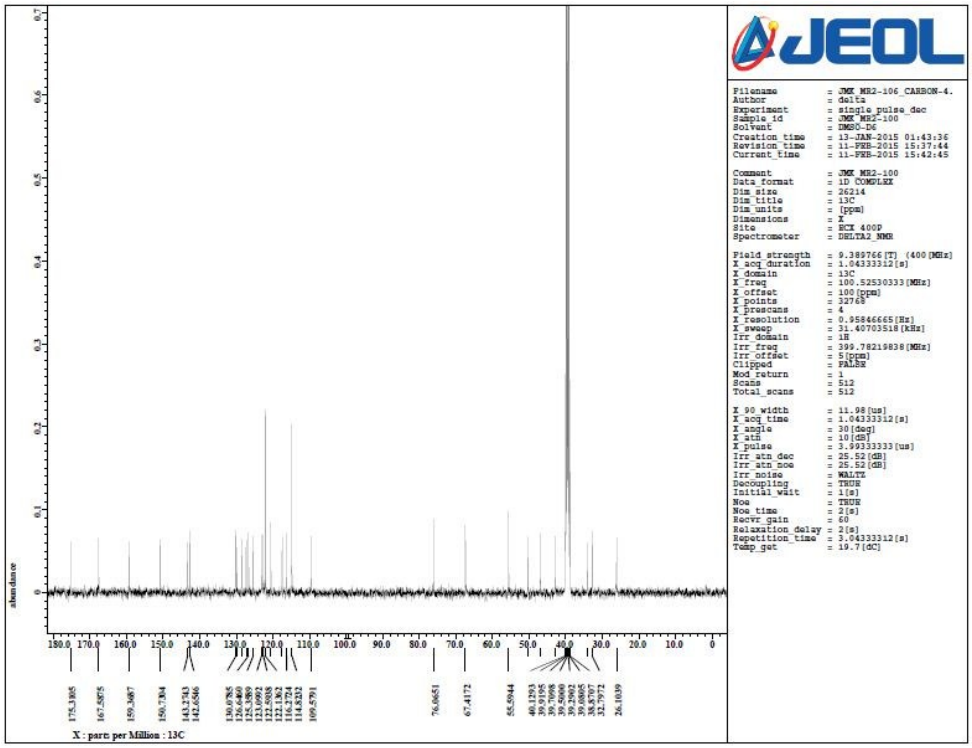
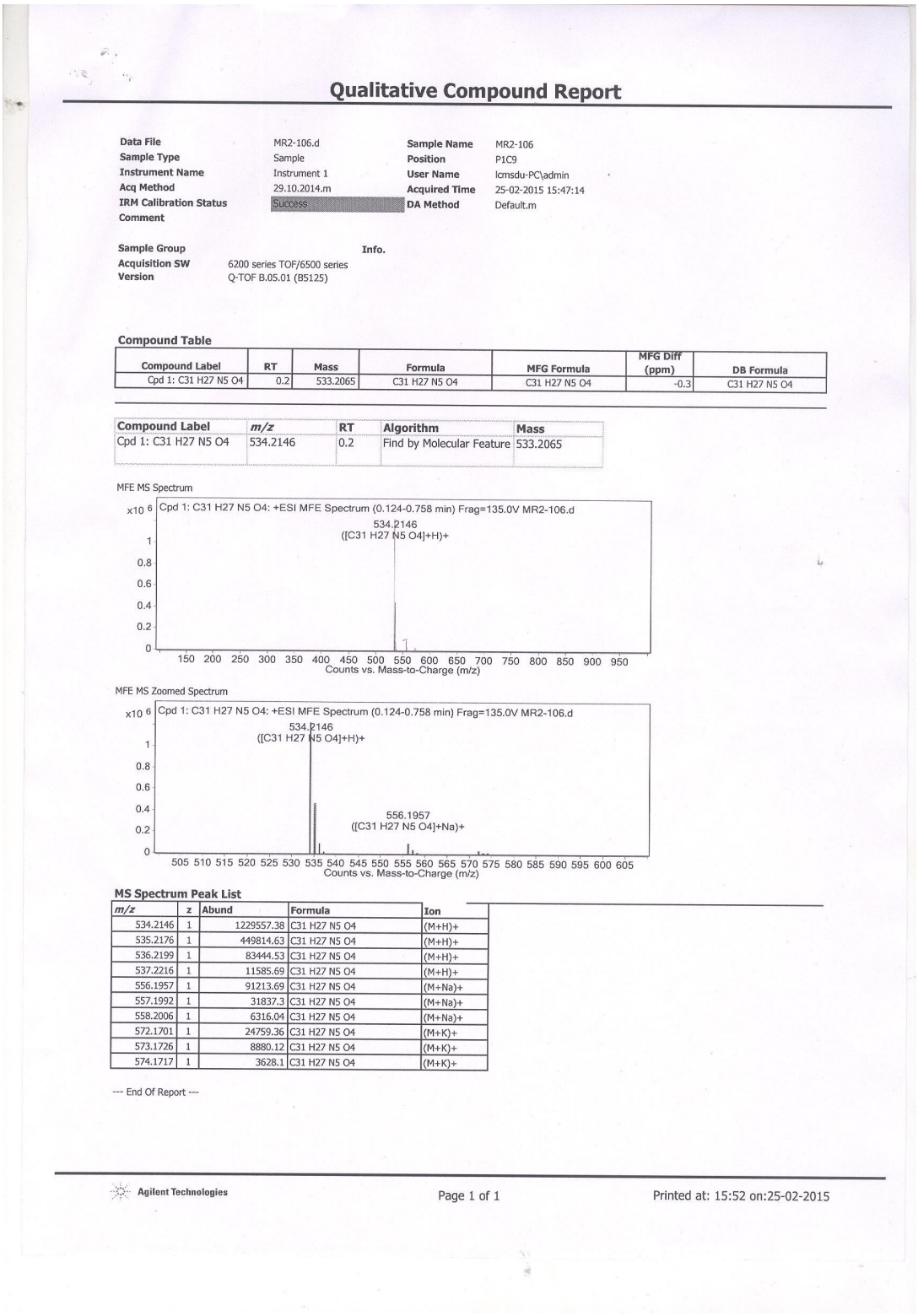


**Figure 6.** Mass spectrum of compound **5b**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2-106.d | **Sample Name** | MR2-106 |
| **Sample Type** | Sample | **Position** | P1C9 |
| **Instrument Name** | Instrument 1 | **User Name** | lcmsdu-PC\admin |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 25-02-2015 15:47:14 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

| | |
|---|---|
| **Sample Group** | **Info.** |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125) |

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C31 H27 N5 O4 | 0.2 | 533.2065 | C31 H27 N5 O4 | C31 H27 N5 O4 | -0.3 | C31 H27 N5 O4 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C31 H27 N5 O4 | 534.2146 | 0.2 | Find by Molecular Feature | 533.2065 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 534.2146 | 1 | 1229557.38 | C31 H27 N5 O4 | (M+H)+ |
| 535.2176 | 1 | 449814.63 | C31 H27 N5 O4 | (M+H)+ |
| 536.2199 | 1 | 83444.53 | C31 H27 N5 O4 | (M+H)+ |
| 537.2216 | 1 | 11585.69 | C31 H27 N5 O4 | (M+H)+ |
| 556.1957 | 1 | 91213.69 | C31 H27 N5 O4 | (M+Na)+ |
| 557.1992 | 1 | 31837.3 | C31 H27 N5 O4 | (M+Na)+ |
| 558.2006 | 1 | 6316.04 | C31 H27 N5 O4 | (M+Na)+ |
| 572.1701 | 1 | 24759.36 | C31 H27 N5 O4 | (M+K)+ |
| 573.1726 | 1 | 8880.12 | C31 H27 N5 O4 | (M+K)+ |
| 574.1717 | 1 | 3628.1 | C31 H27 N5 O4 | (M+K)+ |

--- End Of Report ---

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**Figure 7.** $^{1}$H NMR spectrum of compound **5c** (400 MHz, DMSO).

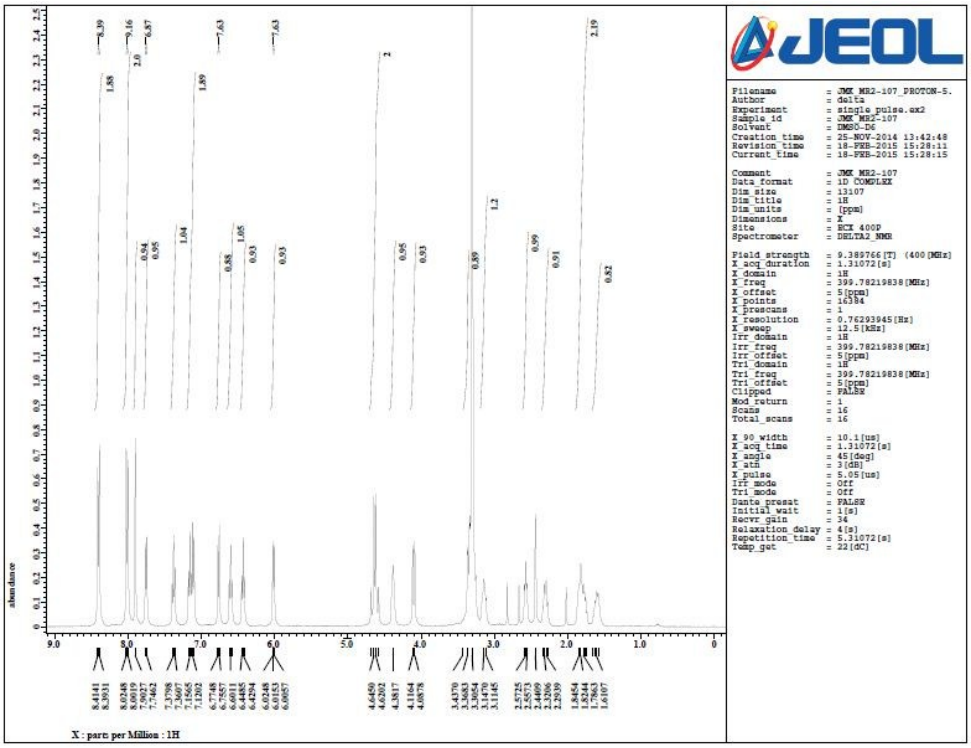


**Figure 8.** $^{13}$C NMR spectrum of compound **5c** (100 MHz, DMSO).

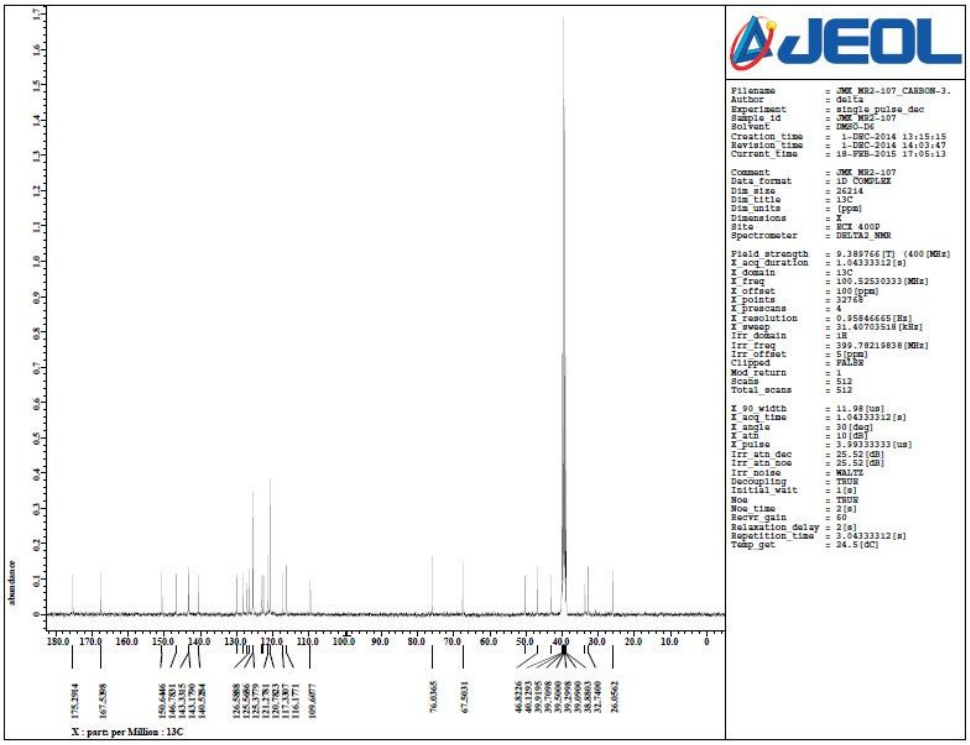
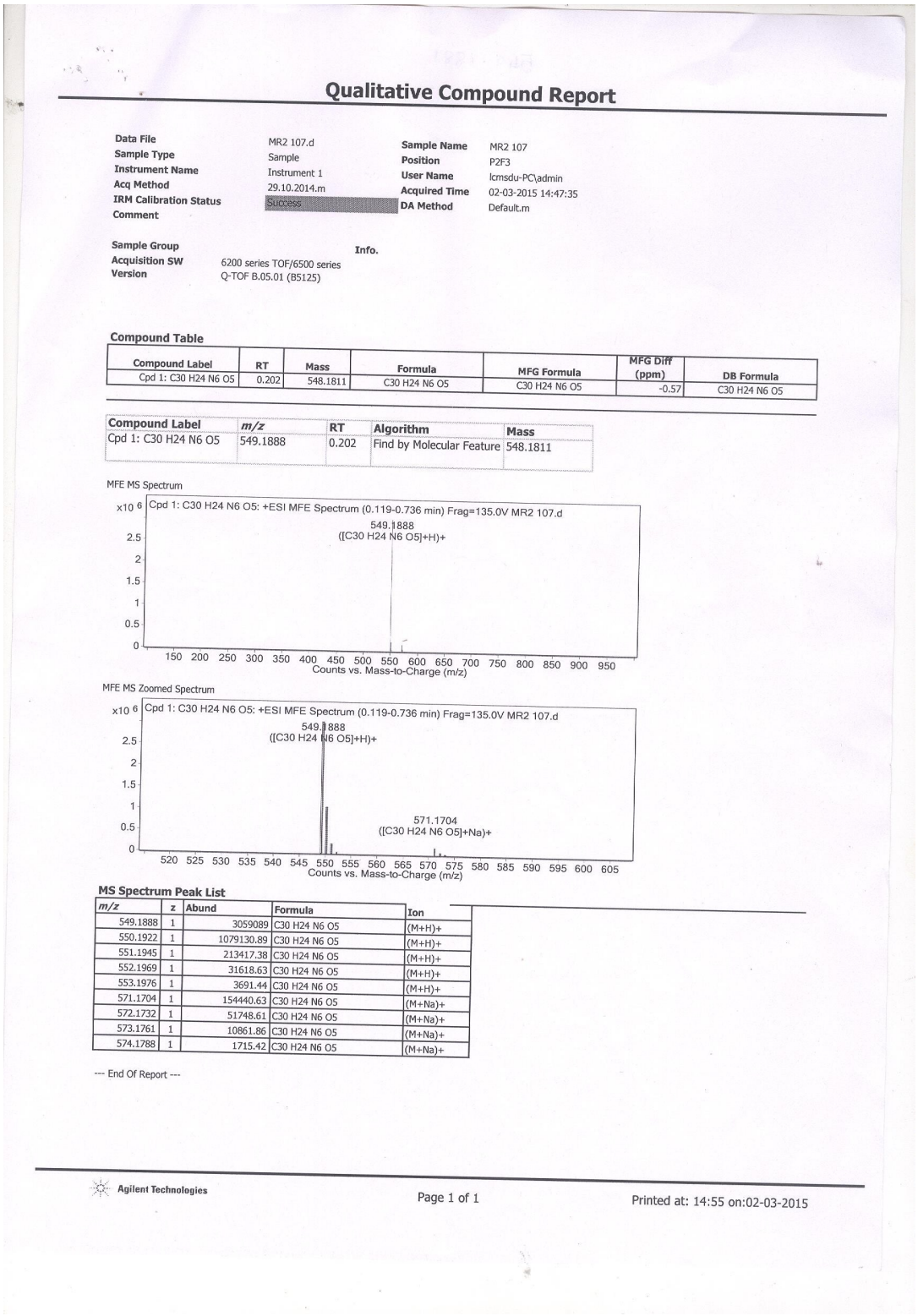


**Figure 9.** Mass spectrum of compound **5c**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2 107.d | **Sample Name** | MR2 107 |
| **Sample Type** | Sample | **Position** | P2F3 |
| **Instrument Name** | Instrument 1 | **User Name** | lcmsdu-PC\admin |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 02-03-2015 14:47:35 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

| | |
|---|---|
| **Sample Group** | **Info.** |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125) |

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C30 H24 N6 O5 | 0.202 | 548.1811 | C30 H24 N6 O5 | C30 H24 N6 O5 | -0.57 | C30 H24 N6 O5 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C30 H24 N6 O5 | 549.1888 | 0.202 | Find by Molecular Feature | 548.1811 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 549.1888 | 1 | 3059089 | C30 H24 N6 O5 | (M+H)+ |
| 550.1922 | 1 | 1079130.89 | C30 H24 N6 O5 | (M+H)+ |
| 551.1945 | 1 | 213417.38 | C30 H24 N6 O5 | (M+H)+ |
| 552.1969 | 1 | 31618.63 | C30 H24 N6 O5 | (M+H)+ |
| 553.1976 | 1 | 3691.44 | C30 H24 N6 O5 | (M+H)+ |
| 571.1704 | 1 | 154440.63 | C30 H24 N6 O5 | (M+Na)+ |
| 572.1732 | 1 | 51748.61 | C30 H24 N6 O5 | (M+Na)+ |
| 573.1761 | 1 | 10861.86 | C30 H24 N6 O5 | (M+Na)+ |
| 574.1788 | 1 | 1715.42 | C30 H24 N6 O5 | (M+Na)+ |

--- End Of Report ---

**Figure 10.** $^{1}$H NMR spectrum of compound **5d** (400 MHz, DMSO).

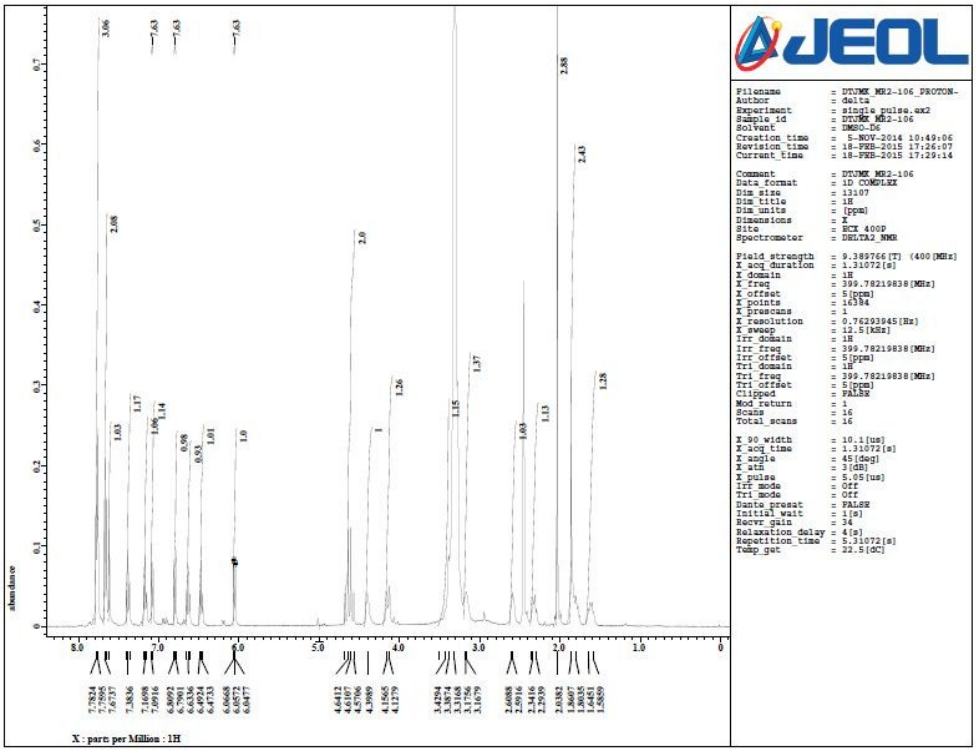


**Figure 11.** $^{13}$C NMR spectrum of compound **5d** (100 MHz, DMSO).

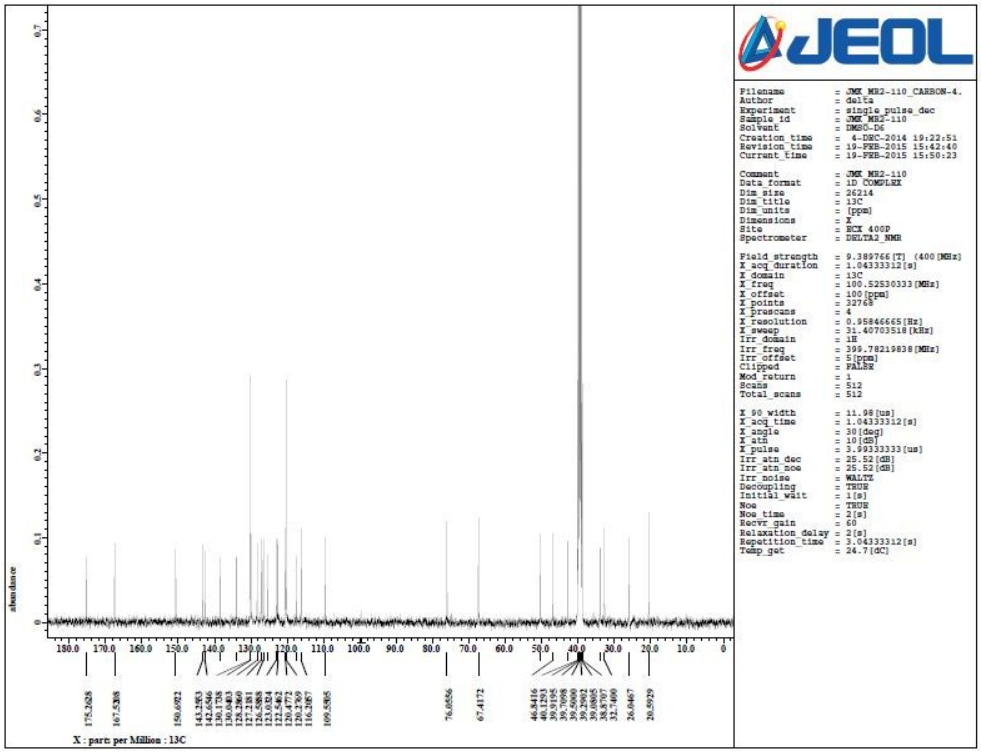
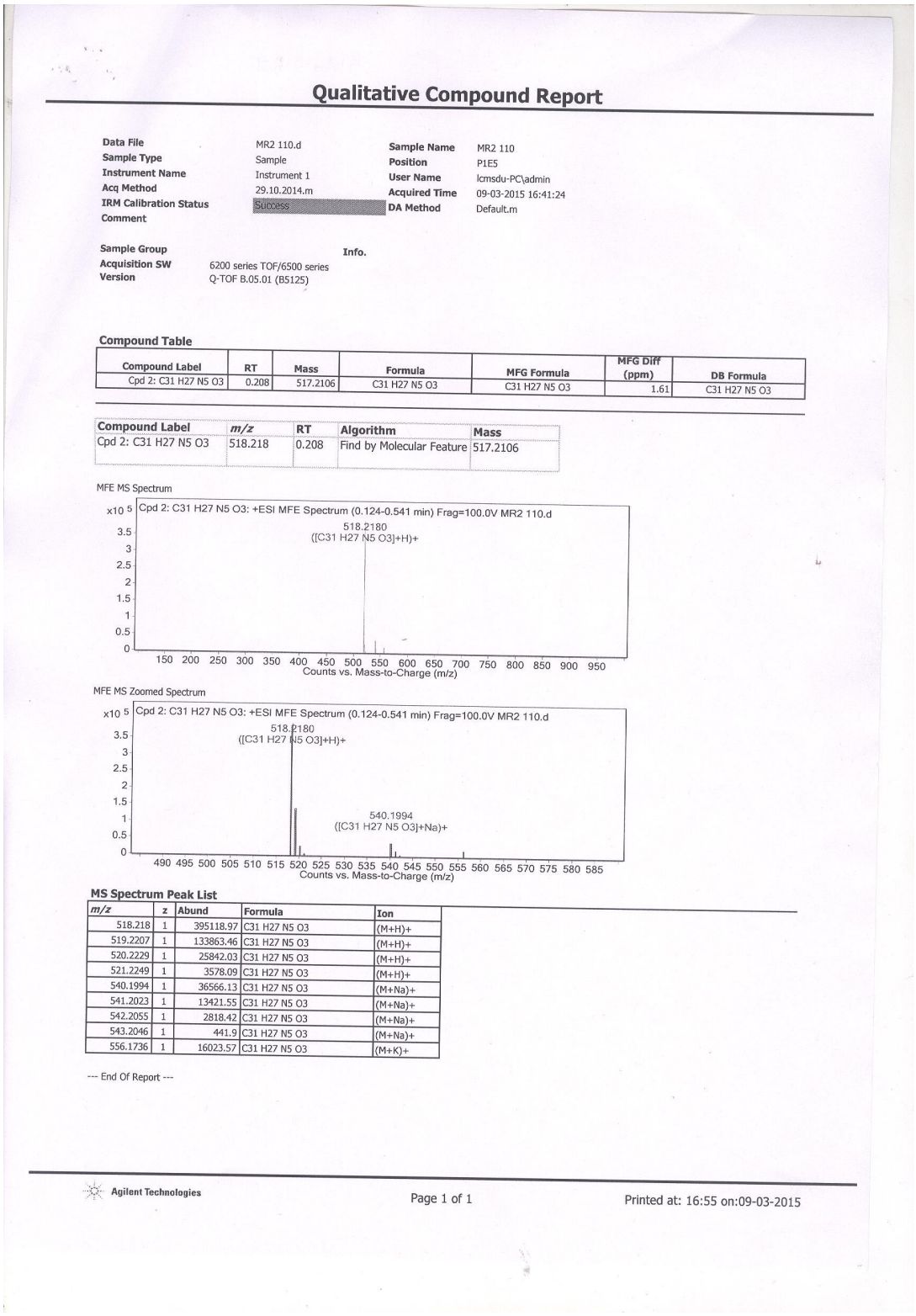


**Figure 12.** Mass spectrum of compound **5d**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2 110.d | **Sample Name** | MR2 110 |
| **Sample Type** | Sample | **Position** | P1E5 |
| **Instrument Name** | Instrument 1 | **User Name** | lcmsdu-PC\admin |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 09-03-2015 16:41:24 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

| | |
|---|---|
| **Sample Group** | **Info.** |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125) |

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 2: C31 H27 N5 O3 | 0.208 | 517.2106 | C31 H27 N5 O3 | C31 H27 N5 O3 | 1.61 | C31 H27 N5 O3 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 2: C31 H27 N5 O3 | 518.218 | 0.208 | Find by Molecular Feature | 517.2106 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 518.218 | 1 | 395118.97 | C31 H27 N5 O3 | (M+H)+ |
| 519.2207 | 1 | 133863.46 | C31 H27 N5 O3 | (M+H)+ |
| 520.2229 | 1 | 25842.03 | C31 H27 N5 O3 | (M+H)+ |
| 521.2249 | 1 | 3578.09 | C31 H27 N5 O3 | (M+H)+ |
| 540.1994 | 1 | 36566.13 | C31 H27 N5 O3 | (M+Na)+ |
| 541.2023 | 1 | 13421.55 | C31 H27 N5 O3 | (M+Na)+ |
| 542.2055 | 1 | 2818.42 | C31 H27 N5 O3 | (M+Na)+ |
| 543.2046 | 1 | 441.9 | C31 H27 N5 O3 | (M+Na)+ |
| 556.1736 | 1 | 16023.57 | C31 H27 N5 O3 | (M+K)+ |

--- End Of Report ---

**Figure 13.** $^{1}$H NMR spectrum of compound **5e** (400 MHz, DMSO).

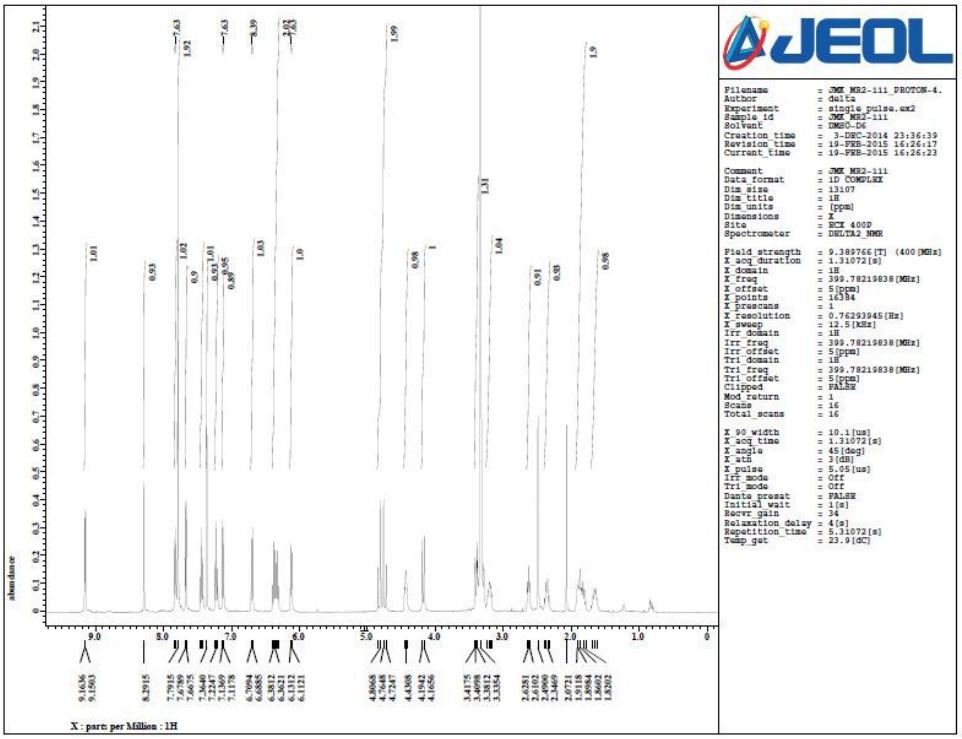



**Figure 14.** $^{13}$C NMR spectrum of compound **5e** (100 MHz, DMSO).

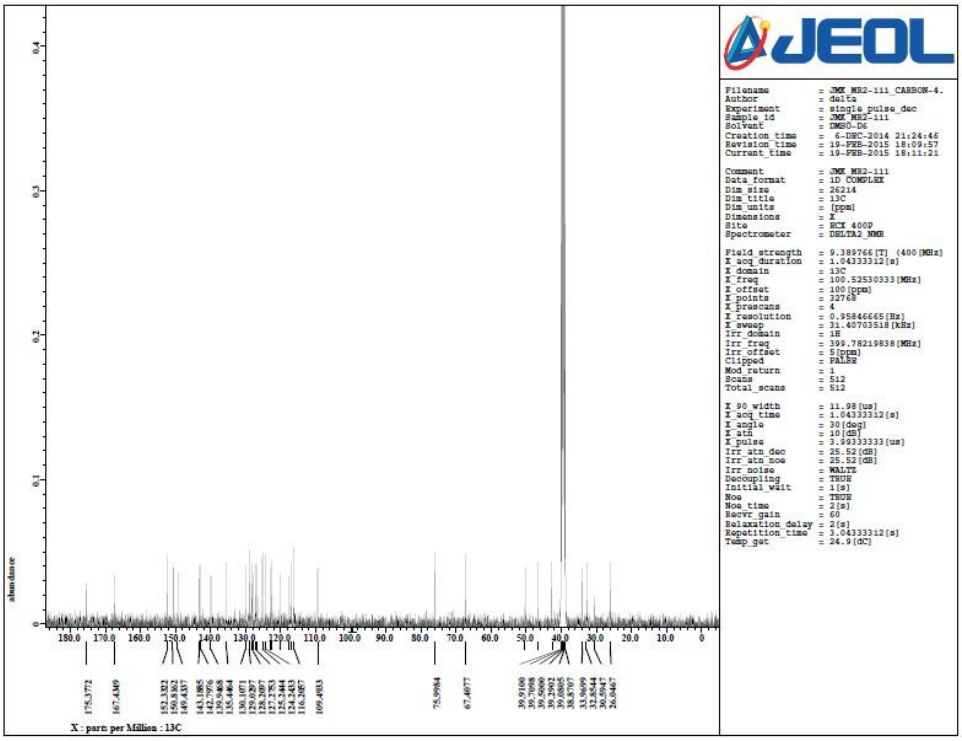
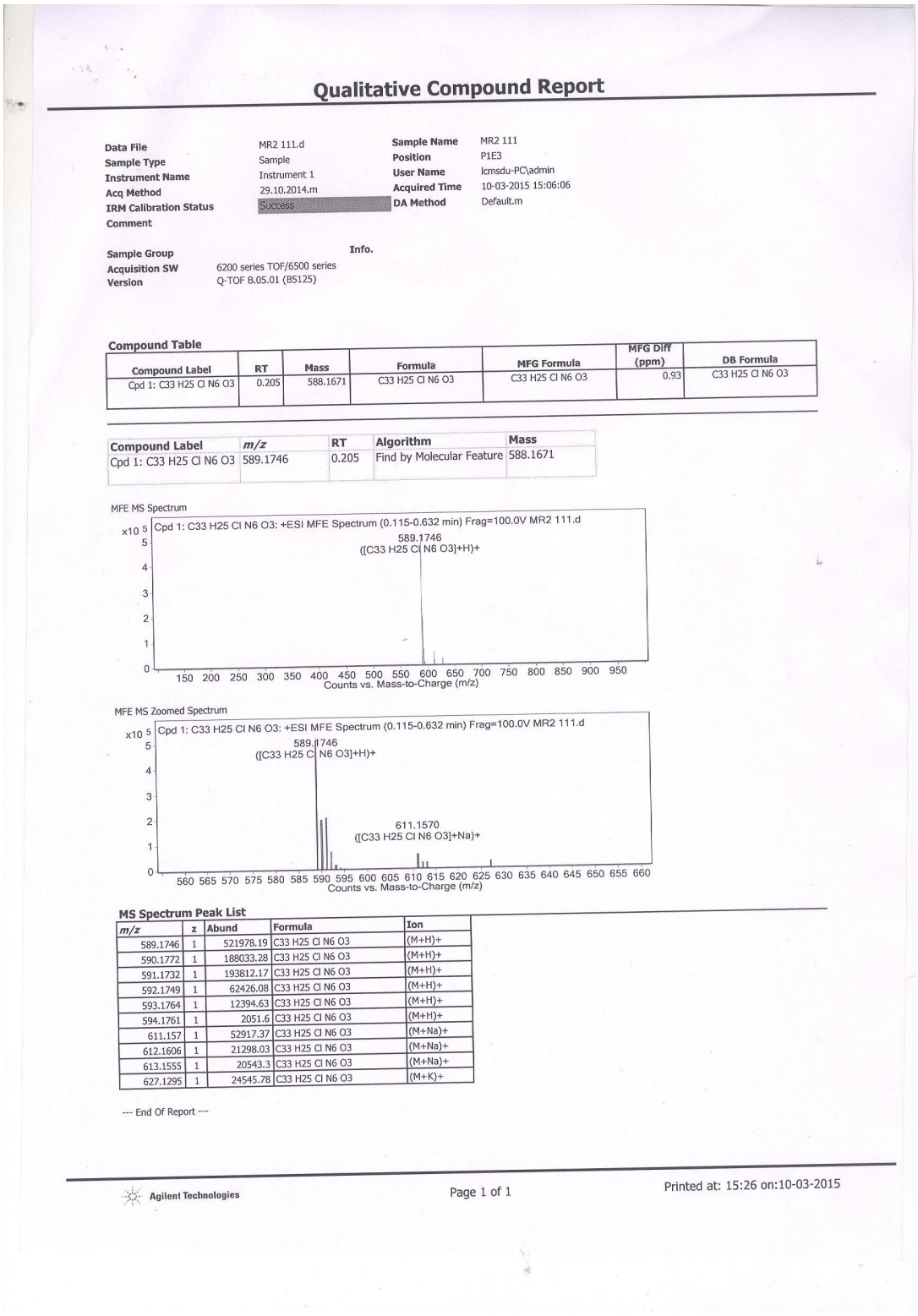


**Figure 15.** Mass spectrum of compound **5e**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2 111.d | **Sample Name** | MR2 111 |
| **Sample Type** | Sample | **Position** | P1E3 |
| **Instrument Name** | Instrument 1 | **User Name** | lcmsdu-PC\admin |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 10-03-2015 15:06:06 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Info.**

| | |
|---|---|
| **Sample Group** | |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125) |

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C33 H25 Cl N6 O3 | 0.205 | 588.1671 | C33 H25 Cl N6 O3 | C33 H25 Cl N6 O3 | 0.93 | C33 H25 Cl N6 O3 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C33 H25 Cl N6 O3 | 589.1746 | 0.205 | Find by Molecular Feature | 588.1671 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 589.1746 | 1 | 521978.19 | C33 H25 Cl N6 O3 | (M+H)+ |
| 590.1772 | 1 | 188033.28 | C33 H25 Cl N6 O3 | (M+H)+ |
| 591.1732 | 1 | 193812.17 | C33 H25 Cl N6 O3 | (M+H)+ |
| 592.1749 | 1 | 62426.08 | C33 H25 Cl N6 O3 | (M+H)+ |
| 593.1764 | 1 | 12394.63 | C33 H25 Cl N6 O3 | (M+H)+ |
| 594.1761 | 1 | 2051.6 | C33 H25 Cl N6 O3 | (M+H)+ |
| 611.157 | 1 | 52917.37 | C33 H25 Cl N6 O3 | (M+Na)+ |
| 612.1606 | 1 | 21298.03 | C33 H25 Cl N6 O3 | (M+Na)+ |
| 613.1555 | 1 | 20543.3 | C33 H25 Cl N6 O3 | (M+Na)+ |
| 627.1295 | 1 | 24545.78 | C33 H25 Cl N6 O3 | (M+K)+ |

--- End Of Report ---

**Figure 16.** $^{1}$H NMR spectrum of compound **5f** (400 MHz, DMSO).

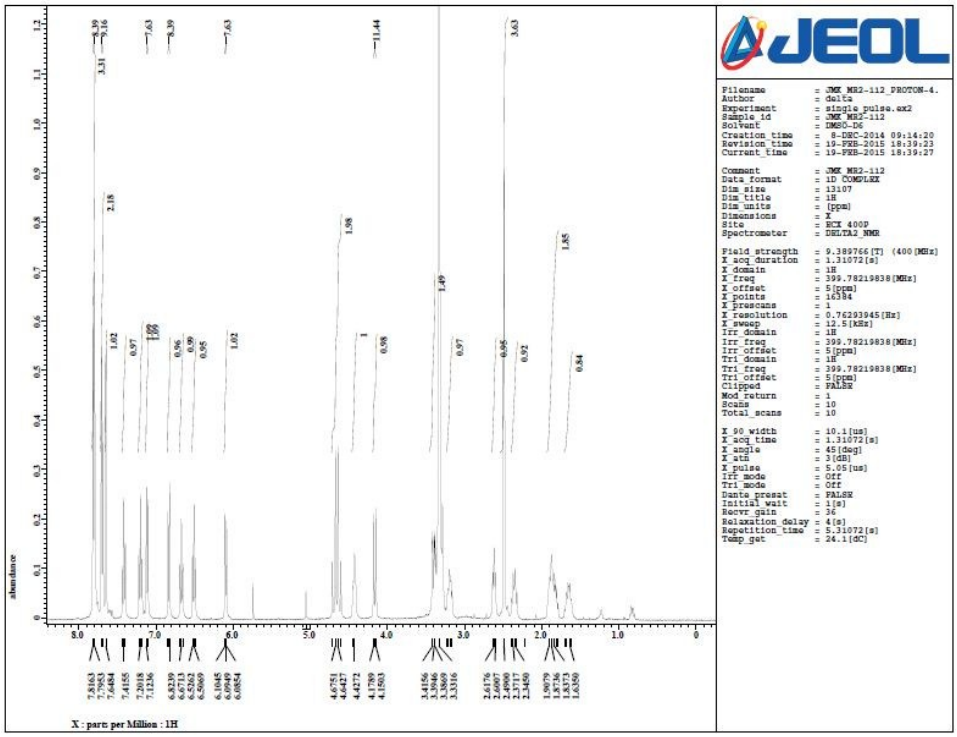



**Figure 17.** $^{13}$C NMR spectrum of compound **5f** (100 MHz, DMSO).

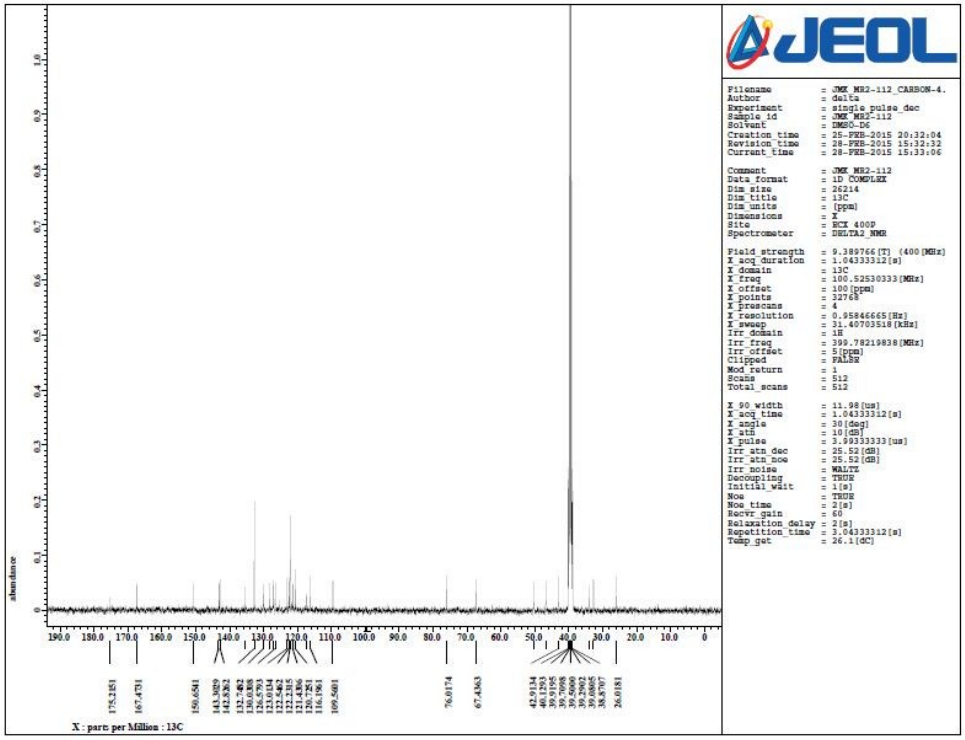
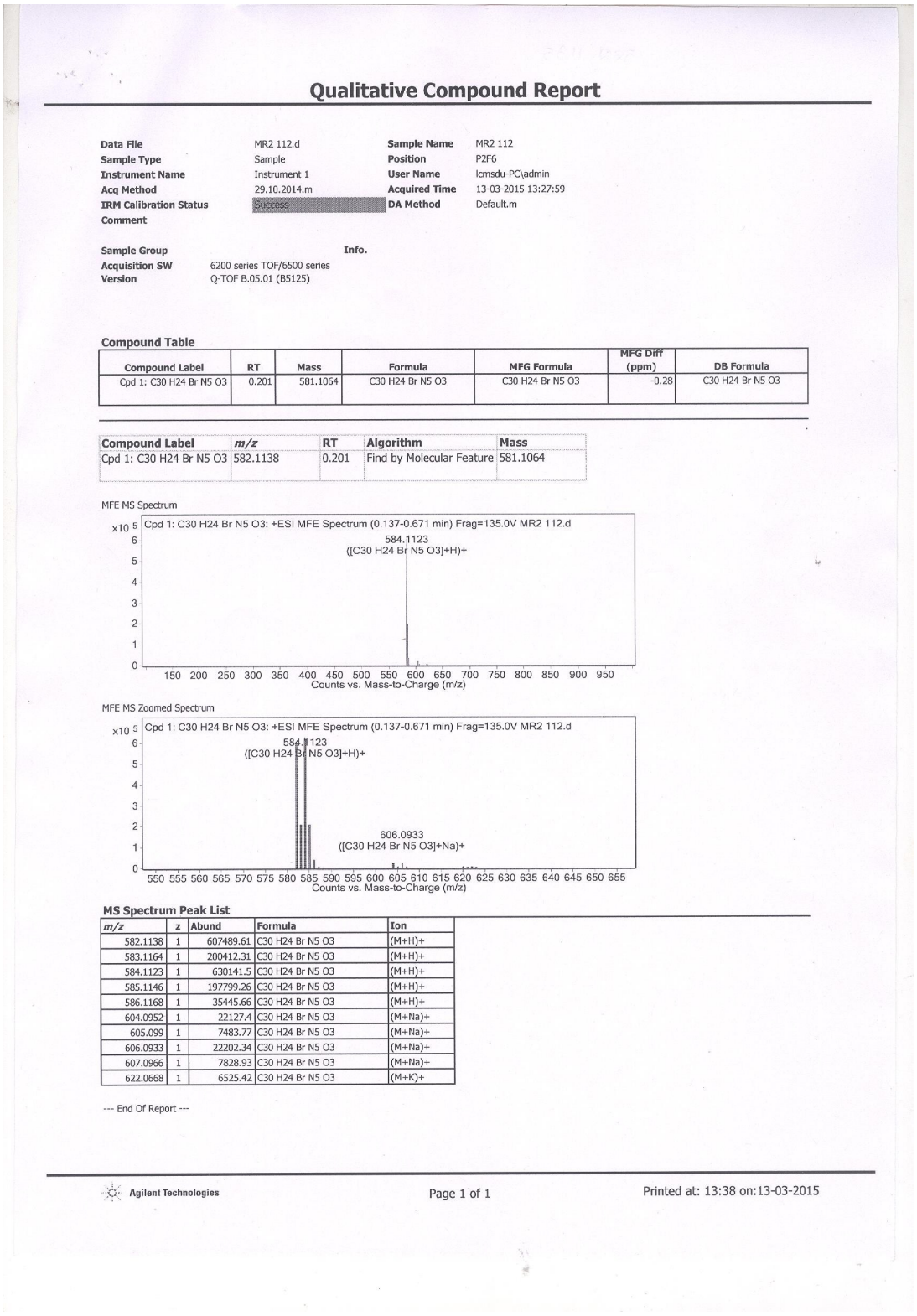


**Figure 18.** Mass spectrum of compound **5f**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2 112.d | **Sample Name** | MR2 112 |
| **Sample Type** | Sample | **Position** | P2F6 |
| **Instrument Name** | Instrument 1 | **User Name** | lcmsdu-PC\admin |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 13-03-2015 13:27:59 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Sample Group** **Info.**
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C30 H24 Br N5 O3 | 0.201 | 581.1064 | C30 H24 Br N5 O3 | C30 H24 Br N5 O3 | -0.28 | C30 H24 Br N5 O3 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C30 H24 Br N5 O3 | 582.1138 | 0.201 | Find by Molecular Feature | 581.1064 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 582.1138 | 1 | 607489.61 | C30 H24 Br N5 O3 | (M+H)+ |
| 583.1164 | 1 | 200412.31 | C30 H24 Br N5 O3 | (M+H)+ |
| 584.1123 | 1 | 630141.5 | C30 H24 Br N5 O3 | (M+H)+ |
| 585.1146 | 1 | 197799.26 | C30 H24 Br N5 O3 | (M+H)+ |
| 586.1168 | 1 | 35445.66 | C30 H24 Br N5 O3 | (M+H)+ |
| 604.0952 | 1 | 22127.4 | C30 H24 Br N5 O3 | (M+Na)+ |
| 605.099 | 1 | 7483.77 | C30 H24 Br N5 O3 | (M+Na)+ |
| 606.0933 | 1 | 22202.34 | C30 H24 Br N5 O3 | (M+Na)+ |
| 607.0966 | 1 | 7828.93 | C30 H24 Br N5 O3 | (M+Na)+ |
| 622.0668 | 1 | 6525.42 | C30 H24 Br N5 O3 | (M+K)+ |

--- End Of Report ---

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**Figure 19.** [1]H NMR spectrum of compound **5g** (400 MHz, DMSO).

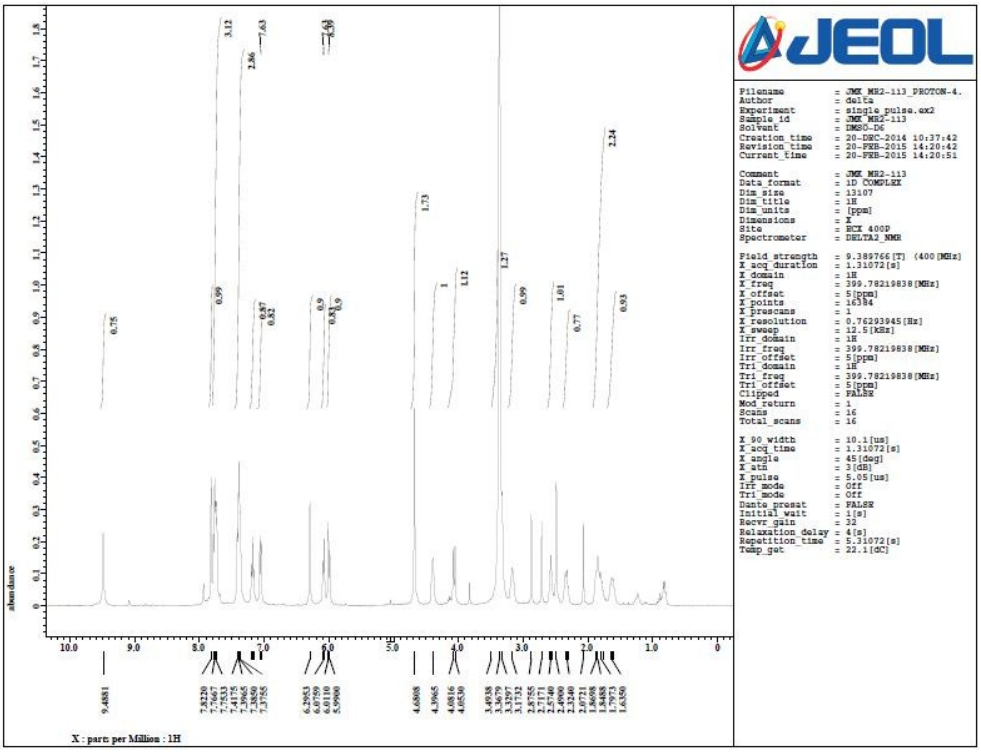


**Figure 20.** [13]C NMR spectrum of compound **5g** (100 MHz, DMSO).

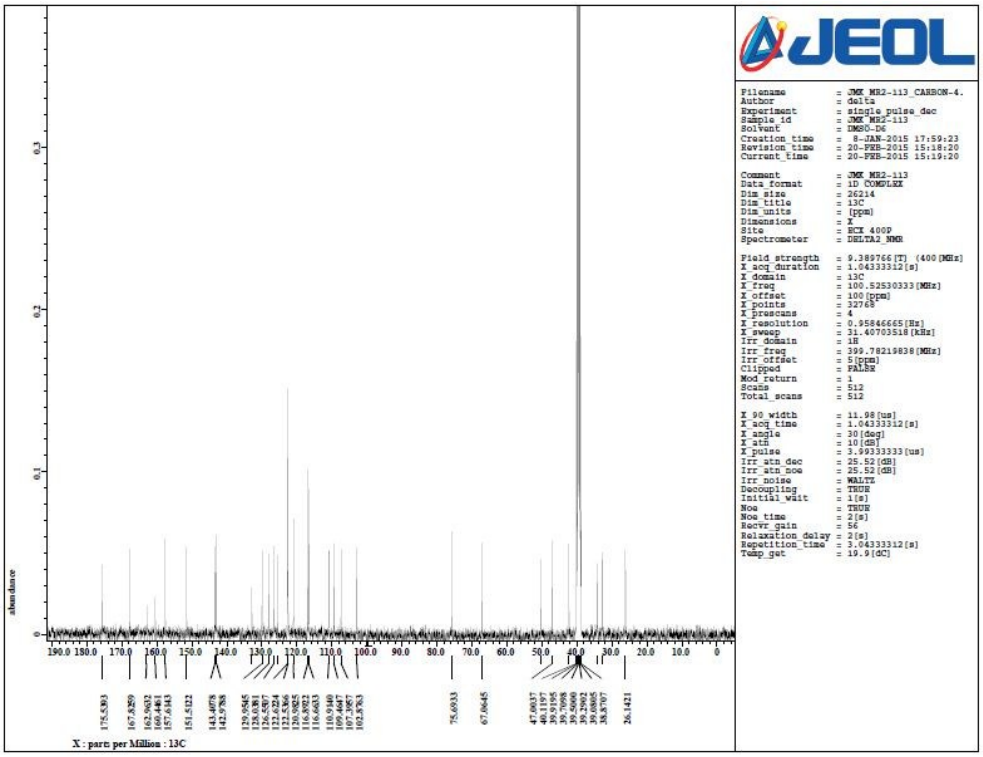
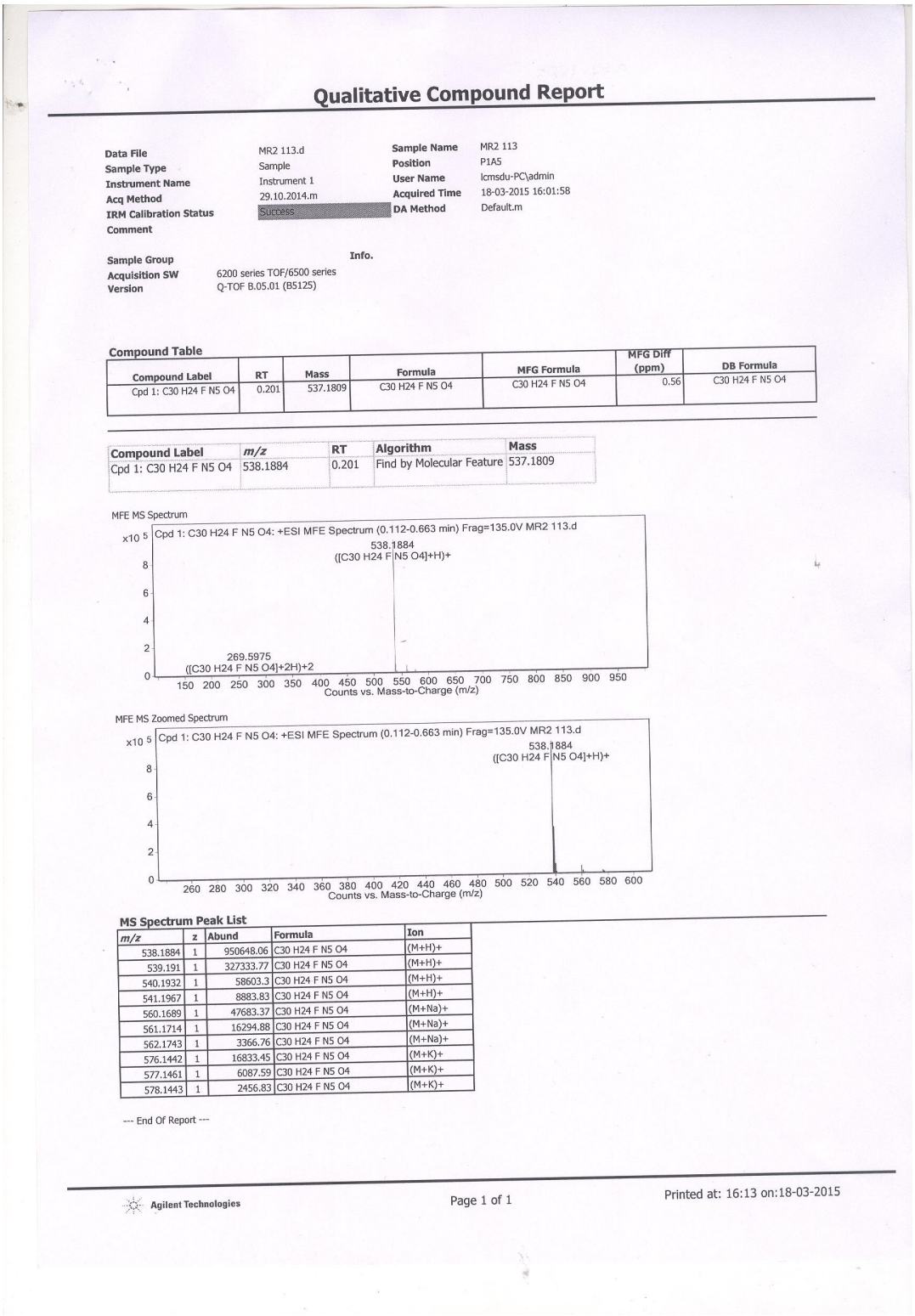


**Figure 21.** Mass spectrum of compound **5g**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2 113.d | **Sample Name** | MR2 113 |
| **Sample Type** | Sample | **Position** | P1A5 |
| **Instrument Name** | Instrument 1 | **User Name** | lcmsdu-PC\admin |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 18-03-2015 16:01:58 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Sample Group** — **Info.**

**Acquisition SW Version**: 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C30 H24 F N5 O4 | 0.201 | 537.1809 | C30 H24 F N5 O4 | C30 H24 F N5 O4 | 0.56 | C30 H24 F N5 O4 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C30 H24 F N5 O4 | 538.1884 | 0.201 | Find by Molecular Feature | 537.1809 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 538.1884 | 1 | 950648.06 | C30 H24 F N5 O4 | (M+H)+ |
| 539.191 | 1 | 327333.77 | C30 H24 F N5 O4 | (M+H)+ |
| 540.1932 | 1 | 58603.3 | C30 H24 F N5 O4 | (M+H)+ |
| 541.1967 | 1 | 8883.83 | C30 H24 F N5 O4 | (M+H)+ |
| 560.1689 | 1 | 47683.37 | C30 H24 F N5 O4 | (M+Na)+ |
| 561.1714 | 1 | 16294.88 | C30 H24 F N5 O4 | (M+Na)+ |
| 562.1743 | 1 | 3366.76 | C30 H24 F N5 O4 | (M+Na)+ |
| 576.1442 | 1 | 16833.45 | C30 H24 F N5 O4 | (M+K)+ |
| 577.1461 | 1 | 6087.59 | C30 H24 F N5 O4 | (M+K)+ |
| 578.1443 | 1 | 2456.83 | C30 H24 F N5 O4 | (M+K)+ |

--- End Of Report ---

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**Figure 22.** [1]H NMR spectrum of compound **5h** (400 MHz, DMSO).

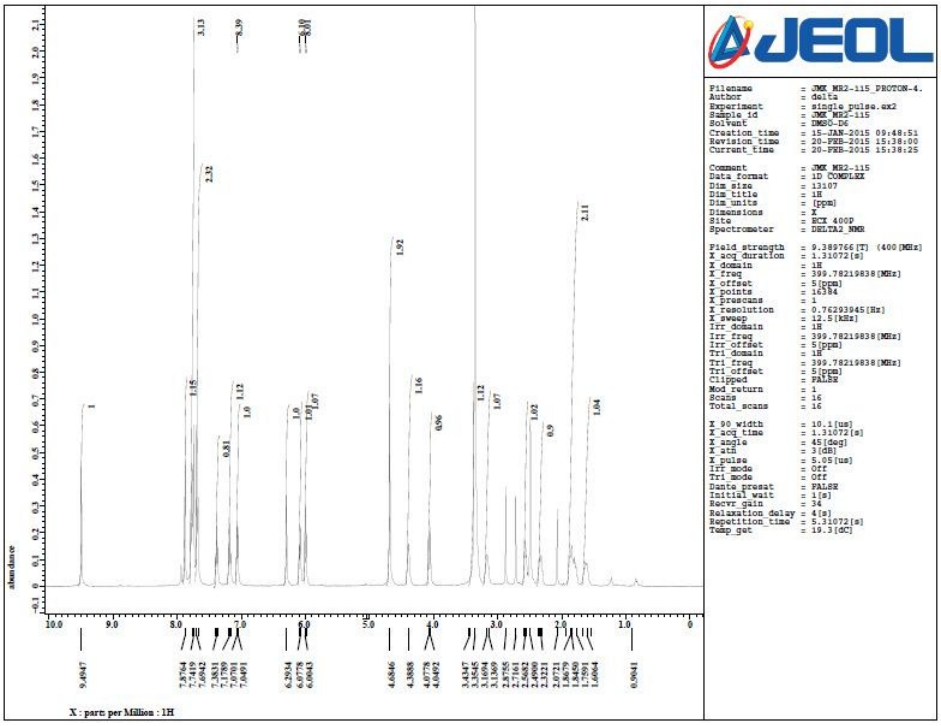


**Figure 23.** [13]C NMR spectrum of compound **5h** (100 MHz, DMSO).

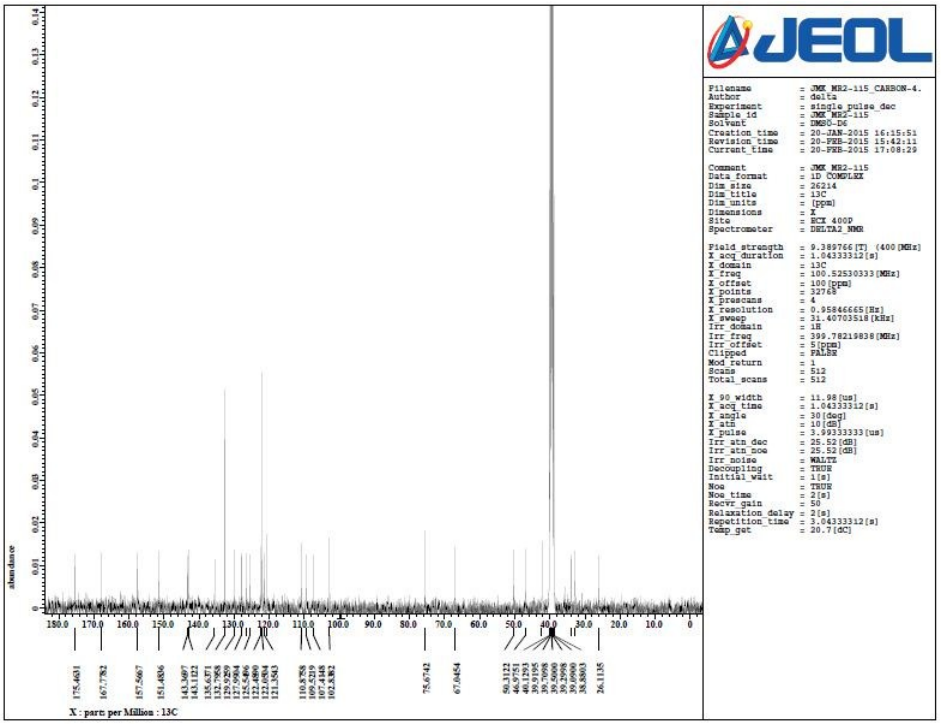
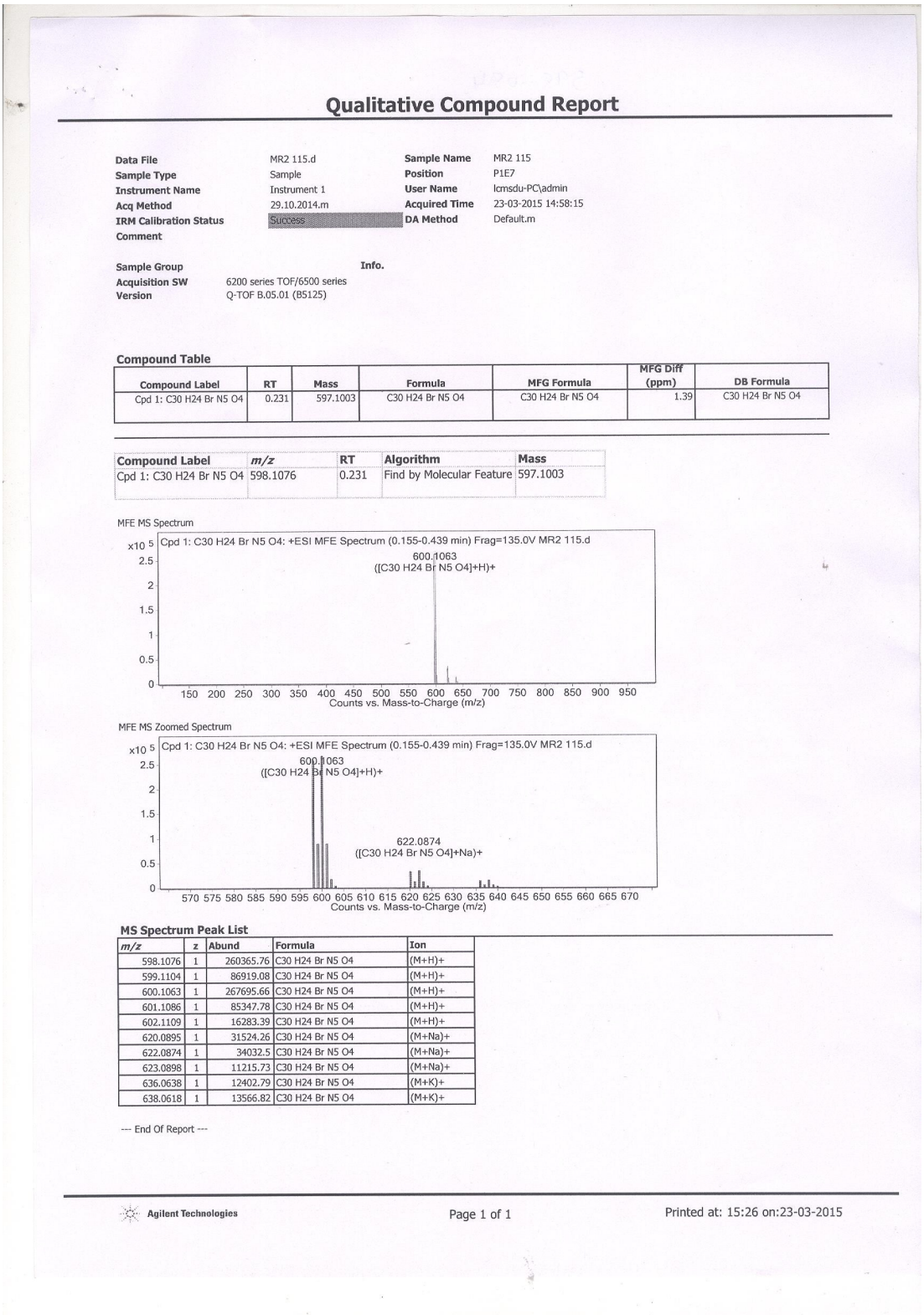


**Figure 24.** Mass spectrum of compound **5h**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2 115.d | **Sample Name** | MR2 115 |
| **Sample Type** | Sample | **Position** | P1E7 |
| **Instrument Name** | Instrument 1 | **User Name** | lcmsdu-PC\admin |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 23-03-2015 14:58:15 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Sample Group** **Info.**
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C30 H24 Br N5 O4 | 0.231 | 597.1003 | C30 H24 Br N5 O4 | C30 H24 Br N5 O4 | 1.39 | C30 H24 Br N5 O4 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C30 H24 Br N5 O4 | 598.1076 | 0.231 | Find by Molecular Feature | 597.1003 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 598.1076 | 1 | 260365.76 | C30 H24 Br N5 O4 | (M+H)+ |
| 599.1104 | 1 | 86919.08 | C30 H24 Br N5 O4 | (M+H)+ |
| 600.1063 | 1 | 267695.66 | C30 H24 Br N5 O4 | (M+H)+ |
| 601.1086 | 1 | 85347.78 | C30 H24 Br N5 O4 | (M+H)+ |
| 602.1109 | 1 | 16283.39 | C30 H24 Br N5 O4 | (M+H)+ |
| 620.0895 | 1 | 31524.26 | C30 H24 Br N5 O4 | (M+Na)+ |
| 622.0874 | 1 | 34032.5 | C30 H24 Br N5 O4 | (M+Na)+ |
| 623.0898 | 1 | 11215.73 | C30 H24 Br N5 O4 | (M+Na)+ |
| 636.0638 | 1 | 12402.79 | C30 H24 Br N5 O4 | (M+K)+ |
| 638.0618 | 1 | 13566.82 | C30 H24 Br N5 O4 | (M+K)+ |

--- End Of Report ---

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**Figure 25.** $^{1}$H NMR spectrum of compound **5i** (400 MHz, DMSO).

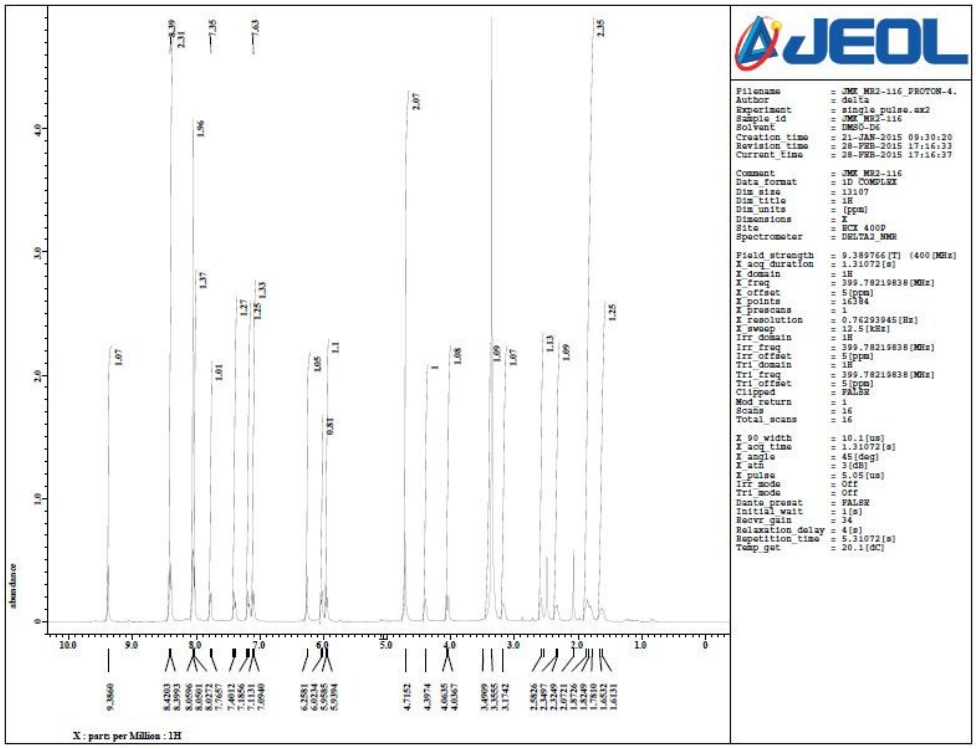


**Figure 26.** $^{13}$C NMR spectrum of compound **5i** (100 MHz, DMSO).

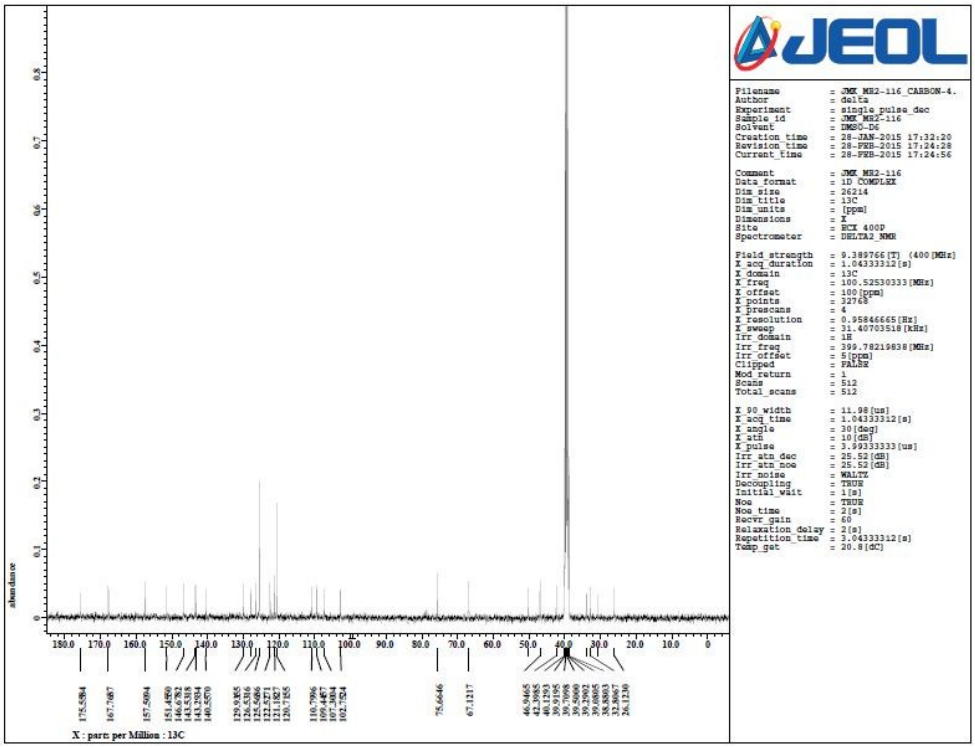
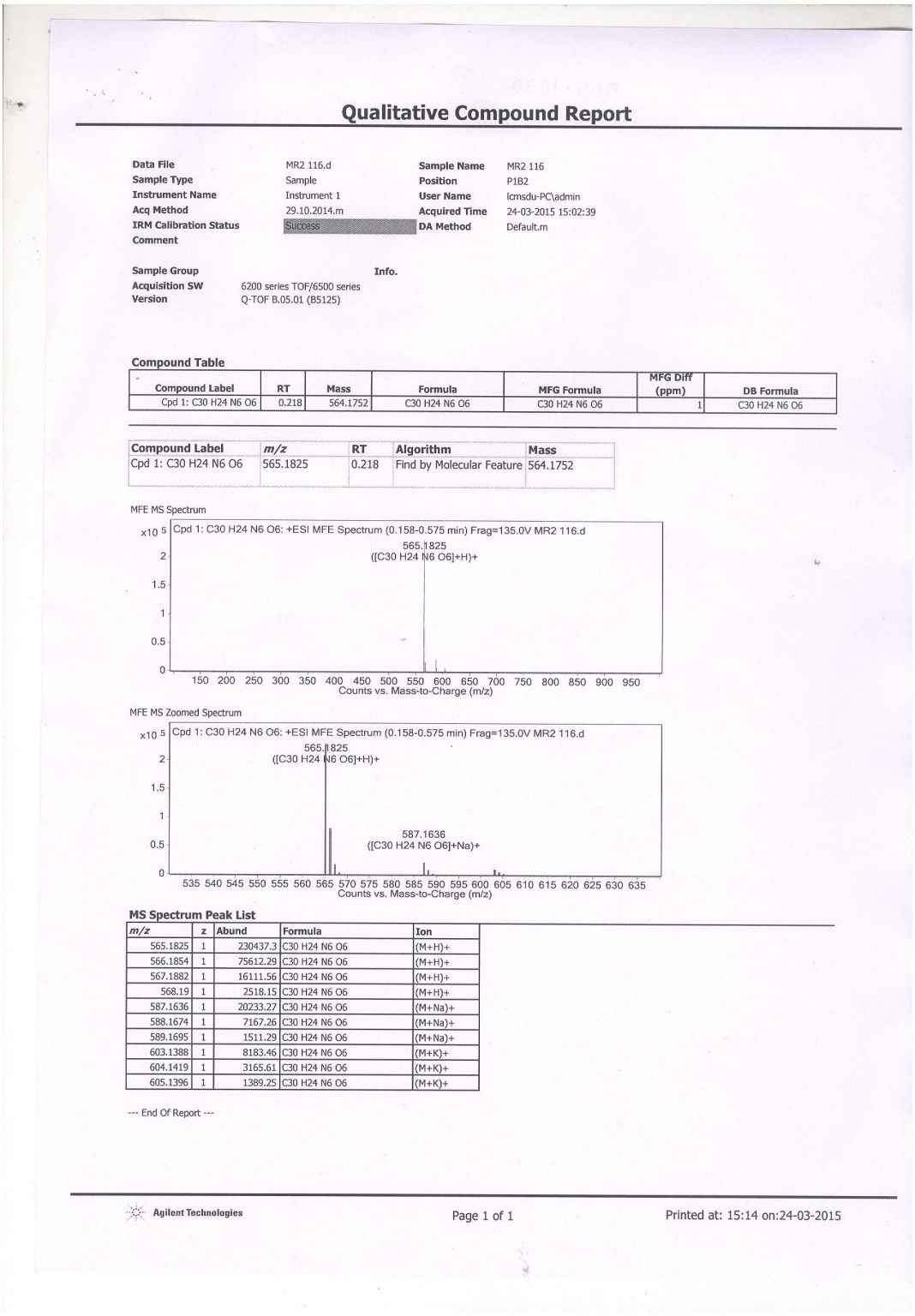


**Figure 27.** Mass spectrum of compound **5i**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2 116.d | **Sample Name** | MR2 116 |
| **Sample Type** | Sample | **Position** | P1B2 |
| **Instrument Name** | Instrument 1 | **User Name** | lcmsdu-PC\admin |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 24-03-2015 15:02:39 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Sample Group** **Info.**
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C30 H24 N6 O6 | 0.218 | 564.1752 | C30 H24 N6 O6 | C30 H24 N6 O6 | 1 | C30 H24 N6 O6 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C30 H24 N6 O6 | 565.1825 | 0.218 | Find by Molecular Feature | 564.1752 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 565.1825 | 1 | 230437.3 | C30 H24 N6 O6 | (M+H)+ |
| 566.1854 | 1 | 75612.29 | C30 H24 N6 O6 | (M+H)+ |
| 567.1882 | 1 | 16111.56 | C30 H24 N6 O6 | (M+H)+ |
| 568.19 | 1 | 2518.15 | C30 H24 N6 O6 | (M+H)+ |
| 587.1636 | 1 | 20233.27 | C30 H24 N6 O6 | (M+Na)+ |
| 588.1674 | 1 | 7167.26 | C30 H24 N6 O6 | (M+Na)+ |
| 589.1695 | 1 | 1511.29 | C30 H24 N6 O6 | (M+Na)+ |
| 603.1388 | 1 | 8183.46 | C30 H24 N6 O6 | (M+K)+ |
| 604.1419 | 1 | 3165.61 | C30 H24 N6 O6 | (M+K)+ |
| 605.1396 | 1 | 1389.25 | C30 H24 N6 O6 | (M+K)+ |

--- End Of Report ---

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**Figure 28.** [1]H NMR spectrum of compound **5j** (400 MHz, DMSO).

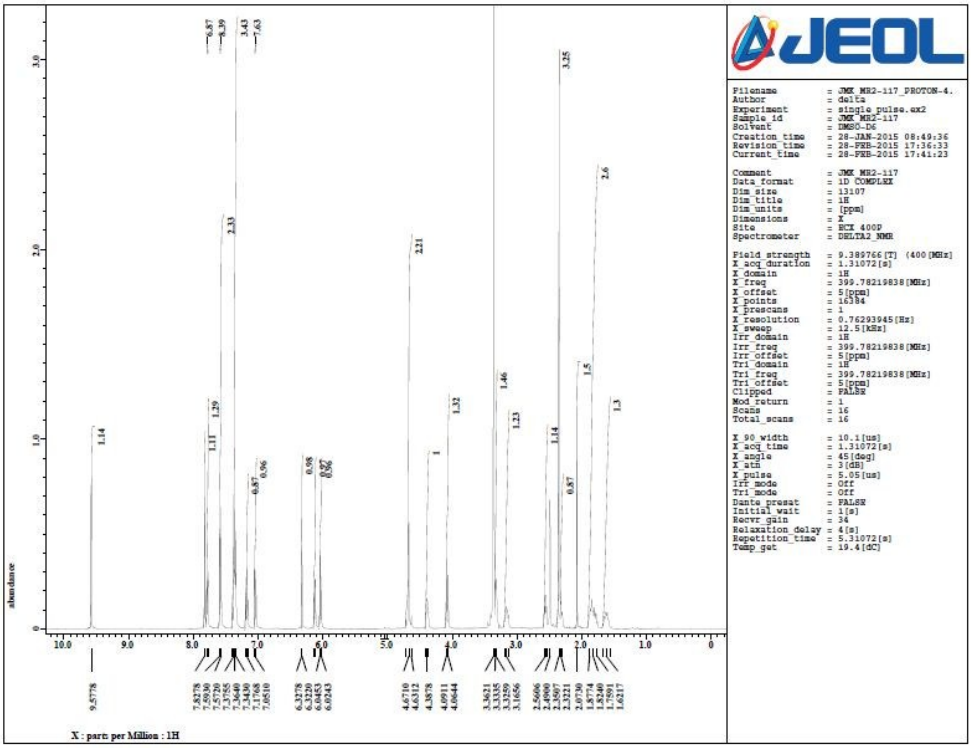


**Figure 29.** [13]C NMR spectrum of compound **5j** (100 MHz, DMSO).

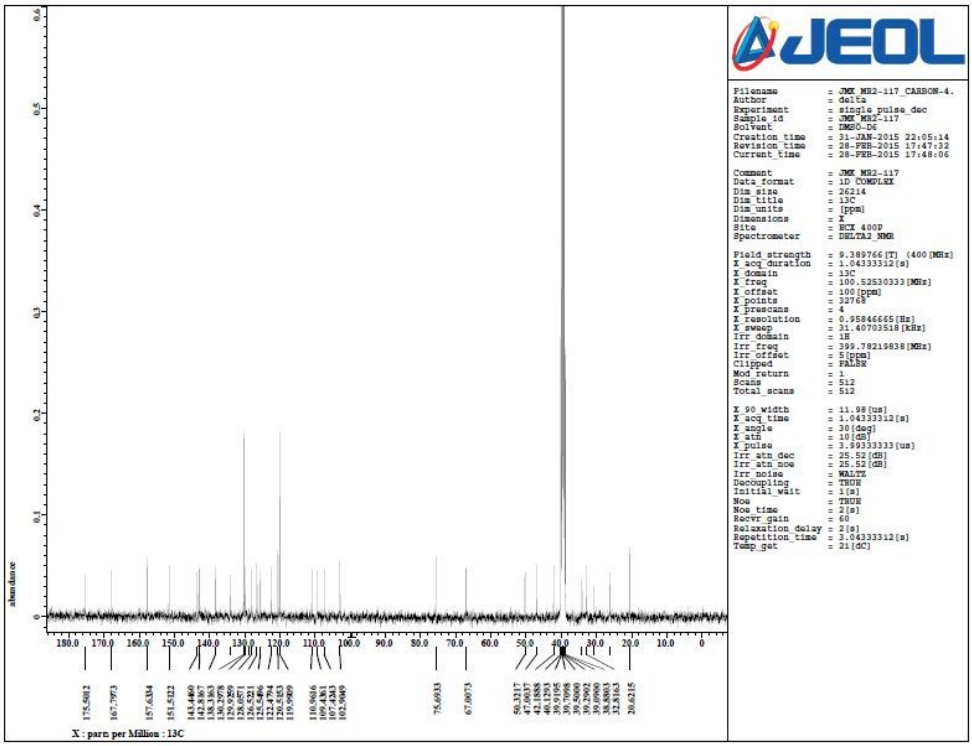
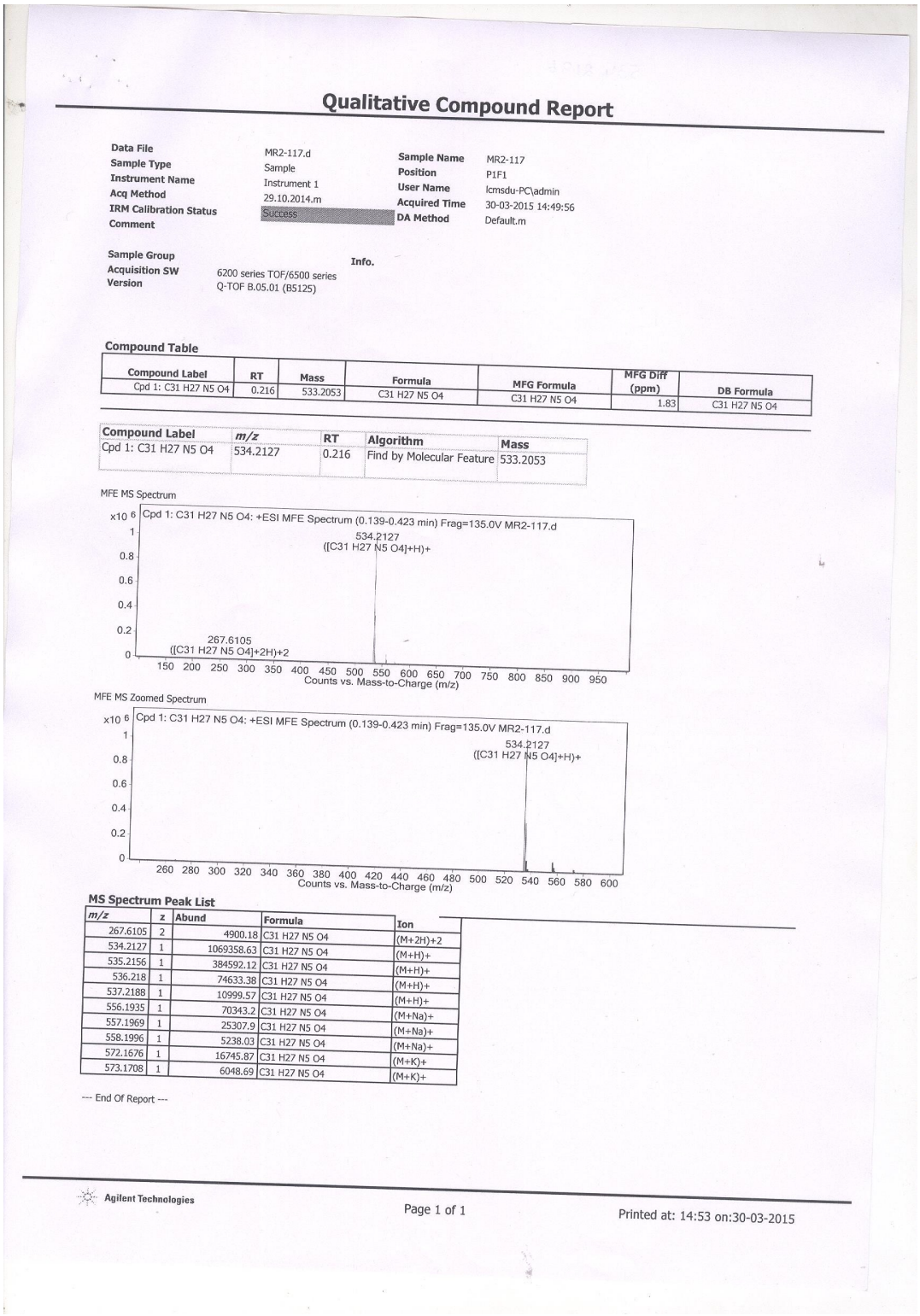


**Figure 30.** Mass spectrum of compound **5j**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2-117.d | **Sample Name** | MR2-117 |
| **Sample Type** | Sample | **Position** | P1F1 |
| **Instrument Name** | Instrument 1 | **User Name** | lcmsdu-PC\admin |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 30-03-2015 14:49:56 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

| | |
|---|---|
| **Sample Group** | **Info.** |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125) |

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C31 H27 N5 O4 | 0.216 | 533.2053 | C31 H27 N5 O4 | C31 H27 N5 O4 | 1.83 | C31 H27 N5 O4 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C31 H27 N5 O4 | 534.2127 | 0.216 | Find by Molecular Feature | 533.2053 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 267.6105 | 2 | 4900.18 | C31 H27 N5 O4 | (M+2H)+2 |
| 534.2127 | 1 | 1069358.63 | C31 H27 N5 O4 | (M+H)+ |
| 535.2156 | 1 | 384592.12 | C31 H27 N5 O4 | (M+H)+ |
| 536.218 | 1 | 74633.38 | C31 H27 N5 O4 | (M+H)+ |
| 537.2188 | 1 | 10999.57 | C31 H27 N5 O4 | (M+H)+ |
| 556.1935 | 1 | 70343.2 | C31 H27 N5 O4 | (M+Na)+ |
| 557.1969 | 1 | 25307.9 | C31 H27 N5 O4 | (M+Na)+ |
| 558.1996 | 1 | 5238.03 | C31 H27 N5 O4 | (M+Na)+ |
| 572.1676 | 1 | 16745.87 | C31 H27 N5 O4 | (M+K)+ |
| 573.1708 | 1 | 6048.69 | C31 H27 N5 O4 | (M+K)+ |

--- End Of Report ---

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Printed at: 14:53 on:30-03-2015

**Figure 31.** [1]H NMR spectrum of compound **5k** (400 MHz, DMSO).

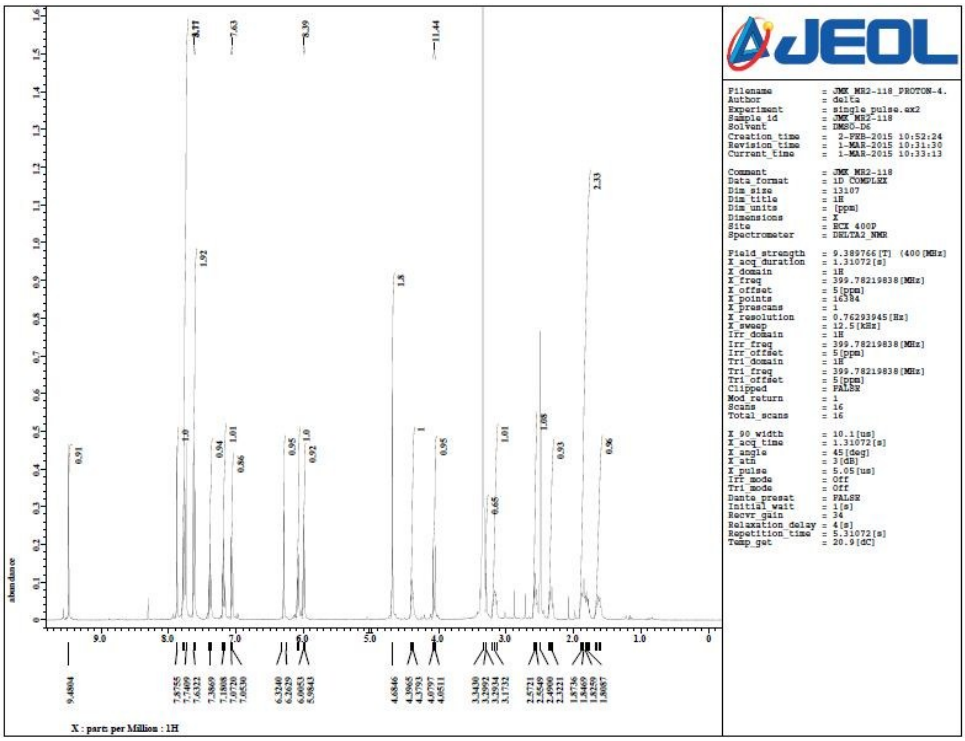



**Figure 32.** [13]C NMR spectrum of compound **5k** (100 MHz, DMSO).

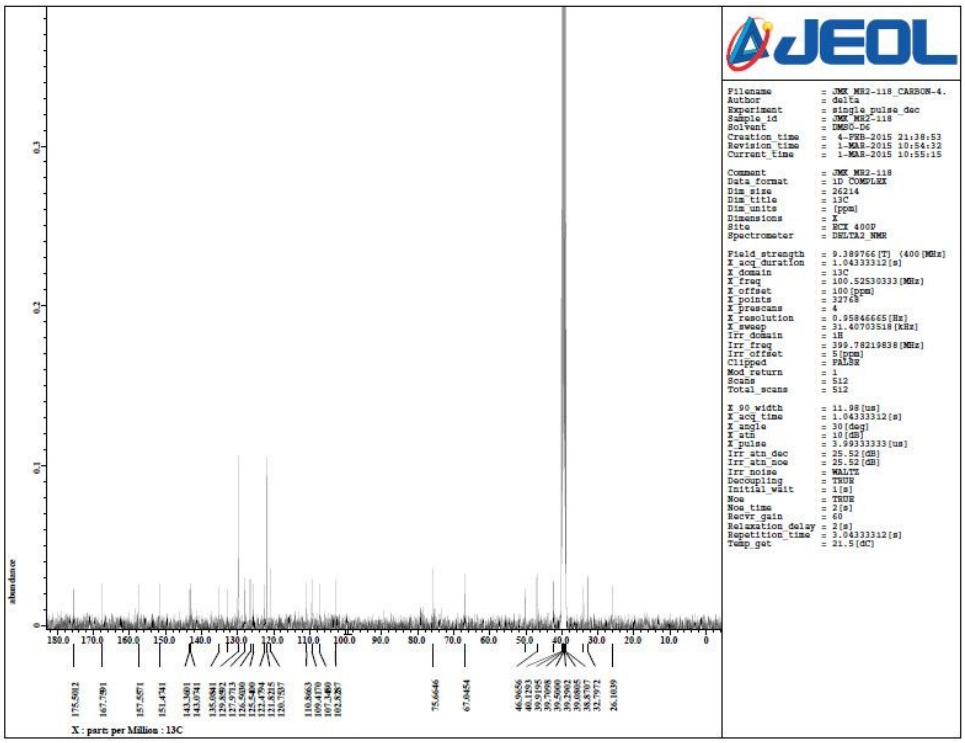
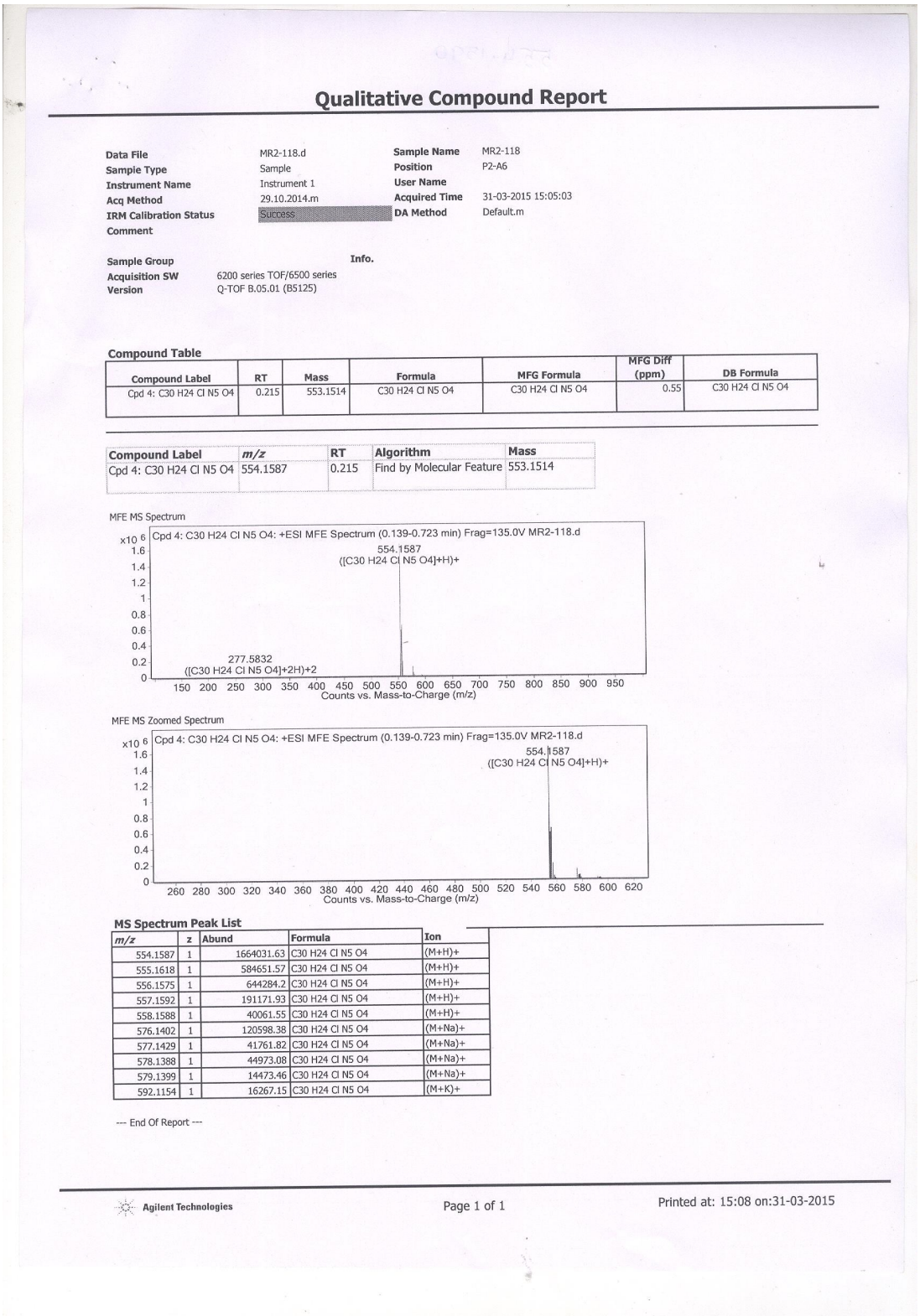


**Figure 33.** Mass spectrum of compound **5k**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2-118.d | **Sample Name** | MR2-118 |
| **Sample Type** | Sample | **Position** | P2-A6 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 31-03-2015 15:05:03 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

| | |
|---|---|
| **Sample Group** | **Info.** |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125) |

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 4: C30 H24 Cl N5 O4 | 0.215 | 553.1514 | C30 H24 Cl N5 O4 | C30 H24 Cl N5 O4 | 0.55 | C30 H24 Cl N5 O4 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 4: C30 H24 Cl N5 O4 | 554.1587 | 0.215 | Find by Molecular Feature | 553.1514 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 554.1587 | 1 | 1664031.63 | C30 H24 Cl N5 O4 | (M+H)+ |
| 555.1618 | 1 | 584651.57 | C30 H24 Cl N5 O4 | (M+H)+ |
| 556.1575 | 1 | 644284.2 | C30 H24 Cl N5 O4 | (M+H)+ |
| 557.1592 | 1 | 191171.93 | C30 H24 Cl N5 O4 | (M+H)+ |
| 558.1588 | 1 | 40061.55 | C30 H24 Cl N5 O4 | (M+H)+ |
| 576.1402 | 1 | 120598.38 | C30 H24 Cl N5 O4 | (M+Na)+ |
| 577.1429 | 1 | 41761.82 | C30 H24 Cl N5 O4 | (M+Na)+ |
| 578.1388 | 1 | 44973.08 | C30 H24 Cl N5 O4 | (M+Na)+ |
| 579.1399 | 1 | 14473.46 | C30 H24 Cl N5 O4 | (M+Na)+ |
| 592.1154 | 1 | 16267.15 | C30 H24 Cl N5 O4 | (M+K)+ |

--- End Of Report ---

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**Figure 34.** $^{1}$H NMR spectrum of compound **5l** (400 MHz, DMSO).

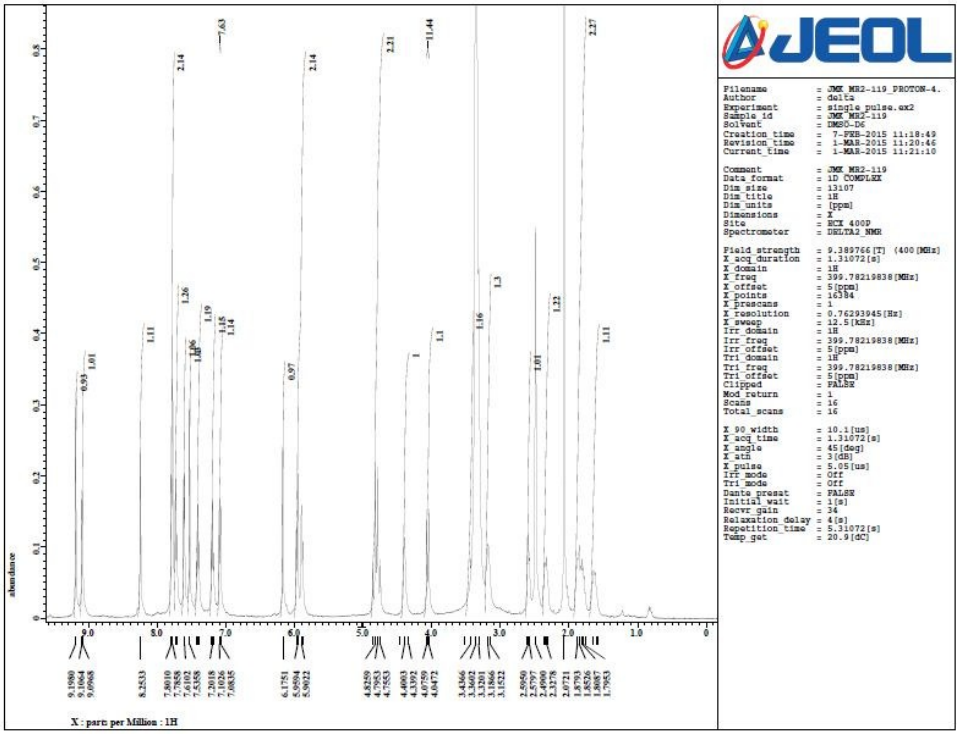


**Figure 35.** $^{13}$C NMR spectrum of compound **5l** (100 MHz, DMSO).

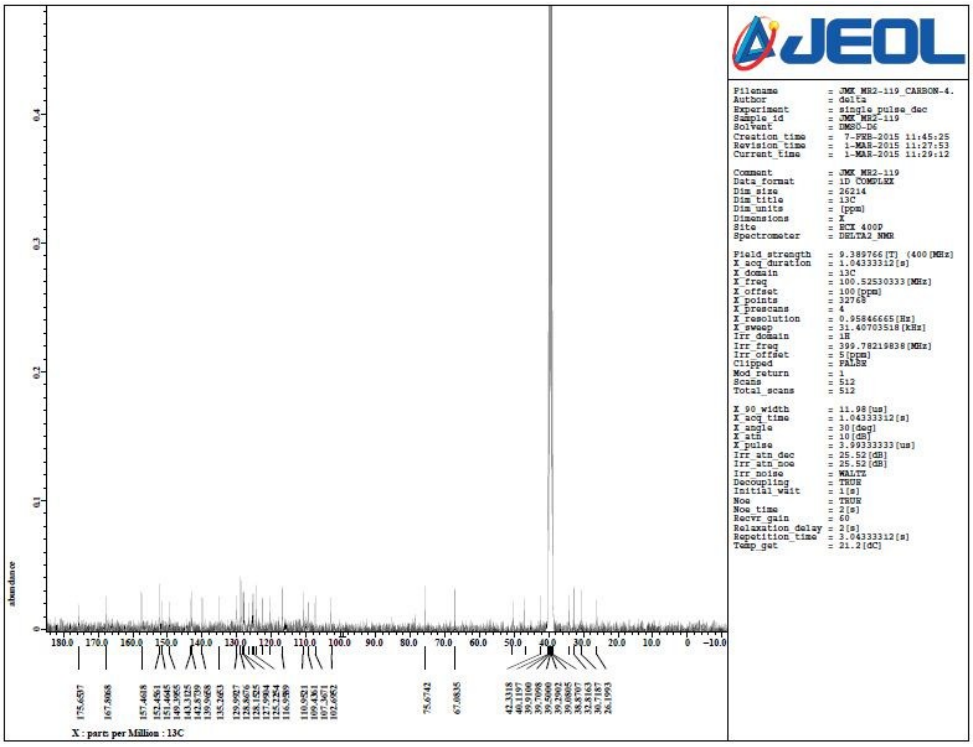
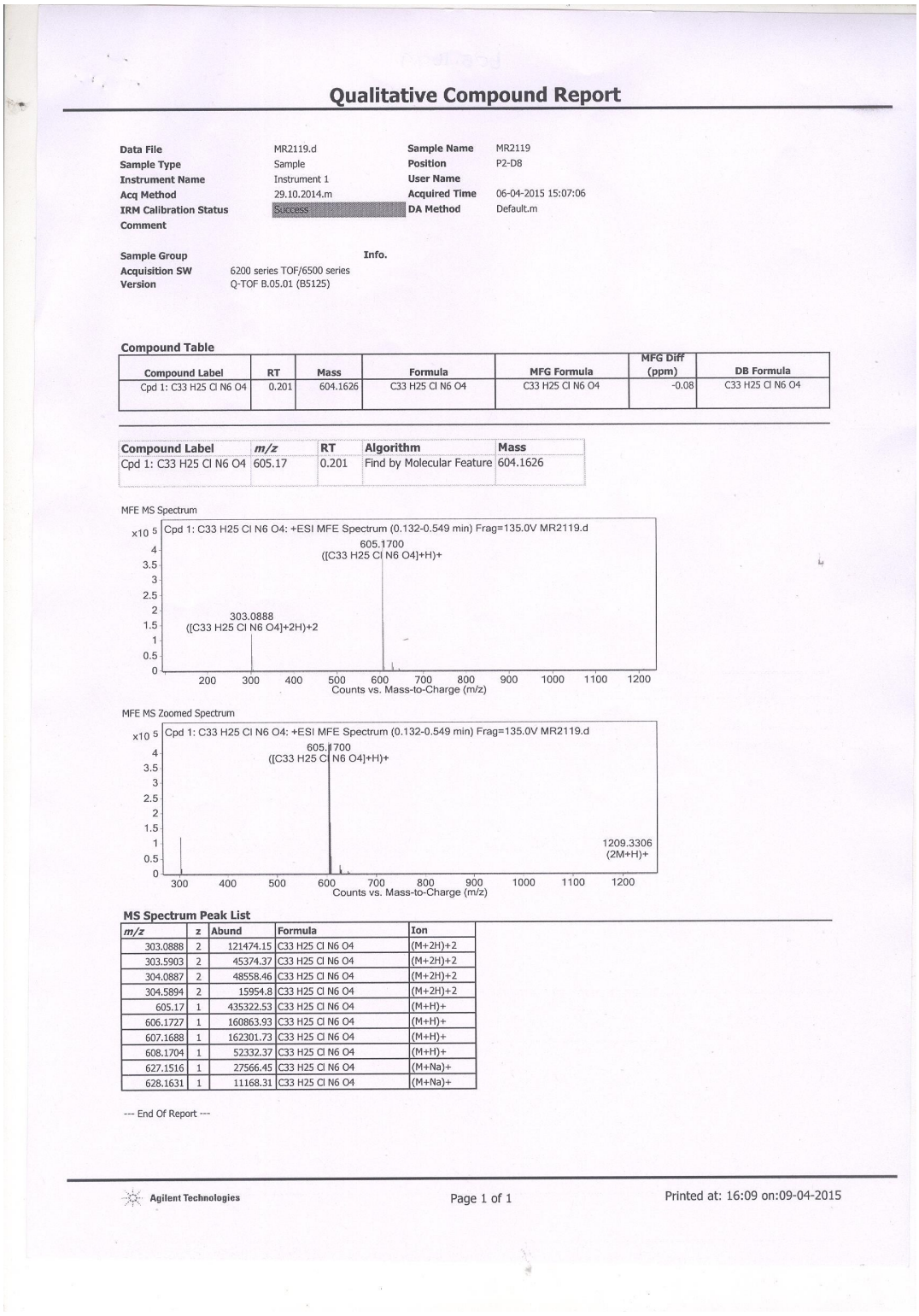


**Figure 36.** Mass spectrum of compound **5l**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2119.d | **Sample Name** | MR2119 |
| **Sample Type** | Sample | **Position** | P2-D8 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 06-04-2015 15:07:06 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Sample Group** **Info.**
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C33 H25 Cl N6 O4 | 0.201 | 604.1626 | C33 H25 Cl N6 O4 | C33 H25 Cl N6 O4 | -0.08 | C33 H25 Cl N6 O4 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C33 H25 Cl N6 O4 | 605.17 | 0.201 | Find by Molecular Feature | 604.1626 |

MFE MS Spectrum



MFE MS Zoomed Spectrum



**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 303.0888 | 2 | 121474.15 | C33 H25 Cl N6 O4 | (M+2H)+2 |
| 303.5903 | 2 | 45374.37 | C33 H25 Cl N6 O4 | (M+2H)+2 |
| 304.0887 | 2 | 48558.46 | C33 H25 Cl N6 O4 | (M+2H)+2 |
| 304.5894 | 2 | 15954.8 | C33 H25 Cl N6 O4 | (M+2H)+2 |
| 605.17 | 1 | 435322.53 | C33 H25 Cl N6 O4 | (M+H)+ |
| 606.1727 | 1 | 160863.93 | C33 H25 Cl N6 O4 | (M+H)+ |
| 607.1688 | 1 | 162301.73 | C33 H25 Cl N6 O4 | (M+H)+ |
| 608.1704 | 1 | 52332.37 | C33 H25 Cl N6 O4 | (M+H)+ |
| 627.1516 | 1 | 27566.45 | C33 H25 Cl N6 O4 | (M+Na)+ |
| 628.1631 | 1 | 11168.31 | C33 H25 Cl N6 O4 | (M+Na)+ |

--- End Of Report ---

**Figure 37.** $^{1}$H NMR spectrum of compound **5m** (400 MHz, DMSO).

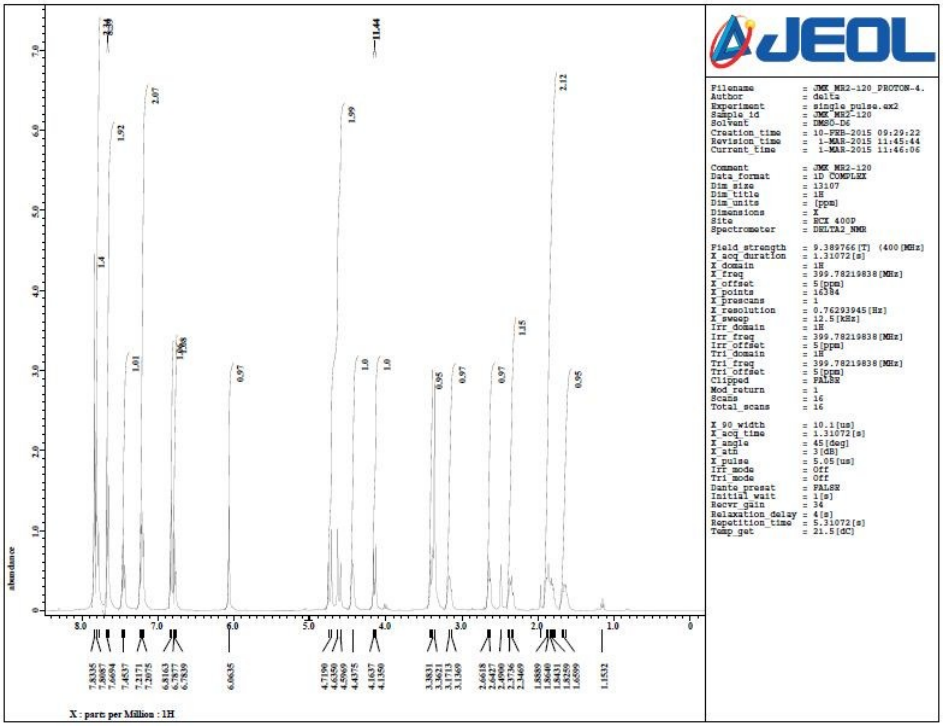


**Figure 38.** $^{13}$C NMR spectrum of compound **5m** (100 MHz, DMSO).

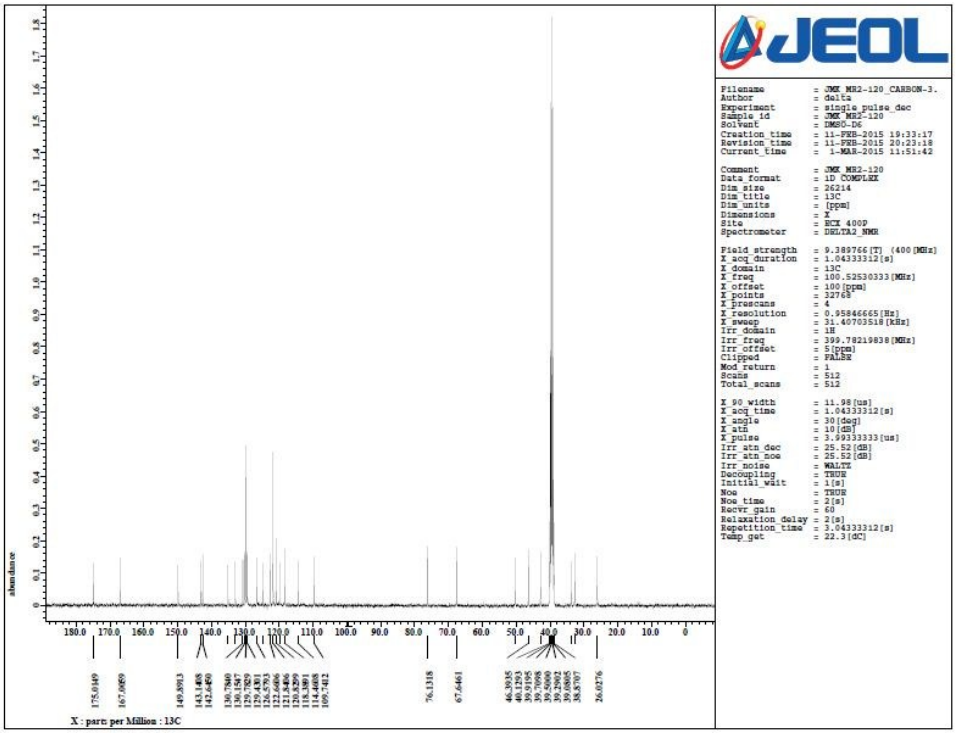
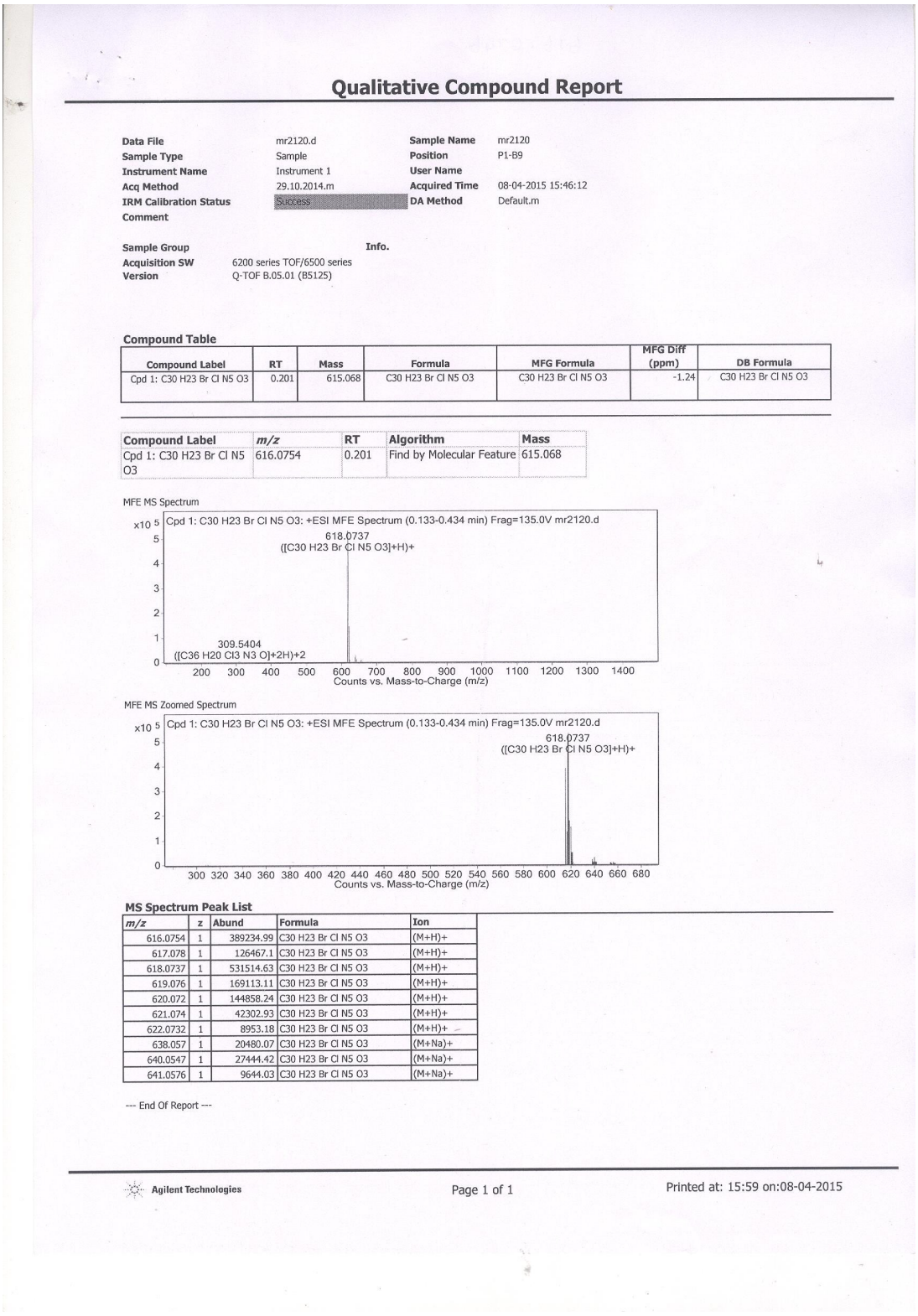


**Figure 39.** Mass spectrum of compound **5m**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | mr2120.d | **Sample Name** | mr2120 |
| **Sample Type** | Sample | **Position** | P1-B9 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 08-04-2015 15:46:12 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Sample Group** **Info.**
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C30 H23 Br Cl N5 O3 | 0.201 | 615.068 | C30 H23 Br Cl N5 O3 | C30 H23 Br Cl N5 O3 | -1.24 | C30 H23 Br Cl N5 O3 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C30 H23 Br Cl N5 O3 | 616.0754 | 0.201 | Find by Molecular Feature | 615.068 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 616.0754 | 1 | 389234.99 | C30 H23 Br Cl N5 O3 | (M+H)+ |
| 617.078 | 1 | 126467.1 | C30 H23 Br Cl N5 O3 | (M+H)+ |
| 618.0737 | 1 | 531514.63 | C30 H23 Br Cl N5 O3 | (M+H)+ |
| 619.076 | 1 | 169113.11 | C30 H23 Br Cl N5 O3 | (M+H)+ |
| 620.072 | 1 | 144858.24 | C30 H23 Br Cl N5 O3 | (M+H)+ |
| 621.074 | 1 | 42302.93 | C30 H23 Br Cl N5 O3 | (M+H)+ |
| 622.0732 | 1 | 8953.18 | C30 H23 Br Cl N5 O3 | (M+H)+ |
| 638.057 | 1 | 20480.07 | C30 H23 Br Cl N5 O3 | (M+Na)+ |
| 640.0547 | 1 | 27444.42 | C30 H23 Br Cl N5 O3 | (M+Na)+ |
| 641.0576 | 1 | 9644.03 | C30 H23 Br Cl N5 O3 | (M+Na)+ |

--- End Of Report ---

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**Figure 40.** [1]H NMR spectrum of compound **5n** (400 MHz, DMSO).

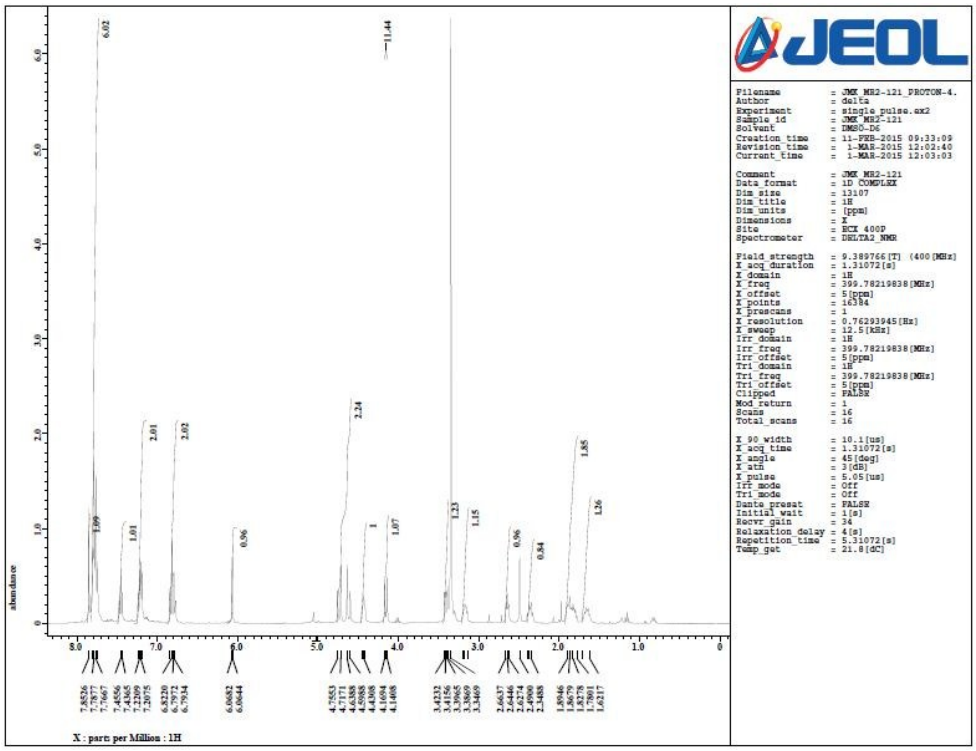



**Figure 41.** [13]C NMR spectrum of compound **5n** (100 MHz, DMSO).

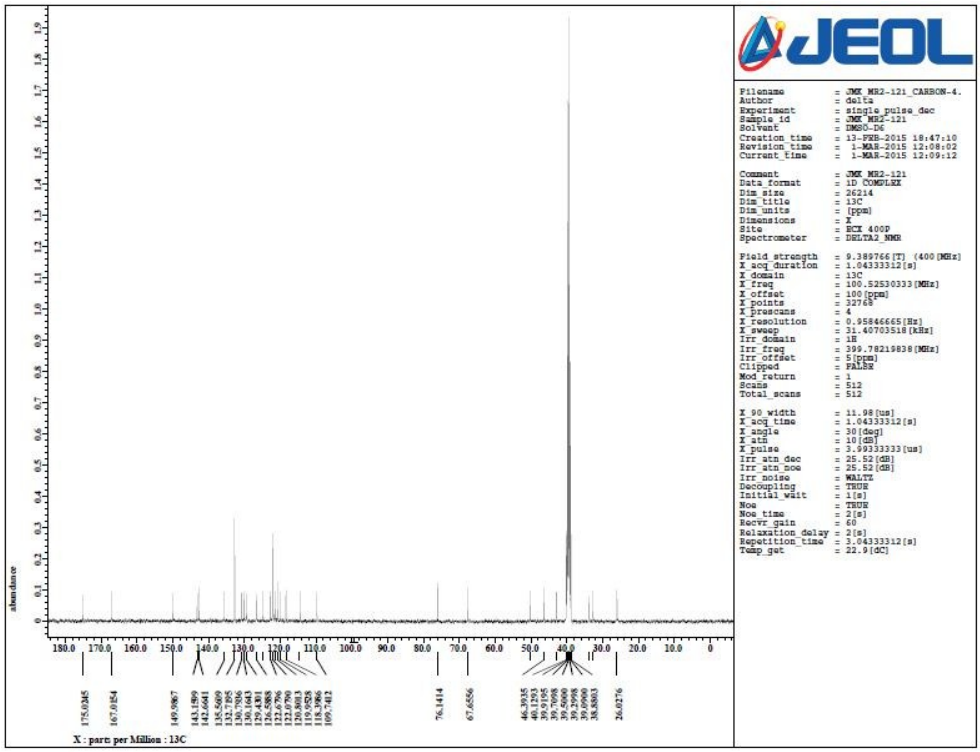
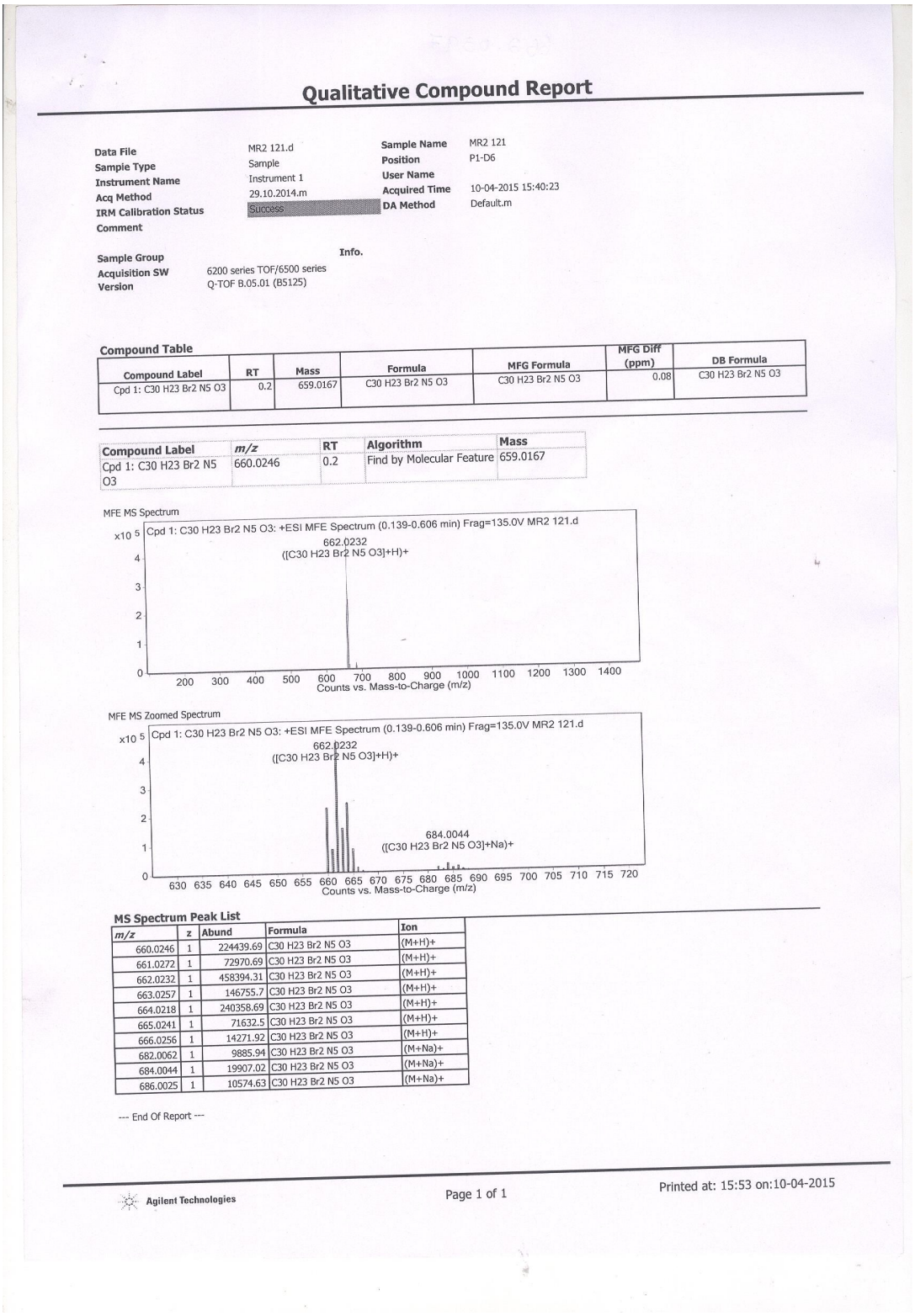


**Figure 42.** Mass spectrum of compound **5n**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2 121.d | **Sample Name** | MR2 121 |
| **Sample Type** | Sample | **Position** | P1-D6 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 10-04-2015 15:40:23 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Info.**

| | |
|---|---|
| **Sample Group** | |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125) |

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C30 H23 Br2 N5 O3 | 0.2 | 659.0167 | C30 H23 Br2 N5 O3 | C30 H23 Br2 N5 O3 | 0.08 | C30 H23 Br2 N5 O3 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C30 H23 Br2 N5 O3 | 660.0246 | 0.2 | Find by Molecular Feature | 659.0167 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 660.0246 | 1 | 224439.69 | C30 H23 Br2 N5 O3 | (M+H)+ |
| 661.0272 | 1 | 72970.69 | C30 H23 Br2 N5 O3 | (M+H)+ |
| 662.0232 | 1 | 458394.31 | C30 H23 Br2 N5 O3 | (M+H)+ |
| 663.0257 | 1 | 146755.7 | C30 H23 Br2 N5 O3 | (M+H)+ |
| 664.0218 | 1 | 240358.69 | C30 H23 Br2 N5 O3 | (M+H)+ |
| 665.0241 | 1 | 71632.5 | C30 H23 Br2 N5 O3 | (M+H)+ |
| 666.0256 | 1 | 14271.92 | C30 H23 Br2 N5 O3 | (M+H)+ |
| 682.0062 | 1 | 9885.94 | C30 H23 Br2 N5 O3 | (M+Na)+ |
| 684.0044 | 1 | 19907.02 | C30 H23 Br2 N5 O3 | (M+Na)+ |
| 686.0025 | 1 | 10574.63 | C30 H23 Br2 N5 O3 | (M+Na)+ |

--- End Of Report ---

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Printed at: 15:53 on:10-04-2015

**Figure 43.** $^{1}$H NMR spectrum of compound **5o** (400 MHz, DMSO).

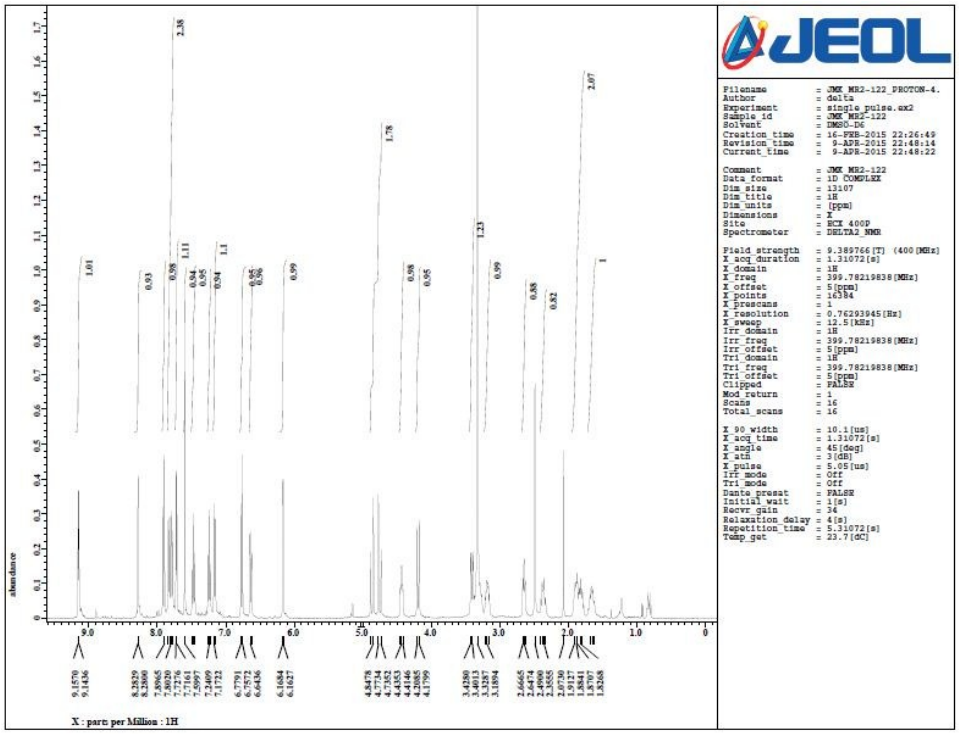



**Figure 44.** $^{13}$C NMR spectrum of compound **5o** (100 MHz, DMSO).

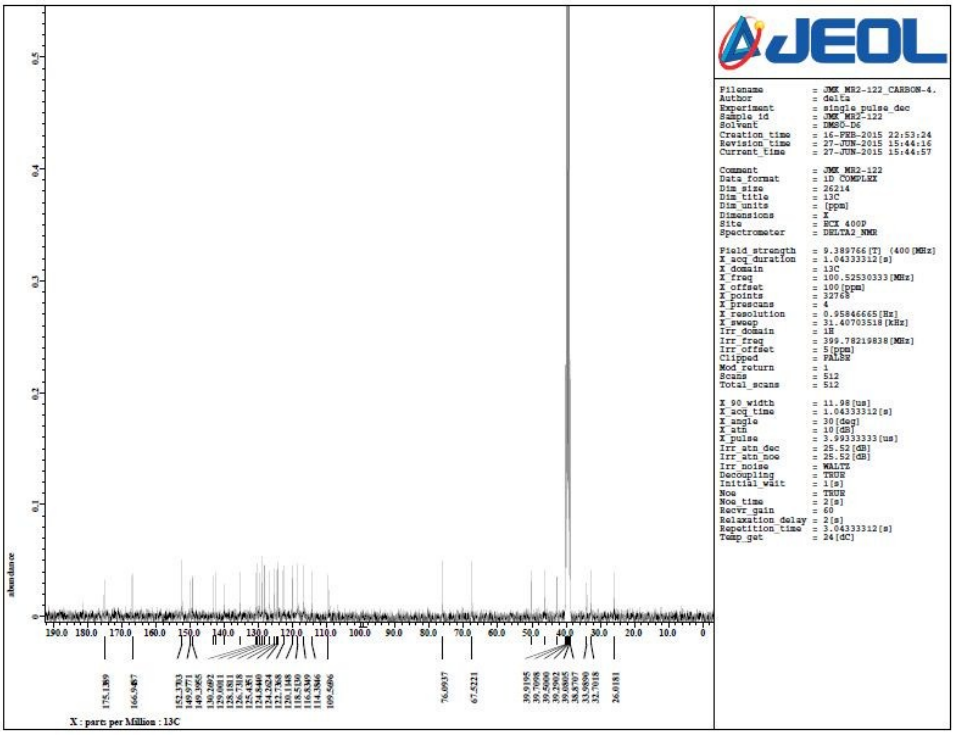
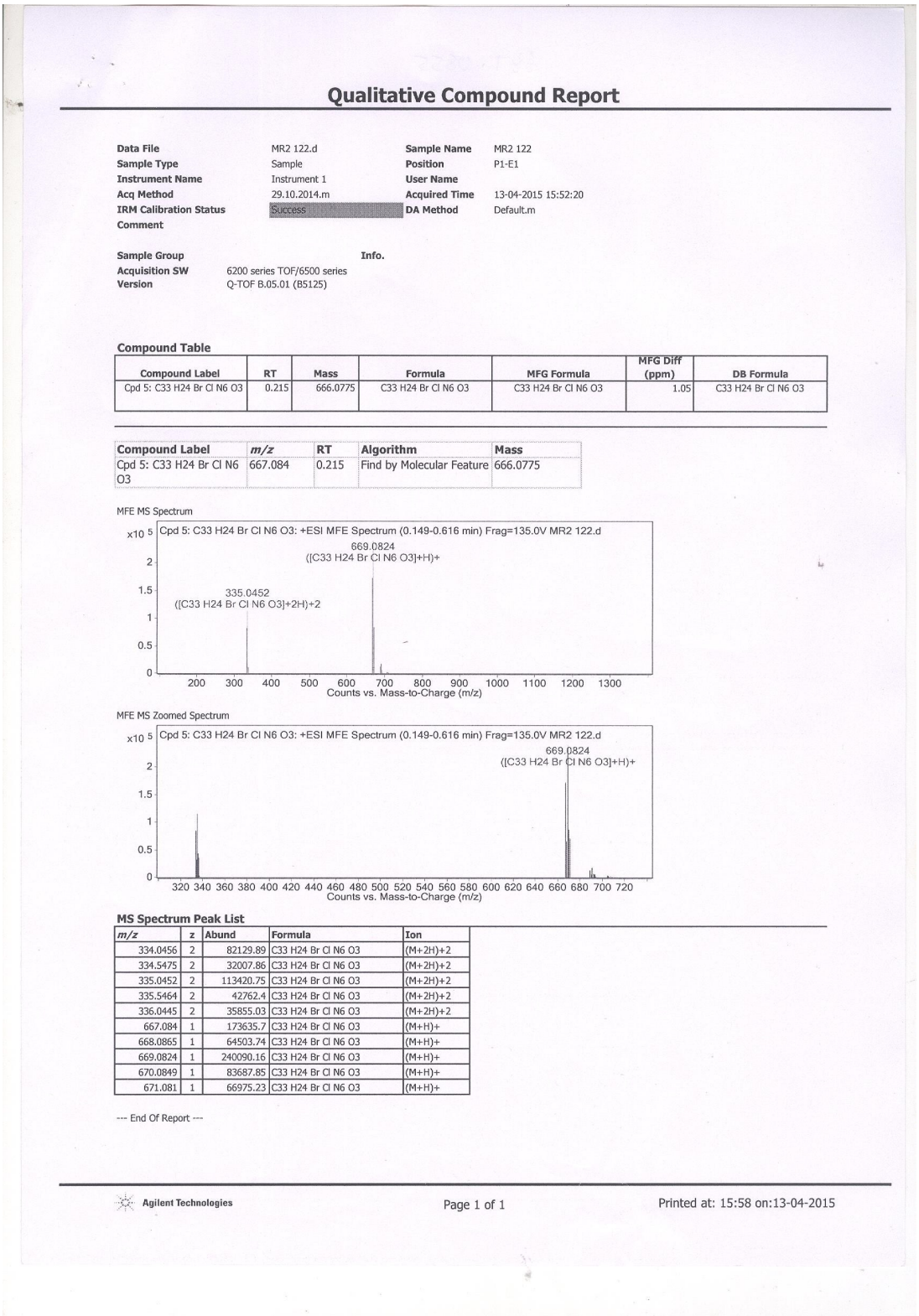


**Figure 45.** Mass spectrum of compound **5o**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2 122.d | **Sample Name** | MR2 122 |
| **Sample Type** | Sample | **Position** | P1-E1 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 13-04-2015 15:52:20 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

| | |
|---|---|
| **Sample Group** | **Info.** |
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125) |

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 5: C33 H24 Br Cl N6 O3 | 0.215 | 666.0775 | C33 H24 Br Cl N6 O3 | C33 H24 Br Cl N6 O3 | 1.05 | C33 H24 Br Cl N6 O3 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 5: C33 H24 Br Cl N6 O3 | 667.084 | 0.215 | Find by Molecular Feature | 666.0775 |

MFE MS Spectrum



MFE MS Zoomed Spectrum



**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 334.0456 | 2 | 82129.89 | C33 H24 Br Cl N6 O3 | (M+2H)+2 |
| 334.5475 | 2 | 32007.86 | C33 H24 Br Cl N6 O3 | (M+2H)+2 |
| 335.0452 | 2 | 113420.75 | C33 H24 Br Cl N6 O3 | (M+2H)+2 |
| 335.5464 | 2 | 42762.4 | C33 H24 Br Cl N6 O3 | (M+2H)+2 |
| 336.0445 | 2 | 35855.03 | C33 H24 Br Cl N6 O3 | (M+2H)+2 |
| 667.084 | 1 | 173635.7 | C33 H24 Br Cl N6 O3 | (M+H)+ |
| 668.0865 | 1 | 64503.74 | C33 H24 Br Cl N6 O3 | (M+H)+ |
| 669.0824 | 1 | 240090.16 | C33 H24 Br Cl N6 O3 | (M+H)+ |
| 670.0849 | 1 | 83687.85 | C33 H24 Br Cl N6 O3 | (M+H)+ |
| 671.081 | 1 | 66975.23 | C33 H24 Br Cl N6 O3 | (M+H)+ |

--- End Of Report ---

**Figure 46.** [1]H NMR spectrum of compound **5p** (400 MHz, DMSO).

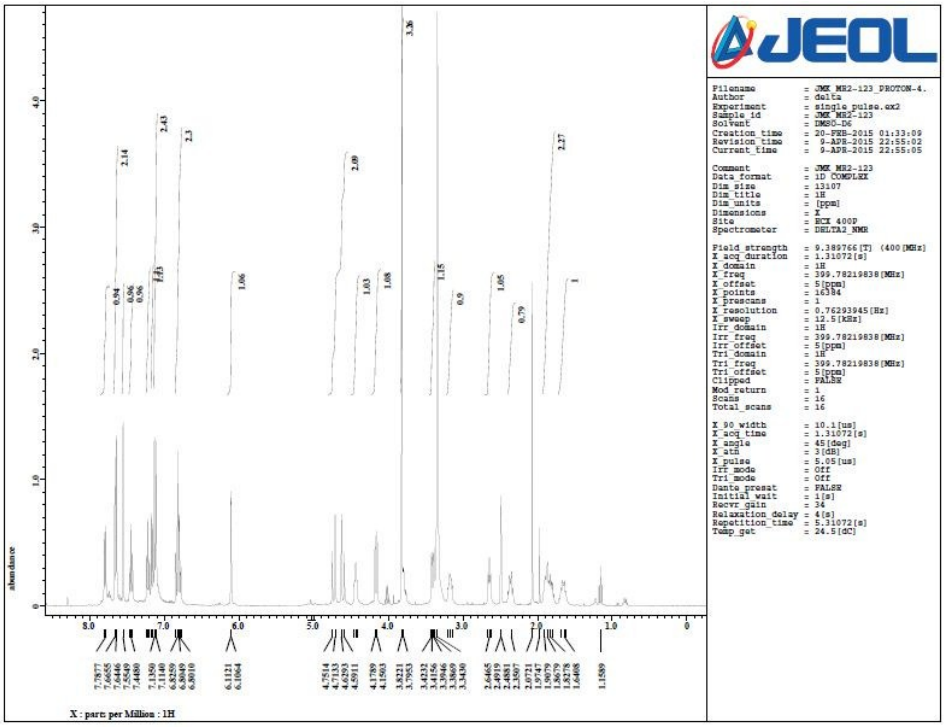


**Figure 47.** [13]C NMR spectrum of compound **5p** (100 MHz, DMSO).

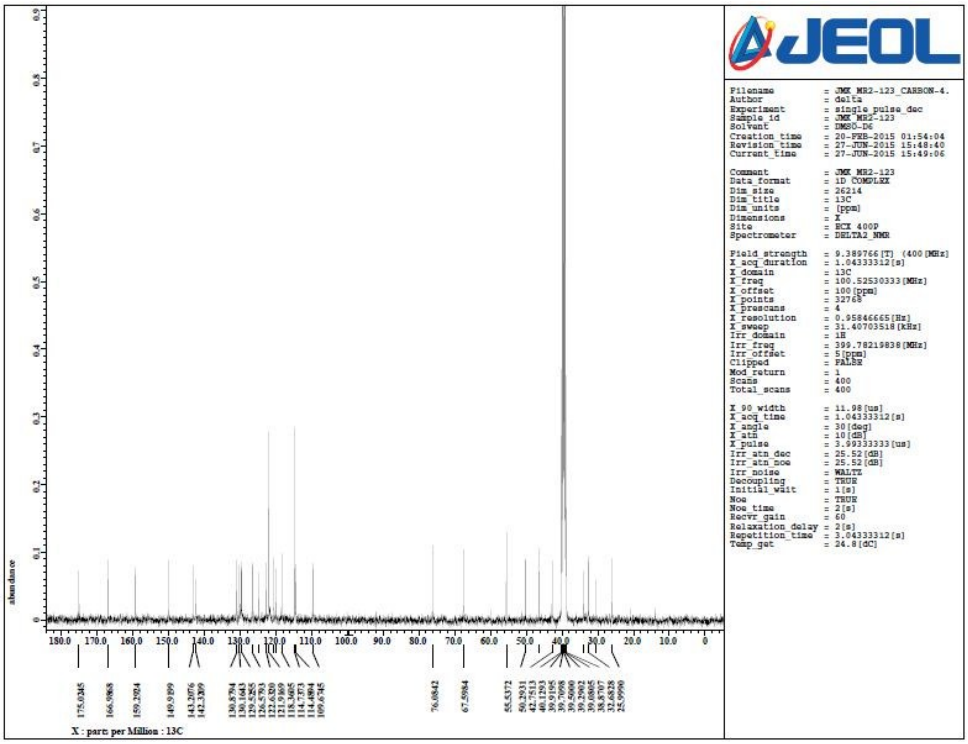
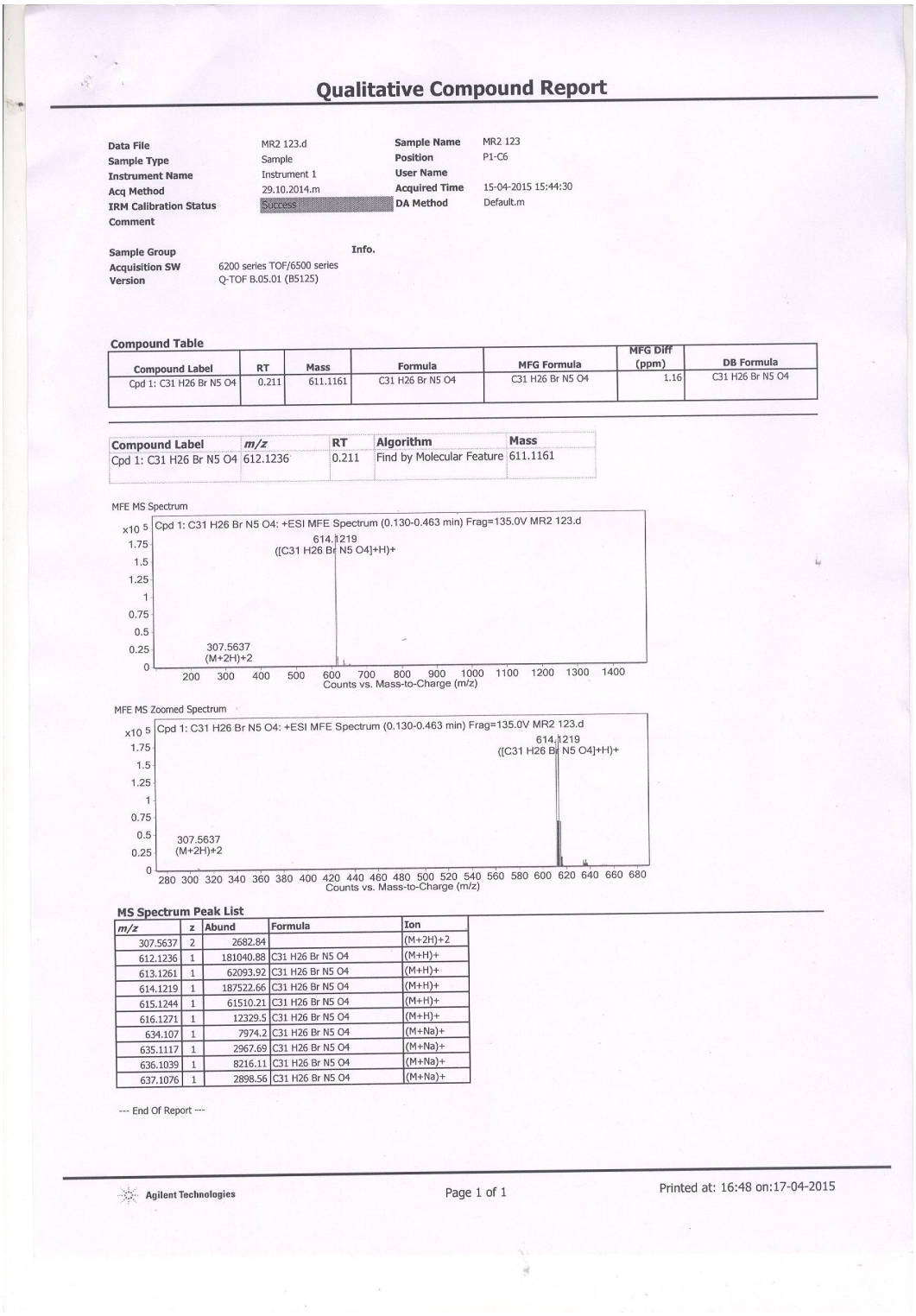


**Figure 48.** Mass spectrum of compound **5p**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2 123.d | **Sample Name** | MR2 123 |
| **Sample Type** | Sample | **Position** | P1-C6 |
| **Instrument Name** | Instrument 1 | **User Name** | |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 15-04-2015 15:44:30 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Sample Group** **Info.**
**Acquisition SW Version** 6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 1: C31 H26 Br N5 O4 | 0.211 | 611.1161 | C31 H26 Br N5 O4 | C31 H26 Br N5 O4 | 1.16 | C31 H26 Br N5 O4 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 1: C31 H26 Br N5 O4 | 612.1236 | 0.211 | Find by Molecular Feature | 611.1161 |

MFE MS Spectrum




MFE MS Zoomed Spectrum




**MS Spectrum Peak List**

| *m/z* | z | Abund | Formula | Ion |
|---|---|---|---|---|
| 307.5637 | 2 | 2682.84 | | (M+2H)+2 |
| 612.1236 | 1 | 181040.88 | C31 H26 Br N5 O4 | (M+H)+ |
| 613.1261 | 1 | 62093.92 | C31 H26 Br N5 O4 | (M+H)+ |
| 614.1219 | 1 | 187522.66 | C31 H26 Br N5 O4 | (M+H)+ |
| 615.1244 | 1 | 61510.21 | C31 H26 Br N5 O4 | (M+H)+ |
| 616.1271 | 1 | 12329.5 | C31 H26 Br N5 O4 | (M+H)+ |
| 634.107 | 1 | 7974.2 | C31 H26 Br N5 O4 | (M+Na)+ |
| 635.1117 | 1 | 2967.69 | C31 H26 Br N5 O4 | (M+Na)+ |
| 636.1039 | 1 | 8216.11 | C31 H26 Br N5 O4 | (M+Na)+ |
| 637.1076 | 1 | 2898.56 | C31 H26 Br N5 O4 | (M+Na)+ |

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**Figure 49.** $^{1}$H NMR spectrum of compound **5q** (400 MHz, DMSO).

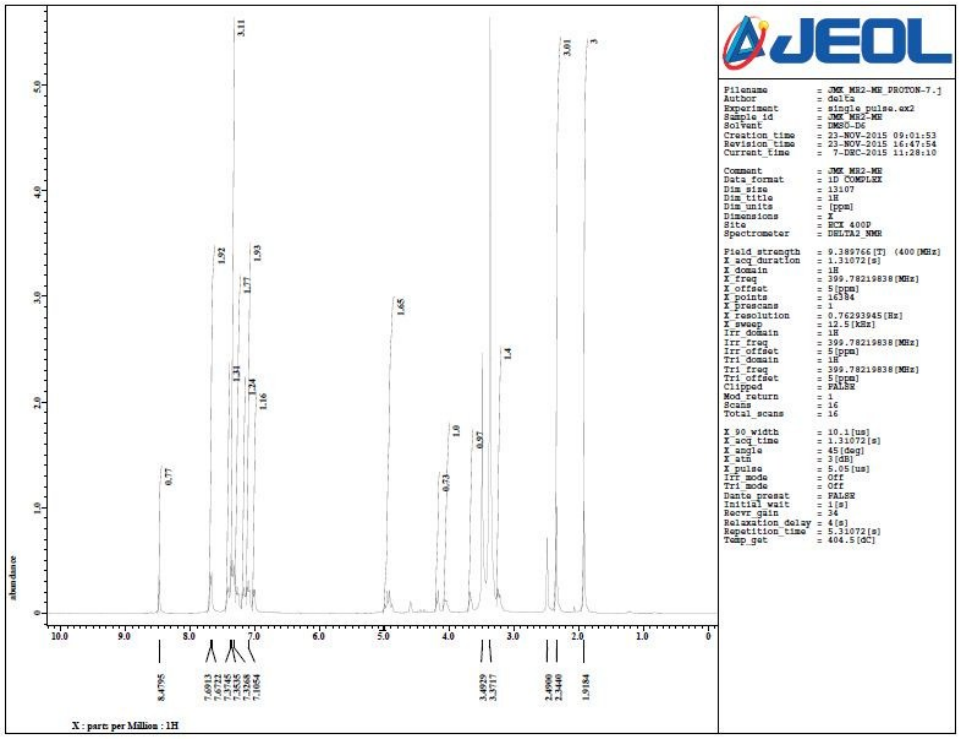



**Figure 50.** $^{13}$C NMR spectrum of compound **5q** (100 MHz, DMSO).

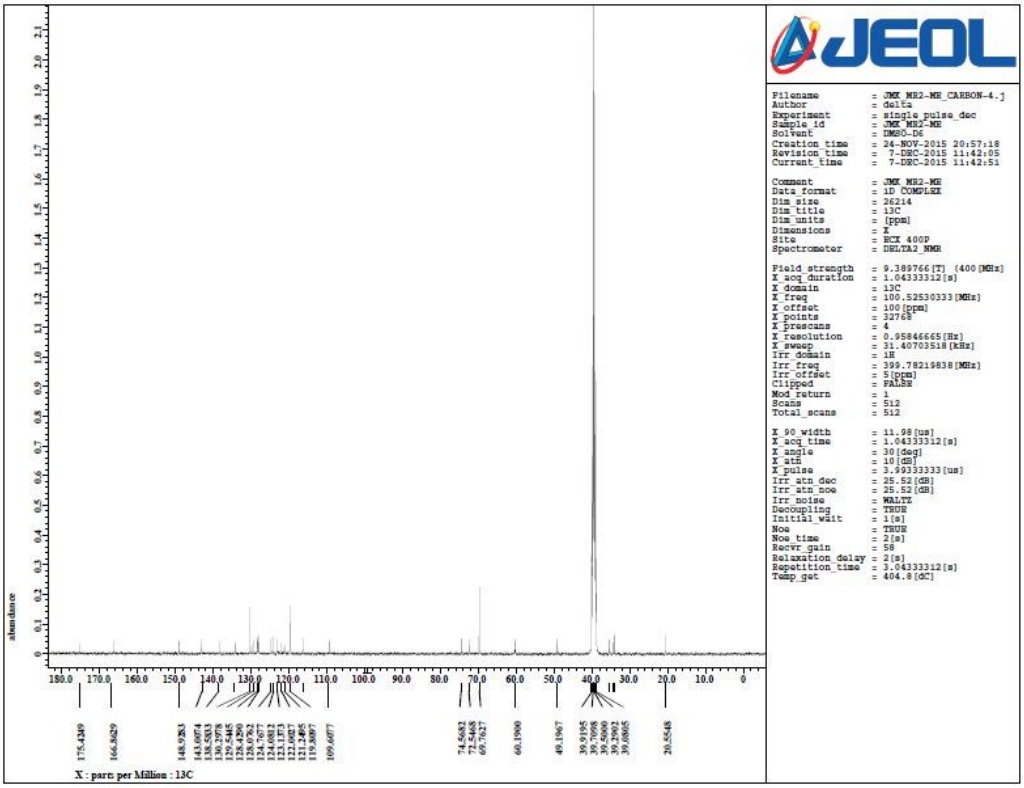


**Figure 51.** Mass spectrum of compound **5q**.

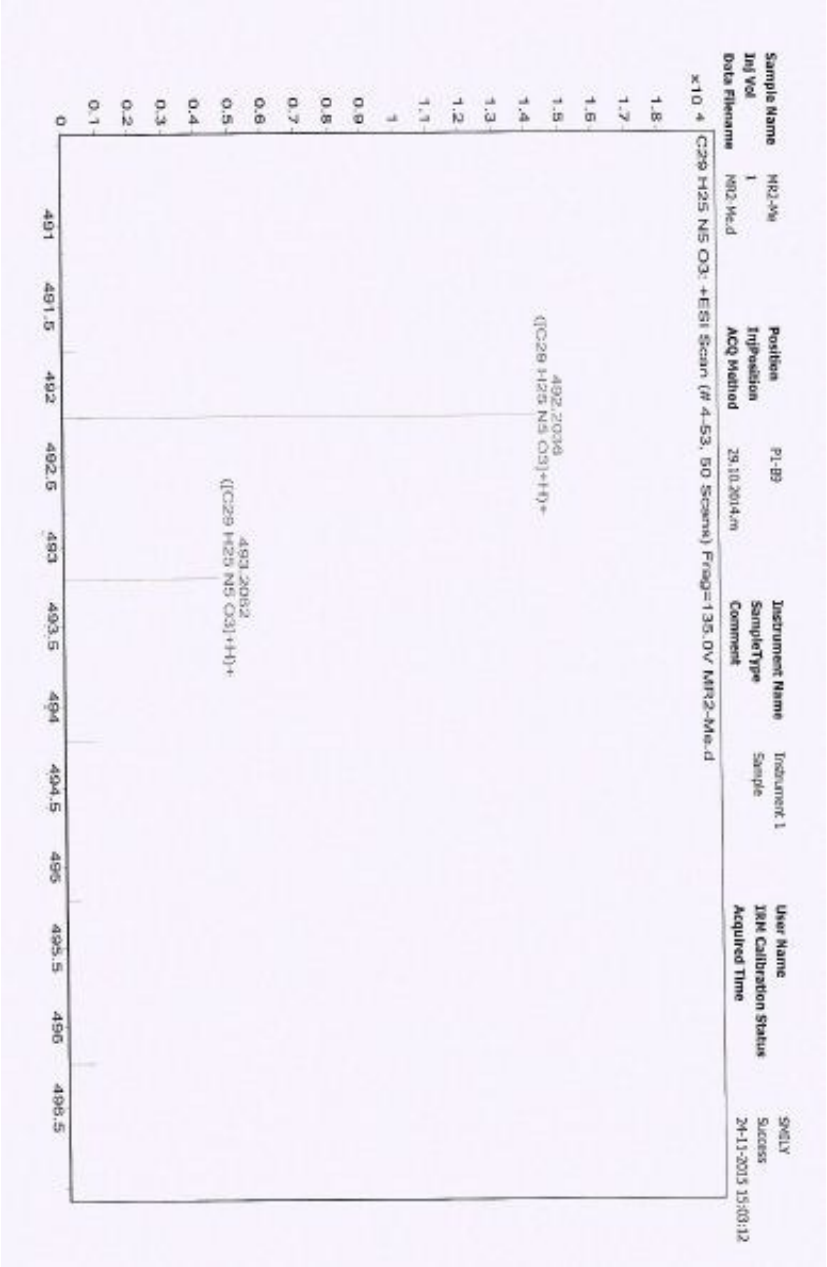


**Figure 52.** $^{1}$H NMR spectrum of compound **5r** (400 MHz, DMSO).

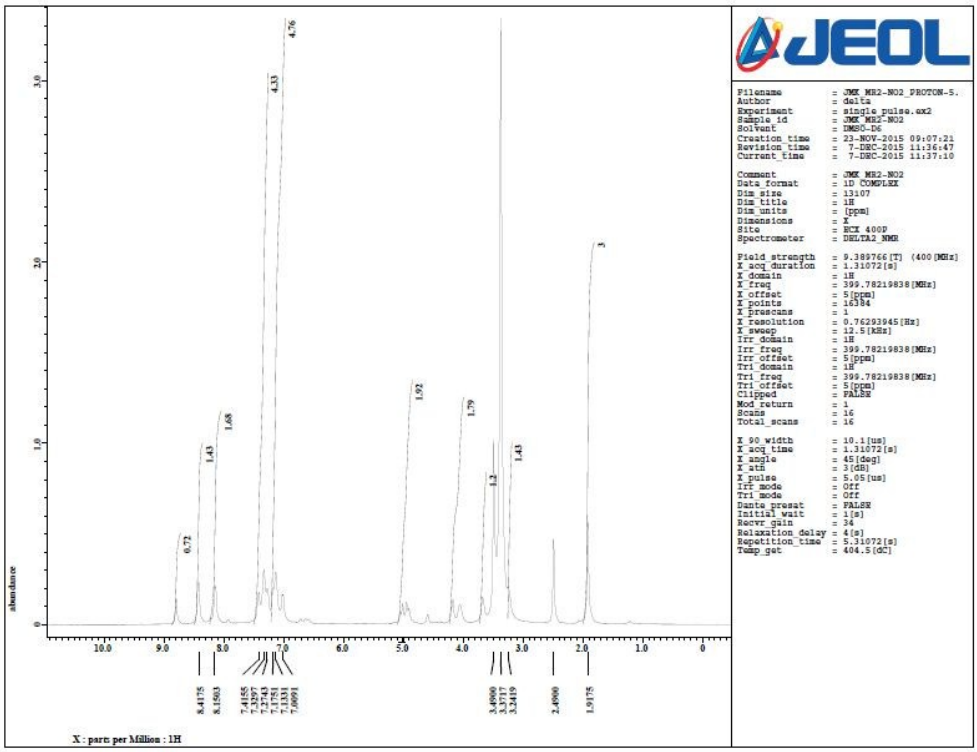


**Figure 53.** $^{13}$C NMR spectrum of compound **5r** (100 MHz, DMSO).

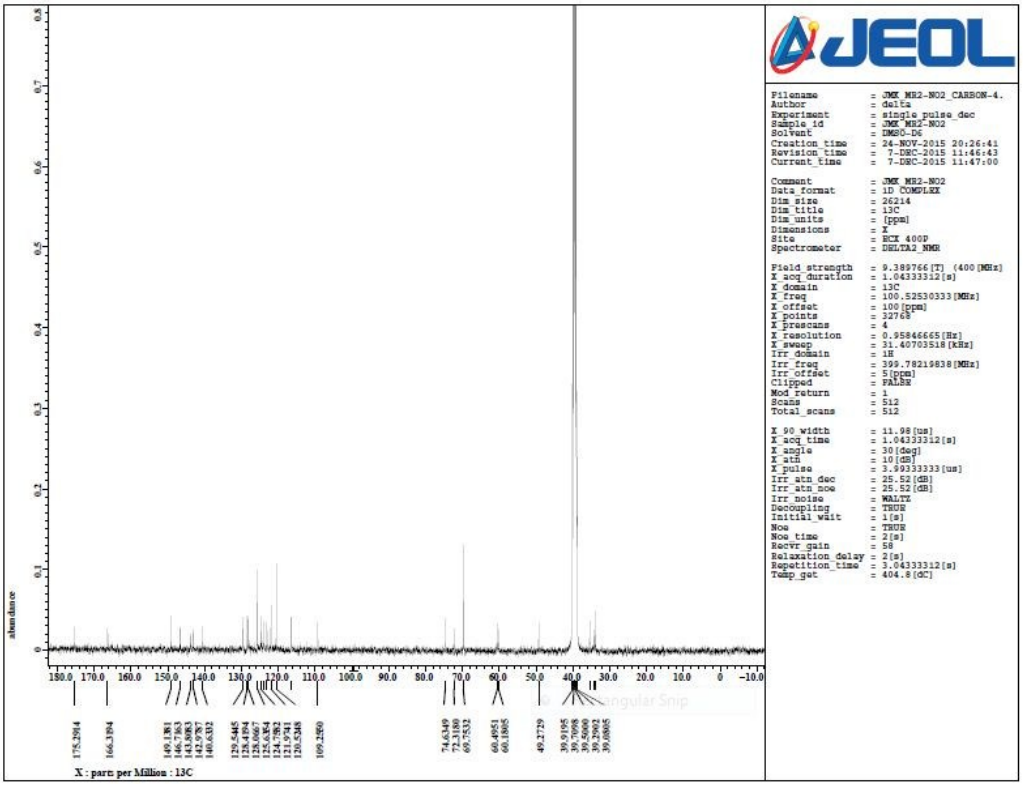
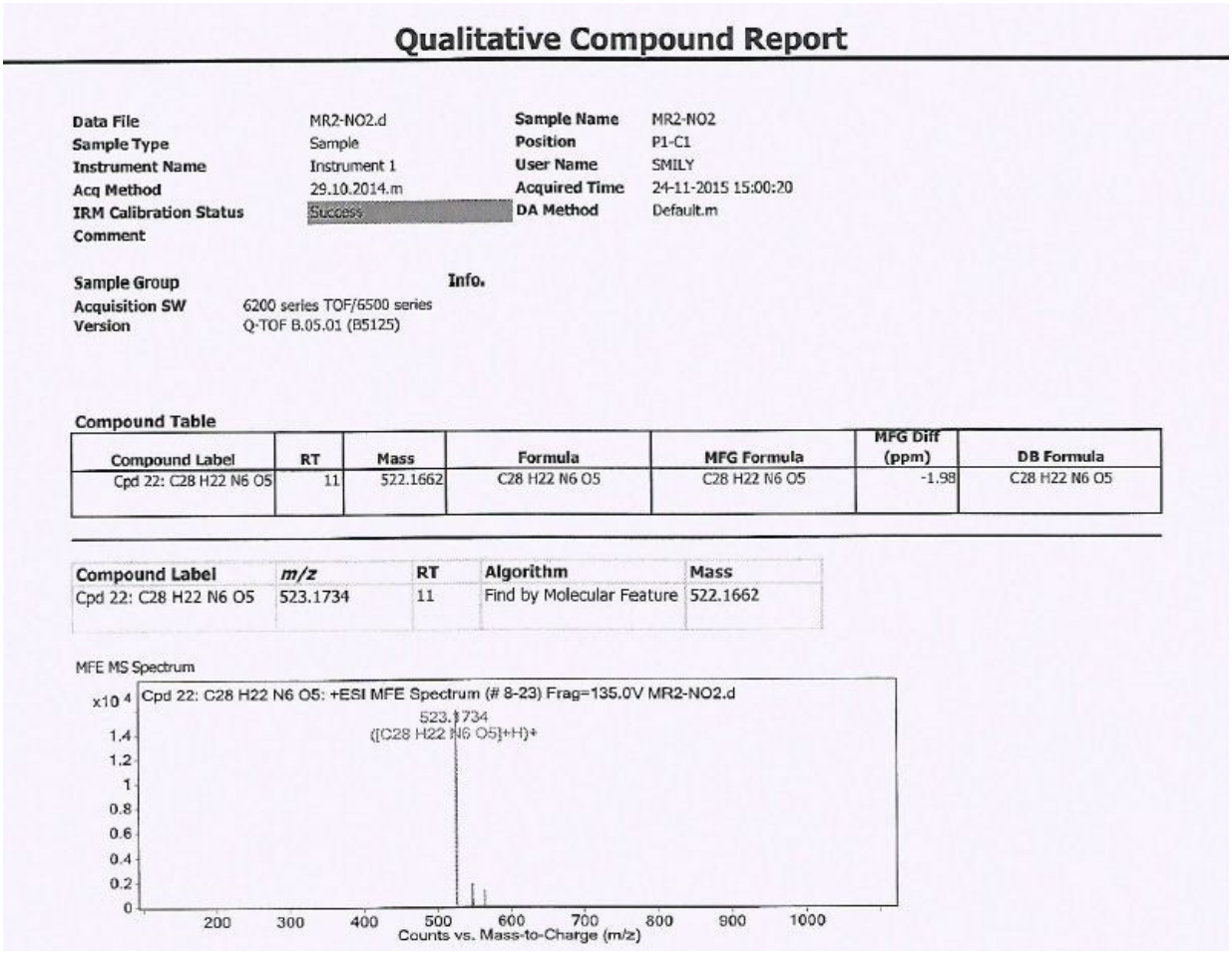


**Figure 54.** Mass spectrum of compound **5r**.



## Qualitative Compound Report

| | | | |
|---|---|---|---|
| **Data File** | MR2-NO2.d | **Sample Name** | MR2-NO2 |
| **Sample Type** | Sample | **Position** | P1-C1 |
| **Instrument Name** | Instrument 1 | **User Name** | SMILY |
| **Acq Method** | 29.10.2014.m | **Acquired Time** | 24-11-2015 15:00:20 |
| **IRM Calibration Status** | Success | **DA Method** | Default.m |
| **Comment** | | | |

**Sample Group** **Info.**

| | |
|---|---|
| **Acquisition SW Version** | 6200 series TOF/6500 series Q-TOF B.05.01 (B5125) |

**Compound Table**

| Compound Label | RT | Mass | Formula | MFG Formula | MFG Diff (ppm) | DB Formula |
|---|---|---|---|---|---|---|
| Cpd 22: C28 H22 N6 O5 | 11 | 522.1662 | C28 H22 N6 O5 | C28 H22 N6 O5 | -1.98 | C28 H22 N6 O5 |

| Compound Label | *m/z* | RT | Algorithm | Mass |
|---|---|---|---|---|
| Cpd 22: C28 H22 N6 O5 | 523.1734 | 11 | Find by Molecular Feature | 522.1662 |

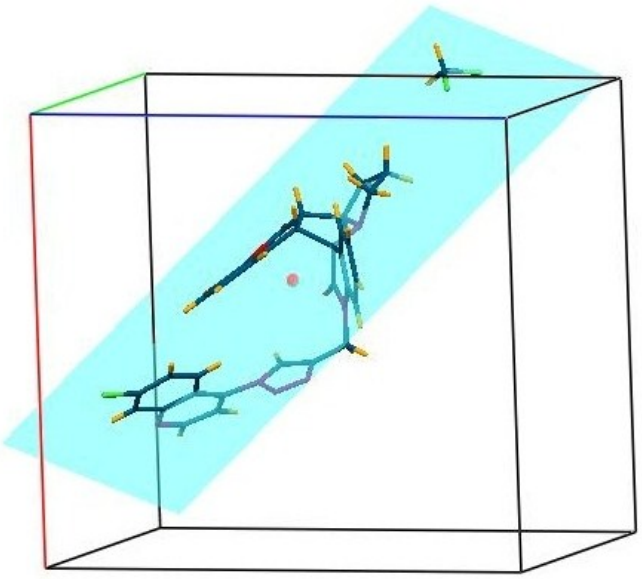
MFE MS Spectrum

**Table 1:** Crystal data and structure refinement information for the title compound **5e**.

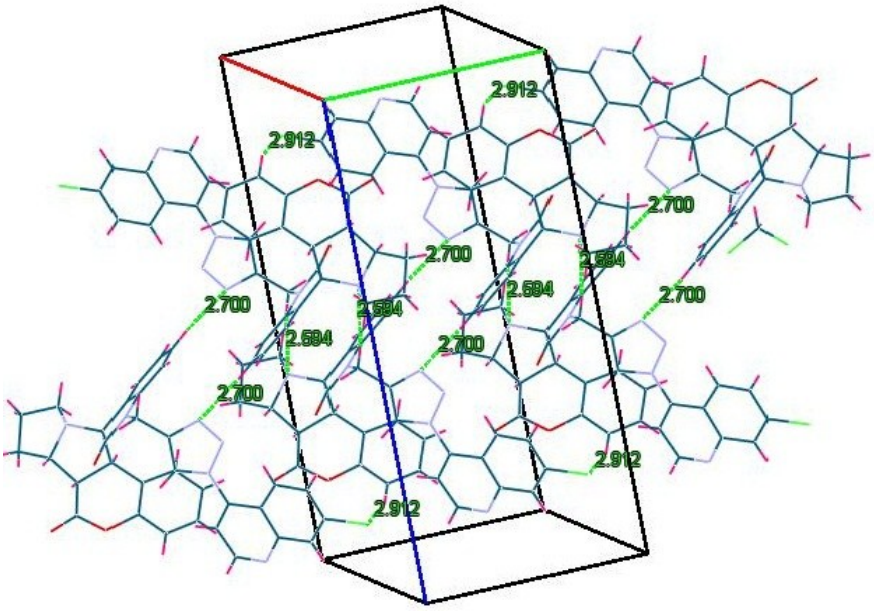
| **Formula** | **'$C_{34}$ $H_{27}$ $Cl_3$ $N_6$ $O_3$'** |
|---|---|
| Formula weight (g) | 673.97 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 Å |
| Crystal system, space group Triclinic | Monoclinic, P21/n |
| Unit cell dimensions | a = 17.3297(8) Å alpha = 90.00 deg. |
| | b = 9.1210(6) Å beta = 92.608(4) deg. |
| | c = 20.2870(10) Å gamma = 90.00 deg. |
| Volume | 3203.3(3)$A^3$ |
| Z | 4 $g/cm^3$ |
| Absorption coefficient | 0.332 $mm^{-1}$ |
| F(000) | 1224 |
| Crystal size | 0.04 X 0.03 X 0.01 mm |
| Theta range for data collection | 3.17 to 21.36 deg |
| Limiting indices | -19<=h<=20, -9<=k<=10, -24<=l<=19 |
| Completeness to theta | 99.9% |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 1.00000 and 0.86823 |
| Refinement method | Full-matrix least-squares on F2 |
| Data / restraints / parameters | 5632/ 0 / 411 |
| Goodness-of-fit on F^2 | 0.955 |
| Final R indices [I>2sigma(I)] | R1 = 0.1009, wR2 = 0.2792 |
| R indices (all data) | R1 = 0.1793, wR2 = 0.3310 |
| Largest diff. peak and hole | 0.60 and -0.54 $e.A^{-3}$ |

The crystal structure shows that spiro carbon of *N*-propargyl isatin and pyrrolizine ring deviates by an angle 110.26° (Fig. 55a). The compound reveals two types of interactions, namely,

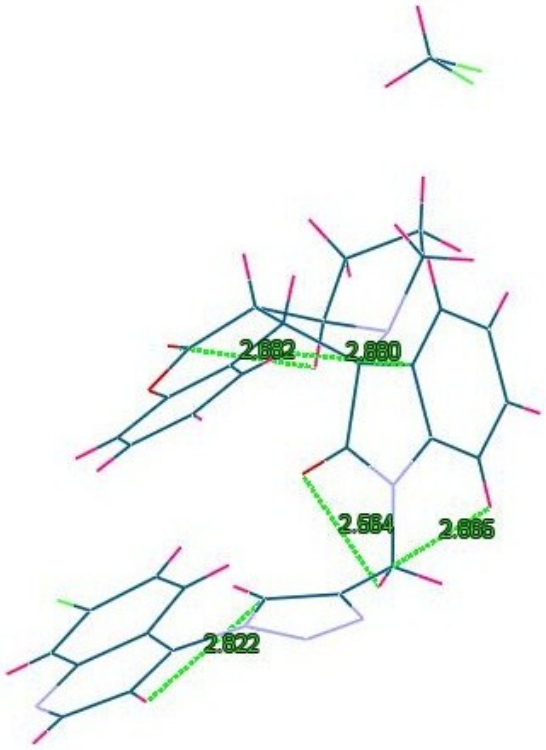
intermolecular and intramolecular hydrogen bonding (Table 2). The intermolecular H-bonding stabilized the molecule by C4-H4…Cl2 and C20-H20A…N6 and C16-H16…N2 with a distance of 2.912, 2.594 and 2.700 Å, respectively (Fig. 55b). The structure also involves intramolecular hydrogen bonding interaction between the C7-H7…O1, C24-H24…C22, C1-H1…C19, C17-H17…C20 and C20-H20B…O3 with a distance of 2.683, 2.822, 2.880, 2.865 and 2.563 Å respectively (Fig. 55c).



(a)




(b)




(c)

**Figure 55:** (a) View of crystal **5e** shown along b axis; (b) Intermolecular hydrogen bonding interactions in the crystal of **5e**; (c) Intramolecular hydrogen bonding in the crystal **5e**.

**Table 2:** Intermolecular and intramolecular hydrogen bonding geometries of **5e**

| D-H…A | D-H (Å) | H…A (Å) | D…A (Å) | D-H…A (°) |
|---|---|---|---|---|
| C7-H7…O3 | 0.98 | 2.52 | 2.9946 (2) | 109 |
| C20-H20A…N6 [i] | 0.97 | 2.59 | 3.5498 (2) | 171 |
| C26-H26…N3 [ii] | 0.93 | 2.56 | 3.0629 (2) | 115 |
| C34-H34A…O1 | 0.97 | 2.59 | 3.5393 (2) | 166 |

Note: D, Donor; A, Acceptor; Symmetry code: i) –x, 1-y, -z; ii) 1/2-x, -1/2+y, 1/2-z.