A facile sulfochlorination of alkenes with $Me_2SO/(COCl)_2$

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CONTENTS

SUPPORTING INFORMATION

Experimental

All the alkenes and reagents were purchased from Beijing Bailingwei Science and Technology Company (Beijing, China). NMR spectra were obtained on a Bruker AV300 or 600 MHz NMR (^{1}H NMR at 300 or 600 MHz, ^{13}C NMR at 75 or 150MHz) in $CDCl_3$ using TMS as internal standard. Chemical shifts (δ) are given in ppm and coupling constants (*J*) in Hz. HRMS data were obtained on a Solarix, Autoflex III Mass Spectrometer or Q Exactive GC.

General reaction procedure of alkenes with dimethyl sulfoxide-oxalyl chloride

To a 50 mL three necked round bottom flask, fitted with a condenser and adapted with a $CaCl_2$ valve, dimethylsulfoxide (DMSO 1.7 mL, 24 mmol, 2.4 equivalents) and CH_2Cl_2 (10 mL) were poured. The stirred solution was cooled to 0 °C and then a solution of oxalyl chloride (1.0 mL, 12 mmol, 1.2 equivalents) in CH_2Cl_2 (10 mL) was added dropwise from a dropping funnel. After 10 min, a solution of the alkene (10 mmol, 1.0 equivalent) in CH_2Cl_2 (10 mL) was added. The reaction mixture was then allowed to warm to room temperature and stirred until TLC showed the reaction completed. Triethylamine (7.0 mL, 50 mmol, 5.0 equivalents) was added in one portion. After stirring for 10 min, the mixture was successively washed with a saturated aqueous solution of NH_4Cl (30 mL) and brine (2 × 30 mL). The combined organic extracts were dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The product was purified by chromatography on silica gel.

1-Methylthio-2-chlorodecane **2a**: colorless oil; 1.94 g, 87% yield. ^{1}H NMR ($CDCl_3$) δ 0.88 (t, *J* = 7.2 Hz, 3 H, H-C-10), 1.22~1.46 (m, 11 H, H-C-4~9), 1.55 (m, 1 H, H′-C-4), 1.68 (m, 1 H, H-C-3), 1.97 (m, 1 H, H′-C-3), 2.17 (s, 3 H, SC$\underline{H}_3$), 2.81 (dd, *J* = 13.8, 7.8 Hz, 1 H, H-C-1), 2.90 (dd, *J* = 13.8, 6.0 Hz, 1 H, H′-C-1), 4.01 (m, 1 H, H-C-2); ^{13}C NMR ($CDCl_3$) δ 14.2 (C-10), 16.6 (S$\underline{C}H_3$), 22.7 (C-9), 26.3 (C-4), 29.2 (C-5), 29.3 (C-7), 29.5 (C-6), 31.9 (C-8), 36.8 (C-3), 42.6 (C-1), 62.0 (C-2). The ^{1}H NMR data were matched with those reported in lit.[1]

1-Methylthio-2-chloro-3-phenylpropane **2b**: colorless oil; 1.80 g, 90% yield. ^{1}H NMR ($CDCl_3$) δ 2.19 (s, 3 H, SC$\underline{H}_3$), 2.85 (dd, *J* = 13.8, 7.2 Hz, 1 H, H-C-1), 2.89 (dd,

J = 13.8, 6.0 Hz, 1 H, H′-C-1), 3.01 (dd, *J* = 14.4, 7.8 Hz, 1 H, H-C-3), 3.33 (dd, *J* = 14.4, 4.8 Hz, 1 H, H′-C-3), 4.23 (m, 1 H, H-C-2), 7.26 (m, 3 H, H(phenyl)), 7.33 (m, 2 H, H(phenyl)); ^{13}C NMR ($CDCl_3$) δ 16.7 (S<u>C</u>H$_3$), 41.8 (C-1), 42.9 (C-3), 62.0 (C-2), 127.0 (C-4′(phenyl)), 128.5 (C-2′and C-6′ (phenyl)), 129.6 (C-3′and C-5′ (phenyl)), 137.4 (C-1′(phenyl)). The ^{1}H NMR data were matched with those reported in lit.[1]

1-Methylthio-2-chloro-3,3-dimethylbutane **2c**: colorless oil; 1.53 g, 92% yield. ^{1}H NMR ($CDCl_3$) δ 0.99 (s, 9 H, C<u>H</u>$_3$-C-3), 2.13 (s, 3 H, SC<u>H</u>$_3$), 2.64 (dd, *J* = 13.8, 10.5 Hz, 1 H, H-C-1), 2.93 (dd, *J* = 13.8, 2.4 Hz, 1 H, H′-C-1), 3.77 (dd, *J* = 10.5, 2.4 Hz, 1 H, H-C-2); ^{13}C NMR ($CDCl_3$) δ 16.3 (S<u>C</u>H$_3$), 26.5 (<u>C</u>H$_3$-C-3), 36.2 (C-3), 38.8 (C-1), 73.6 (C-2). The ^{1}H NMR data were matched with those reported in lit.[1]

1-Cyclohexyl-1-chloro-2-methylthioethane **2d**: colorless oil; 1.69 g, 88% yield. ^{1}H NMR ($CDCl_3$) δ 1.52-1.92 and 1.20-1.40 (m, 11 H, cyclohexyl), 2.17 (s, 3 H, SC<u>H</u>$_3$), 2.85 (dd, *J* = 13.8, 6.9 Hz, 1 H, H-C-2), 2.92 (dd, *J* = 13.8, 6.9 Hz, 1 H, H′-C-2), 3.95 (td, *J* = 6.9, 3.9 Hz, 1 H, H-C-1); ^{13}C NMR ($CDCl_3$) δ 16.3 (S<u>C</u>H$_3$), 25.7 (<u>C</u>H$_2$ (cyclohexyl)), 26.0 (<u>C</u>H$_2$ (cyclohexyl)), 26.1 (<u>C</u>H$_2$ (cyclohexyl)), 26.6 (<u>C</u>H$_2$ (cyclohexyl)), 30.6 (<u>C</u>H$_2$ (cyclohexyl)), 39.7 (C-2), 41.8 (C-1 (cyclohexyl)), 67.5 (C-1). The ^{1}H NMR data were matched with those reported in lit.[1]

1-Chloro-2-methylthio-1-phenylethane **2e**: light yellow oil; 1.68 g, 90% yield. ^{1}H NMR ($CDCl_3$) δ 1.97 (s, 3 H, SC<u>H</u>$_3$), 3.13 (dd, *J* = 14.1, 8.7 Hz, 1 H, H-C-2), 3.23 (dd, *J* = 14.1, 6.6 Hz, 1 H, H′-C-2), 4.98 (dd, *J* = 8.7, 6.6 Hz, 1 H, H-C-1), 7.28~7.46 (m, 5 H, H(phenyl)); ^{13}C NMR ($CDCl_3$) δ 16.0 (S<u>C</u>H$_3$), 43.0 (C-2), 61.8 (C-1), 127.1(C-2′and C-6′ (phenyl)), 127.6 (C-4′(phenyl)), 128.4 (C-3′and C-5′ (phenyl)), 139.8 (C-1′(phenyl)). The ^{1}H NMR data were matched with those reported in lit.[2]

1-Chloro-2-methylthiocyclohexane **2f**: light yellow oil; 1.58 g, 96% yield. ^{1}H NMR ($CDCl_3$) δ 1.30-1.85 (m, 6 H, H-C-3~6), 2.17 (s, 3 H, SC<u>H</u>$_3$), 2.20~2.35 (m, 2 H, H′-C-3 and H′-C-6), 2.75 (td, *J* = 7.8, 4.2 Hz, 1 H, H-C-2), 4.01 (td, *J* = 7.8, 3.9 Hz, 1 H, H-C-1); ^{13}C NMR ($CDCl_3$) δ 15.0 (S<u>C</u>H$_3$), 23.6 (C-5), 23.9 (C-4), 30.7 (C-3), 34.4 (C-6), 52.0 (C-2), 63.5 (C-1). The ^{1}H NMR data were matched with those reported in lit.[1]

1-Chloro-2-methylthiocyclopentane **2g**: light yellow oil; 1.37 g, 91% yield. ^{1}H NMR ($CDCl_3$) δ 1.45-1.58 and 1.65-2.00 (m, 4 H, H-C-3 and H-C-4), 2.15 (s, 3 H, SC<u>H</u>$_3$),

2.22-2.42 (m, 2 H, H-C-5), 3.20 (m, 1 H, H-C-2), 4.23 (m, 1 H, H-C-1); ^{13}C NMR ($CDCl_3$) δ 16.4 (S$\underline{C}H_3$), 22.2 (C-4), 30.6 (C-3), 34.9 (C-5), 54.8 (C-2), 65.5 (C-1). The ^{1}H NMR data were matched with those reported in lit.[1]

1-Chloro-2-methylthiocycloheptane **2h**: light yellow oil; 1.54 g, 86% yield. ^{1}H NMR ($CDCl_3$) δ 1.38-1.82 and 1.86-2.20 (m, 10 H, H-C-3~7), 2.10 (s, 3 H, SC$\underline{H}_3$), 2.98 (ddd, *J* = 8.7, 5.7, 2.7 Hz, 1 H, H-C-2), 4.25 (td, *J* = 5.7, 3.3 Hz, 1 H, H-C-1); ^{13}C NMR ($CDCl_3$) δ 15.0 (S$\underline{C}H_3$), 22.2 (C-4), 24.4 (C-6), 27.8 (C-5), 29.8 (C-3), 33.3 (C-7), 55.5 (C-2), 66.0 (C-1). The ^{1}H NMR data were matched with those reported in lit.[1]

anti-1,3-Dichloro-2-methylthiohexane 2i and *syn*-1-methylthio-2,3-dichlorohexane 2i′: light yellow oil; 1.30 g, 65% yield. ^{1}H NMR ($CDCl_3$) δ 0.89 (t, *J* = 7.2 Hz, 3 H) and 0.90 (t, *J* = 7.5 Hz, 3 H) (partly overlapping, H-C-6 (2i and 2i′)), 1.30~1.95 (m, 8 H, H-C-4 and H-C-5 (2i and 2i′)), 2.11 (s, 3 H, SC$\underline{H}_3$ (2i′)), 2.17 (s, 3 H, SC$\underline{H}_3$ (2i)), 2.84 (dd, *J* = 13.8, 6.0 Hz, 1 H, H-C-1 (2i′)), 2.95 (td, *J* = 6.3, 5.1 Hz, 1 H, H-C-2 (2i)), 3.04 (dd, *J* = 13.8, 8.4 Hz, 1 H, H′-C-1 (2i′)), 3.77 (dd, *J* = 11.7, 6.3 Hz, 1 H, H-C-1 (2i)), 3.89 (dd, *J* = 11.7, 5.1 Hz, 1 H, H′-C-1 (2i)), 4.06 (ddd, *J* = 8.4, 6.0, 2.1 Hz, 1 H, H-C-2 (2i′)), 4.21 (ddd, *J* = 9.6, 6.3, 3.0 Hz, 1 H, H-C-3 (2i)), 4.37 (ddd, *J* = 8.7, 4.8, 2.1 Hz, 1 H, H-C-3 (2i′)). ^{13}C NMR ($CDCl_3$) δ 13.36 and 13.41 (C-6 (2i and 2i′)), 16.1 (S$\underline{C}H_3$ (2i)), 16.3 (S$\underline{C}H_3$ (2i′)), 19.7 and 19.8 (C-5 (2i and 2i′)), 36.5 (C-4 (2i)), 37.7 (C-4) (2i′)), 39.2 (C-1 (2i′)), 45.6 (C-1 (2i)), 55.7 (C-2 (2i)), 62.7 (C-3 (2i′)), 63.2 (C-3 (2i)), 63.6 (C-2 (2i′)). The characteristic ^{1}H NMR signals of 2i in the lower field were matched with those reported in lit.[20] HRMS (Autoflex III Mass Spectrometer, EI): calcd. for $C_8H_{16}OS$ M^+ 200.0193, found 200.0196.

syn-1,3-Dichloro-2-methylthiohexane 2j and *anti*-1-methylthio-2,3-dichlorohexane 2j′: light yellow oil; 1.36 g, 68% yield. ^{1}H NMR ($CDCl_3$) δ 0.89 (t, *J* = 7.5 Hz, 3 H, H-C-6 (2j′)) and 0.90 (t, *J* = 7.2 Hz, 0.75 H, H-C-6 (2j)) (partly overlapping), 1.30~2.05 (m, 5 H, H-C-4 and H-C-5 (2j and 2j′)), 2.14 (s, 0.75 H, SC$\underline{H}_3$ (2j)), 2.15 (s, 3 H, SC$\underline{H}_3$ (2j′)), 2.82 (ddd, *J* = 10.5, 5.1, 2.1 Hz, 0.25 H, H-C-2 (2j)), 2.94 (dd, *J* = 14.4, 6.6 Hz, 1 H, H-C-1 (2j′)), 3.05 (dd, *J* = 14.4, 4.8 Hz, 1 H, H′-C-1 (2j′)), 3.72 (dd, *J* = 10.5, 5.1 Hz, 0.25 H, H-C-1 (2j)), 3.84 (dd, *J* = 10.5 Hz, 0.25 H, H′-C-1 (2j)), 4.11 (td, *J* = 6.6, 4.8 Hz, 1 H, H-C-2 (2j′)), 4.17 (td, *J* = 6.6, 2.7 Hz, 1 H, H-C-3 (2j′)), 4.42 (ddd, *J* = 9.6,

5.5, 2.1 Hz, 0.25 H, H-C-3 (2j)). ^{13}C NMR ($CDCl_3$) δ 13.40 (C-6 (2j′)), 13.41 (C-6 (2j)), 15.6 (S<u>C</u>H$_3$ (2j)), 16.8 (S<u>C</u>H$_3$ (2j′)), 19.2 (C-5 (2j′)), 20.1 (C-5 (2j)), 36.0 (C-4 (2j′)), 38.2 (C-4) (2j)), 39.7 (C-1 (2j′)), 44.7 (C-1 (2j)), 55.7 (C-2 (2j)), 61.8 (C-3 (2j)), 63.9 (C-3 (2j′)), 64.8 (C-2 (2j′)). HRMS (Autoflex III Mass Spectrometer, EI): calcd. for $C_8H_{16}OS$ M^+ 200.0193, found 200.0195.

syn-1-Methylthio-2-chloro-3-hexanol 2k: light yellow oil; 0.60 g, 33% yield. ^{1}H NMR ($CDCl_3$) δ 0.89 (t, *J* = 7.2 Hz, 3 H, H-C-6), 1.30~1.52 (m, 4 H, H-C-4 and H-C-5), 1.68 (d, *J* = 9.3 Hz, 1 H, -OH), 2.11 (s, 3 H, SC<u>H</u>$_3$), 2.83 (dd, *J* = 13.8, 5.7 Hz, 1 H, H-C-1), 3.00 (dd, *J* = 13.8, 8.7 Hz, 1 H, H′-C-1), 3.87-3.97 (m, 1 H, H-C-3) and 3.97 (ddd, *J* = 8.7, 5.7, 2.1 Hz, 1 H, H-C-2) (partly overlapping). ^{13}C NMR ($CDCl_3$) δ 13.9 (C-6), 16.4 (S<u>C</u>H$_3$), 18.9 (C-5), 37.2 (C-4), 38.6 (C-1), 65.9 (C-2), 70.7 (C-3). HRMS (Solarix Mass Spectrometer, ESI): calcd. for $C_7H_{15}ClNaOS$ $[M + Na]^+$ 205.0424, found 205.0423.

anti-1-Methylthio-2-chloro-3-hexanol 2k′: light yellow oil; 0.75 g, 41% yield. ^{1}H NMR ($CDCl_3$) δ 0.89 (t, *J* = 7.2 Hz, 3 H, H-C-6), 1.40~1.60 (m, 4 H, H-C-4 and H-C-5), 2.12 (s, 3 H, SC<u>H</u>$_3$), 2.83 (dd, *J* = 14.1, 7.2 Hz, 1 H, H-C-1), 2.89 (dd, *J* = 14.1, 6.0 Hz, 1 H, H′-C-1), 3.81-3.91 (m, 1 H, H-C-3), 4.08 (ddd, *J* = 7.2, 6.0, 4.2 Hz, 1 H, H-C-2). ^{13}C NMR ($CDCl_3$) δ 13.9 (C-6), 16.4 (S<u>C</u>H$_3$), 18.9 (C-5), 34.4 (C-4), 37.8 (C-1), 66.3 (C-2), 73.4 (C-3). HRMS (Solarix Mass Spectrometer, ESI): calcd. for $C_7H_{15}ClNaOS$ $[M + Na]^+$ 205.0424, found 205.0424.

2′-Methylthio-3′-chloropropyl *trans*-2-butenoate 2l: light yellow oil; 1.66 g, 80% yield. ^{1}H NMR ($CDCl_3$) δ 1.82 (dd, *J* = 6.9, 1.8 Hz, 3 H, H-C-4), 2.13 (s, 3 H, SC<u>H</u>$_3$), 3.01 (m, 1 H, H-C-2′), 3.66 (dd, *J* = 11.1, 7.5 Hz, 1 H, H-C-3′), 3.72 (dd, *J* = 11.1, 5.1 Hz, 1 H, H′-C-3′), 4.28 (dd, *J* = 11.7, 6.0 Hz, 1 H, H-C-1′), 4.38 (dd, *J* = 11.7, 5.1 Hz, 1 H, H′-C-1′), 5.79 (dq, *J* = 15.3, 1.8 Hz, 1 H, H-C-2), 6.94 (dq, *J* = 15.3, 6.9 Hz, 1 H, H-C-3). ^{13}C NMR ($CDCl_3$) δ 14.6 (S<u>C</u>H$_3$), 18.0 (C-4), 44.2 (C-3′), 47.3 (C-2′), 62.9 (C-1′), 122.1 (C-3), 145.6 (C-2), 165.9 (C-1(C=O)). HRMS (Solarix Mass Spectrometer, ESI): calcd. for $C_8H_{13}ClNaO_2S$ $[M + Na]^+$ 231.0217, found 231.0218.

1-Hydoxy-2-methylthio-1-phenylethane **3a**: colorless oil; 1.46 g, 87% yield. ^{1}H NMR ($CDCl_3$) δ 2.13 (s, 3 H, SC<u>H</u>$_3$), 2.73 (dd, *J* = 13.8, 9.6 Hz, 1 H, H-C-2), 2.87 (dd,

J = 13.8, 3.6 Hz, 1 H, H′-C-2), 2.97 (br, 1 H, OH), 4.77 (dd, *J* = 9.6, 3.6 Hz, 1 H, H-C-1), 7.28~7.40 (m, 5 H, H(phenyl)); ^{13}C NMR ($CDCl_3$) δ 15.4 (S<u>C</u>H$_3$), 44.1 (C-2), 71.1 (C-1), 125.8 (C-2′and C-6′ (phenyl)), 127.8 (C-4′(phenyl)), 128.5 (C-3′and C-5′ (phenyl)), 142.4 (C-1′(phenyl)). The ^{1}H NMR data were matched with those reported in lit.[3]

1-Hydroxy-2-methylthiocyclohexane **3b**: colorless oil; 1.36 g, 93% yield. ^{1}H NMR ($CDCl_3$) δ 1.20~1.58 (m, 4 H, H-C-4 and H-C-5), 1.65~1.82 (m, 2 H, H-C-3 and H-C-6), 2.00~2.22 (m, 2 H, H′-C-3 and H′-C-6), 2.07 (s, 3 H, SC<u>H</u>$_3$), 2.34 (ddd, *J* = 12.0, 9.9, 3.9 Hz, 1 H, H-C-2), 2.98 (br, 1 H, OH), 3.34 (td, *J* = 9.9, 4.5 Hz, 1 H, H-C-1); ^{13}C NMR ($CDCl_3$) δ 11.3 (S<u>C</u>H$_3$), 24.5 (C-5), 26.2 (C-4), 31.5 (C-3), 33.8 (C-6), 53.2 (C-2), 71.0 (C-1). The ^{1}H NMR data were matched with those reported in lit.[4]

1-Hydroxy-2-methylthiocycloheptane **3c**: light yellow oil; 1.30 g, 81% yield. ^{1}H NMR ($CDCl_3$) δ 1.38-1.85 and 1.90-2.10 (m, 10 H, H-C-3~7), 2.07 (s, 3 H, SC<u>H</u>$_3$), 2.46 (td, *J* = 9.3, 3.0 Hz, 1 H, H-C-2), 2.60-2.87 (br, 1 H, OH), 3.47 (ddd, *J* = 9.3, 8.1, 3.6 Hz, 1 H, H-C-1); ^{13}C NMR ($CDCl_3$) δ 11.9 (S<u>C</u>H$_3$), 21.8 (C-6), 25.9 (C-4), 27.3 (C-5), 31.0 (C-3), 33.6 (C-7), 55.9 (C-2), 73.6 (C-1). HRMS (Q Exactive GC, EI): calcd. for $C_8H_{16}OS$ M^+ 160.0916, found 160.0917.

1-Acetoxy-2-methylthiocyclohexane **4**

The mixture of 1-hydroxy-2-methylthiocyclohexane **3b** (0.7 g, 4.8 mmol), acetic anhydride (1.0 g, 9.6 mmol) in pyridine (15 mL) was stirred for 3 h at room temperature. Then the solution was poured into diluted HCl and extracted with CH_2Cl_2 (3 × 20 mL). The combined organic extracts were dried with anhydrous $MgSO_4$, filtered and concentrated. Purification by column chromatography on silica gel (petroleum/EtOAc, 15: 1) gave 1-acetoxy-2-methylthiocyclohexane **4** (0.87 g, 96%) as a colorless oil. ^{1}H NMR ($CDCl_3$) δ 1.15~1.56 (m, 4 H, H-C-4 and H-C-5), 1.60~1.80 (m, 2 H, H-C-3 and H-C-6), 1.96~2.14 (m, 2 H, H′-C-3 and H′-C-6), 2.07 (s, 3 H, C<u>H</u>$_3$(Ac)), 2.09 (s, 3 H, SC<u>H</u>$_3$), 2.55 (ddd, *J* = 10.5, 9.3, 3.9 Hz, 1 H, H-C-2), 4.73 (td, *J* = 9.3, 4.5 Hz, 1 H, H-C-1); ^{13}C NMR ($CDCl_3$) δ 13.4 (S<u>C</u>H$_3$), 21.3 (<u>C</u>H$_3$(Ac)), 23.7 (C-5), 25.1 (C-4), 31.1 (C-3), 31.3 (C-6), 48.4 (C-2), 74.4 (C-1), 170.4 (C=O). The ^{1}H NMR data were matched with those reported in lit.[5]

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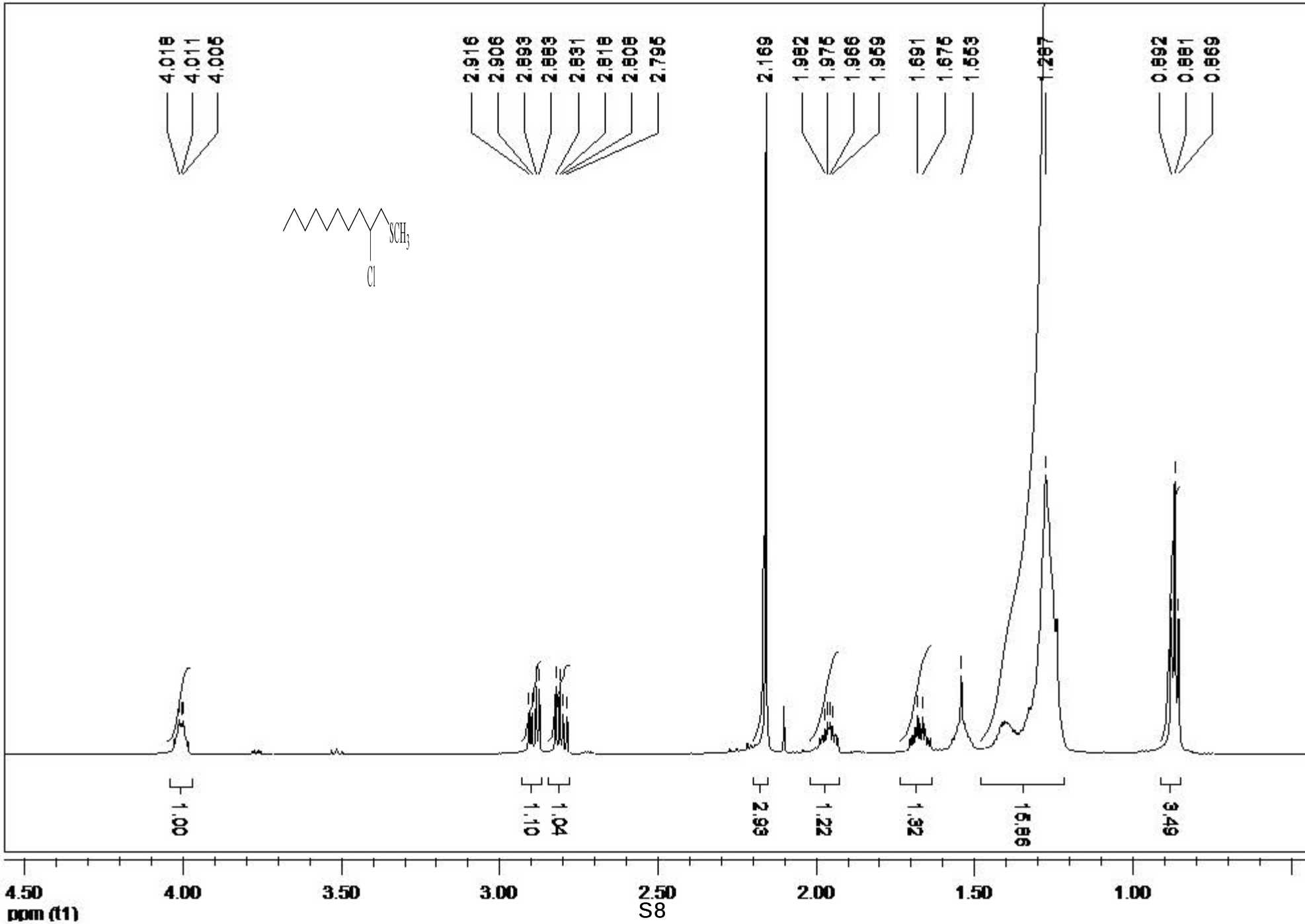
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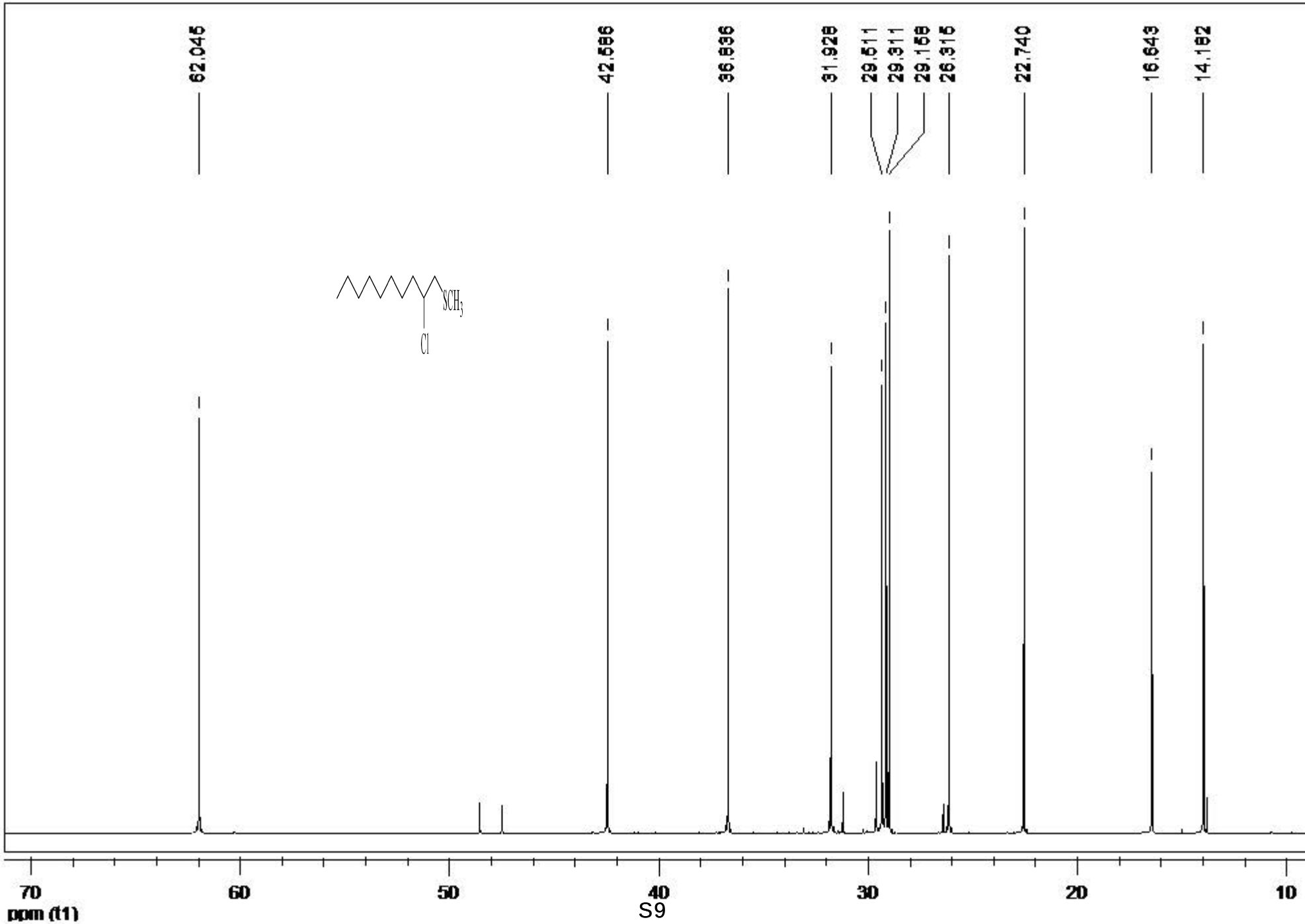
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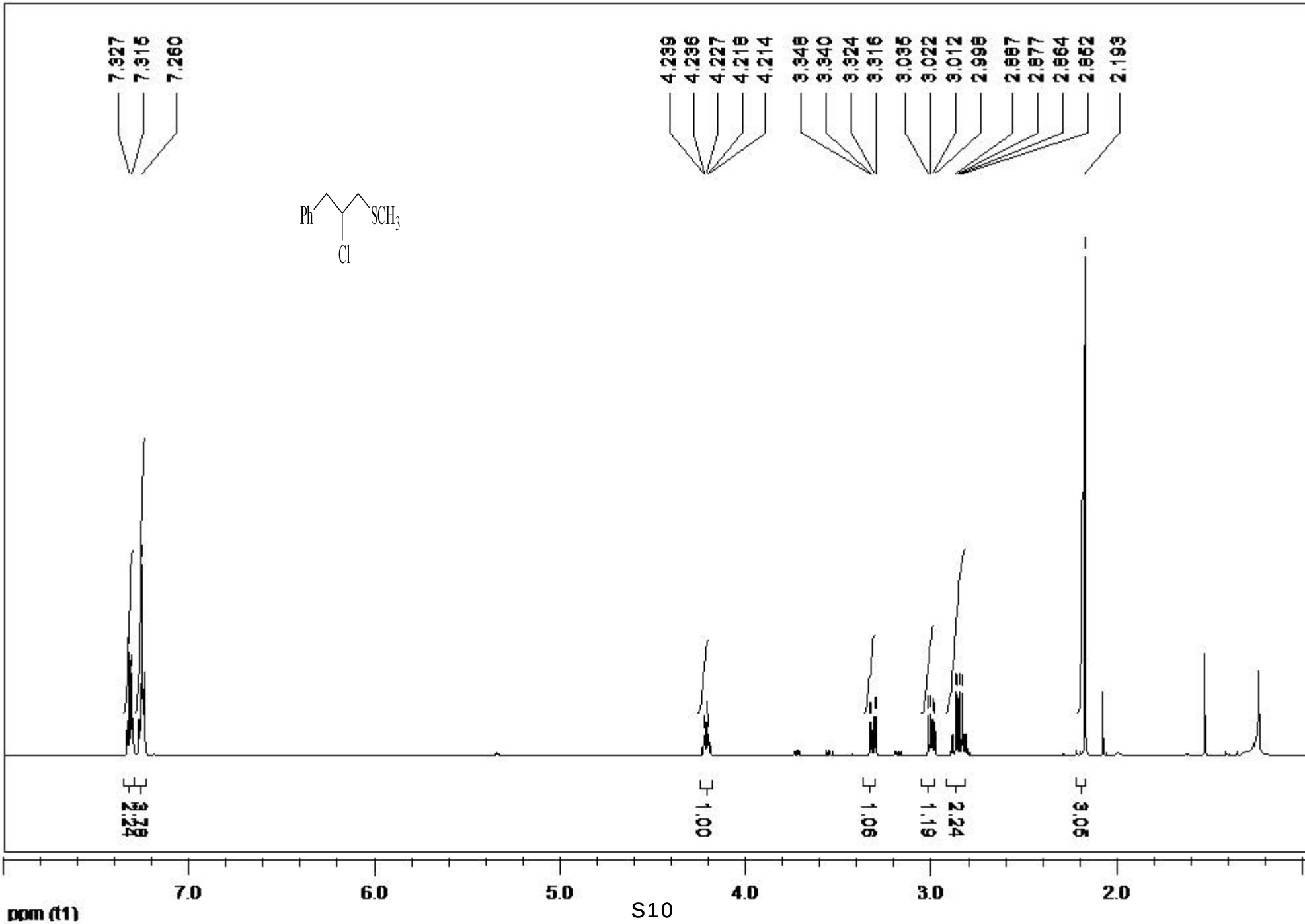
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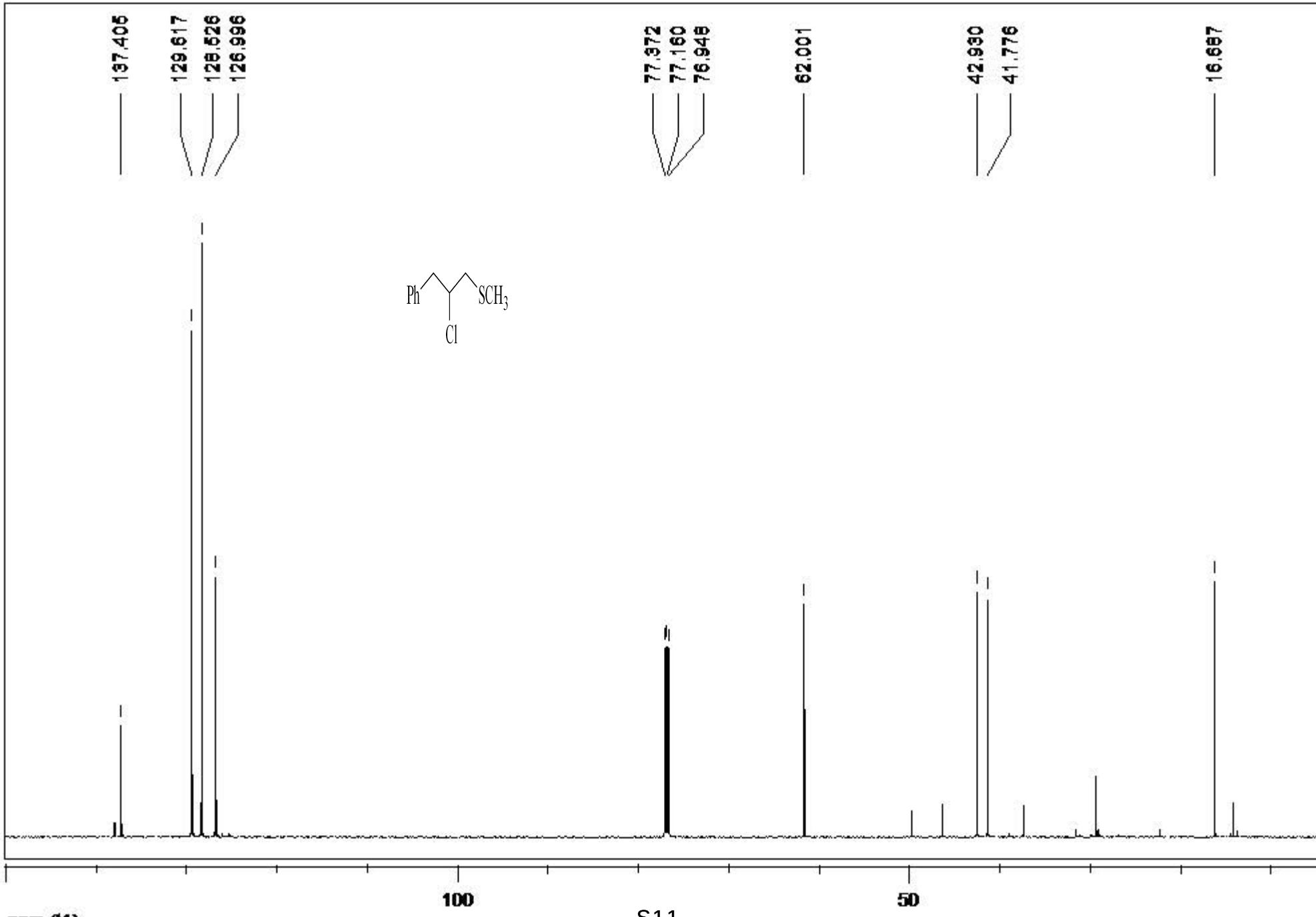
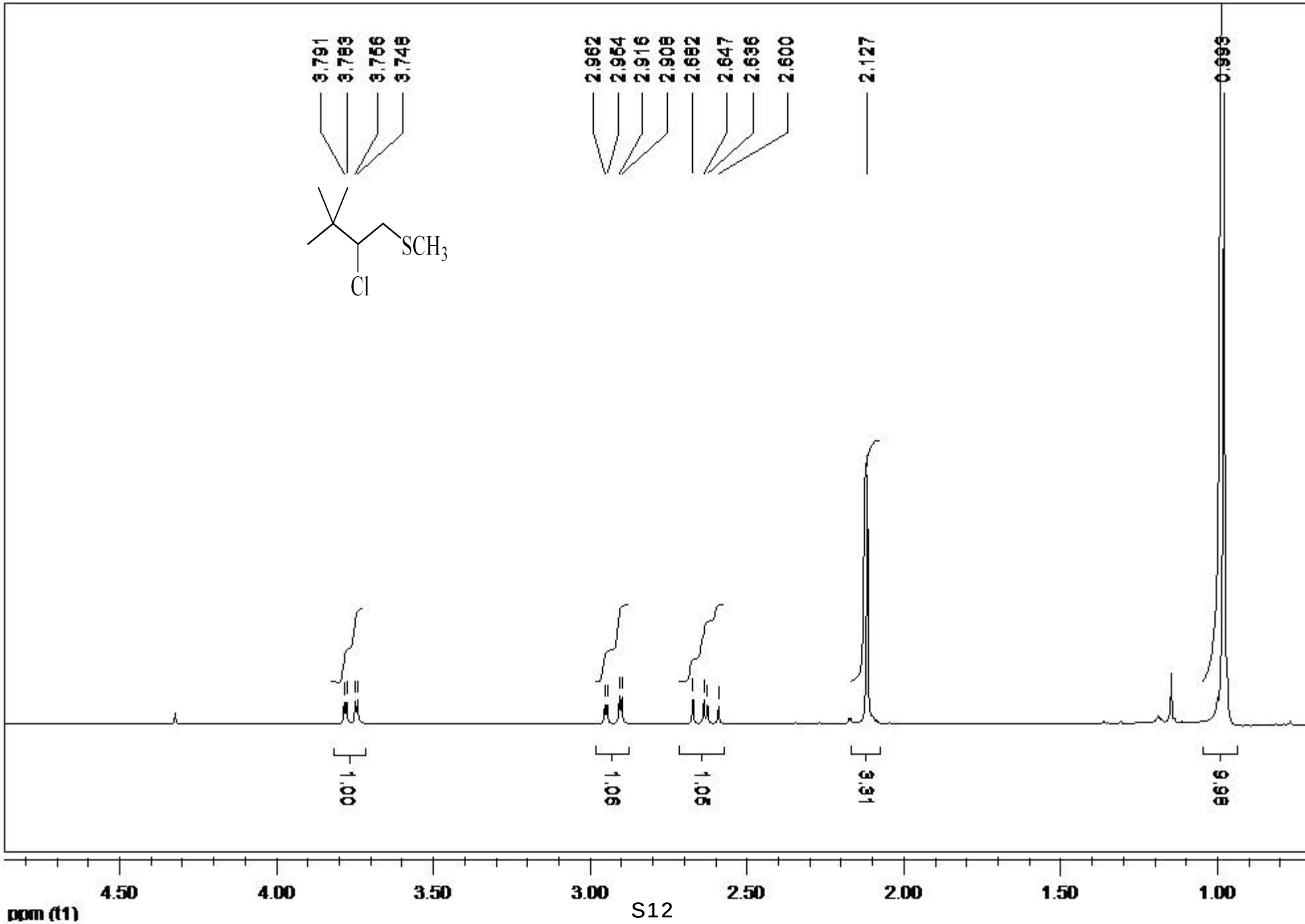
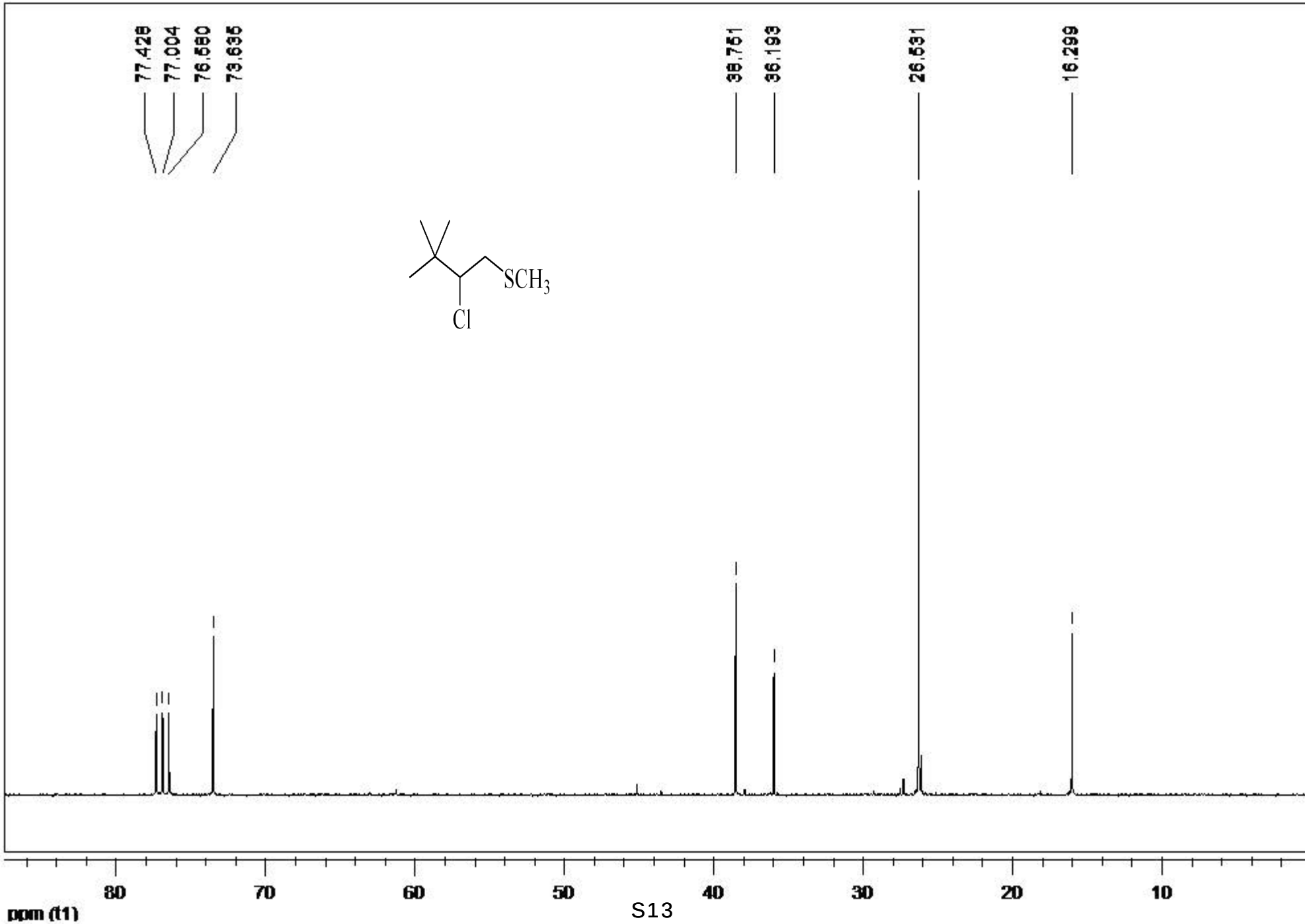
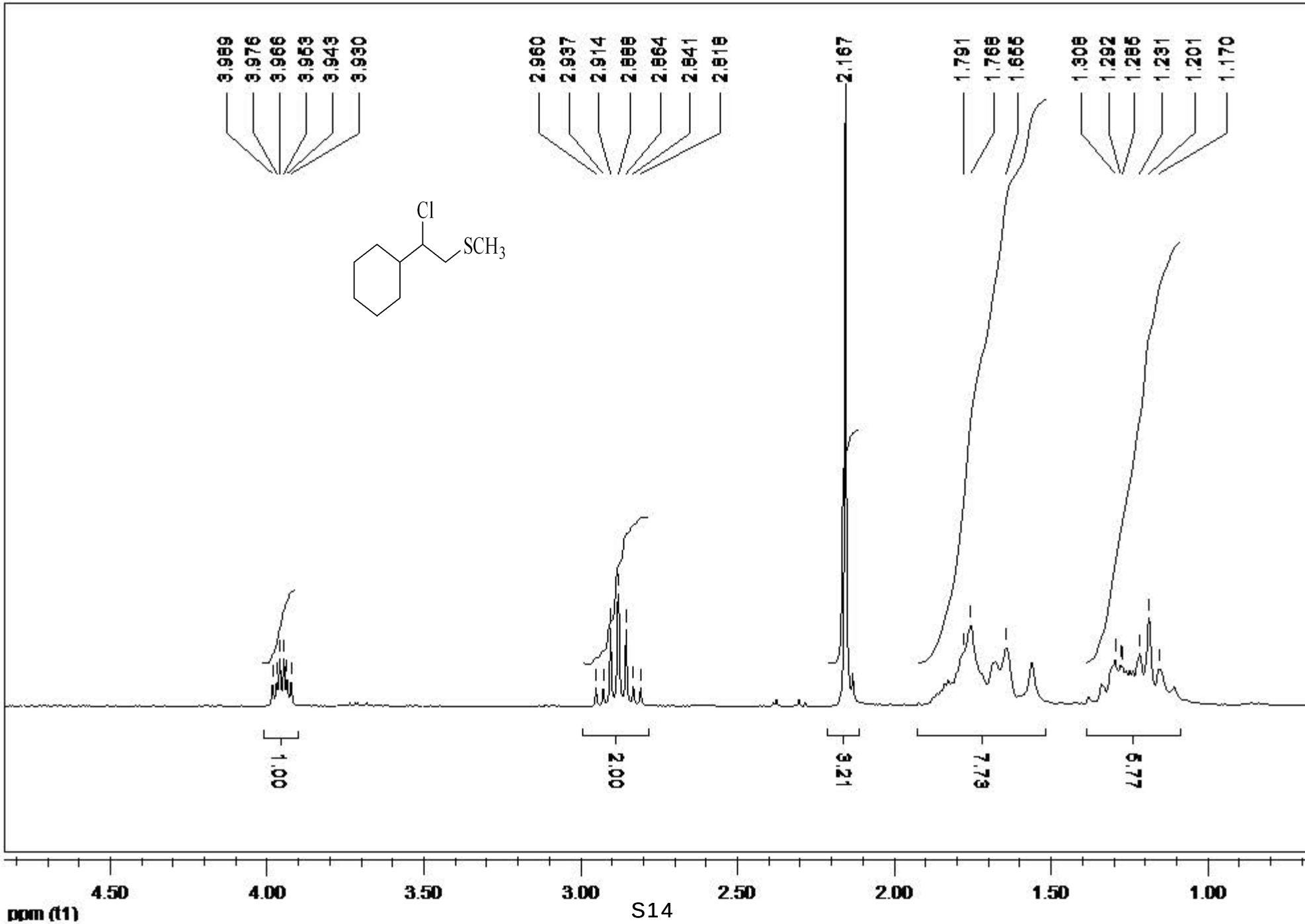
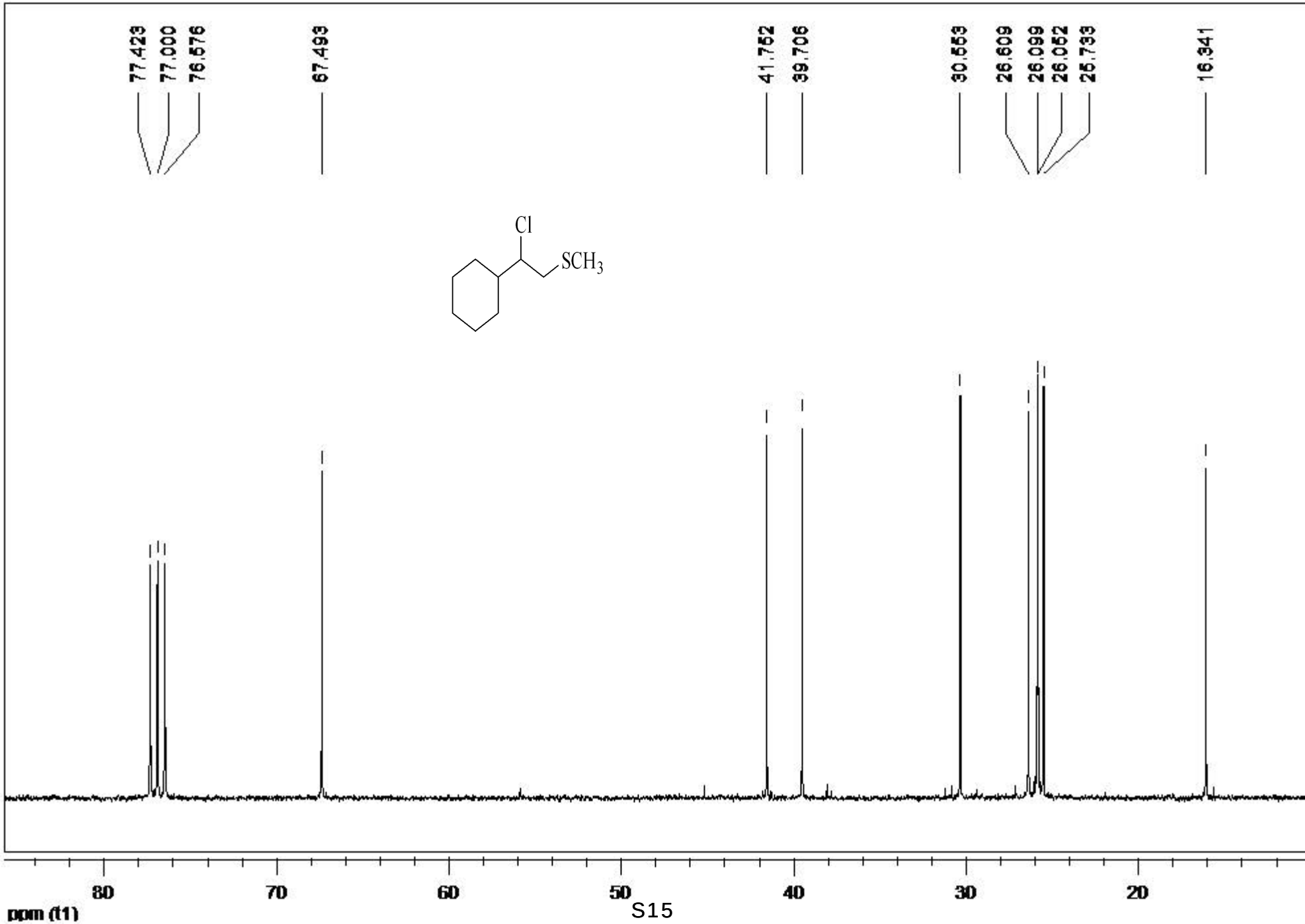
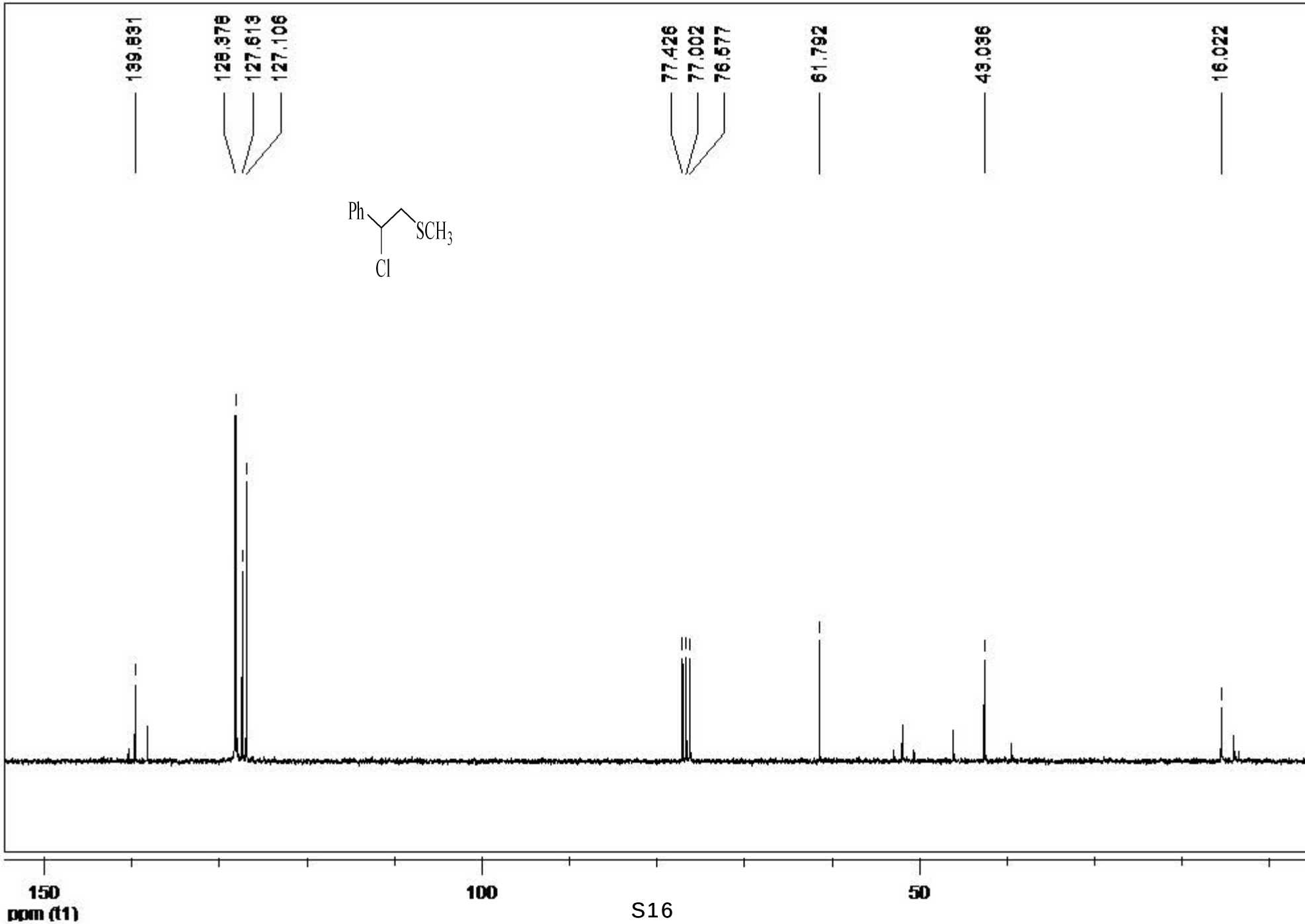

137.405
129.617
128.526
126.996
77.372
77.160
76.948
62.001
42.930
41.776
16.687
Ph
SCH3
Cl
100
50
ppm (t1)

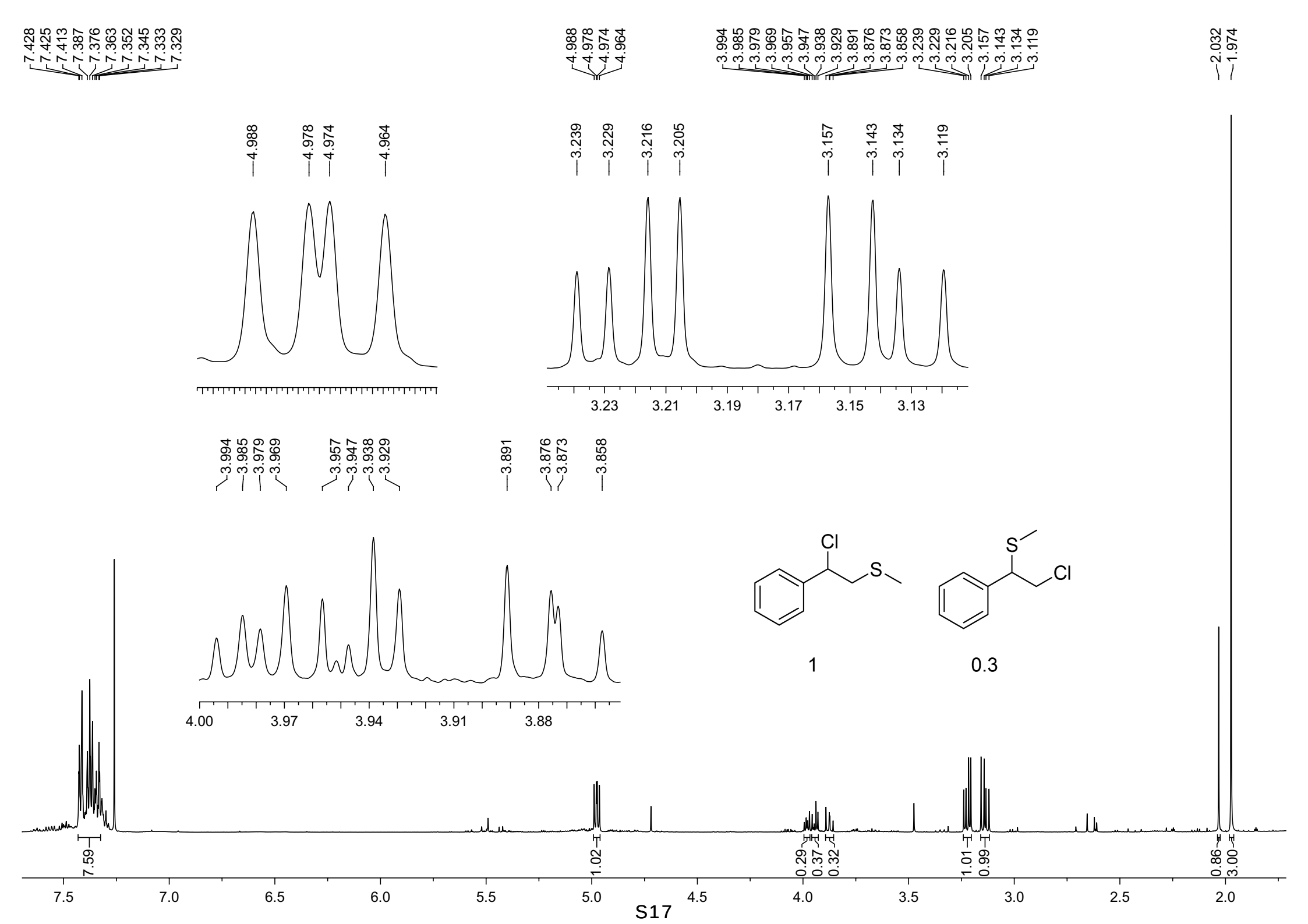

3.791
3.783
3.756
3.748
2.962
2.954
2.916
2.908
2.682
2.647
2.636
2.600
2.127
0.993
SCH3
Cl
1.00
1.06
1.06
3.31
9.96
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
ppm (t1)

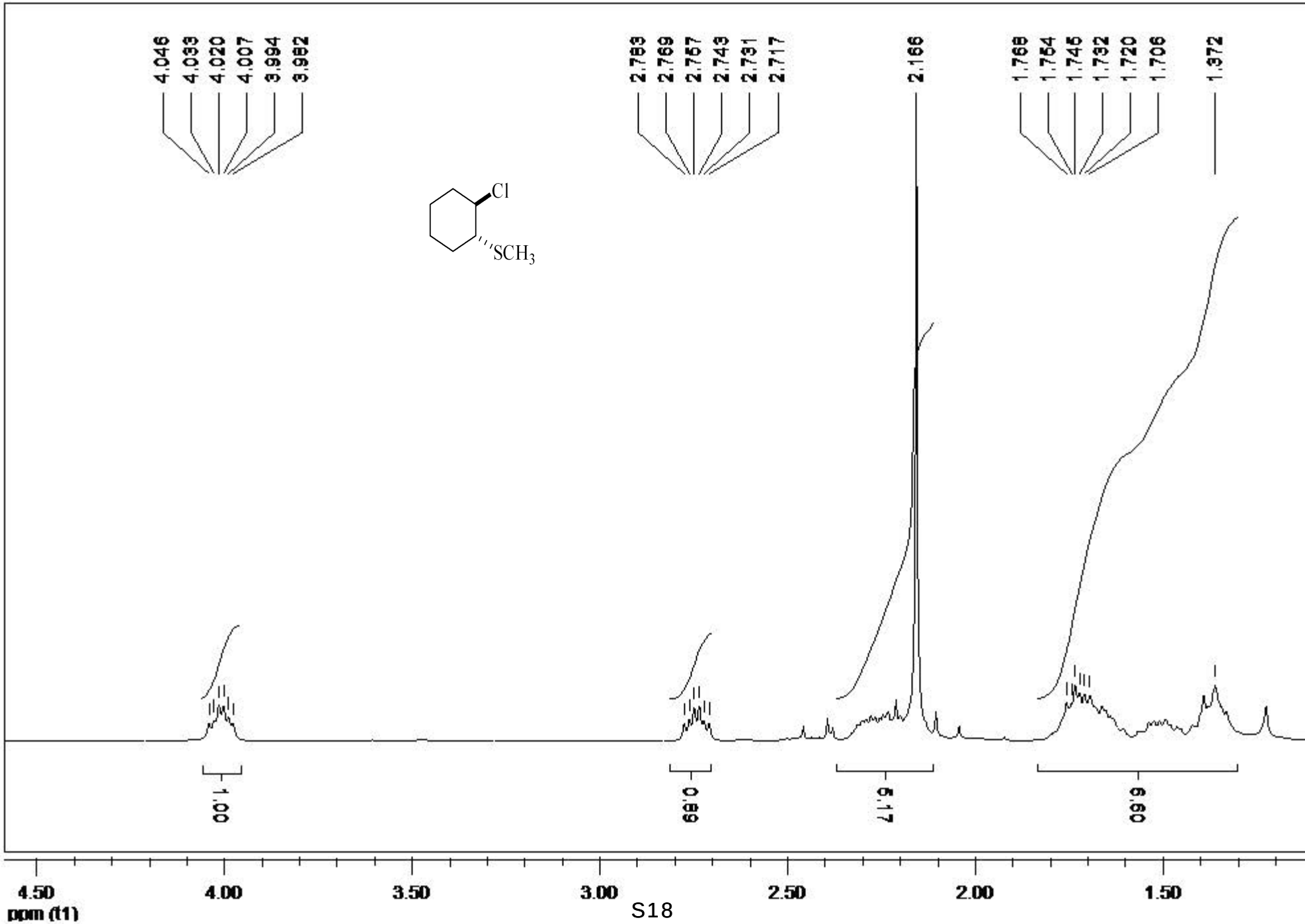

77.428
77.004
76.580
73.635
38.751
36.193
26.531
16.299
SCH3
Cl
80
70
60
50
40
30
20
10
ppm (t1)

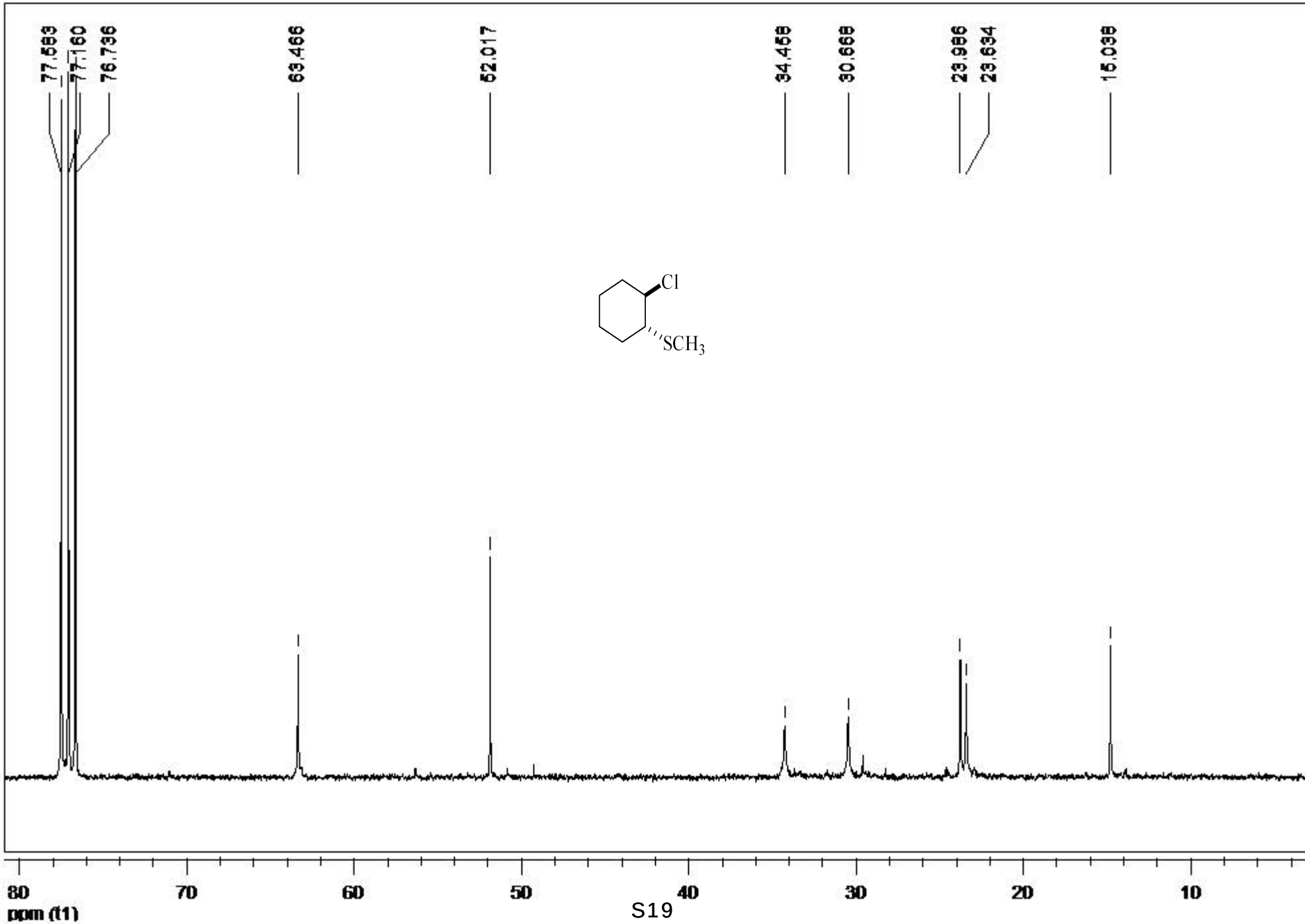

3.989
3.976
3.966
3.953
3.943
3.930
2.960
2.937
2.914
2.888
2.864
2.841
2.818
2.167
1.791
1.768
1.655
1.308
1.292
1.285
1.231
1.201
1.170
Cl
SCH3
1.00
2.00
3.21
7.79
6.77
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
ppm (t1)

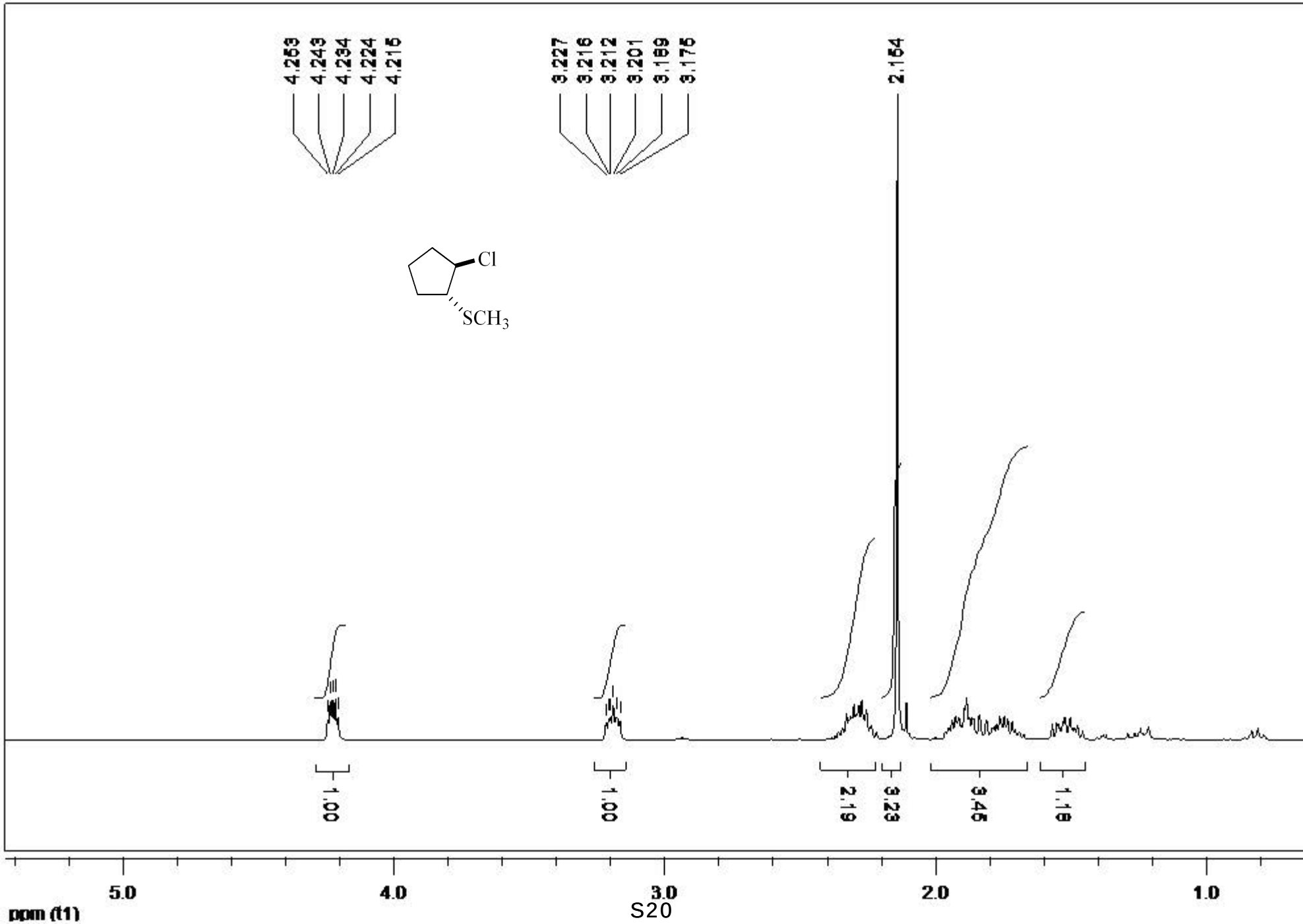

77.423
77.000
76.576
67.493
41.752
39.706
30.553
26.609
26.099
26.052
25.733
16.341
Cl
SCH3
80
70
60
50
40
30
20
ppm (t1)

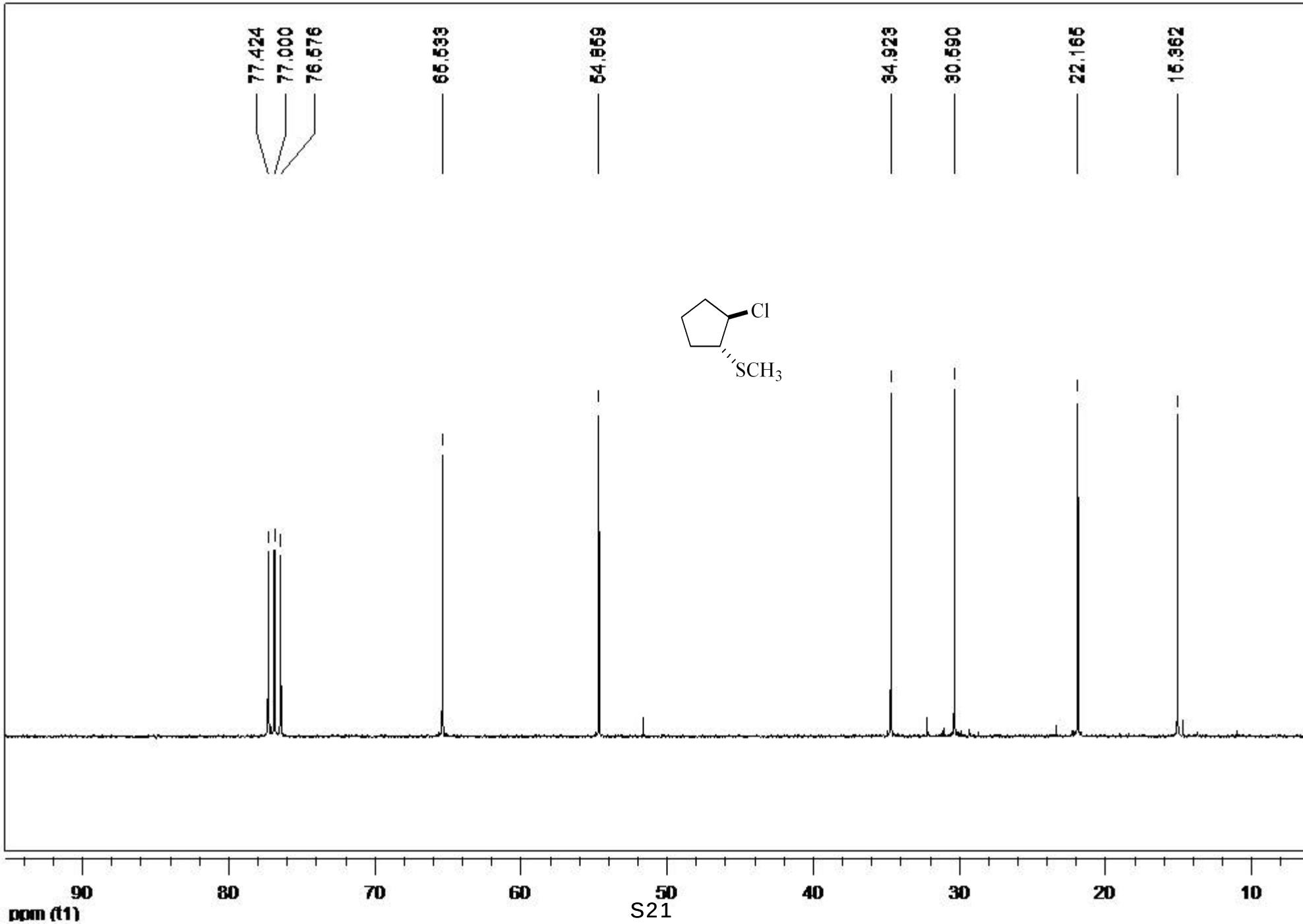

139.631
128.378
127.619
127.106
77.426
77.002
76.577
61.792
43.036
16.022
Ph
SCH3
Cl
150
100
50
ppm (t1)

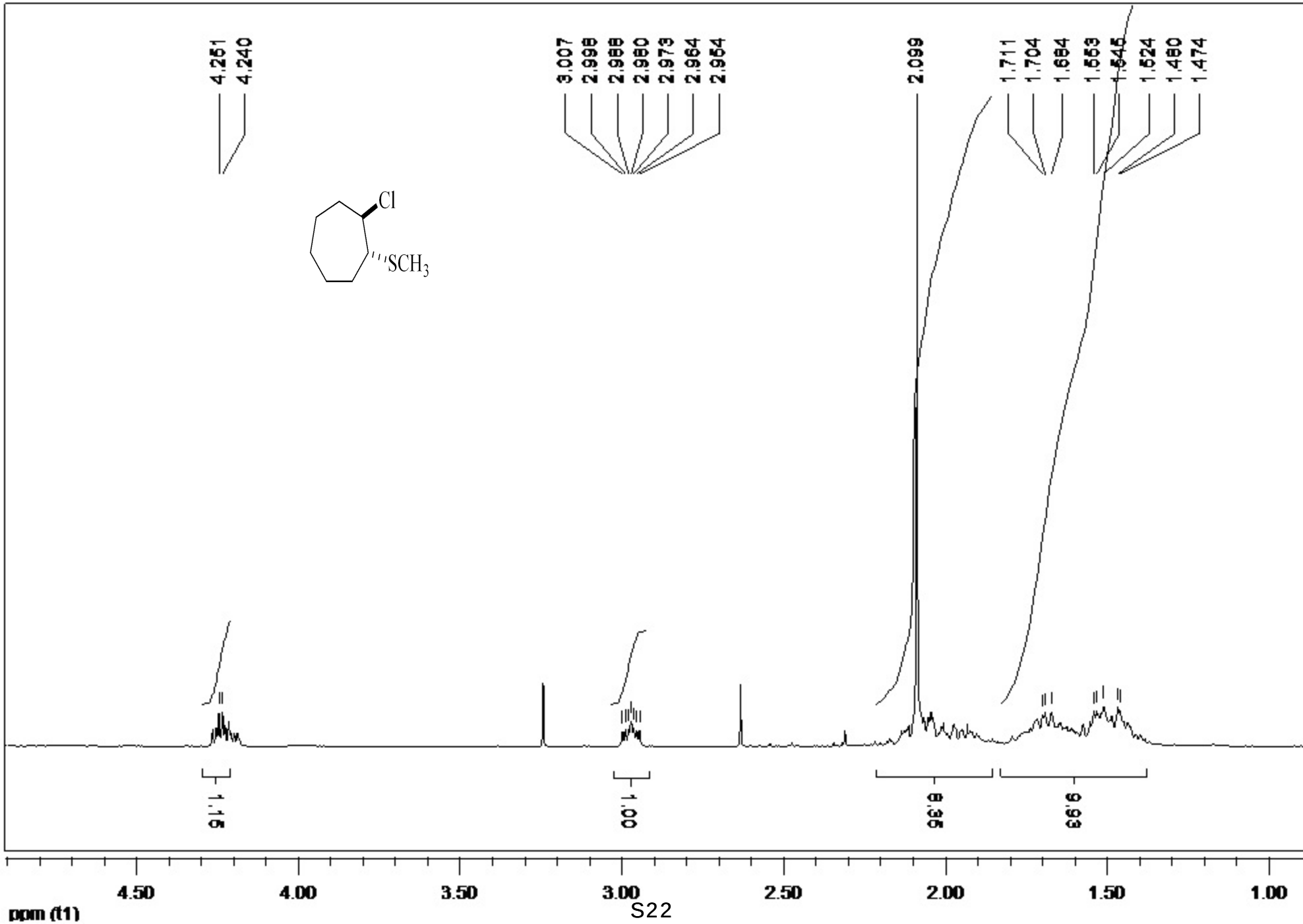

7.428
7.425
7.413
7.387
7.376
7.363
7.352
7.345
7.333
7.329
4.988
4.978
4.974
4.964
3.994
3.985
3.979
3.969
3.957
3.947
3.938
3.929
3.891
3.876
3.873
3.858
3.239
3.229
3.216
3.205
3.157
3.143
3.134
3.119
2.032
1.974
3.23
3.21
3.19
3.17
3.15
3.13
4.00
3.97
3.94
3.91
3.88
Cl
S
S
Cl
1
0.3
7.59
1.02
0.29
0.37
0.32
1.01
0.99
0.86
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0

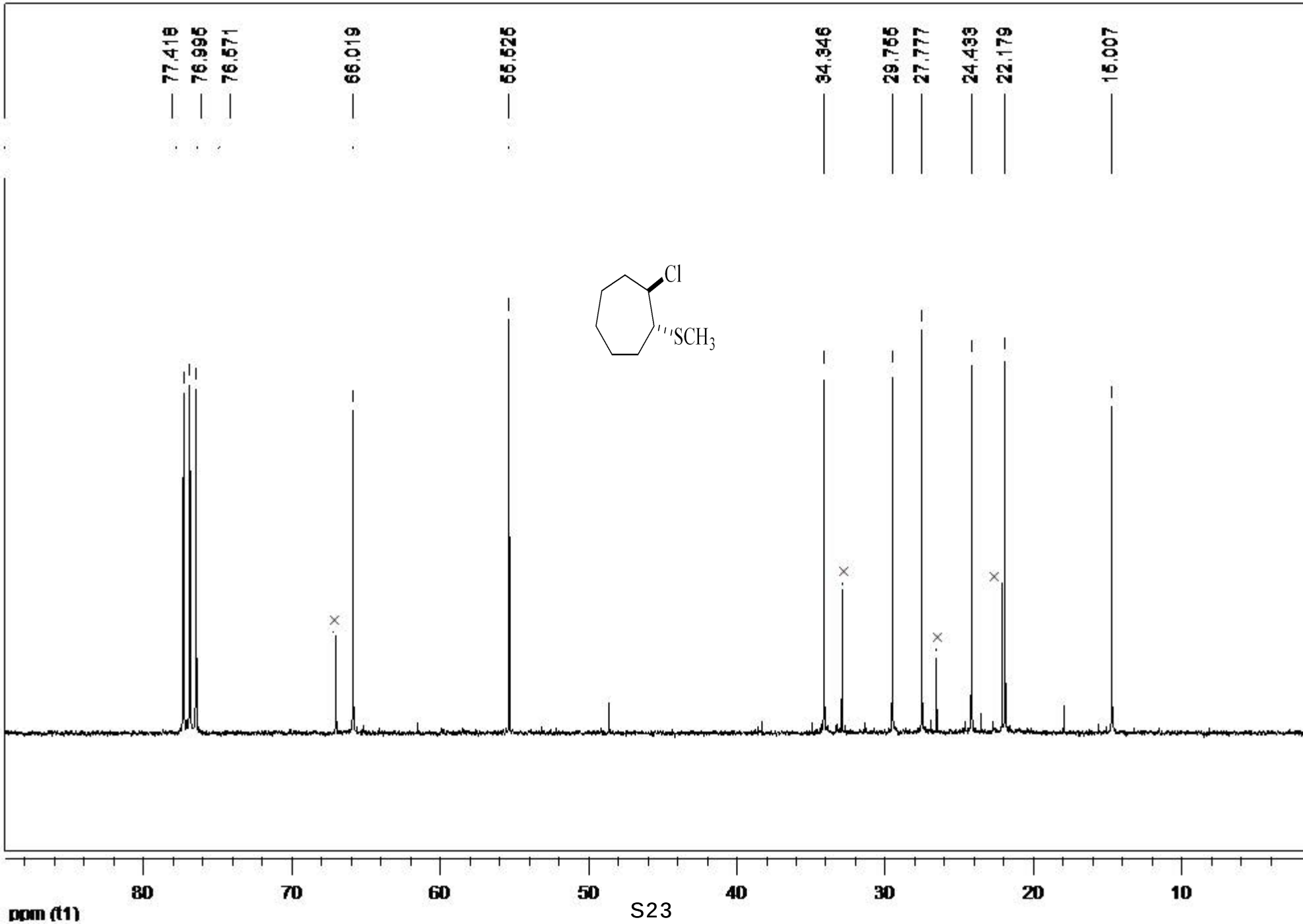

4.046
4.033
4.020
4.007
3.994
3.982
2.783
2.769
2.757
2.743
2.731
2.717
2.166
1.768
1.754
1.745
1.732
1.720
1.706
1.372
Cl
SCH3
1.00
0.89
6.17
6.60
4.50
4.00
3.50
3.00
2.50
2.00
1.50
ppm (t1)

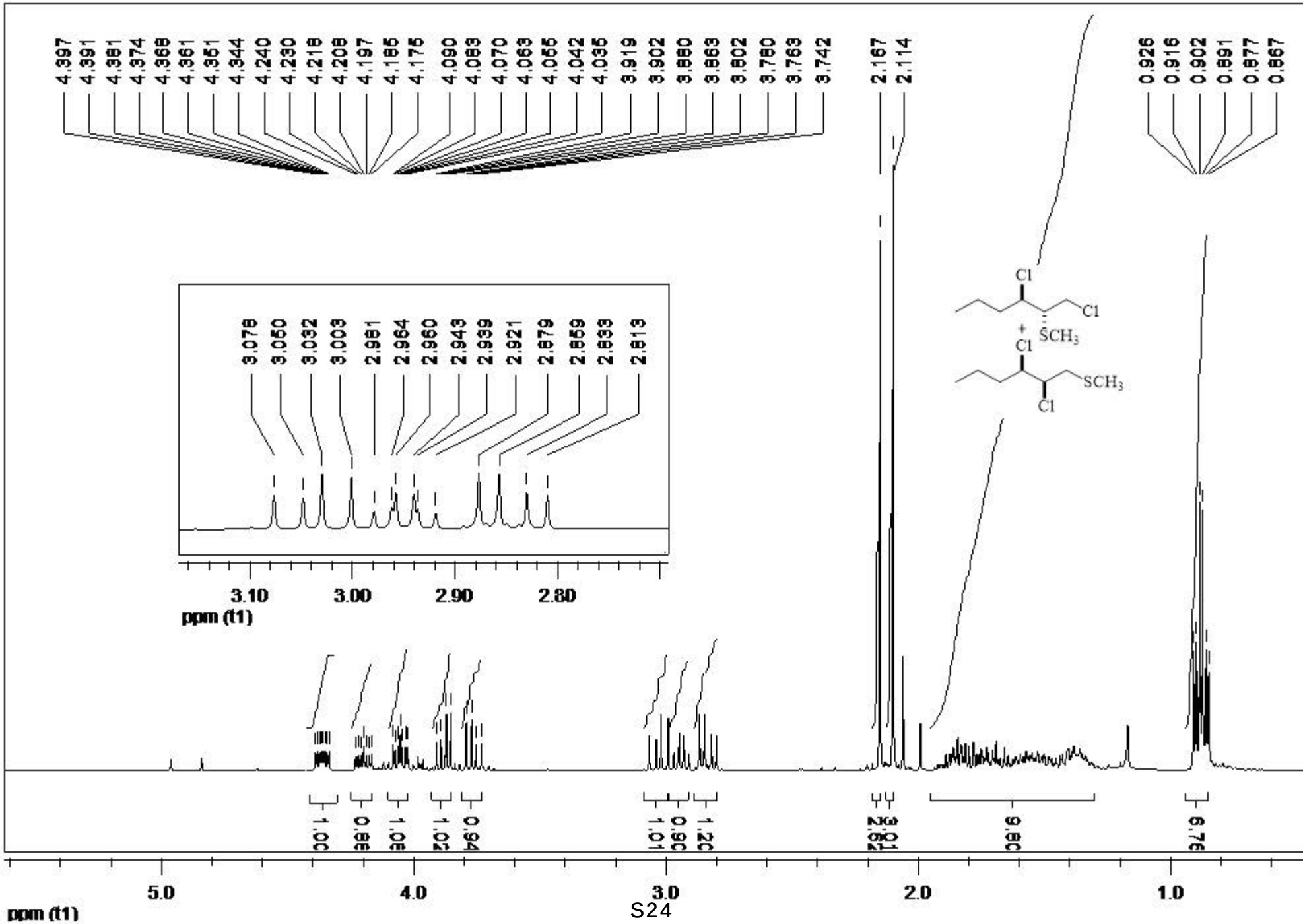

77.583
77.160
76.736
63.466
52.017
34.458
30.668
23.986
23.634
15.038
Cl
SCH3
80
70
60
50
40
30
20
10
ppm (t1)

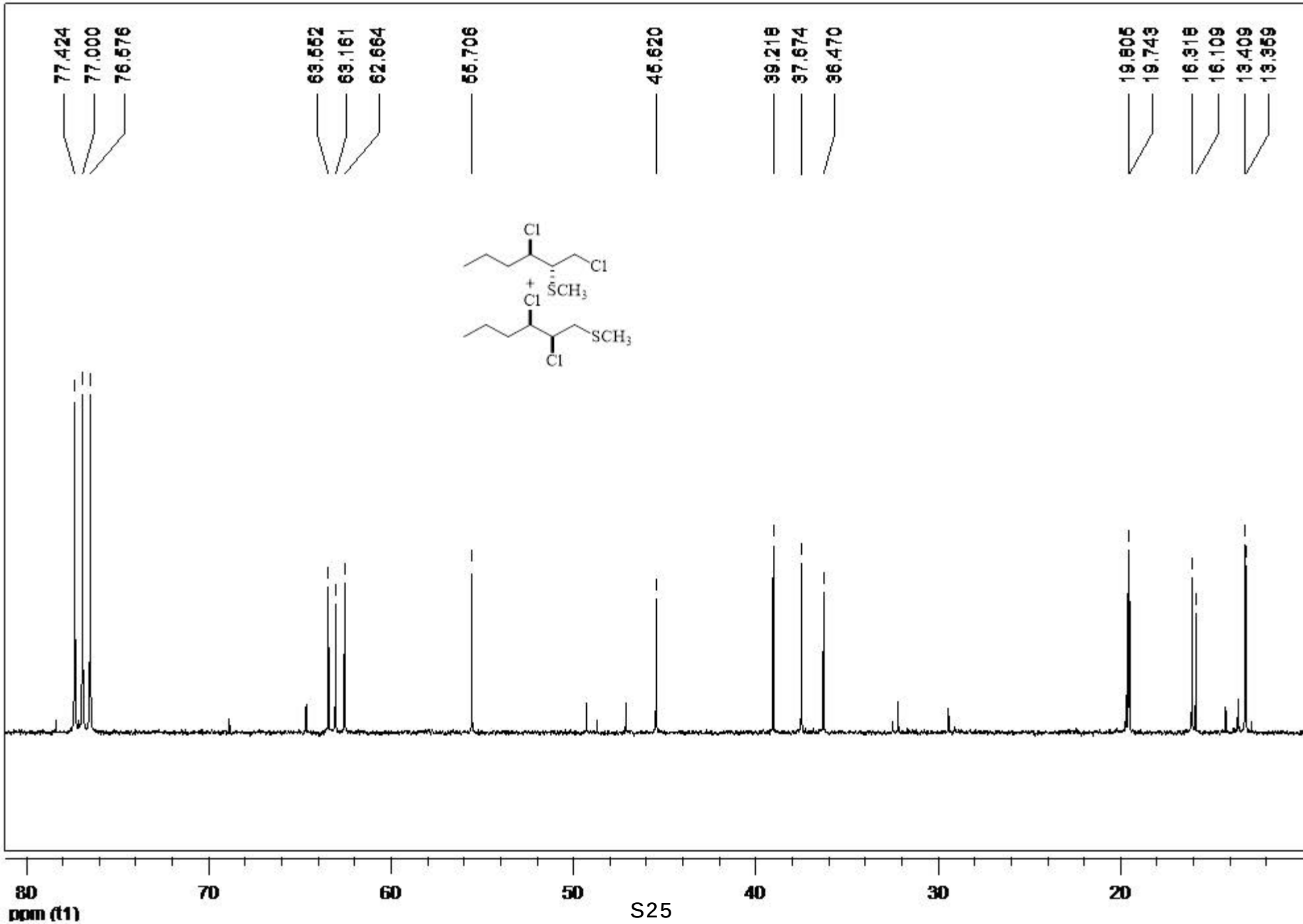

4.253
4.243
4.234
4.224
4.215
3.227
3.216
3.212
3.201
3.189
3.175
2.154
Cl
SCH3
1.00
1.00
2.19
3.23
6.45
1.18
5.0
4.0
3.0
2.0
1.0
ppm (t1)

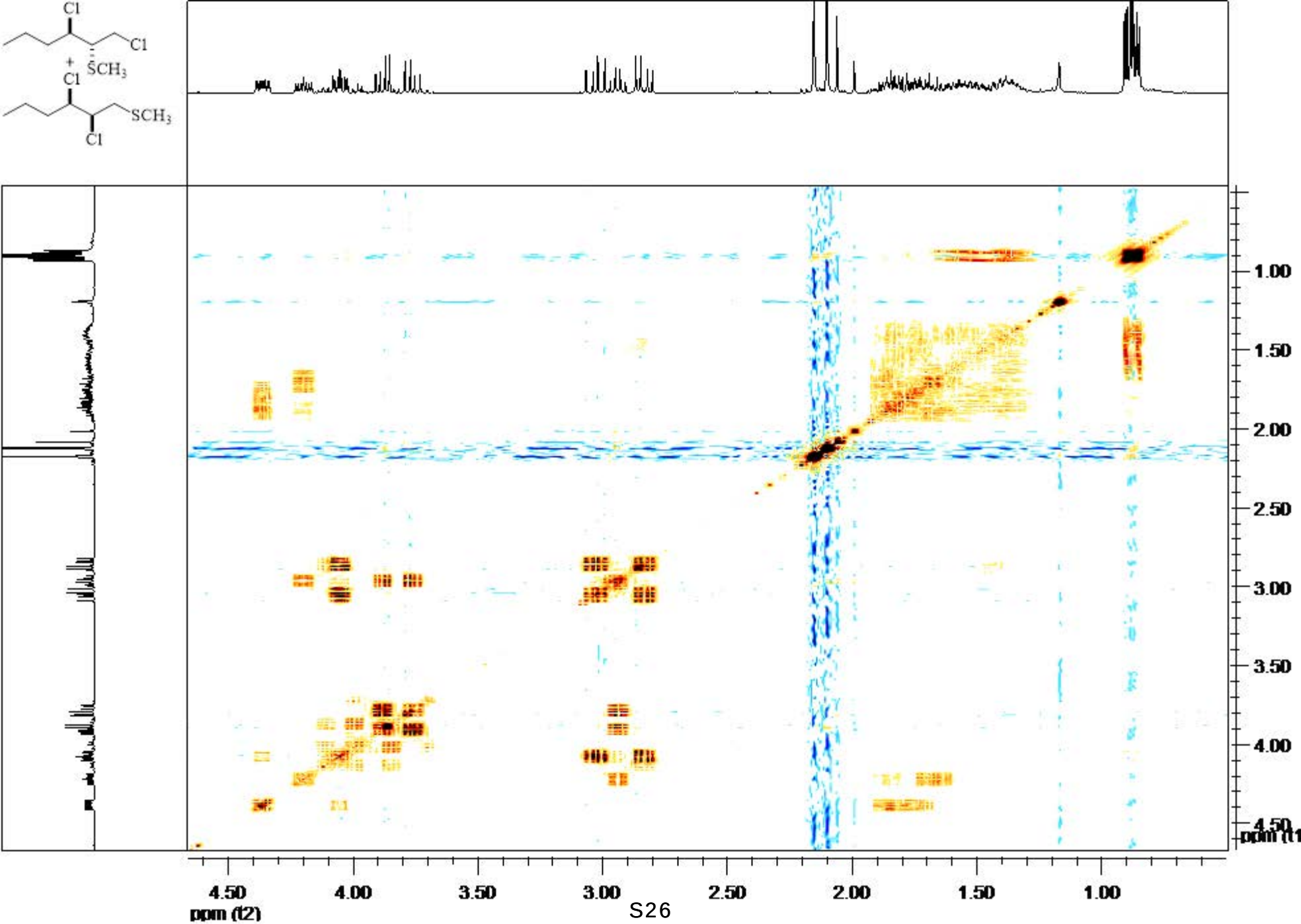

77.424
77.000
76.576
65.533
54.859
34.923
30.590
22.165
15.362
Cl
SCH3
90
80
70
60
50
40
30
20
10
ppm (t1)

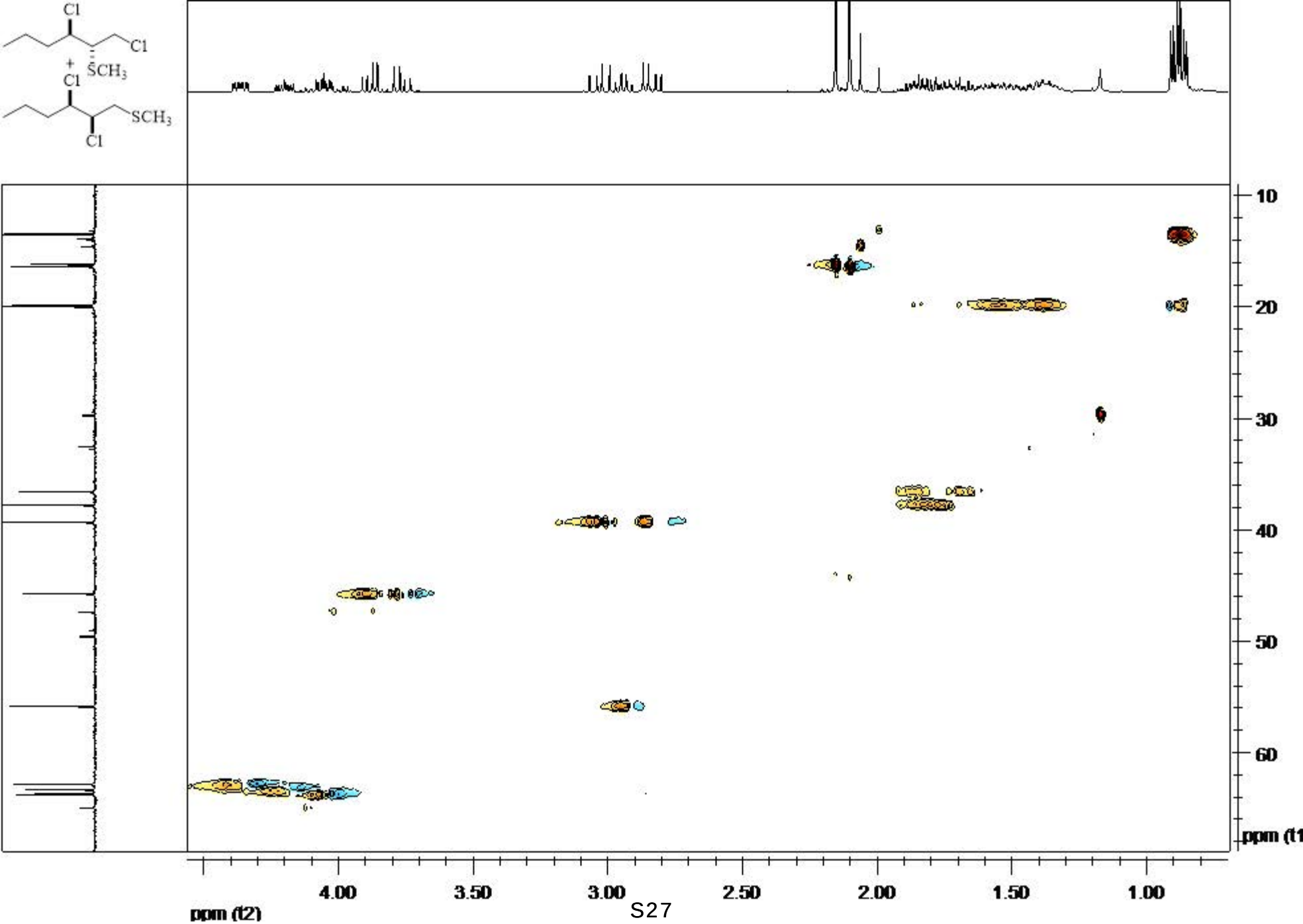

4.251
4.240
3.007
2.998
2.988
2.980
2.973
2.964
2.954
2.099
1.711
1.704
1.684
1.553
1.545
1.524
1.480
1.474
Cl
SCH3
1.16
1.00
8.36
9.99
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
ppm (t1)

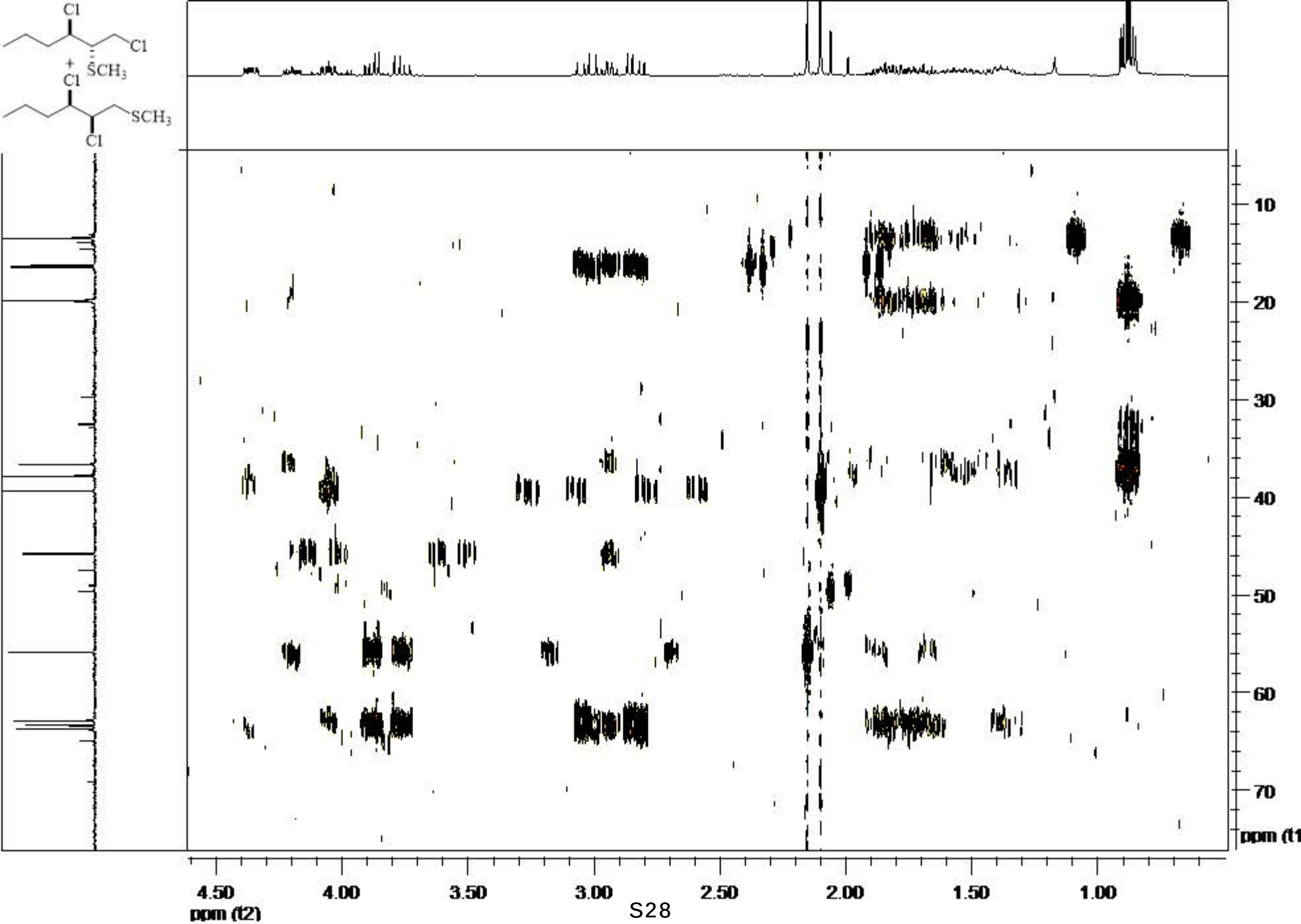

77.418
76.995
76.571
66.019
55.525
34.946
29.765
27.777
24.433
22.179
15.007
Cl
SCH3
80
70
60
50
40
30
20
10
ppm (t1)

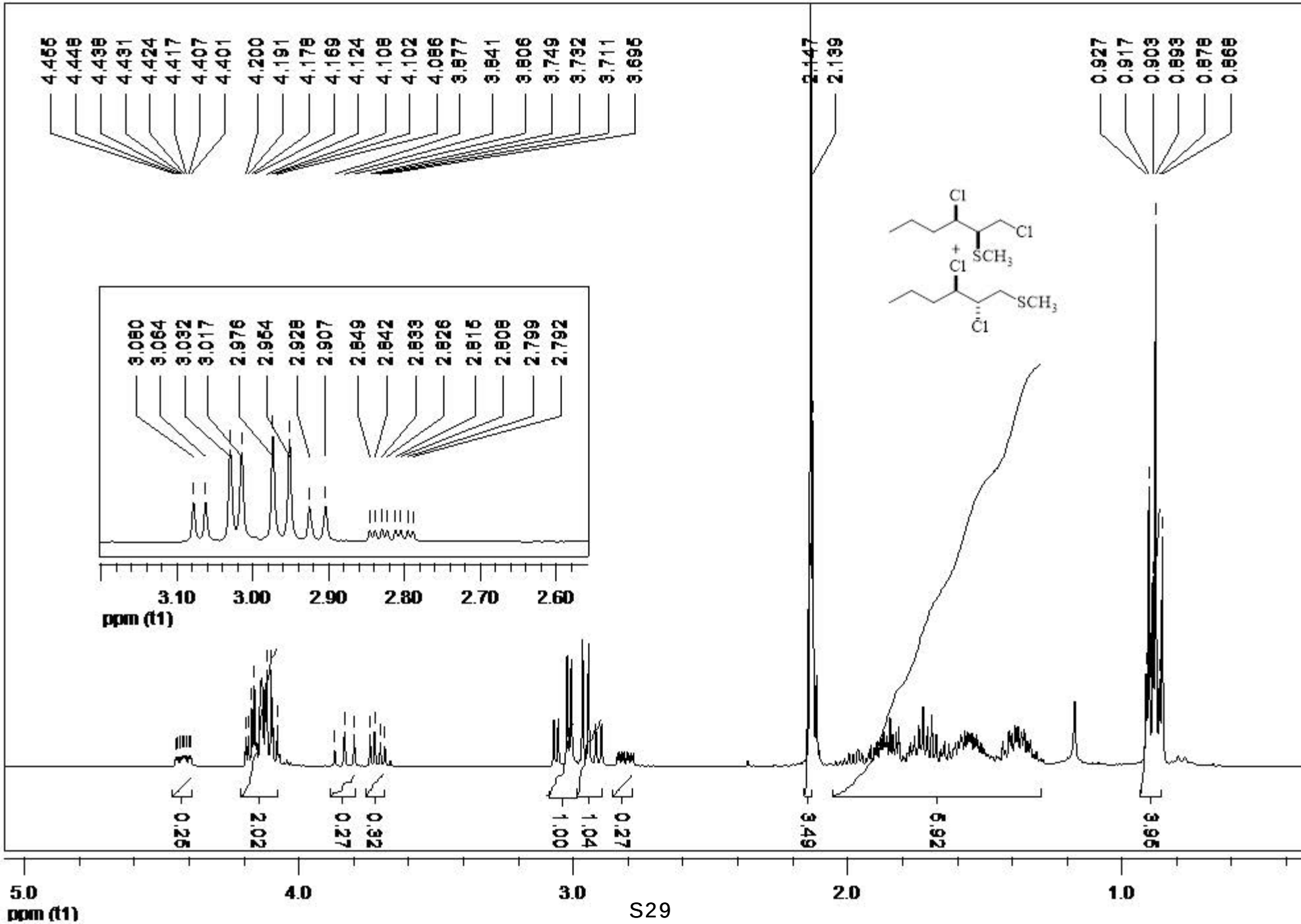

4.397
4.391
4.381
4.374
4.368
4.361
4.351
4.344
4.240
4.230
4.218
4.208
4.197
4.185
4.175
4.090
4.083
4.070
4.063
4.055
4.042
4.035
3.919
3.902
3.880
3.863
3.802
3.780
3.763
3.742
2.167
2.114
0.926
0.916
0.902
0.891
0.877
0.867
3.078
3.050
3.032
3.003
2.981
2.964
2.960
2.943
2.939
2.921
2.879
2.859
2.833
2.813
Cl
Cl
+ SCH3
Cl
SCH3
Cl
3.10
3.00
2.90
2.80
ppm (t1)
1.00
0.88
1.06
1.02
0.94
1.01
0.90
1.20
2.82
3.01
9.80
6.76
5.0
4.0
3.0
2.0
1.0
ppm (t1)

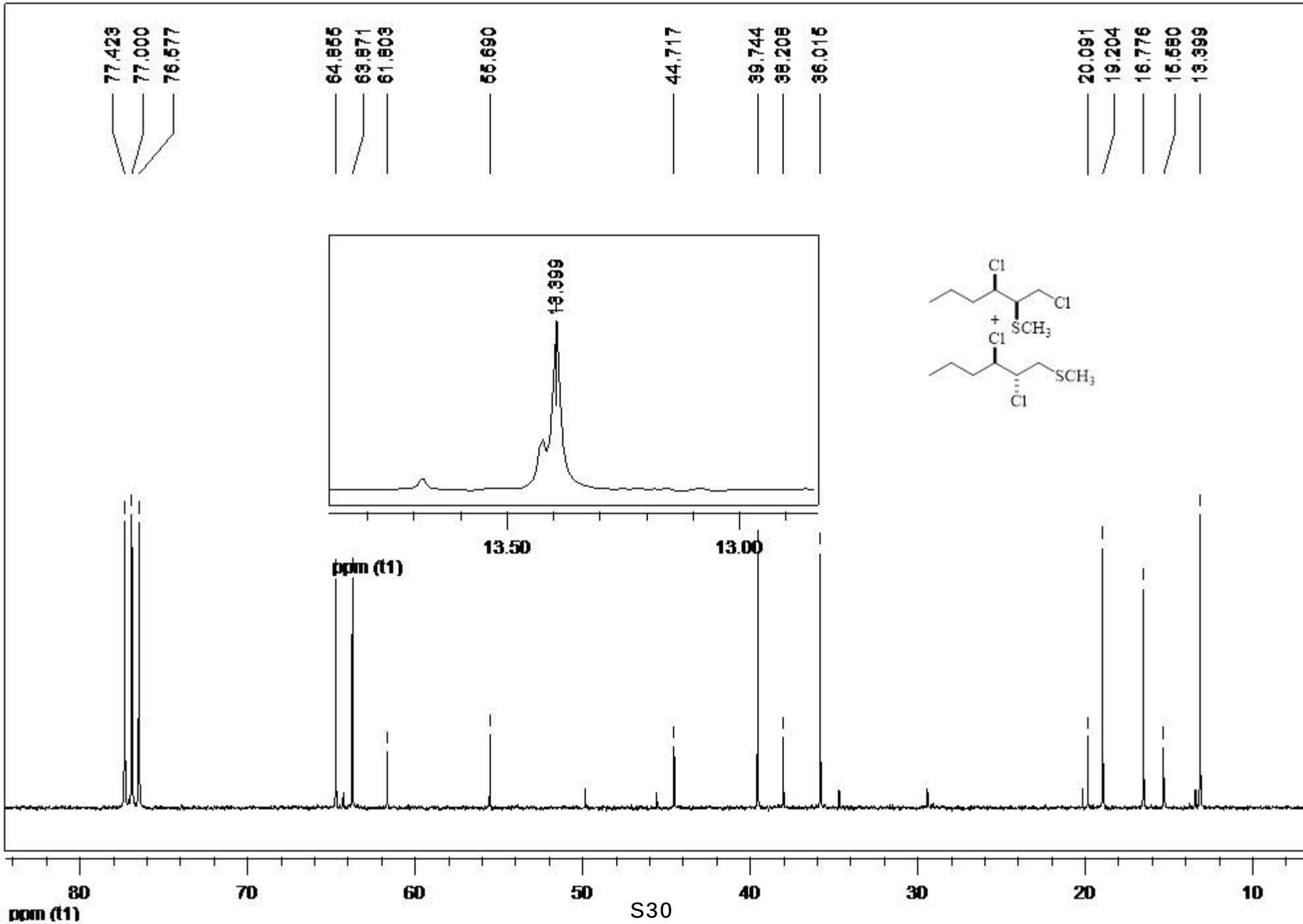

77.424
77.000
76.576
63.552
63.161
62.664
55.706
45.620
39.218
37.674
36.470
19.805
19.743
16.318
16.109
13.409
13.359
Cl
Cl
+ SCH3
Cl
SCH3
Cl
80
70
60
50
40
30
20
ppm (t1)

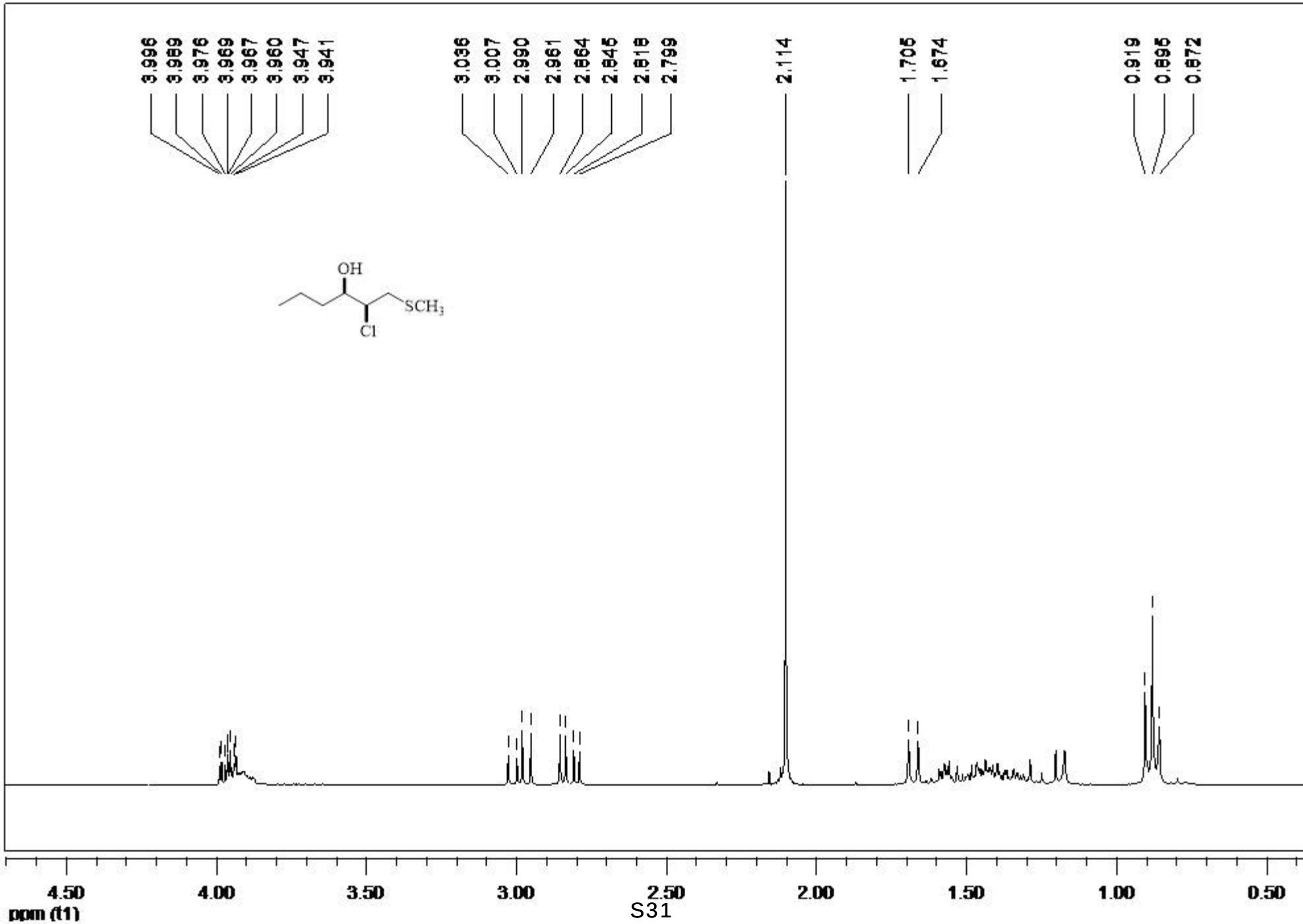

Cl
Cl
+
SCH3
Cl
SCH3
Cl
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
ppm (t1)
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
ppm (t2)

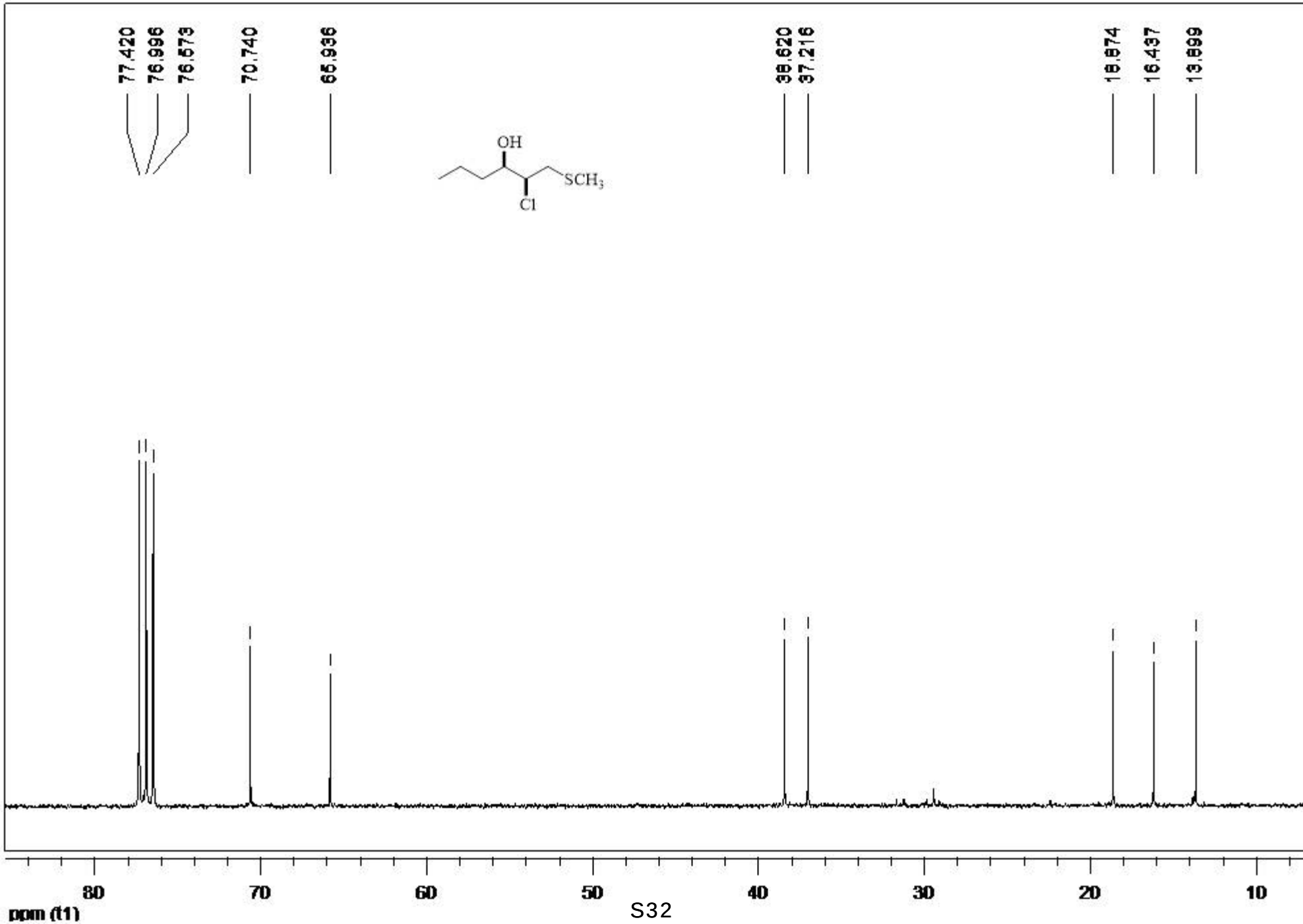

Cl
Cl
+
SCH3
Cl
SCH3
Cl
10
20
30
40
50
60
ppm (t1
4.00
3.50
3.00
2.50
2.00
1.50
1.00
ppm (t2)

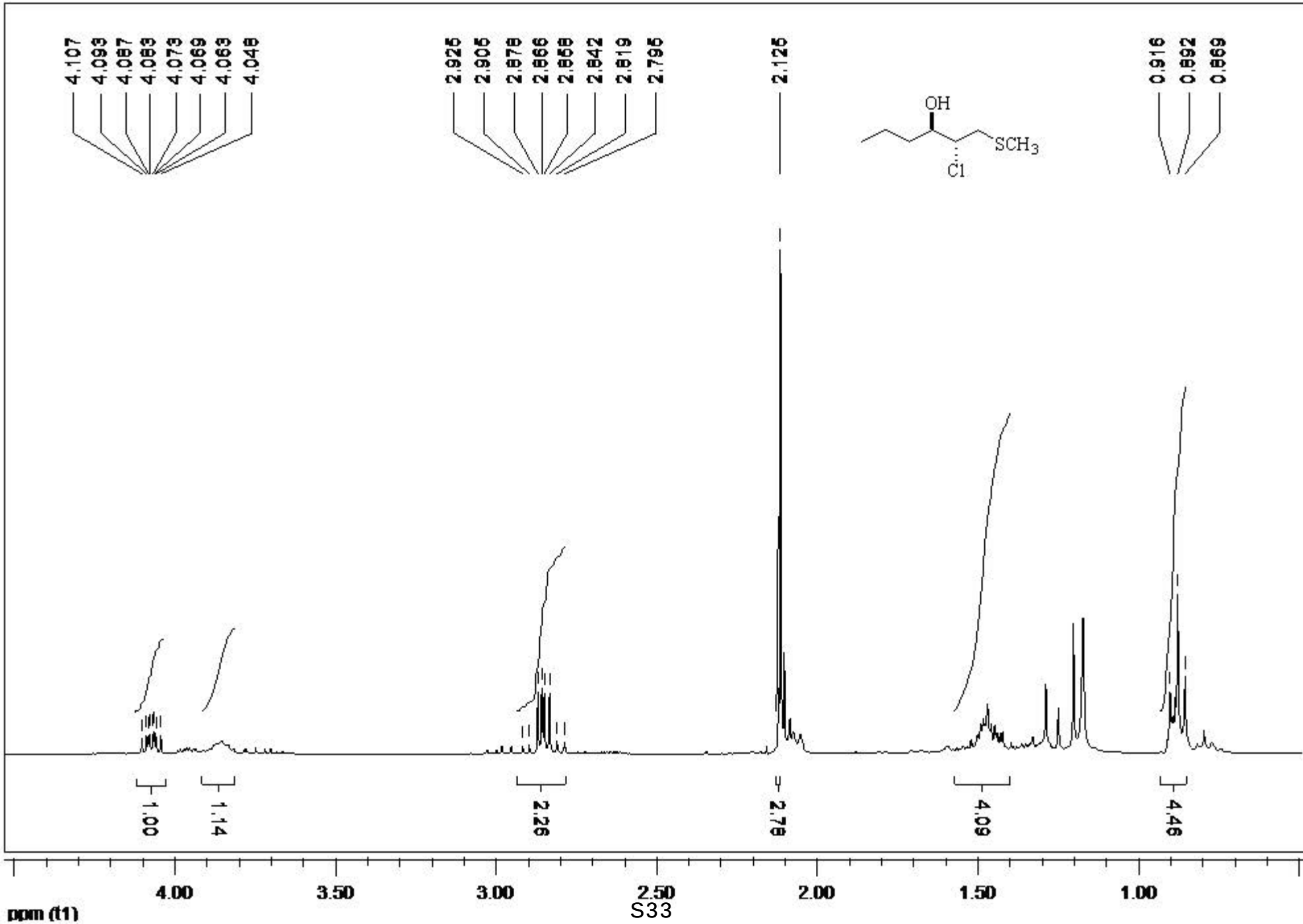

Cl
Cl
+
SCH3
Cl
SCH3
Cl
10
20
30
40
50
60
70
ppm (t1
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
ppm (t2)

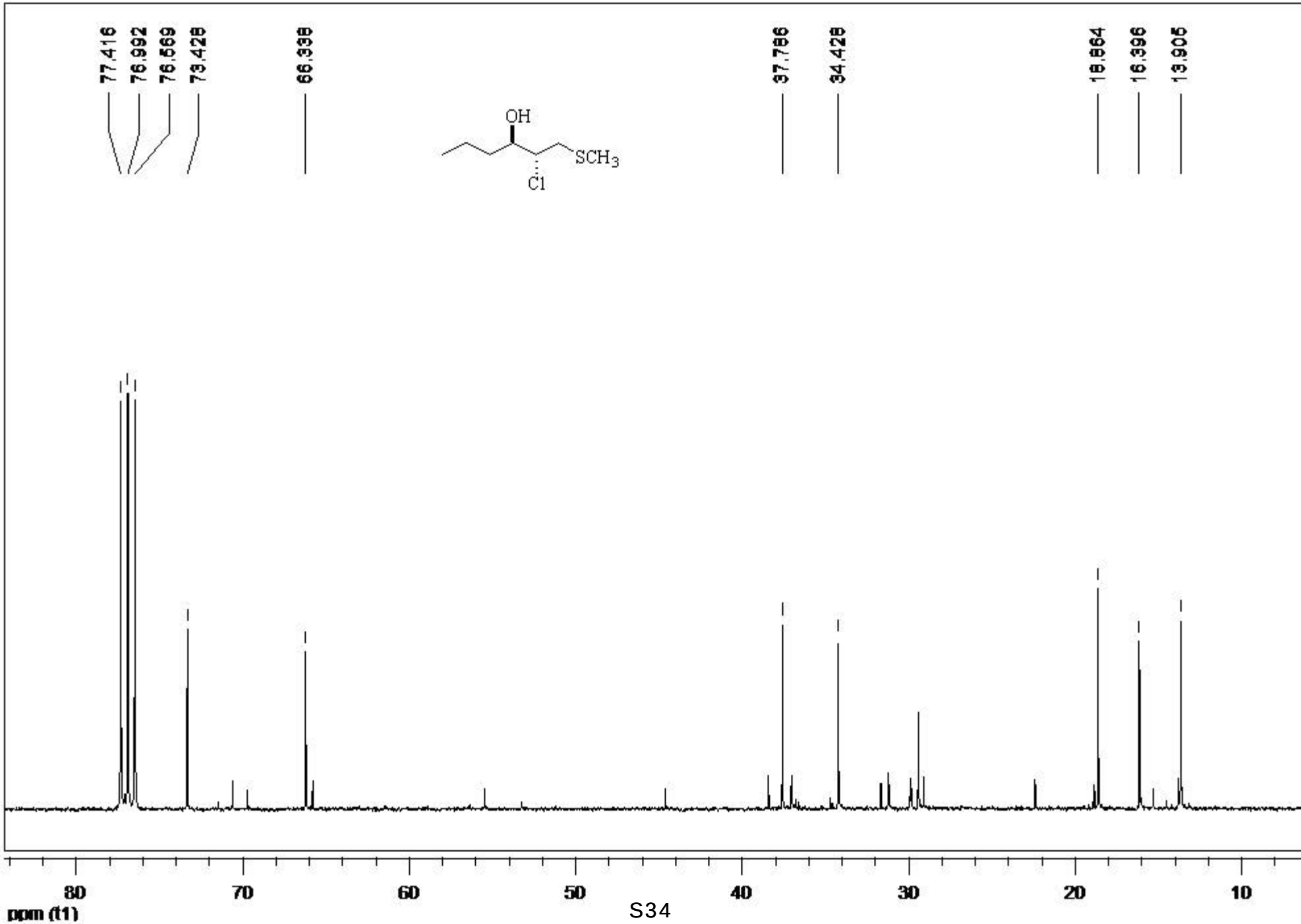

4.455
4.448
4.438
4.431
4.424
4.417
4.407
4.401
4.200
4.191
4.178
4.169
4.124
4.108
4.102
4.086
3.877
3.841
3.806
3.749
3.732
3.711
3.695
2.147
2.139
0.927
0.917
0.903
0.893
0.878
0.868
Cl
Cl
+
SCH3
Cl
SCH3
Cl
3.080
3.064
3.032
3.017
2.976
2.954
2.928
2.907
2.849
2.842
2.833
2.826
2.815
2.808
2.799
2.792
3.10
3.00
2.90
2.80
2.70
2.60
ppm (t1)
0.26
2.02
0.27
0.32
1.00
1.04
0.27
3.49
6.92
3.96
5.0
4.0
3.0
2.0
1.0
ppm (t1)

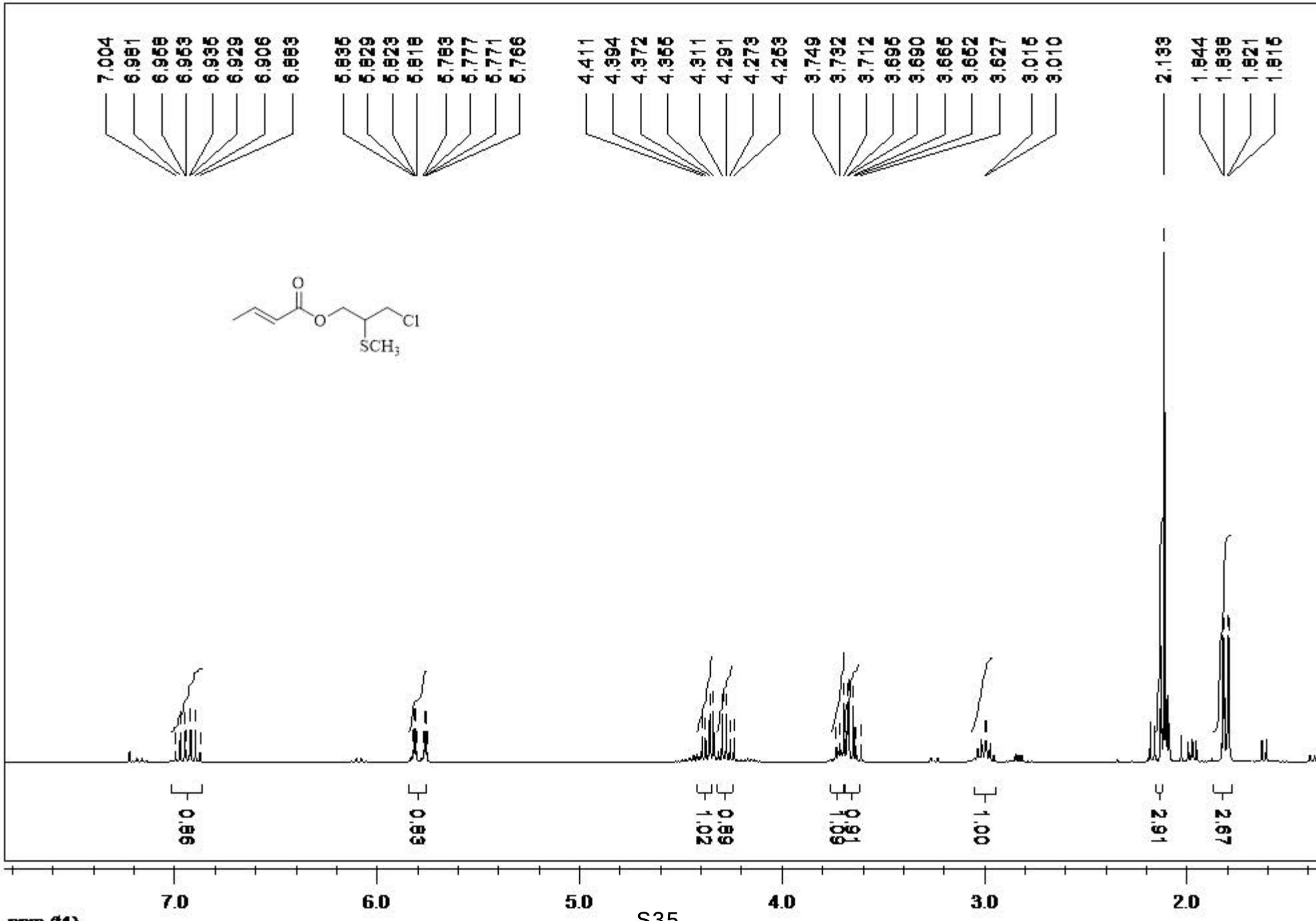

77.423
77.000
76.577
64.855
63.871
61.803
55.690
44.717
39.744
38.208
36.015
20.091
19.204
16.776
15.580
13.399
13.399
13.50
13.00
ppm (t1)
Cl
Cl
+
SCH3
Cl
SCH3
Cl
80
70
60
50
40
30
20
10
ppm (t1)

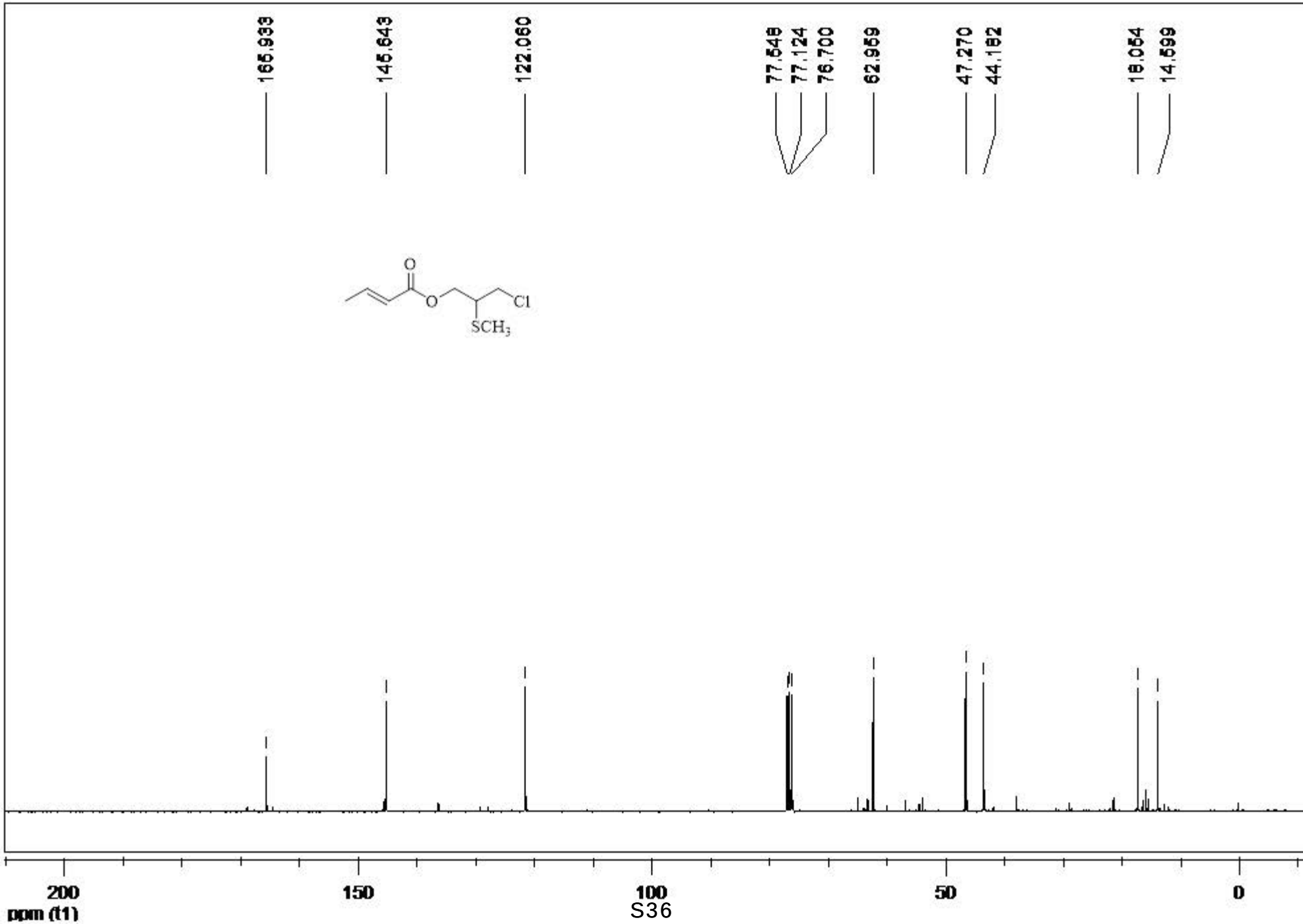

3.996
3.989
3.976
3.969
3.967
3.960
3.947
3.941
3.036
3.007
2.990
2.961
2.864
2.845
2.818
2.799
2.114
1.705
1.674
0.919
0.895
0.872
OH
SCH3
Cl
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
ppm (t1)

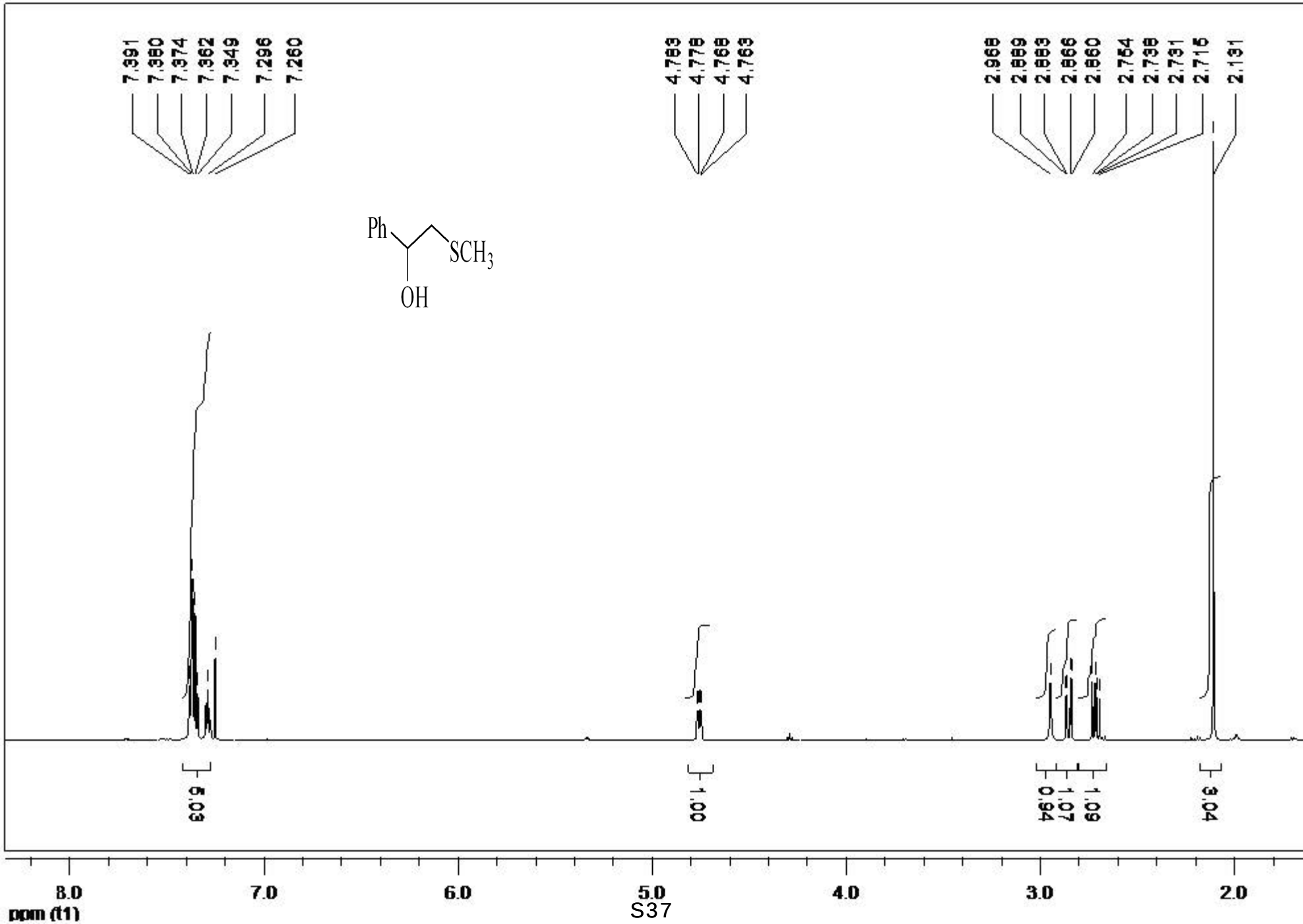

77.420
76.996
76.573
70.740
65.936
38.620
37.216
18.674
16.437
13.899
OH
SCH3
Cl
80
70
60
50
40
30
20
10
ppm (t1)

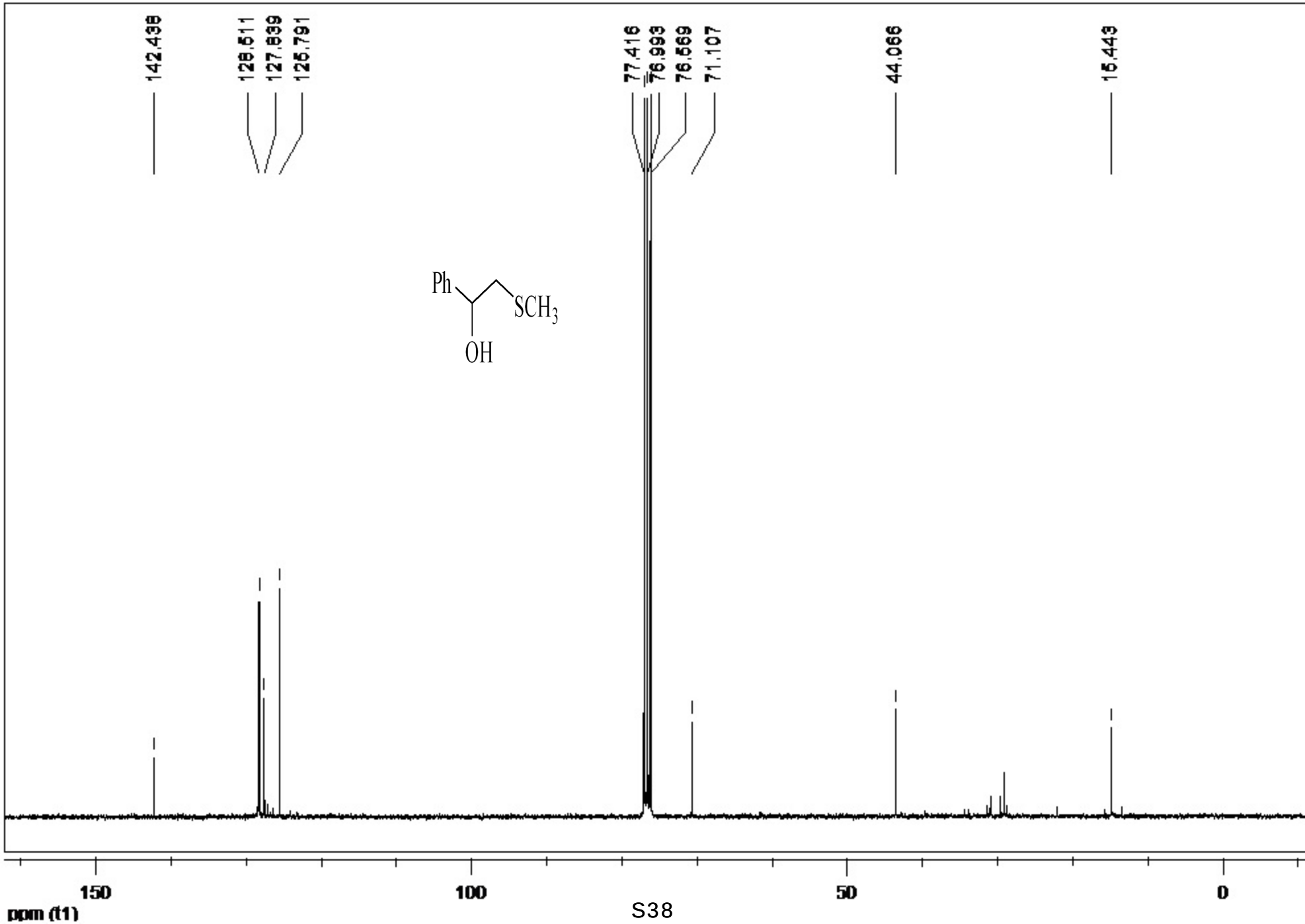

4.107
4.093
4.087
4.083
4.073
4.069
4.063
4.048
2.925
2.905
2.878
2.866
2.858
2.842
2.819
2.795
2.125
OH
SCH3
Cl
0.916
0.892
0.869
1.00
1.14
2.26
2.78
4.09
4.46
4.00
3.50
3.00
2.50
2.00
1.50
1.00
ppm (t1)

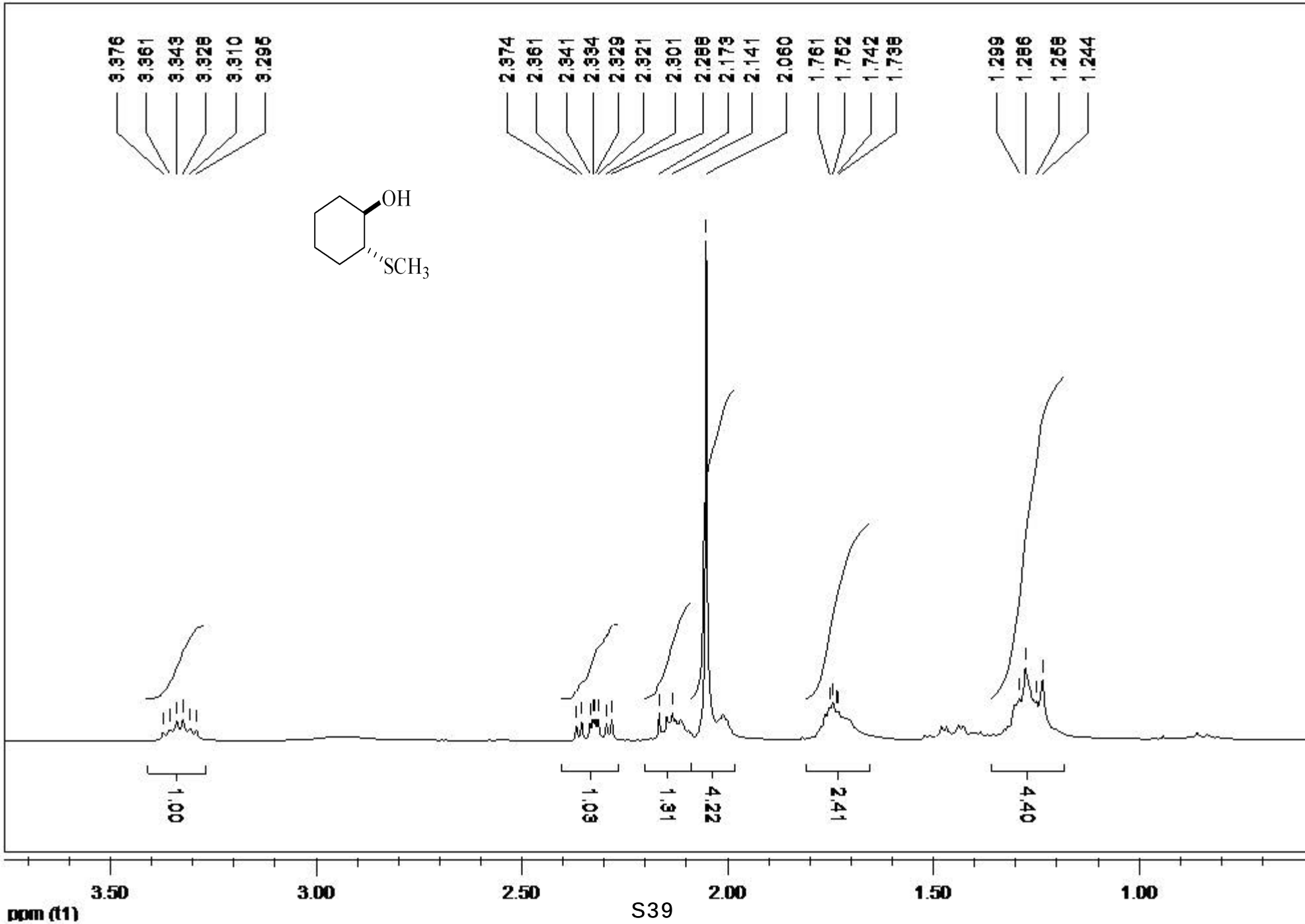

77.416
76.992
76.569
73.428
66.338
37.786
34.428
18.864
16.396
13.905
OH
SCH3
Cl
80
70
60
50
40
30
20
10
ppm (t1)

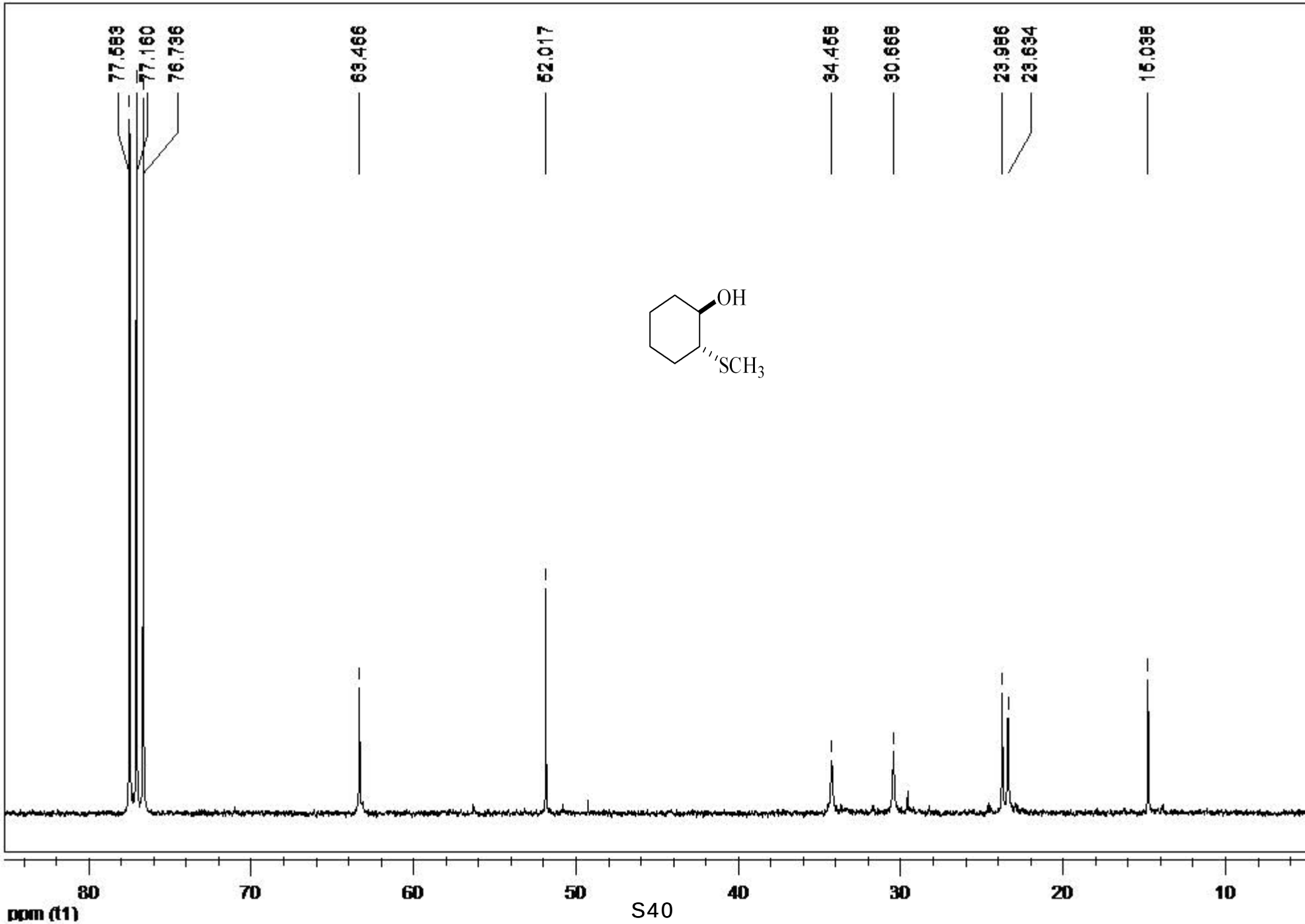

7.004
6.981
6.958
6.953
6.935
6.929
6.906
6.883
5.835
5.829
5.823
5.818
5.783
5.777
5.771
5.766
4.411
4.394
4.372
4.355
4.311
4.291
4.273
4.253
3.749
3.732
3.712
3.695
3.690
3.665
3.652
3.627
3.015
3.010
2.133
1.844
1.838
1.821
1.815
O
O
Cl
SCH3
0.66
0.89
1.02
0.89
0.91
1.09
1.00
2.91
2.67
7.0
6.0
5.0
4.0
3.0
2.0
ppm (t1)

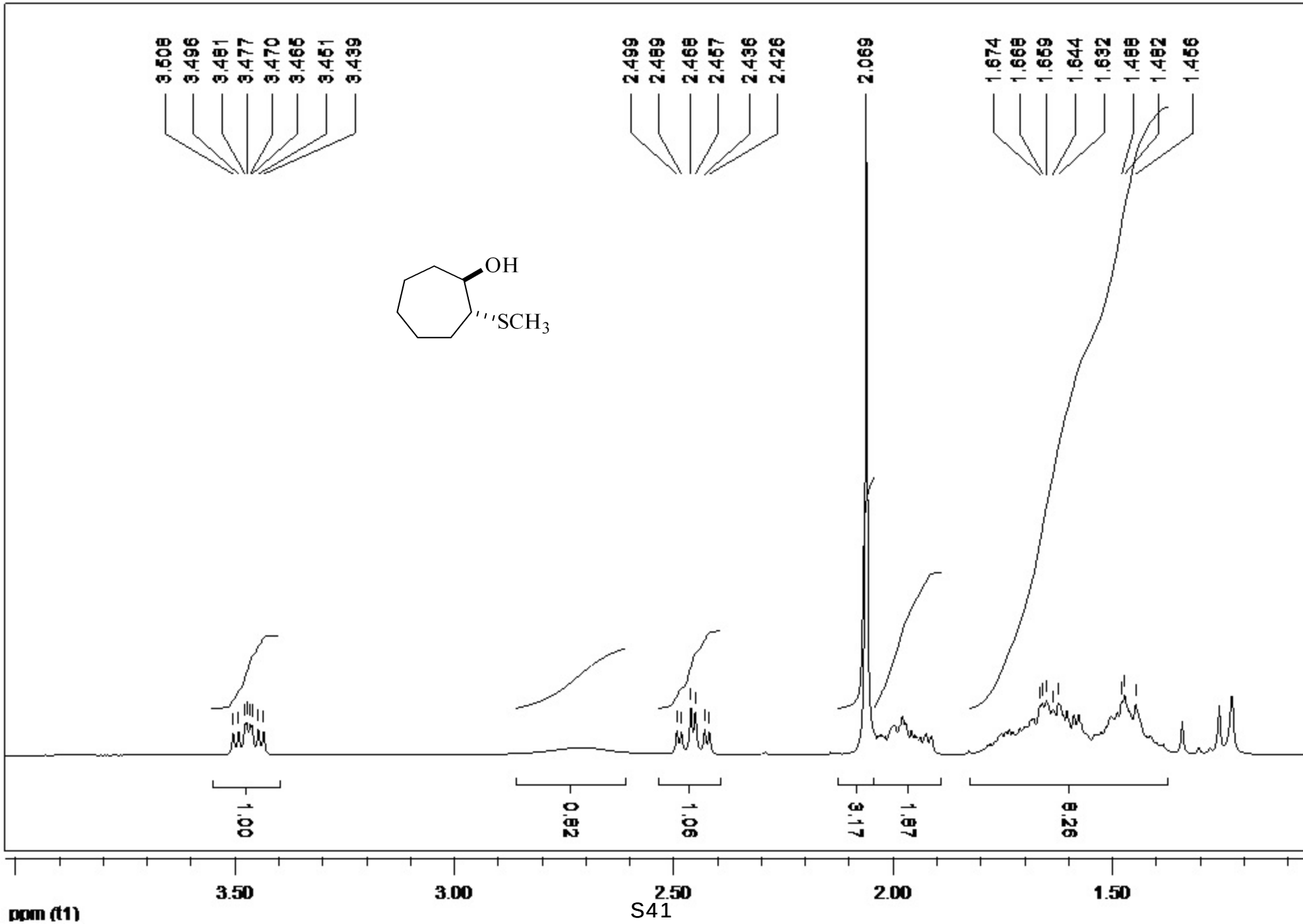

165.933
145.643
122.060
77.548
77.124
76.700
62.959
47.270
44.182
18.054
14.599
O
O
Cl
SCH3
200
150
100
50
0
ppm (t1)

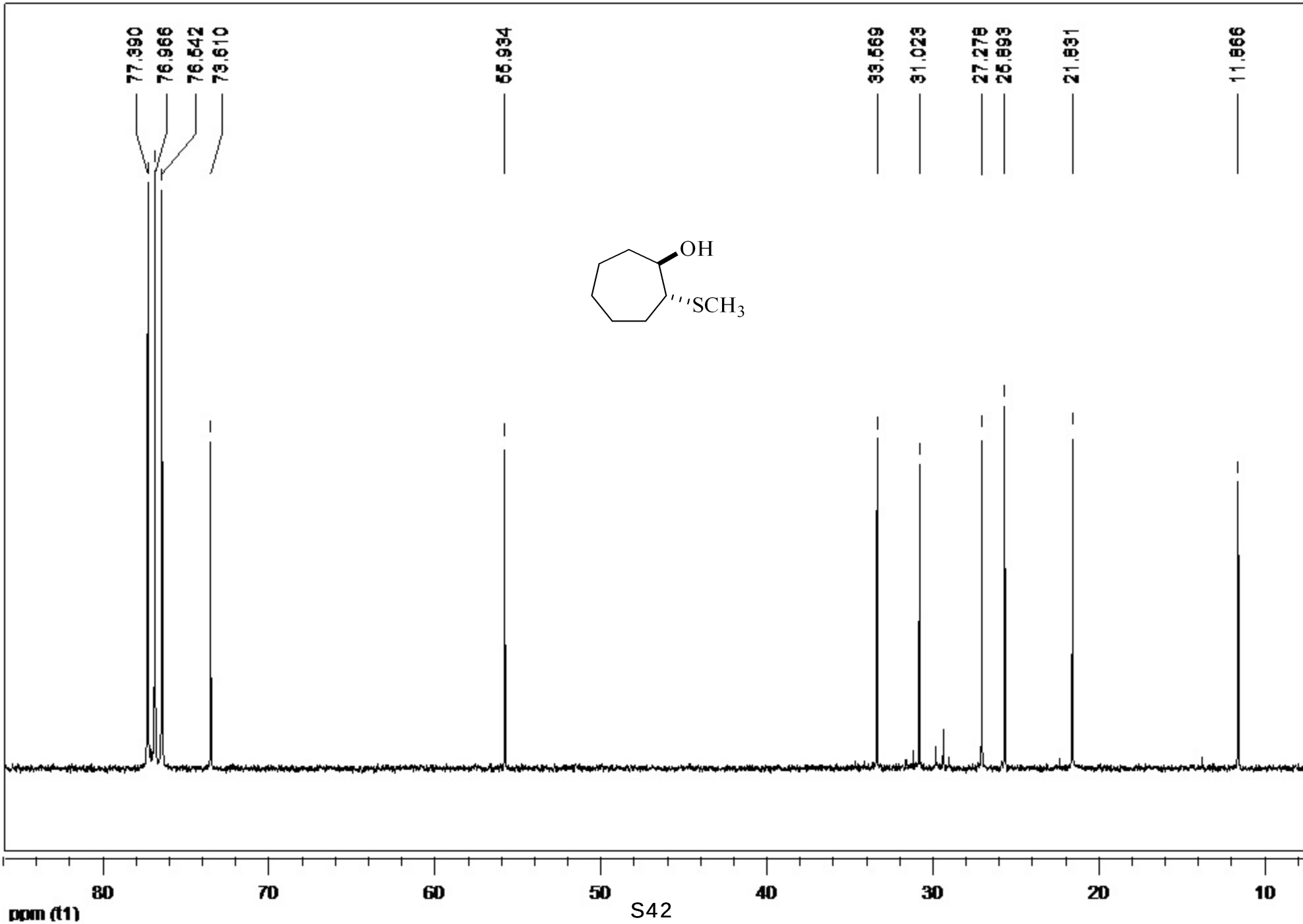

7.391
7.380
7.374
7.362
7.349
7.296
7.260
4.783
4.778
4.768
4.763
2.968
2.889
2.883
2.866
2.860
2.754
2.738
2.731
2.715
2.131
Ph
SCH3
OH
6.03
1.00
0.94
1.07
1.09
3.04
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (t1)

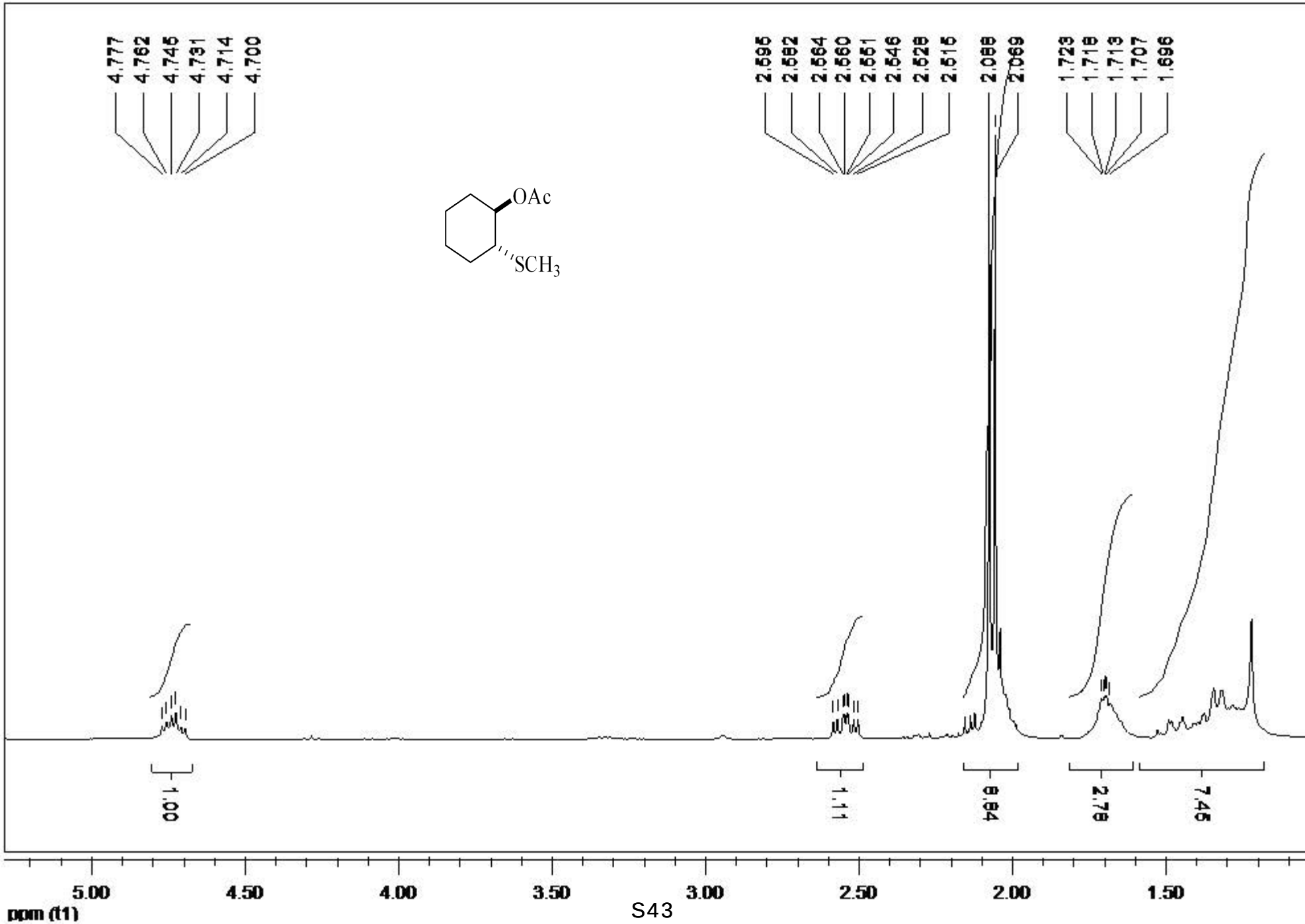

142.438
128.511
127.839
125.791
77.416
76.993
76.569
71.107
44.066
15.443
Ph
SCH3
OH
150
100
50
0
ppm (t1)

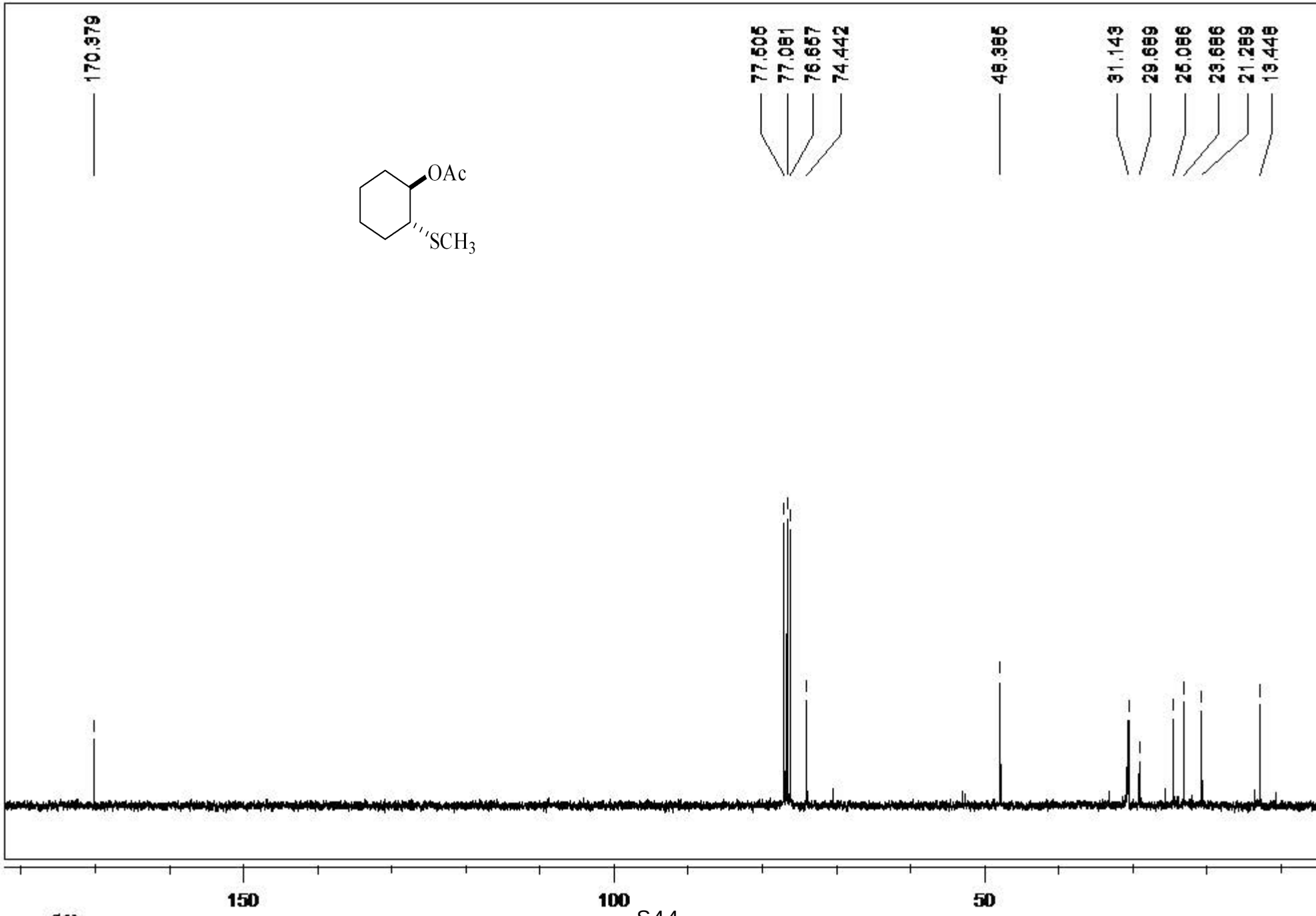

3.376
3.361
3.343
3.328
3.310
3.295
2.374
2.361
2.341
2.334
2.329
2.321
2.301
2.288
2.173
2.141
2.060
1.761
1.752
1.742
1.738
1.299
1.286
1.258
1.244
OH
SCH3
1.00
1.03
1.31
4.22
2.41
4.40
3.50
3.00
2.50
2.00
1.50
1.00
ppm (t1)


77.583
77.160
76.736
63.466
52.017
34.458
30.668
23.986
23.634
15.038
OH
SCH3
80
70
60
50
40
30
20
10
ppm (t1)

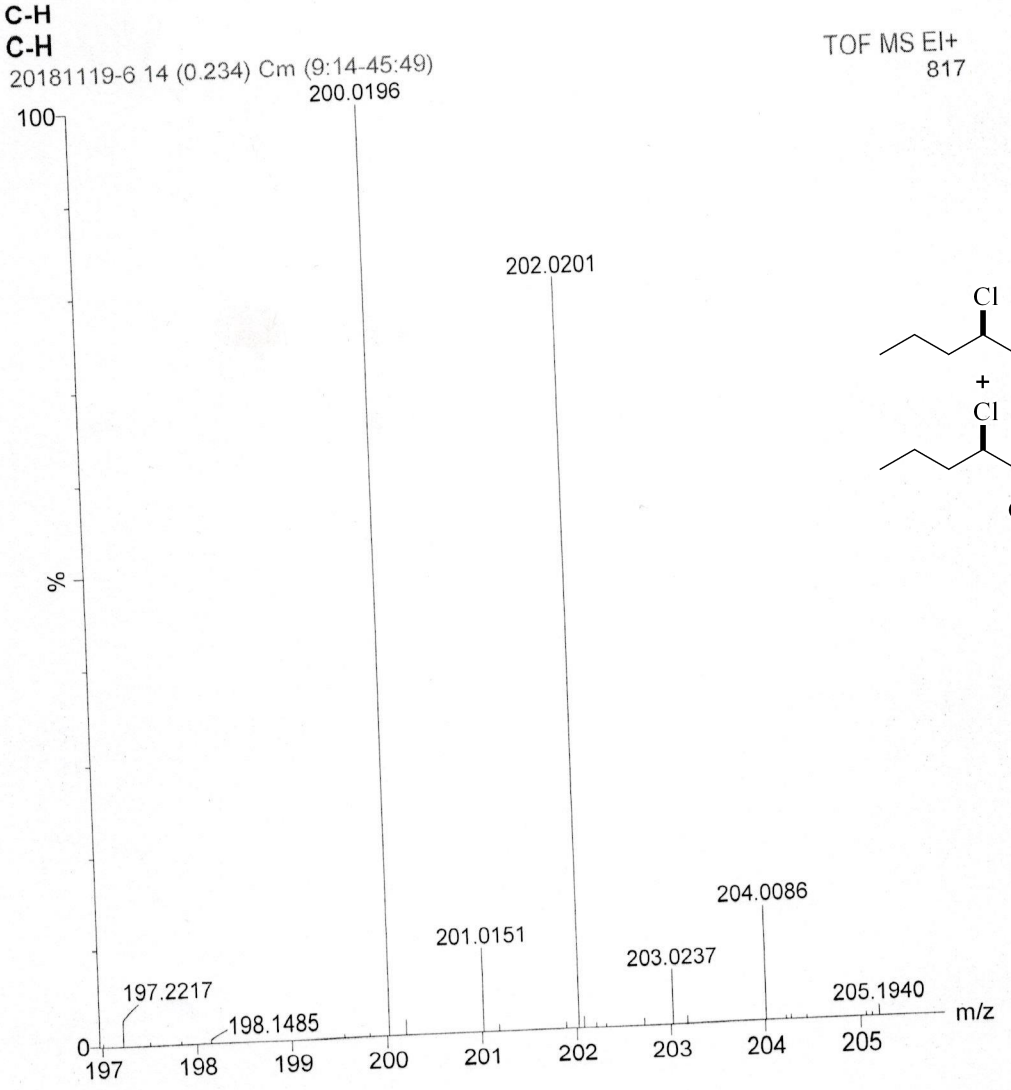

3.508
3.496
3.481
3.477
3.470
3.465
3.451
3.439
2.499
2.489
2.468
2.457
2.436
2.426
2.069
1.674
1.668
1.659
1.644
1.632
1.488
1.482
1.456
OH
SCH3
1.00
0.82
1.06
3.17
1.87
8.26
3.50
3.00
2.50
2.00
1.50
ppm (t1)

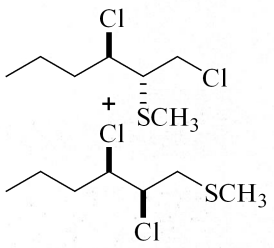

77.390
76.966
76.542
73.610
55.934
33.569
31.023
27.278
25.893
21.831
11.866
OH
SCH3
80
70
60
50
40
30
20
10
ppm (t1)


4.777
4.762
4.745
4.731
4.714
4.700
2.595
2.582
2.564
2.560
2.551
2.546
2.528
2.515
2.088
2.069
1.723
1.718
1.713
1.707
1.696
OAc
SCH3
1.00
1.11
6.84
2.78
7.45
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
ppm (t1)


170.379
77.505
77.081
76.657
74.442
48.385
31.143
29.689
25.086
23.686
21.289
13.448
OAc
SCH3
150
100
50
ppm (t1)

C-H
C-H





Elemental Composition Report

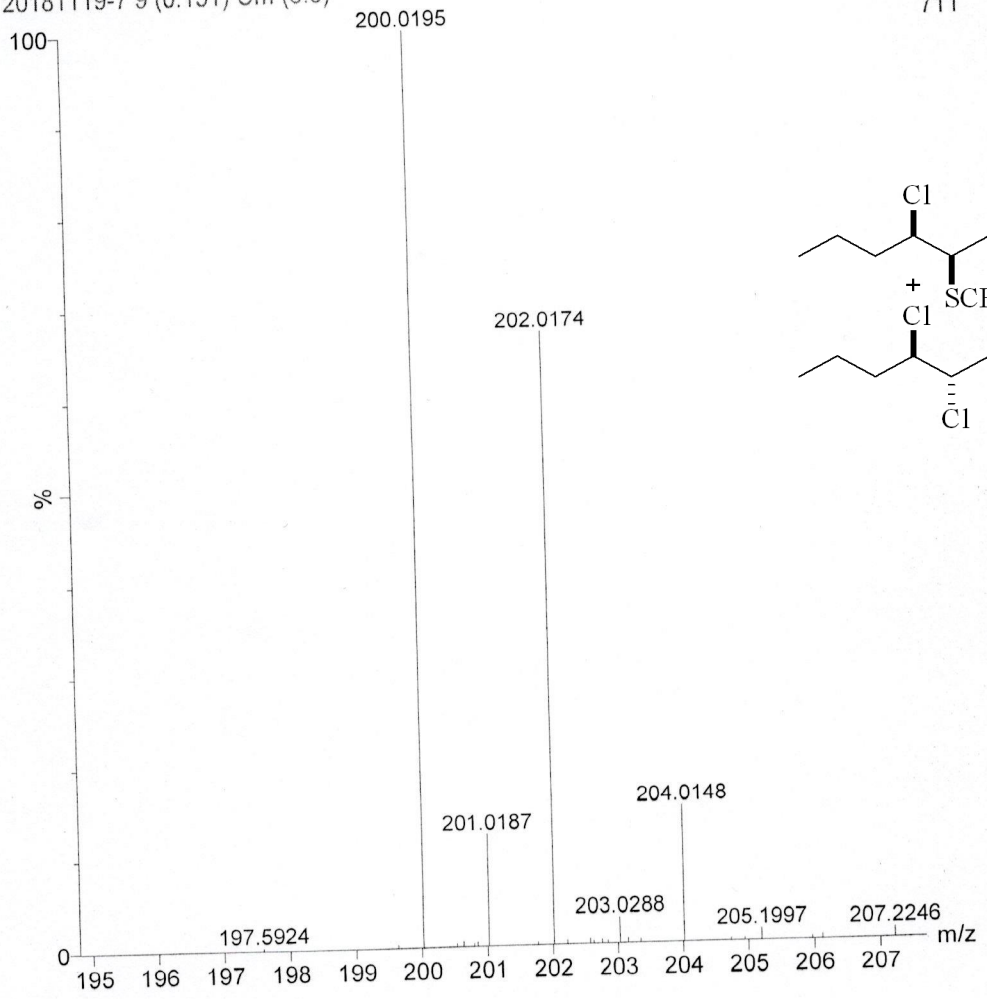
Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 100.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
11 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Minimum:	30.00				-1.5		
Maximum:	100.00		200.0	100.0	50.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	Score	Formula
200.0196	100.00	200.0193	0.3	1.4	0.0	1	C7 H14 S Cl2

T-H
T-H
20181119-7 9 (0.151) Cm (8:9)

TOF MS EI+
711



Elemental Composition Report

Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 100.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
11 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Minimum:	30.00				-1.5		
Maximum:	100.00		200.0	100.0	50.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	Score	Formula
200.0195	100.00	200.0193	0.2	0.9	0.0	1	C7 H14 S Cl2

ESI(P),S-M-11-13,20181128

Analysis Info

Acquisition Date 11/28/2018 3:59:09 PM

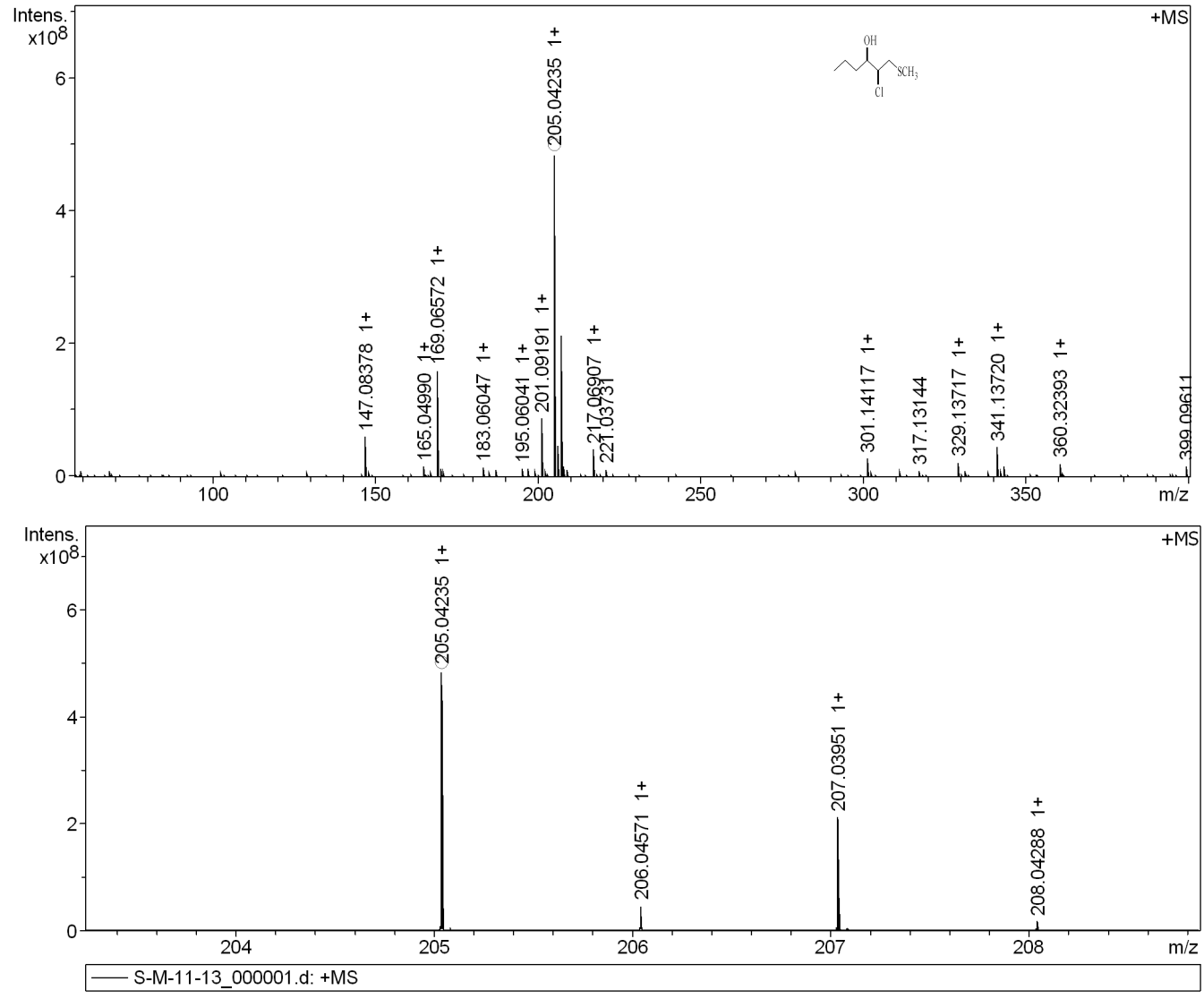
Analysis Name D:\Data\ESI\2018\2018-11\1128\S-M-11-13_000001.d

Sample Name S-M-11-13

Instrument solariX

Acquisition Parameter

Acquisition Mode	Single MS	Acquired Scans	10	Calibration Date	Mon Nov 19 10:49:01 2018
Polarity	Positive				
Broadband Low Mass	57.7 m/z				
Broadband High Mass	400.0 m/z				



Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSigma	rdb	e¯ Conf	N-Rule
205.042352	1	C7H15ClNaOS	100.00	205.042434	-0.4	0.2	29.0	-0.5	even	ok

ESI(P),S-M-24-26,20181128

Analysis Info

Acquisition Date 11/28/2018 3:53:37 PM

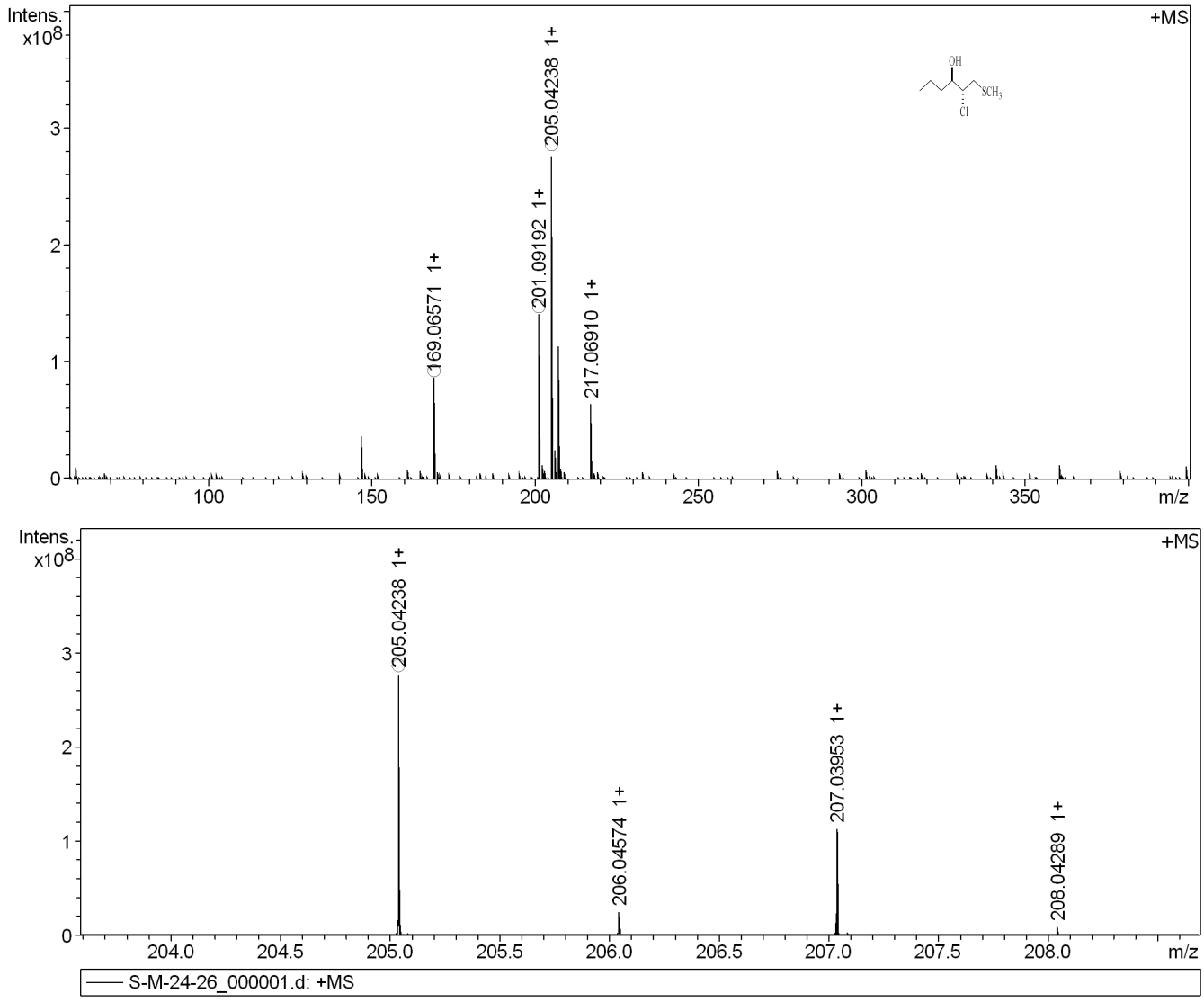
Analysis Name D:\Data\ESI\2018\2018-11\1128\S-M-24-26_000001.d

Sample Name S-M-24-26

Instrument solariX

Acquisition Parameter

Acquisition Mode	Single MS	Acquired Scans	10	Calibration Date	Mon Nov 19 10:49:01 2018
Polarity	Positive				
Broadband Low Mass	57.7 m/z				
Broadband High Mass	400.0 m/z				



Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSigma	rdb	e¯ Conf	N-Rule
169.065713	1	C7H14NaOS	100.00	169.065757	-0.3	0.4	6.4	0.5	even	ok
201.091918	1	C8H18NaO2S	100.00	201.091972	0.3	0.5	6.6	-0.5	even	ok
205.042383	1	C7H15ClNaOS	100.00	205.042434	-0.3	0.0	16.0	-0.5	even	ok

ESI(P),R-M-4-6,20181128

Analysis Info

Acquisition Date 11/28/2018 2:51:02 PM

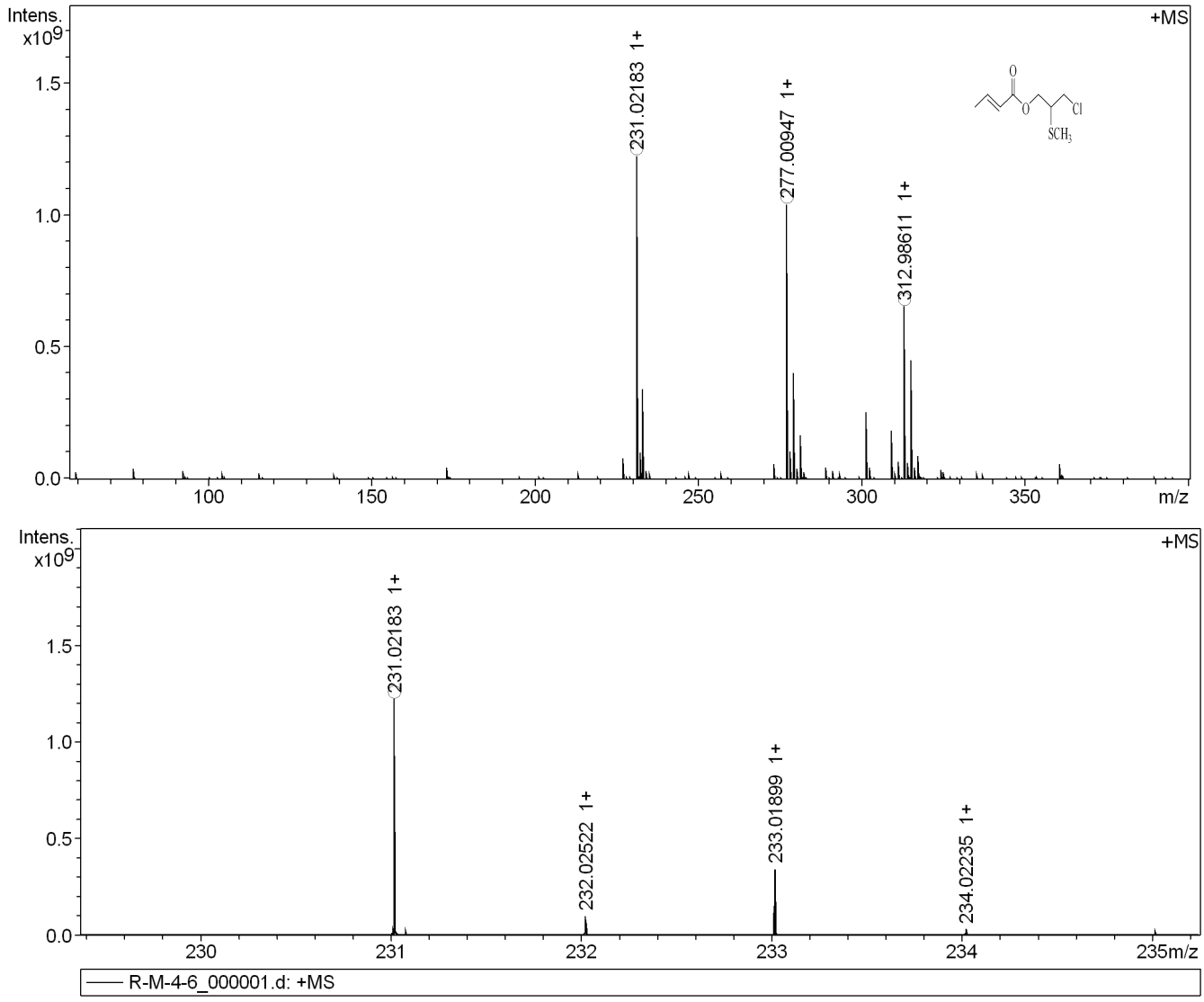
Analysis Name D:\Data\ESI\2018\2018-11\1128\R-M-4-6_000001.d

Sample Name R-M-4-6

Instrument solariX

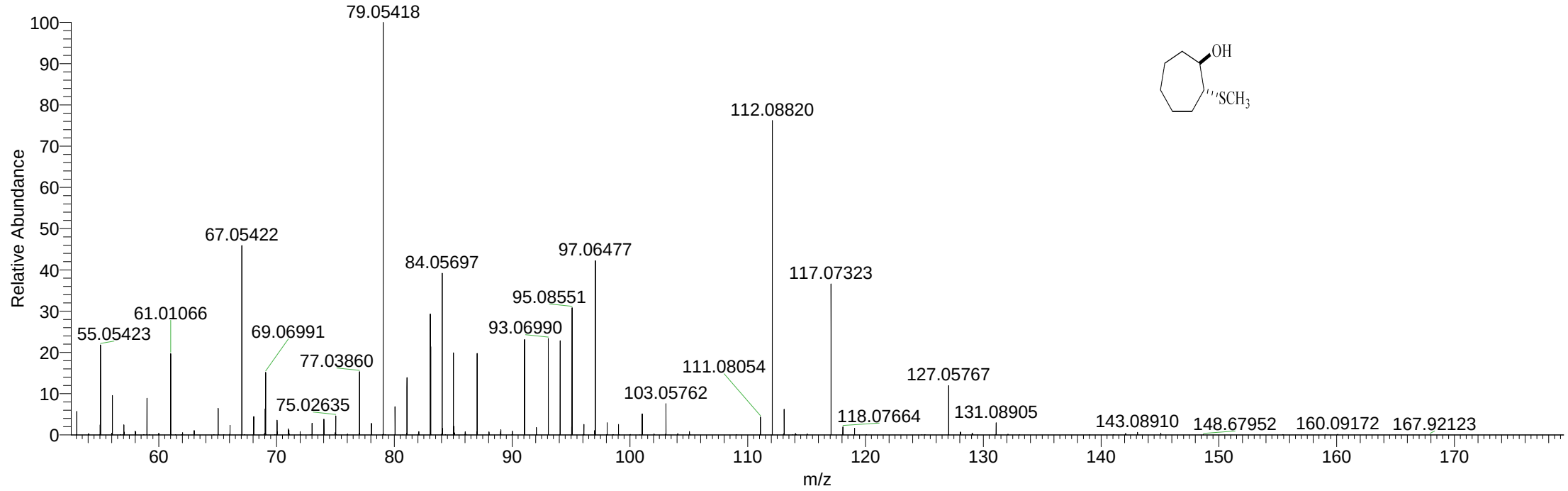
Acquisition Parameter

Acquisition Mode	Single MS	Acquired Scans	10	Calibration Date	Wed Nov 14 09:46:54 2018
Polarity	Positive				
Broadband Low Mass	57.7 m/z				
Broadband High Mass	400.0 m/z				



Meas. m/z	#	Ion Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
231.021833	1	C8H13ClNaO2S	100.00	231.021699	0.6	-0.7	42.3	1.5	even	ok
277.009468	1	C9H15ClNaO2S2	100.00	277.009420	0.2	-0.2	14.4	1.5	even	ok
312.986110	1	C9H16Cl2NaO2S2	100.00	312.986098	0.0	-0.1	25.0	0.5	even	ok

FTMS-18100352 #1405 RT: 5.66 AV: 1 NL: 7.10E9
T: FTMS + p EI Full ms [50.0000-550.0000]



FTMS-18100352 #1405 RT: 5.66 AV: 1 NL: 1.28E7
T: FTMS + p EI Full ms [50.0000-550.0000]