Supplementary Material (ESI) for Chemical Communications

Palladium-Catalyzed Decarboxylative 1,2-Addition of Carboxylic Acids to Aldehydes or Imines

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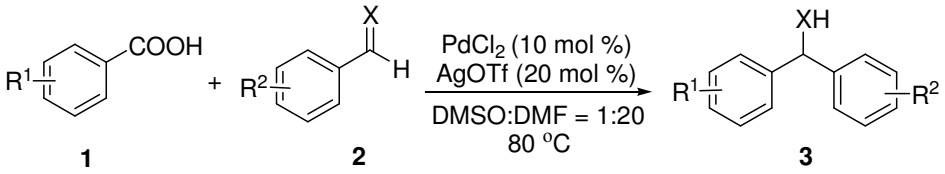
Supporting Information

1. General experimental method (S2)
2. General Experimental procedure (S2)
3. Characterization data of compounds **3** and **4** (S2-S9)
4. ^{1}H and ^{13}C NMR spectra of compounds **3** and **4** (S10-S55)

General experimental method:

All reactions were performed in test tubes under nitrogen atmosphere at 80 °C. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63 μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated at ~20 Torr (house vacuum) at 25–35 °C. Solvents were re-distilled prior to use in the reactions. Other commercial reagents were used as received. (All the products are known compounds. The characterizations of these compounds are identical with the literature reports.)

General procedure for palladium-catalyzed decarboxylative 1,2-addition of carboxylic acids to aldehydes or imines



1 **2** **3**

Carboxylic acid **1** (0.24 mmol) and aldehyde or imine **2** (0.20 mmol) were added to a mixture of $PdCl_2$ (10 mol %, 3.6 mg) and AgOTf (20 mol %, 10.3 mg) in DMF (2.0 mL) and DMSO (0.1 mL). The reaction was stirred at 80 °C. After completion of reaction as indicated in TLC, the mixture was diluted by EtOAc (20 mL), washed with saturated brine (2 x 20 mL), and dried by Na_2SO_4. Evaporation the solvent followed by purification on silica gel provided the product **3**.

(2,6-Dimethoxyphenyl)(4-nitrophenyl)methanol **3a**

White solid, melting point: 136-137 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 8.12 (d, J = 7.8 Hz, 2H), 7.50 (d, J = 8.7 Hz, 2H), 7.26 (m, 1H), 6.60 (d, J = 8.3 Hz, 2H), 6.38 (s, 1H), 4.31 (br, 1H), 3.80 (s, 6H). ^{13}C NMR (100M Hz): δ 157.5, 152.6, 146.6, 129.6, 126.2, 123.1, 118.3, 104.5, 67.8, 55.8. IR (thin film): ν(cm^{-1}) 3536, 1597, 1518, 1476.

HRMS (ESI) calculated for $C_{15}H_{15}NO_5$ $[M+Na]^+$ requires 312.0848, found 312.0848.

(2,4-Dichlorophenyl)(2,6-dimethoxyphenyl)methanol **3b**

White solid, melting point: 106-107 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.35 (d, J = 1.8 Hz, 2H), 7.28-7.23 (m, 2H), 7.14-7.11 (m, 1H), 6.59 (d, J = 8.7 Hz, 2H), 6.50 (br, 1H), 4.19 (br, 1H), 3.78 (s, 6H). ^{13}C NMR (100M Hz): δ 158.0, 139.6, 134.4, 133.1, 129.50, 129.45, 129.36, 126.3, 116.9, 104.5, 66.5, 55.8. IR (thin film): ν(cm^{-1}) 3530, 1594, 1560, 1474. HRMS (ESI) calculated for $C_{15}H_{14}Cl_2O_3$ $[M+Na]^+$ requires 335.0218, found 335.0223.

(2-Chlorophenyl)(2,6-dimethoxyphenyl)methanol **3c**

White solid, melting point: 99-100 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.36-7.34 (m, 1H), 7.30-7.23 (m, 2H), 7.12-7.18 (m, 2H), 6.60 (d, J = 8.3 Hz, 2H), 6.57 (s, 1H), 4.27(br, 1H) 3.79 (s, 6H). ^{13}C NMR (100M Hz): δ 158.1, 140.8, 133.9, 129.7, 129.2, 128.5, 128.3, 126.1, 117.3, 104.5, 67.0, 55.8. IR (thin film): ν(cm^{-1}) 3531, 1595, 1475. HRMS (ESI) calculated for $C_{15}H_{15}ClO_3$ $[M+Na]^+$ requires 301.0607, found 301.0618.

(2-Bromo-4-fluorophenyl)(2,6-dimethoxyphenyl)methanol **3d**

White solid, melting point: 110-111 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.33-7.29 (m, 1H) , 7.24-7.27 (m, 1H), 7.21-7.23 (m, 1H), 6.87-6.92 (m, 1H), 6.61 (d, J = 8.2 Hz, 2H), 6.47 (s, 1H), 4.33(br, 1H), 3.80 (s, 6H). ^{13}C NMR (100M Hz): δ 161.4 (d, J (C,F) = 248.9 Hz), 158.0, 138.4, 129.7 (d, J (C,F) = 8.6 Hz), 129.4, 124.1 (d, J (C,F) = 9.5 Hz), 120.1(d, J (C,F) = 24.8 Hz), 117.2, 113.7(d, J (C,F) = 20.0 Hz), 104.5, 68.6, 55.8. IR (thin film): ν(cm^{-1}) 3531, 1595, 1476. HRMS (ESI) calculated for $C_{15}H_{14}BrFO_3$ $[M+Na]^+$ requires 363.0008, found 363.0015.

(2,6-Dimethoxyphenyl)(4-(trifluoromethyl)phenyl)methanol **3e**

White solid, melting point: 100.6-101.7 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.52 (d, J = 8.2 Hz, 2H), 7.46 (d, J = 8.7 Hz, 2H), 7.25-7.23 (m, 1H), 6.60 (d, J = 8.7 Hz, 2H), 6.35 (d, J = 11.0 Hz, 2H), 4.36 (d, J = 11.9 Hz, 1H), 3.79(s, 6H). ^{13}C NMR (100M

Hz): δ 157.6, 148.9, 129.3, 128.6 (q, J (C,F) = 31.4 Hz), 125.8, 124.3 (q, J (C,F) = 270.7 Hz), 124.8 (q, J (C,F) = 3.8 Hz), 118.8, 104.5, 68.0, 55.8. IR (thin film): ν(cm^{-1}) 3564, 1594, 1475. HRMS (ESI) calculated for $C_{16}H_{15}F_3O_3$ $[M+Na]^+$ requires 335.0871, found 335.0875.

(2,6-Dimethoxyphenyl)(2-nitrophenyl)methanol **3f**

White solid, melting point: 93-94 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.66-7.69 (m, 1H), 7.32-7.42 (m, 3H), 7.24-7.28 (m, 1H), 6.84 (d, J = 10.1 Hz, 1H), 6.60 (d, J = 8.2 Hz, 2H), 4.40 (d, J = 10.5 Hz, 1H), 3.76 (s, 6H). ^{13}C NMR (100M Hz): δ 157.9, 149.6, 137.9, 131.5, 129.7, 128.2, 127.7, 123.8, 116.4, 104.4, 65.2, 55.7. IR (thin film): ν(cm^{-1}) 3523, 1595, 1530, 1475. HRMS (ESI) calculated for $C_{15}H_{15}NO_5$ $[M+Na]^+$ requires 312.0848, found 312.0856.

Methyl 4-((2,6-dimethoxyphenyl)(hydroxy)methyl)benzoate **3g**

White solid, melting point: 129-130 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.94(d, J = 8.2 Hz, 2H), 7.41 (d, J = 7.8 Hz, 2H), 7.23 (d, J = 8.3 Hz, 2H), 6.59 (d, J = 8.3 Hz, 2H), 6.36 (d, J = 10.5 Hz, 1H), 4.30(d, J = 11.4 Hz, 1H), 3.88(s, 3H), 3.78 (s, 6H). ^{13}C NMR (100M Hz): δ 167.2, 157.6, 150.3, 129.2, 128.2, 125.4, 119.0, 104.5, 68.1, 55.8, 51.9. IR (thin film): ν(cm^{-1}) 3539, 1719, 1596, 1475. HRMS (ESI) calculated for $C_{17}H_{18}O_5$ $[M+Na]^+$ requires 325.1052, found 325.1055.

(2-Bromo-5-fluorophenyl)(2,6-dimethoxyphenyl)methanol **3h**

White solid, melting point: 81-82 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.51-7.47 (m, 1H), 7.29-7.24 (m, 1H), 7.07-7.04 (m, 1H), 6.83-6.79 (m, 1H), 6.60 (d, J = 8.7 Hz, 2H), 6.44 (dr, 1H), 4.25 (dr, 1H), 3.80 (s, 6H). ^{13}C NMR (100M Hz): δ 161.8 (d, J (C,F) = 244.1 Hz), 158.0, 144.8(d, J (C,F) = 6.6 Hz), 134.0 (d, J (C,F) = 7.6 Hz), 129.6, 117.8, 116.7, 116.0(d, J (C,F) = 23.9 Hz), 115.5 (d, J (C,F) = 21.9 Hz), 104.5, 68.9, 55.8. IR (thin film): ν(cm^{-1}) 3518, 1595, 1475. HRMS (ESI) calculated for $C_{15}H_{14}BrFO_3$ $[M+Na]^+$ requires 363.0008, found 363.0014.

N-((2,6-Dimethoxyphenyl)(4-nitrophenyl)methyl)-4-methylbenzenesulfonamide **3i**

White solid, melting point: 115-116 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 8.06 (d, J = 8.7 Hz, 2H), 7.52 (d, J = 8.2 Hz, 2H), 7.43 (d, J = 8.7 Hz, 2H), 7.10 (t, J = 8.2 Hz, 1H), 7.01 (d, J = 8.2 Hz, 2H), 6.41 (d, J = 10.5 Hz, 1H), 6.35 (d, J = 8.2 Hz, 2H), 6.25 (d, J = 10.5 Hz, 1H), 3.65 (s, 6H), 2.30 (s, 3H). ^{13}C NMR (100M Hz): δ 156.9, 148.9, 146.7, 142.9, 137.1, 129.7, 128.8, 127.1, 126.8, 123.2, 115.1, 103.9, 55.7, 51.1, 21.3. IR (thin film): ν(cm^{-1}) 3358, 3262, 1597, 1519, 1476. HRMS (ESI) calculated for $C_{22}H_{22}N_2O_6S$ $[M+Na]^+$ requires 465.1096, found 465.1106.

4-Methyl-*N*-((4-nitrophenyl)(2,4,6-trimethoxyphenyl)methyl)benzenesulfonamide **3j**

White solid, melting point: 138-139 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 8.03 (d, J = 8.7 Hz, 2H), 7.54 (d, J = 8.2 Hz, 2H), 7.43 (d, J = 8.7 Hz, 2H), 7.04 (d, J = 8.2 Hz, 2H), 6.37 (d, J = 10.5 Hz, 1H), 6.15 (d, J = 10.5 Hz, 1H), 5.9 (s, 2H), 3.74 (s, 3H), 3.62 (s, 6H), 2.31 (s, 3H). ^{13}C NMR (100M Hz): δ 161.3, 157.6, 149.4, 146.6, 142.8, 137.2, 128.8, 127.1, 126.8, 123.1, 107.7, 90.4, 55.5, 55.3, 51.0, 21.3. IR (thin film): ν(cm^{-1}) 3317, 1597, 1518, 1495. HRMS (ESI) calculated for $C_{23}H_{24}N_2O_7S$ $[M+Na]^+$ requires 495.1202, found 495.1209. Elem. Anal. Calcd for $C_{23}H_{24}N_2O_7S$: C, 58.46; H, 5.12; N, 5.93; Found: C, 58.76; H, 4.96; N, 5.88.

N-((2,4-Dichlorophenyl)(2,6-dimethoxyphenyl)methyl)-4-methylbenzenesulfonamid **3k**

White solid, melting point: 185-186 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.51 (d, J = 8.2 Hz, 2H), 7.33 (d, J = 8.7 Hz, 1H), 7.27 (d, J = 2.3 Hz, 1H), 7.08-7.05 (m, 2H), 7.01 (d, J = 8.2 Hz, 2H), 6.44 (d, J = 10.0 Hz, 1H), 6.31 (d, J = 8.7 Hz, 2H), 6.14 (d, J = 10.5 Hz, 1H), 3.65 (s, 6H), 2.30 (s, 3H). ^{13}C NMR (100M Hz): δ 157.3, 142.6, 137.4, 136.8, 134.1, 133.2, 130.5, 129.4, 128.7, 126.8, 126.2, 114.2, 103.9, 55.5, 50.0, 21.3. IR (thin film): ν(cm^{-1}) 3290, 1595, 1475. HRMS (ESI) calculated for $C_{22}H_{21}Cl_2NO_4S$ $[M+Na]^+$ requires 488.0466, found 488.0475.

N-((2,4-Dichlorophenyl)(2,4,6-trimethoxyphenyl)methyl)-4-methylbenzenesulfonami

de **3l**

White solid, melting point: 213-214 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.52 (d, J = 8.3 Hz, 2H), 7.34 (d, J = 8.7 Hz, 1H), 7.26 (m, 1H), 7.07-7.02 (m, 3H), 6.32 (d, J = 10.1 Hz, 1H), 6.04 (m, 1H), 5.85 (s, 2H), 2.73 (s, 3H), 3.61 (s, 6H), 2.32 (s, 3H). ^{13}C NMR (100M Hz): δ 161.2, 158.0, 142.5, 137.5, 137.1, 134.0, 133.0, 130.5, 129.3, 128.7, 126.8, 126.1, 106.9, 90.4, 55.4, 55.2, 49.8, 21.3. IR (thin film): ν(cm^{-1}) 3273, 1592, 1494, 1466. HRMS (ESI) calculated for $C_{23}H_{23}Cl_2NO_5S$ $[M+Na]^+$ requires 518.0572, found 518.0574.

N-((2-Bromo-4-fluorophenyl)(2,6-dimethoxyphenyl)methyl)-4-methylbenzenesulfonamide **3m**

White solid, melting point: 180-181 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.49 (d, J = 8.2 Hz, 2H), 7.33-7.30 (m, 1H), 7.26-7.22 (m, 1H), 7.06 (t, J = 8.2 Hz, 1H), 6.99 (d, J = 8.2 Hz, 2H), 6.86-6.82 (m, 1H), 6.45 (d, J = 10.5 Hz, 1H), 6.30 (d, J = 8.3 Hz, 2H), 6.12 (d, J = 10.5 Hz, 1H), 3.66 (s, 6H), 2.29 (s, 3H). ^{13}C NMR (100M Hz): δ 161.3 (d, J (C,F) = 248.9 Hz), 157.3, 142.6, 137.4, 135.6, 130.9, 130.8(d, J (C,F) = 8.6 Hz), 129.3, 128.6, 126.7, 123.8(d, J (C,F) = 9.5 Hz), 120.1(d, J (C,F) = 24.8 Hz), 114.3, 113.8 (d, J (C,F) = 21.0 Hz), 103.9, 55.5, 52.3, 21.3. IR (thin film): ν(cm^{-1}) 3286, 1595, 1476. HRMS (ESI) calculated for $C_{22}H_{21}BrFNO_4S$ $[M+Na]^+$ requires 516.0256, found 516.0247.

N-((2-Bromo-4-fluorophenyl)(2,4,6-trimethoxyphenyl)methyl)-4-methylbenzenesulfonamide **3n**

White solid, melting point: 206-207 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.50 (d, J = 8.2 Hz, 2H), 7.33-7.30 (m, 1H), 7.21-7.24 (m, 1H), 7.02 (d, J = 8.2 Hz, 2H), 6.87-6.81 (m, 1H), 6.33 (d, J = 10.1 Hz, 1H), 6.00 (d, J = 10.5 Hz, 1H), 5.86 (s, 2H), 3.73 (s, 3H), 3.63 (s, 6H), 2.32 (s, 3H). ^{13}C NMR (100M Hz): δ 161.2 (d, J (C,F) = 248.9 Hz), 161.1, 158.0, 142.4, 137.5, 135.8, 130.9 (d, J (C,F) = 8.6 Hz), 128.6, 126.8, 123.7 (d, J (C,F) = 9.6 Hz), 120.0 (d, J (C,F) = 23.8 Hz), 113.6 (d, J (C,F) = 21.0 Hz), 107.0, 90.5, 55.4, 55.2, 52.1, 21.3. IR (thin film): ν(cm^{-1}) 3270, 1594, 1494, 1461.

HRMS (ESI) calculated for $C_{23}H_{23}BrFNO_5S$ $[M+Na]^+$ requires 546.0362, found 546.0350.

N-((2-Chlorophenyl)(2,4,6-trimethoxyphenyl)methyl)-4-methylbenzenesulfonamide **3o**

White solid, melting point: 197-198 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.51 (d, J = 8.2 Hz, 2H), 7.37-7.35 (m, 1H), 7.28-7.26 (m, 1H), 7.11-7.08 (m, 2H), 7.01 (d, J = 8.2 Hz, 2H), 6.41 (d, J = 10.5 Hz, 1H), 6.04 (d, J = 10.5 Hz, 1H), 5.86 (s, 2H), 3.72 (s, 3H), 3.62 (s, 6H), 2.31 (s, 3H). ^{13}C NMR (100M Hz): δ 161.0, 158.1, 142.3, 138.2, 137.6, 133.5, 129.7, 129.4, 128.6, 128.1, 128.1, 126.8, 125.9, 107.3, 90.5, 55.4, 55.2, 50.3, 21.3. IR (thin film): ν(cm^{-1}) 3279, 1592, 1494, 1439. HRMS (ESI) calculated for $C_{23}H_{24}ClNO_5S$ $[M+Na]^+$ requires 484.0961, found 484.0958. Elem. Anal. Calcd for $C_{23}H_{24}ClNO_5S$: C, 59.80; H, 5.24; N, 3.03; Found: C, 59.63; H, 5.32; N, 2.93.

N-((2,6-Dimethoxyphenyl)(2-nitrophenyl)methyl)-4-methylbenzenesulfonamide **3p**

White solid, melting point: 162-163 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.52 (d, J = 8.2 Hz, 2H), 7.43 (m, 2H), 7.10 (t, J = 8.2 Hz, 1H), 7.01 (d, J = 7.8 Hz, 2H), 6.40-6.34 (m, 3H), 6.25 (d, J = 10.6 Hz, 1H), 3.69 (s, 6H), 2.30 (s, 3H). ^{13}C NMR (100M Hz): δ 156.9, 148.9, 146.7, 142.9, 137.1, 129.7, 128.8, 127.2, 126.8, 123.2, 115.1, 103.9, 55.7, 51.1, 21.4. IR (thin film): ν(cm^{-1}) 3270, 1597, 1518, 1477. HRMS (ESI) calculated for $C_{22}H_{22}N_2O_6S$ $[M+Na]^+$ requires 465.1096, found 465.1093. Elem. Anal. Calcd for $C_{22}H_{22}N_2O_6S$: C, 59.72; H, 5.01; N, 6.33; Found: C, 59.86; H, 4.77; N, 6.22.

N-((2-Bromo-5-fluorophenyl)(2,6-dimethoxyphenyl)methyl)-4-methylbenzenesulfonamide **3q**

White solid, melting point: 194-195 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.51 (d, J = 8.2 Hz, 2H), 7.45-7.41 (m, 1H), 7.13-7.05 (m, 2H), 7.00 (d, J = 7.8 Hz, 2H), 6.79-6.76 (m, 1H), 6.44 (d, J = 10.6 Hz, 1H), 6.31 (d, J = 8.7 Hz, 2H), 6.08 (d, J = 10.6 Hz, 1H), 3.67 (s, 6H), 2.30 (s, 3H). ^{13}C NMR (100M Hz): δ 161.6 (d, J (C,F) =

244.1 Hz), 157.4, 142.6, 141.8 (d, *J* (C,F) = 7.6 Hz), 137.4, 134.2(d, *J* (C,F) = 7.6 Hz), 129.5, 128.7, 126.8, 117.6, 117.1 (d, *J* (C,F) = 24.8 Hz), 115.7 (d, *J* (C,F) = 21.9 Hz), 114.0, 104.0, 55.5, 52.8, 21.3. IR (thin film): ν(cm^{-1}) 3292, 1596, 1475. HRMS (ESI) calculated for $C_{22}H_{21}BrFNO_4S$ $[M+Na]^+$ requires 516.0256, found 516.0247.

N-((2,6-Dimethoxyphenyl)(4-(trifluoromethyl)phenyl)methyl)-4-methylbenzenesulfonamide **3r**

White solid, melting point: 168-169 °C. ^1H NMR (400M Hz, $CDCl_3$): δ 7.50(d, *J* = 7.8 Hz, 2H), 7.45 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.2 Hz, 2H), 7.07 (t, *J* = 8.2 Hz, 1H), 6.98 (d, *J* = 8.2 Hz, 2H), 6.43 (d, *J* = 10.6 Hz, 1H), 6.33 (d, *J* = 8.2 Hz, 2H), 6.23 (d, *J* = 11.0 Hz, 1H), 3.64 (s, 6H), 2.28 (s, 3H). ^{13}C NMR (100M Hz): δ 157.0, 145.2, 142.7, 137.2, 129.3, 128.8 (q, *J* (C,F) = 31.5 Hz), 128.7, 126.7, 126.6, 124.8 (q, *J* (C,F) = 3.8 Hz), 124.2 (q, *J* (C,F) = 269.8 Hz), 115.4, 103.9, 55.6, 51.2, 21.3. IR (thin film): ν(cm^{-1}) 3288, 1596, 1494, 1462. HRMS (ESI) calculated for $C_{23}H_{22}F_3NO_4S$ $[M+Na]^+$ requires 488.1119, found 488.1126.

4-Methyl-*N*-((4-(trifluoromethyl)phenyl)(2,4,6-trimethoxyphenyl)methyl)benzenesulfonamide **3s**

White solid, melting point: 121-122 °C. ^1H NMR (400M Hz, $CDCl_3$): δ 7.51 (d, *J* = 8.2 Hz, 2H), 7.45 (d, *J* = 8.2 Hz, 2H), 7.37 (d, *J* = 8.2 Hz, 2H), 7.01 (d, *J* = 7.8 Hz, 2H), 6.32 (d, *J* = 10.6 Hz, 1H), 6.12 (d, *J* = 10.6 Hz, 1H), 5.90 (s, 2H), 3.74 (s, 3H), 3.62 (s, 6H), 2.30 (s, 3H). ^{13}C NMR (100M Hz): δ 161.1, 157.7, 145.6, 142.6, 137.3, 128.8(q, *J* (C,F) = 42.4 Hz), 128.7, 126.8, 126.6, 124.7(q, *J* (C,F) = 3.8 Hz), 124.2 (q, *J* (C,F) = 270.8 Hz), 108.1, 90.5, 55.5, 55.3, 51.1, 21.3. IR (thin film): ν(cm^{-1}) 3296, 1609, 1496. HRMS (ESI) calculated for $C_{24}H_{24}F_3NO_5S$ $[M+Na]^+$ requires 518.1225, found 518.1236.

2,2'-((2,4-Dichlorophenyl)methylene)bis(1,3,5-trimethoxybenzene) **4a**

White solid, melting point: 162-163 °C. ^1H NMR (400M Hz, $CDCl_3$): δ 7.25 (s, 1H), 7.03-7.00(m, 1H), 6.94 (d, *J* = 8.7 Hz, 1H), 6.22 (s, 1H), 6.09 (s, 4H), 3.77 (s, 6H),

3.51 (s, 12H). ^{13}C NMR (100M Hz): δ 159.7, 159.4, 141.8, 134.1, 131.5, 130.6, 127.9, 125.9, 113.0, 91.9, 56.3, 55.2, 35.5. IR (thin film): ν(cm^{-1}) 1590, 1491, 1465. HRMS (ESI) calculated for $C_{25}H_{26}Cl_2O_6$ [M+Na]$^+$ requires 515.1004, found 515.1010.

2,2'-((2-Chlorophenyl)methylene)bis(1,3,5-trimethoxybenzene) **4b**

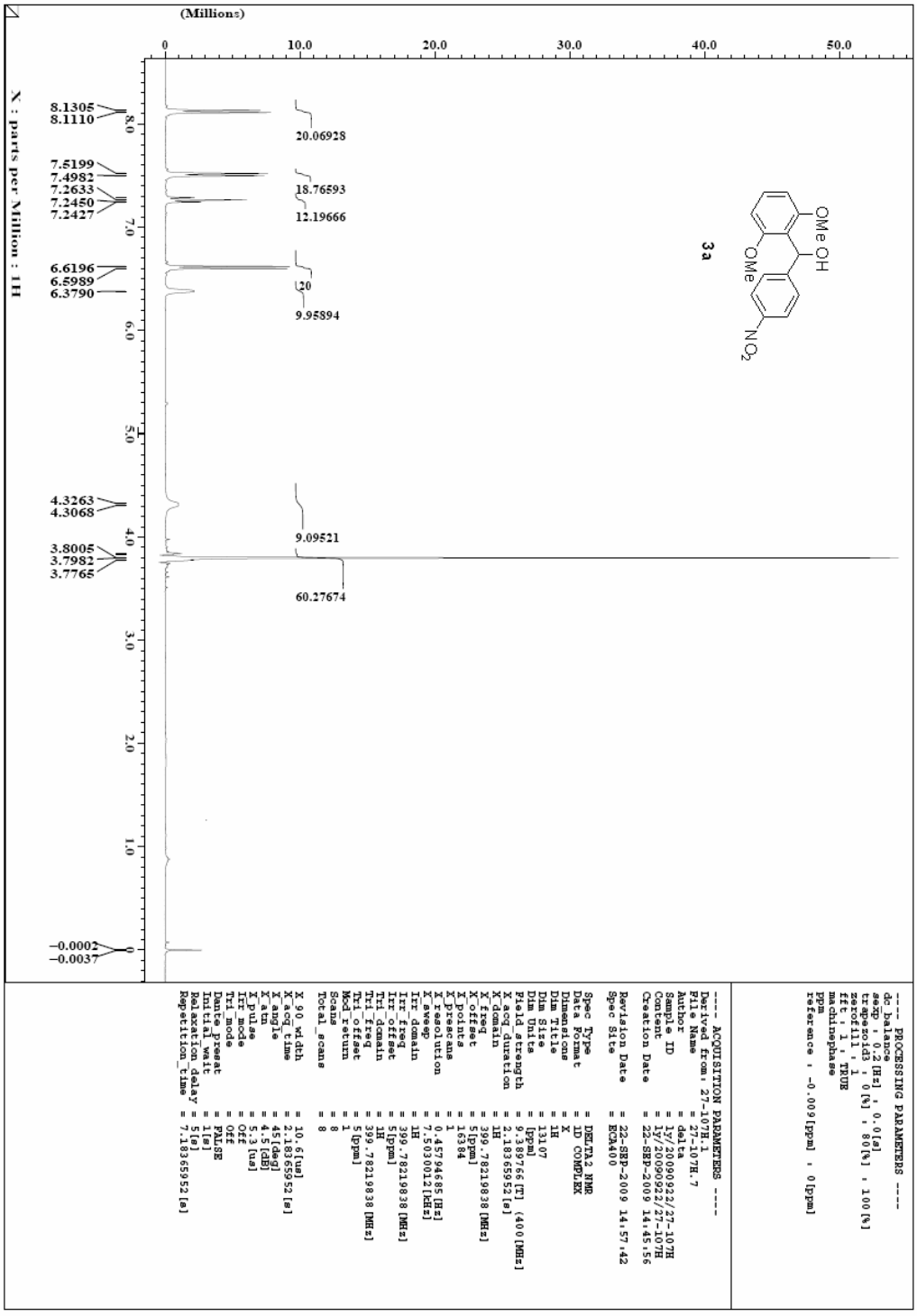
White solid, melting point: 193-194 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.25-7.23 (m, 1H), 7.04-7.00 (m, 3H), 6.29 (s, 1H), 6.10 (s, 4H), 3.77 (s, 6H), 3.49 (s, 12H). ^{13}C NMR (100M Hz): δ 159.7, 159.1, 142.8, 133.4, 130.4, 128.2, 125.8, 125.6, 113.6, 92.0, 56.3, 55.1, 35.8. IR (thin film): ν(cm^{-1}) 1591, 1492, 1466. HRMS (ESI) calculated for $C_{25}H_{27}ClO_6$ [M+Na]$^+$ requires 481.1394, found 481.1392. Elem. Anal. Calcd for $C_{25}H_{27}ClO_6$: C, 65.43; H, 5.93; Found: C, 65.29; H, 6.01.

2,2'-((2-Bromo-4-fluorophenyl)methylene)bis(1,3,5-trimethoxybenzene) **4c**

White solid, melting point: 192-193 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.20-7.17 (m, 1H), 6.98-7.02(m, 1H), 6.79-6.84 (m, 1H), 6.19 (s, 1H), 6.10 (s, 4H), 3.78 (s, 6H), 3.51 (s, 12H). ^{13}C NMR (100M Hz): δ 160.0 (d, J (C,F) = 244.0 Hz), 159.6, 159.3, 140.3, 131.4 (d, J (C,F) = 7.6 Hz), 123.3 (d, J (C,F) = 9.6 Hz), 118.4 (d, J (C,F) = 23.8 Hz), 113.4, 113.0 (d, J (C,F) = 20.0 Hz), 91.9, 56.3, 55.1, 37.6. IR (thin film): ν(cm^{-1}) 1592, 1481, 1455. HRMS (ESI) calculated for $C_{25}H_{26}BrFO_6$ [M+Na]$^+$ requires 543.0794, found 543.0786.

2,2'-((4-(Trifluoromethyl)phenyl)methylene)bis(1,3,5-trimethoxybenzene) **4d**

White solid, melting point: 175-176 °C. ^{1}H NMR (400M Hz, $CDCl_3$): δ 7.39 (d, J = 8.2 Hz, 2H), 7.13 (d, J = 7.8 Hz, 2H), 6.24 (s, 1H), 6.11 (s, 4H), 3.79 (s, 6H), 3.51 (s, 12H). ^{13}C NMR (100M Hz): δ 159.7, 159.4, 150.2, 127.9, 126.3(q, J (C,F) = 31.4 Hz), 124.8 (q, J (C,F) = 271.7 Hz), 123.9(q, J (C,F) = 3.8 Hz), 113.1, 91.6, 56.0, 55.1, 37.0. IR (thin film): ν(cm^{-1}) 1590, 1492, 1456. HRMS (ESI) calculated for $C_{26}H_{27}F_3O_6$ [M+Na]$^+$ requires 515.1657, found 515.1655.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7.2427
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6.5989
6.3790
4.3263
4.3068
3.8005
3.7982
3.7765
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−0.0037
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12.19666
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9.09521
60.27674
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OMe
NO2
3a
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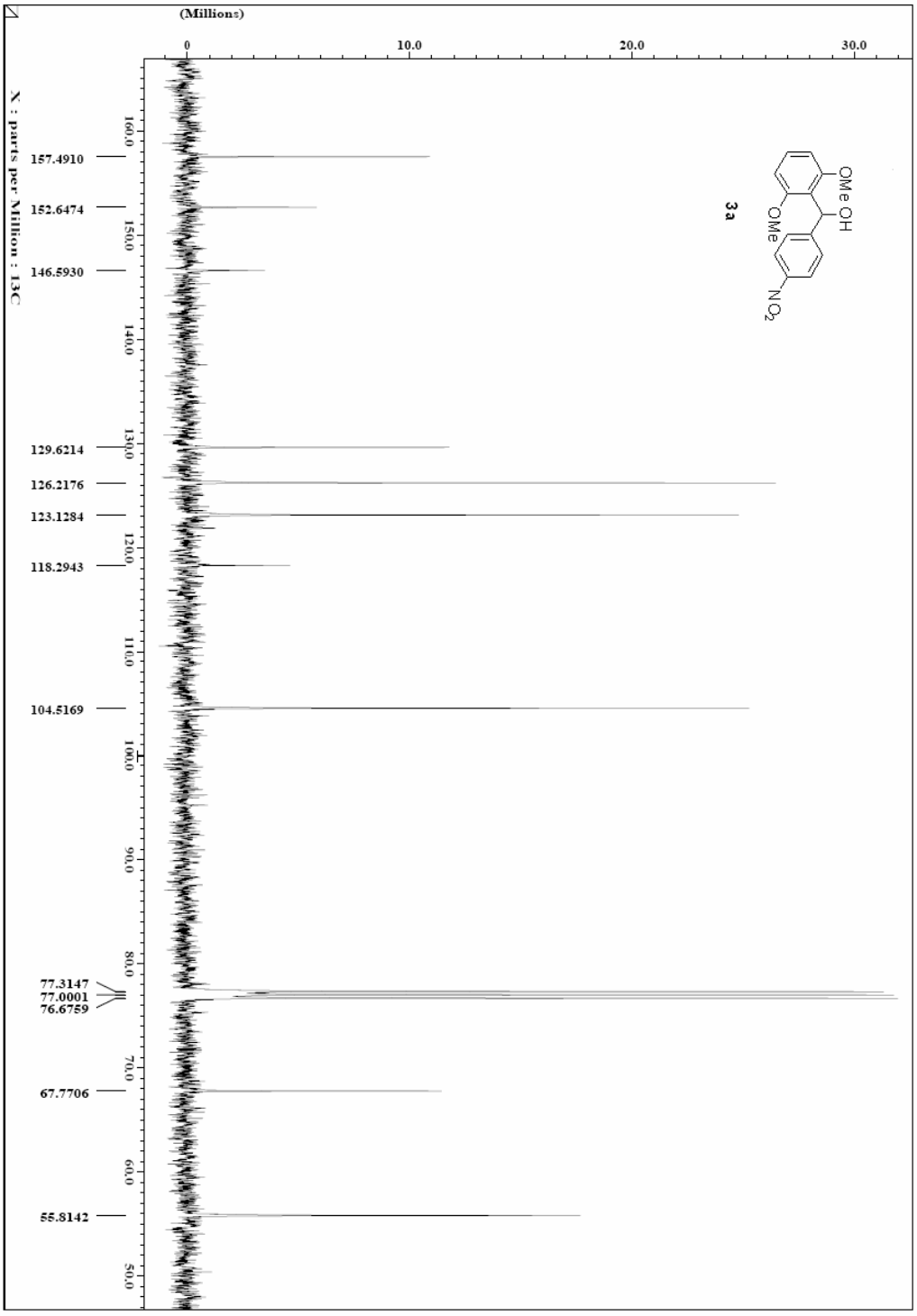
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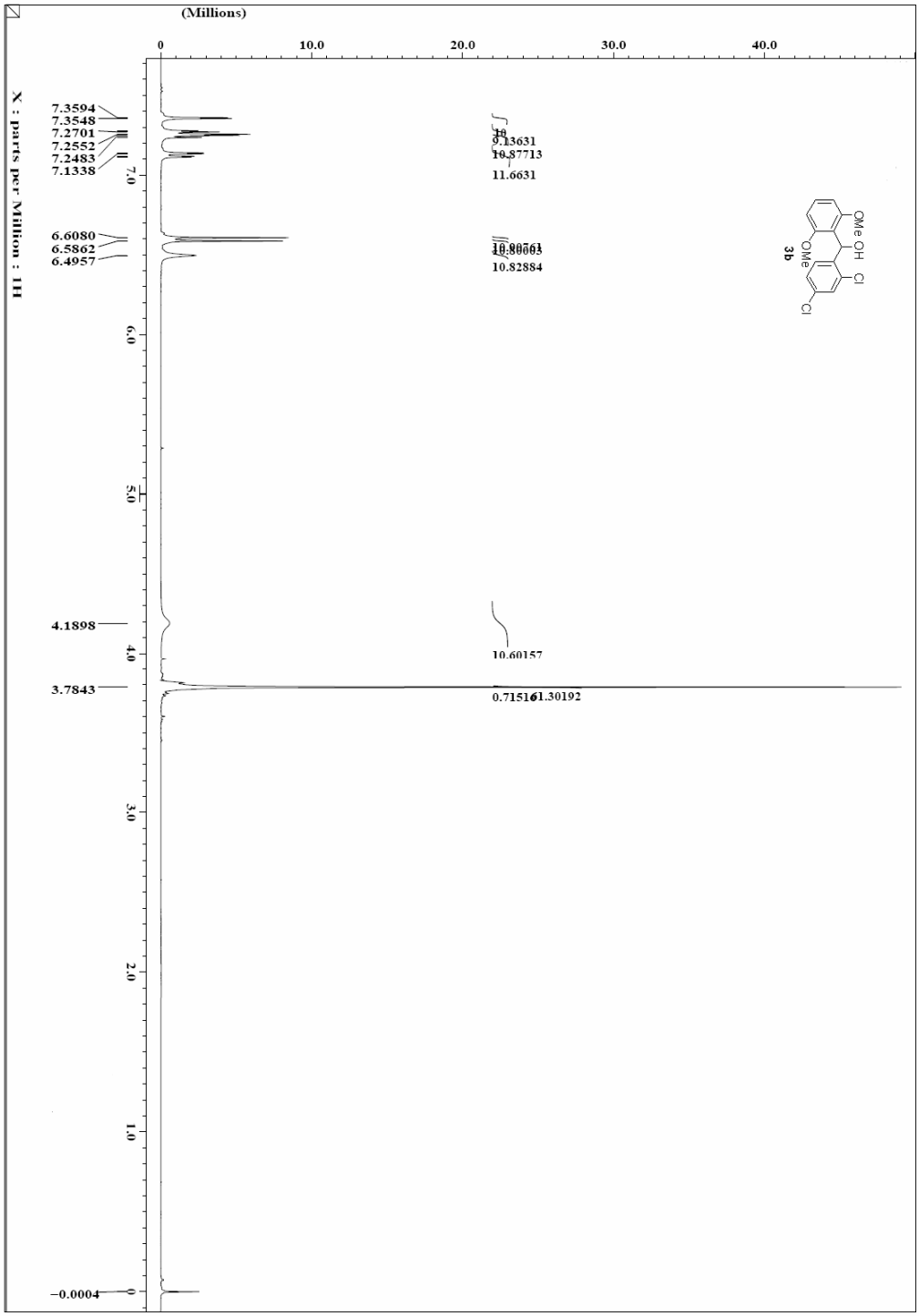
OMe
NO2
3a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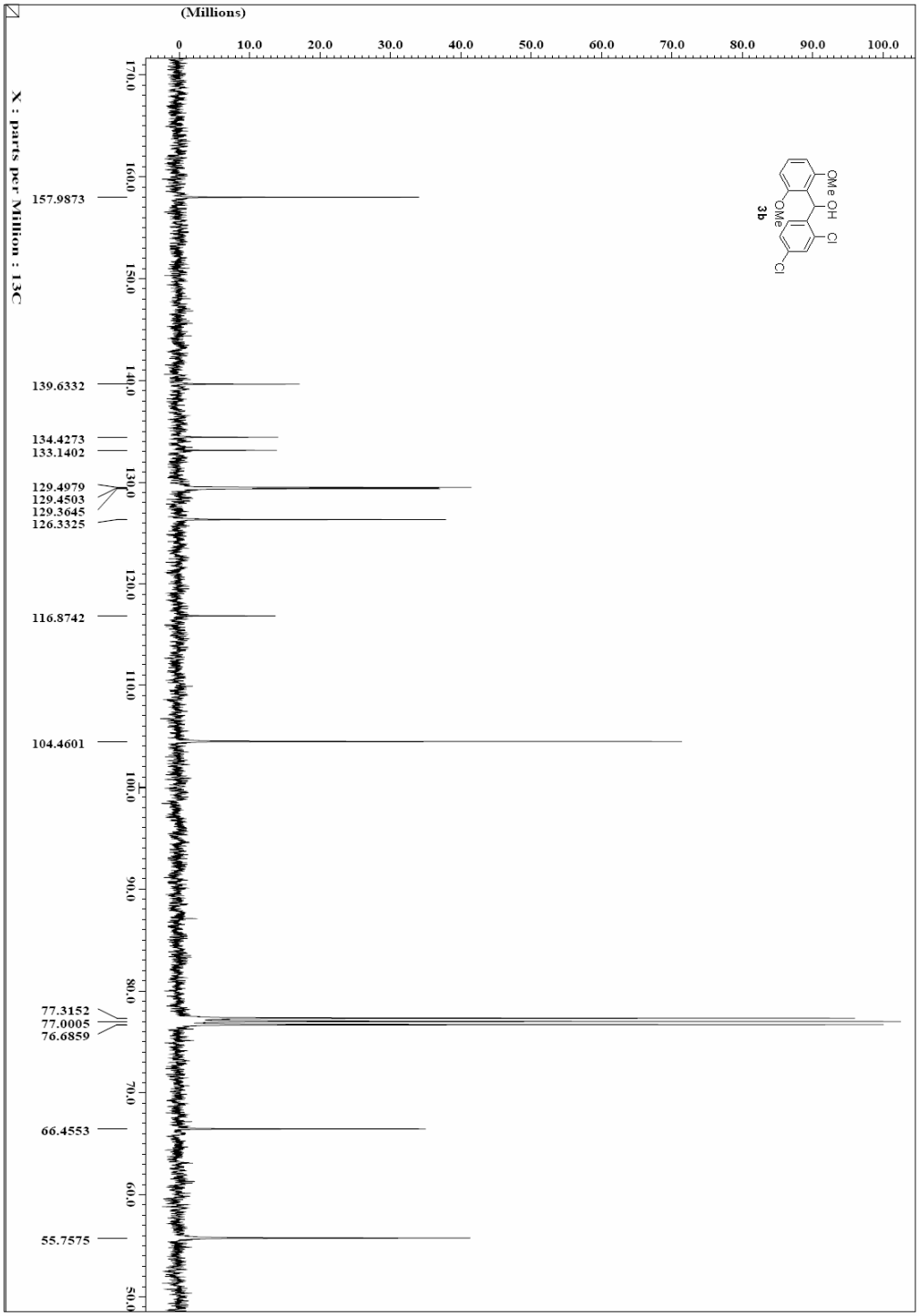
Cl
OMe
3b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Cl
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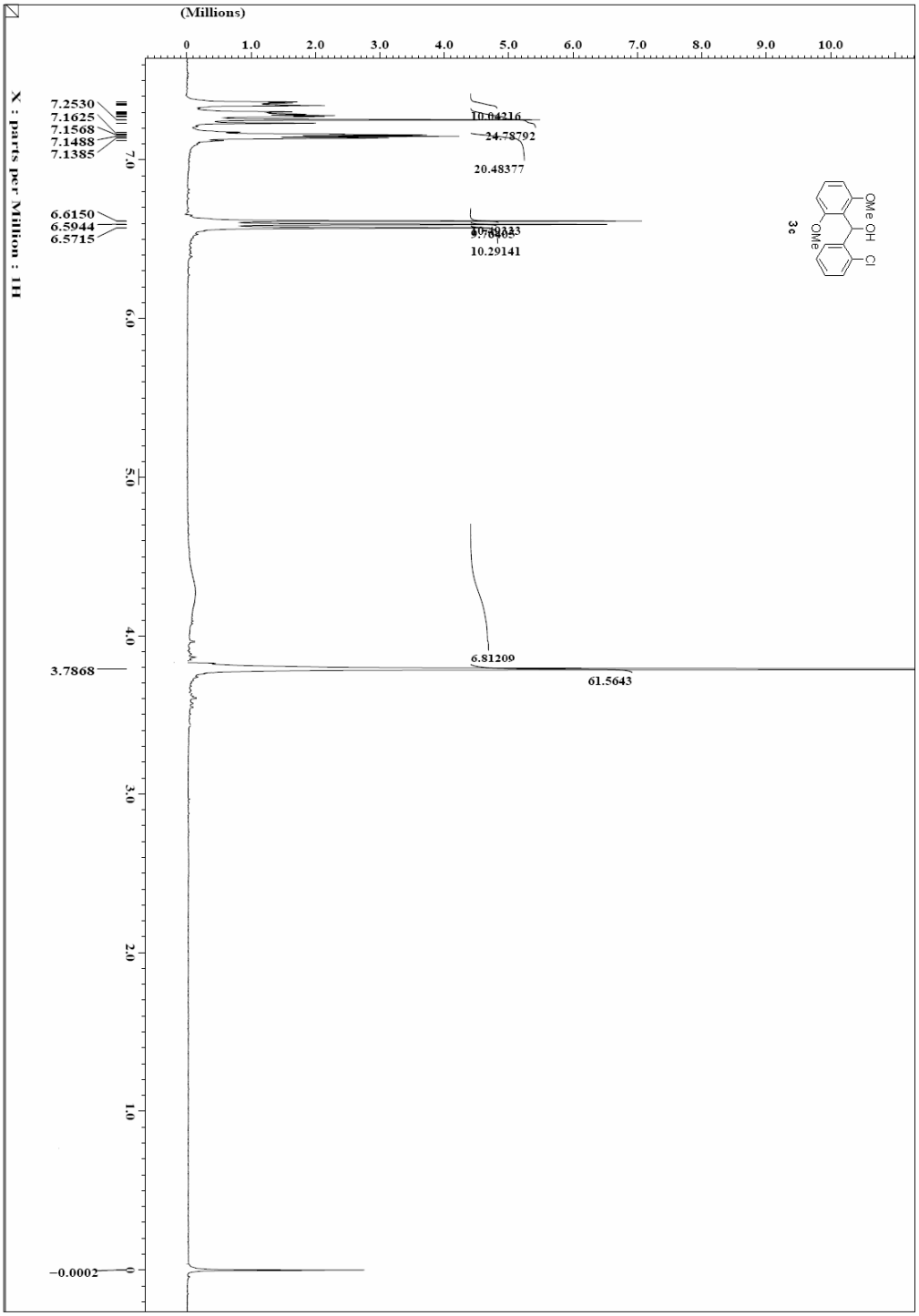
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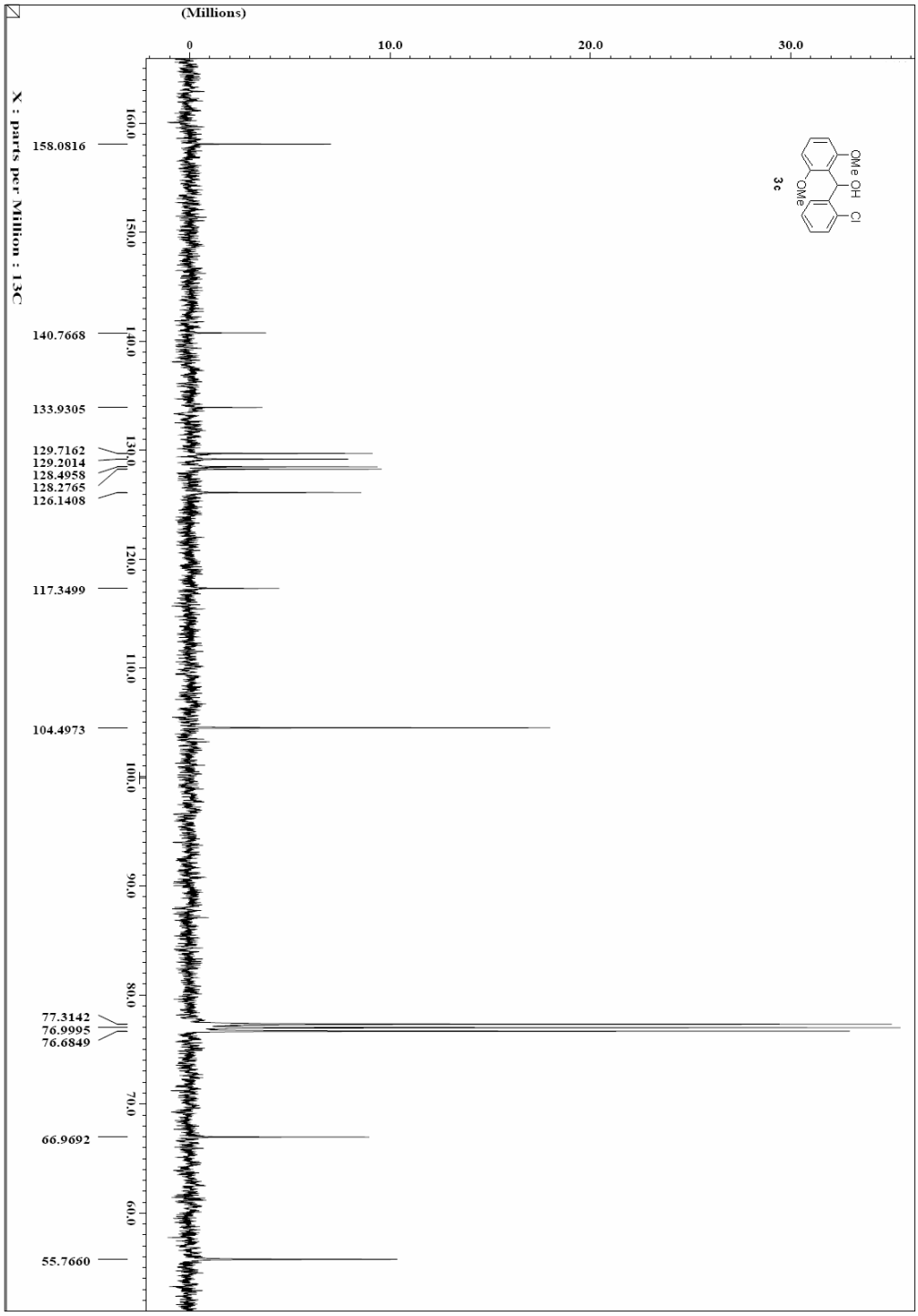
OMe
3c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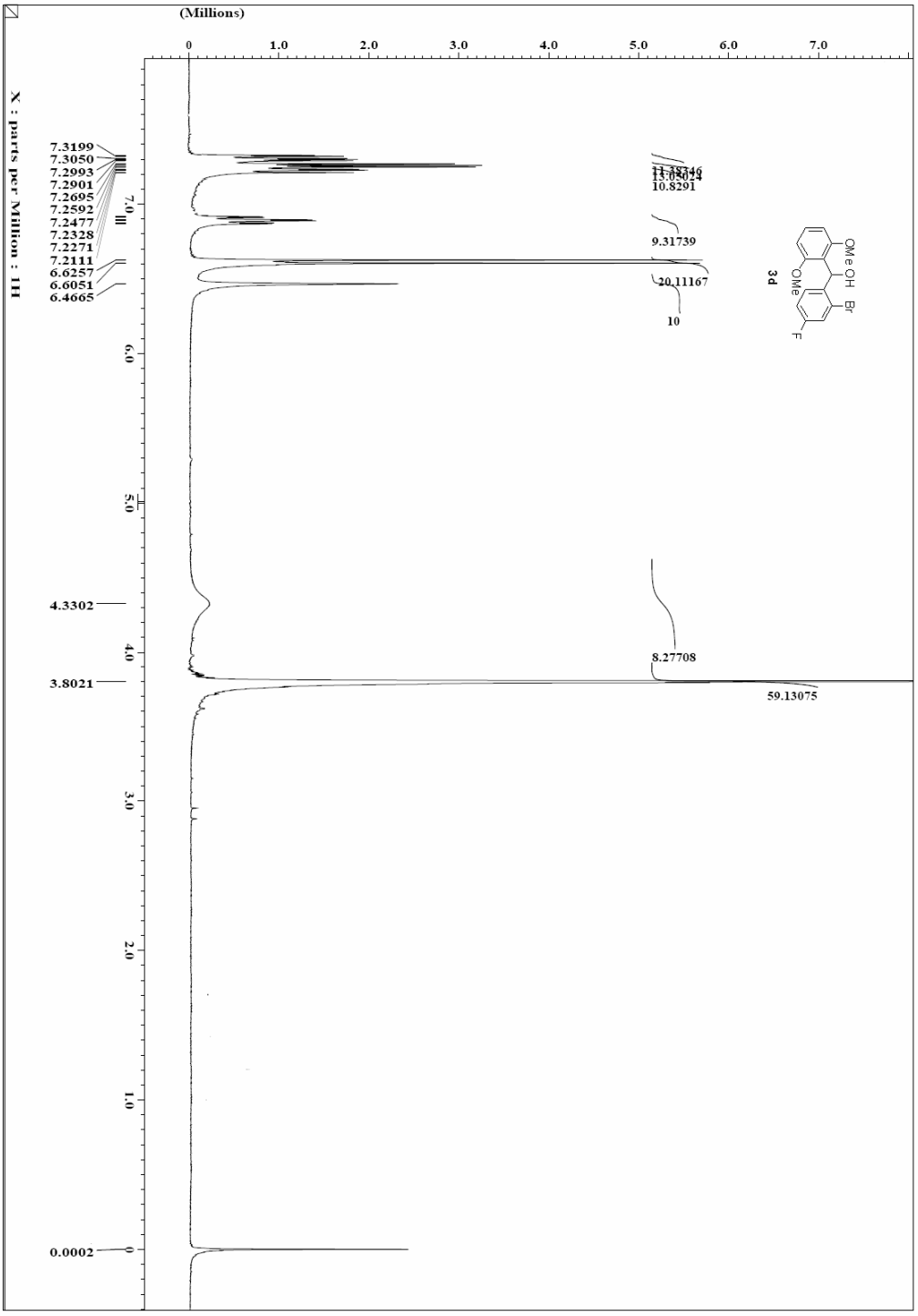
OH
Cl
OMe
3c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6.6051
6.4665
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10
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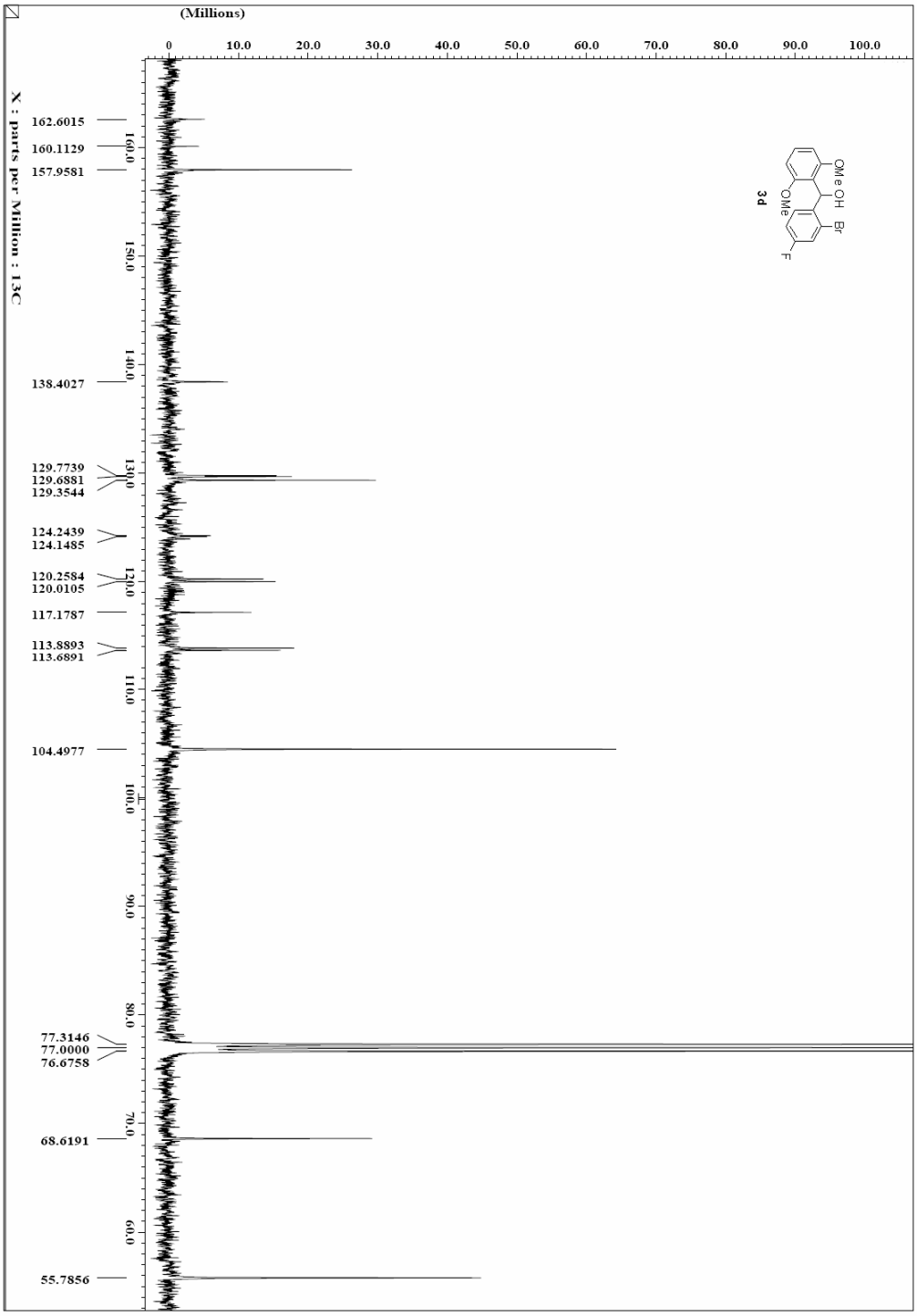
OH
Br
OMe
F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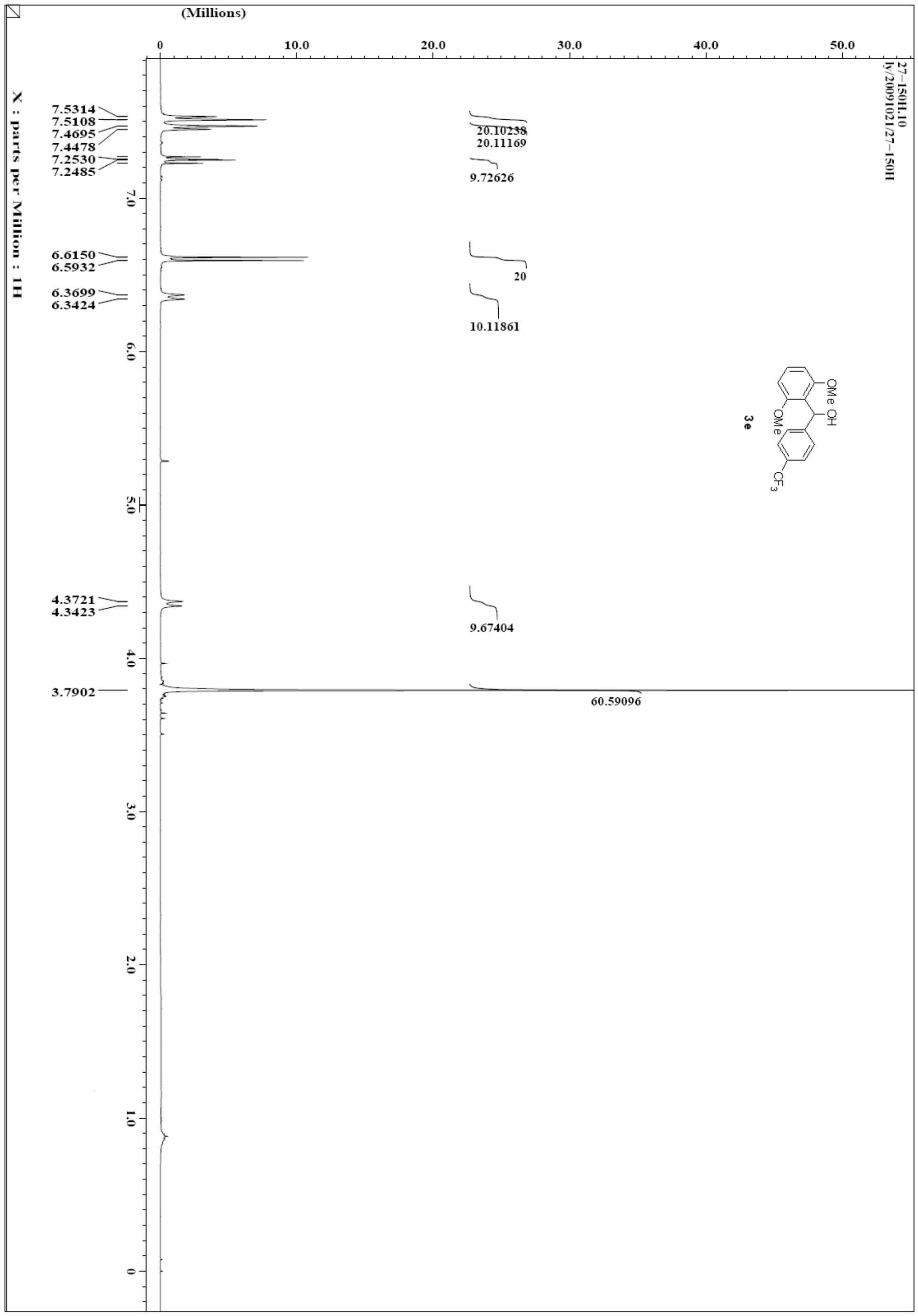
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7.2485
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6.5932
6.3699
6.3424
4.3721
4.3423
3.7902
7.0
6.0
5.0
4.0
3.0
2.0
1.0
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20
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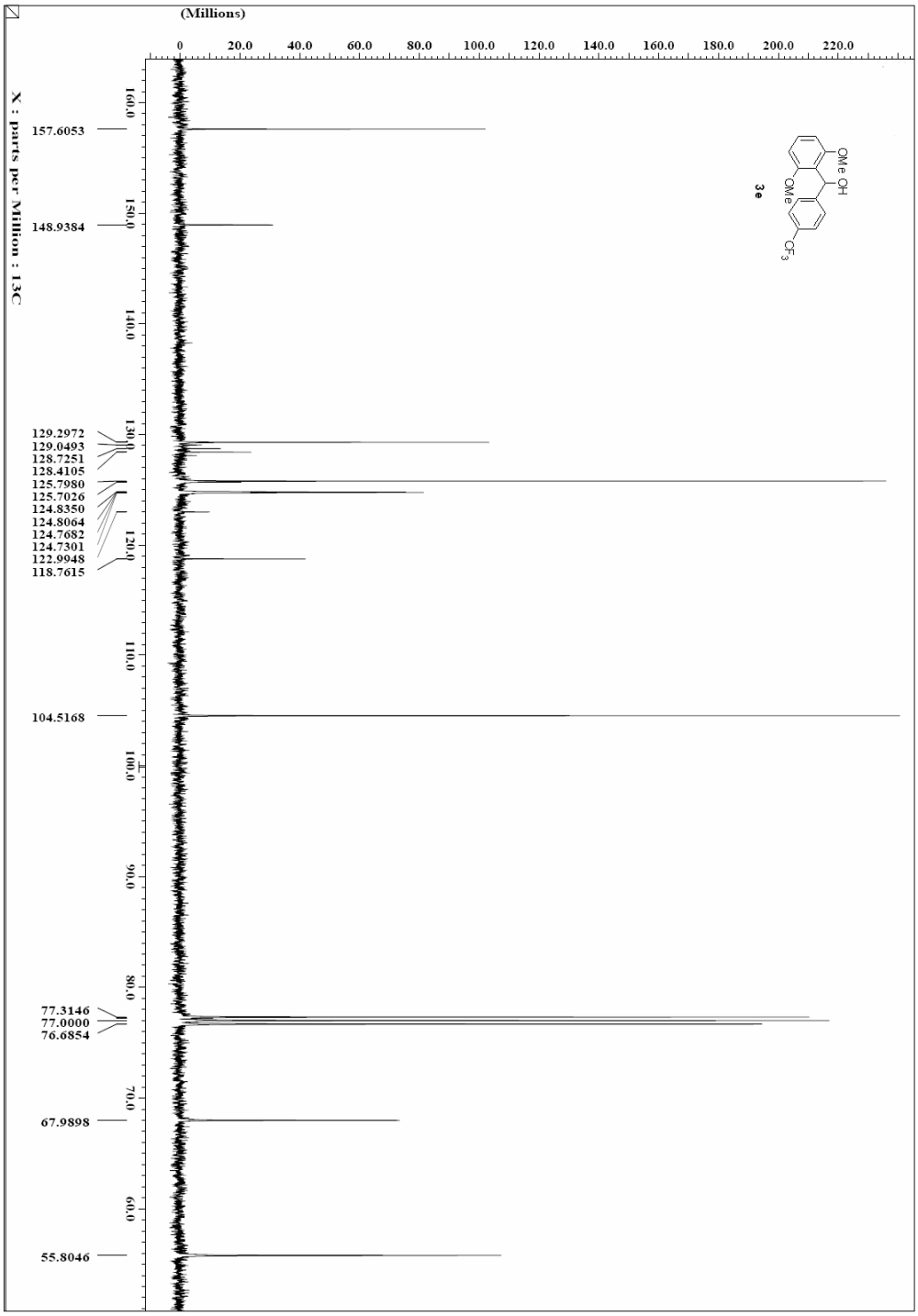
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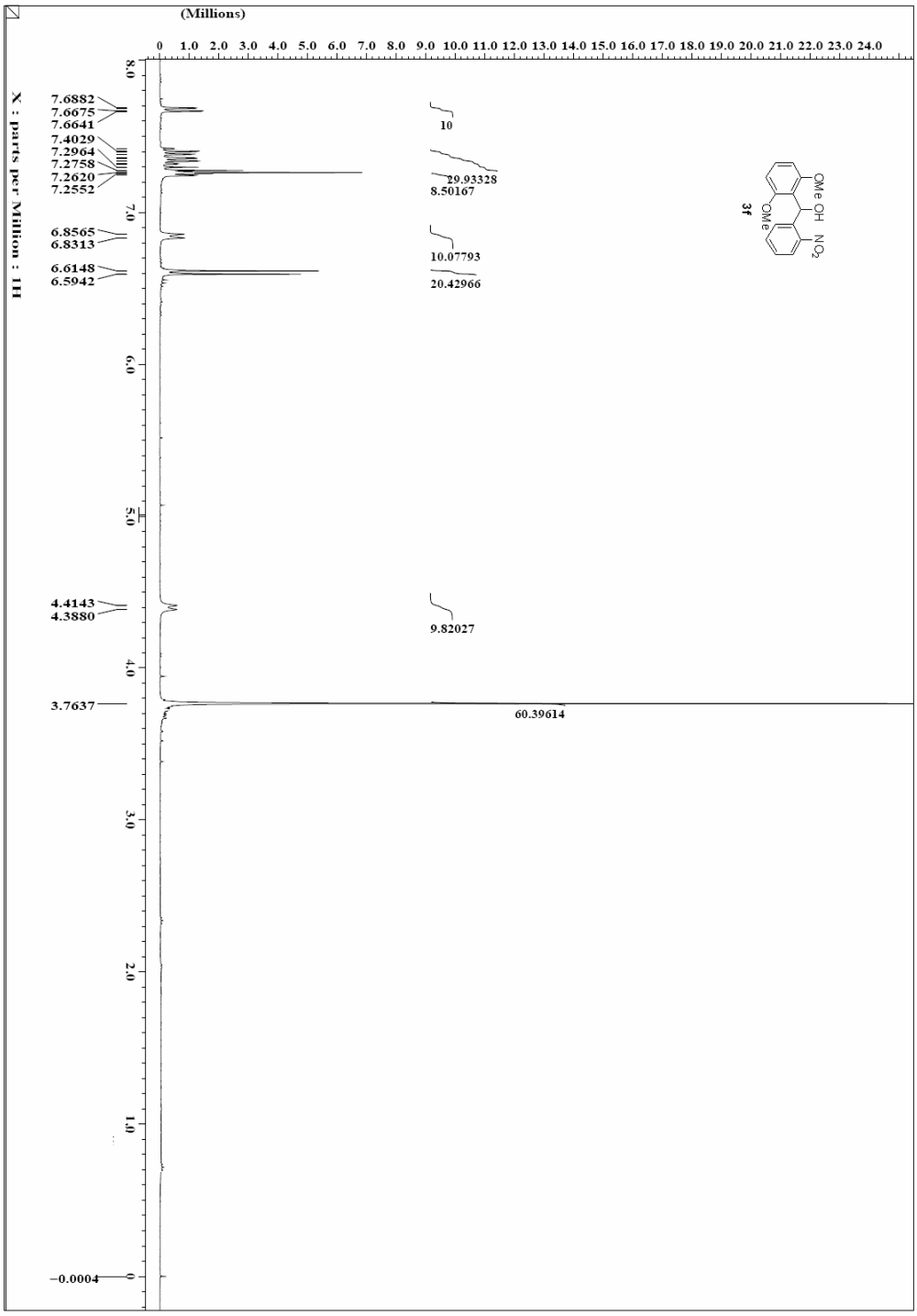
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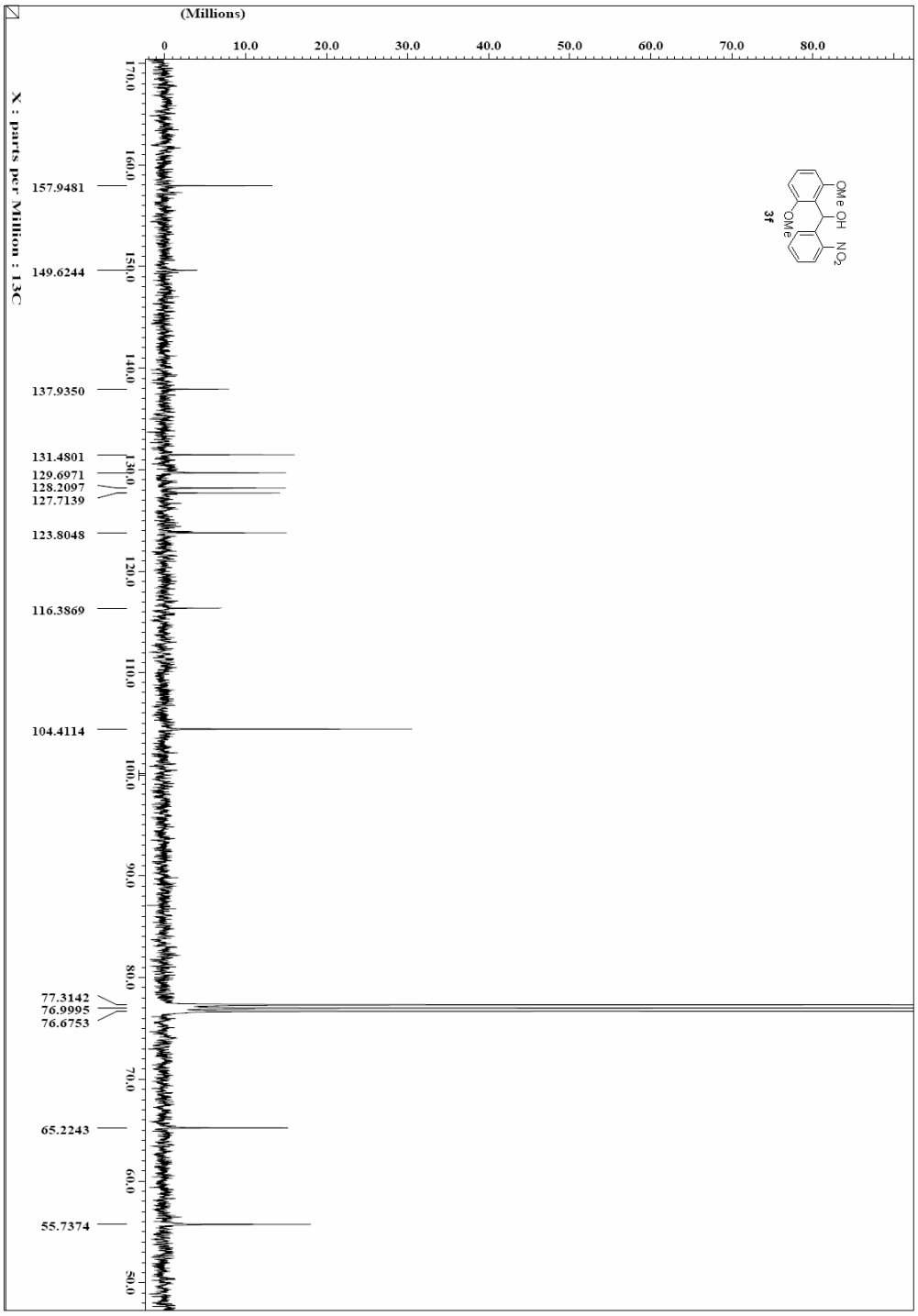
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OMe
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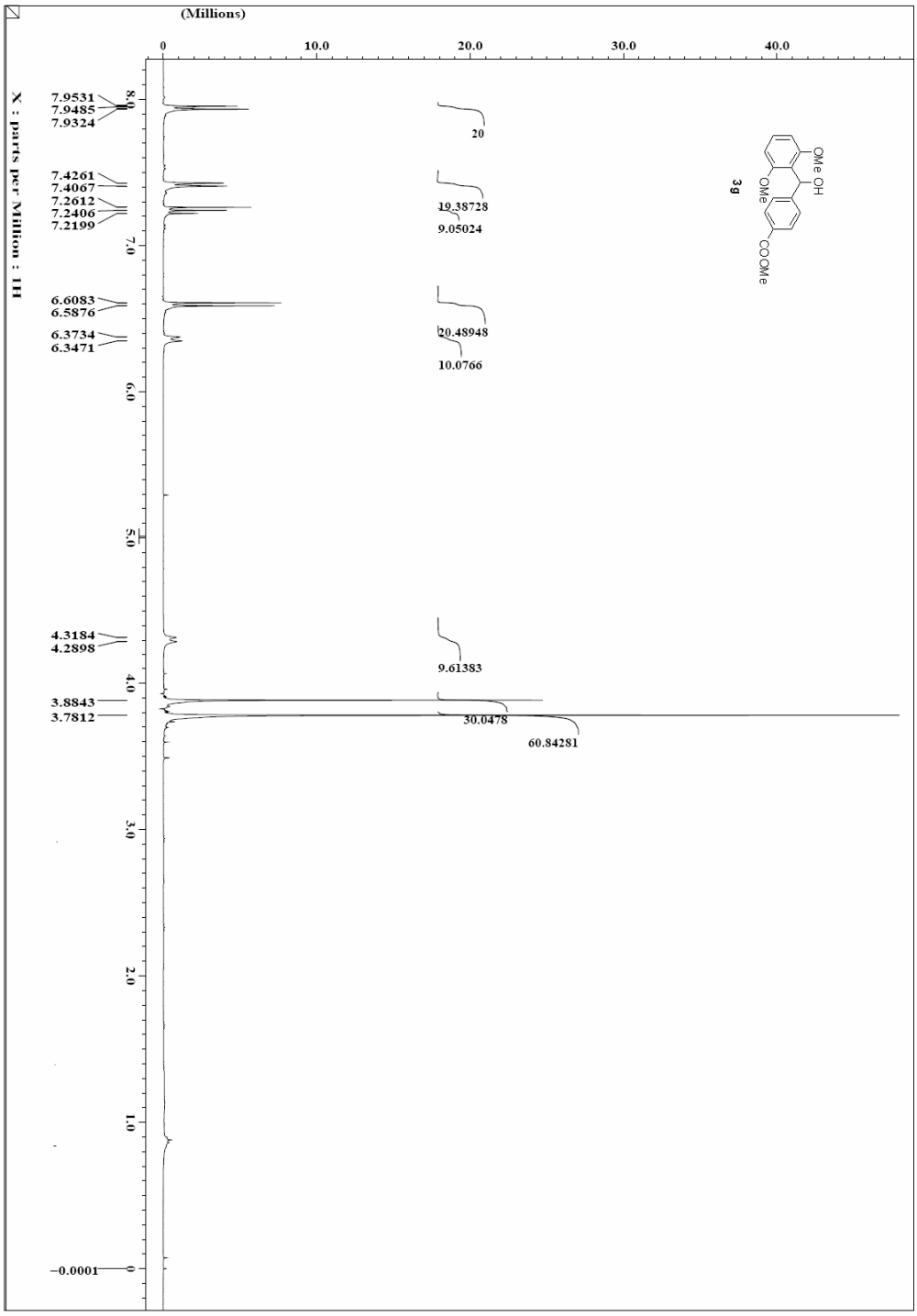
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OMe
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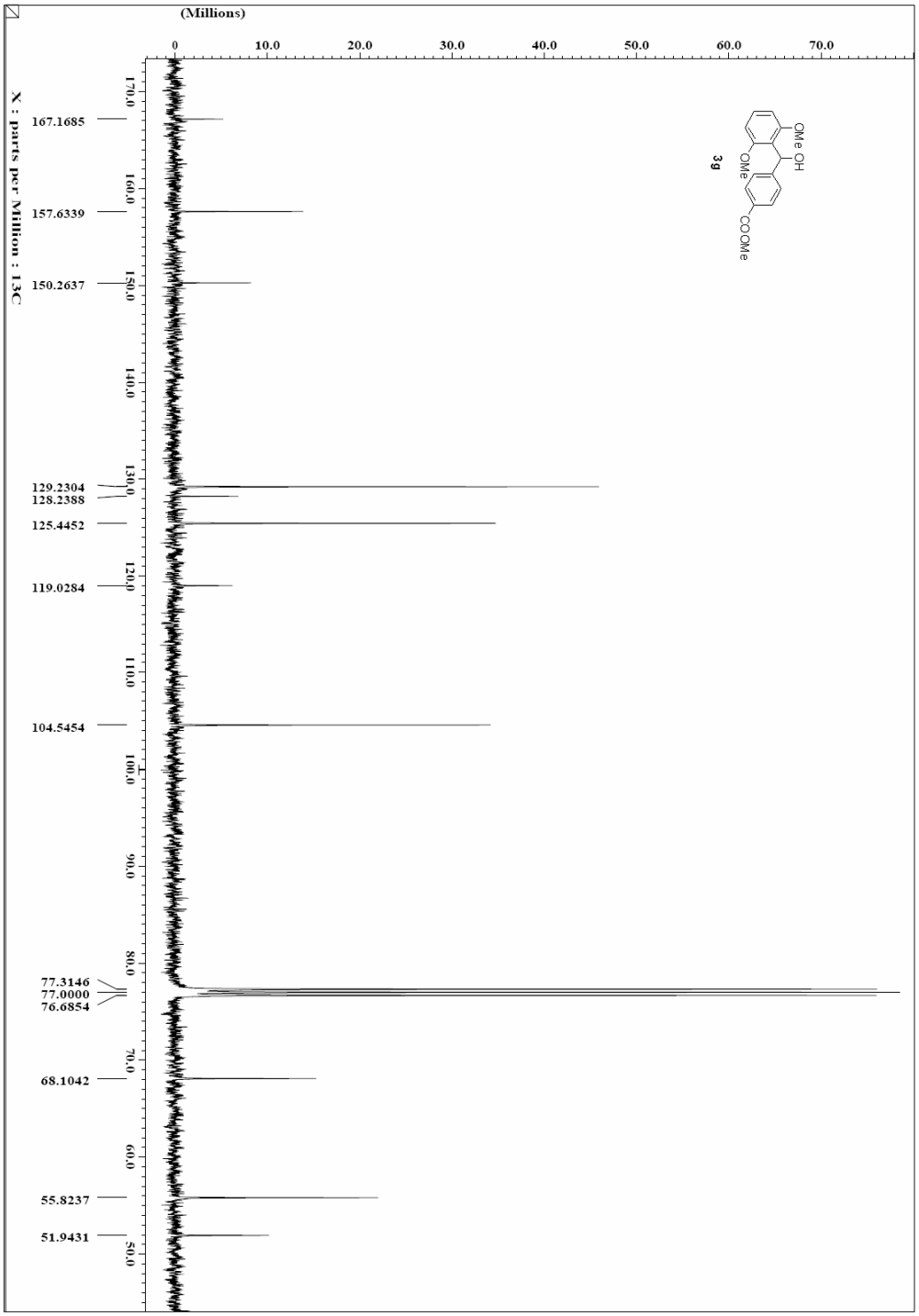
3g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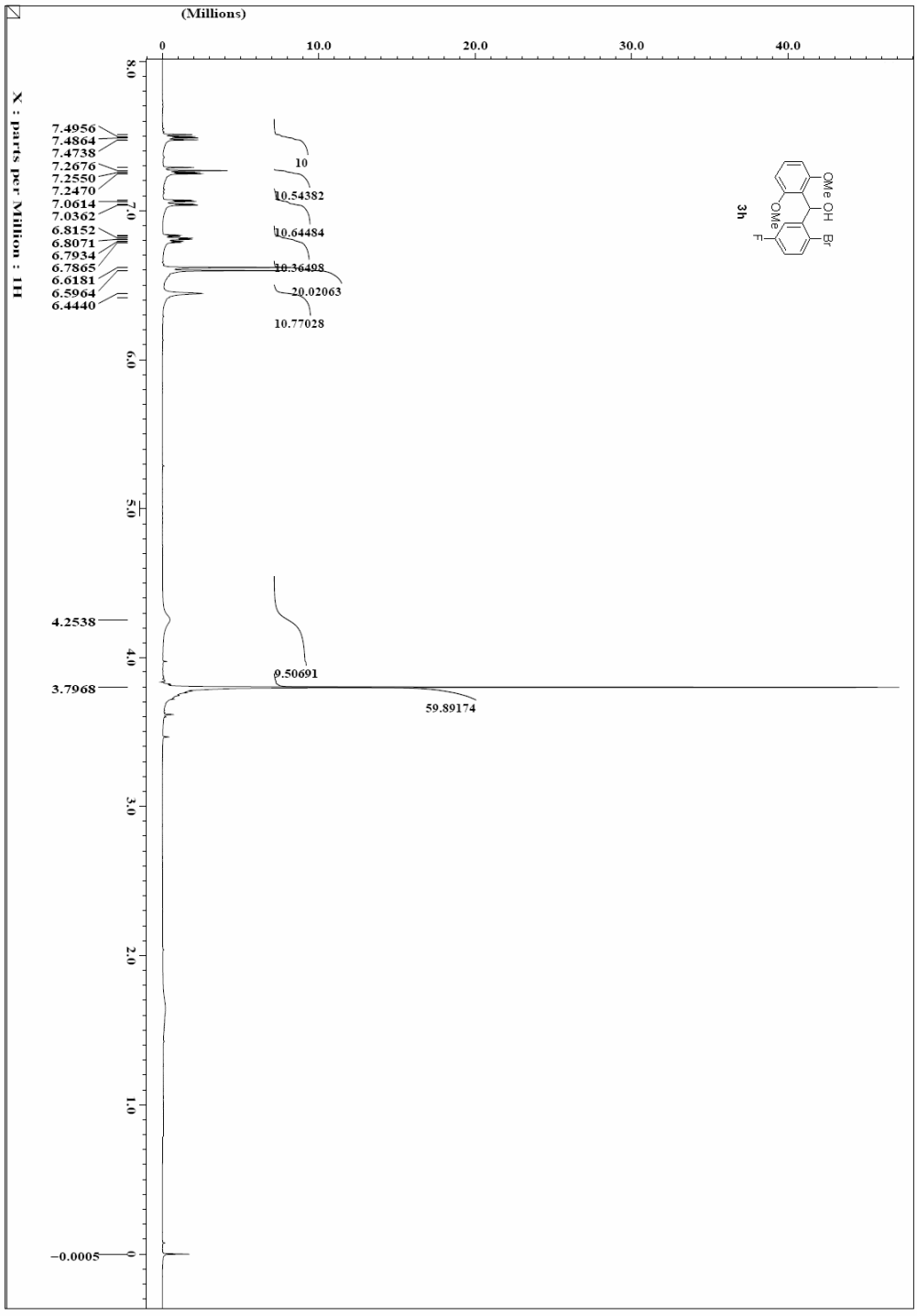
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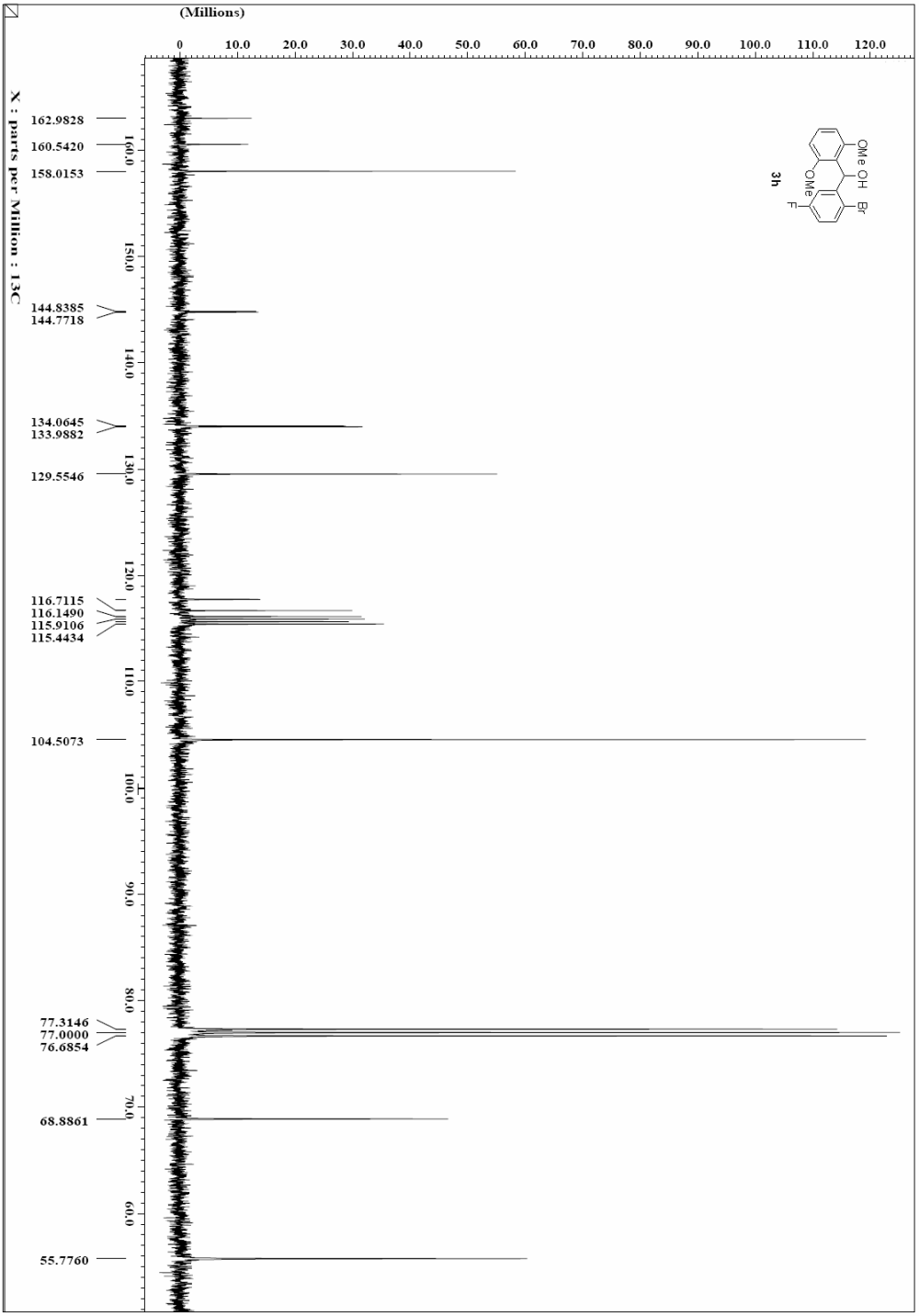
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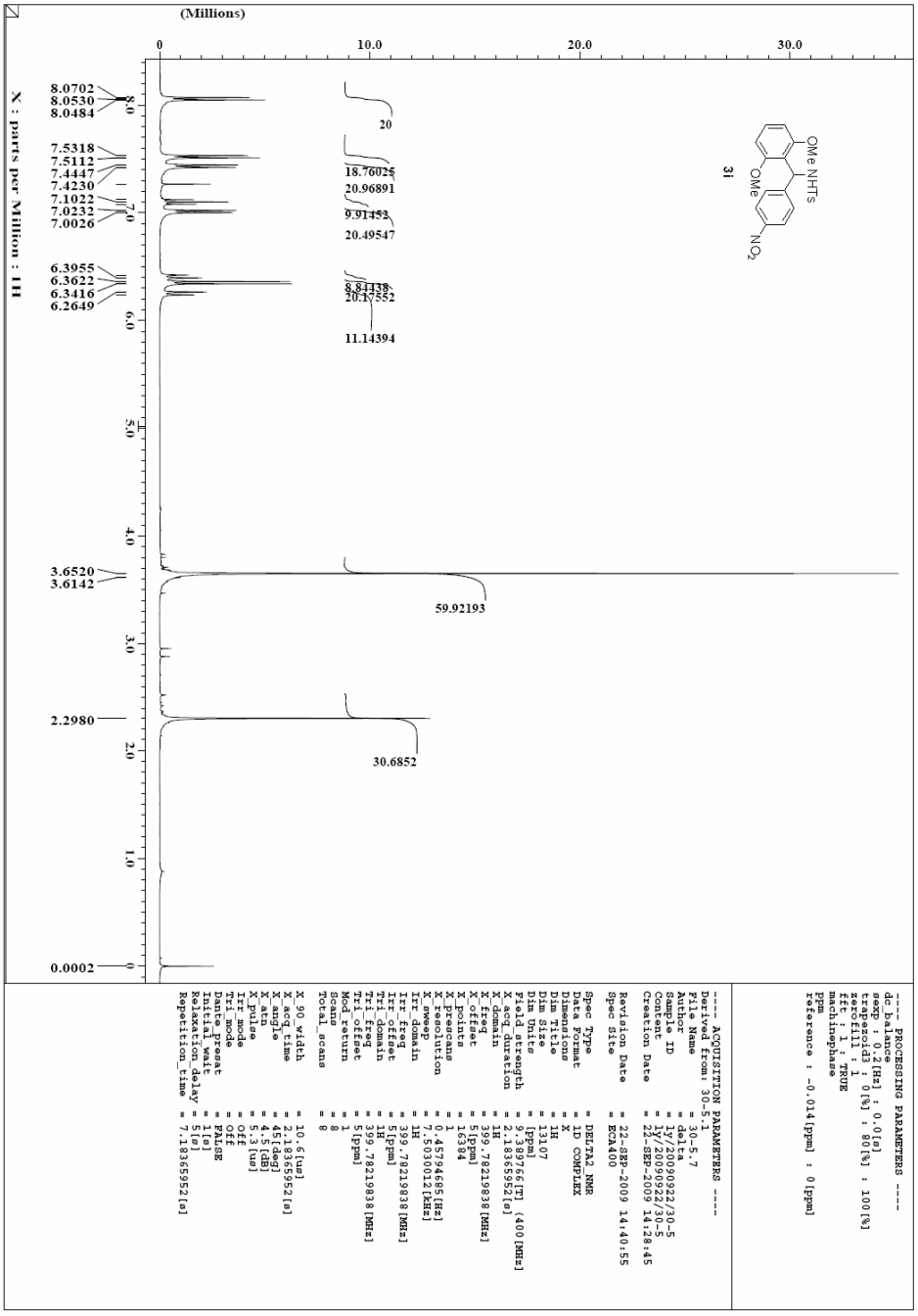
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6.3416
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20
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OMe NHTs
OMe
NO2
3l
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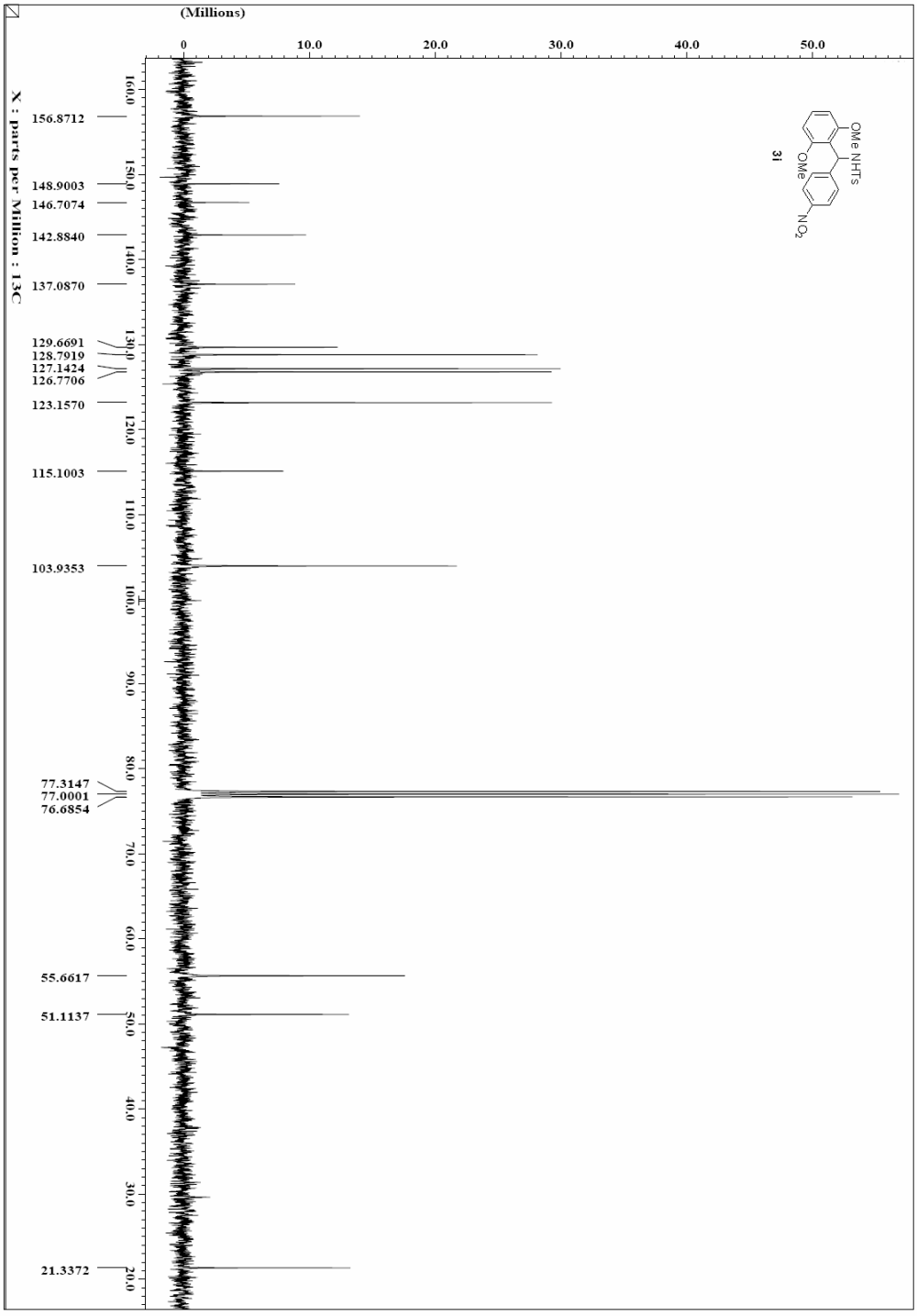
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NHTs
OMe
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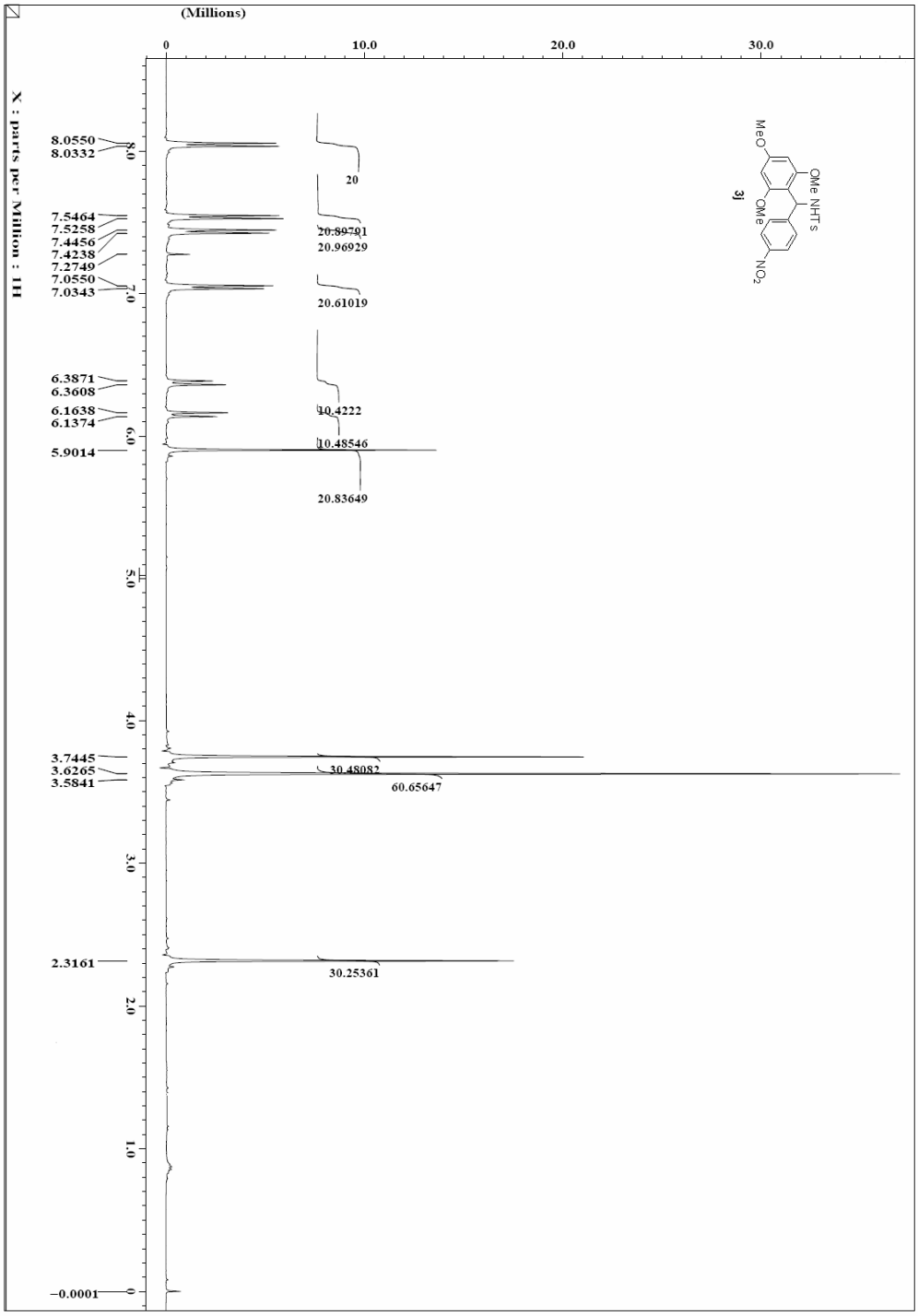
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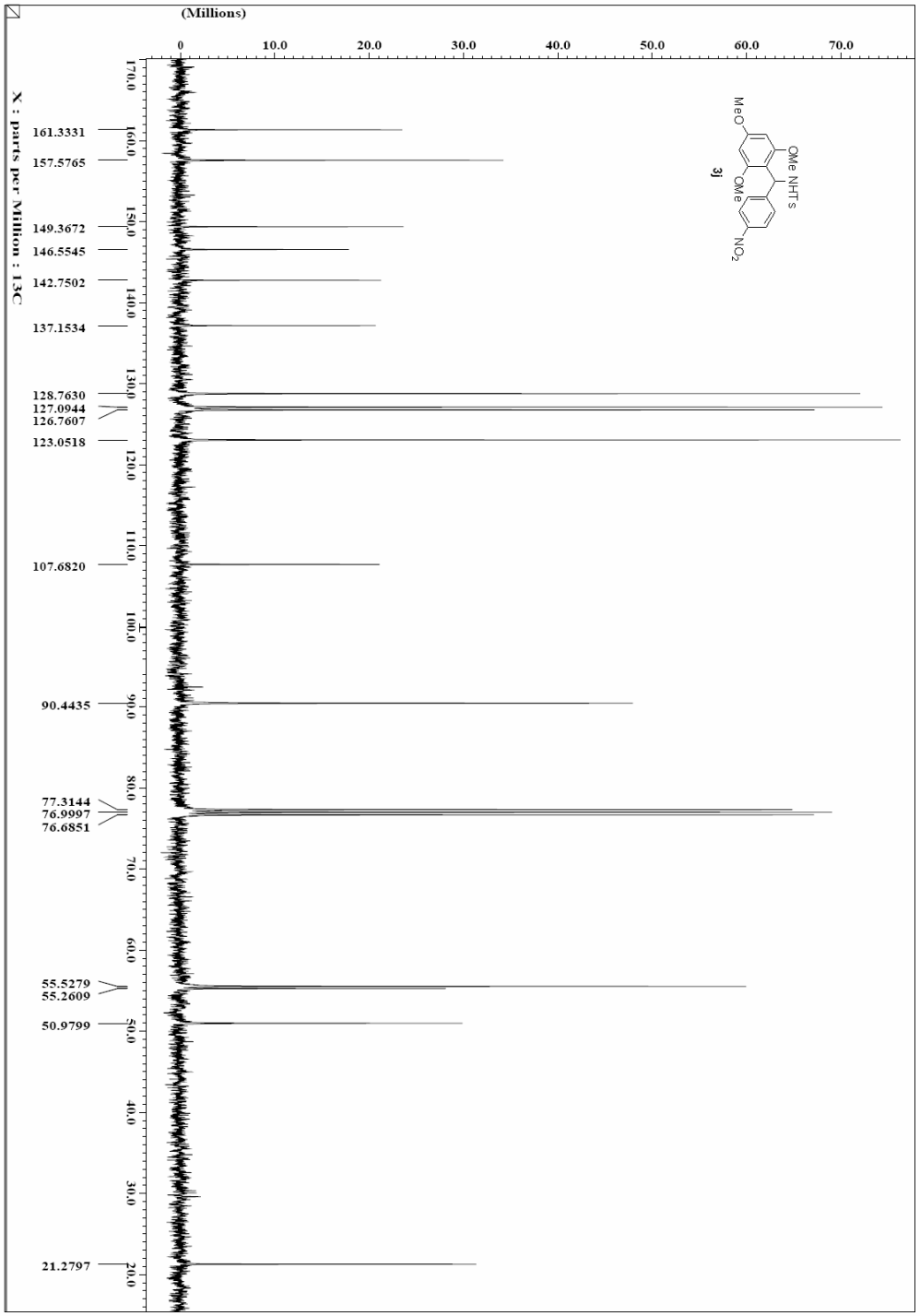
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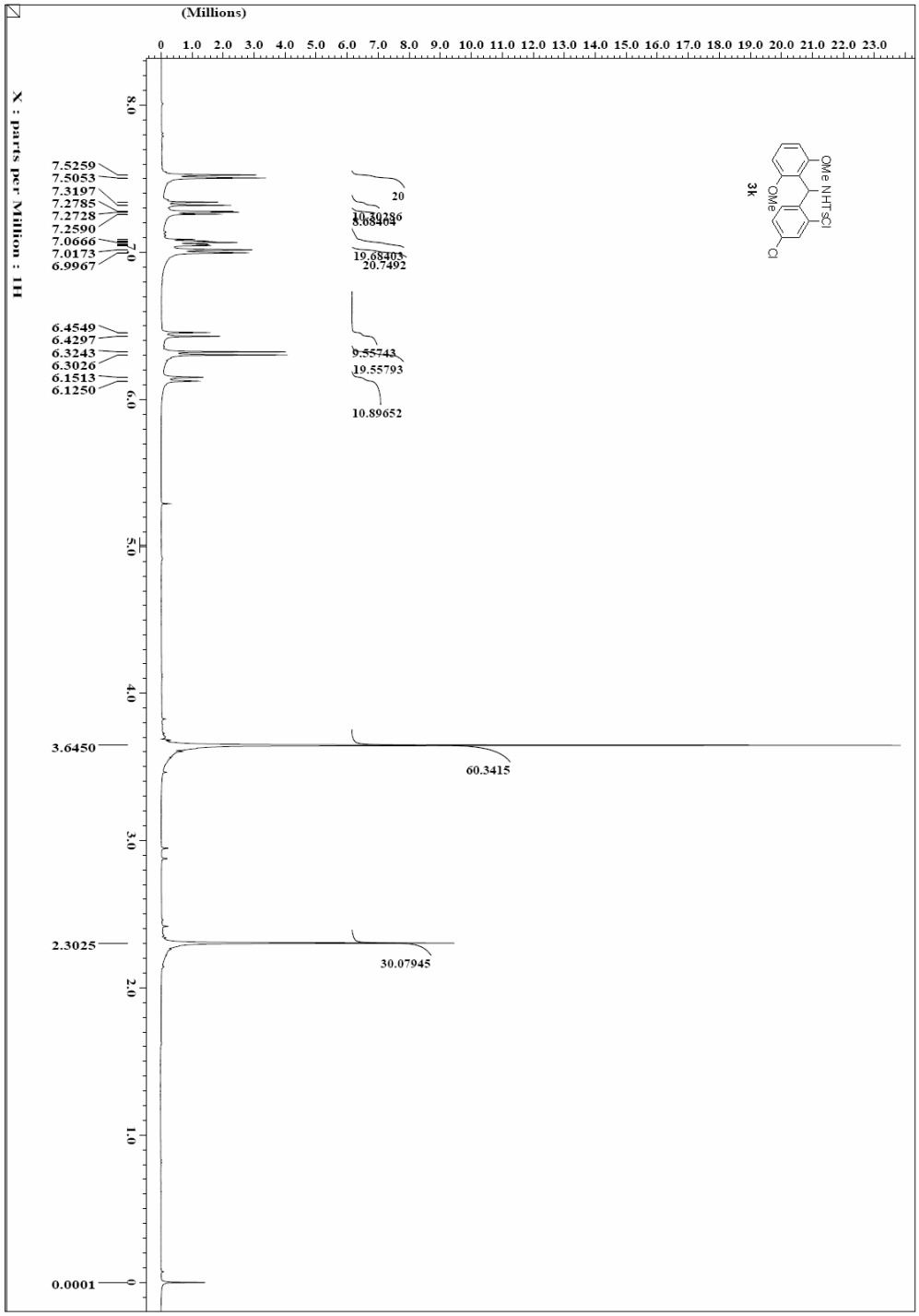
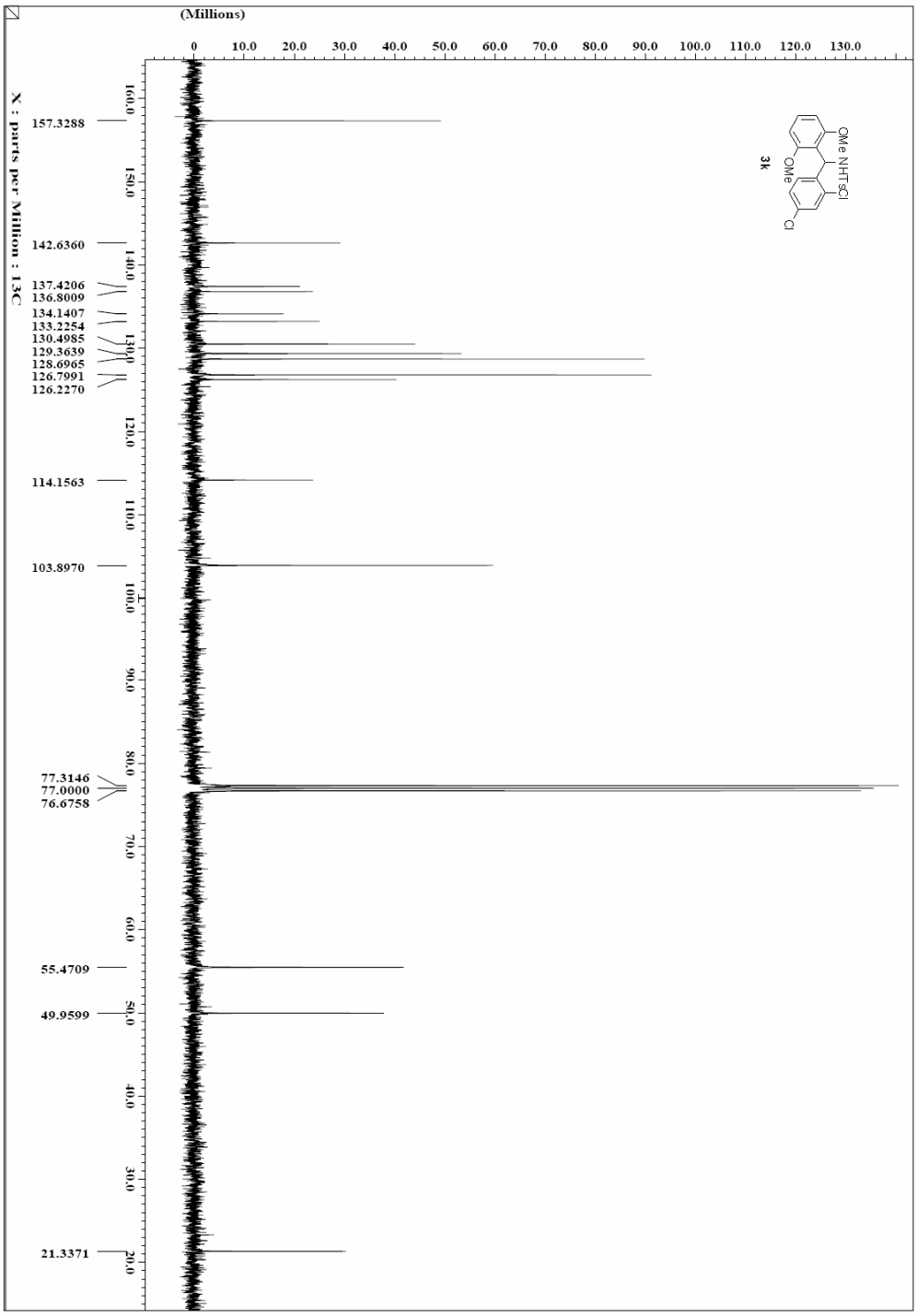
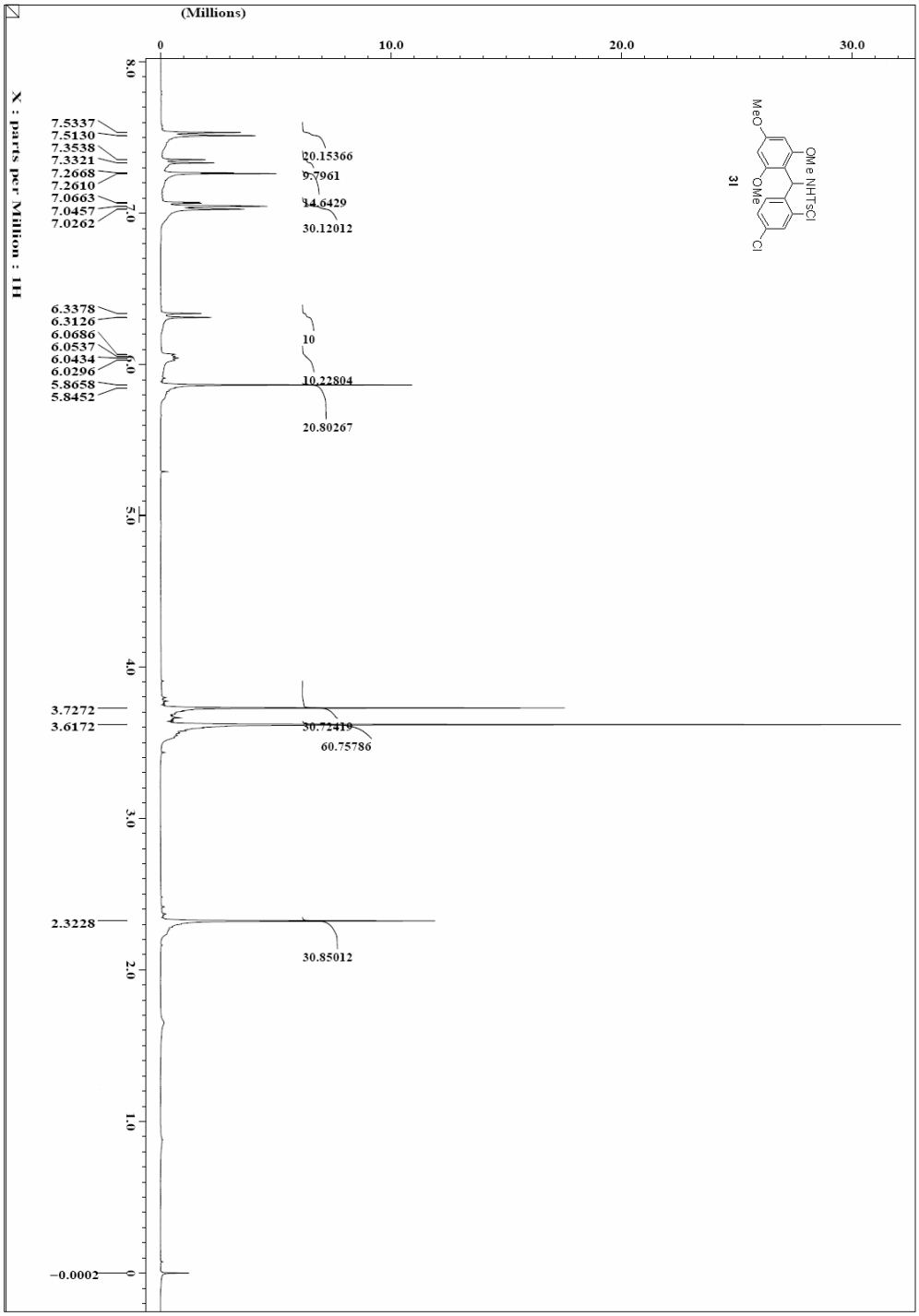
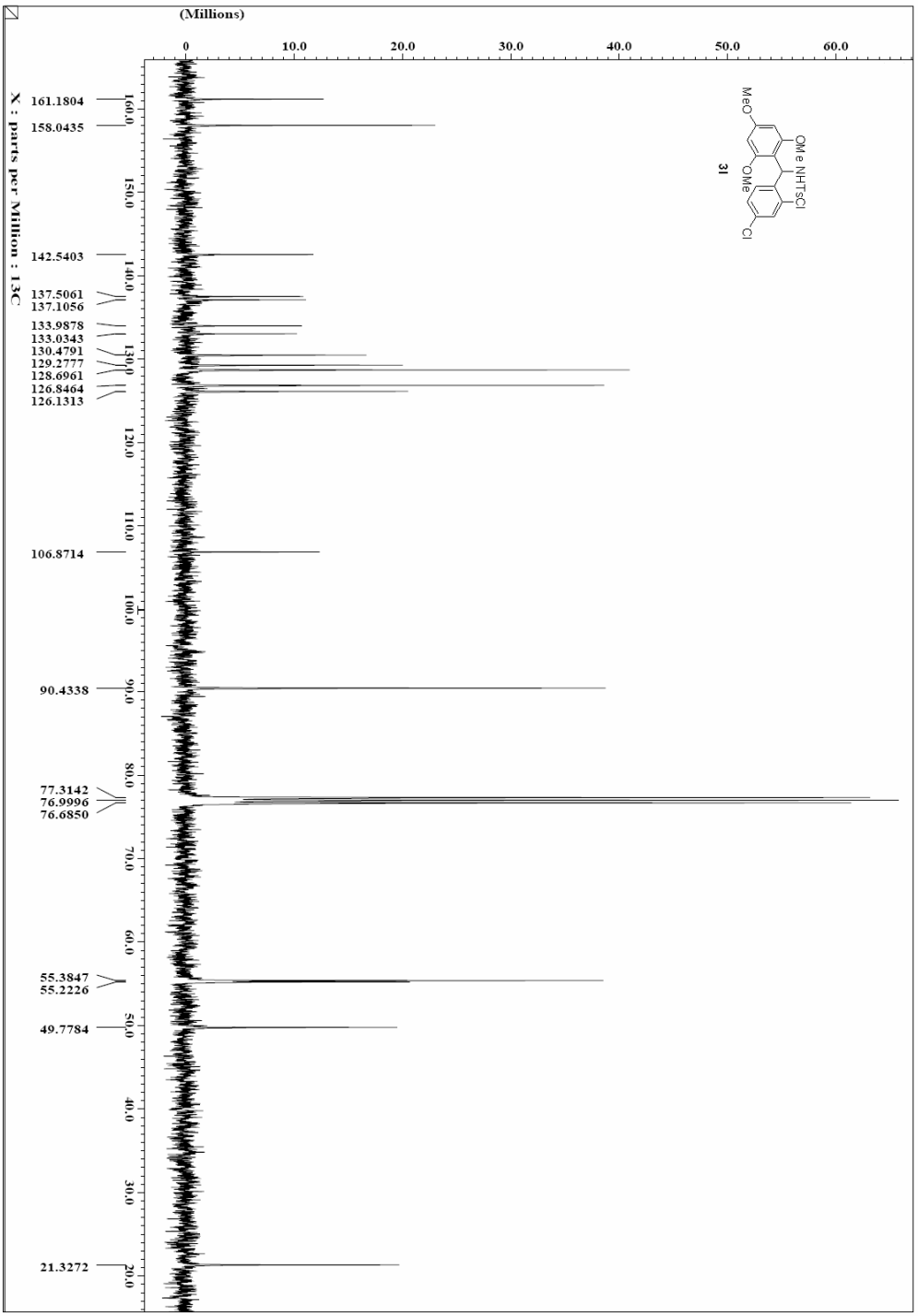
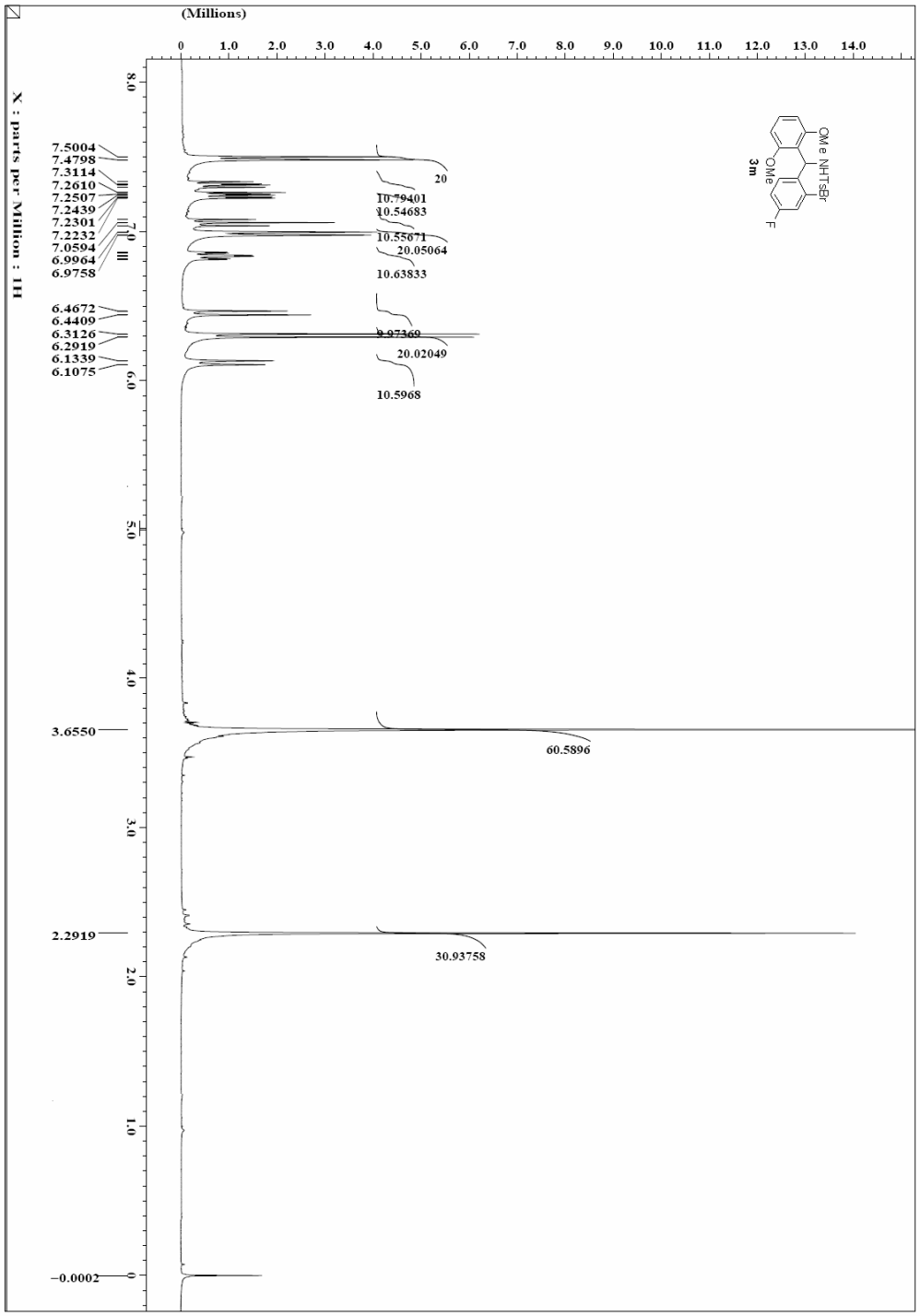
142.7502
137.1534
128.7630
127.0944
126.7607
123.0518
107.6820
90.4435
77.3144
76.9997
76.6851
55.5279
55.2609
50.9799
21.2797
MeO
OMe
NHTs
NO2
3j

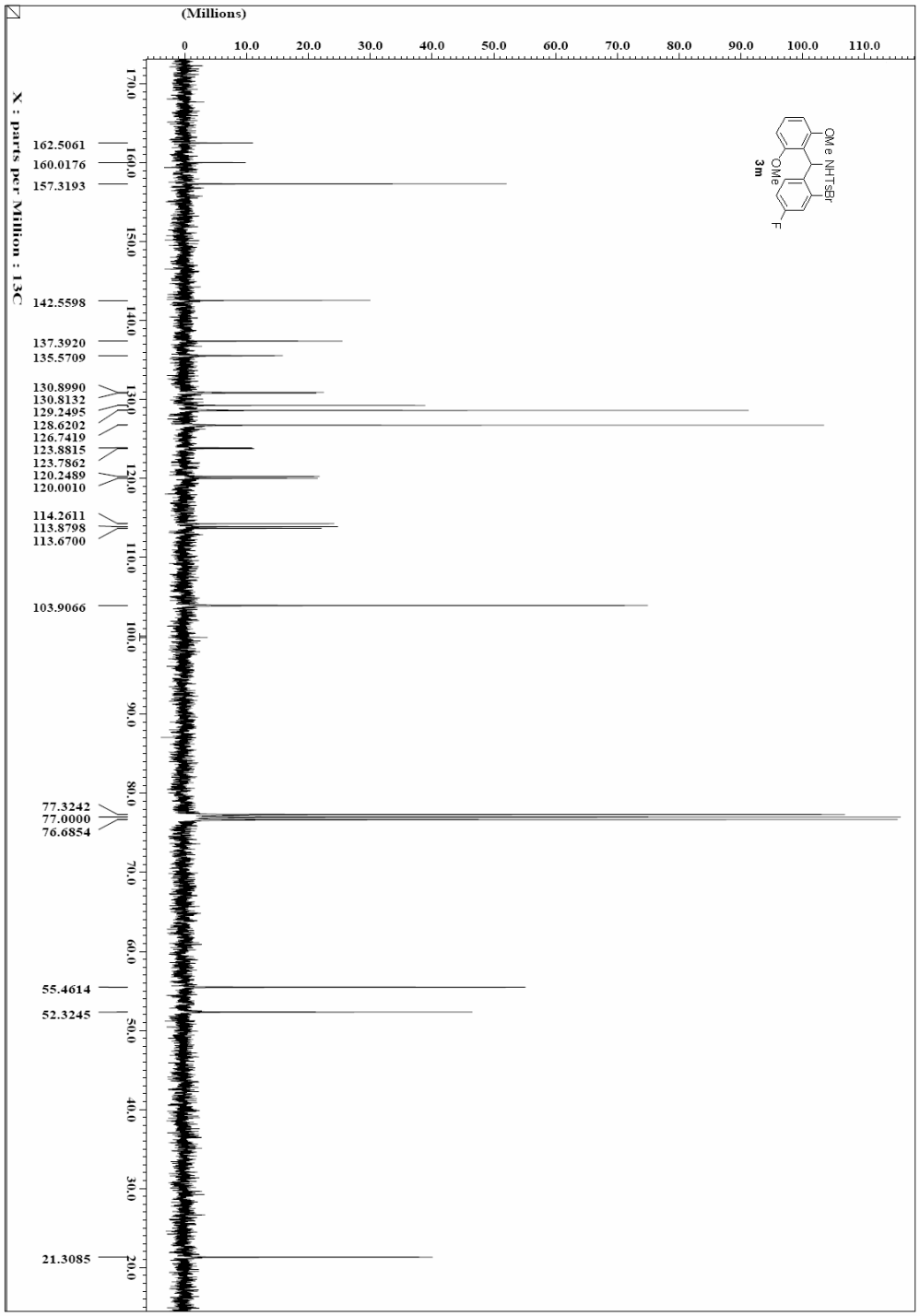

(Millions)
X : parts per Million : 1H
7.5259
7.5053
7.3197
7.2785
7.2728
7.2590
7.0666
7.0173
6.9967
6.4549
6.4297
6.3243
6.3026
6.1513
6.1250
3.6450
2.3025
0.0001
20
19.68403
20.7492
9.55743
19.55793
10.89652
60.3415
30.07945
3k
OMe
NHTsCl
OMe
Cl

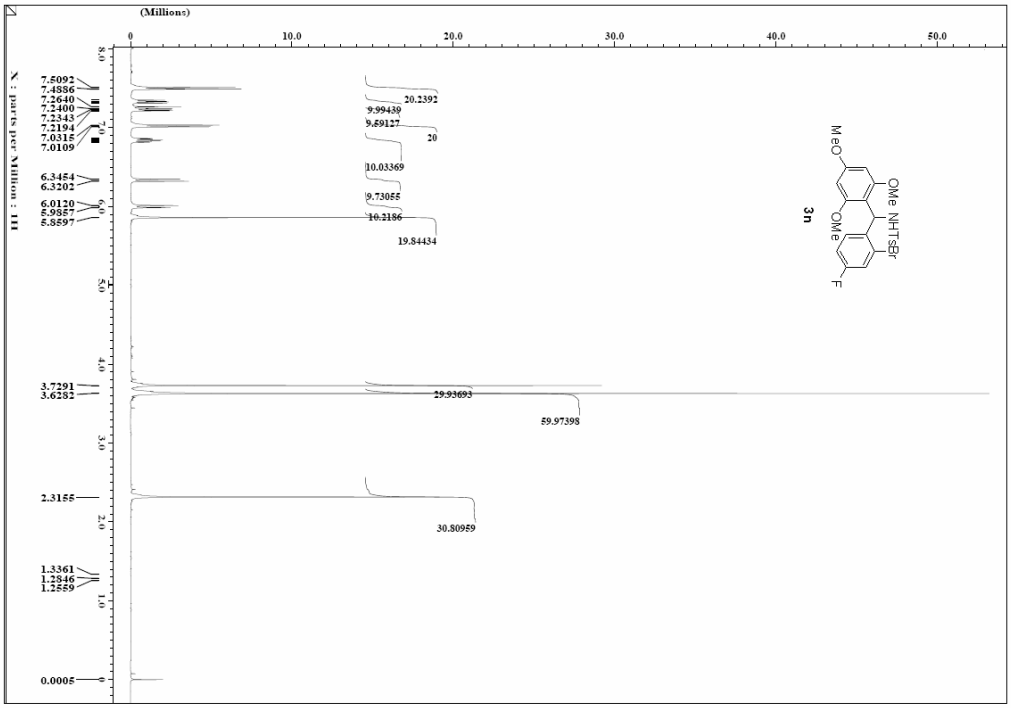

(Millions)
X : parts per Million : 13C
157.3288
142.6360
137.4206
136.8009
134.1407
133.2254
130.4985
129.3639
128.6965
126.7991
126.2270
114.1563
103.8970
77.3146
77.0000
76.6758
55.4709
49.9599
21.3371
3k
OMe
NHTsCl
Cl

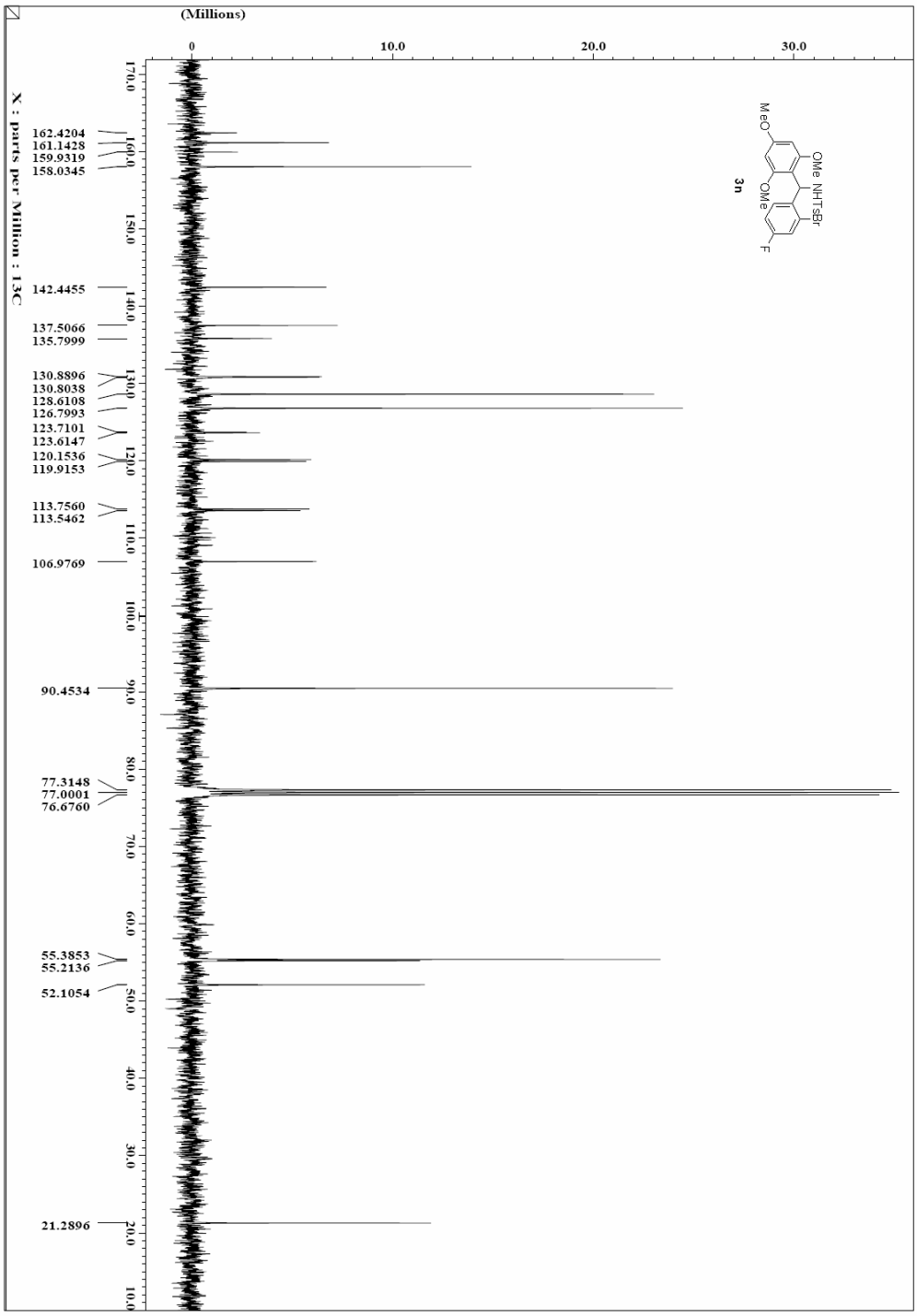

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 1H
7.5337
7.5130
7.3538
7.3321
7.2668
7.2610
7.0663
7.0457
7.0262
6.3378
6.3126
6.0686
6.0537
6.0434
6.0296
5.8658
5.8452
3.7272
3.6172
2.3228
−0.0002
20.15366
9.7961
14.6429
30.12012
10
10.22804
20.80267
30.72419
60.75786
30.85012
MeO
OMe
NHTsCl
Cl
31

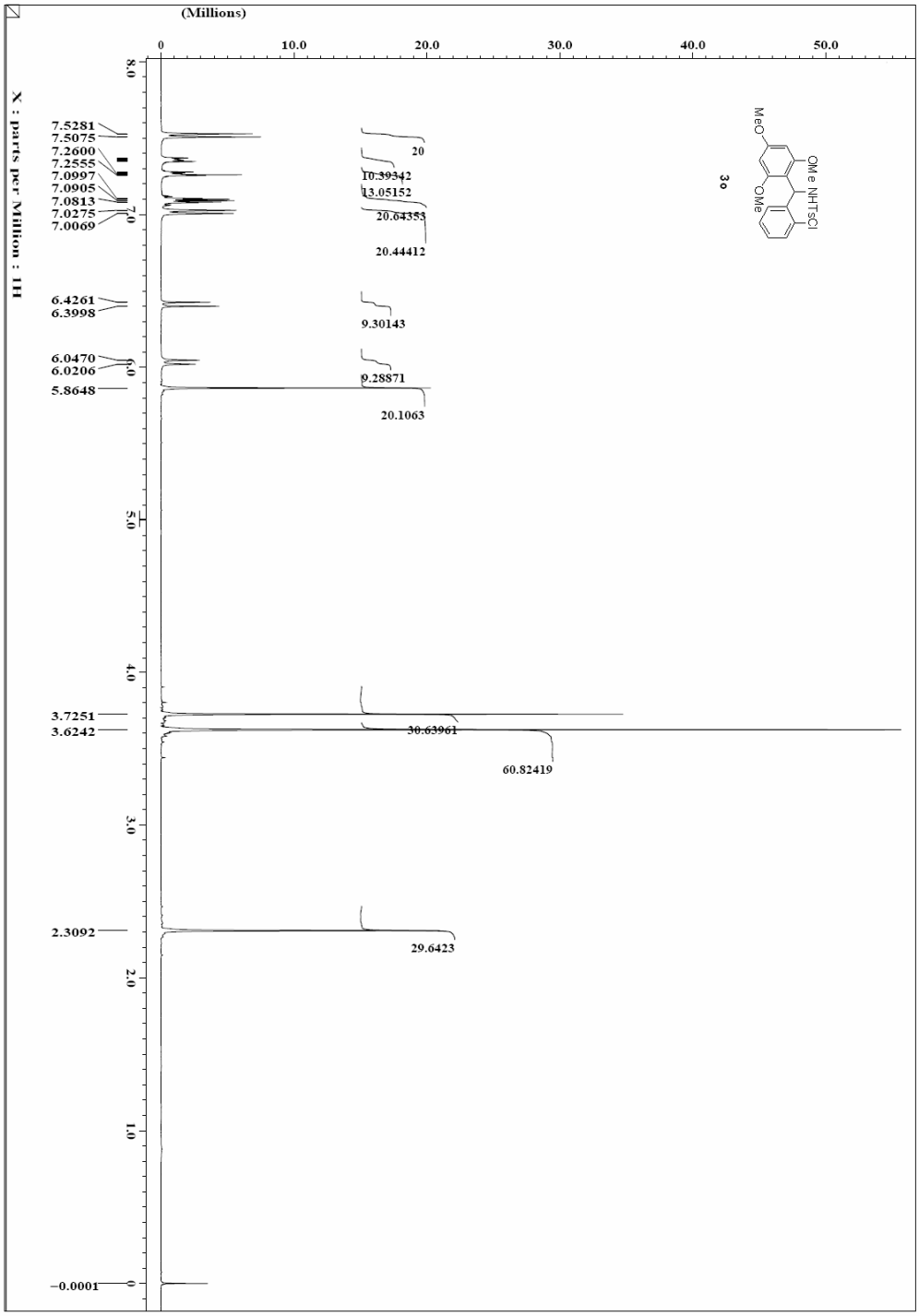

(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
X : parts per Million : 13C
161.1804
158.0435
142.5403
137.5061
137.1056
133.9878
133.0343
130.4791
129.2777
128.6961
126.8464
126.1313
106.8714
90.4338
77.3142
76.9996
76.6850
55.3847
55.2226
49.7784
21.3272
MeO
OMe
NHTsCl
Cl
31

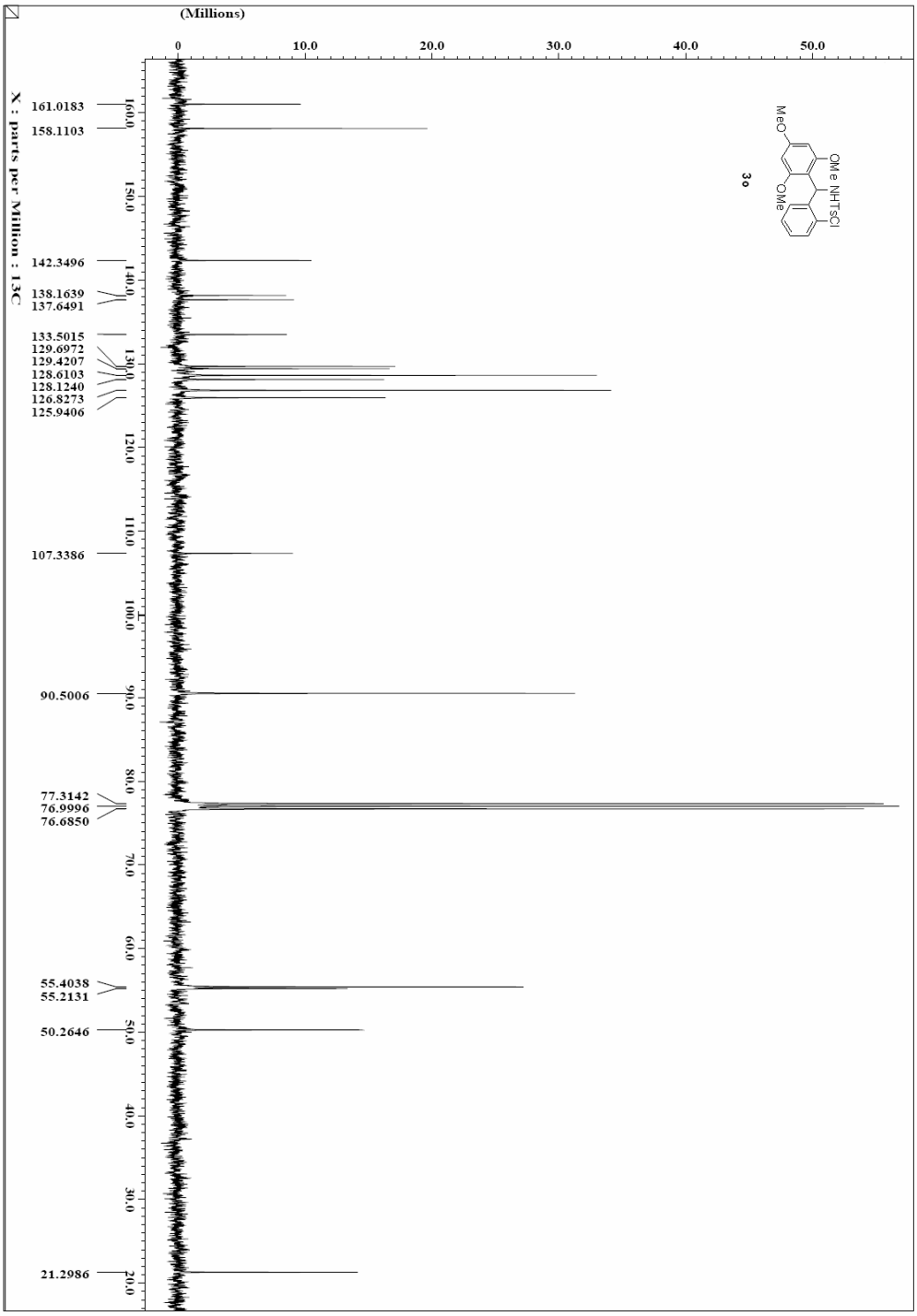

(Millions)
X : parts per Million : 1H
7.5004
7.4798
7.3114
7.2610
7.2507
7.2439
7.2301
7.2232
7.0594
6.9964
6.9758
6.4672
6.4409
6.3126
6.2919
6.1339
6.1075
3.6550
2.2919
-0.0002
20
10.79401
10.54683
10.55671
20.05064
10.63833
9.97369
20.02049
10.5968
60.5896
30.93758
OMe NHTsBr
OMe
F
3m

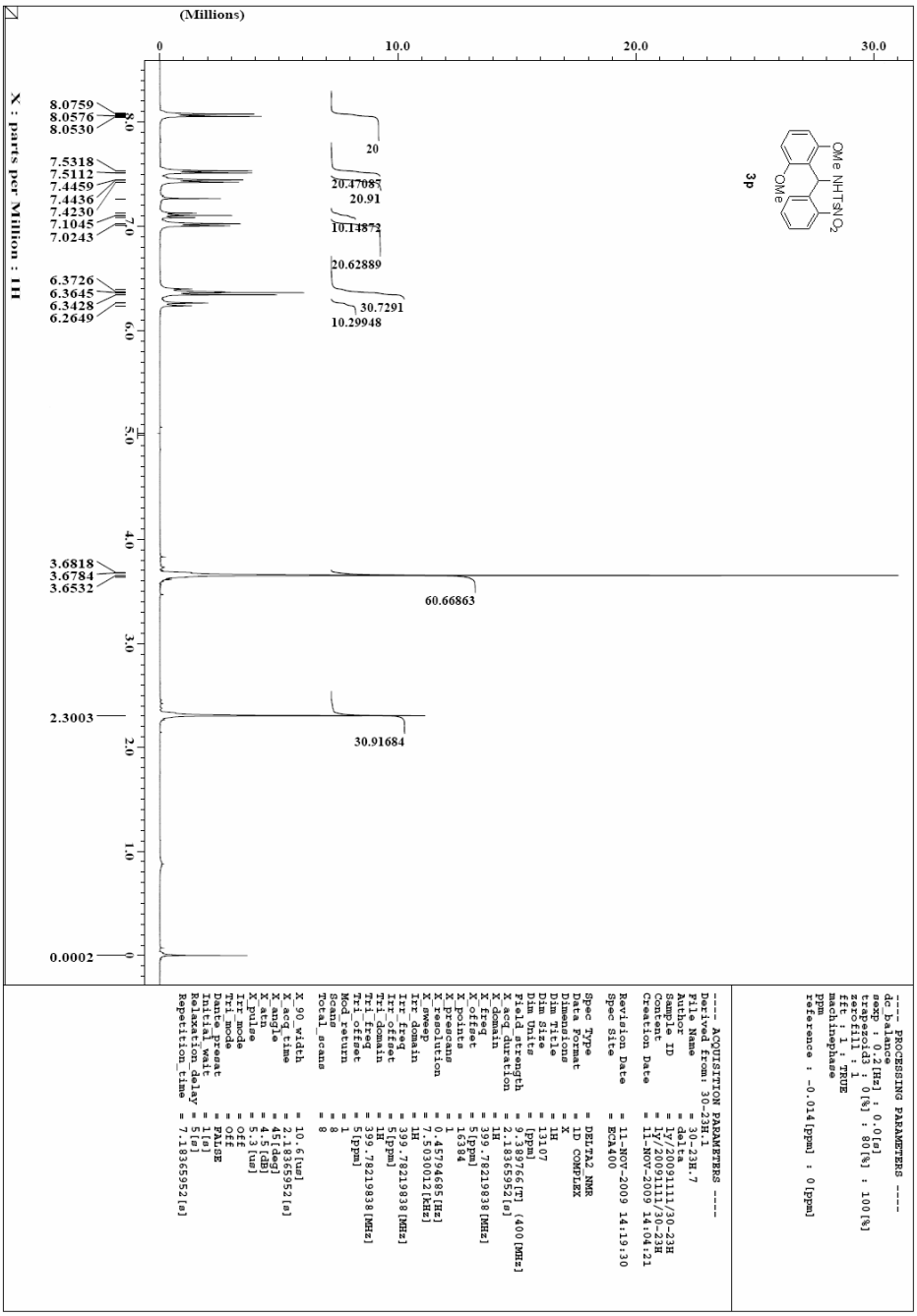

(Millions)
X : parts per Million : 13C
162.5061
160.0176
157.3193
142.5598
137.3920
135.5709
130.8990
130.8132
129.2495
128.6202
126.7419
123.8815
123.7862
120.2489
120.0010
114.2611
113.8798
113.6700
103.9066
77.3242
77.0000
76.6854
55.4614
52.3245
21.3085
OMe NHTsBr
OMe
F
3m

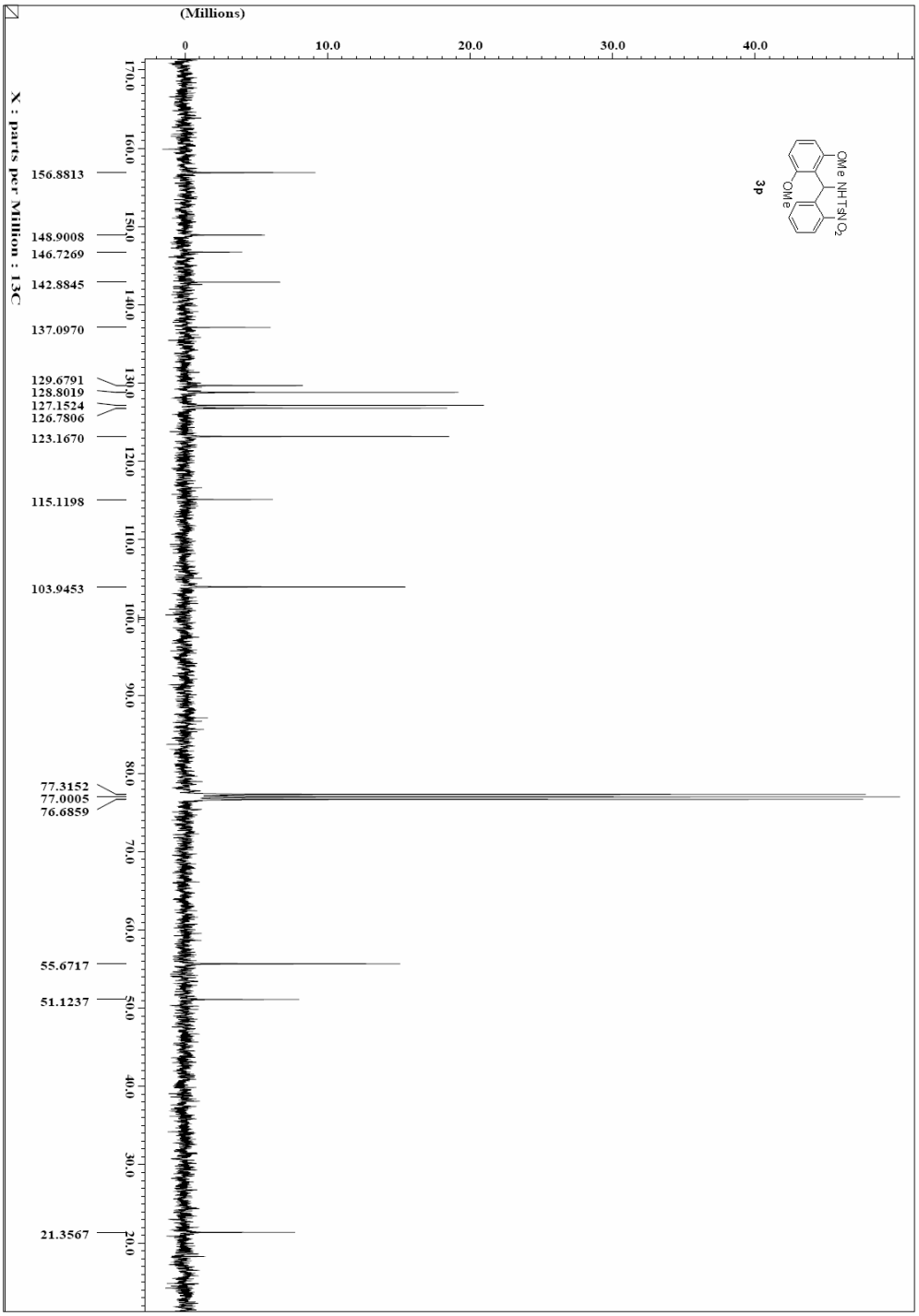

(Millions)
0
10.0
20.0
30.0
40.0
50.0
X : parts per Million : 1H
7.5092
7.4886
7.2640
7.2400
7.2343
7.2194
7.0315
7.0109
6.3454
6.3202
6.0120
5.9857
5.8597
3.7291
3.6282
2.3155
1.3361
1.2846
1.2559
0.0005
20.2392
9.99439
9.89127
20
10.03369
9.73055
10.2186
19.84434
29.93693
59.97398
30.80959
MeO
OMe
NHTsBz
F
3n

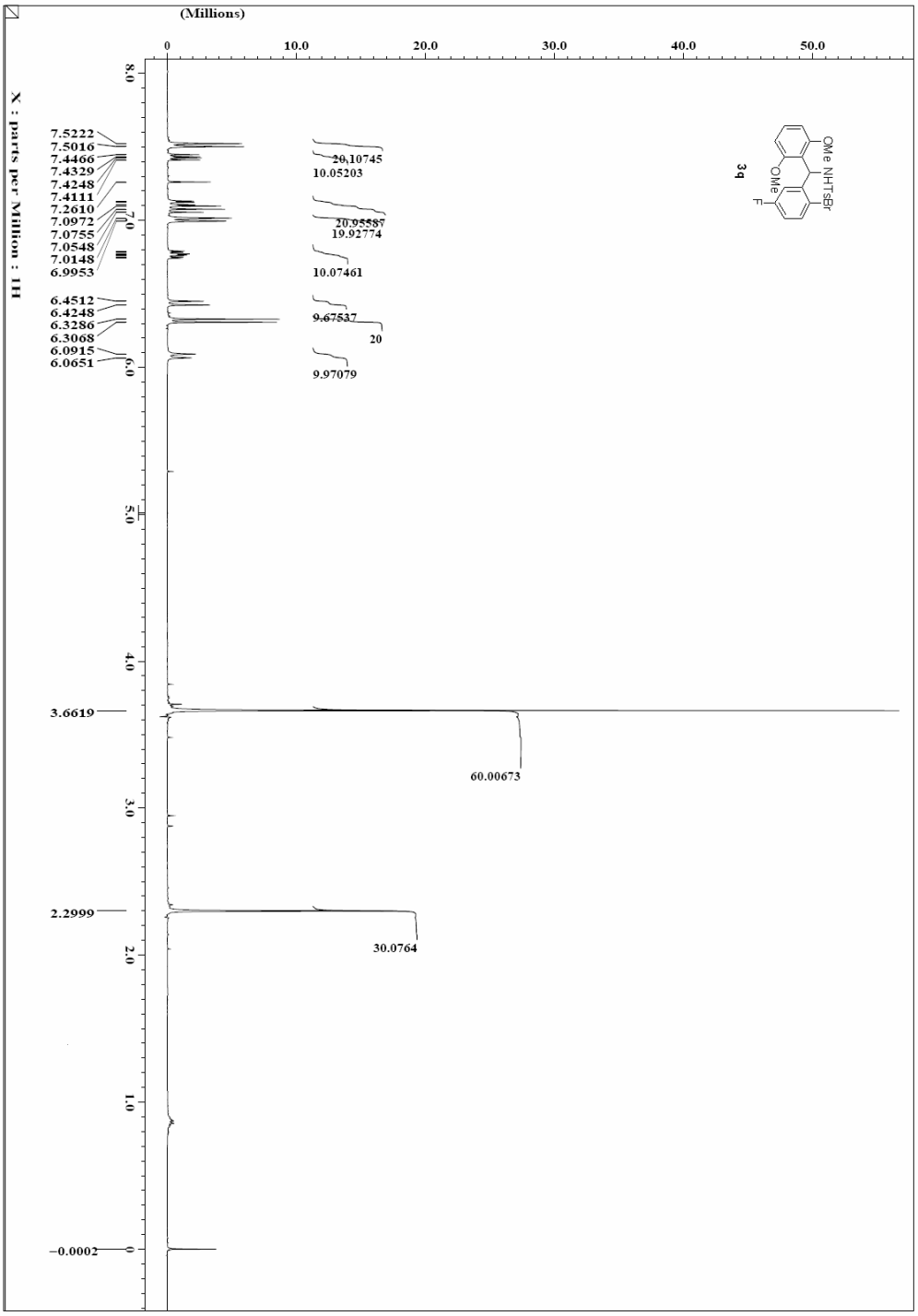

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 13C
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
162.4204
161.1428
159.9319
158.0345
142.4455
137.5066
135.7999
130.8896
130.8038
128.6108
126.7993
123.7101
123.6147
120.1536
119.9153
113.7560
113.5462
106.9769
90.4534
77.3148
77.0001
76.6760
55.3853
55.2136
52.1054
21.2896
MeO
OMe
OMe NHTsBr
F
3n

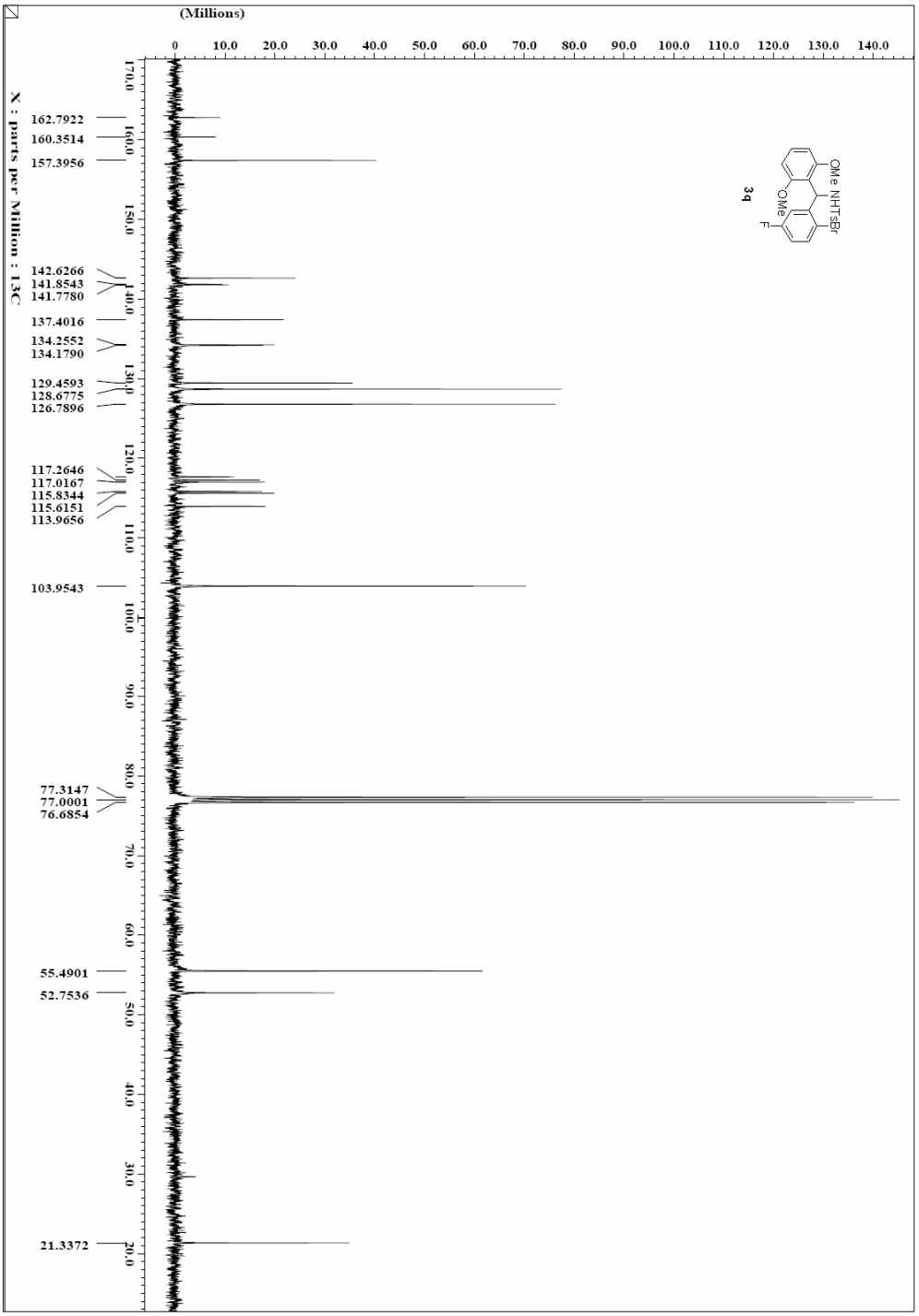

(Millions)
X : parts per Million : 1H
7.5281
7.5075
7.2600
7.2555
7.0997
7.0905
7.0813
7.0275
7.0069
6.4261
6.3998
6.0470
6.0206
5.8648
3.7251
3.6242
2.3092
−0.0001
20
10.39342
13.05152
20.64353
20.44412
9.30143
9.28871
20.1063
30.63961
60.82419
29.6423
3o
MeO
OMe
OMe NHTsCl

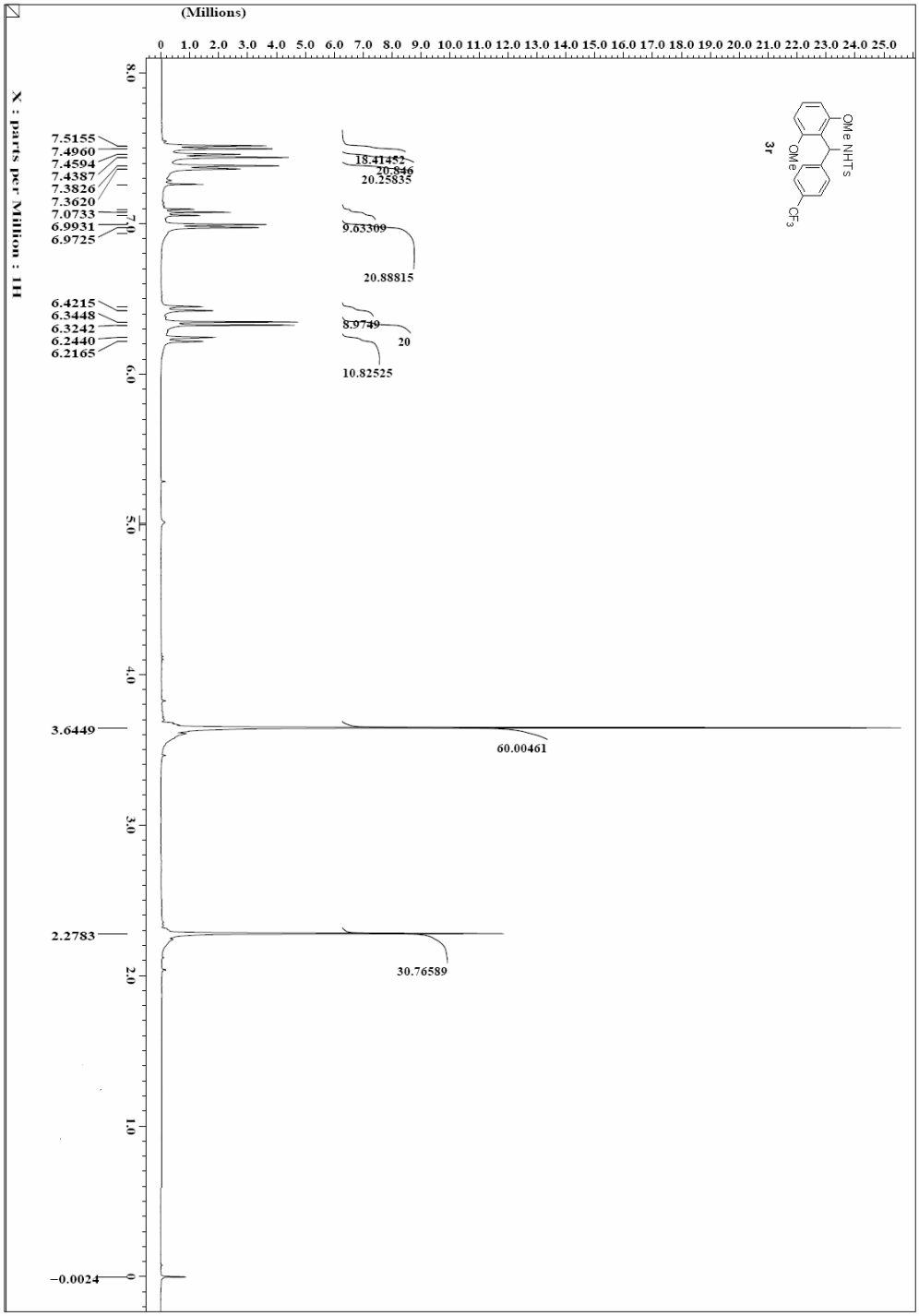

(Millions)
0
10.0
20.0
30.0
40.0
50.0
X : parts per Million : 13C
161.0183
158.1103
142.3496
138.1639
137.6491
133.5015
129.6972
129.4207
128.6103
128.1240
126.8273
125.9406
107.3386
90.5006
77.3142
76.9996
76.6850
55.4038
55.2131
50.2646
21.2986
MeO
OMe
NHTsCl
3o

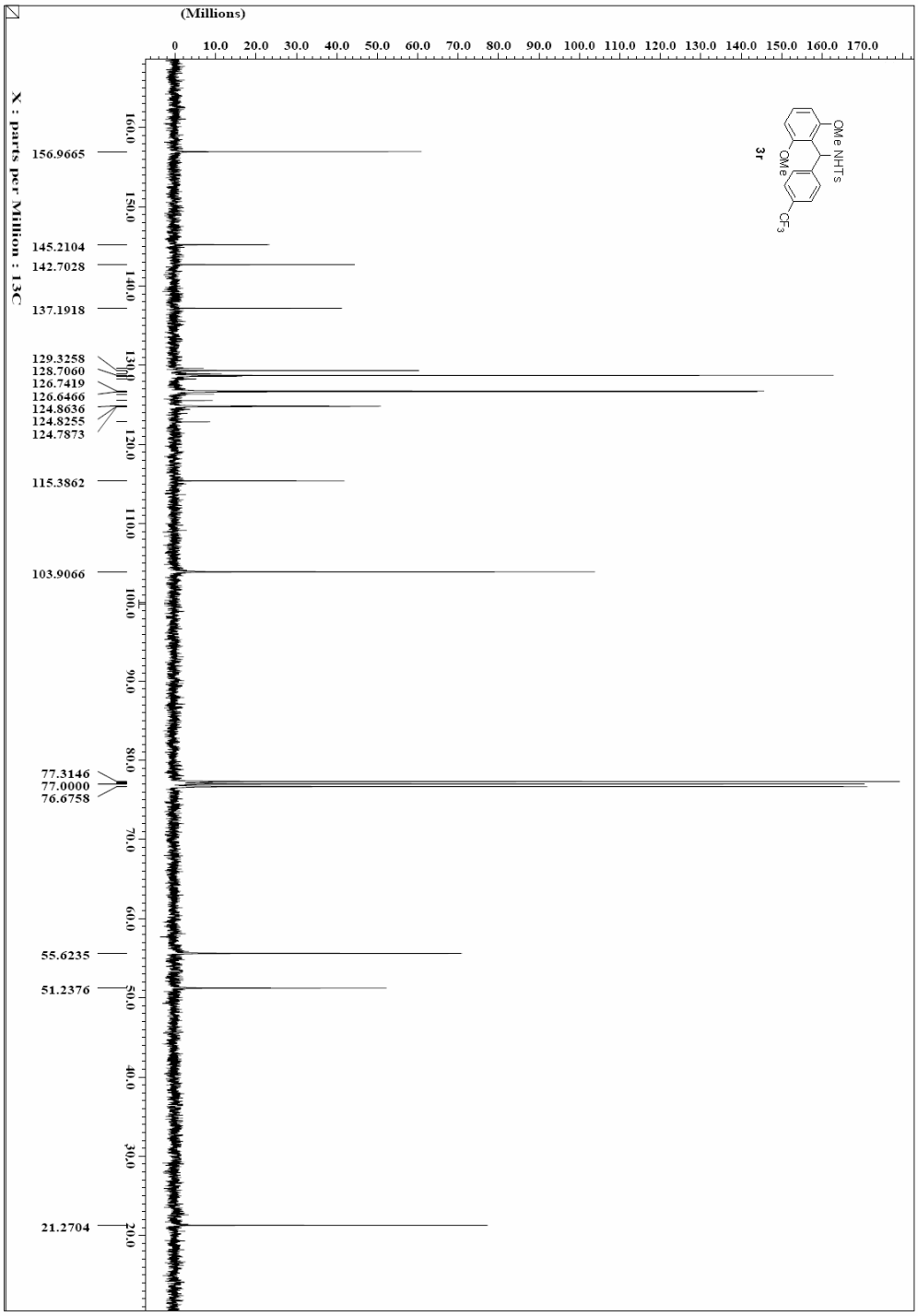

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 1H
8.0759
8.0576
8.0530
7.5318
7.5112
7.4450
7.4436
7.4230
7.1045
7.0243
6.3726
6.3645
6.3428
6.2649
3.6818
3.6784
3.6532
2.3003
0.0002
20
20.47087
20.91
10.14872
20.62889
30.7291
10.29948
60.66863
30.91684
OMe
NHTsNO2
OMe
3p
---- PROCESSING PARAMETERS ----
dc_balance : 0.2[Hz] ; 0.0[g]
sweep : 0.2[Hz] ; 0[%] ; 80[%] ; 100[%]
trapezoid3 : 0[%] ; 80[%] ; 100[%]
zerofill : 1
fft : 1 ; TRUE
magnitude
phase
reference : -0.014[ppm] ; 0[ppm]
---- ACQUISITION PARAMETERS ----
Derived from: 30-23H.1
File Name = 30-23H.7
Author = dw/20091111/30-23H
Sample ID = ly/20091111/30-23H
Content = 11-NOV-2009 14:04:21
Creation Date = 11-NOV-2009 14:19:30
Revision Date = ECA400
Spec Site
Spec Type = DELTA2_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 1H
Dim Size = 13107
Dim Units = [ppm]
Field_strength = 9.399766[T] (400[MHz]
X_acq_duration = 2.18365952[s]
X_domain = 1H
X_freq = 399.78219838[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.45794685[Hz]
X_sweep = 7.50300012[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Irr_points = 16384
Tri_freq = 399.78219838[MHz]
Tri_offset = 5[ppm]
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 10.6[us]
X_acq_time = 2.18365952[s]
X_angle = 45[deg]
X_atn = 4.5[dB]
X_pulse = 5.3[us]
Irr_mode = Off
Tri_mode = Off
Decpl_preset = FALSE
Initial_wait = 1[s]
Relaxation_delay = 5[s]
Repetition_time = 7.18365952[s]

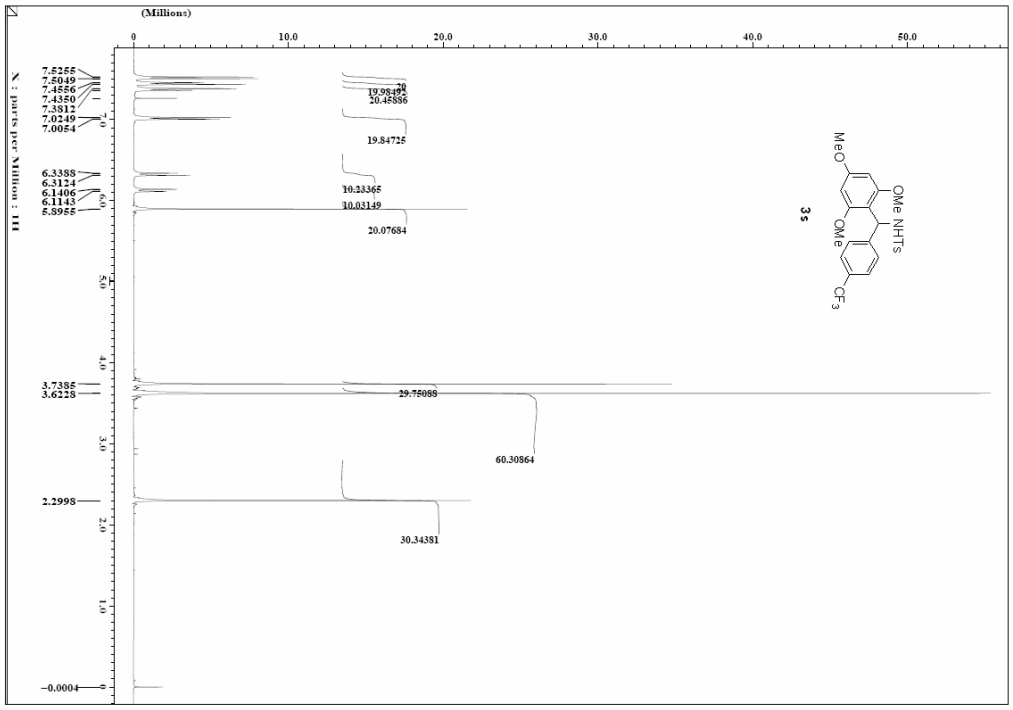

(Millions)
0
10.0
20.0
30.0
40.0
X : parts per Million : 13C
156.8813
148.9008
146.7269
142.8845
137.0970
129.6791
128.8019
127.1524
126.7806
123.1670
115.1198
103.9453
77.3152
77.0005
76.6859
55.6717
51.1237
21.3567
OMe
NHTsNO2
OMe
3p

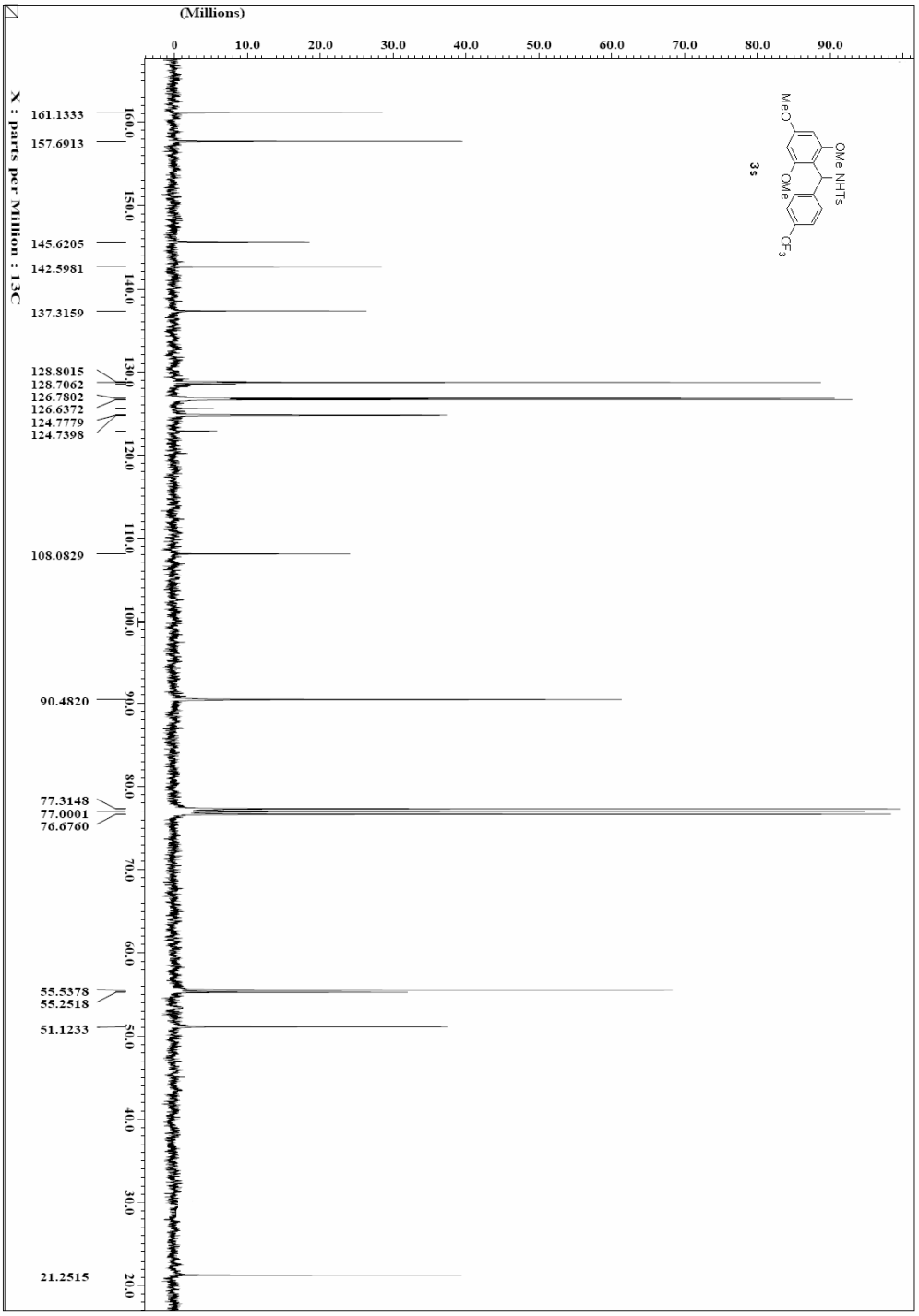

(Millions)
X : parts per Million : 1H
7.5222
7.5016
7.4466
7.4329
7.4248
7.4111
7.2610
7.0972
7.0755
7.0548
7.0148
6.9953
6.4512
6.4248
6.3286
6.3068
6.0915
6.0651
3.6619
2.2999
−0.0002
20.10745
10.05203
20.95587
19.92774
10.07461
9.67537
20
9.97079
60.00673
30.0764
3b
OMe
NHTs
Br
OMe
F


(Millions)
X : parts per Million : 13C
162.7922
160.3514
157.3956
142.6266
141.8543
141.7780
137.4016
134.2552
134.1790
129.4593
128.6775
126.7896
117.2646
117.0167
115.8344
115.6151
113.9656
103.9543
77.3147
77.0001
76.6854
55.4901
52.7536
21.3372
OMe NHTsBr
OMe
F
3q


(Millions)
X : parts per Million : 1H
7.5155
7.4960
7.4594
7.4387
7.3826
7.3620
7.0733
6.9931
6.9725
6.4215
6.3448
6.3242
6.2440
6.2165
3.6449
2.2783
−0.0024
18.41452
20.846
20.25835
9.63309
20.88815
8.9749
20
10.82525
60.00461
30.76589
3r
OMe
NHTs
CF3


(Millions)
0 10.0 20.0 30.0 40.0 50.0 60.0 70.0 80.0 90.0 100.0 110.0 120.0 130.0 140.0 150.0 160.0 170.0
X : parts per Million : 13C
160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0
156.9665
145.2104
142.7028
137.1918
129.3258
128.7060
126.7419
126.6466
124.8636
124.8255
124.7873
115.3862
103.9066
77.3146
77.0000
76.6758
55.6235
51.2376
21.2704
OMe NHTs
OMe
CF3
3r


(Millions)
0
10.0
20.0
30.0
40.0
50.0
X : parts per Million : 1H
7.5255
7.5049
7.4556
7.4350
7.3812
7.0249
7.0054
6.3388
6.3124
6.1406
6.1143
5.8955
3.7385
3.6228
2.2998
-0.0004
19.98492
20.45886
19.84725
10.33365
10.03149
20.07684
29.75089
60.30864
30.34381
MeO
OMe NHTs
OMe
CF3
3s


(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
X : parts per Million : 13C
161.1333
157.6913
145.6205
142.5981
137.3159
128.8015
128.7062
126.7802
126.6372
124.7779
124.7398
108.0829
90.4820
77.3148
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55.5378
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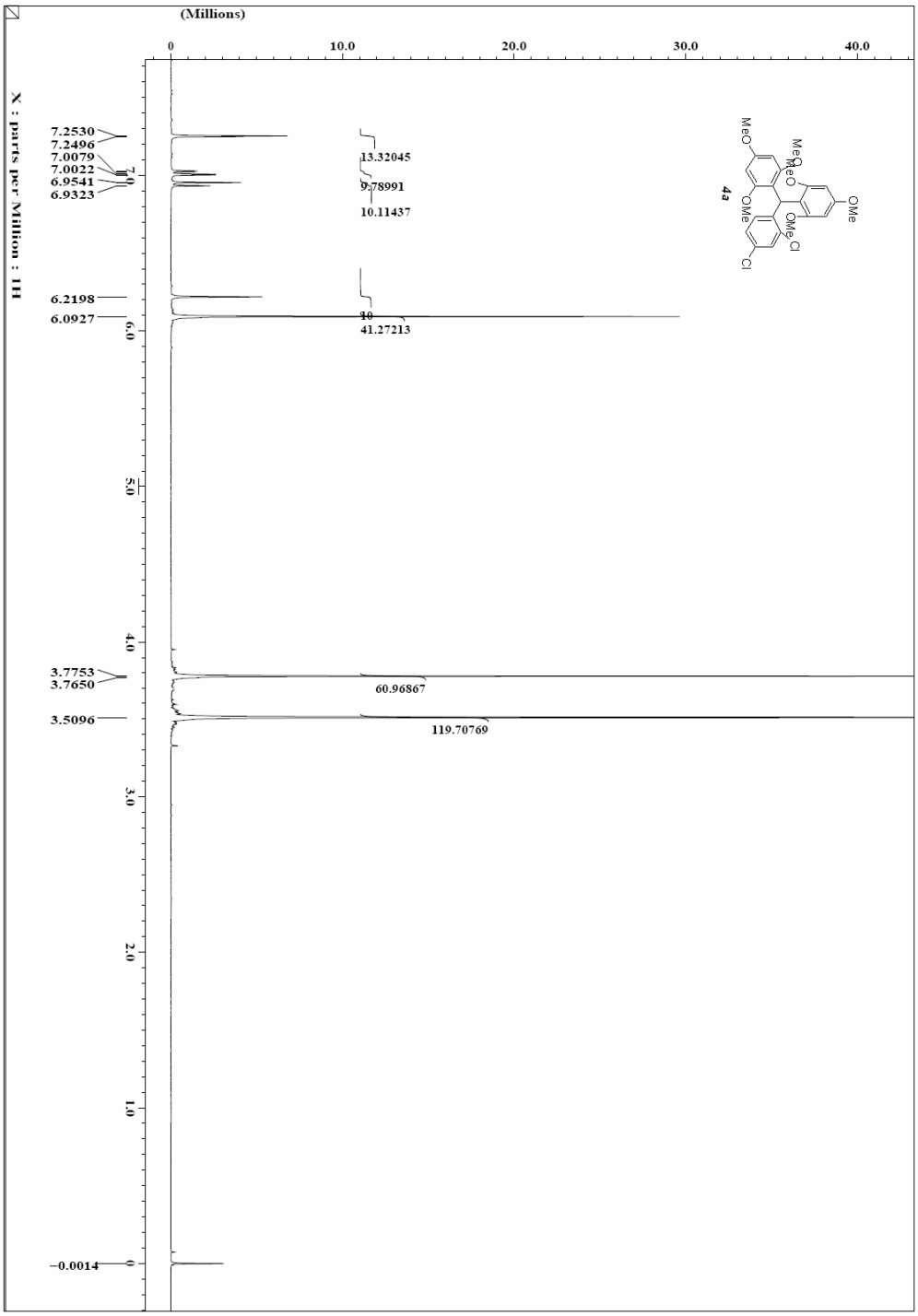
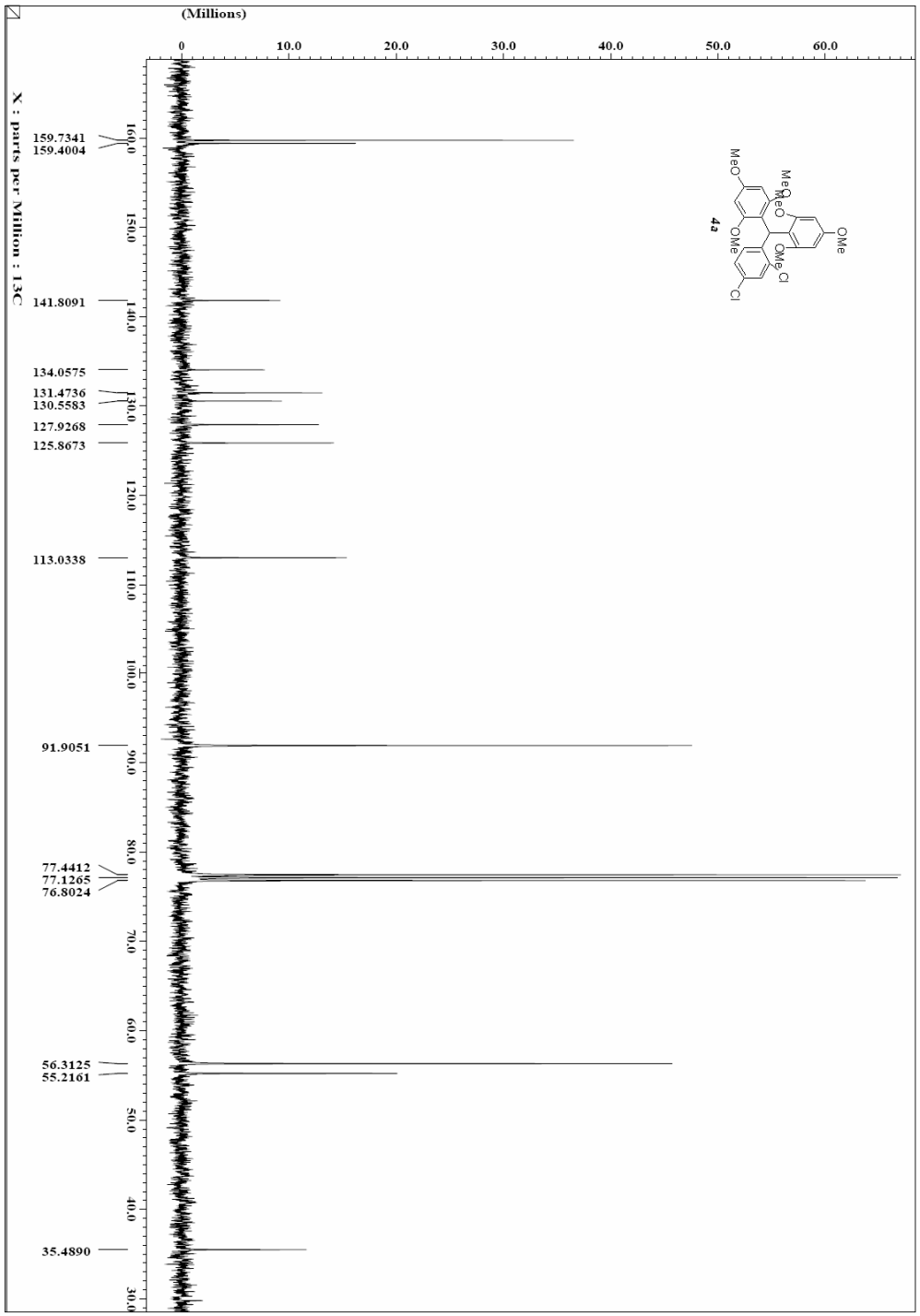
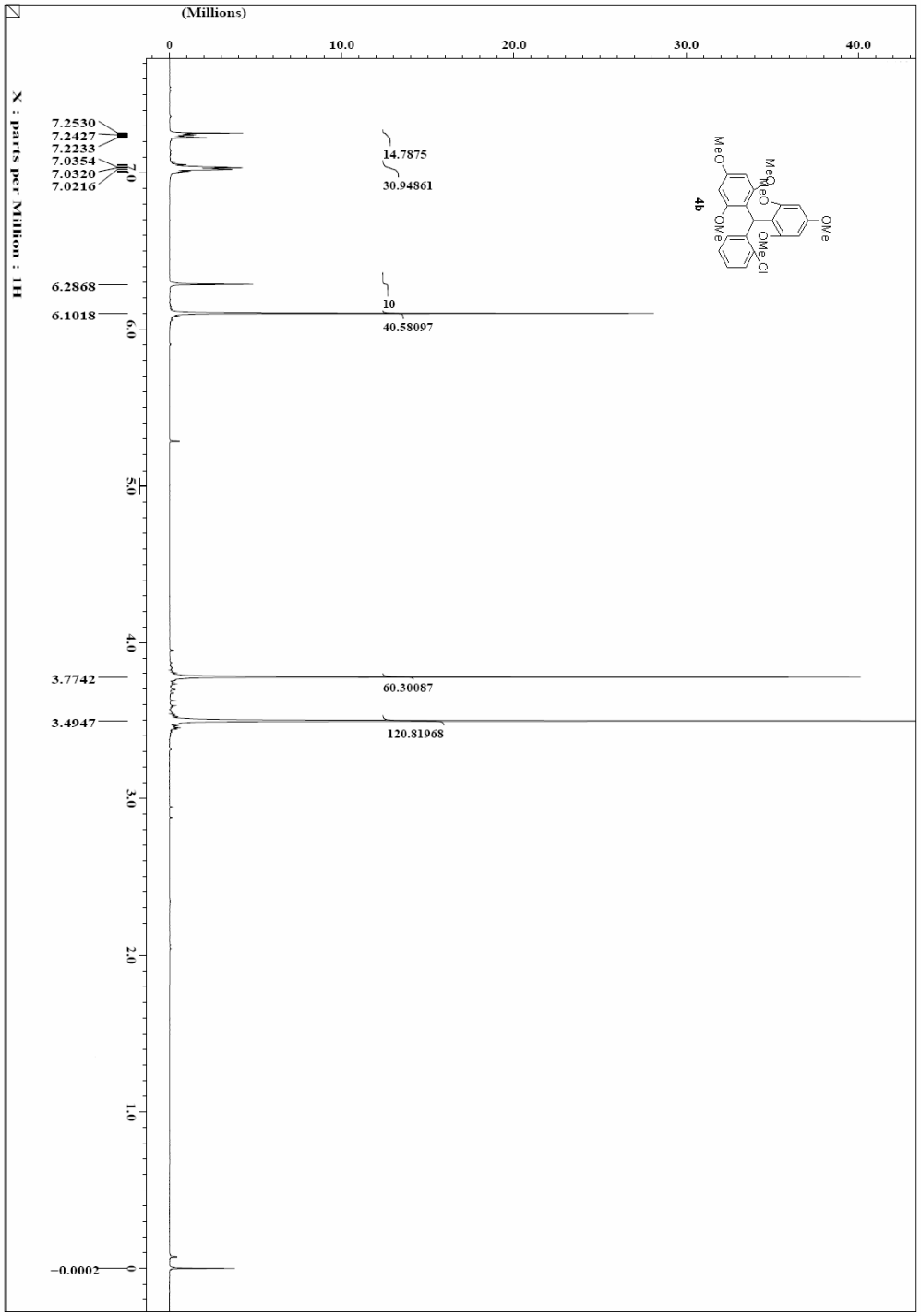
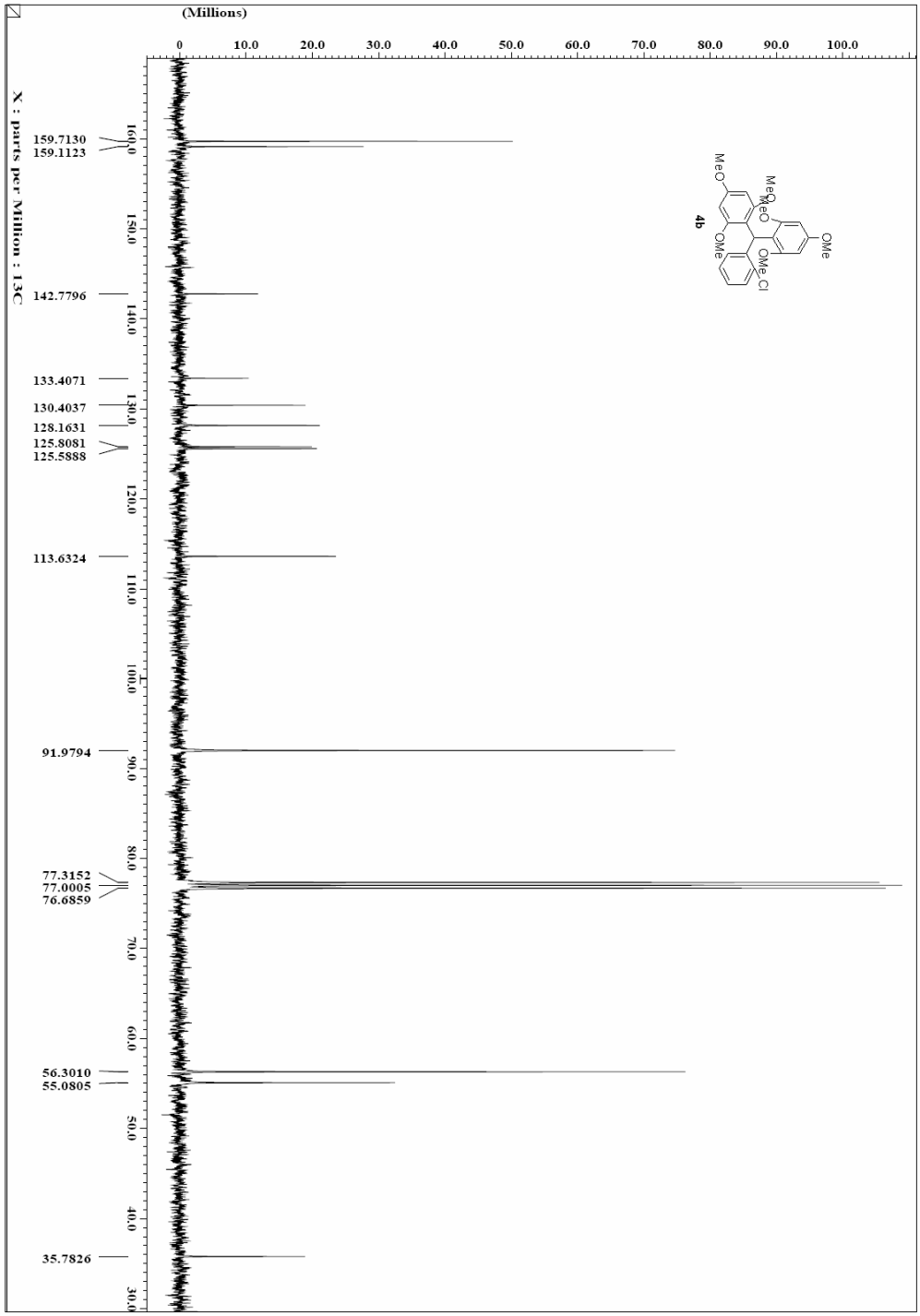
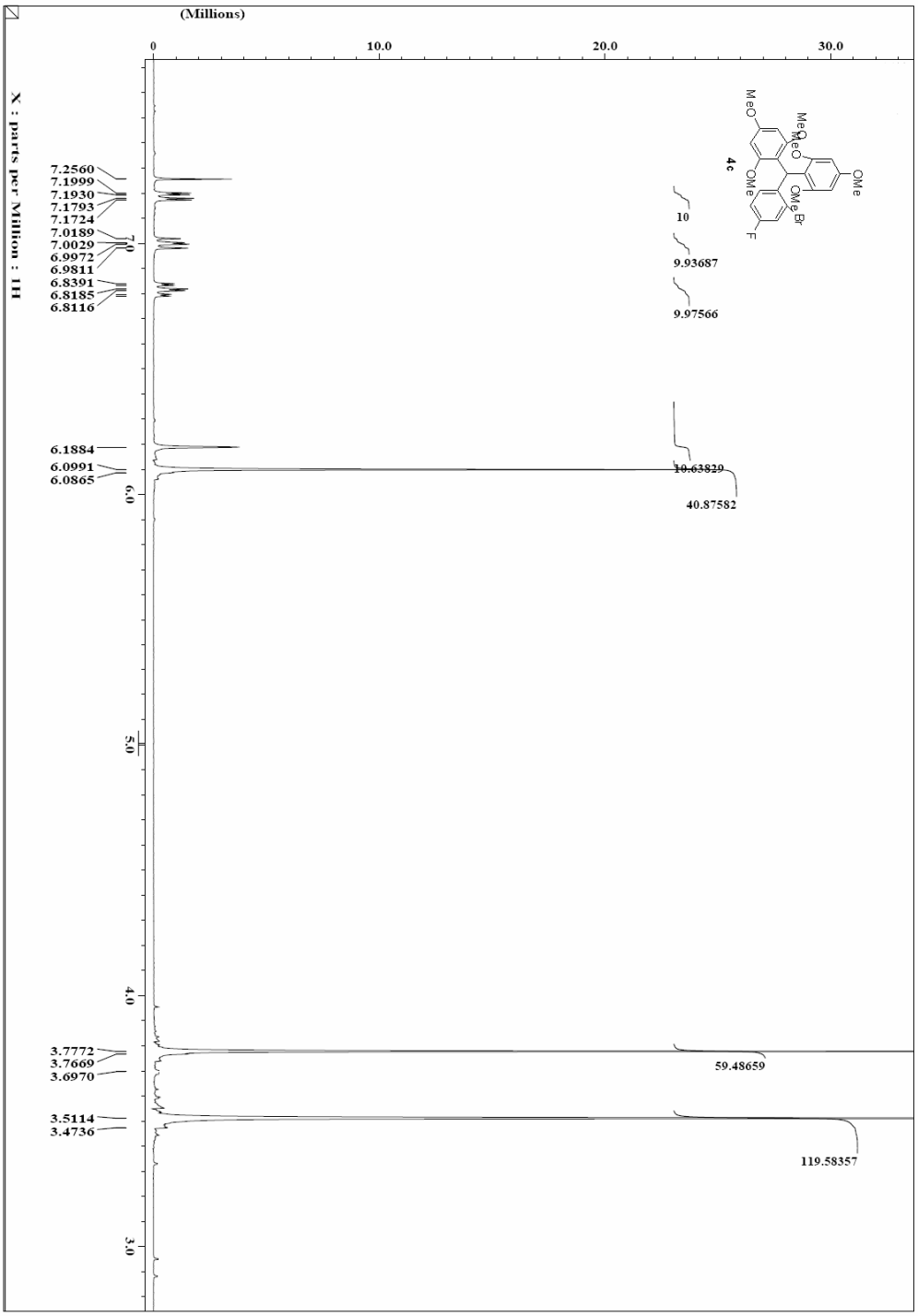
51.1233
21.2515
MeO
OMe
NHTs
CF3
3s

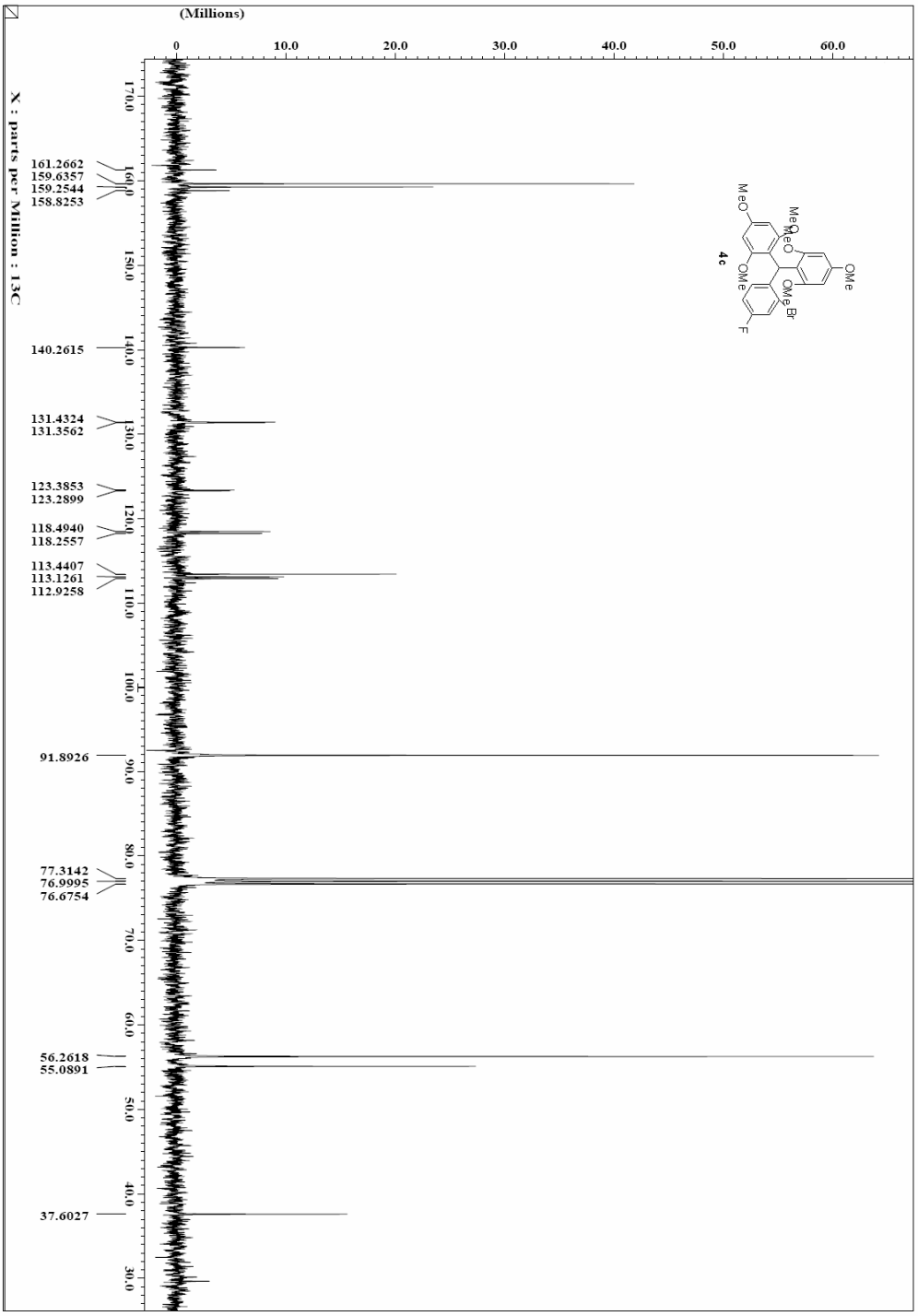

(Millions)
0
10.0
20.0
30.0
40.0
X : parts per Million : 1H
7.2530
7.2496
7.0079
7.0022
6.9541
6.9323
6.2198
6.0927
3.7753
3.7650
3.5096
−0.0014
13.32045
9.78991
10.11437
10
41.27213
60.96867
119.70769
MeO
OMe
Cl
4a

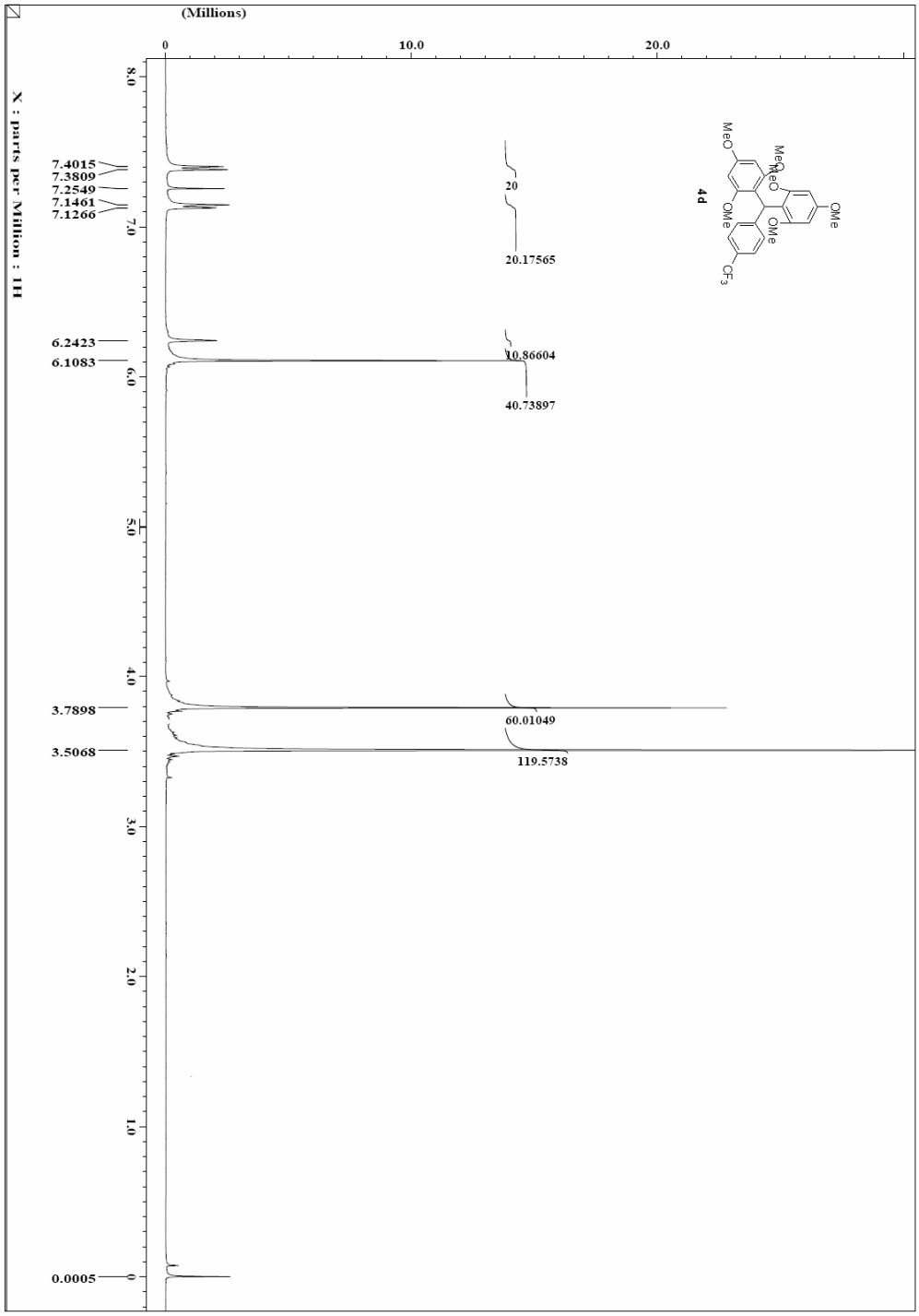

(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
X : parts per Million : 13C
159.7341
159.4004
141.8091
134.0575
131.4736
130.5583
127.9268
125.8673
113.0338
91.9051
77.4412
77.1265
76.8024
56.3125
55.2161
35.4890
MeO
MeO
OMe
OMe
OMe
Cl
Cl
4a

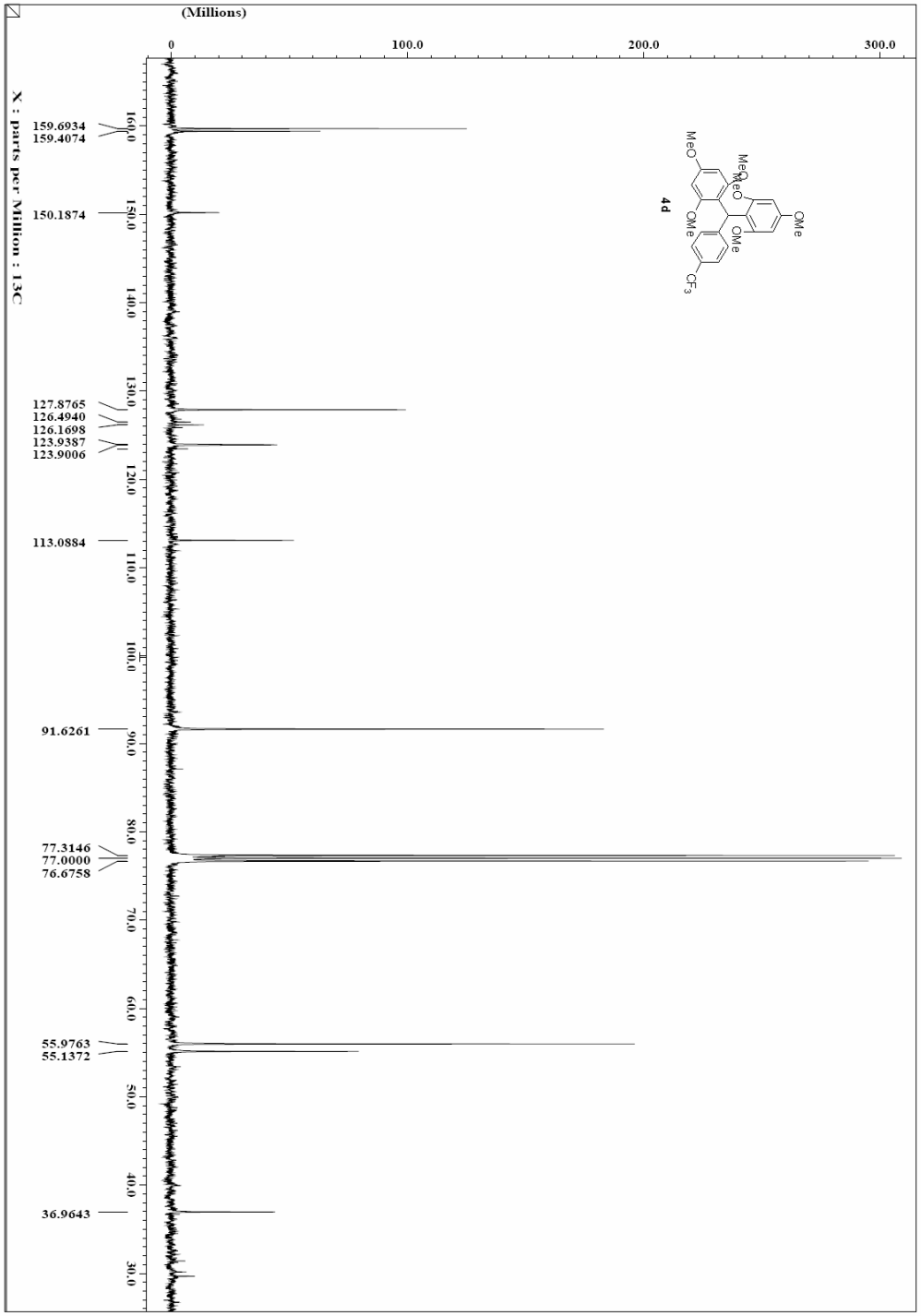

(Millions)
0
10.0
20.0
30.0
40.0
X : parts per Million : 1H
7.2530
7.2427
7.2233
7.0354
7.0320
7.0216
14.7875
30.94861
6.2868
6.1018
10
40.58097
3.7742
60.30087
3.4947
120.81968
−0.0002
4b
MeO
MeO
OMe
OMe
OMe
OMe
Cl


(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
X : parts per Million : 13C
159.7130
159.1123
142.7796
133.4071
130.4037
128.1631
125.8081
125.5888
113.6324
91.9794
77.3152
77.0005
76.6859
56.3010
55.0805
35.7826
MeO
MeO
OMe
OMe
OMe
Cl
4b


(Millions)
0
10.0
20.0
30.0
X : parts per Million : 1H
7.2560
7.1999
7.1930
7.1793
7.1724
7.0189
7.0029
6.9972
6.9811
6.8391
6.8185
6.8116
6.1884
6.0991
6.0865
3.7772
3.7669
3.6970
3.5114
3.4736
7.0
6.0
5.0
4.0
3.0
4c
MeO
MeO
MeO
OMe
OMe
OMe
Br
F
10
9.93687
9.97566
10.63829
40.87582
59.48659
119.58357


(Millions)
X : parts per Million : 13C
161.2662
159.6357
159.2544
158.8253
140.2615
131.4324
131.3562
123.3853
123.2899
118.4940
118.2557
113.4407
113.1261
112.9258
91.8926
77.3142
76.9995
76.6754
56.2618
55.0891
37.6027
4c


(Millions)
0
10.0
20.0
X : parts per Million : 1H
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.4015
7.3809
7.2549
7.1461
7.1266
6.2423
6.1083
3.7898
3.5068
0.0005
20
20.17565
10.86604
40.73897
60.01049
119.5738
4d
MeO
MeO
OMe
OMe
OMe
CF3


(Millions)
0
100.0
200.0
300.0
X : parts per Million : 13C
159.6934
159.4074
150.1874
127.8765
126.4940
126.1698
123.9387
123.9006
113.0884
91.6261
77.3146
77.0000
76.6758
55.9763
55.1372
36.9643
MeO
OMe
OMe
OMe
OMe
CF3
4d