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## *Supporting Information*

# New organosulfur compounds derived from dithioketalization of hydroxyacetone and its tosylate derivative - mechanistic considerations

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## Contents

## *1. General Information*

Reactions were carried out under an atmosphere of argon in heat gun dry flask or an air atmosphere, and room temperature unless stated otherwise. Reactions were monitored by TLC on Merck silica gel 60 $F_{254}$ plates visualized by UV lamp at 254 nm, immersed in a solution of phosphomolybdic acid (PMA) in EtOH (20% wt) and visualized with heat. Column chromatography was performed on Merck silica gel 60 (230-400 mesh). NMR spectra were measured on a Bruker ADVANCE$^{II}$ 500 NMR spectrometer (500 MHz for $^{1}$H spectra and 125 MHz for $^{13}$C spectra), $CDCl_3$ or TMS served as an internal standard. Chemical shifts ($\delta$ values) were quoted in parts per million (ppm), the following abbreviations were used to describe resonance signals splitting patterns when appropriate: s = singlet, d = doublet, dd = doublet of doublets, ddd = doublet of doublet doublets, dqd = doublet of doublet quartets, t = triplet, q = quartet, qd = a quartet of doubles, quin = pentet, m = multiplet, brs = broad singlet, *eq.* = equatorial, *ax.* = axial. Coupling constants (*J* values) were reported in hertz units (Hz). Infrared spectra were measured on a Nicolet 8700 spectrophotometer (ATR or KBr). Low resolution mass spectra (MS) and high resolution mass spectra (HRMS) were measured on an AUTOSPEC (EI+) and on an MALDI-MS ($^{109}$Ag-LGN) mass spectrometers. Melting point were obtained on a Boetius hot plate microscope or MEL-TEMP® apparatus and are uncorrected. In the description of the isolation of products and their purification using the silica gel column chromatography technique, the mass of silica gel is given (g). All liquid physical mixtures are given as mixtures by volume (%).

Sometimes on $^1$H NMR spectra resonance signal at $\delta$ 5.30 ppm is present(residue of DCM) also resonance signal of water (humidity) are present at $\delta$ ~1.54 ppm. In some situations, calibration of the nmr spectra was done on $CDCl_3$ resonance signal ($\delta$ 7.26 ppm).

The process efficiency was calculated based on the mass of tosylate used.

Single crystals suitable for X-ray diffraction measurements were grown from DCM by solvent evaporation. One crystal was chosen under a polarizing microscope and mounted on a Mitegen® loop with ParatoneN® oil. Diffraction data were collected at r.t. using graphite-monochromated Mo Kα radiation on Rigaku Oxford Diffraction Gemini A Ultra four-circle κ-geometry diffractometer. CrysAlis$^{PRO}$ software was used for diffractometer control as well as data reduction. Crystal structure was solved by intrinsic phasing using SHELXT.[1] Initial independent-atom-model structural refinements were carried out using SHELXL and final Hirshfeld-atom-model refinements were carried out using olex2.refine and NoSpherA2. [2-4] All atoms were refined anisotropically. Sulfur atoms were refined anharmonically using 3rd and 4th order Gram-Charlier coefficients, which significantly lowered residual electron density. Crystal structure solution and refinement were carried out using olex2 GUI. [5] CCDC 2364133 contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre [6].

## *2. Materials*

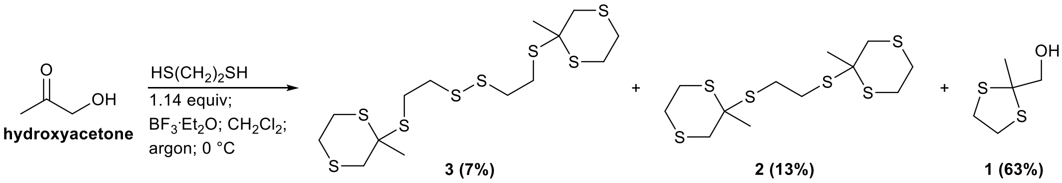
Commercially available reagents were used without purification. The solvents used in the reactions were dried according to the literature [7]. **Boron trifluoride etherate (**for synthesis), **hydroxyacetone** (technical grade 90%) and **ethane-1,2-dithiol** was purchased from Sigma-Aldrich. **Propane-1,3-dithiol** was purchased from TCI. **Hydroxyacetone tosylate** was prepared in accordance with the literature description wherever [8].

The transformation diagrams show the order of products eluted from the chromatographic column.

## *3. Experimental procedures*

### *3.1 Dithioketalization of **hydroxyacetone** with **ethane-1,2-** or **propane-1,3-dithiols***

### *3.1.1 Reaction of **hydroxyacetone** with **ethane-1,2-dithiol** and catalyst **boron trifluoride etherate***

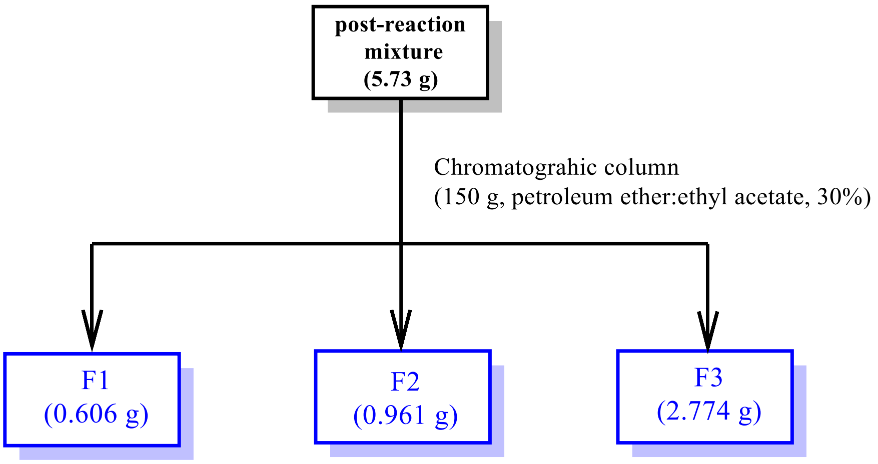


Under an atmosphere of argon in heat gun dry a three-necked round-bottom flask (250 mL) equipped with a magnetic stirrer and a dropping funnel with a pressure equalizer the **hydroxyacetone** (2.16 g; 2.0 mL; 29.2 mmol) in dry DCM (15 mL) was added. Reaction mixture was cooled to 0 °C in ice bath and **ethane-1,2-dithiol** (3.14 g, 2.8 mL, 33.3 mmol, 1.14 equiv) in dry DCM (20 mL) was added dropwise (**Note 1.**). Afterward, **boron trifluoride etherate** (4.14 g, 3.6 mL, 29.2 mmol; 1.0 equiv) in dry DCM (10 mL) was added dropwise (**Note 2.**) and the resulting mixture was stirred for 30 min. The progress of the reaction was monitored by thin layer chromatography (TLC, *n*-hexane:ethyl acetate, 3:1). Next the cooling bath was removed and the reaction mixture with stirring was left for overnight. Then, a mixture of saturated aqueous solution of $NaHCO_3$ (15 mL) and water (30 mL) were added to the reaction mixture and the resulting mixture was stirred for 30 min. After separating the layers, the aqueous phase was extracted with DCM (35 mL). The combined organic layers were dried over anhydrous sodium sulfate. After filtering off the drying agent, volatiles were removed under reduced pressure. The crude reaction mixture was obtained in the form of an oil (5.7 g).

**Note:**

1. **Ethane-1,2-dithiol** was added over 35 min.
2. **Boron trifluoride etherate** was added over 40 min.

Crude mixture of products was isolated and purified by column chromatography. The flowsheet 3.1.1 shows the isolation of the main products.

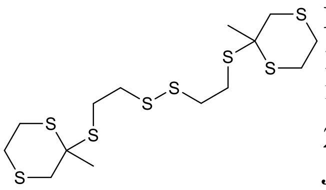


*Legend:*

***F1*** *– was purify with two chromatographic columns: 1st column with 90 g of gel, elution with n-hexane:ethyl acetate 0.15%, and 2nd column with 50 g of gel, elution with n-hexane:ethyl acetate 3%. Chromatographically pure product* ***3*** *(0.46 g) was obtained in the form of a colorless oil.*
***F2*** *– after evaporation of the eluent, 610 mg of product* ***2*** *crystallized and additionally 90 mg was "extracted" from the post-crystalline liquors by chromatography. Product* ***2*** *(0.70 g) was obtained in the form of a colorless crystalline solid.*
***F3*** *– chromatographically pure product* ***1*** *(2.77 g) was obtained in the form of a colorless oil.*

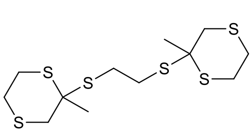
Flowsheet 3.1.1 Scheme illustrates the isolation of products **1**, **2** and **3**.

Spectral data of **2,2'-[disulfanediylbis(ethane-2,1-diylsulfanediyl)]bis(2-methyl-1,4-dithiane)**, **3** (0.46 g, yield 7%):



IR (ATR) ($cm^{-1}$): $\tilde{\nu}$=2956; 2902; 2799; 2543; 1438; 1404; 1368; 1287; 1252; 1207; 1128; 1058; 933; 894; 840; 759; 699; 683; 663.
$^1$H-NMR (500 MHz, $CDCl_3$): δ=3.20 (ddd, *J 13.7, 9.1 and 2.6,* 2H, 2xSCH$H_{ax.}CH_2SCH_2$); 3.08 (d, *J 13.9*, 2H, 2xSCH$H_{ax.}CCH_3$); 2.99 (d, *J 13.9*, 2H, 2xSCH$H_{eq.}CCH_3$); 2.94-2.90 (m, 4H, $2xSCH_2CH_2SCH_2$): 2.88-2.85 (m, 2H, 2xSCH$H_{eq.}CH_2SCH_2$); 2.83-2.75 (m, 8H, $(SCH_2CH_2S)_2$); 1.70 (2s, 6H, 2x$CH_3$).
$^{13}$C-NMR (125 MHz, $CDCl_3$): δ=52.1 (2*C*); 41.9 (2*C*); 33.7 (2*C*); 29.8 (2*C*); 28.8 (2*C*); 28.0 (2*C*); 24.9 (2*C*).
MS (EI+), *m/z* (%): 450 ([M]$^+$, 0.19); 135 (10); 134 (10); 133 (100); 132 (27); 105 (13); 62 (15); 60 (26); 46 (13).
HRMS for $C_{14}H_{26}S_8$ calcd: 449.9800; obsd: 449. 9812.

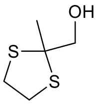
Spectral data of **2,2'-[ethane-1,2-diyldi(sulfanediyl)]bis(2-methyl-1,4-dithiane)**, **2** (0.70 g, yield 13%):



m.p.= 68-69 °C ($CH_2Cl_2$)
IR (ATR) ($cm^{-1}$): $\tilde{\nu}$=2966; 2946; 2934; 2907; 2897; 1428; 1403; 1367; 1290; 1196; 1061; 1045; 882; 826; 760; 708; 676.
$^1$H-NMR (500 MHz, $CDCl_3$): δ=3.21 (ddd, *J 14.2, 9.0* and *2.6,* 2H, 2xSCH$H_{ax.}CH_2SCH_2$); 3.08 (d, *J 13.9*, 2H, 2xSCH$H_{ax.}CCH_3$); 3.01 (d, *J 13.9*, 2H, 2xSCH$H_{eq.}CCH_3$); 2.97-2.90 (m, 4H, $2xSCH_2CH_2SCH_2$): 2.89-2.85 (m, 2H, 2xSCH$H_{eq.}CH_2SCH_2$); 2.84-2.75 (m, 4H, $2xSCSCH_2$); 1.71 (2s, 6H, 2x$CH_3$).
$^{13}$C-NMR (125 MHz, $CDCl_3$): δ=52.1 (2*C*); 41.8 (2*C*); 29.78 (2*C*); 29.74 (2*C*); 28.84 (2*C*); 28.07 (2*C*).
MS (EI+), *m/z* (%): 358 ([M]$^+$, 0.7); 225 (46); 135 (12); 134 (11); 133 (100); 132 (19); 105 (16); 73 (11); 62 (14); 60 (20); 46 (11).
HRMS for $C_{12}H_{22}S_6$ calcd: 358.0046; obsd: 358.0052.

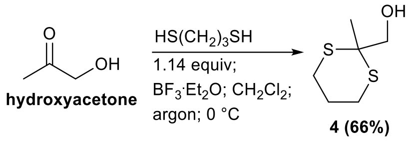
Crystal Data for compound **2**: $C_{12}H_{22}S_6$ ($M$ =358.705 g/mol): triclinic, space group $\boldsymbol{P\bar{1}}$ (no. 2), $a$ = 5.9899(1) Å, $b$ = 7.7228(2) Å, $c$ = 9.8079(3) Å, $\alpha$ = 93.666(2)°, $\beta$ = 92.363(2)°, $\gamma$ = 109.581(2)°, $V$ = 425.640(19) Å$^3$, $Z$ = 1, $T$ = 297 K, μ(Mo Kα) = 0.786 $mm^{-1}$, $\rho$ = 1.40 g/cm$^3$, 29994 reflections measured (6.72° ≤ 2Θ ≤ 65.44°), 3011 unique ($R_{int}$ = 0.033, $R_{sigma}$ = 0.019) which were used in all calculations. The final $R_1$ was 0.026 ($I \geq 2\sigma(I)$), and $wR_2$ was 0.046 (all data).

The spectral data of product **1** were reported by Afonso *et al.* in 1991 [9]. Our spectral data upgrades the literature data.
Spectral data of (**2-methyl-1,3-dithiolan-2-yl)methanol**, **1** (2.77 g, yield 63%):



IR (ATR) (cm$^{-1}$): $\tilde{\nu}$=3400 (OH); 2961; 2921; 2857; 1438; 1423; 1373; 1276; 1152; 1100; 1071; 1026; 973; 936; 851; 732; 681.
$^{1}$H-NMR(500 MHz, $CDCl_3$): δ=3.58 (d, *J* 7.1, 2H, $\mathrm{C}H_2\mathrm{OH}$); 3.39-3.34 (m, 2H, $\mathrm{SCH}H_{ax.}$); 3.32-3.27 (m, 2H, $\mathrm{SCH}H_{eq.}$); 2.29 (t, *J 7.1*, 1H, $\mathrm{O}H$); 1.77 (s, 3H, $\mathrm{C}H_3$).
$^{13}$C-NMR (125 MHz, $CDCl_3$): δ=71.0; 68.1; 39.6 (2*C*); 26.6.
MS (EI+), *m/z* (%): 150 ([M]$^+$,11); 121 (9); 119 (100); 61 (10); 59 (39); 58 (7); 57 (3); 46 (7).
HRMS for $C_5H_{10}OS_2$ calcd: 150.0173; obsd: 150.0171.

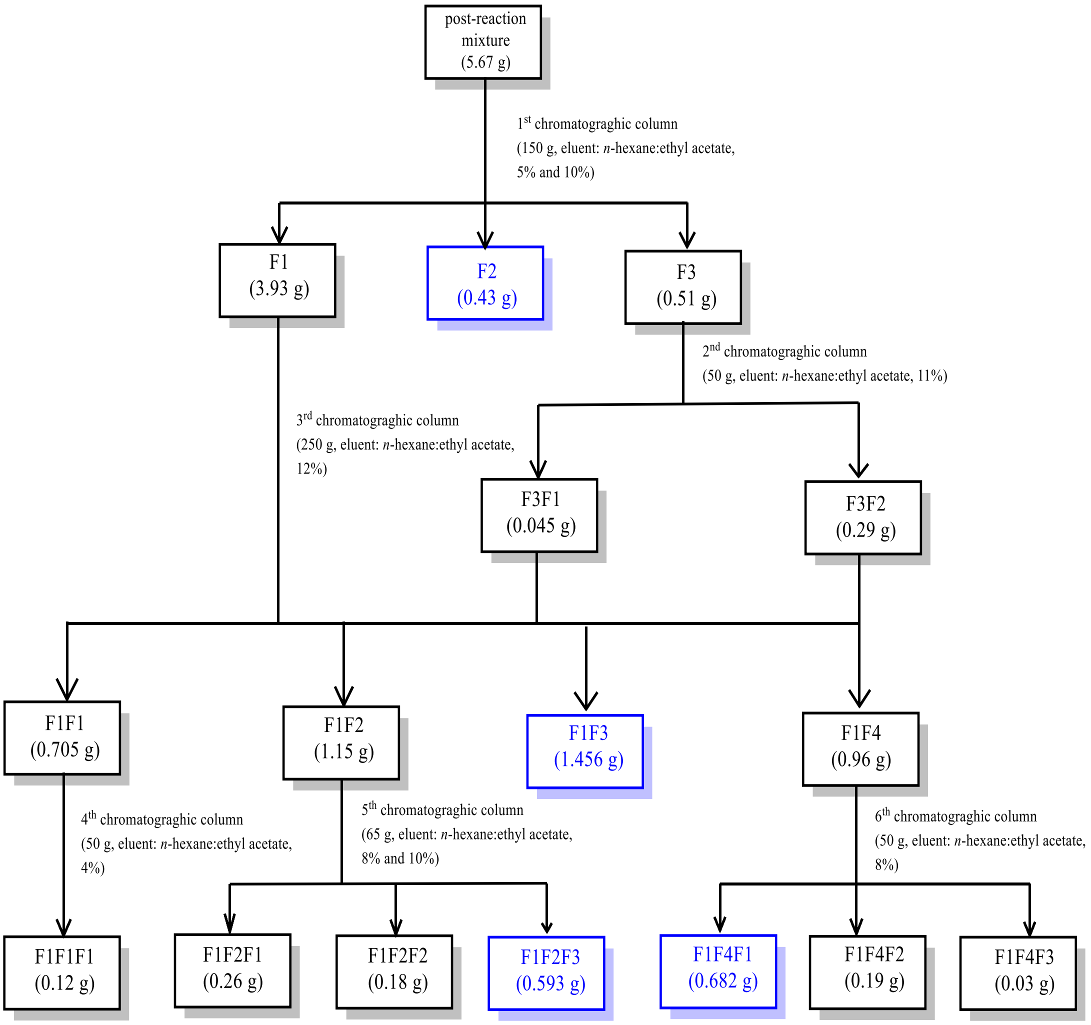
### *3.1.2 Reaction of* ***hydroxyacetone*** *with* ***propane-1,3-dithiol***



Under an atmosphere of argon in heat gun dry a three-necked round-bottom flask (250 mL) equipped with a magnetic stirrer and a dropping funnel with a pressure equalizer the **hydroxyacetone** (2.15 g, 2 mL, 29.0 mmol) in DCM (10 mL) was added. Reaction mixture was cooled to 0 °C in ice bath and **propane-1,3-dithiol** (3.20 g, 3 mL, 29.6 mmol, 1.02 equiv) was added **(Note 1.)**. The reaction mixture was held and stirred for 30 min. Then it was heated to r.t. and left for 20 h. The progress of the reaction was monitored by thin layer chromatography (TLC, *n*-hexane:ethyl acetate, 3:1) (**Note 2.**). Again reaction mixture was cooled to 0 °C in ice bath and **boron trifluoride etherate** (3.45 g, 3 mL, 24.3 mmol, 0.84 equiv) was added **(Note 3.)**. The reaction was stirred for approximately 10 min. (substrate disappearance according to TLC). Then, a mixture of saturated aqueous solution of $NaHCO_3$ (15 mL) and water (30 mL) were added to the reaction mixture and the resulting mixture was stirred for 25 min. After separating the layers, the aqueous phase was extracted with DCM (70 mL). The combined organic layers were dried over anhydrous sodium sulfate. After filtering off the drying agent, volatiles were removed under reduced pressure. The crude reaction mixture was obtained in the form of an oil (5.67 g). Flowsheet 3.1.2 illustrates the isolation and purification of main product. Chromatographically pure product **4** was isolated as a pale yellow oil (3.16 g, yield 66%).

**Note:**

1. **Propane-1,3-dithiol** was added over 45 min.
2. After 1 hour of reaction at r.t., according to TLC, unreacted substrate is present in the reaction mixture ($R_f$=0.25).
3. **Boron trifluoride etherate** was added over 40 min. (an exothermic effect occurs). After the catalyst was added dropwise, the reaction mixture became cloudy and yellow.

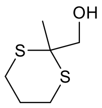


*Legend:*

*After column chromatography a $^1$H-NMR spectrum was done for each fraction and decisions were made about disposal or re-chromatography. The $^1$H-NMR spectra of fractions F2, F1F3, F1F2F3 and F1F4F1 (blue color) were the identical. These fractions were combined and selected as chromatographically pure product* ***4****.*

Flowsheet 3.1.2 Isolation of product **4** from the reaction mixture.

Spectral data of **(2-methyl-1,3-dithian-2-yl)methanol**, **4** (3.16 g, 66%):



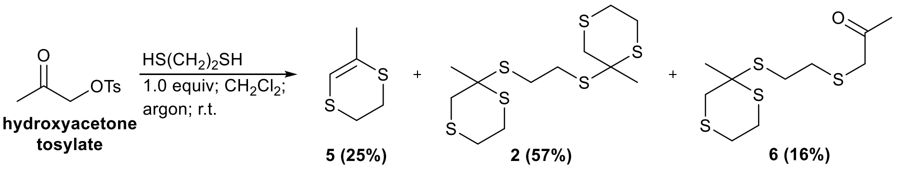
IR (ATR) (cm$^{-1}$): $\tilde{\nu}$=3419 (OH); 2921; 2907; 2867; 2828; 1423; 1367; 1277; 1239; 1213; 1147; 1029; 908; 866; 817; 746; 677; 659.

$^1$H-NMR (500 MHz, CDCl$_3$): δ=3.70 (d, *J* 6.65, 2H, C$H_2$OH); 2.96-2.90 (m, 2H, 2xSCH$H_{ax.}$); 2.62-2.58 (m, 2H, 2xSCH$H_{eq.}$); 2.14-2.08 (m, 1H, SCH$_2$CH$H_{ax.}$); 2.04 (t, *J* 6.65, 1H, O$H$); 1.88-1.79 (m, 1H, SCH$_2$CH$H_{eq.}$); 1.45 (s, 3H, C$H_3$).

$^{13}$C-NMR (125 MHz, CDCl$_3$): δ=65.5; 49.8 (*C*$_{IV}$); 26.2 (2*C*); 25.6; 24.5.
MS (EI+), *m*/*z* (%): 164 ([M]$^+$, 11); 135 (9); 133 (100); 73 (5); 61 (6); 59 (45); 46 (7); 42 (9).
HRMS for $C_6H_{12}OS_2$ calcd:164.0330; obsd: 164.0327.

## *3.2 Dithioketalization of* ***hydroxyacetone tosylate*** *with* ***ethane-1,2-dithiol*** *at room temperature in an atmosphere of argon or air*

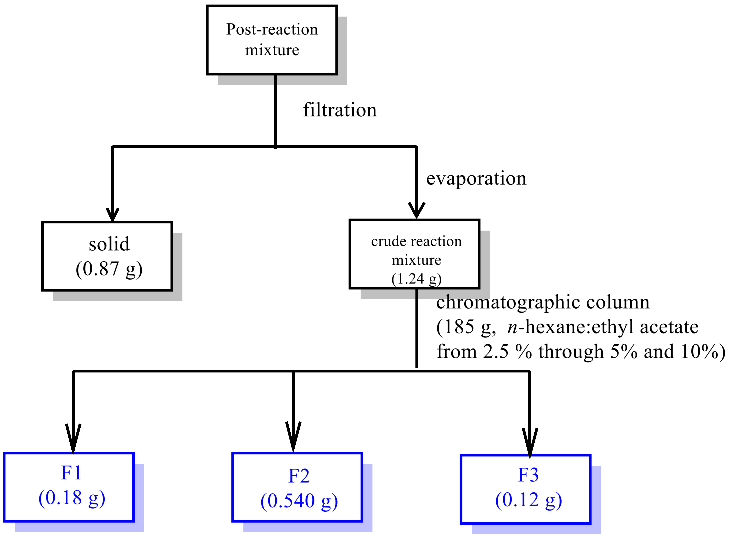
### *3.2.1 Dithioketalization of* ***hydroxyacetone tosylate*** *with* ***ethane-1,2-dithiol*** *at room temperature in an atmosphere of argon*



Under an atmosphere of argon in heat gun dry a two-necked round-bottom flask (250 mL) equipped with a magnetic stirrer and a dropping funnel with a pressure equalizer the **hydroxyacetone tosylate** in dry DCM (1.23 g, 5.39 mmol, in 10 mL DCM) was added. Then, **ethane-1,2-dithiol** (0.51 g, 0.5 mL, 5.41 mmol, 1.0 equiv) in dry DCM (5 mL) was added dropwise to the reaction mixture (**Note 1.)**. The progress of the reaction was monitored by thin layer chromatography (TLC, *n*-hexane:ethyl acetate, 3:1). After adding dithiol, the reaction mixture was left to stir for 24 hours. Then, the crystalline precipitate (0.87 g) was filtered off and washed with DCM (10 mL). The filtrate was concentrated to 1/2 volume. Then, a mixture of saturated aqueous solution of $NaHCO_{3sat.}$ (10 mL) and water (20 mL) were added to the reaction mixture and the resulting mixture was stirred for 30 min. After separating the layers, the aqueous phase was extracted with DCM (30 mL). The combined organic layers were dried over anhydrous sodium sulfate. After filtering off the drying agent, volatiles were removed under reduced pressure. The crude reaction mixture was obtained in the form of an oil (1.24 g). Flowsheet 3.2.1 shows isolation and purification of three main products. A $^1$H-NMR spectrum was done for each fraction after column chromatography. Chromatographically pure product **5** (0.18 g, yield 25%) was obtained as an oil, also product **2** (0.54 g, yield 57%) as a crystalline precipitate, and product **6** (0.12 g, yield 16%) as an oil.

### Note 1.

**Ethane-1,2-dithiol** was added over 30 min. During the dropwise addition of dithiol a precipitate of PTSA occurred.

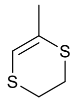


*Legend:*

*Fractions was identified: F1 as a product **5**; F2 as a product **2** and F3 as a product **6**.*

Flowsheet 3.2.1 Scheme of isolation of products **5**, **2** and **6**.

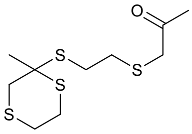
Spectral data of **5-methyl-2,3-dihydro-1,4-dithiine**, **5** (0.18 g, yield 25%):



Spectral data are consistent with the literature.[9, 10]
$^1$H-NMR (500 MHz, $CDCl_3$): δ=5.83 (s, 1H, =C*H*); 3.22-3.20 (m, 2H, 2xSCH$H_{ax.}$); 3.10-3.08 (m, 2H, 2xSCH$H_{eq.}$); 1.92 (d, *J* 1.0, 3H, C$H_3$).
$^{13}$C-NMR (125 MHz, $CDCl_3$): δ=124.1; 108.1; 27.89; 25.30; 25.04.

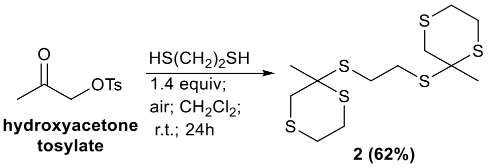
Spectral data of **2,2'-[ethane-1,2-diyldi(sulfanediyl)]bis(2-methyl-1,4-dithiane)**, **2** (0.54 g, yield 57%) are consistent with the spectral data for product obtained in experiment 3.1.1.

Spectral data of **1-({2-[(2-methyl-1,4-dithian-2-yl)sulfanyl]ethyl}sulfanyl)propan-2-one, 6** (0.12 g, yield 16%):



IR (ATR) (cm$^{-1}$): $\tilde{\nu}$=2957; 2905; 2854; 1700 (C=O); 1406; 1354; 1275; 1235; 1202; 1178; 1139; 1060; 999; 933; 894; 840; 758; 683; 663.
$^1$H-NMR (500 MHz, $CDCl_3$): δ=3.26 (s, 2H, C$H_2$C(O)); 3.16 (ddd, *J 13.6, 9.0* and *2.6,* 1H, SC$H_{ax.}$HC$H_2$SC$H_2$); 3.05 (d, *J 13.9*, 1H, SC$H_{ax.}$HCC$H_3$); 2.96 (d, *J 13.9*, 1H, SC$H_{eq.}$HCC$H_3$); 2.88-2.71 (m, 7H, C$H_2$C$H_2$SC(C$H_3$)SCH$H_{eq.}$C$H_2$); 2.28 (s, 3H, C(O)C$H_3$); 1.67 (s, 3H, CC$H_3$).
$^{13}$C-NMR (125 MHz, $CDCl_3$): δ=203.6; 52.2; 41.78; 41.73; 32.1; 29.8; 29.0; 28.8; 28.0; 27.8.
MS (EI+), *m/z* (%): 282 ([M]$^+$, 7); 135 (12); 134 (11); 133 (100); 132(18); 119 (15); 117 (9); 105 (17); 73 (16); 62 (15) 60 (18); 46 (12); 44 (20).
HRMS for $C_{10}H_{18}0S_4$ calcd: 282.0241; obsd: 282.0250.

### *3.2.2 Dithioketalization of **hydroxyacetone tosylate** with **ethane-1,2-dithiol** at room temperature in an air atmosphere*

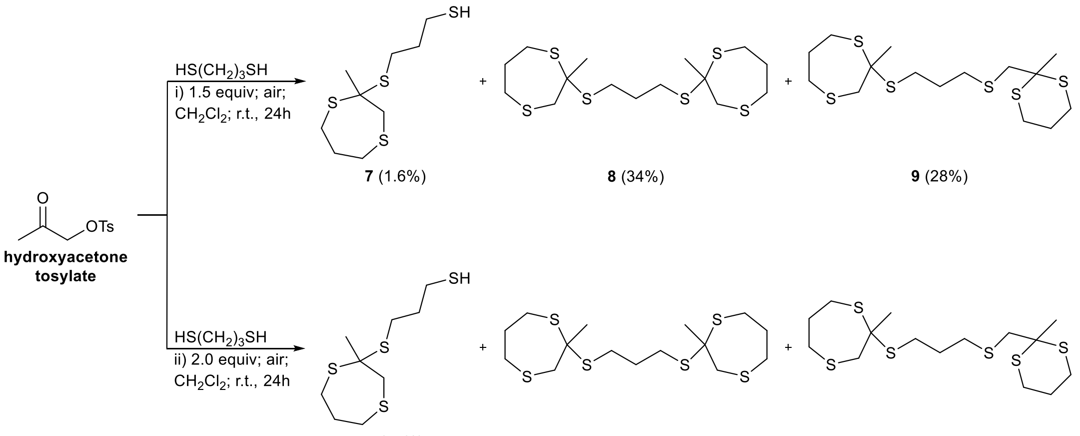


To a two-necked round-bottom flask (100 mL) equipped with a magnetic stirrer and a dropping funnel with a pressure equalizer the **hydroxyacetone tosylate** (0.85 g; 0.7 mL; 3.7 mmol) in DCM (15 mL) was added. **Ethane-1,2-dithiol** (0.48 g, 0.57 mL, 5.1 mmol, 1.4 equiv) in DCM (10 mL) was added dropwise to the reaction mixture (**Note 1.**). The progress of the reaction was monitored by thin layer chromatography (TLC, *n*-hexane:ethyl acetate, 3:1). The reaction mixture was left overnight. The crystalline precipitate of PTSA (0.73 g) was filtered off and washed with additional DCM (10 mL). An aqueous solution of $NaHCO_3$ *satur.* was added to the filtrate in portions (30 mL, 1:2) and the resulting mixture was stirred for 20 min. After separating the layers, the aqueous phase was extracted with DCM (30 mL). The combined organic layers were dried over anhydrous sodium sulfate. After filtering off the drying agent, volatiles were removed under reduced pressure. The crude reaction mixture was obtained in the form of an oil (0.989 g). Main product was isolated and purified by two column chromatography (1st column: 100 g, *n*-hexane (0.71 g), 2nd column: 100 g, *n*-hexane:ethyl acetate, 0.25% up to 1.0%) yielding **2** as solid (0.41 g, 62%). Spectral data of **2** are consistent with the spectral data ($^1$H-NMR) for product obtained in experiment 3.1.1.

**Note 1.**

**Ethane-1,2-dithiol** was added over 20 min. During the dropwise addition of dithiol reagent, a precipitate of PTSA occurred.

### 3.3 *Dithioketalization of* ***hydroxyacetone tosylate*** *with* ***propane-1,3-dithiol*** *in an atmosphere of air*



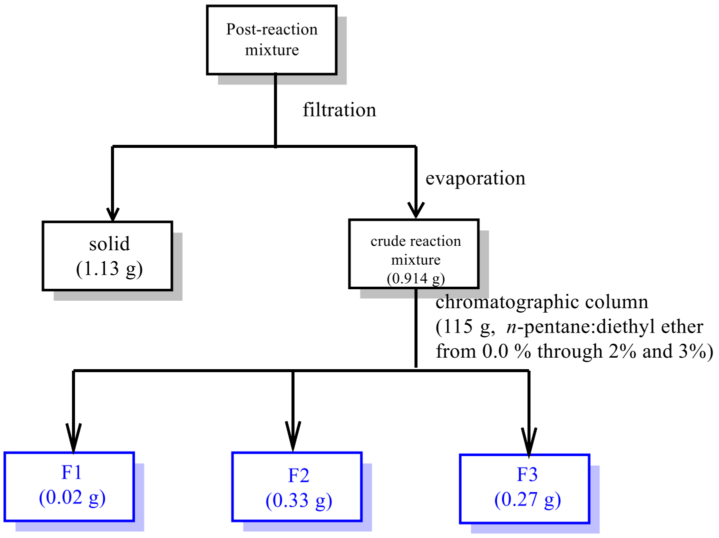
Reactor load:

| Mode i) 1.5 equiv dithiol reagent | Mode ii) 2.0 equiv dithiol reagent |
|---|---|
| **Hydroxyacetone tosylate**: 1.10 g; 0.85 mL; 4.8 mmol.<br>**Propane-1,3-dithiol:** 0.78 g; 0.72 mL; 7.2 mmol.<br>Dry DCM: 30 mL. | **Hydroxyacetone tosylate**: 1.10 g; 0.85 mL, 4.8 mmol.<br>**Propane-1,3-dithiol:** 1.04 g; 1.1 mL, 9.6 mmol.<br>Dry DCM: 30 mL. |

*General procedure*

To a two-necked round-bottom flask (100 mL) equipped with a magnetic stirrer and a dropping funnel with a pressure equalizer the **hydroxyacetone tosylate** in dry DCM (15 mL) was added. Then, **propane-1,2-dithiol** in dry DCM (10 mL) was added dropwise to the reaction mixture (**Note**). The progress of the reaction was monitored by thin layer chromatography (TLC, *n*-hexane:ethyl acetate, 3:1). The reaction mixture was left overnight. The crystalline precipitate of TsOH was filtered off and washed with additional DCM (10 mL). An aqueous solution of saturated $NaHCO_3$ was added to the filtrate in portions (30 mL, 1:2) and the resulting mixture was stirred for 15 min. After separating the layers, the aqueous phase was extracted with DCM (30 mL). The combined organic layers were dried over anhydrous sodium sulfate. After filtering off the drying agent, volatiles were removed under reduced pressure. The crude reaction mixture were obtained in the form of an oil. Products was isolated and purified by column chromatography. Flowsheet 3.3.1 shows isolation and purification of three main products.

**Note:Propane-1,2-dithiol** was added over 20 min. During the dropwise addition of dithiol reagent a precipitate of PTSA occurred.



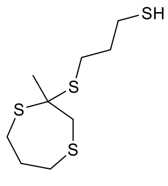
*Legend:*

*Fractions was identified: F1 as a product **7**; F2 as a product **8** and F3 as a product **9**.*

Flowsheet 3.3.1 Scheme of isolation of products **7**, **8** and **9** in mode i) 1.5 equiv dithiol reagent.

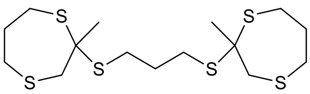
Isolation of the main products in mode ii) (2.0 equiv dithiol reagent) was carried out analogously such as mode i) and delivered product **7** (0.61 g) as a oil; product **8** (0.04g) and product **9** (0.03 g) as a oil.

Spectral data of **3-[(2-methyl-1,4-dithiepan-2-yl)sulfanyl]propane-1-thiol**, **7**, (mode i) 0.02g, 1.6% yield; mode ii) 0.61 g, 50% yield).



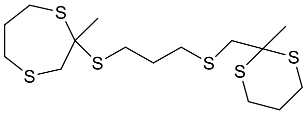
IR (ATR) ($cm^{-1}$): $\tilde{\nu}$=2955; 2916; 2843; 2537 (S-H); 1439; 1408; 1367; 1342; 1305; 1253; 1218; 1060; 907; 844; 730; 686.
$^1H$ NMR (500 MHz, $CDCl_3$): δ=3.29-3.22 (m, 1H, $SCSCH\mathit{H}_{ax.}$); 3.04 (s, 2H, $CH_3CC\mathit{H}_2$); 3.02-2.97 (m, 1H, $SCSCH\mathit{H}_{eq.}$); 2.95-2.89 (m, 1H, $CH_3CCH_2SCH\mathit{H}_{ax.}$); 2.83-2.77 (2m, 3H, $C\mathit{H}_2SCCH_2SCH\mathit{H}_{eq.}$); 2.68-2.64 (m, 2H, $C\mathit{H}_2SH$); 2.13-1.99 (m, 2H, $CH_3CCH_2SCH_2C\mathit{H}_2$); 1.93 (p, *J* 7.0, 2H, $C\mathit{H}_2CH_2SH$); 1.66 (s, 3H, $C\mathit{H}_3$); 1.41 (t, *J* 8.1, 1H, S*H*) ppm.
$^{13}C$ NMR (125 MHz, $CDCl_3$): δ=60.8; 52.0; 34.1; 33.1; 31.7; 29.1 (2*C*); 27.6; 23.7 ppm.
MS (EI+), *m/z* (%): 254 ($[M]^+$, 5); 149 (12); 148 (14); 147 (100); 133 (5); 106 (49); 74 (10); 73 (21); 60 (13); 46 (11); 42 (17).
HRMS for $C_9H_{18}S_4$ calcd: 254.0291; obsd: 254.0292.

Spectral data of **2,2'-[propane-1,3-diyldi(sulfanediyl)]bis(2-methyl-1,4-dithiepan)**, **8** (mode i) 0.33 g, 34% yield; mode ii) 0.04 g, yield 4%).



IR (ATR) ($cm^{-1}$): $\tilde{\nu}$=2953; 2914; 2851; 1438; 1408; 1366; 1305; 1253; 1217; 1059; 889; 844; 731; 686.
$^1H$ NMR (500 MHz, $CDCl_3$): δ=3.28-3.22 (m, 2H, $2xSCSCH\mathit{H}_{ax.}$); 3.03 (s, 4H, $2xCH_3CC\mathit{H}_2$); 3.02-2.96 (m, 2H, $2xSCSCH\mathit{H}_{eq.}$); 2.94-2.89 (m, 2H, $2xCH_3CCH_2SCH\mathit{H}_{ax.}$); 2.83-2.75 (m, 6H, $2xC\mathit{H}_2SCCH_2SCH\mathit{H}_{eq.}$); 2.13-1.99 (m, 4H, $2xCH_3CCH_2SCH_2C\mathit{H}_2$); 1.95 (p, *J* 7.3, 2H, $C\mathit{H}_2(CH_2SCCH_3)_2$); 1.65 (s, 6H, $2xC\mathit{H}_3$) ppm.
$^{13}C$ NMR (125 MHz, $CDCl_3$): δ=60.9 (2*C*); 52.1 (2*C*); 34.3 (2*C*); 31.9 (4*C*); 29.3 (2*C*); 29.3 (2*C*); 28.9 ppm.
HRMS for $C_{15}H_{28}S_6Ag$ calcd: 508.9562; obsd: 508.9522.

Spectral data of **2-methyl-2-[(3-{[(2-methyl-1,3-dithian-2-yl)methyl]sulfanyl}propyl) sulfanyl]-1,4-dithiepane**, **9** (mode i) 0.27 g, 28% yield; mode ii) 0.03 g, yield 3%).

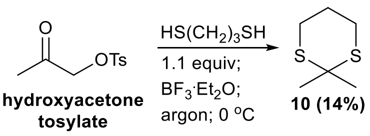


IR (ATR) ($cm^{-1}$): $\tilde{\nu}$=2950; 2908; 2853; 1438; 1423; 1409; 1366; 1305; 1275; 1252; 1217; 1140; 1060; 906; 884; 728; 674.
$^1H$ NMR (500 MHz, $CDCl_3$): δ=3.28-3.22 (m, 1H, $C(CH_3)SCH\mathit{H}_{ax.}$ (7-membered ring)); 3.18 (s, 2H, $CH_3C$(6-membered ring)$C\mathit{H}_2$); 3.03 (s, 2H, $C(CH_3)C\mathit{H}_2$, (7-membered ring); 3.02-2.89 (m, 4H, $C(CH_3)SCH\mathit{H}_{eq.}$ (7-membered ring) and $2xSCH\mathit{H}_{ax.}$ (6-membered ring) and $CH_2SCH\mathit{H}_{ax.}$ (7-membered ring)); 2.83-2.73 (m, 7H, $CH_2SCH\mathit{H}_{eq.}$ (7-membered ring) and $2xSCH\mathit{H}_{eq.}$ (6-membered ring) and $CH_3CSC\mathit{H}_2CH_2C\mathit{H}_2SCH_2CCH_3$); 2.13-1.86 (m, 6H, $3xSCH_2C\mathit{H}_2$); 1.67 (s, 3H, C(6-membered ring)$C\mathit{H}_3$); 1.66 (s, 3H, C(7-membered ring)$C\mathit{H}_3$) ppm.

$^{13}$C NMR (125 MHz, $CDCl_3$): δ=60.8; 52.0; 49.3; 44.0; 34.1; 33.4; 31.8; 29.4; 29.1 (2*C*); 28.2; 27.5; 26.8 (2*C*); 24.8 ppm.
MS (EI+), *m/z* (%): 400 ($[M]^+$, 0.3), 253 (37), 149 (15), 148 (14), 147 (100), 133 (63), 107 (8), 106 (10), 105 (5), 73 (21), 60 (13), 46 (9), 42 (13).
HRMS for $C_{15}H_{28}S_6$ calcd: 400.0515; obsd: 400.0521.

### *3.4 Dithioketalization of* ***hydroxyacetone tosylate*** *with* ***propane-1,3-dithiol*** *in an atmosphere of argon*

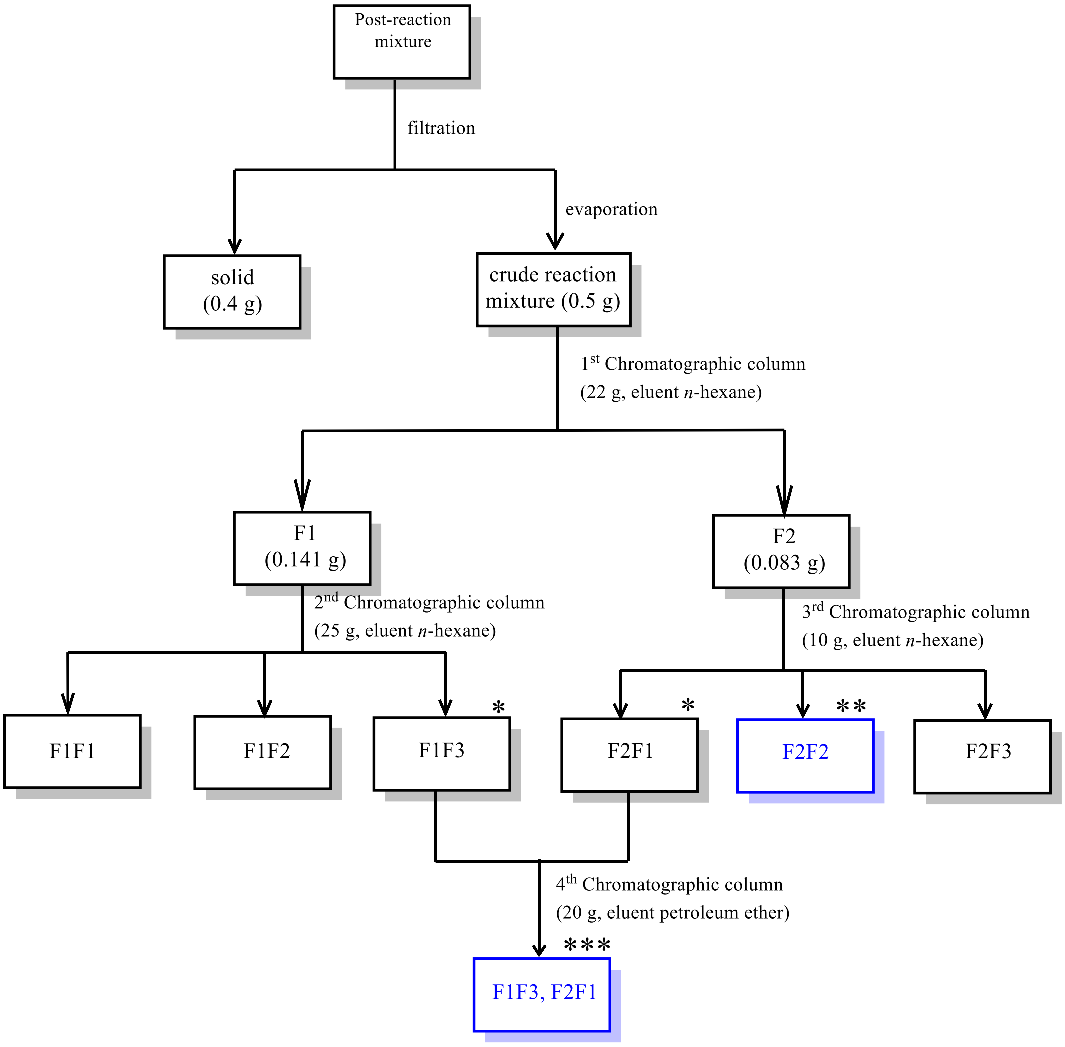
### *3.4.1 Dithioketalization of* ***hydroxyacetone tosylate*** *with catalyst and 1.1 equiv dithiol reagent and argon atmosphere*



Under an atmosphere of argon in heat gun dry a two-necked round-bottom flask (250 mL) equipped with a magnetic stirrer and a dropping funnel with a pressure equalizer the **hydroxyacetone tosylate** in dry DCM (0.91 g; 3.99 mmol; in 10 mL of DCM) was added. Reaction mixture was cooled to 0 °C in ice bath and **boron trifluoride etherate** (0.62 g; 0.54 mL; 4.4 mmol; 1.1 eqiuv) was added dropwise followed by the dropwise addition of **propane-1,3-dithiol** (0.47 g; 0.44 mL; 4.34 mmol; 1.1 equiv) in DCM (20 mL) (**Note 1**.). The progress of the reaction was monitored by thin layer chromatography (TLC, *n*-hexane:ethyl acetate, 4:1). The reaction mixture was kept at a 0 °C for 20 min., then water bath was removed (**Note 2.**). The reaction mixture was left to stir for 24 hours. Then, a mixture of saturated solution of $NaHCO_3$ (10 mL) and water (20 mL) were added to the reaction mixture and the resulting mixture was stirred for 20 min. After separating the layers, the aqueous phase was extracted with DCM (30 mL). The combined organic layers were dried over anhydrous sodium sulfate. After filtering off the drying agent, volatiles were removed under reduced pressure. The crude reaction mixture was obtained in the form of an oil (0.89 g) **(Note 3.)**. Flowsheet 3.4.1 shows isolation and purification of main product. Product **10** was obtained as an oil (0.08 g, 14%).

**Note:**

1. **Propane-1,2-dithiol** was added over 45 min.
2. After warming the reaction mixture to room temperature, the reaction changes color from colorless to yellow.
3. After standard work-up a precipitate of PTSA occurred.



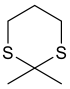
* - The $^{1}$H-NMR spectra of fractions F1F3 and F2F1 showed the presence of product **10**,
** - The $^{1}$H-NMR spectrum of the F2F2 fraction showed chromatographically pure product **10**,
***** -** The 1H-NMR spectrum of the F1F3, F2F1 fractions showed chromatographically pure product **10**.

Fractions F2F2 and F1F3, F2F1 were combined and labeled as product **10**.

Flowsheet 3.4.1 Scheme isolation and purification of product **10**.

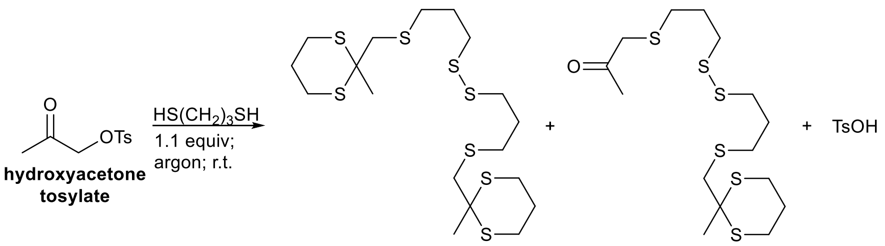
Spectral data of **2,2-dimethyl-1,3-dithiane**, **10** (0.08 g, yield 14%):

$^{1}$H-NMR spectral data for **10** are consistent with the literature [11].



$^{1}$H-NMR (500 MHz, CDCl$_3$): δ=2.89 (t$_{pseudo}$, $J$ 5.7, 4H, 2xSC$H_2$); 1.99-1.95 (m, 2H, SCH$_2$C$H_2$); 1.70 (s, 6H, 2xC$H_3$).
$^{13}$C-NMR (125 MHz, CDCl$_3$): δ=45.3; 30.8 (2$C$); 27.1 (2$C$); 25.2.

### 3.4.2 *Dithioketalization of* **hydroxyacetone tosylate** *with out catalyst and 1.1 equiv dithiol reagent and argon atmosphere*



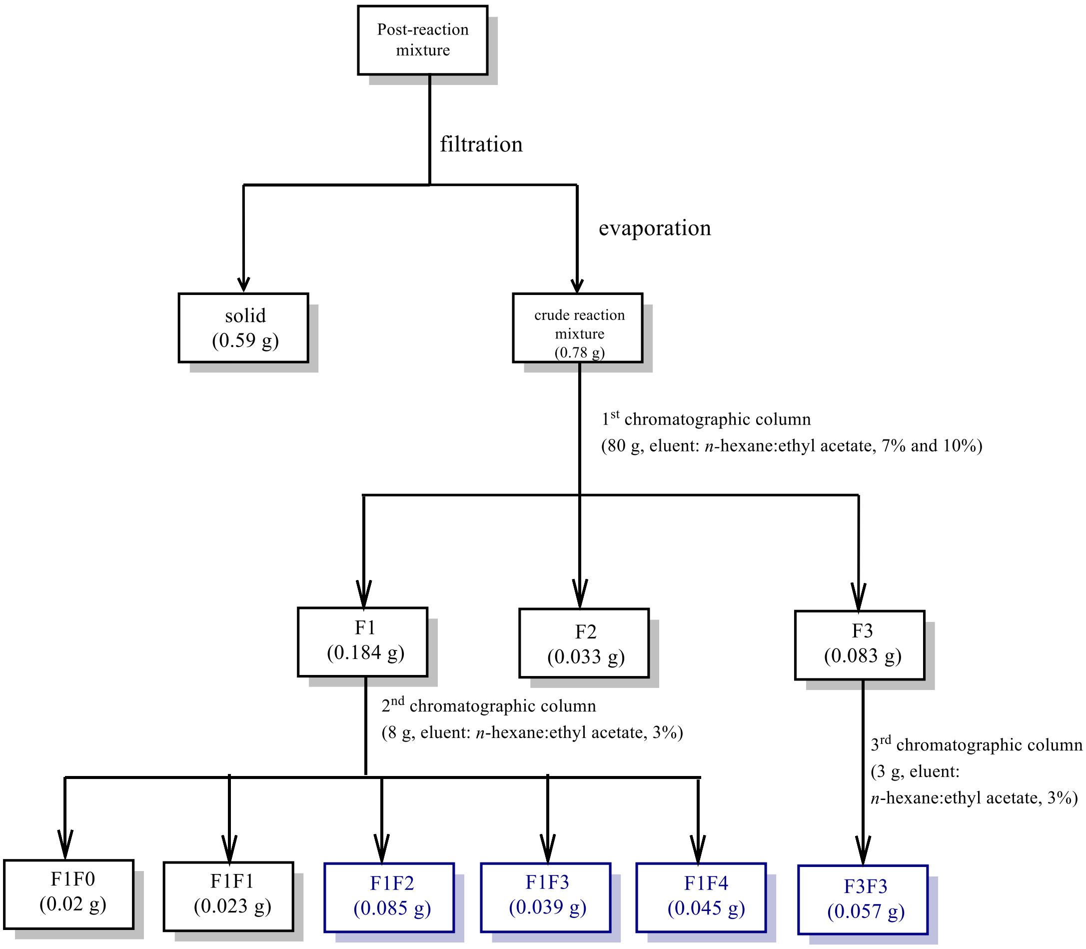
**11 (17%)** **12 (7%)** **13**

Under an atmosphere of argon in heat gun dry a two-necked round-bottom flask (50 mL) equipped with a magnetic stirrer and a dropping funnel with a pressure equalizer the **hydroxyacetone tosylate** in dry DCM (0.92 g; 4.03 mmol in 10 mL) was added. Then, it was added dropwise to the reaction mixture **propane-1,3-dithiol** (0.49 g; 0.45 mL; 4.53 mmol; 1.1 equiv) in dry DCM (5 mL) **(Note)**. The progress of the reaction was monitored by thin layer chromatography (TLC, *n*-hexane:ethyl acetate, 3:1). The reaction mixture was left overnight. The crystalline precipitate of TsOH (0.59 g) was filtered off and washed with additional DCM (10 mL). Then, a mixture of saturated solution of $NaHCO_3$ (10 mL) and water (20 mL) were added to the filtrate, and the resulting mixture was stirred for 20 min.. After separating the layers, the aqueous phase was extracted with methylene chloride (30 mL). The combined organic layers were dried over anhydrous sodium sulfate. After filtering off the drying agent, volatiles were removed under reduced pressure. The crude reaction mixture were obtained in the form of an oil (0.78 g).

Flowsheet 3.4.2. illustrates the isolation of the main products. Chromatographically pure product **11** (0.17 g; 17%) and product **12** (0.06 g; 7%) was obtained as an oil.

**Note:**

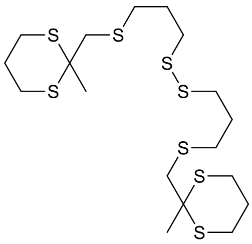
After approximately 5 min., during the dropwise addition of **propane-1,3-dithiol**, a precipitate of PTSA occured.



*Legend:*
*The $^1$H-NMR spectra of the F1F2, F1F3 and F1F4 fractions were the same. These fractions were combined and labeled as product **11**. F3F3 fraction, chromatographically homogeneous in the confirmed eluent system (TLC, n-hexane:ethyl acetate, 3:1), was labeled as product **12**.*

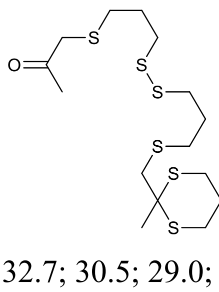
Flowsheet 3.4.2 Scheme of isolation of main products **11** and **12** from the reaction mixture.

Spectral data of **2,2'-[disulfanediylbis(propane-3,1-diylsulfanediyl)]bis(2-methyl-1,3-dithiane)**, **11** (0.17 g; yield 17%):



IR (ATR) (cm$^{-1}$): $\tilde{\nu}$=2904; 2850; 2826; 1436; 1421; 1365; 1276; 1238; 1140; 1065; 998; 906; 867; 852; 815; 728; 704; 673; 658.
$^1$H-NMR (500 MHz, CDCl$_3$): δ=3.18 (s, 4H, 2xCH$_3$CC*H*$_2$); 2.96-2.90 (m, 4H, 4xSCH*H*$_{ax.}$); 2.81-2.74 (m, 12H, 4xSCH*H*$_{eq.}$ and (SC*H*$_2$CH2C*H*$_2$S)$_2$); 2.08-1.99 (m+p, 2H+4H, *J* 7.1, 2xSCH2CH*H*$_{ax.}$ and (SCH$_2$C*H*$_2$)$_2$); 1.95-1.86 (m, 2H, 2xSCH2CH*H*$_{ax.}$); 1.67 (s, 6H, 2xC*H*$_3$).
$^{13}$C-NMR (125 MHz, CDCl$_3$): δ=49.4 (2*C*); 44.1 (2*C*); 37.2 (2*C*); 32.7 (2*C*); 29.0 (2*C*); 27.6 (2*C*), 26.8 (4*C*); 24.8 (2*C*).
MS (EI+); *m/z* (%): 506 ([M]$^+$, 0.4); 253 (19); 149 (7); 148 (7); 147 (76); 135 (10); 134 (8), 133 (100); 107 (8); 106 (11); 105 (5); 73 (16); 60 (12); 46 (8); 42 (13).
HRMS for $C_{18}H_{34}S_8$: calcd: 506.0426; obsd: 506.0411.

Spectral data of **1-({3-[(3-{[(2-methyl-1,3-dithian-2-yl)methyl]sulfanyl}propyl)disulfanyl] propyl}sulfanyl)propan-2-one**, **12** (0.06 g; yield 7%):



IR (ATR) (cm$^{-1}$): $\tilde{\nu}$=2915; 2850; 2829; 1702 (C=O); 1421; 1354; 1294; 1277; 1236; 1147; 1065; 999; 906; 729; 705; 673.

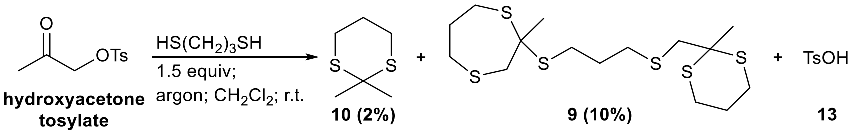
$^{1}$H-NMR (500 MHz, CDCl$_3$): δ=3.23 (s, 2H, C(O)C$H_2$); 3.18 (s, 2H, CH$_3$CC$H_2$); 2.96-2.90 (m, 2H, 2xSCH$H_{ax.}$); 2.81-2.74 (m, 8H, (SC$H_2$CH$_2$C$H_2$S)$_2$); 2.61 (t$_{pseudo}$, 2H, *J* 7.1, 2xSCH$H_{eq.}$); 2.30 (s, 3H, C(O)C$H_3$); 2.08-1.86 (m, 6H, 3xSCH2C$H_2$); 1.67 (s, 3H, C$H_3$).

$^{13}$C-NMR (125 MHz, CDCl$_3$): δ=203.7 (*C*=O); 49.4; 44.1; 41.8; 37.2; 37.1; 32.7; 30.5; 29.0; 28.1; 27.8; 27.6; 26.8 (2*C*); 24.8.

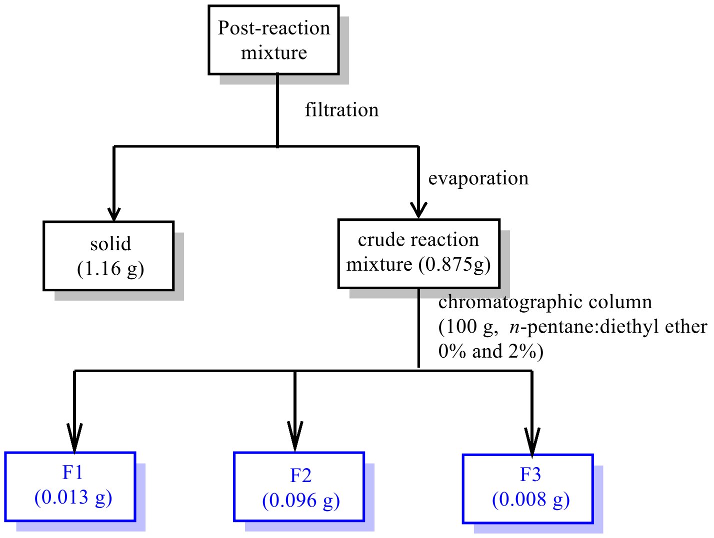
MS (EI+); *m/z* (%): 416 ([M]$^+$, 0.4); 253 (21); 163 (12); 149 (6); 148 (6);147 (69); 135 (11); 134 (8); 133 (100); 107 (7); 106 (15); 105 (6); 73 (19); 60 (9); 46 (8); 42 (12).

HRMS for C$_{15}$H$_{28}$OS$_6$: calcd: 416.0464; obsd: 416.0463.

### *3.4.3 Dithioketalization of* **hydroxyacetone tosylate** *without catalyst and 1.5 equiv dithiol reagent and argon atmosphere*

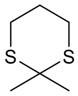


To a two-necked round-bottom flask (100 mL) heated in a stream of argon then cooled to room temperature, equipped with a magnetic stirrer and a dropping funnel with a pressure equalizer the **hydroxyacetone tosylate** (1.10 g; 0.85 mL; 4.8 mmol) in dry DCM (10 mL) was added. **Propane-1,3-dithiol** (0.77 g, 0.72 mL, 7.12 mmol, 1.5 equiv) in DCM (15 mL) was added dropwise to the reaction mixture. The progress of the reaction was monitored by thin layer chromatography (TLC, *n*-pentane:ethyl acetate, 9:1). The reaction mixture was left overnight. The crystalline precipitate of TsOH (1.16 g) was filtered off and washed with additional DCM (10 mL). Then, a mixture of saturated solution of NaHCO$_3$ (10 mL) and water (20 mL) were added to the filtrate and the resulting mixture was stirred for 25 min.. After separating the layers, the aqueous phase was extracted with methylene chloride (2x15 mL). The combined organic layers were dried over anhydrous sodium sulfate. After filtering off the drying agent, volatiles were removed under reduced pressure. The crude reaction mixture were obtained in the form of an oil (0.875 g). The oil was colorless in the beginning, during workup and storage it has changed color from colorless to almost black. Mixture of product was isolated and purified by column chromatography. The flowsheet 3.4.3 shows the isolation of the main products **9** and **10** and trace of **13**.

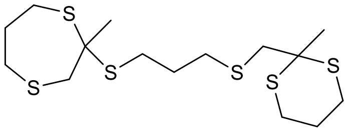


*Legend:*
*Fractions was identified: F1 as a product **10**; F2 as a product **9** and F3 as a TsOH, **13**.*

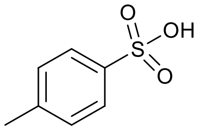
Flowsheet 3.4.3 Scheme shows the isolation of the main products **9** and **10**.



Spectral data of **2,2-dimethyl-1,3-dithiane**, **10** (0.01 g; 2% yield) are consistent with the compound **10** obtained above (pkt 3.4.1.).



Spectral data of **2-methyl-2-((3-(((2-methyl-1,3-dithian-2-yl)methyl)thio)propyl)thio)-1,4-dithiepane**, **9** (0.10 g; 10% yield) are consistent with the compound **9** obtained above. (pkt 3.3.)



Spectral data of **4-methylbenzenesulfonic acid** are consistent with literature [12]:
$^1$H NMR: (500 MHz, acetone-$d_6$): δ=7.79 and 7.42 (2d, *J* 8.2, 4H, 4xAr*H*); 2.44 (s, 3H, C$H_3$).

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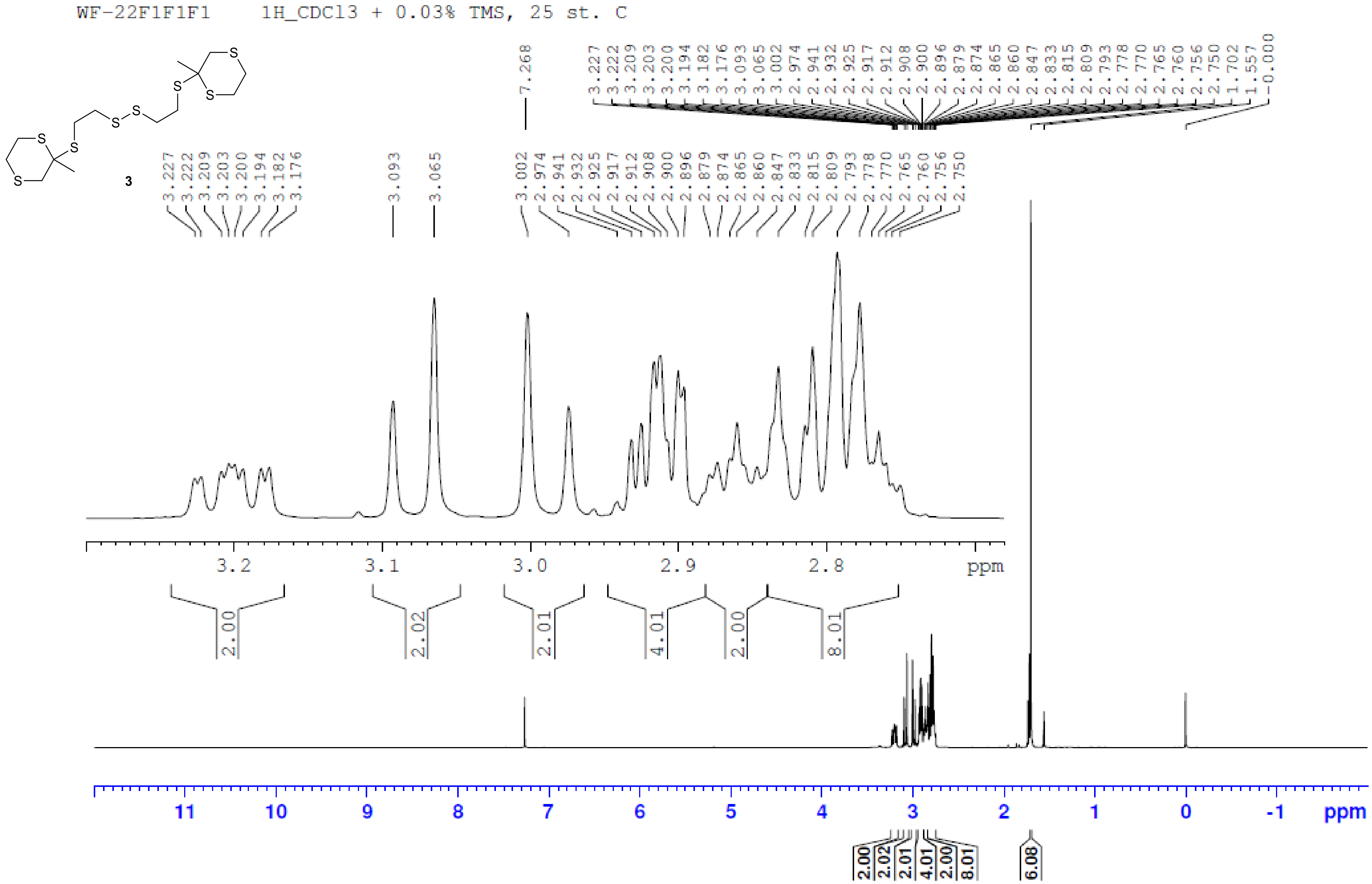
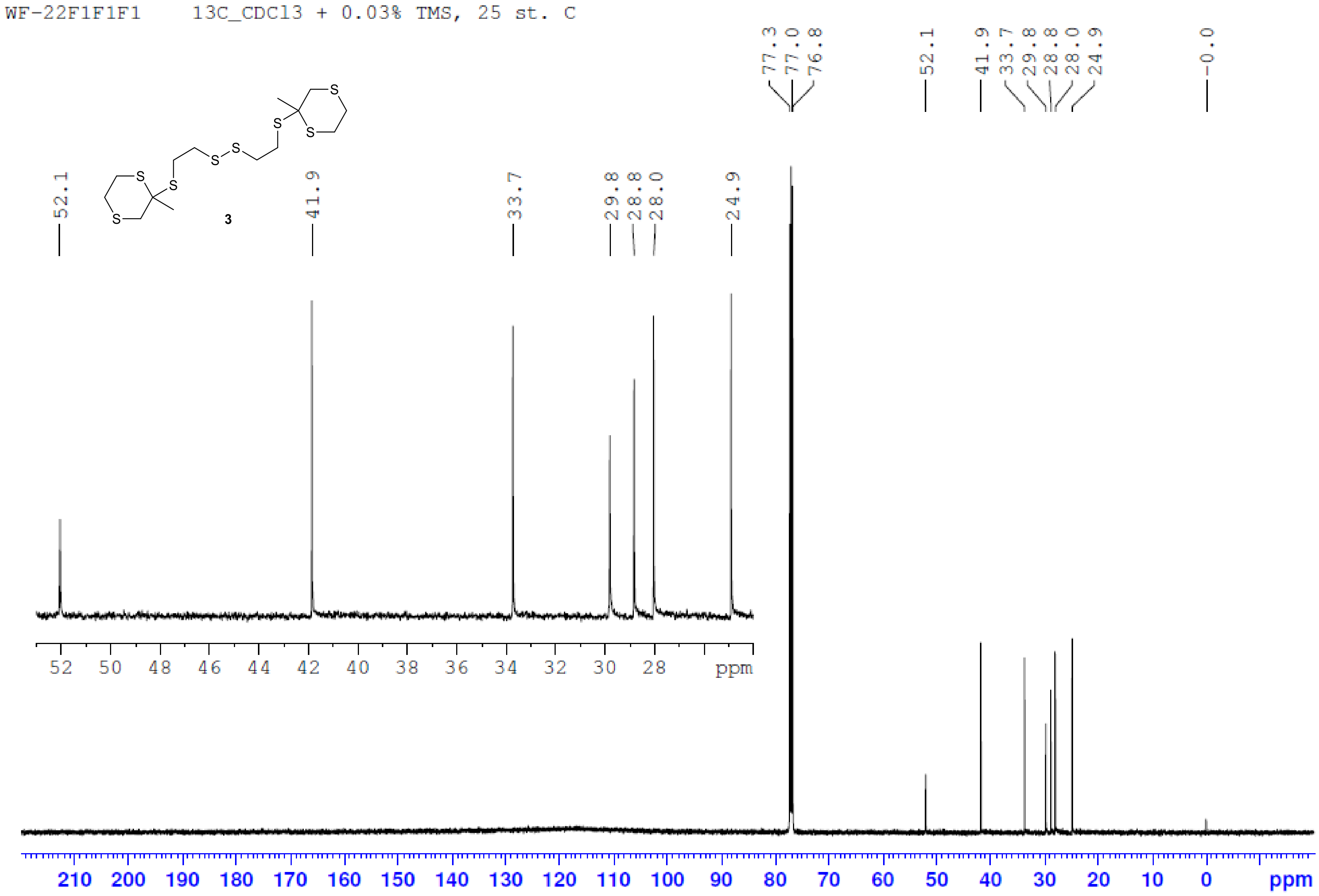
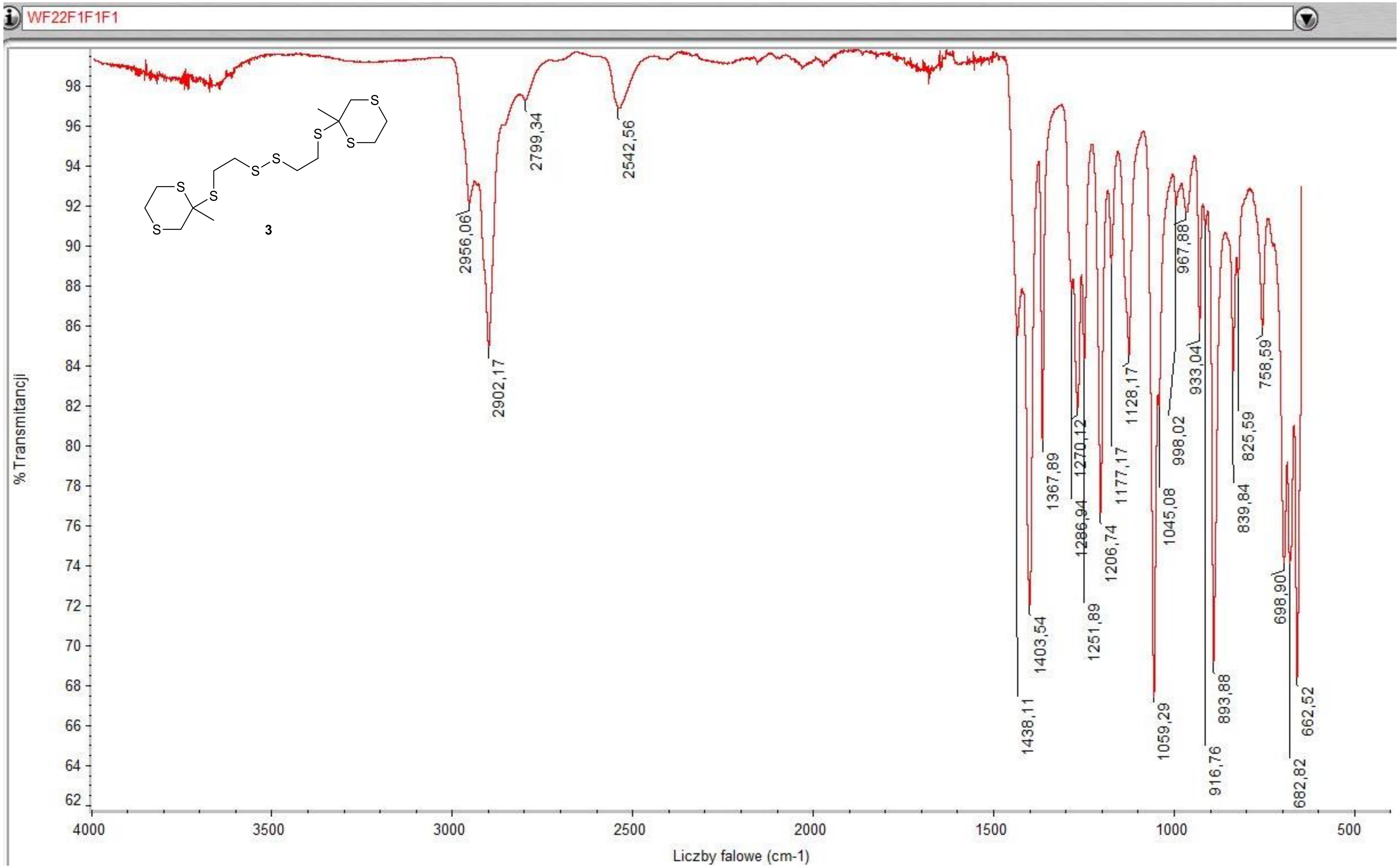
[11] Jnaneshwara, G.K.; Barhate, N.B.; Sudalai, A,; Deshpande, V.H.; R. D. Wakharkar, R.D.; Gajare, A.S. and 2 more, "Transdithioacetalization of acetals, ketals, oximes, enamines and tosylhydrazones catalysed by natural kaolinitic clay", *J. Chem. Soc. Perkin Transactions 1*, **1998**, *5*, 965-968. https://doi.org/10.1039/A706475F

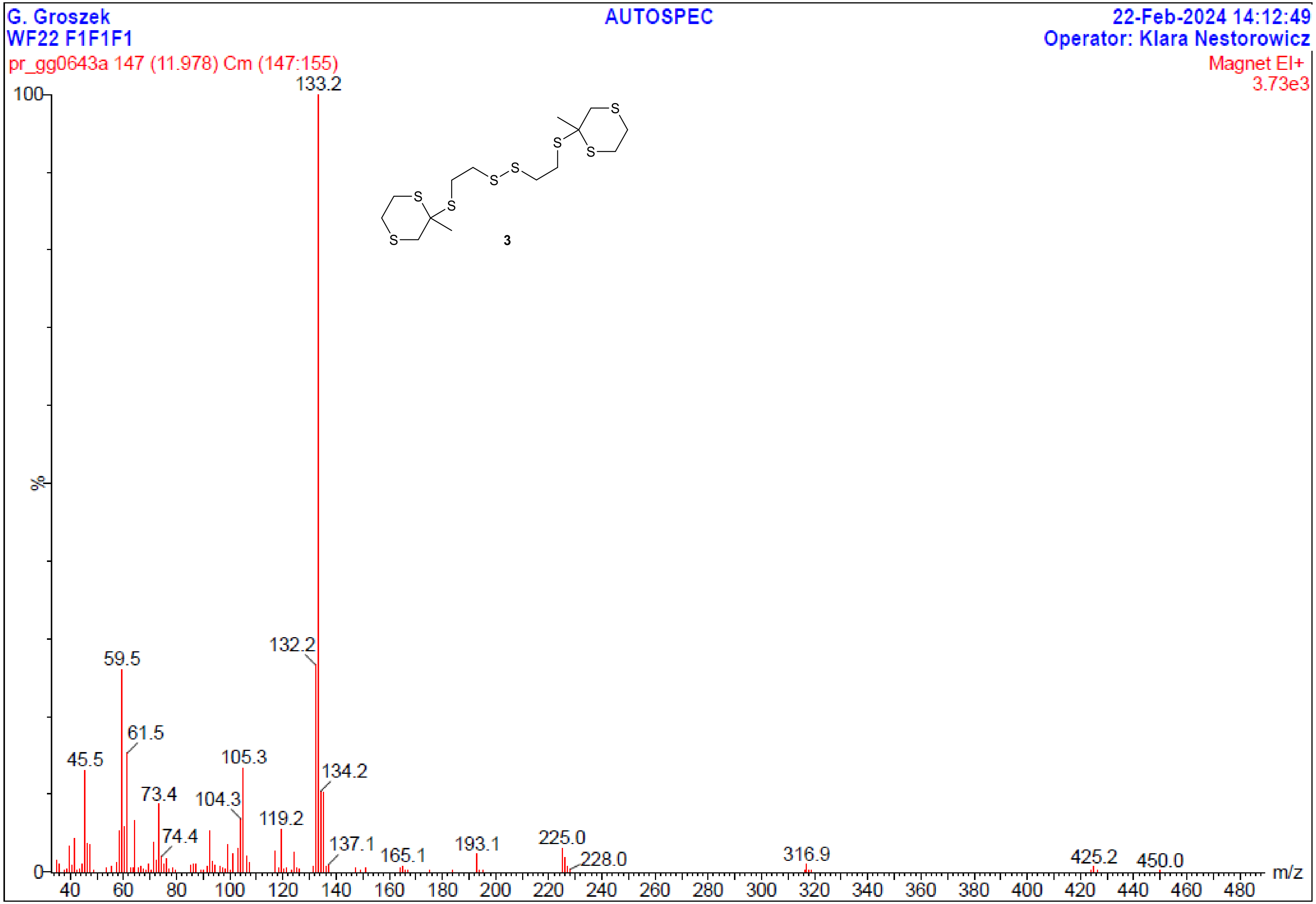
[12] Fieser, L.F. *Experiments in Organic Chemistry*, Boston. 3rd Ed., **1955**, 144.

## 5. Spectra $^1$H-, $^{13}$C-NMR, MS, HRMS and IR of obtained compounds

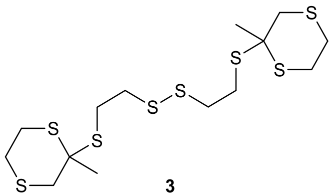



WF-22F1F1F1 13C_CDCl3 + 0.03% TMS, 25 st. C
77.3
77.0
76.8
52.1
41.9
33.7
29.8
28.8
28.0
24.9
-0.0
3
S
52 50 48 46 44 42 40 38 36 34 32 30 28 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


WF22F1F1F1
% Transmitancji
98
96
94
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
4000
3500
3000
2500
2000
1500
1000
500
Liczby falowe (cm-1)
3
2956,06
2902,17
2799,34
2542,56
1438,11
1403,54
1367,89
1286,94
1270,12
1251,89
1206,74
1177,17
1128,17
1059,29
1045,08
998,02
967,88
933,04
916,76
893,88
839,84
825,59
758,59
698,90
682,82
662,52


G. Groszek
WF22 F1F1F1
AUTOSPEC
22-Feb-2024 14:12:49
Operator: Klara Nestorowicz
pr_gg0643a 147 (11.978) Cm (147:155)
Magnet EI+
3.73e3
100
%
0
133.2
132.2
59.5
61.5
45.5
105.3
134.2
73.4
104.3
119.2
74.4
137.1
165.1
193.1
225.0
228.0
316.9
425.2
450.0
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440
460
480
m/z
S
3

# Elemental Composition Report



3

Page 1

## Single Mass Analysis

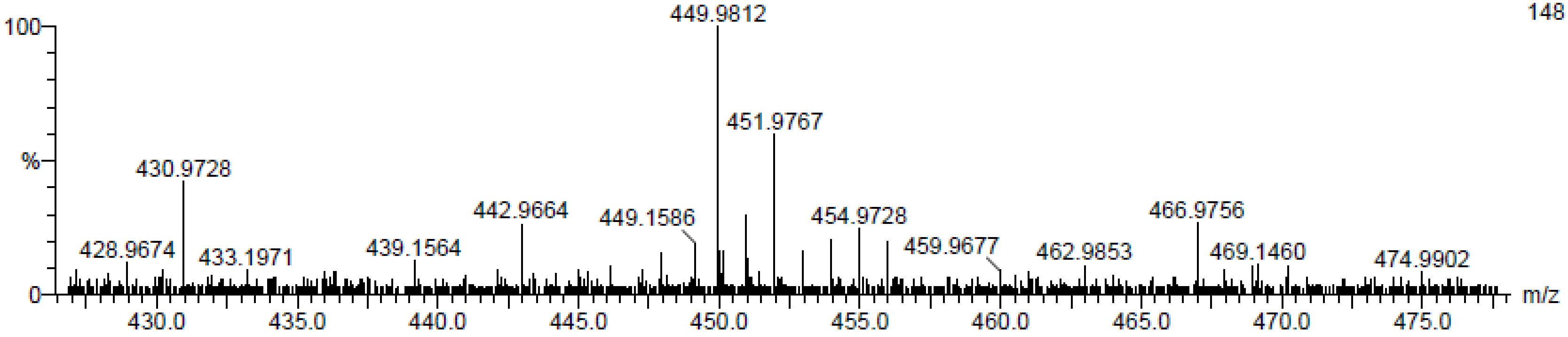
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 150.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
50 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-200 S: 0-11

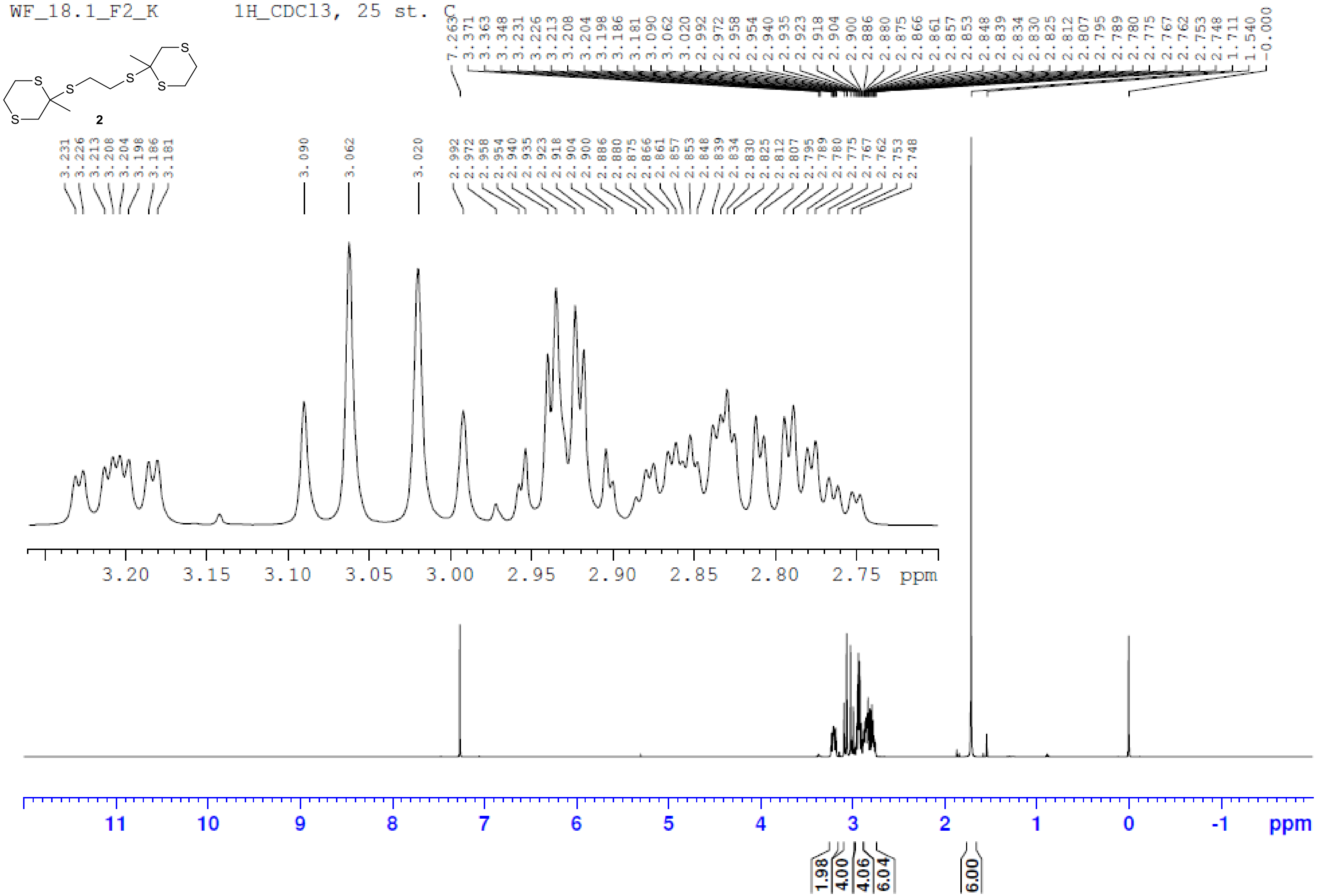
G. Groszek
WF22 F1F1F1
pr_gg0643h 143 (5.453) Cm (143:149)

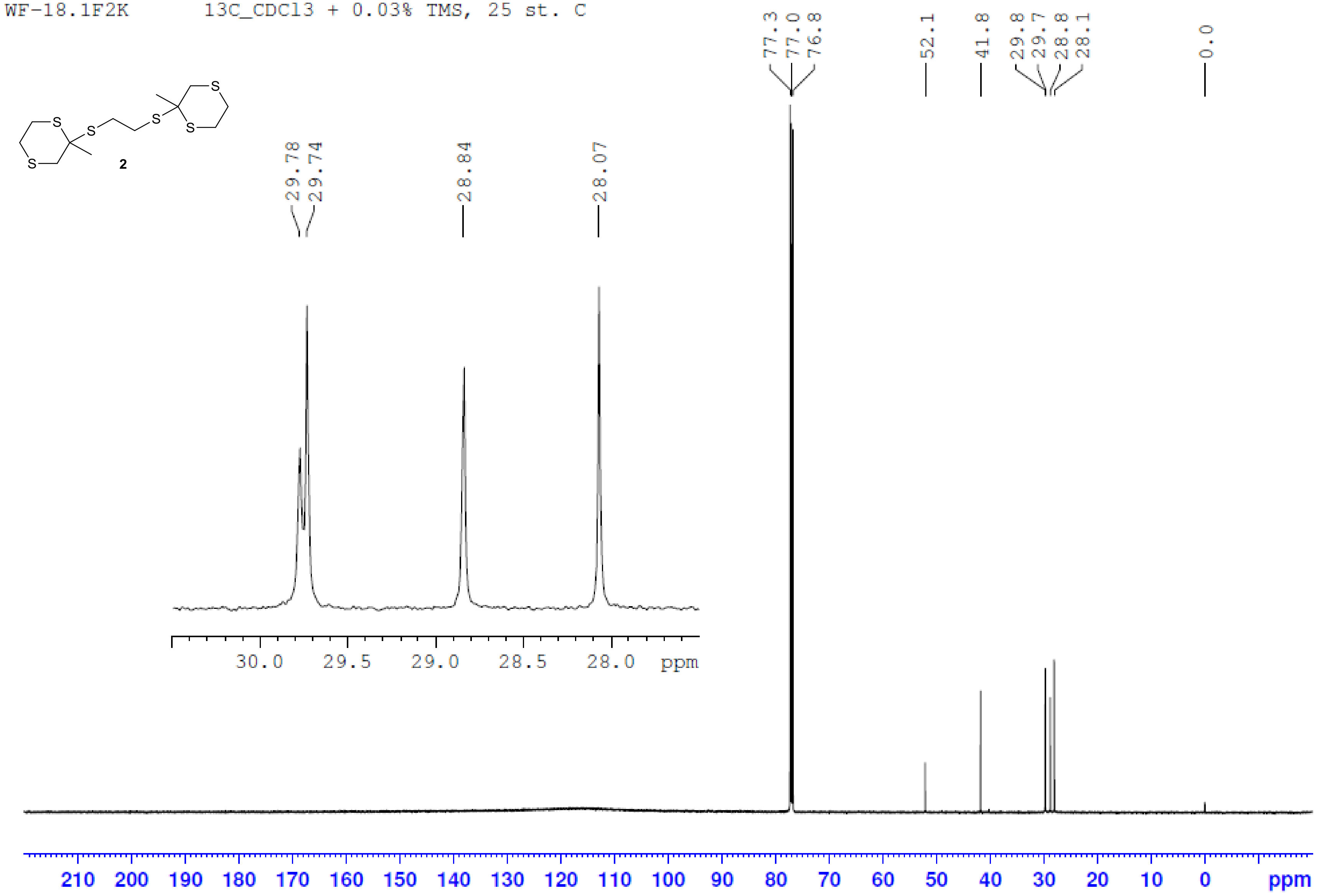
AUTOSPEC

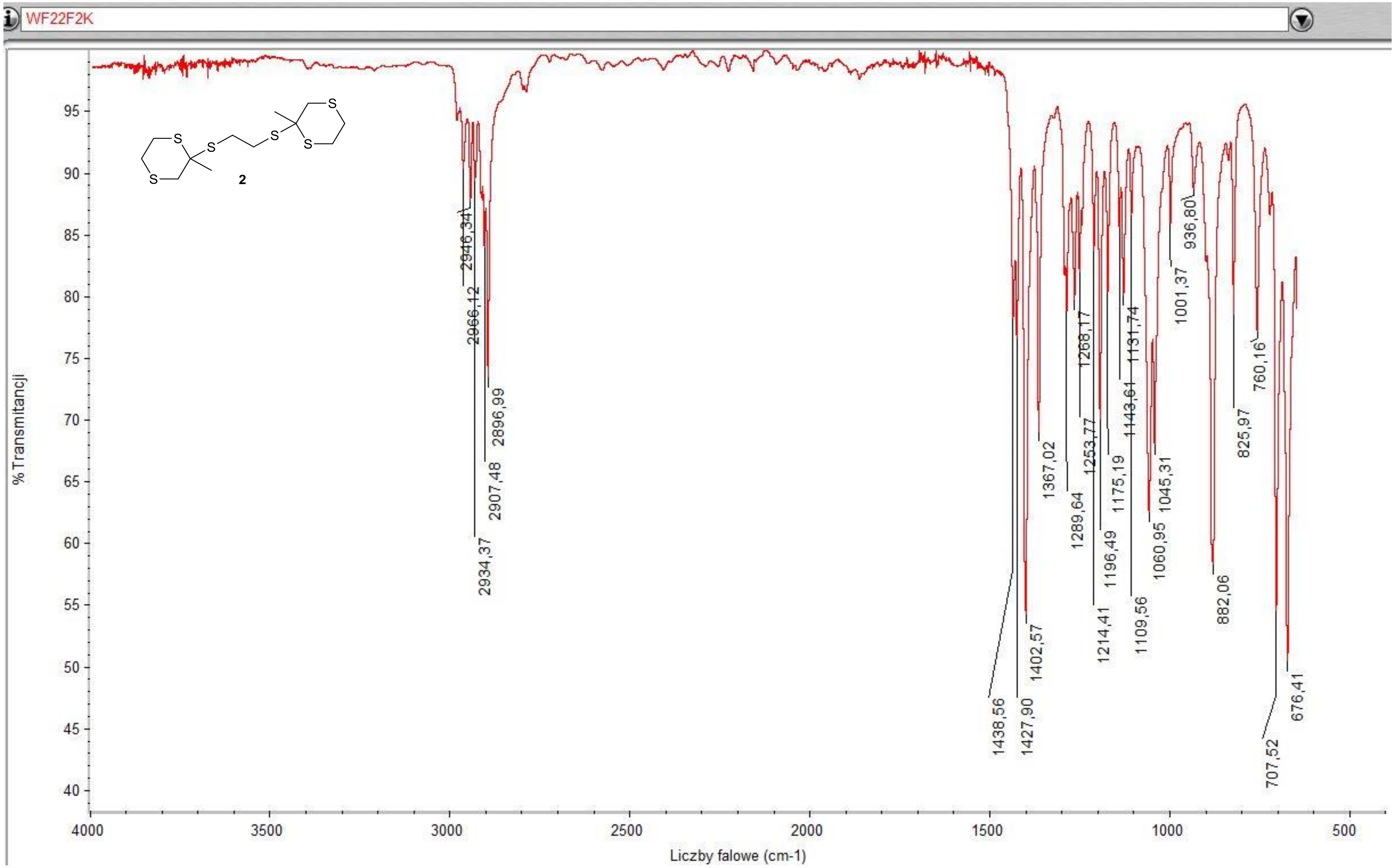
23-Feb-2024 14:22:59
Operator: Klara Nestorowicz
Voltage EI+
148

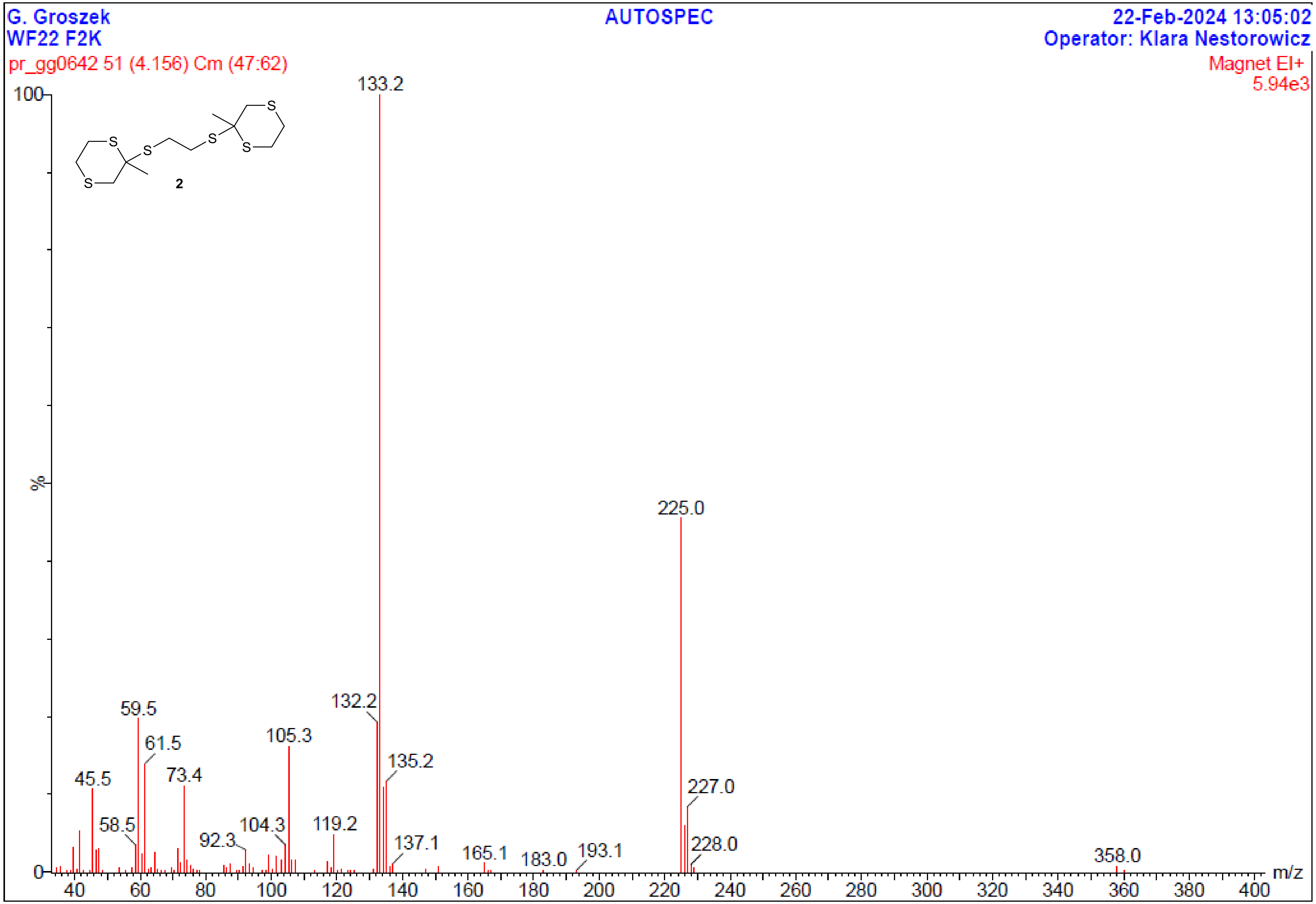


```
Minimum:                                   -1.5
Maximum:                  5.0      20.0    150.0

Mass       Calc. Mass     mDa      PPM     DBE       i-FIT      Formula

449.9812   449.9800       1.2      2.7     2.0       5.2        C14  H26  S8
           449.9767       4.5      10.0    7.0       6.9        C17  H22  S7
           449.9733       7.9      17.6    12.0      9.5        C20  H18  S6
```

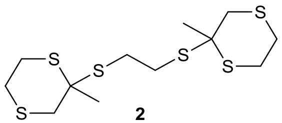

WF_18.1_F2_K
1H_CDCl3, 25 st. C
2
7.263 3.371 3.363 3.348 3.231 3.226 3.213 3.208 3.204 3.198 3.186 3.181 3.090 3.062 3.020 2.992 2.972 2.958 2.954 2.940 2.935 2.923 2.918 2.904 2.900 2.886 2.880 2.875 2.866 2.861 2.857 2.853 2.848 2.839 2.834 2.830 2.825 2.812 2.807 2.795 2.789 2.780 2.775 2.767 2.762 2.753 2.748 1.711 1.540 -0.000
3.231 3.226 3.213 3.208 3.204 3.198 3.186 3.181
3.090 3.062 3.020
2.992 2.972 2.958 2.954 2.940 2.935 2.923 2.918 2.904 2.900 2.886 2.880 2.875 2.866 2.861 2.857 2.853 2.848 2.839 2.834 2.830 2.825 2.812 2.807 2.795 2.789 2.780 2.775 2.767 2.762 2.753 2.748
3.20 3.15 3.10 3.05 3.00 2.95 2.90 2.85 2.80 2.75 ppm
11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
1.98 4.00 4.06 6.04
6.00


WF-18.1F2K
13C_CDCl3 + 0.03% TMS, 25 st. C
2
77.3
77.0
76.8
52.1
41.8
29.8
29.7
28.8
28.1
0.0
29.78
29.74
28.84
28.07
30.0
29.5
29.0
28.5
28.0
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


WF22F2K
2
% Transmitancji
Liczby falowe (cm-1)
95
90
85
80
75
70
65
60
55
50
45
40
4000
3500
3000
2500
2000
1500
1000
500
2946,34
2966,12
2934,37
2907,48
2896,99
1438,56
1427,90
1402,57
1367,02
1289,64
1268,17
1253,77
1214,41
1196,49
1175,19
1143,61
1131,74
1109,56
1060,95
1045,31
1001,37
936,80
882,06
825,97
760,16
707,52
676,41


G. Groszek
WF22 F2K
pr_gg0642 51 (4.156) Cm (47:62)
AUTOSPEC
22-Feb-2024 13:05:02
Operator: Klara Nestorowicz
Magnet EI+
5.94e3
2
100
%
0
133.2
225.0
59.5
61.5
45.5
73.4
58.5
92.3
104.3
105.3
119.2
132.2
135.2
137.1
165.1
183.0
193.1
227.0
228.0
358.0
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
m/z

## Elemental Composition Report



2

Page 1

## Single Mass Analysis

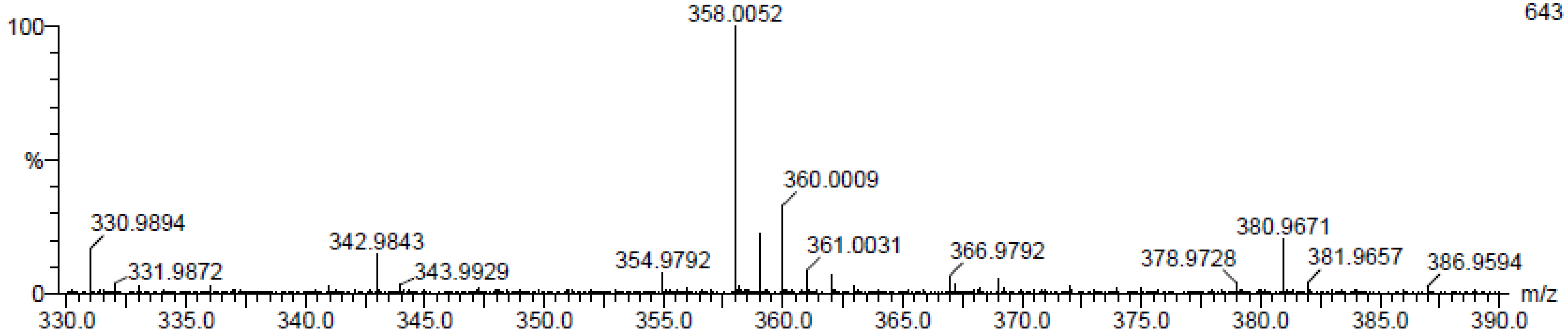
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 150.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
33 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-200 S: 0-8

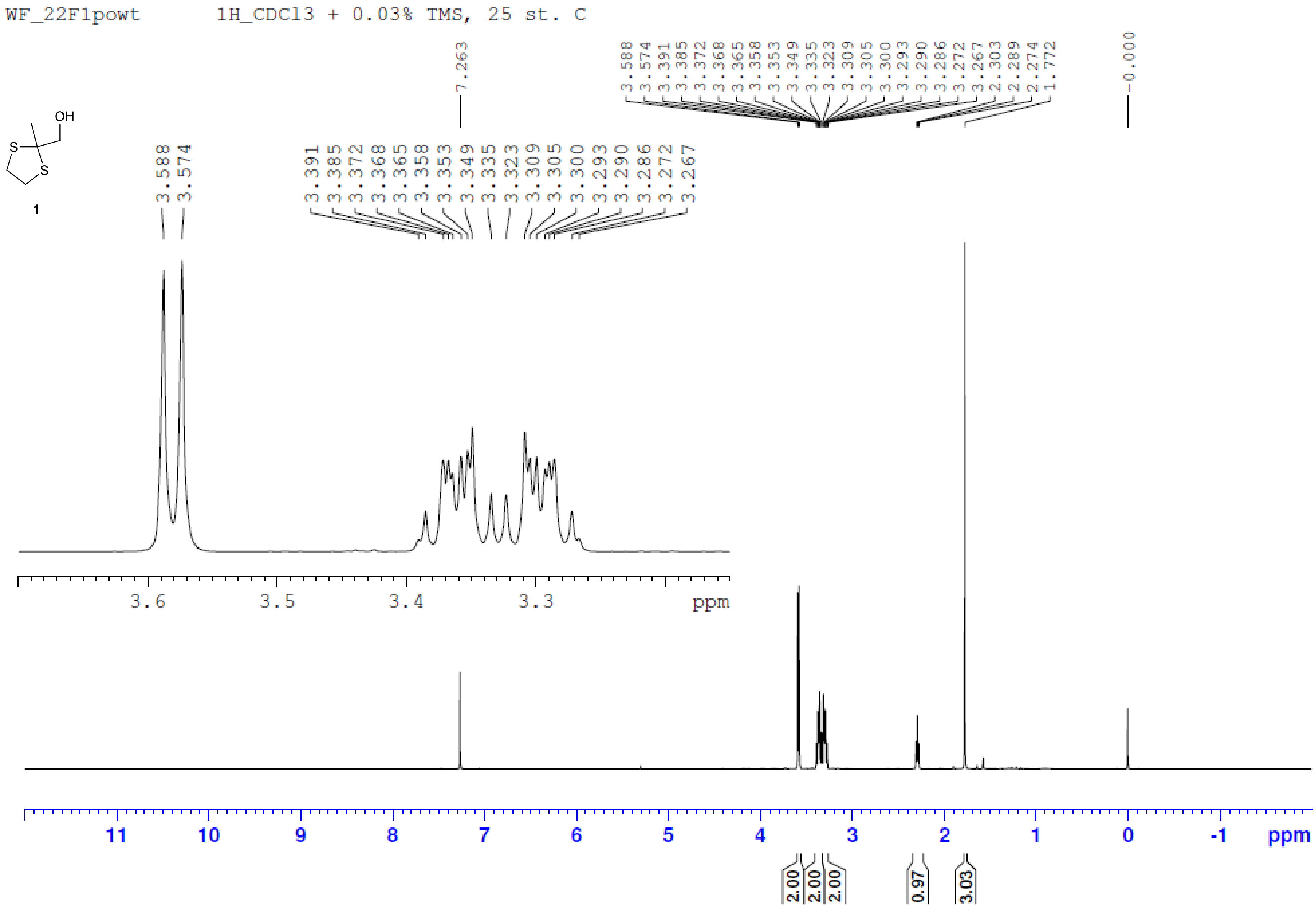
G. Groszek
WF22 F2K
pr_gg0642h 270 (10.298) Cm (268:281)

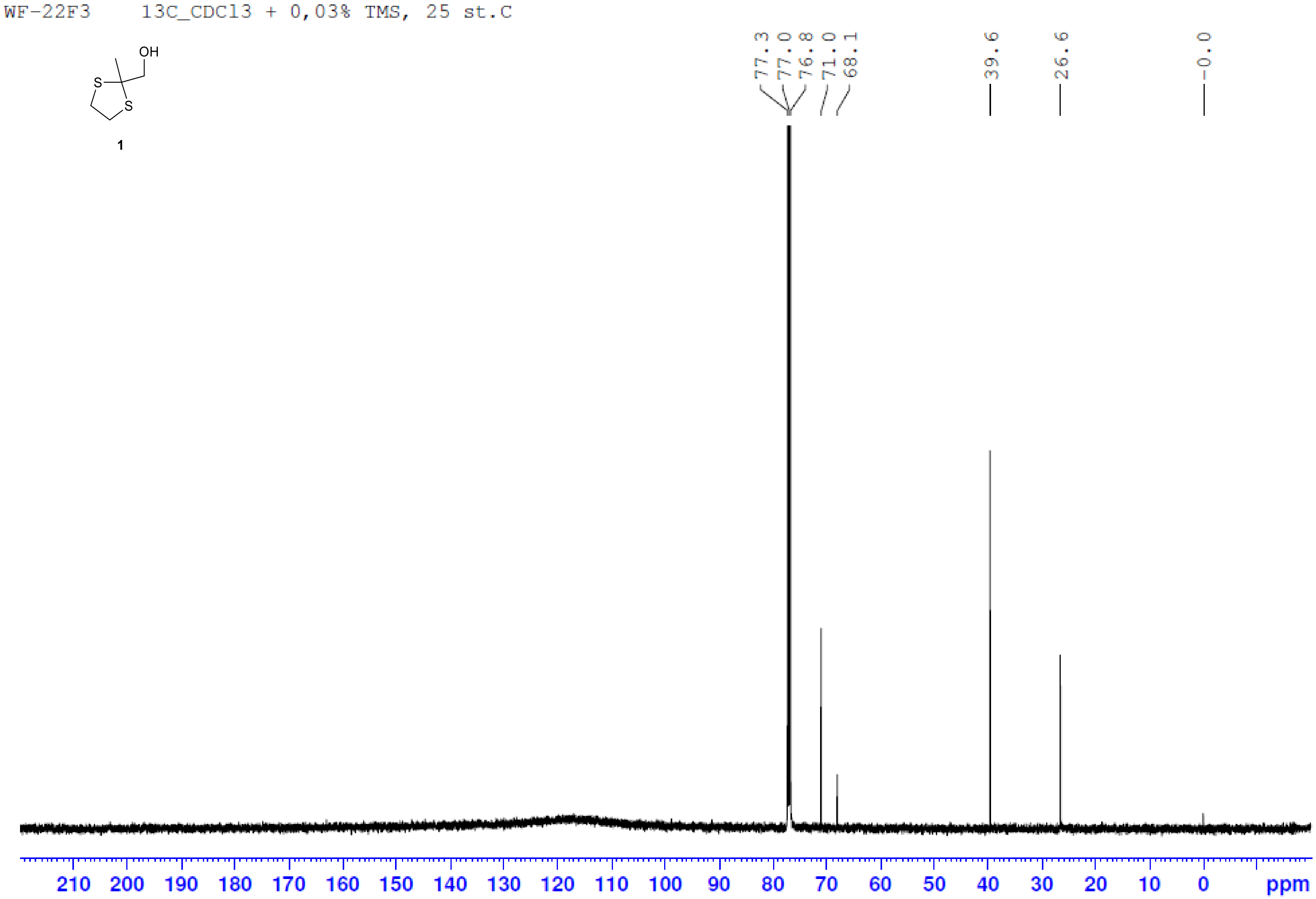
AUTOSPEC

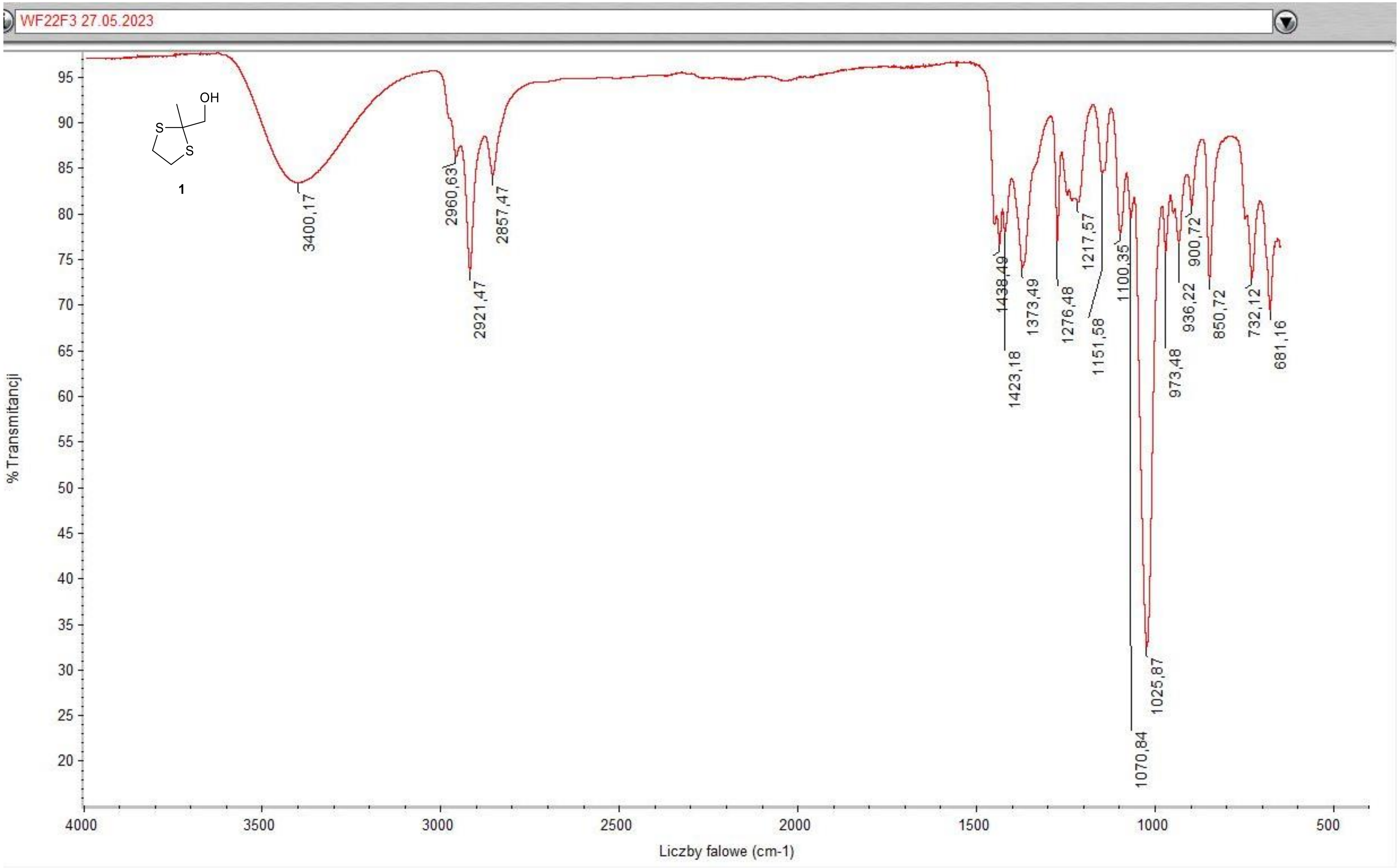
23-Feb-2024 13:50:10
Operator: Klara Nestorowicz
Voltage EI+
643

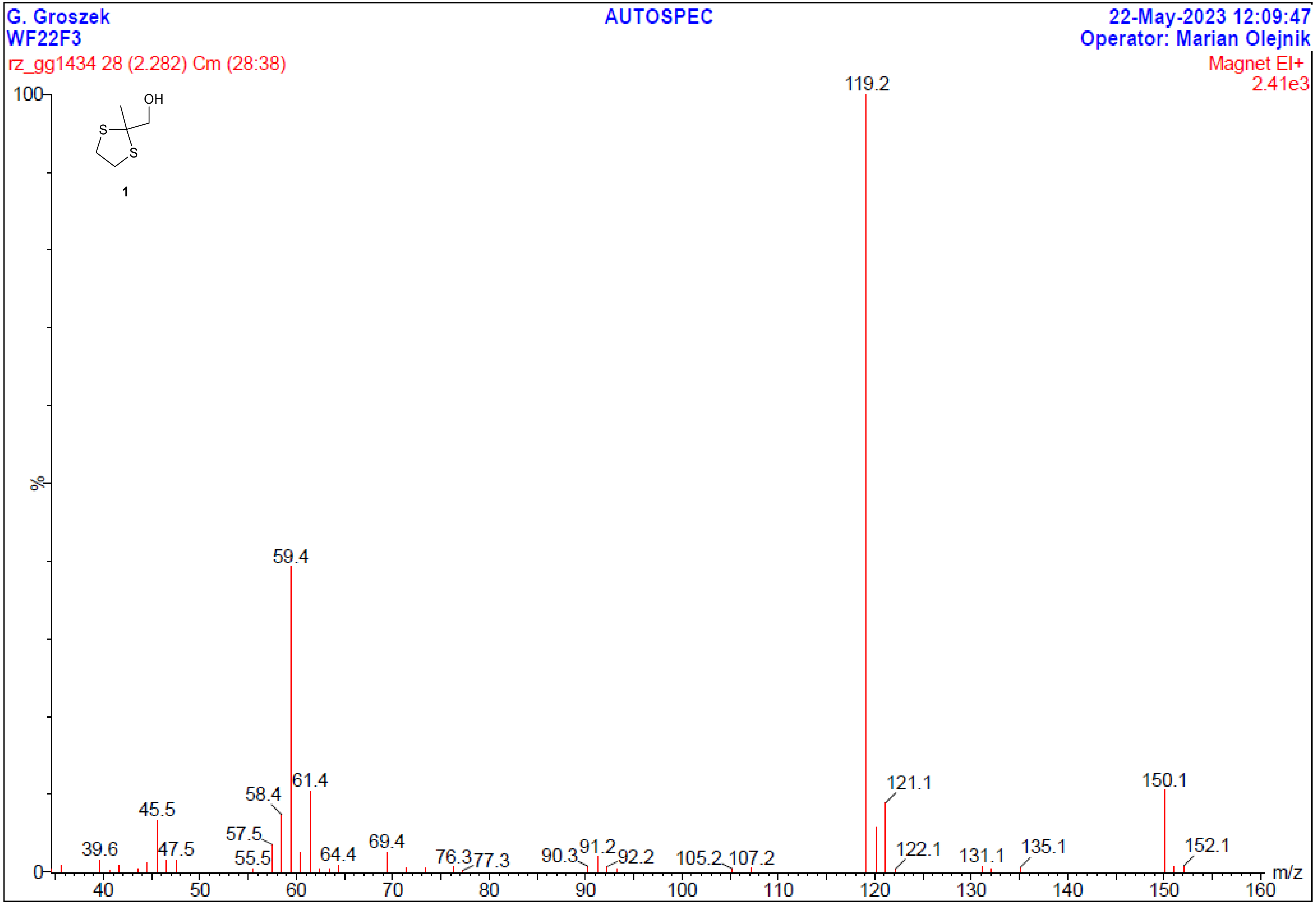


```
Minimum:                                  -1.5
Maximum:                5.0       20.0    150.0

Mass       Calc. Mass   mDa       PPM     DBE       i-FIT     Formula

358.0052   358.0046     0.6       1.7     2.0       3.3       C12  H22  S6
           358.0012     4.0       11.2    7.0       6.6       C15  H18  S5
```

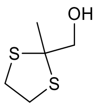

WF_22F1powt
1H_CDCl3 + 0.03% TMS, 25 st. C
OH
S
S
1
7.263
3.588
3.574
3.391
3.385
3.372
3.368
3.365
3.358
3.353
3.349
3.335
3.323
3.309
3.305
3.300
3.293
3.290
3.286
3.272
3.267
2.303
2.289
2.274
1.772
-0.000
3.6
3.5
3.4
3.3
ppm
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.00
2.00
0.97
3.03


WF-22F3 13C_CDCl3 + 0,03% TMS, 25 st.C
OH
S
S
1
77.3
77.0
76.8
71.0
68.1
39.6
26.6
-0.0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


WF22F3 27.05.2023
OH
S
S
1
3400,17
2960,63
2921,47
2857,47
1438,49
1423,18
1373,49
1276,48
1217,57
1151,58
1100,35
1070,84
1025,87
973,48
936,22
900,72
850,72
732,12
681,16
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
%Transmitancji
4000
3500
3000
2500
2000
1500
1000
500
Liczby falowe (cm-1)


G. Groszek
WF22F3
rz_gg1434 28 (2.282) Cm (28:38)
AUTOSPEC
22-May-2023 12:09:47
Operator: Marian Olejnik
Magnet EI+
2.41e3
OH
S
S
1
100
%
0
119.2
59.4
61.4
58.4
45.5
57.5
55.5
39.6
47.5
64.4
69.4
76.3
77.3
90.3
91.2
92.2
105.2
107.2
121.1
122.1
131.1
135.1
150.1
152.1
40
50
60
70
80
90
100
110
120
130
140
150
160
m/z

## Elemental Composition Report



**1**

Page 1

### Single Mass Analysis

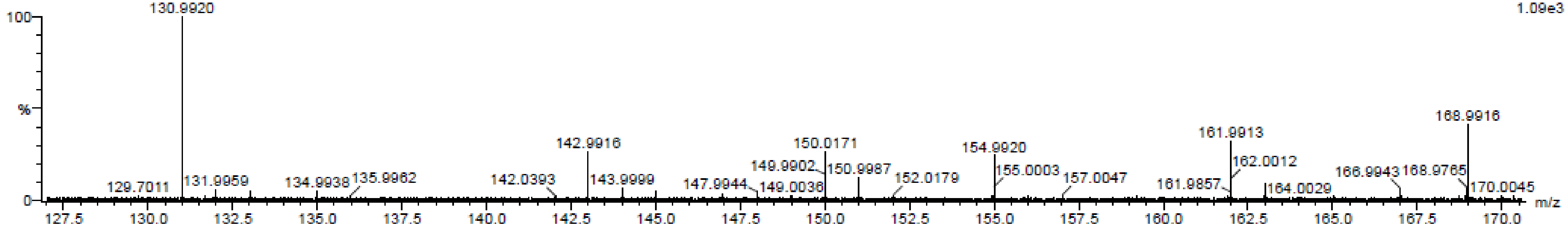
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 150.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
1 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-200 O: 1-1 S: 2-2

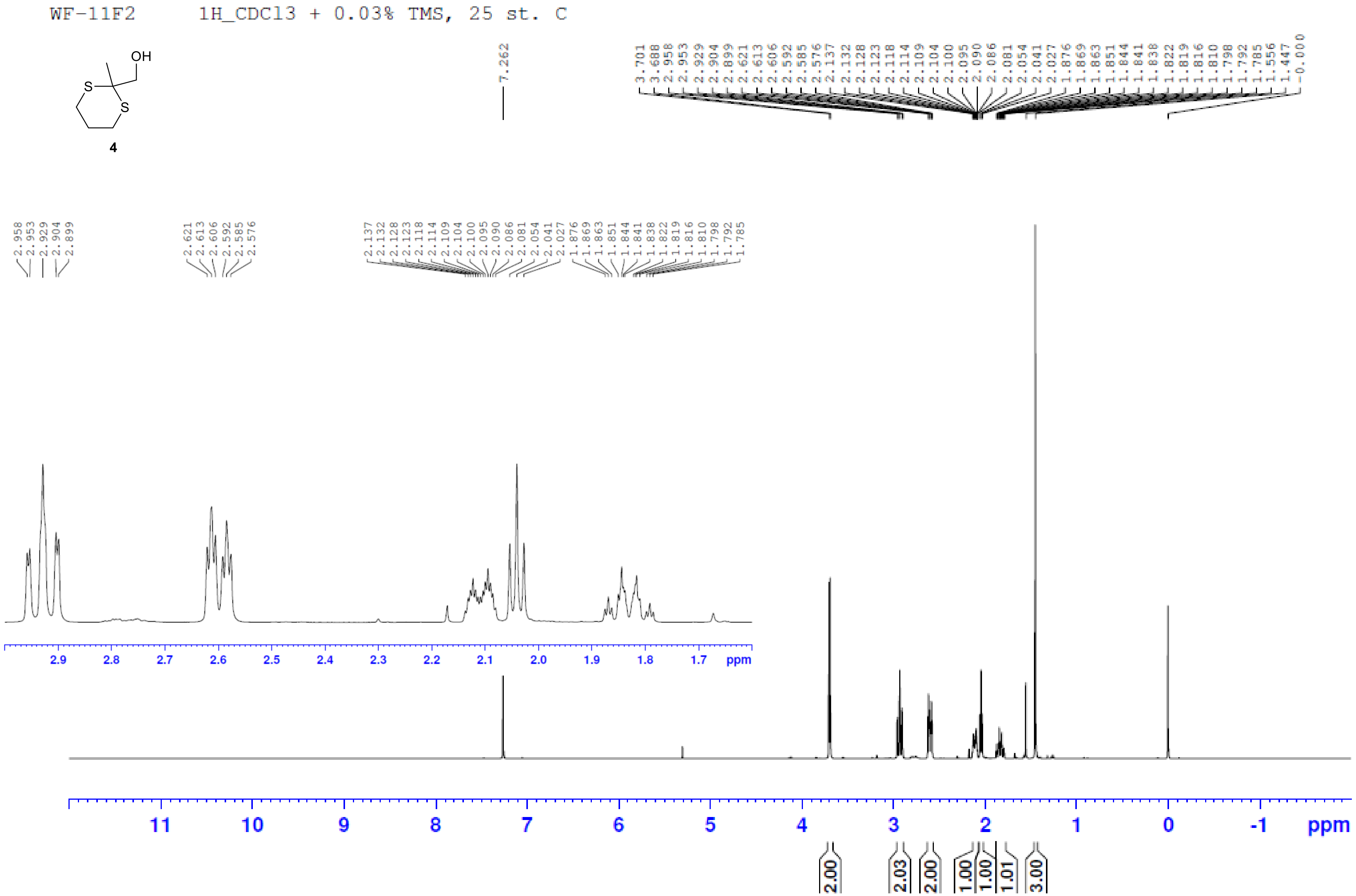
G. Groszek
WF22F3
rz_gg1434h1 43 (1.641) Cm (37:44)

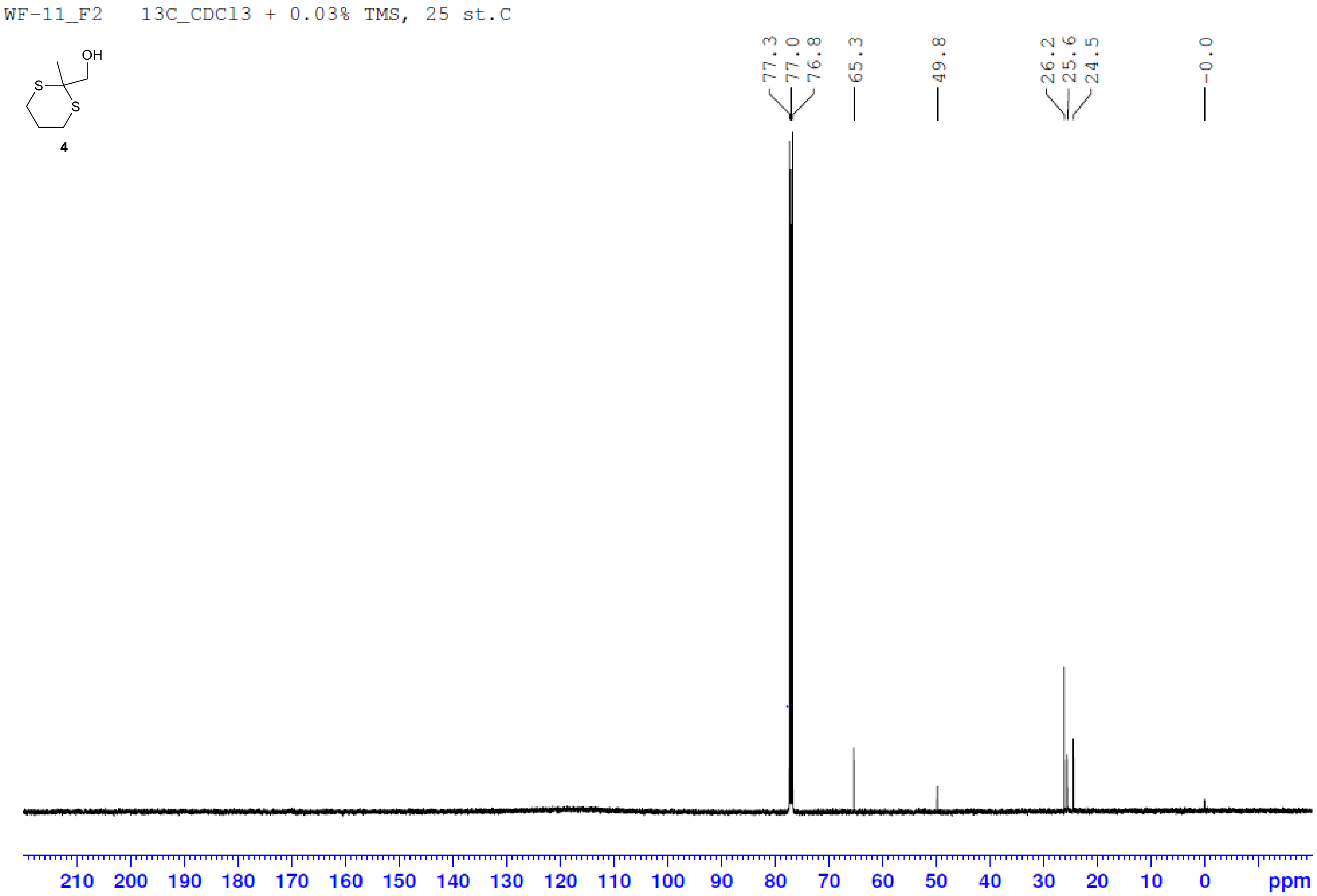
AUTOSPEC

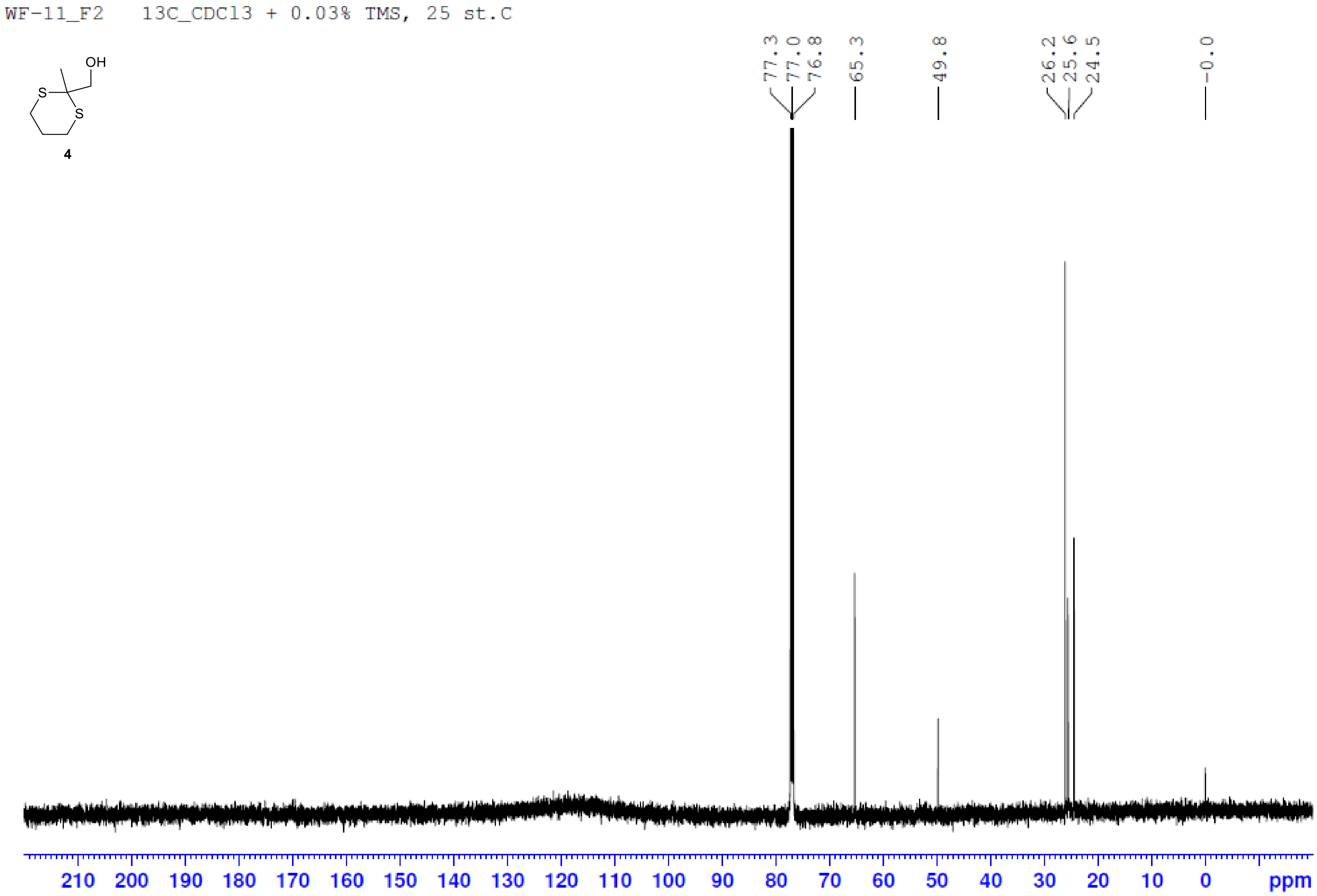
23-May-2023 14:47:44
Operator: Marian Olejnik
Voltage EI+
1.09e3

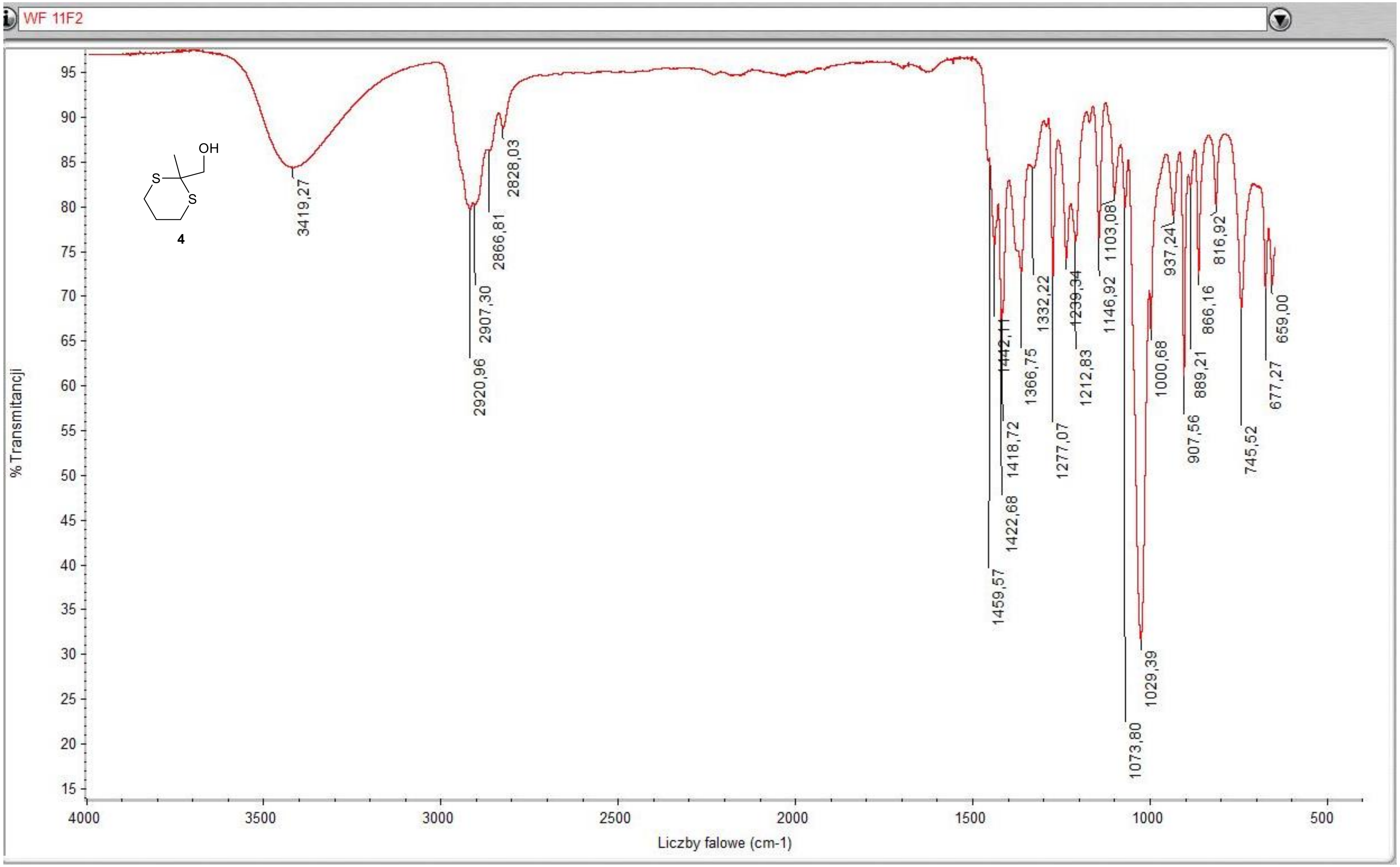


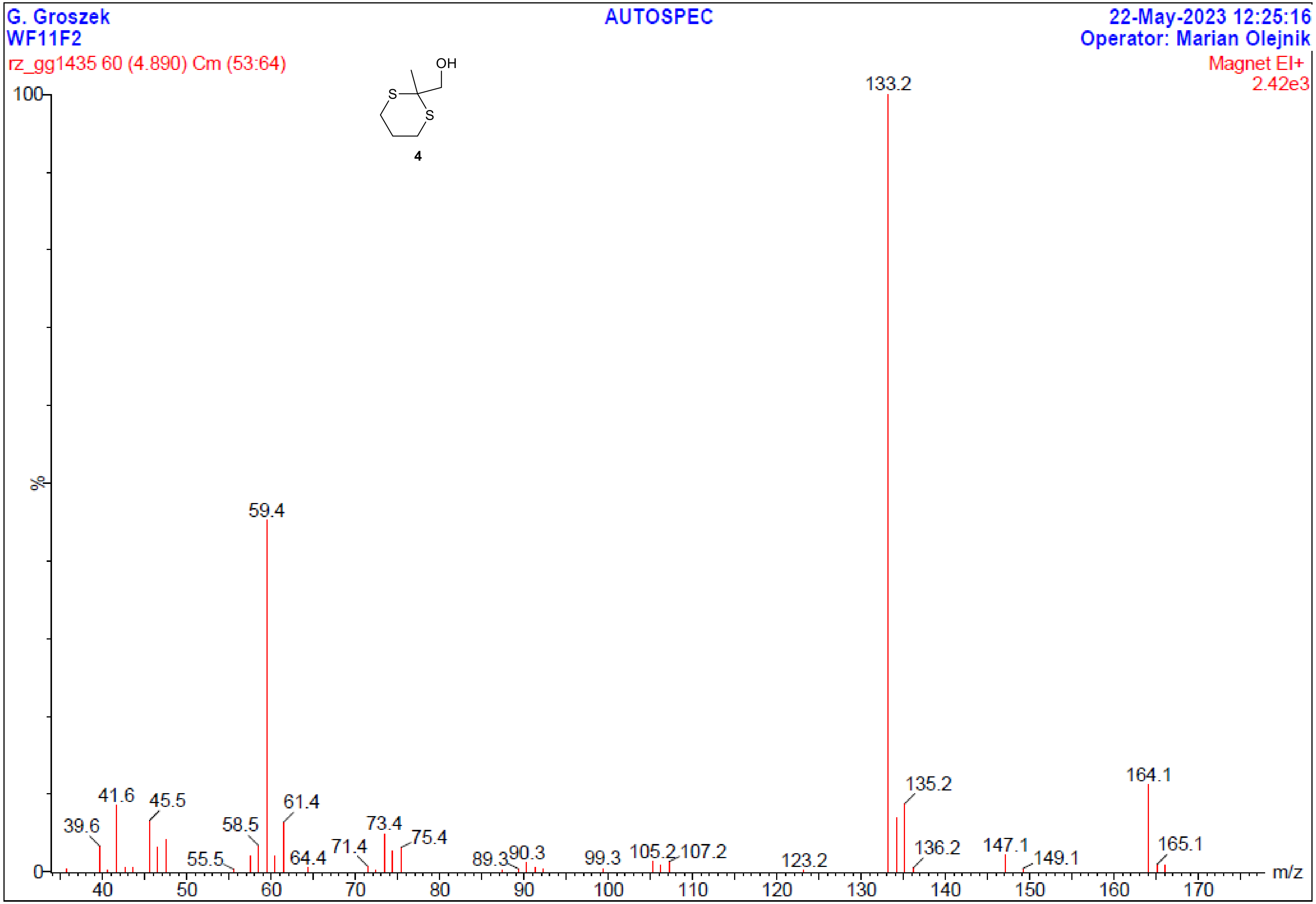
```
Minimum:                           -1.5
Maximum:                5.0    20.0   150.0

Mass      Calc. Mass    mDa    PPM    DBE    i-FIT    Formula

150.0171  150.0173     -0.2   -1.3    1.0    1.8      C5  H10  O  S2
```

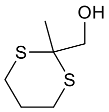

WF-11F2  1H_CDCl3 + 0.03% TMS, 25 st. C
OH
S
S
4
7.262
3.701
3.688
2.958
2.953
2.929
2.904
2.899
2.621
2.613
2.606
2.592
2.585
2.576
2.137
2.132
2.128
2.123
2.118
2.114
2.109
2.104
2.100
2.095
2.090
2.086
2.081
2.054
2.041
2.027
1.876
1.869
1.863
1.851
1.844
1.841
1.838
1.822
1.819
1.816
1.810
1.798
1.792
1.785
1.556
1.447
0.000
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
ppm
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.03
2.00
1.00
1.00
1.01
3.00


WF-11_F2 13C_CDCl3 + 0.03% TMS, 25 st.C
OH
S
S
4
77.3
77.0
76.8
65.3
49.8
26.2
25.6
24.5
-0.0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


WF-11_F2 13C_CDCl3 + 0.03% TMS, 25 st.C
OH
S
S
4
77.3
77.0
76.8
65.3
49.8
26.2
25.6
24.5
-0.0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


WF 11F2
OH
S
S
4
% Transmitancji
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
3419,27
2920,96
2907,30
2866,81
2828,03
1459,57
1442,11
1422,68
1418,72
1366,75
1332,22
1277,07
1239,34
1212,83
1146,92
1103,08
1073,80
1029,39
1000,68
937,24
907,56
889,21
866,16
816,92
745,52
677,27
659,00
4000
3500
3000
2500
2000
1500
1000
500
Liczby falowe (cm-1)


G. Groszek
WF11F2
rz_gg1435 60 (4.890) Cm (53:64)
AUTOSPEC
22-May-2023 12:25:16
Operator: Marian Olejnik
Magnet EI+
2.42e3
OH
S
S
4
100
%
0
133.2
59.4
164.1
135.2
61.4
45.5
41.6
39.6
58.5
73.4
75.4
71.4
55.5
64.4
89.3
90.3
99.3
105.2
107.2
123.2
136.2
147.1
149.1
165.1
40
50
60
70
80
90
100
110
120
130
140
150
160
170
m/z



**4**

## Elemental Composition Report

**Page 1**

### Single Mass Analysis

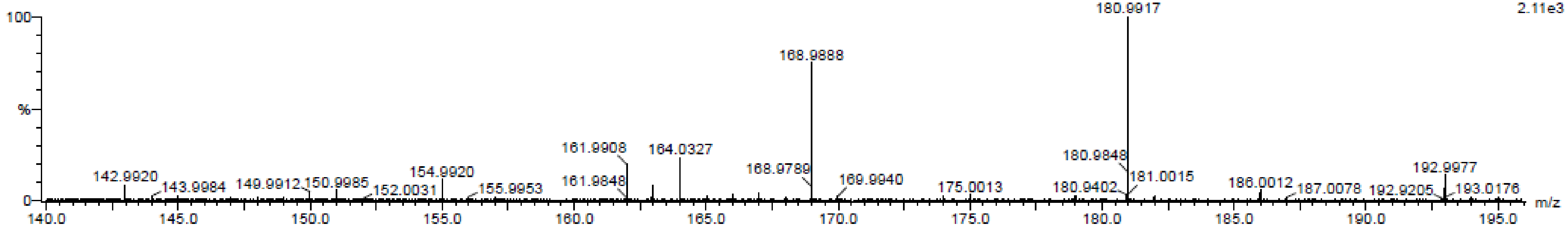
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 150.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
2 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-200 O: 1-1 S: 2-2

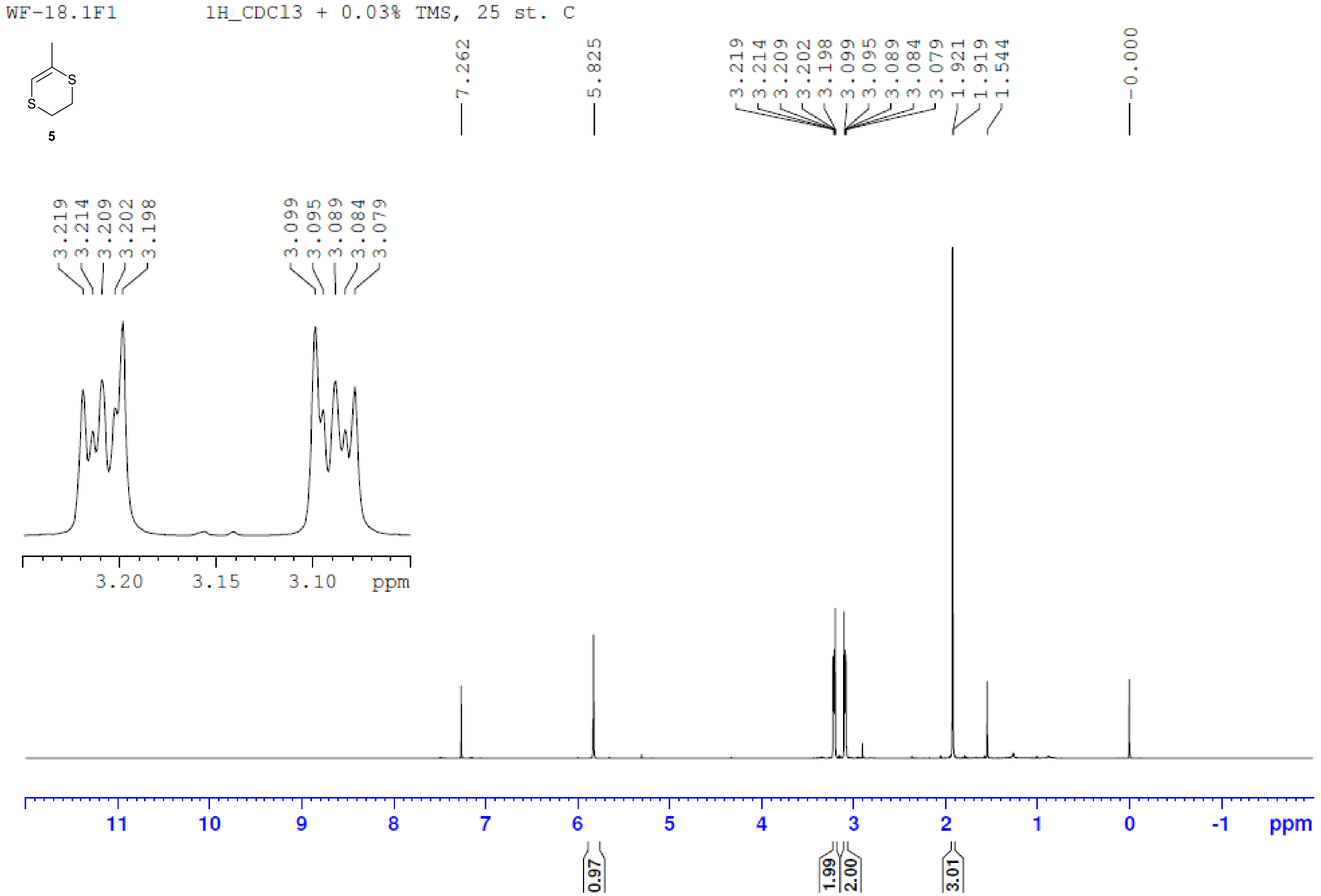
G. Groszek
WF11F2
rz_gg1435h 57 (2.174) Cm (56:66)

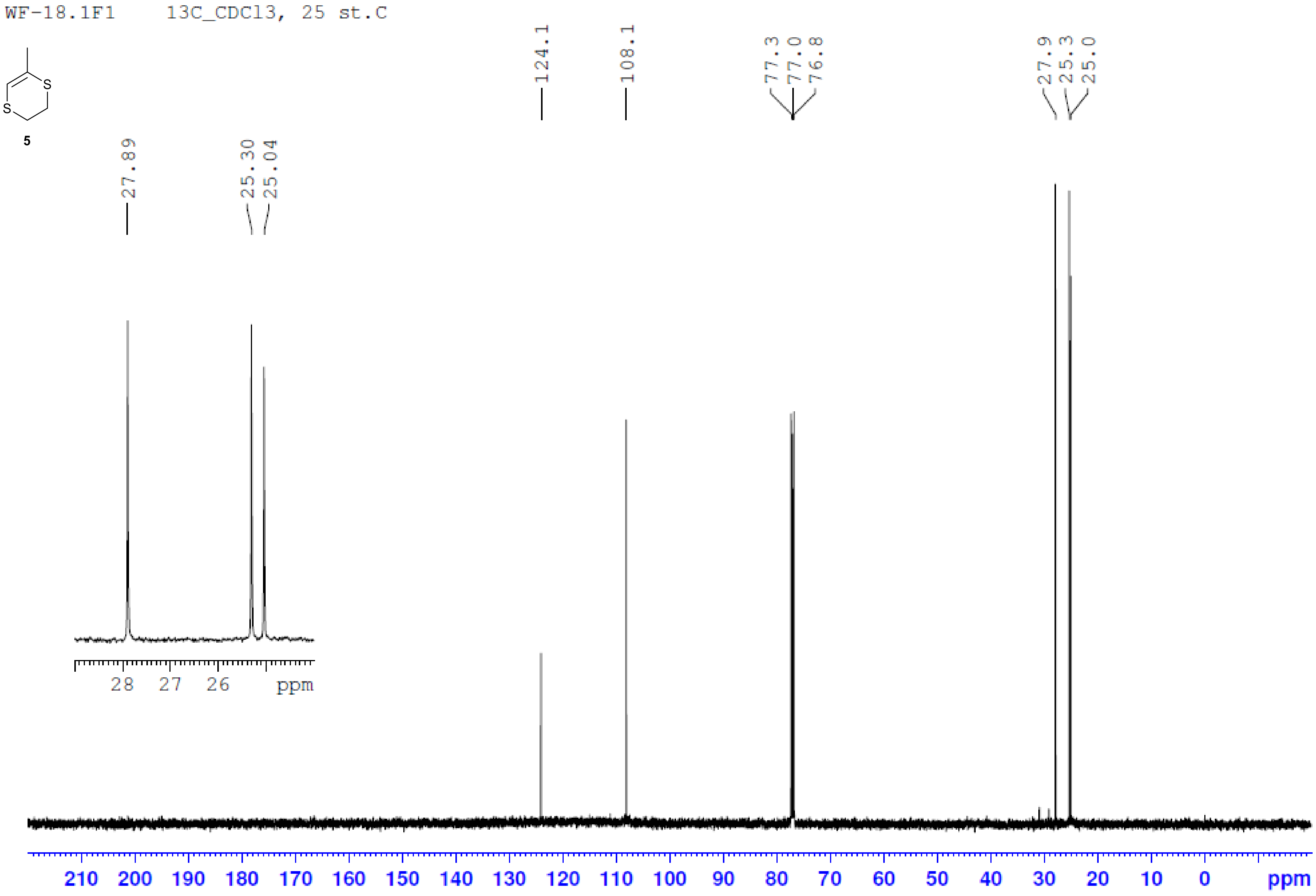
AUTOSPEC

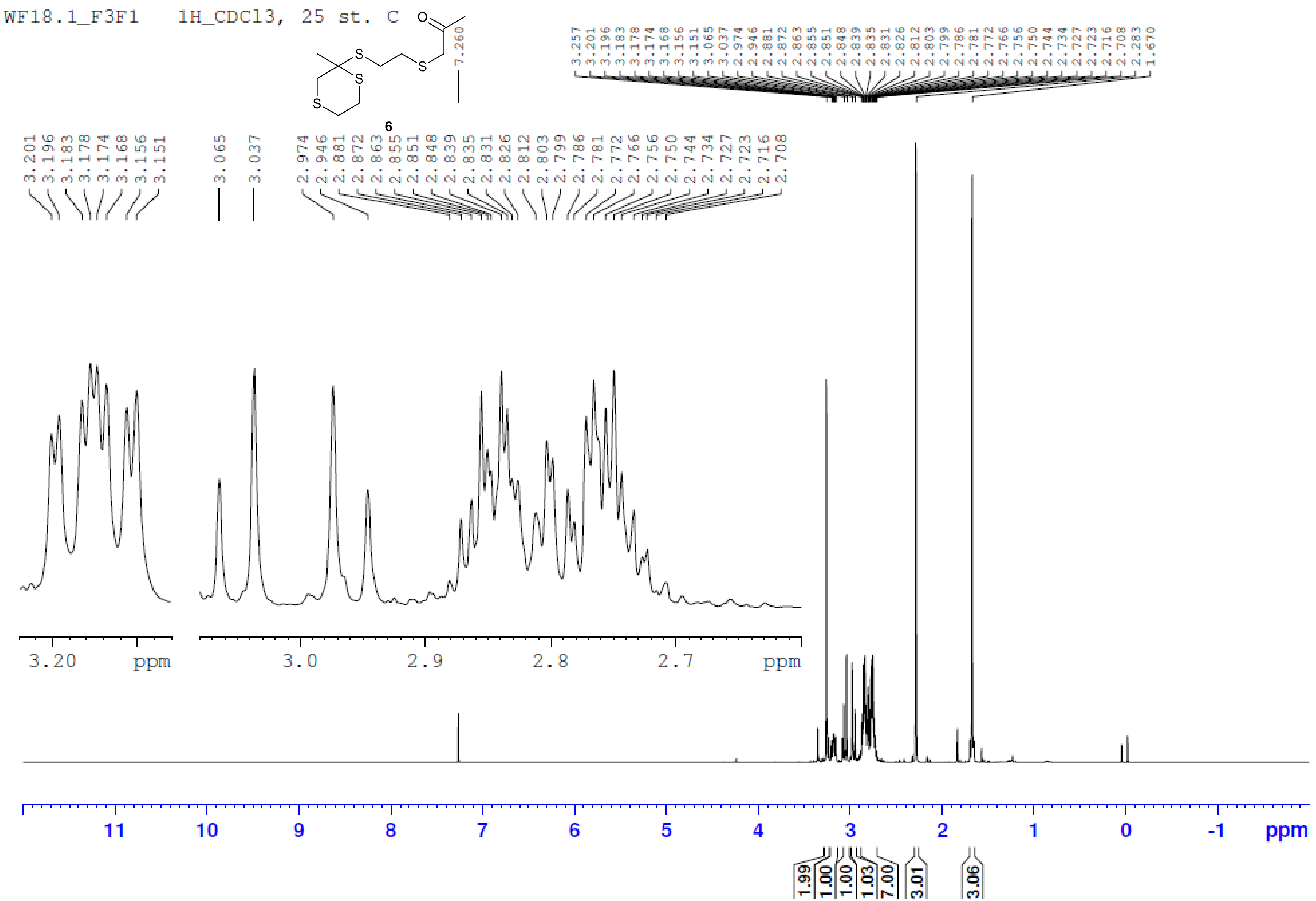
23-May-2023 15:16:23
Operator: Marian Olejnik
Voltage EI+

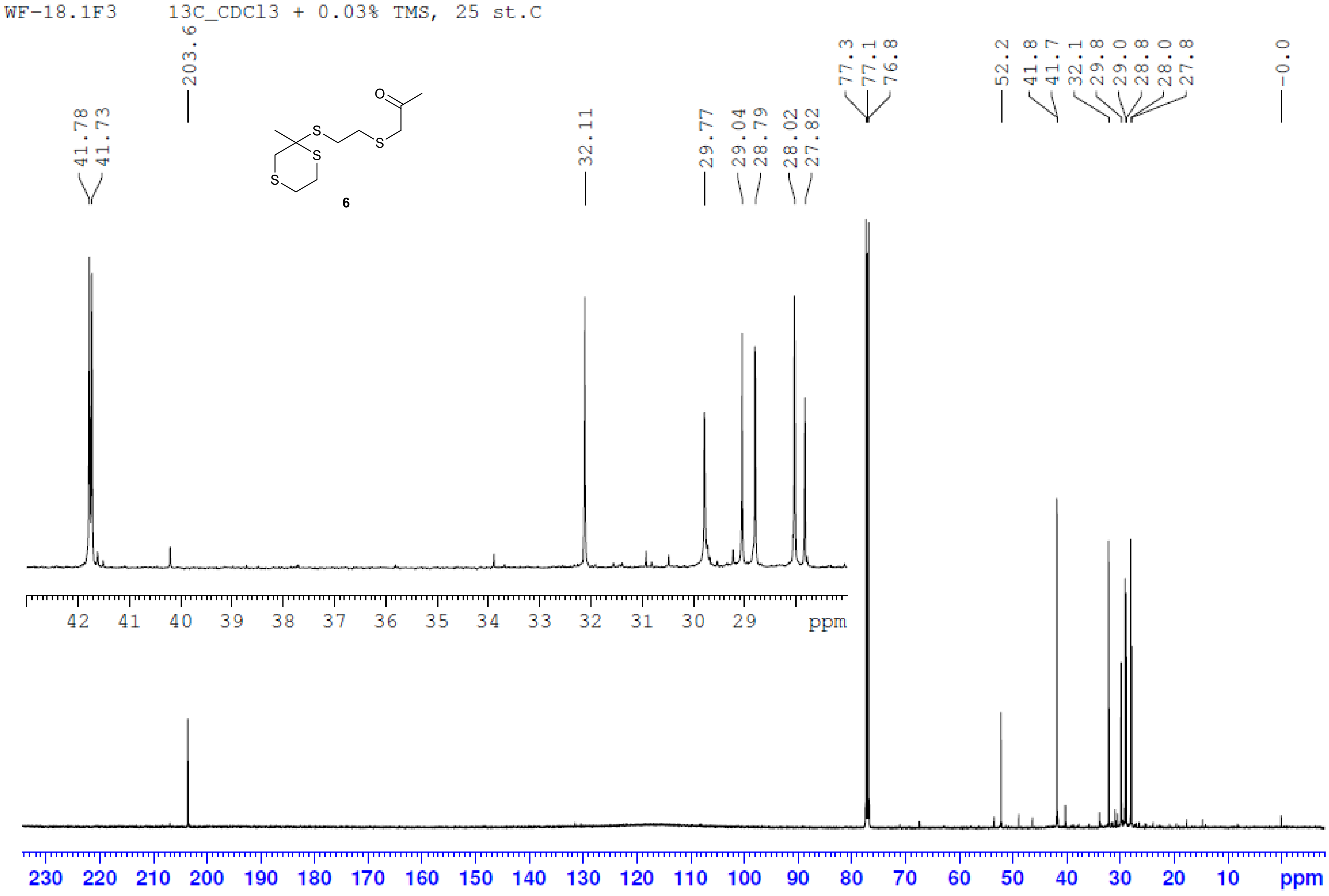


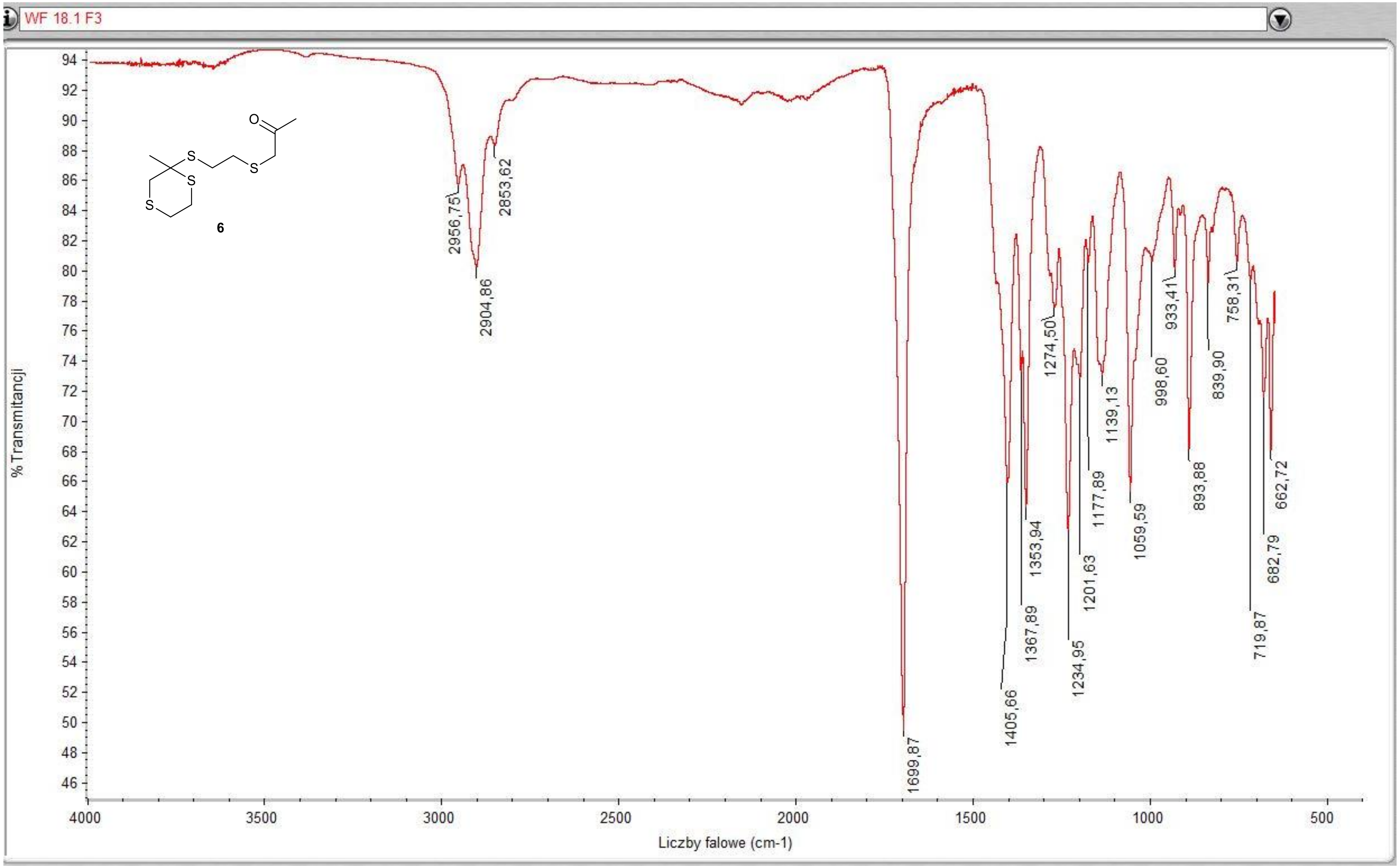
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|---|---|---|---|---|---|---|
| Minimum: | | | | -1.5 | | |
| Maximum: | | 5.0 | 20.0 | 150.0 | | |
| 164.0327 | 164.0330 | -0.3 | -1.8 | 1.0 | 3.2 | C6 H12 O S2 |

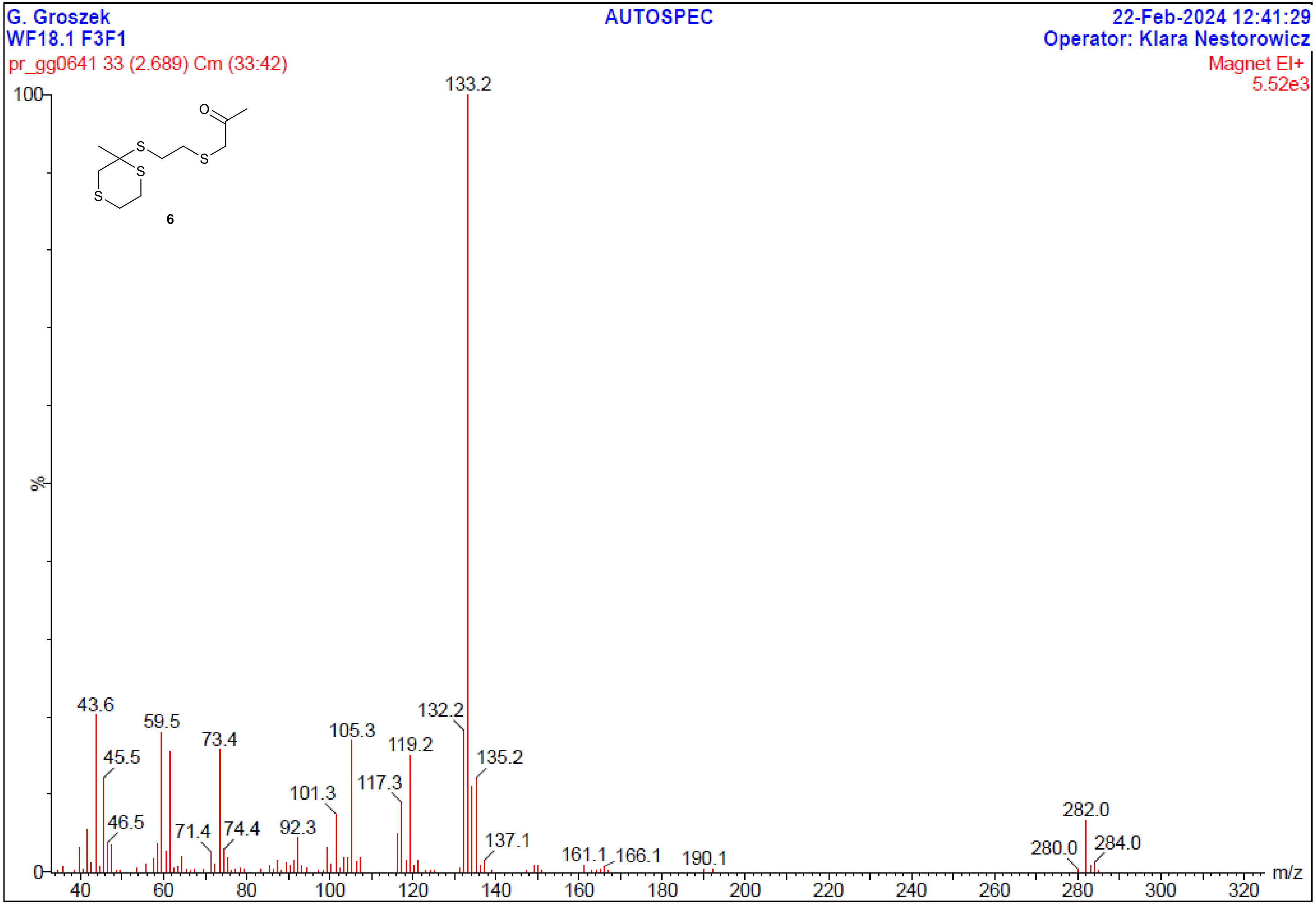

WF-18.1F1
1H_CDCl3 + 0.03% TMS, 25 st. C
S
S
5
7.262
5.825
3.219
3.214
3.209
3.202
3.198
3.099
3.095
3.089
3.084
3.079
1.921
1.919
1.544
-0.000
3.20
3.15
3.10
ppm
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
0.97
1.99
2.00
3.01


WF-18.1F1 13C_CDCl3, 25 st.C
S
S
5
124.1
108.1
77.3
77.0
76.8
27.9
25.3
25.0
27.89
25.30
25.04
28
27
26
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

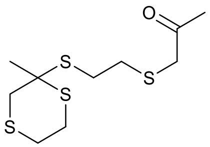

WF18.1_F3F1 1H_CDCl3, 25 st. C
6
7.260
3.257
3.201
3.196
3.183
3.178
3.174
3.168
3.156
3.151
3.065
3.037
2.974
2.946
2.881
2.872
2.863
2.855
2.851
2.848
2.839
2.835
2.831
2.826
2.812
2.803
2.799
2.786
2.781
2.772
2.766
2.756
2.750
2.744
2.734
2.727
2.723
2.716
2.708
2.283
1.670
3.20
ppm
3.0
2.9
2.8
2.7
ppm
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.99
1.00
1.00
1.03
7.00
3.01
3.06


WF-18.1F3 13C_CDCl3 + 0.03% TMS, 25 st.C
203.6
77.3
77.1
76.8
52.2
41.8
41.7
32.1
29.8
29.0
28.8
28.0
27.8
-0.0
41.78
41.73
32.11
29.77
29.04
28.79
28.02
27.82
O
S
S
S
S
6
42
41
40
39
38
37
36
35
34
33
32
31
30
29
ppm
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


WF 18.1 F3
% Transmitancji
Liczby falowe (cm-1)
94
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
50
48
46
4000
3500
3000
2500
2000
1500
1000
500
O
S
S
S
S
6
2956,75
2904,86
2853,62
1699,87
1405,66
1367,89
1353,94
1274,50
1234,95
1201,63
1177,89
1139,13
1059,59
998,60
933,41
893,88
839,90
758,31
719,87
682,79
662,72


G. Groszek
WF18.1 F3F1
pr_gg0641 33 (2.689) Cm (33:42)
AUTOSPEC
22-Feb-2024 12:41:29
Operator: Klara Nestorowicz
Magnet EI+
5.52e3
O
S
S
S
S
6
100
%
0
133.2
43.6
45.5
46.5
59.5
71.4
73.4
74.4
92.3
101.3
105.3
117.3
119.2
132.2
135.2
137.1
161.1
166.1
190.1
280.0
282.0
284.0
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
m/z

## Elemental Composition Report



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Page 1

## Single Mass Analysis

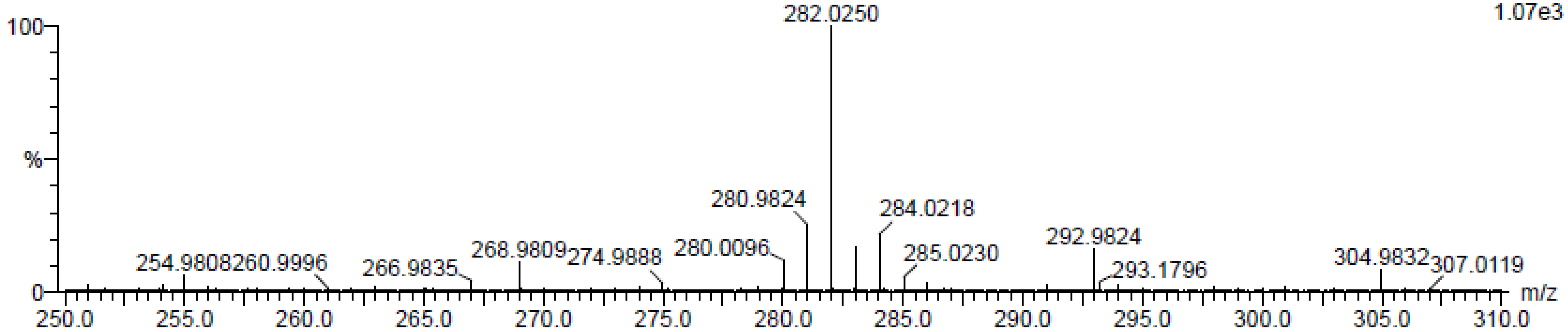
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 150.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
50 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-200 O: 0-2 S: 0-4

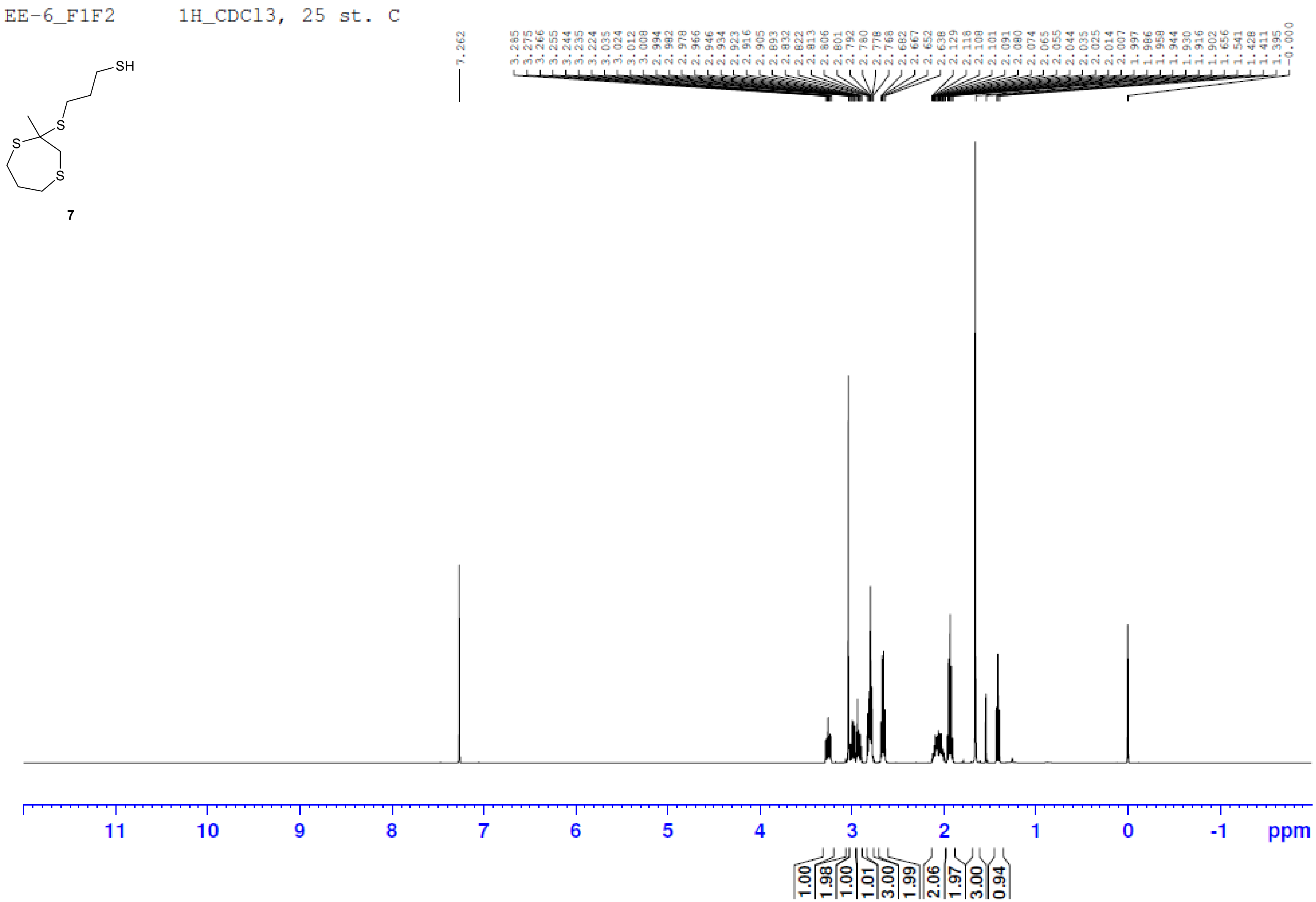
G. Groszek
WF18.1 F3F1
pr_gg0641h 241 (9.192) Cm (240:254)

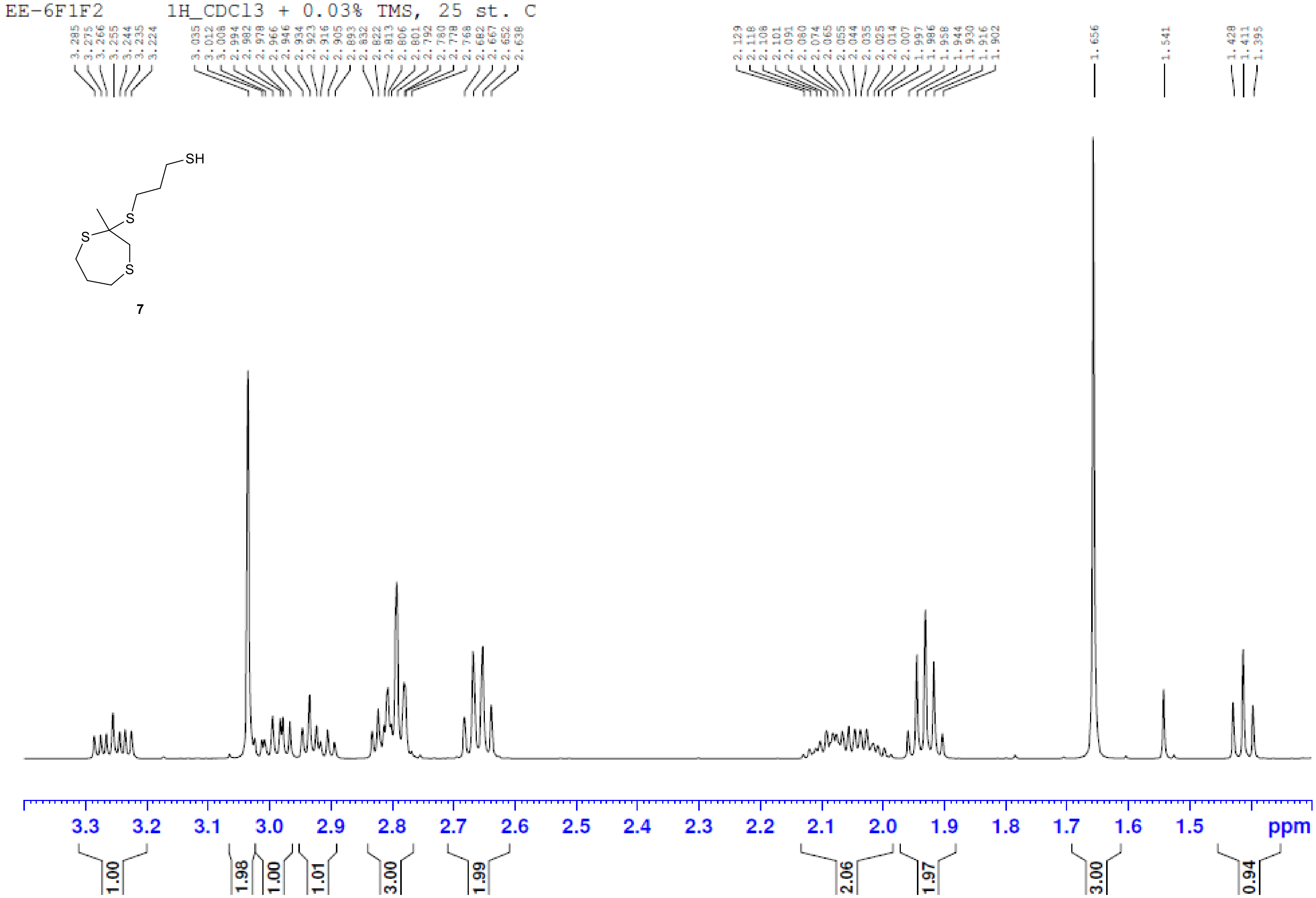
AUTOSPEC

23-Feb-2024 13:13:13
Operator: Klara Nestorowicz
Voltage EI+
1.07e3

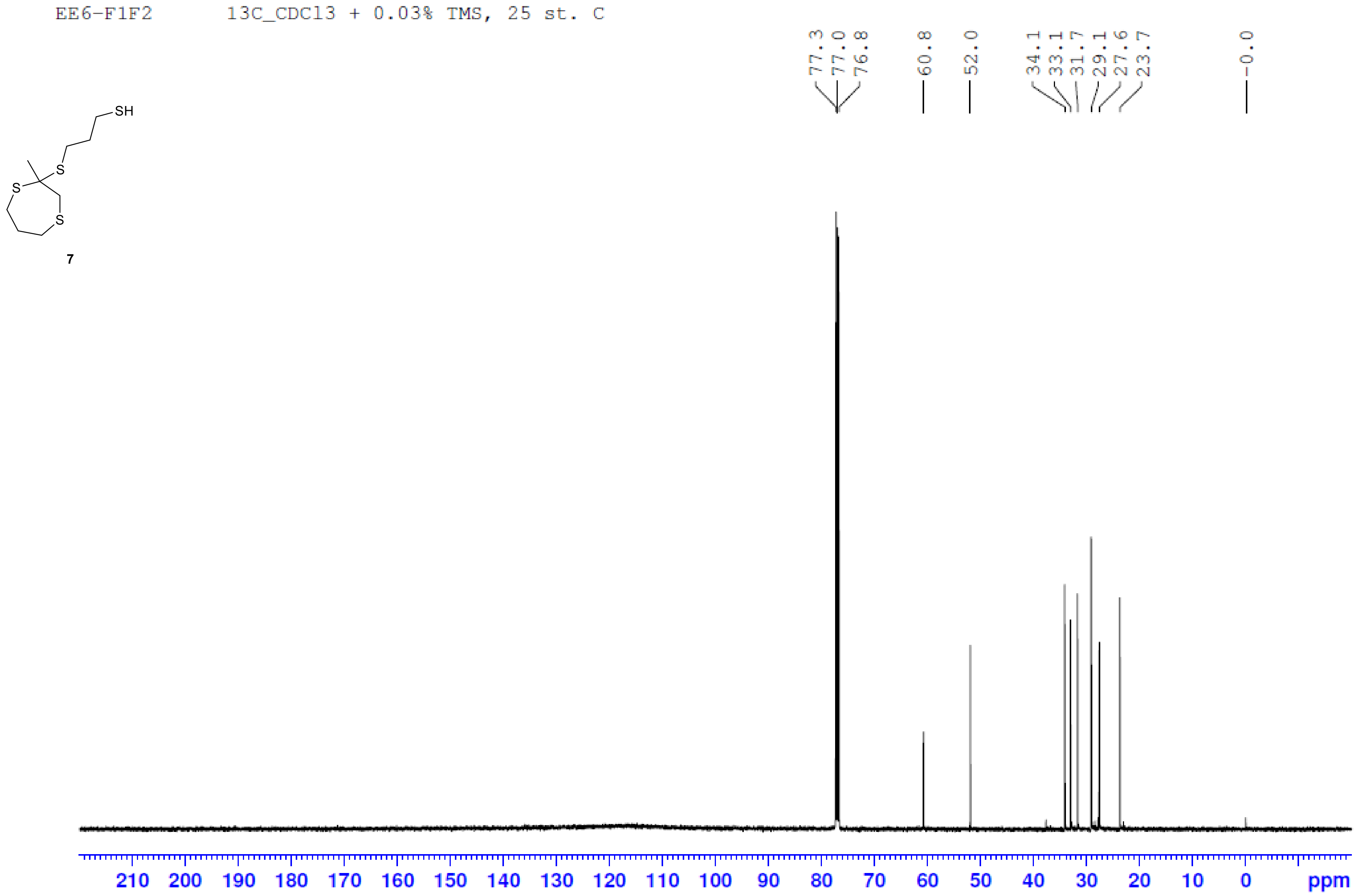


```
Minimum:                                     -1.5
Maximum:                   5.0       20.0    150.0

Mass        Calc. Mass     mDa       PPM     DBE       i-FIT      Formula

282.0250    282.0241       0.9       3.2     2.0       3.1        C10  H18  O  S4
            282.0207       4.3       15.2    7.0       11.1       C13  H14  O  S3
```


EE-6_F1F2 1H_CDCl3, 25 st. C
SH
S
S
S
7
7.262
3.285
3.275
3.266
3.255
3.244
3.235
3.224
3.035
3.024
3.012
3.008
2.994
2.982
2.978
2.966
2.946
2.934
2.923
2.916
2.905
2.893
2.832
2.822
2.813
2.806
2.801
2.792
2.780
2.778
2.768
2.682
2.667
2.652
2.638
2.129
2.118
2.108
2.101
2.091
2.080
2.074
2.065
2.055
2.044
2.035
2.025
2.014
2.007
1.997
1.986
1.958
1.944
1.930
1.916
1.902
1.656
1.541
1.428
1.411
1.395
-0.000
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
1.98
1.00
1.01
3.00
1.99
2.06
1.97
3.00
0.94


EE-6F1F2 1H_CDCl3 + 0.03% TMS, 25 st. C
3.285
3.275
3.266
3.255
3.244
3.235
3.224
3.035
3.012
3.008
2.994
2.982
2.978
2.966
2.946
2.934
2.923
2.916
2.905
2.893
2.832
2.822
2.813
2.806
2.801
2.792
2.780
2.778
2.768
2.682
2.667
2.652
2.638
2.129
2.118
2.108
2.101
2.091
2.080
2.074
2.065
2.055
2.044
2.035
2.025
2.014
2.007
1.997
1.986
1.958
1.944
1.930
1.916
1.902
1.656
1.541
1.428
1.411
1.395
SH
S
S
S
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
ppm
1.00
1.98
1.00
1.01
3.00
1.99
2.06
1.97
3.00
0.94

7



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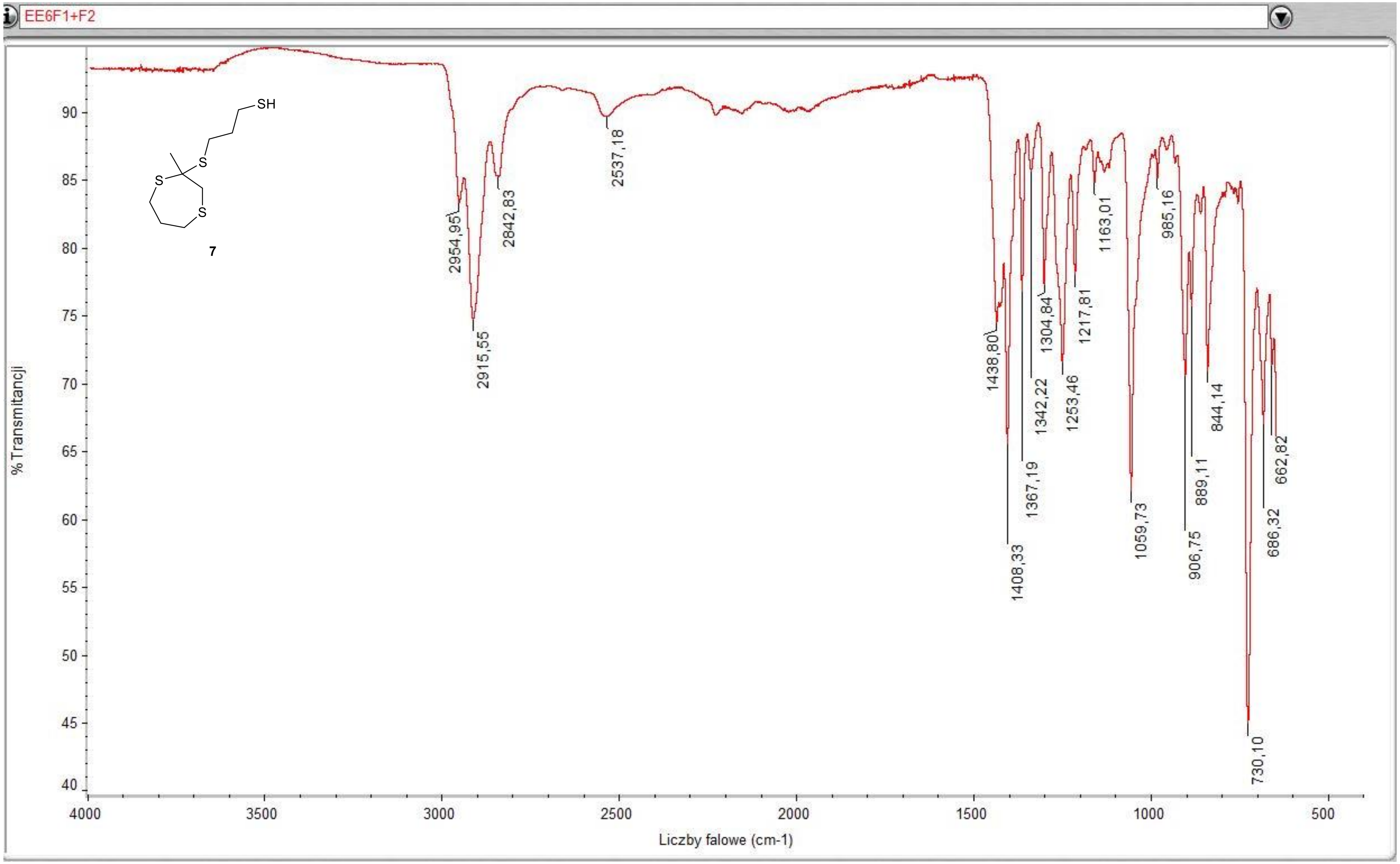
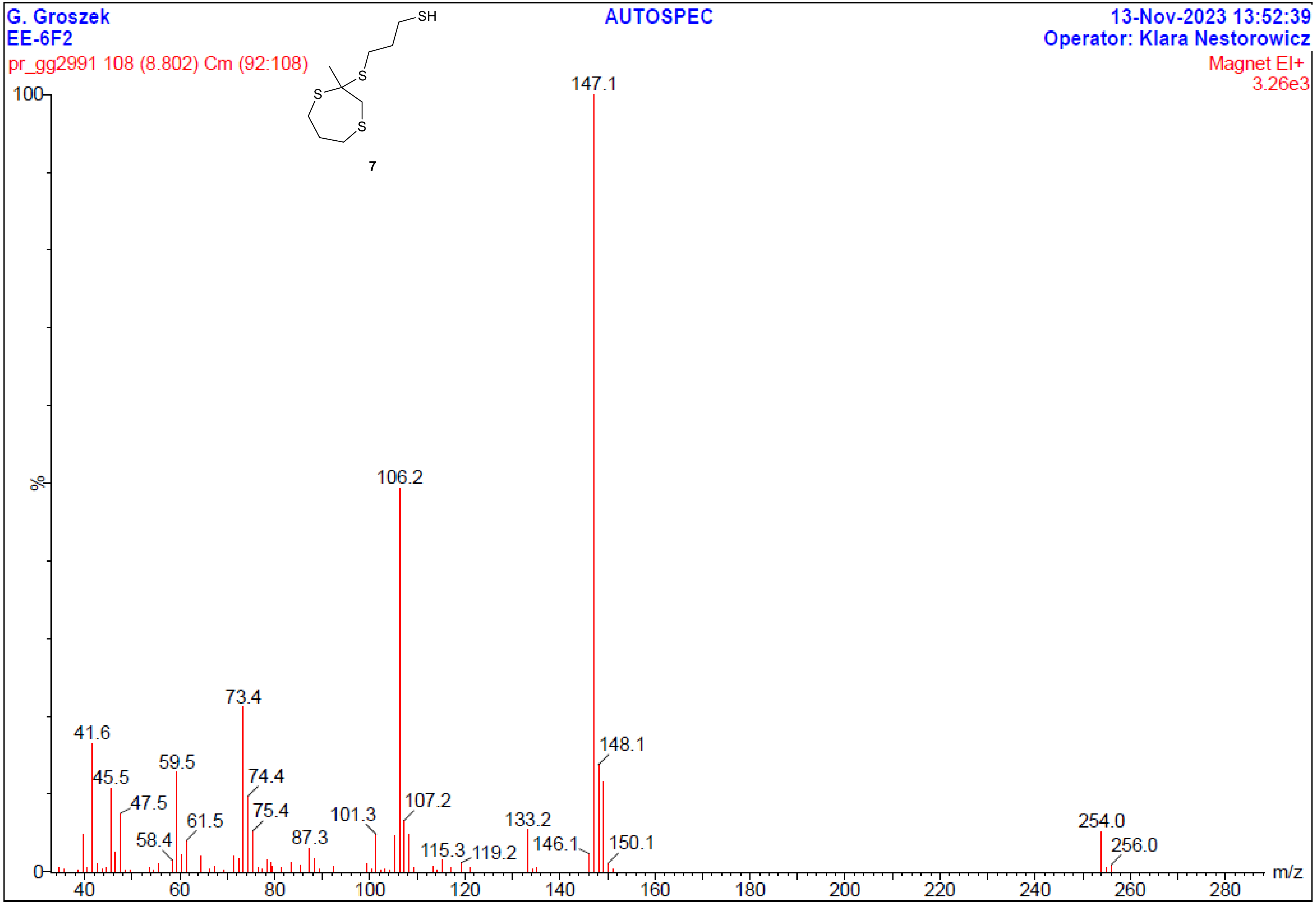
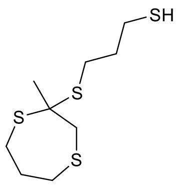
EE6-F1F2 13C_CDCl3 + 0.03% TMS, 25 st. C
SH
S
S
S
77.3
77.0
76.8
60.8
52.0
34.1
33.1
31.7
29.1
27.6
23.7
-0.0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


EE6F1+F2
SH
S
S
S
7
% Transmitancji
Liczby falowe (cm-1)
40
45
50
55
60
65
70
75
80
85
90
4000
3500
3000
2500
2000
1500
1000
500
2954,95
2915,55
2842,83
2537,18
1438,80
1408,33
1367,19
1342,22
1304,84
1253,46
1217,81
1163,01
1059,73
985,16
906,75
889,11
844,14
730,10
686,32
662,82


G. Groszek
EE-6F2
pr_gg2991 108 (8.802) Cm (92:108)
SH
S
S
S
7
AUTOSPEC
13-Nov-2023 13:52:39
Operator: Klara Nestorowicz
Magnet EI+
3.26e3
100
%
0
147.1
106.2
73.4
41.6
148.1
59.5
45.5
74.4
47.5
107.2
75.4
61.5
101.3
133.2
254.0
58.4
87.3
150.1
146.1
256.0
115.3
119.2
40
60
80
100
120
140
160
180
200
220
240
260
280
m/z

## Elemental Composition Report



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Page 1

### Single Mass Analysis

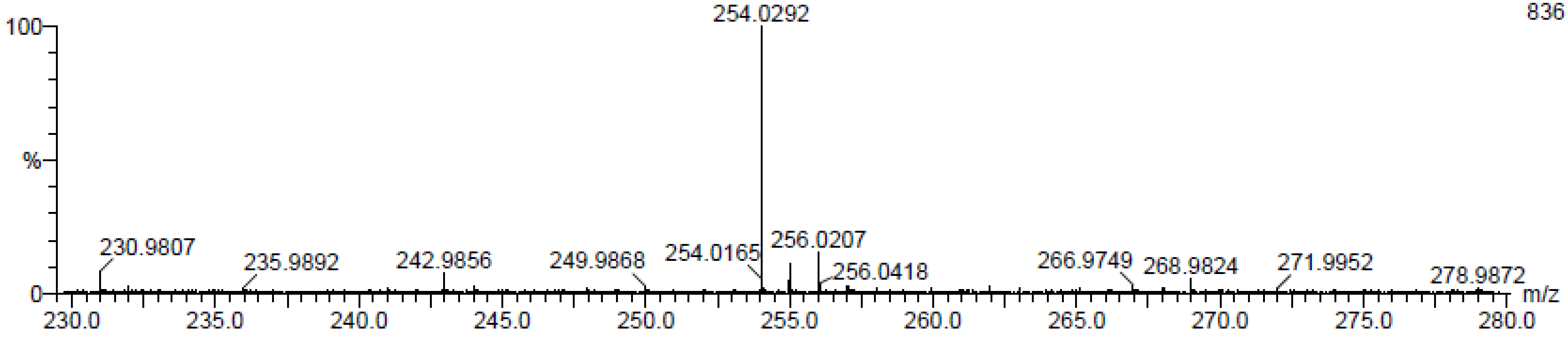
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 150.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
16 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-200 S: 0-4

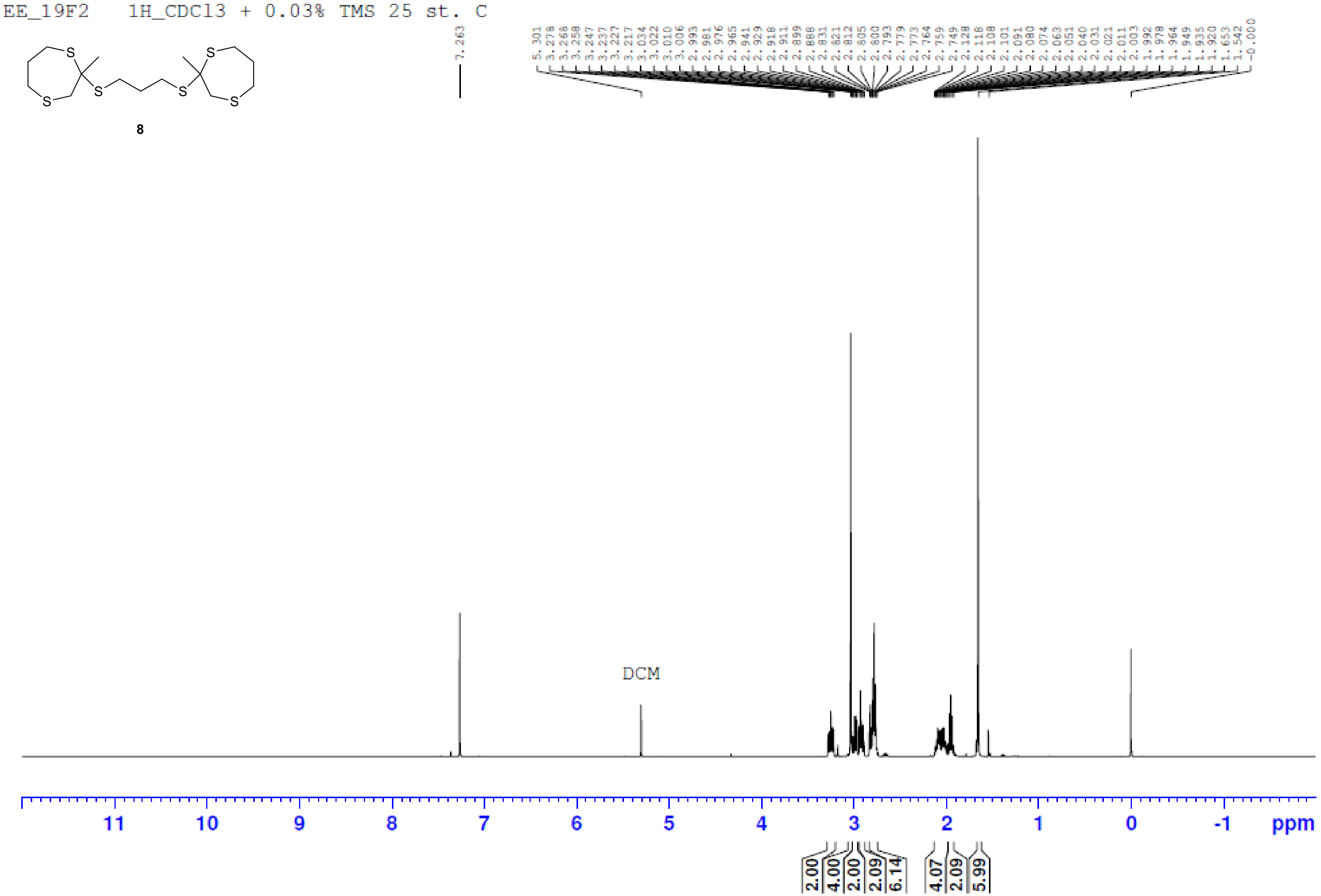
G. Groszek
EE-6F2
pr_gg2991h 136 (5.187) Cm (124:137)

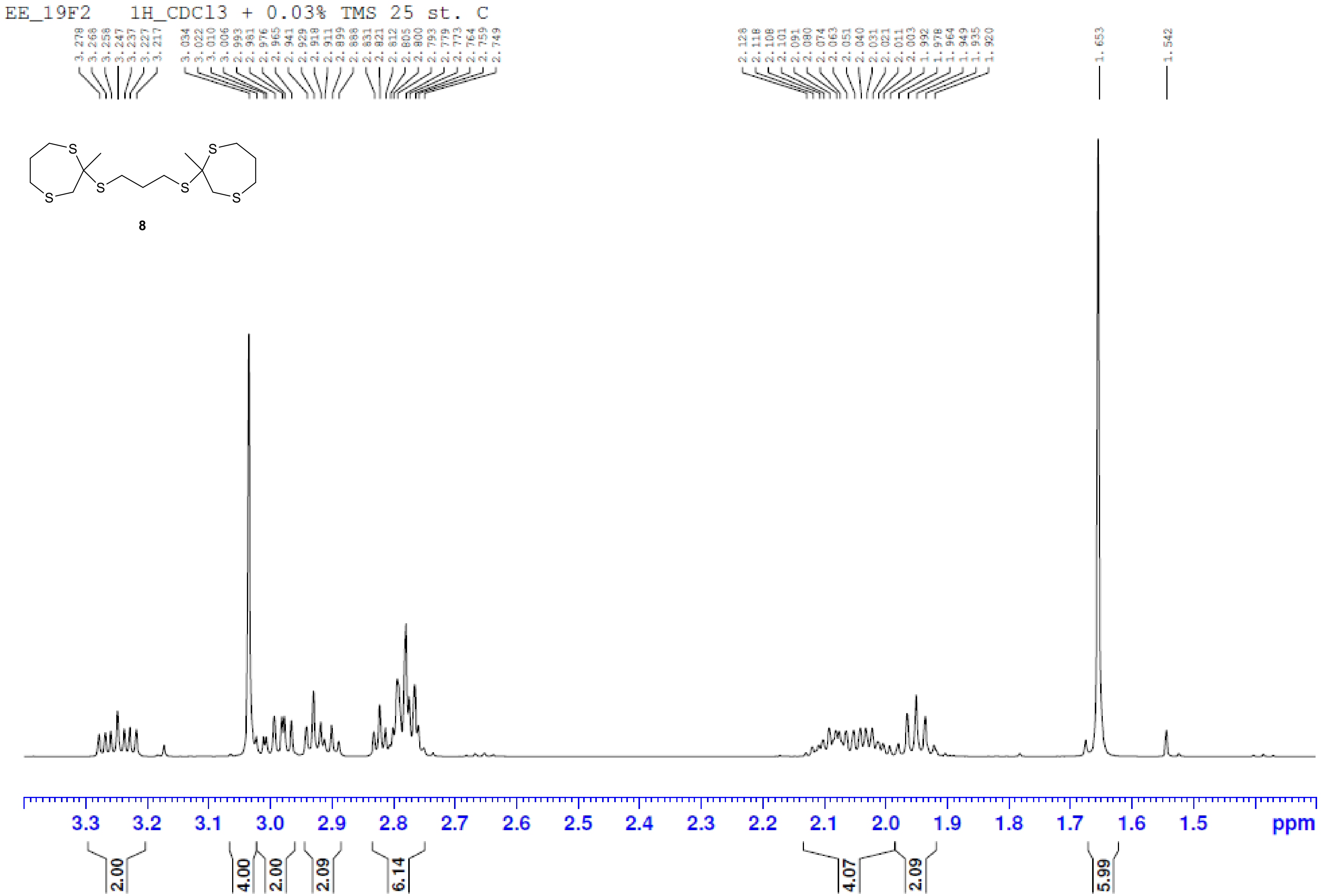
AUTOSPEC

15-Nov-2023 12:52:17
Operator: Klara Nestorowicz
Voltage EI+
836

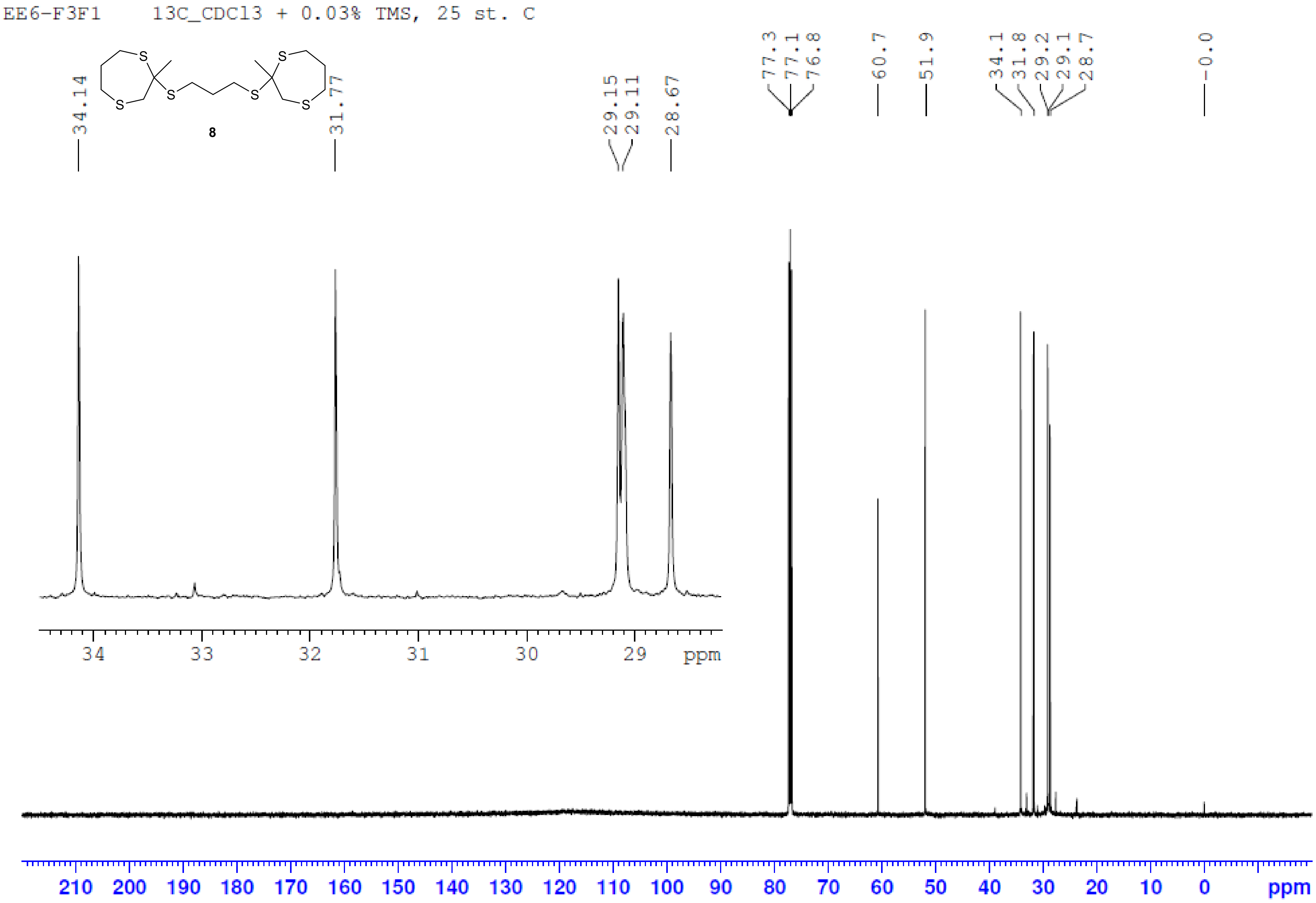


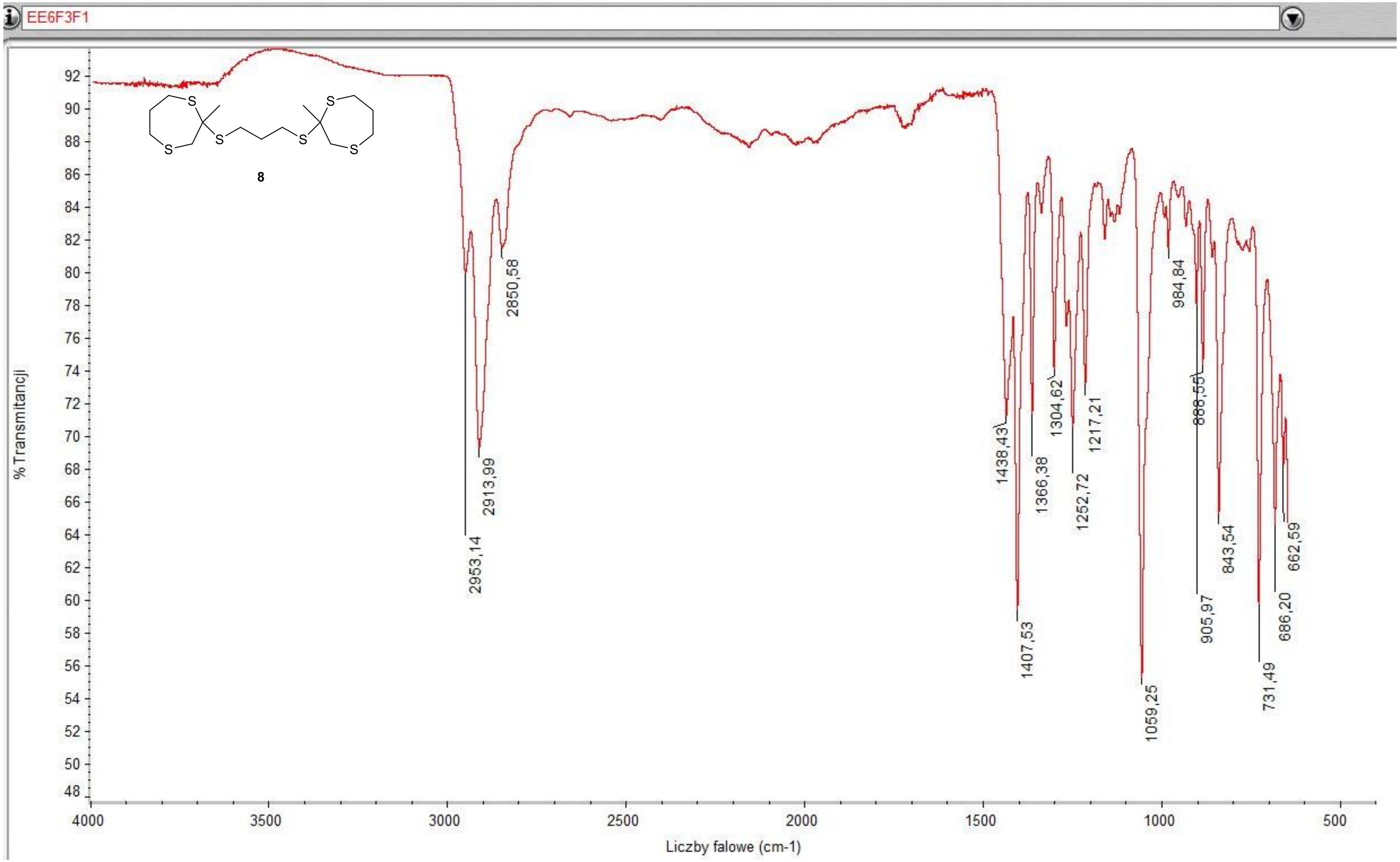
```
Minimum:                                 -1.5
Maximum:                5.0      20.0    150.0

Mass       Calc. Mass   mDa      PPM     DBE      i-FIT    Formula

254.0292   254.0291     0.1      0.4     1.0      3.6      C9  H18  S4
           254.0258     3.4      13.4    6.0      8.2      C12  H14  S3
```

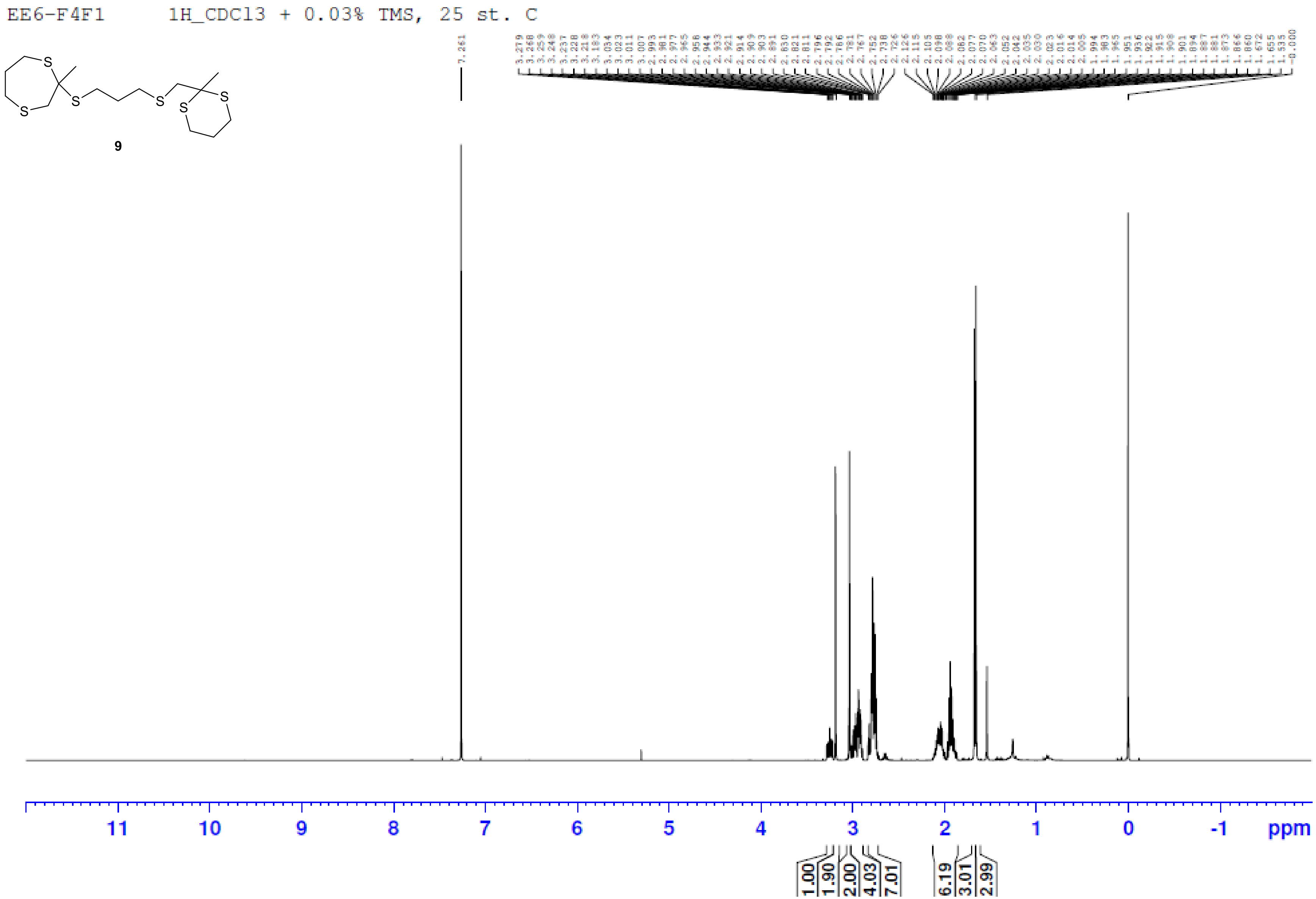

EE_19F2 1H_CDCl3 + 0.03% TMS 25 st. C
8
7.263
5.301
3.278
3.268
3.258
3.247
3.237
3.227
3.217
3.034
3.022
3.010
3.006
2.993
2.981
2.976
2.965
2.941
2.929
2.918
2.911
2.899
2.888
2.831
2.821
2.812
2.805
2.800
2.793
2.779
2.773
2.764
2.759
2.749
2.128
2.118
2.108
2.101
2.091
2.080
2.074
2.063
2.051
2.040
2.031
2.021
2.011
2.003
1.992
1.978
1.964
1.949
1.935
1.920
1.653
1.542
-0.000
DCM
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
4.00
2.00
2.09
6.14
4.07
2.09
5.99


EE_19F2 1H_CDCl3 + 0.03% TMS 25 st. C
3.278
3.268
3.258
3.247
3.237
3.227
3.217
3.034
3.022
3.010
3.006
2.993
2.981
2.976
2.965
2.941
2.929
2.918
2.911
2.899
2.888
2.831
2.821
2.812
2.805
2.800
2.793
2.779
2.773
2.764
2.759
2.749
2.128
2.118
2.108
2.101
2.091
2.080
2.074
2.063
2.051
2.040
2.031
2.021
2.011
2.003
1.992
1.978
1.964
1.949
1.935
1.920
1.653
1.542
S
S
S
S
S
S
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
ppm
2.00
4.00
2.00
2.09
6.14
4.07
2.09
5.99

**8**


EE6-F3F1 13C_CDCl3 + 0.03% TMS, 25 st. C
8
34.14
31.77
29.15
29.11
28.67
77.3
77.1
76.8
60.7
51.9
34.1
31.8
29.2
29.1
28.7
-0.0
34
33
32
31
30
29
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


EE6F3F1
S
S
S
S
S
S
8
%Transmitancji
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
50
48
2953,14
2913,99
2850,58
1438,43
1407,53
1366,38
1304,62
1252,72
1217,21
1059,25
984,84
905,97
888,55
843,54
731,49
686,20
662,59
4000
3500
3000
2500
2000
1500
1000
500
Liczby falowe (cm-1)



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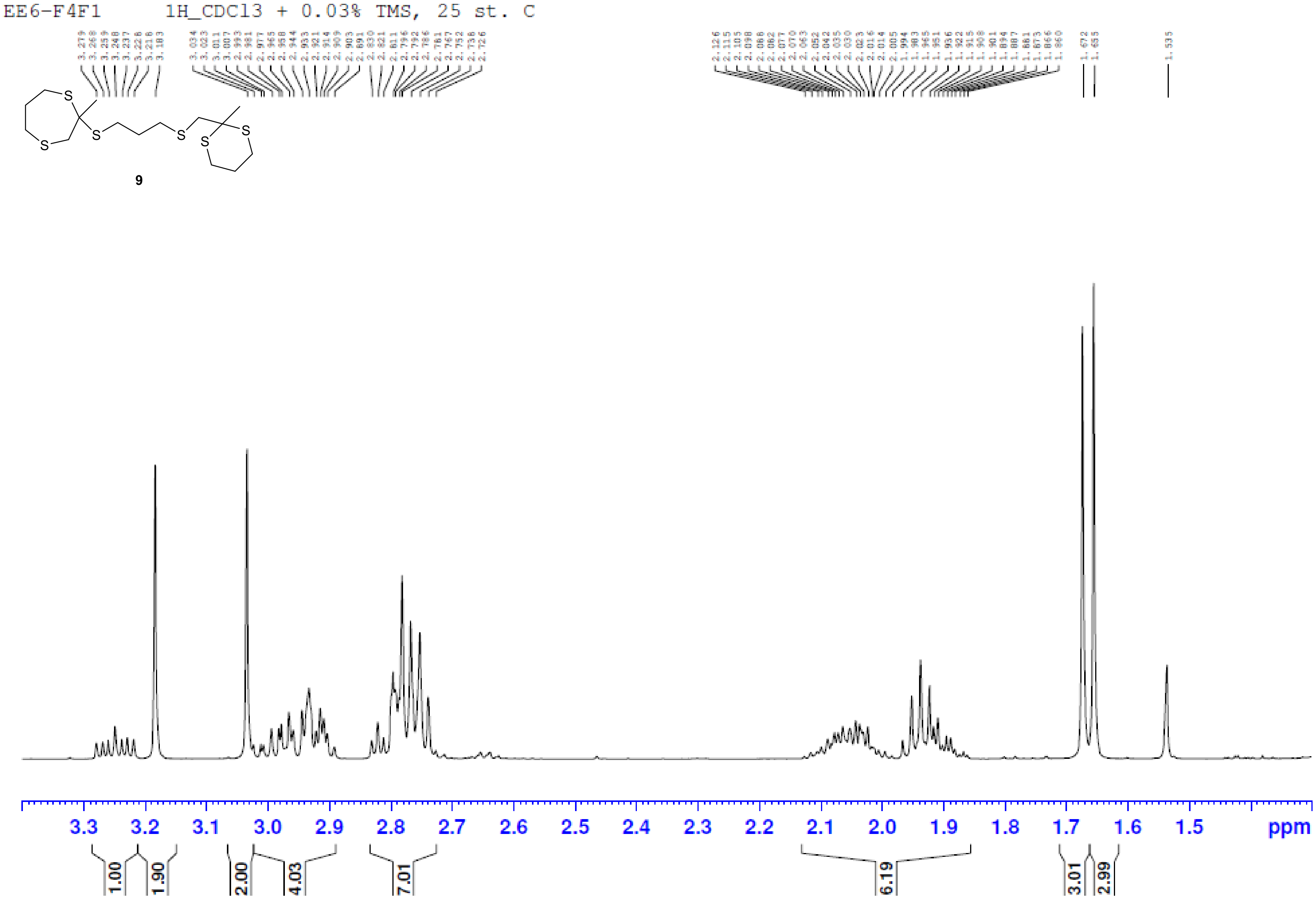
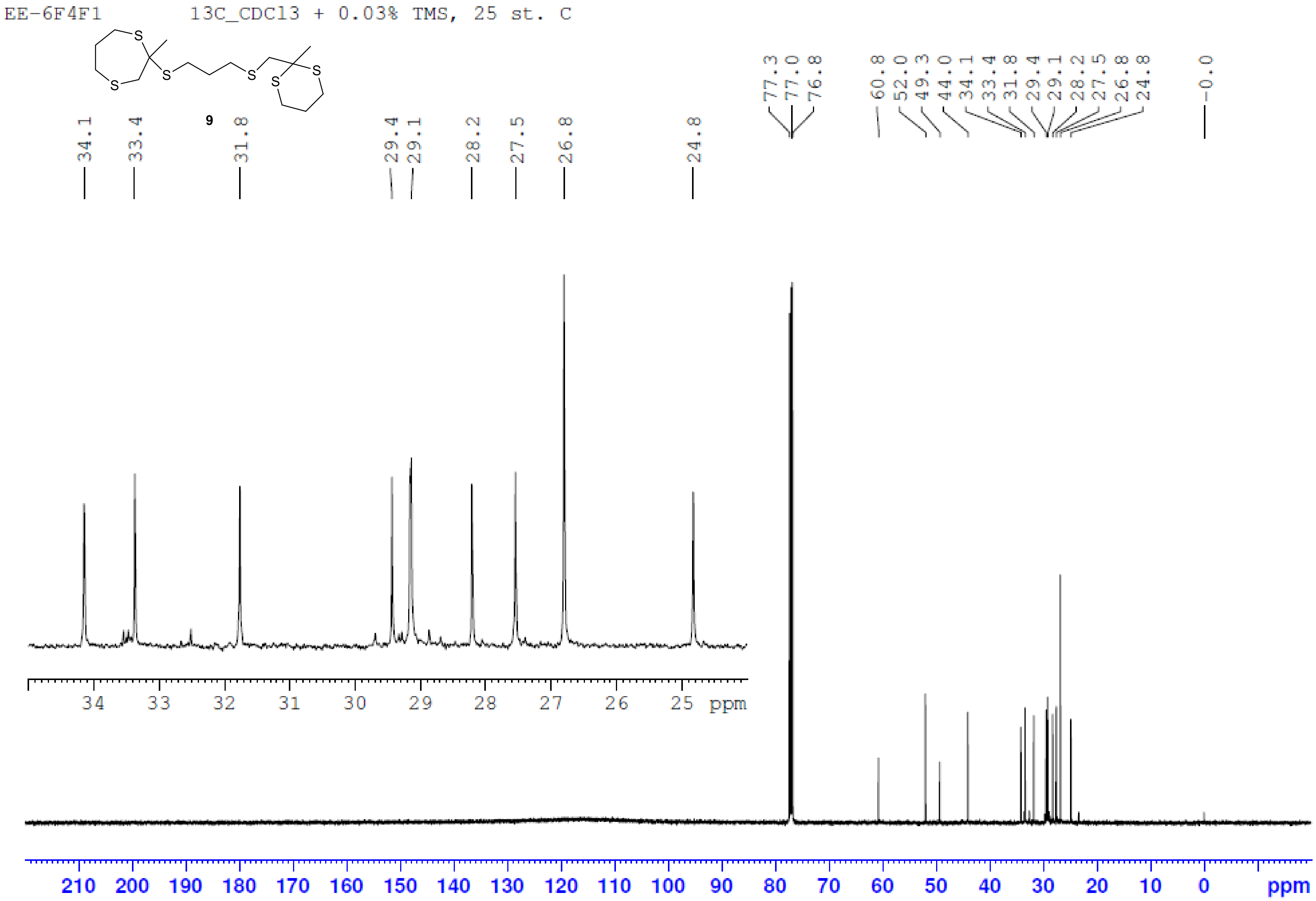
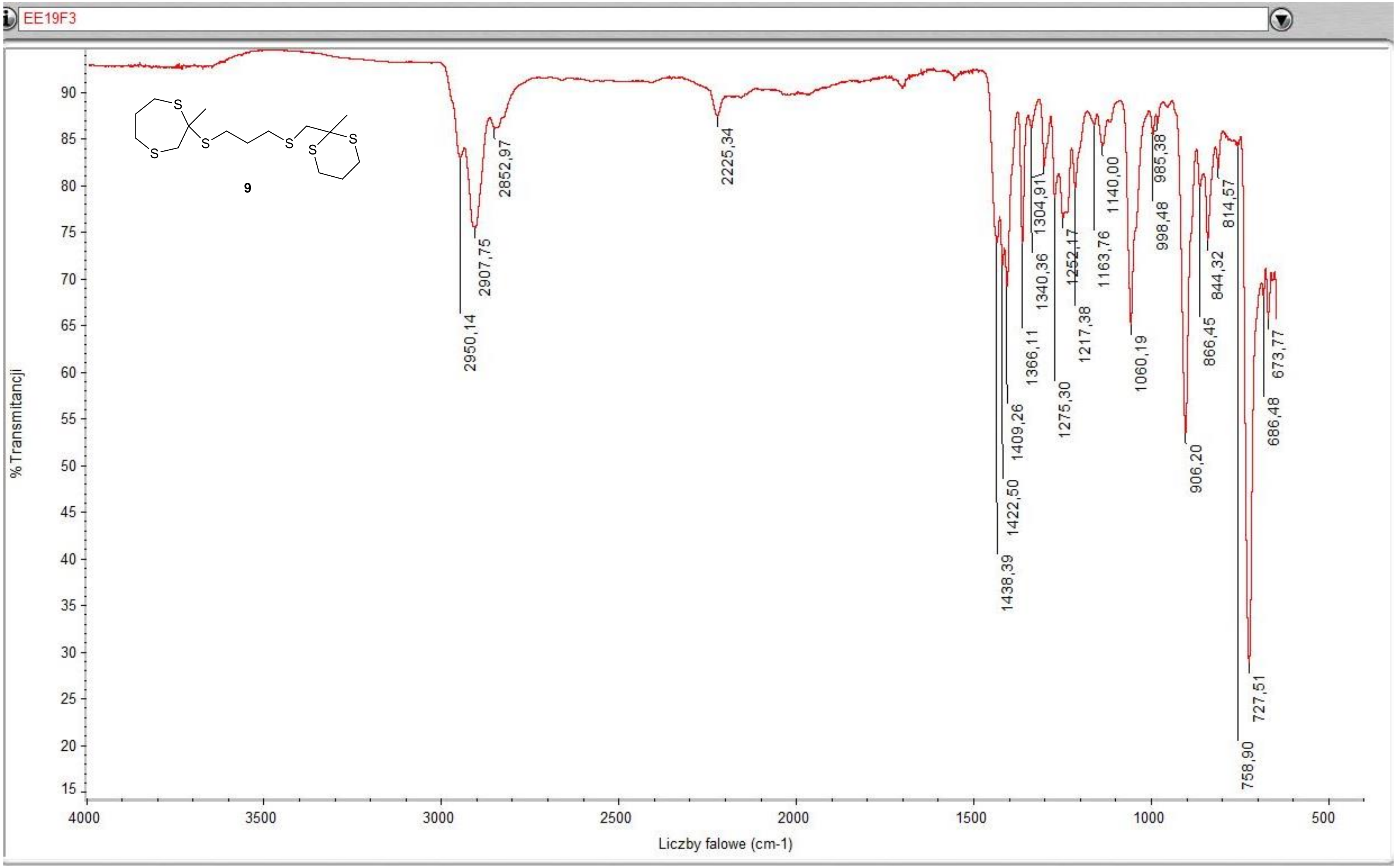
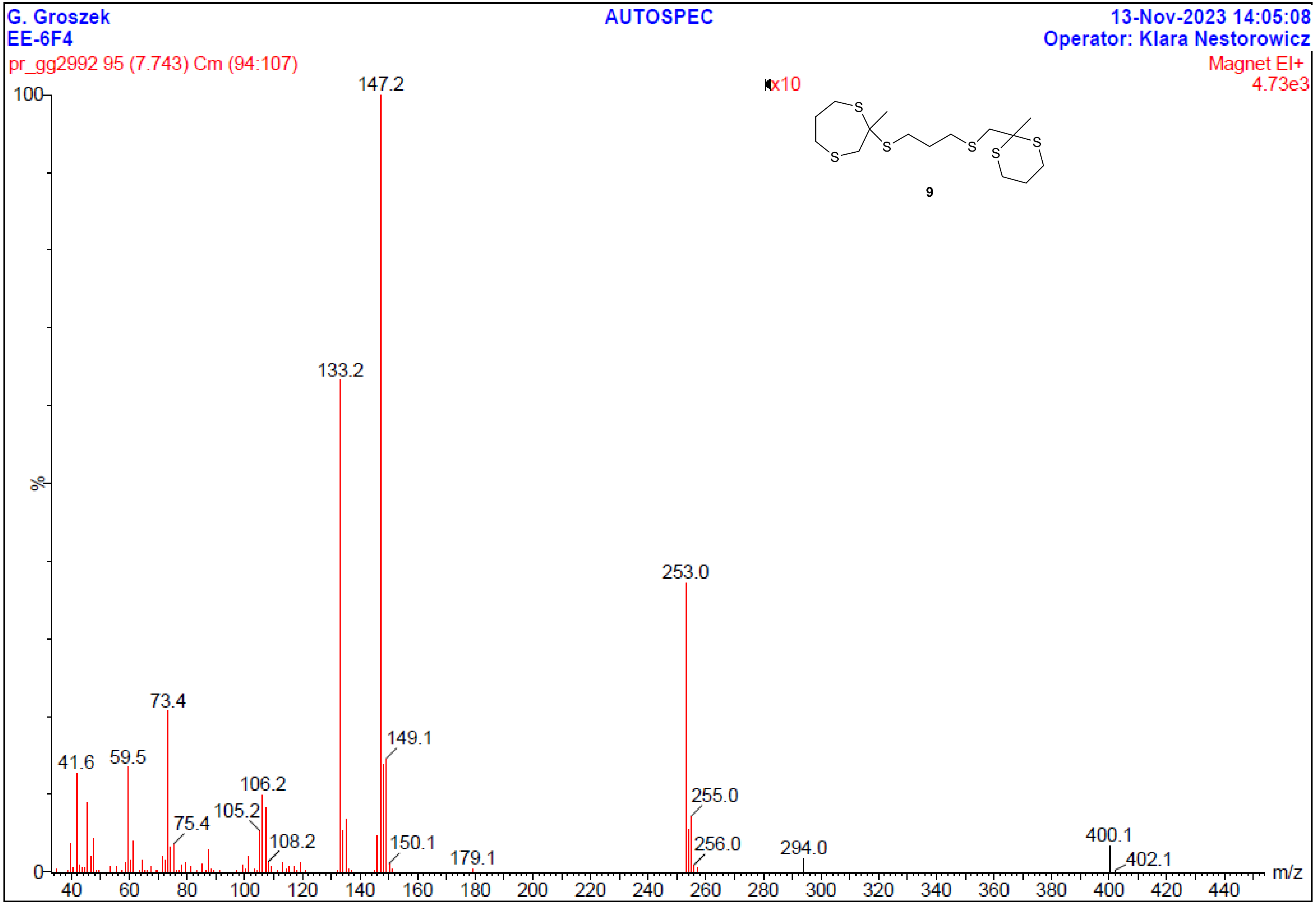
EE6-F4F1 1H_CDCl3 + 0.03% TMS, 25 st. C
7.261
3.279
3.268
3.259
3.248
3.237
3.228
3.218
3.183
3.034
3.023
3.011
3.007
2.993
2.981
2.977
2.965
2.956
2.944
2.933
2.921
2.914
2.909
2.903
2.891
2.830
2.821
2.811
2.796
2.792
2.786
2.781
2.767
2.752
2.738
2.726
2.126
2.115
2.105
2.098
2.088
2.082
2.077
2.070
2.063
2.052
2.042
2.035
2.030
2.023
2.016
2.014
2.005
1.994
1.983
1.965
1.951
1.936
1.922
1.915
1.908
1.901
1.894
1.887
1.881
1.873
1.866
1.860
1.672
1.655
1.535
-0.000
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
1.90
2.00
4.03
7.01
6.19
3.01
2.99


EE6-F4F1 1H_CDCl3 + 0.03% TMS, 25 st. C
3.279
3.268
3.259
3.248
3.237
3.226
3.216
3.183
3.034
3.023
3.011
3.007
2.993
2.981
2.977
2.965
2.958
2.944
2.933
2.921
2.914
2.909
2.903
2.891
2.830
2.821
2.811
2.796
2.792
2.786
2.761
2.757
2.752
2.736
2.726
2.126
2.115
2.105
2.098
2.086
2.082
2.077
2.070
2.063
2.052
2.042
2.035
2.030
2.023
2.016
2.014
2.005
1.994
1.983
1.965
1.951
1.936
1.922
1.915
1.908
1.901
1.894
1.887
1.881
1.873
1.866
1.860
1.672
1.655
1.535
S
S
S
S
S
S
9
3.3
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
1.7
1.6
1.5
ppm
1.00
1.90
2.00
4.03
7.01
6.19
3.01
2.99

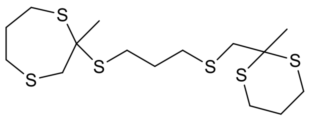

EE-6F4F1
13C_CDCl3 + 0.03% TMS, 25 st. C
S
S
S
S
S
S
34.1
33.4
31.8
29.4
29.1
28.2
27.5
26.8
24.8
77.3
77.0
76.8
60.8
52.0
49.3
44.0
34.1
33.4
31.8
29.4
29.1
28.2
27.5
26.8
24.8
-0.0
34
33
32
31
30
29
28
27
26
25
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

9


EE19F3
9
% Transmitancji
Liczby falowe (cm-1)
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
4000
3500
3000
2500
2000
1500
1000
500
2950,14
2907,75
2852,97
2225,34
1438,39
1422,50
1409,26
1366,11
1340,36
1304,91
1275,30
1252,17
1217,38
1163,76
1140,00
1060,19
998,48
985,38
906,20
866,45
844,32
814,57
758,90
727,51
686,48
673,77


G. Groszek
EE-6F4
AUTOSPEC
13-Nov-2023 14:05:08
Operator: Klara Nestorowicz
pr_gg2992 95 (7.743) Cm (94:107)
Magnet EI+
4.73e3
x10
S
S
S
S
S
S
9
100
%
0
147.2
133.2
253.0
73.4
41.6
59.5
106.2
105.2
75.4
108.2
149.1
150.1
179.1
255.0
256.0
294.0
400.1
402.1
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440
m/z

# Elemental Composition Report



**9**

Page 1

## Single Mass Analysis

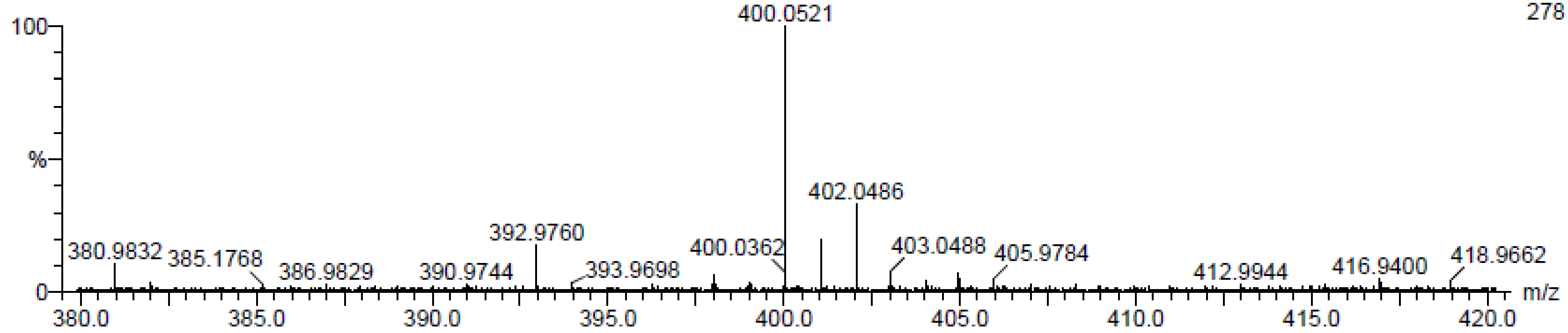
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 150.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
33 formula(e) evaluated with 3 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-200 S: 0-6

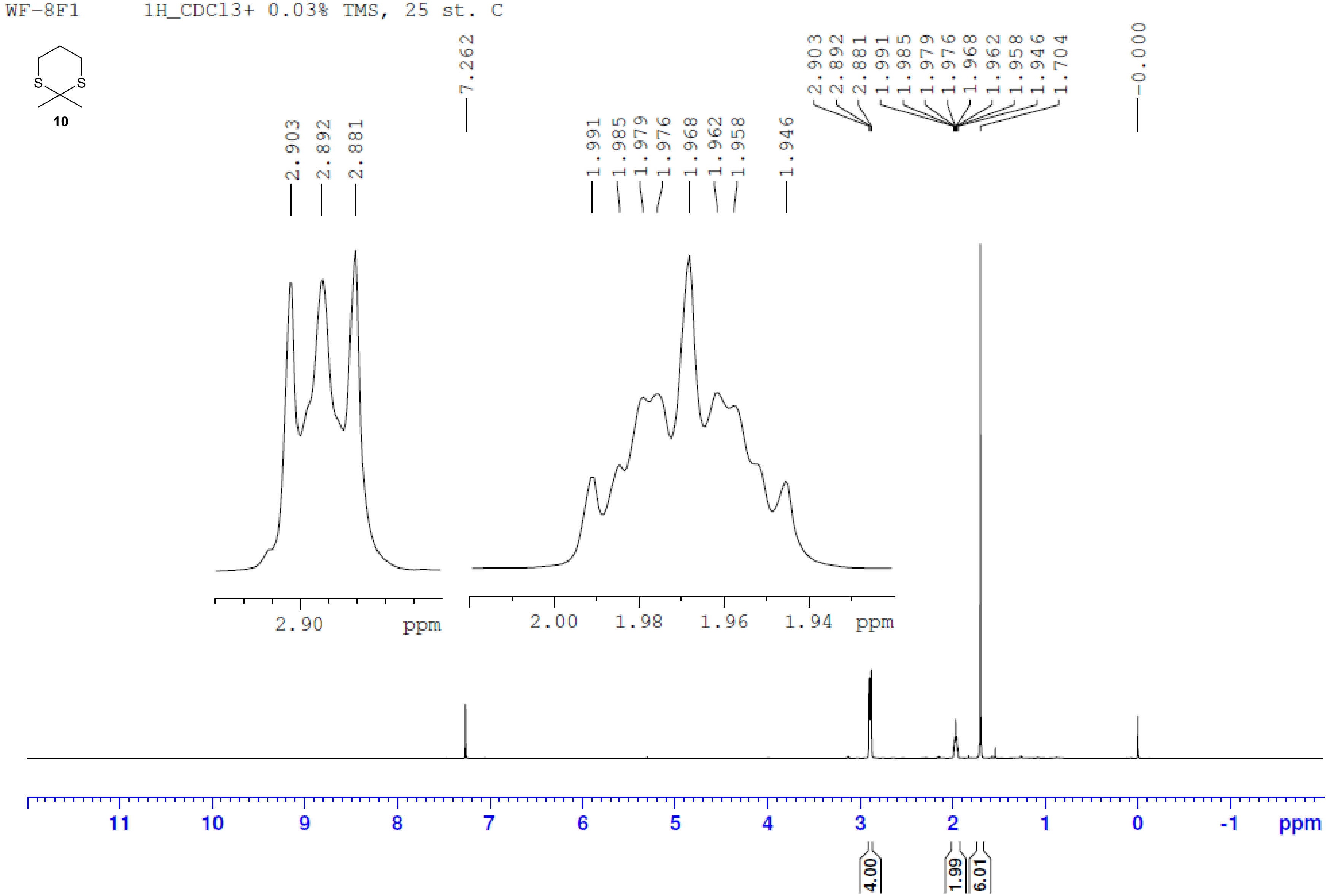
G. Groszek
EE-6F4
pr_gg2992h 682 (26.013) Cm (680:701)

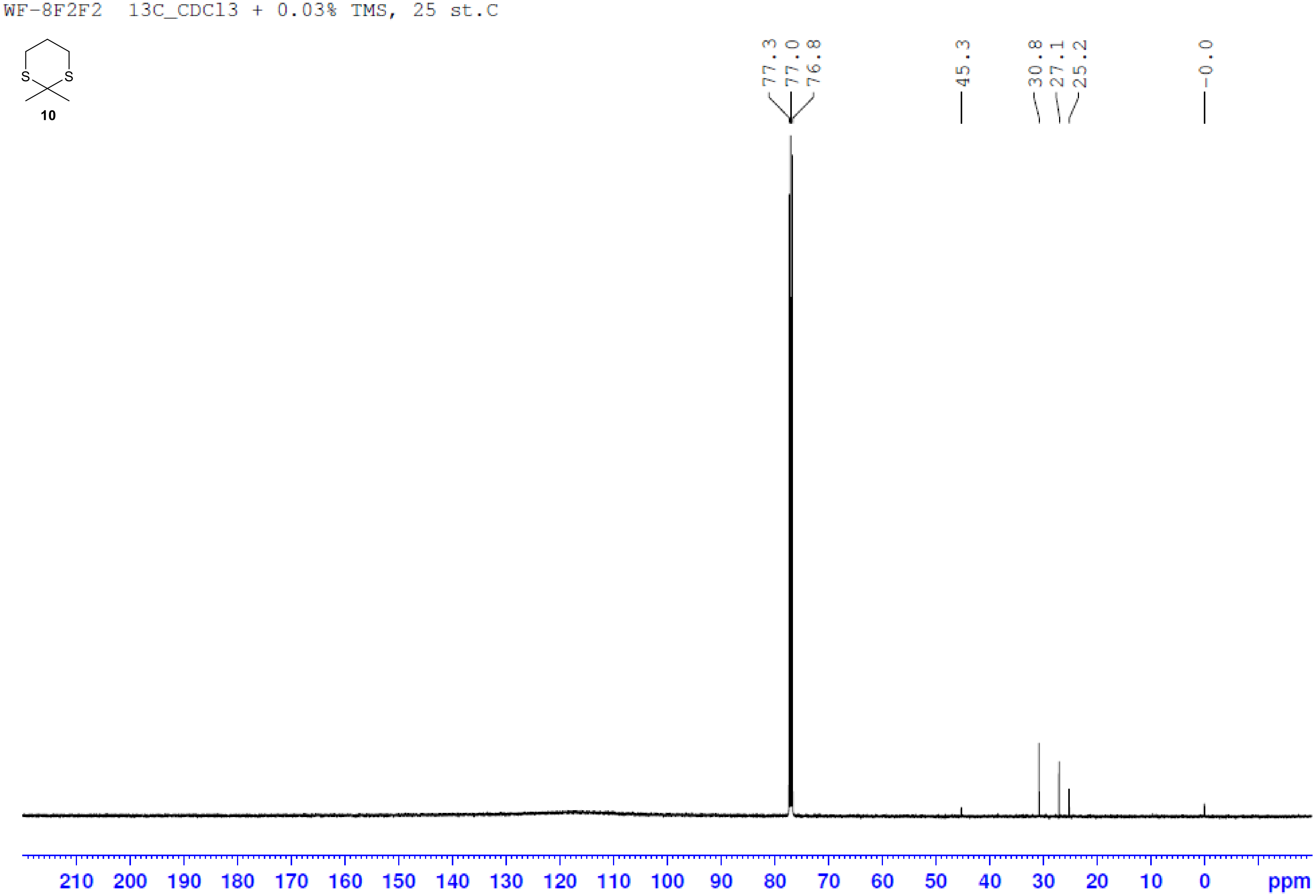
AUTOSPEC

15-Nov-2023 13:12:08
Operator: Klara Nestorowicz
Voltage EI+
278



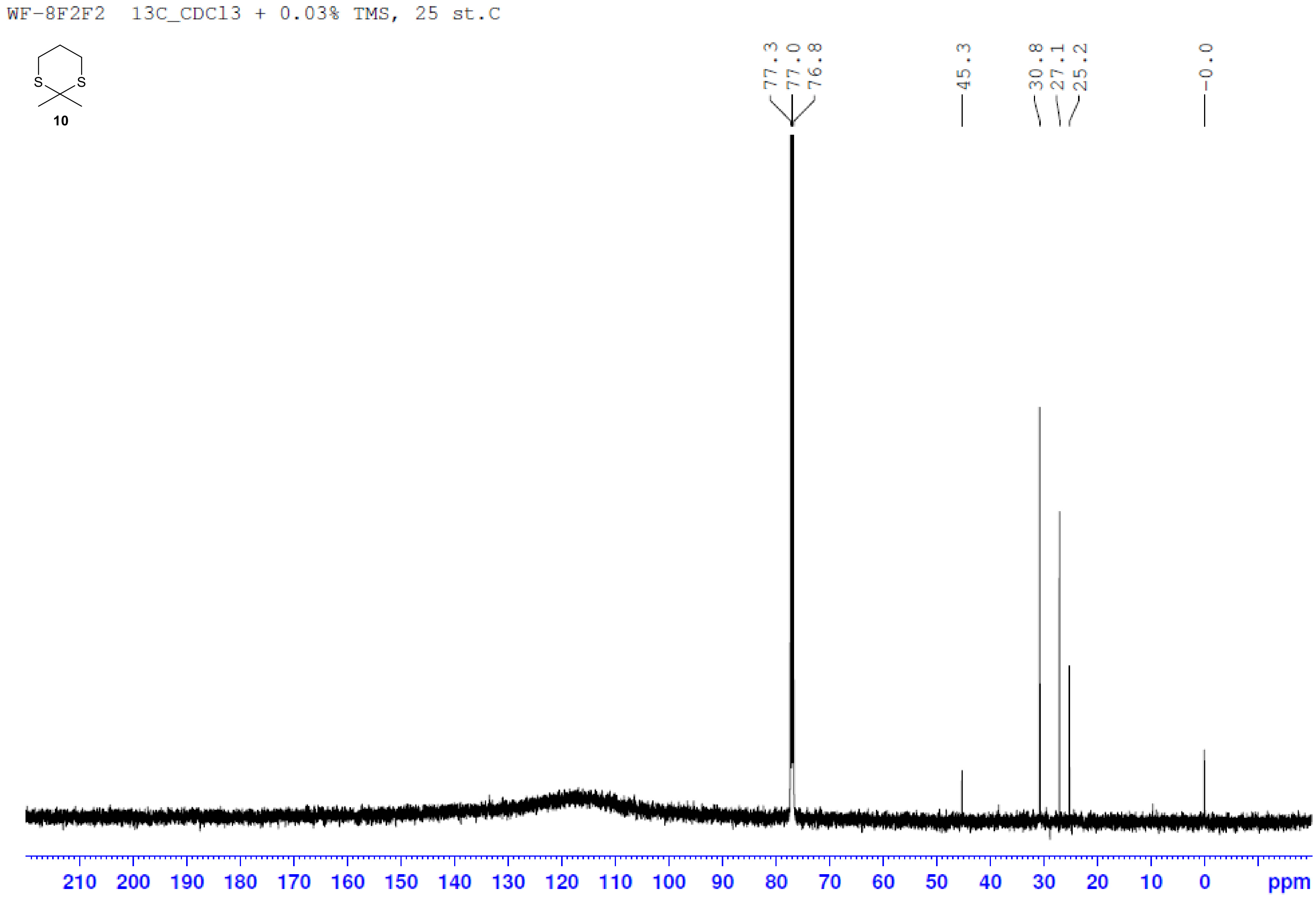
| | | | | | | |
|---|---|---|---|---|---|---|
| Minimum: | | | | -1.5 | | |
| Maximum: | | 5.0 | 20.0 | 150.0 | | |
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
| 400.0521 | 400.0515 | 0.6 | 1.5 | 2.0 | 1.2 | C15 H28 S6 |
| | 400.0482 | 3.9 | 9.7 | 7.0 | 4.6 | C18 H24 S5 |
| | 400.0448 | 7.3 | 18.2 | 12.0 | 10.2 | C21 H20 S4 |

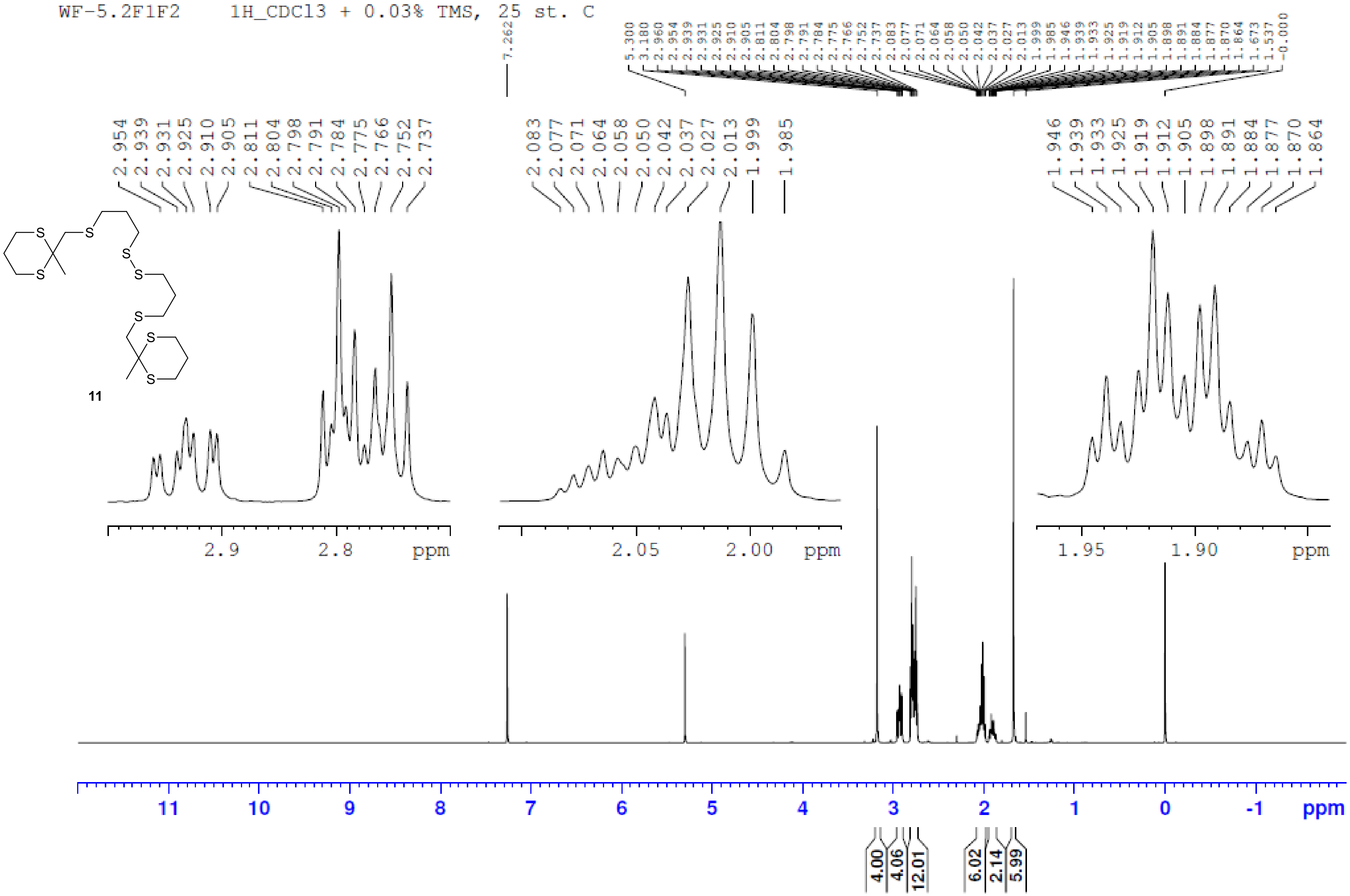

WF-8F1 1H_CDCl3+ 0.03% TMS, 25 st. C
S
S
10
2.903
2.892
2.881
7.262
1.991
1.985
1.979
1.976
1.968
1.962
1.958
1.946
1.704
-0.000
2.90
ppm
2.00
1.98
1.96
1.94
ppm
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.00
1.99
6.01



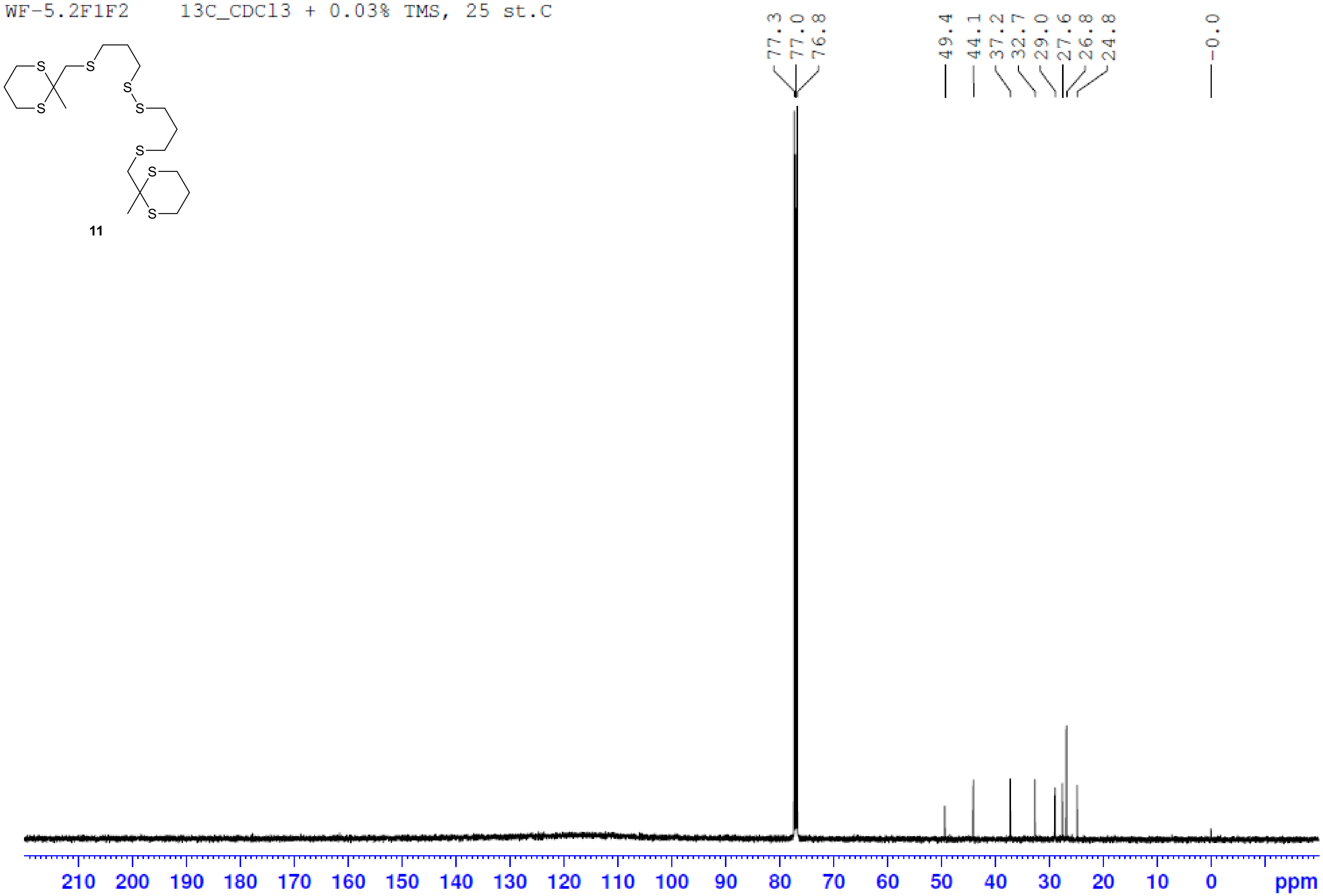
**10**

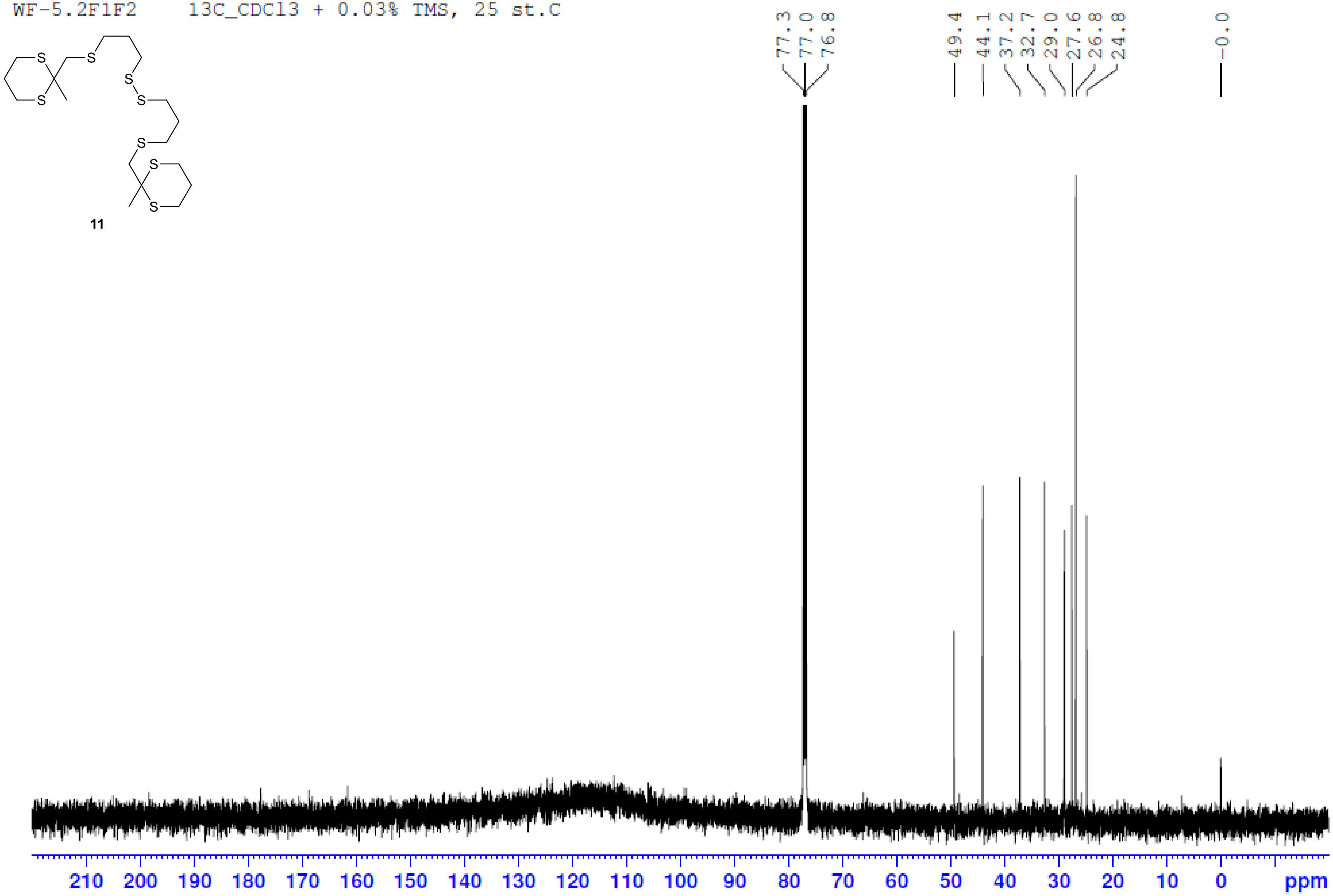
WF-8F2F2 13C_CDCl3 + 0.03% TMS, 25 st.C
77.3
77.0
76.8
45.3
30.8
27.1
25.2
-0.0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


WF-8F2F2 13C_CDCl3 + 0.03% TMS, 25 st.C
S
S
10
77.3
77.0
76.8
45.3
30.8
27.1
25.2
-0.0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

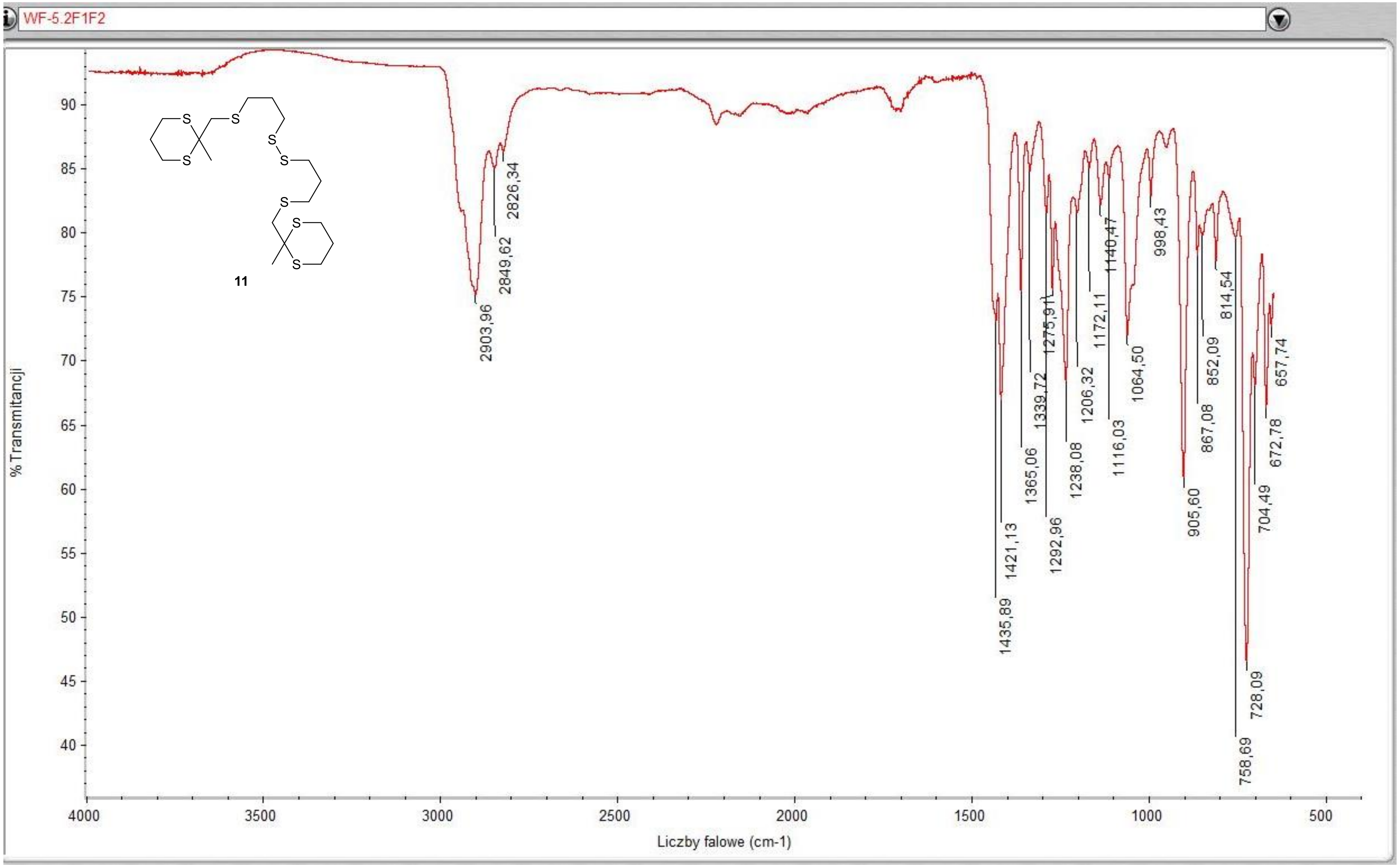

WF-5.2F1F2 1H_CDCl3 + 0.03% TMS, 25 st. C
7.262
5.300
3.180
2.960
2.954
2.939
2.931
2.925
2.910
2.905
2.811
2.804
2.798
2.791
2.784
2.775
2.766
2.752
2.737
2.083
2.077
2.071
2.064
2.058
2.050
2.042
2.037
2.027
2.013
1.999
1.985
1.946
1.939
1.933
1.925
1.919
1.912
1.905
1.898
1.891
1.884
1.877
1.870
1.864
1.673
1.537
-0.000
S
S
S
S
S
S
S
S
2.9
2.8
ppm
2.05
2.00
ppm
1.95
1.90
ppm
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.00
4.06
12.01
6.02
2.14
5.99

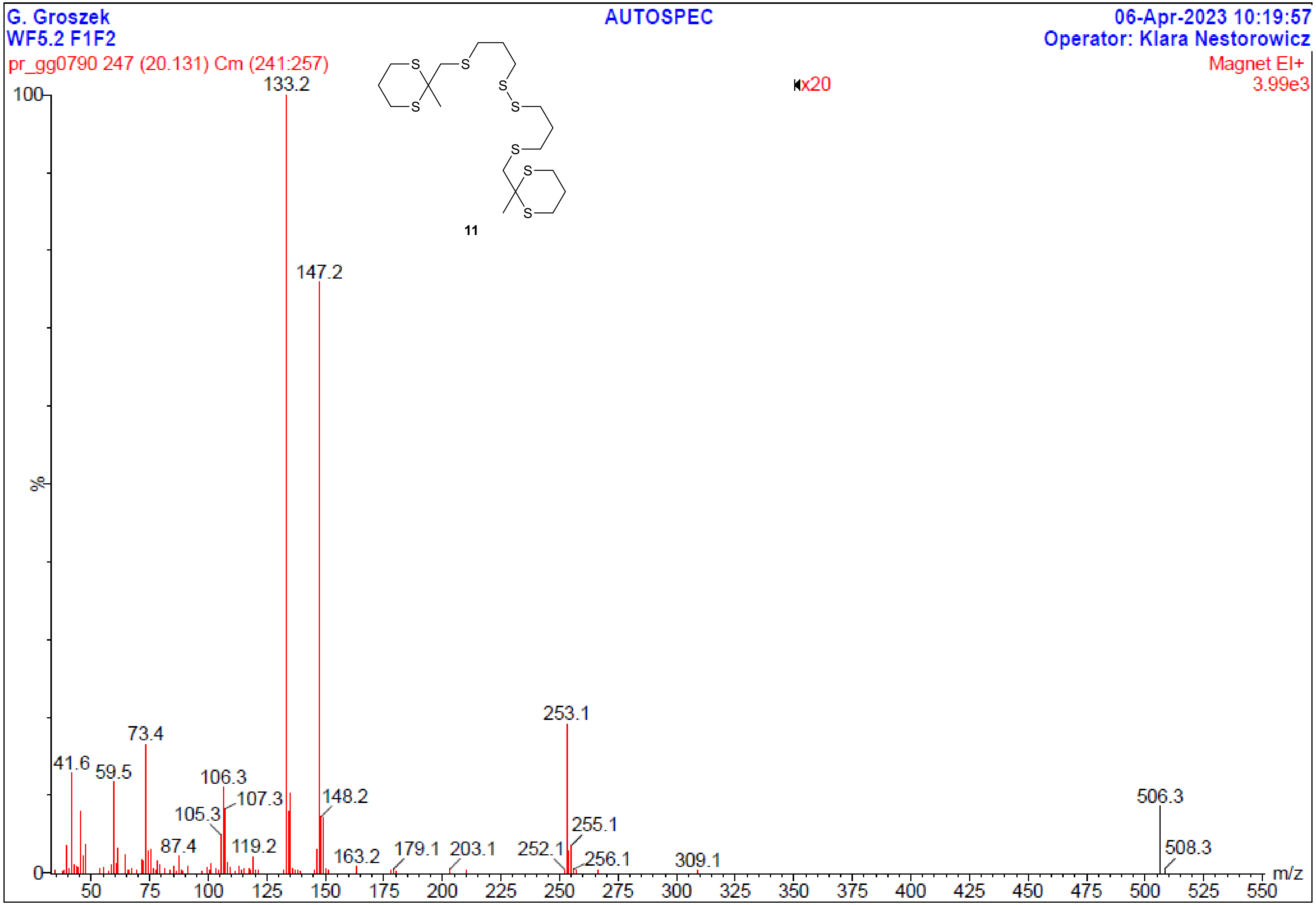
11

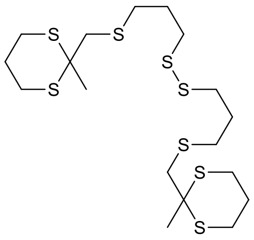

WF-5.2F1F2 13C_CDCl3 + 0.03% TMS, 25 st.C
11
77.3
77.0
76.8
49.4
44.1
37.2
32.7
29.0
27.6
26.8
24.8
-0.0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


WF-5.2F1F2 13C_CDCl3 + 0.03% TMS, 25 st.C
S
S
S
S
S
S
S
S
77.3
77.0
76.8
49.4
44.1
37.2
32.7
29.0
27.6
26.8
24.8
-0.0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

**11**


WF-5.2F1F2
S
S
S
S
S
S
S
S
11
% Transmitancji
90
85
80
75
70
65
60
55
50
45
40
4000
3500
3000
2500
2000
1500
1000
500
Liczby falowe (cm-1)
2903,96
2849,62
2826,34
1435,89
1421,13
1365,06
1339,72
1292,96
1275,91
1238,08
1206,32
1172,11
1140,47
1116,03
1064,50
998,43
905,60
867,08
852,09
814,54
758,69
728,09
704,49
672,78
657,74


G. Groszek
WF5.2 F1F2
pr_gg0790 247 (20.131) Cm (241:257)
AUTOSPEC
06-Apr-2023 10:19:57
Operator: Klara Nestorowicz
Magnet EI+
3.99e3
x20
11
S
100
%
0
133.2
147.2
253.1
73.4
41.6
59.5
106.3
107.3
105.3
148.2
87.4
119.2
163.2
179.1
203.1
252.1
255.1
256.1
309.1
506.3
508.3
50
75
100
125
150
175
200
225
250
275
300
325
350
375
400
425
450
475
500
525
550
m/z



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## Elemental Composition Report

Page 1

### Single Mass Analysis

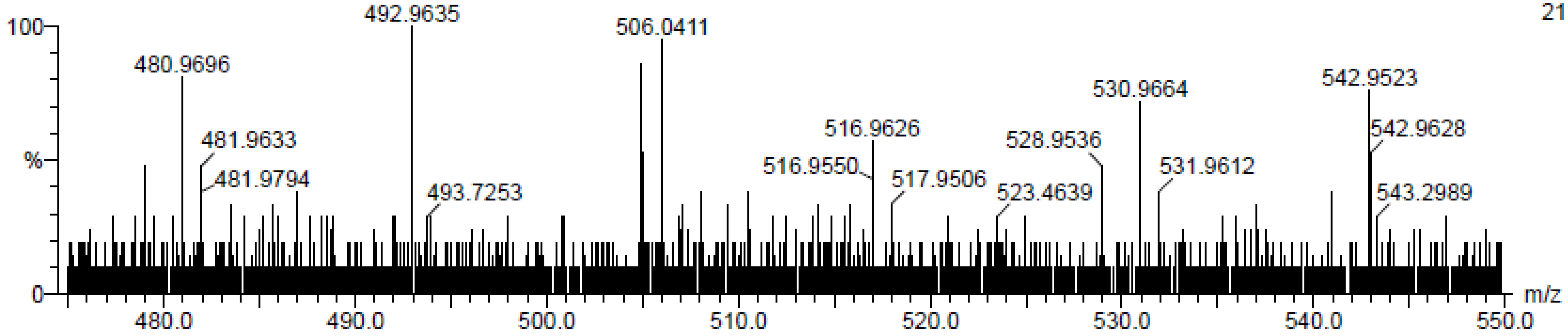
Tolerance = 100.0 PPM / DBE: min = -1.5, max = 150.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
4 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-200 S: 8-8

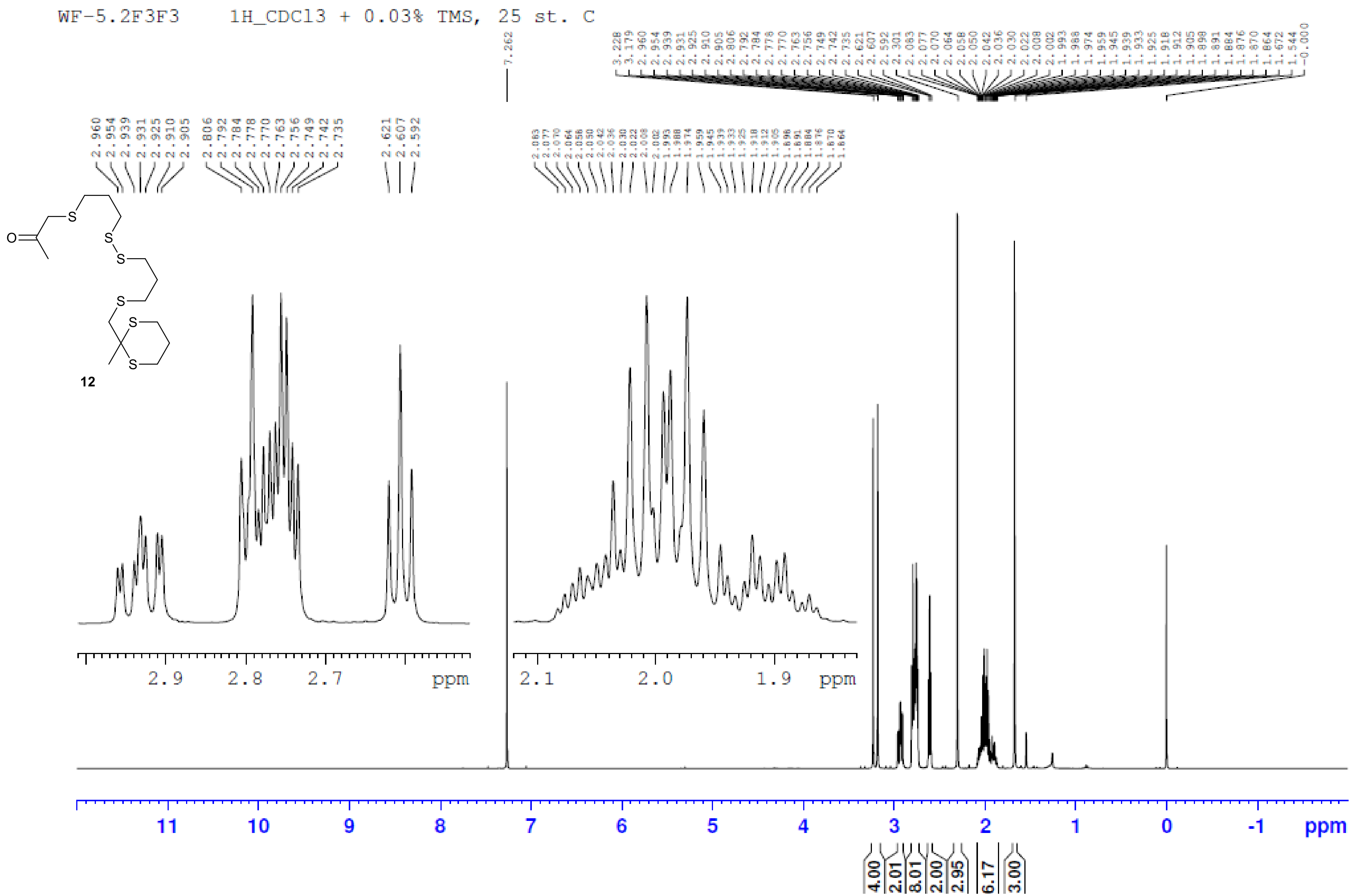
G. Groszek
WF5.2 F1F2
pr_gg0790h 280 (10.680) Cm (270:282)

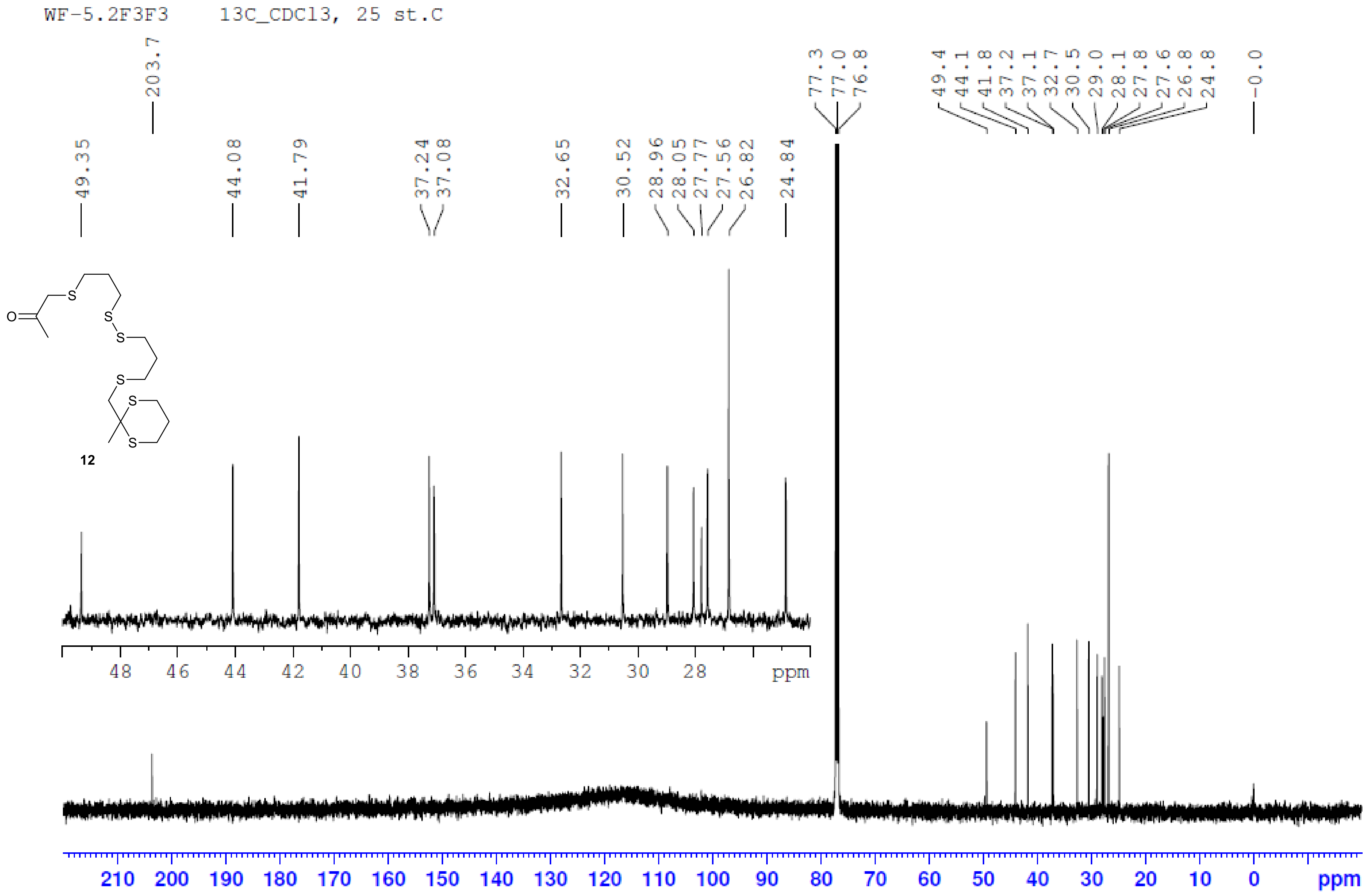
AUTOSPEC

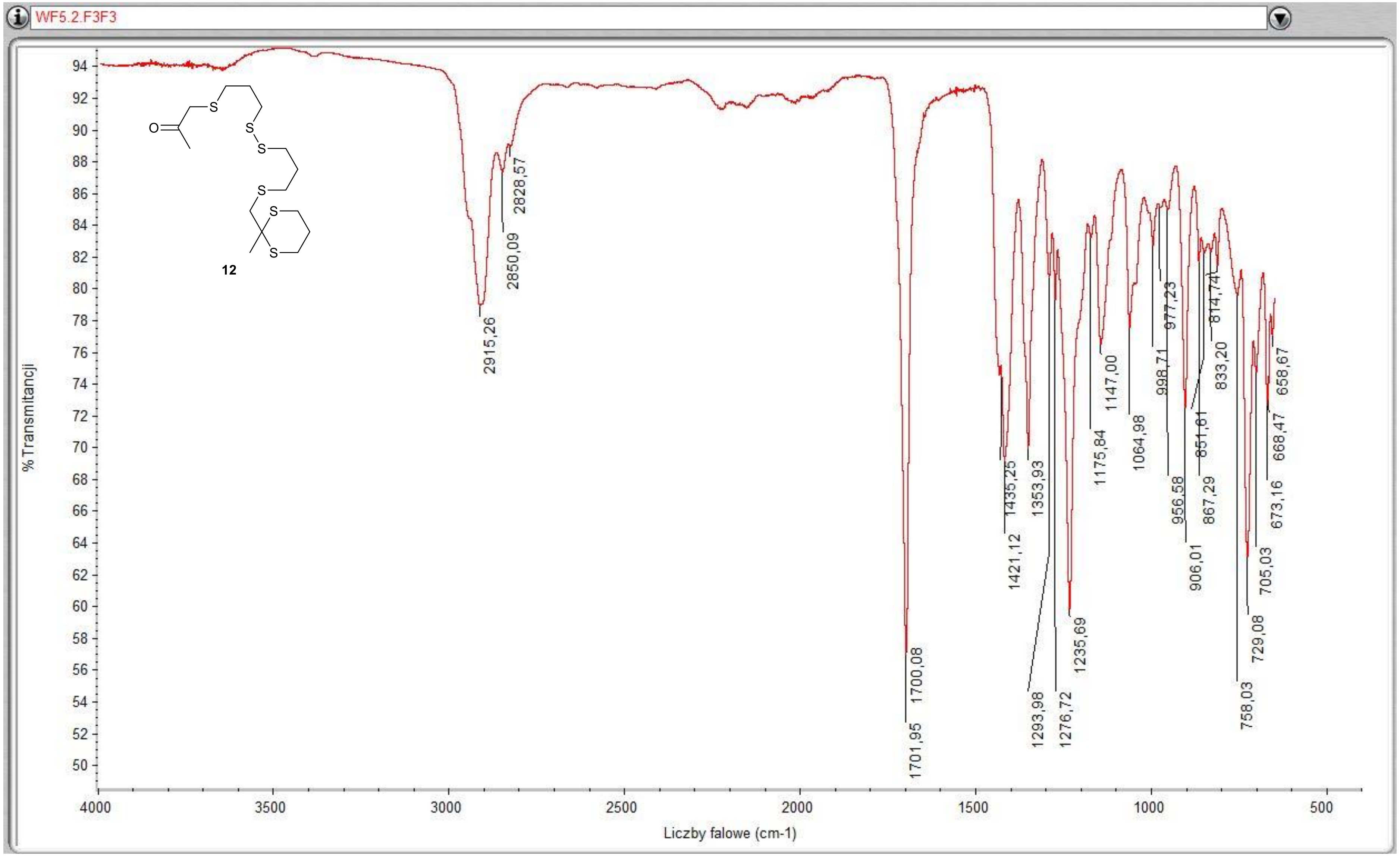
13-Apr-2023 16:36:50
Operator: Marian Olejnik
Voltage EI+
21

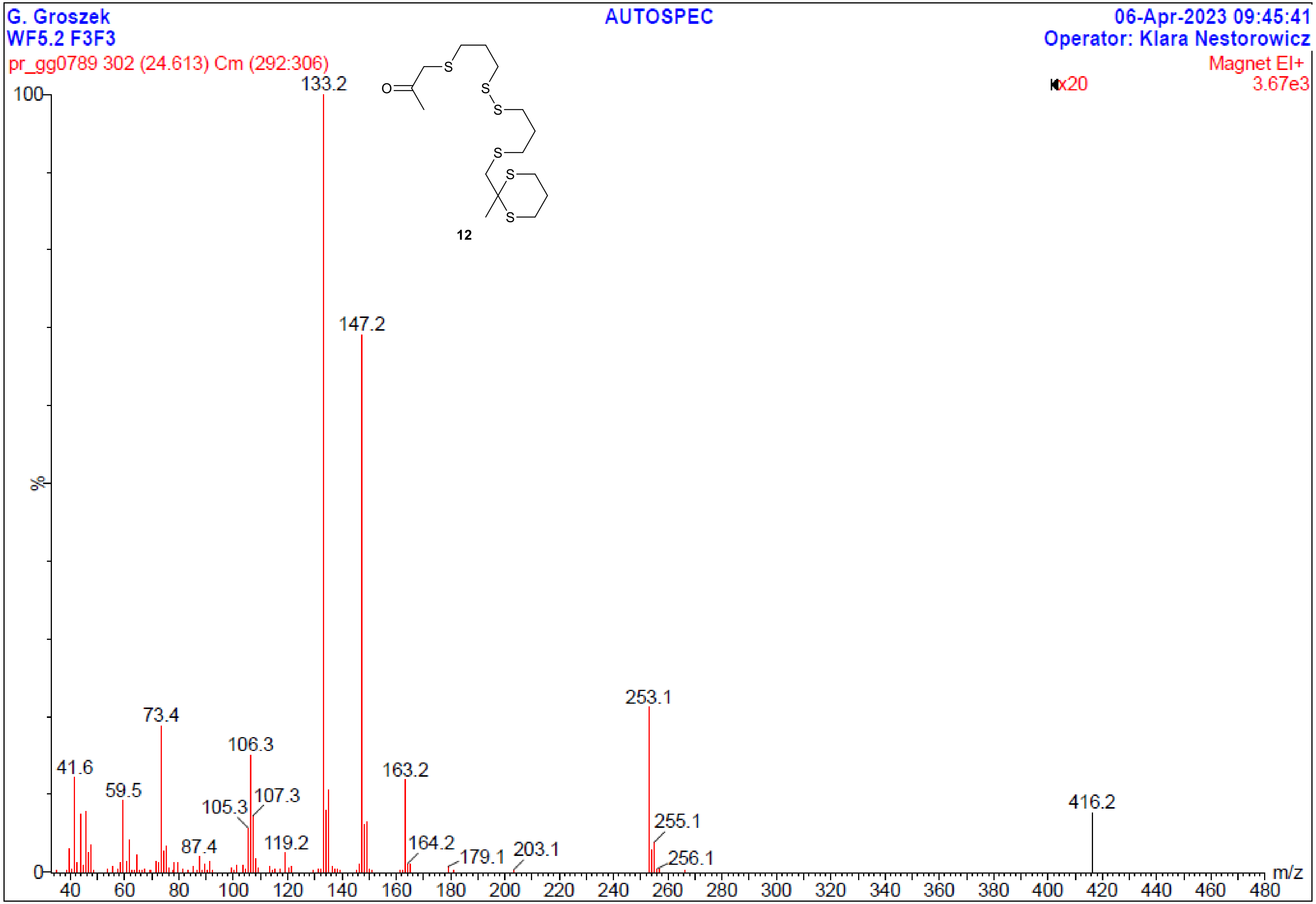


| | Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|---|---|---|---|---|---|---|---|
| Minimum: | | | | | -1.5 | | |
| Maximum: | | | 5.0 | 100.0 | 150.0 | | |
| | 506.0411 | 506.0426 | -1.5 | -3.0 | 2.0 | 0.2 | C18 H34 S8 |

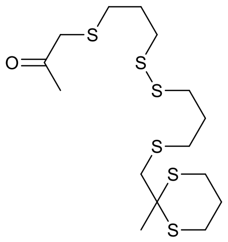

WF-5.2F3F3 1H_CDCl3 + 0.03% TMS, 25 st. C
7.262
3.228
3.179
2.960
2.954
2.939
2.931
2.925
2.910
2.905
2.806
2.792
2.784
2.778
2.770
2.763
2.756
2.749
2.742
2.735
2.621
2.607
2.592
2.301
2.083
2.077
2.070
2.064
2.058
2.050
2.042
2.036
2.030
2.022
2.008
2.002
1.993
1.988
1.974
1.959
1.945
1.939
1.933
1.925
1.918
1.912
1.905
1.898
1.891
1.884
1.876
1.870
1.864
1.672
1.544
-0.000
O
S
S
S
S
S
S
12
2.9
2.8
2.7
ppm
2.1
2.0
1.9
ppm
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.00
2.01
8.01
2.00
2.95
6.17
3.00


WF-5.2F3F3 13C_CDCl3, 25 st.C
203.7
77.3
77.0
76.8
49.4
44.1
41.8
37.2
37.1
32.7
30.5
29.0
28.1
27.8
27.6
26.8
24.8
-0.0
49.35
44.08
41.79
37.24
37.08
32.65
30.52
28.96
28.05
27.77
27.56
26.82
24.84
O
S
S
S
S
S
S
12
48
46
44
42
40
38
36
34
32
30
28
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


WF5.2.F3F3
% Transmitancji
Liczby falowe (cm-1)
94
92
90
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
58
56
54
52
50
4000
3500
3000
2500
2000
1500
1000
500
12
2915,26
2850,09
2828,57
1701,95
1700,08
1435,25
1421,12
1353,93
1293,98
1276,72
1235,69
1175,84
1147,00
1064,98
998,71
977,23
956,58
906,01
867,29
851,61
833,20
814,74
758,03
729,08
705,03
673,16
668,47
658,67


G. Groszek
WF5.2 F3F3
AUTOSPEC
06-Apr-2023 09:45:41
Operator: Klara Nestorowicz
pr_gg0789 302 (24.613) Cm (292:306)
Magnet EI+
3.67e3
x20
12
100
%
0
133.2
147.2
253.1
73.4
106.3
41.6
163.2
59.5
105.3
107.3
416.2
255.1
87.4
119.2
164.2
179.1
203.1
256.1
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440
460
480
m/z

## Elemental Composition Report



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Page 1

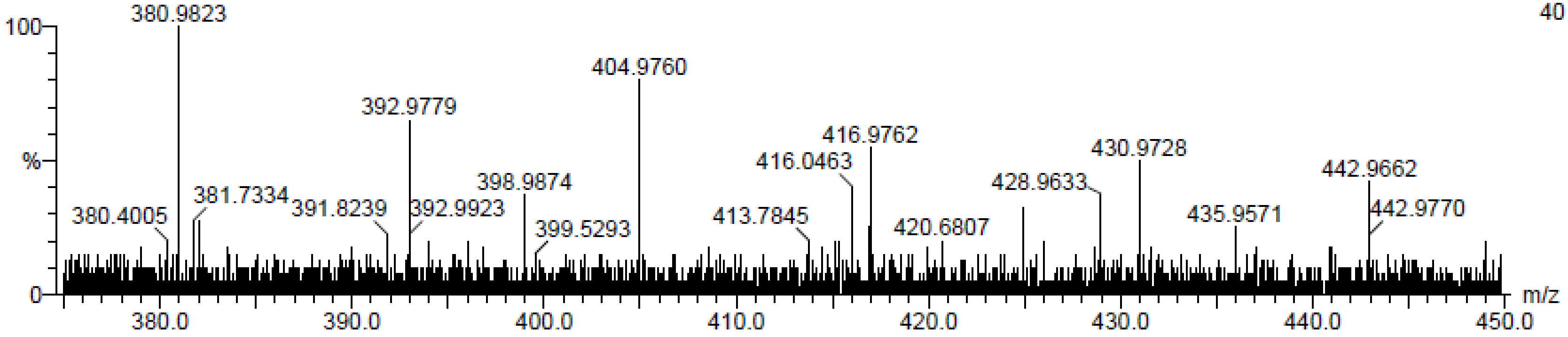
### Single Mass Analysis
Tolerance = 100.0 PPM / DBE: min = -1.5, max = 150.0
Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions
4 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-200 O: 1-1 S: 6-6

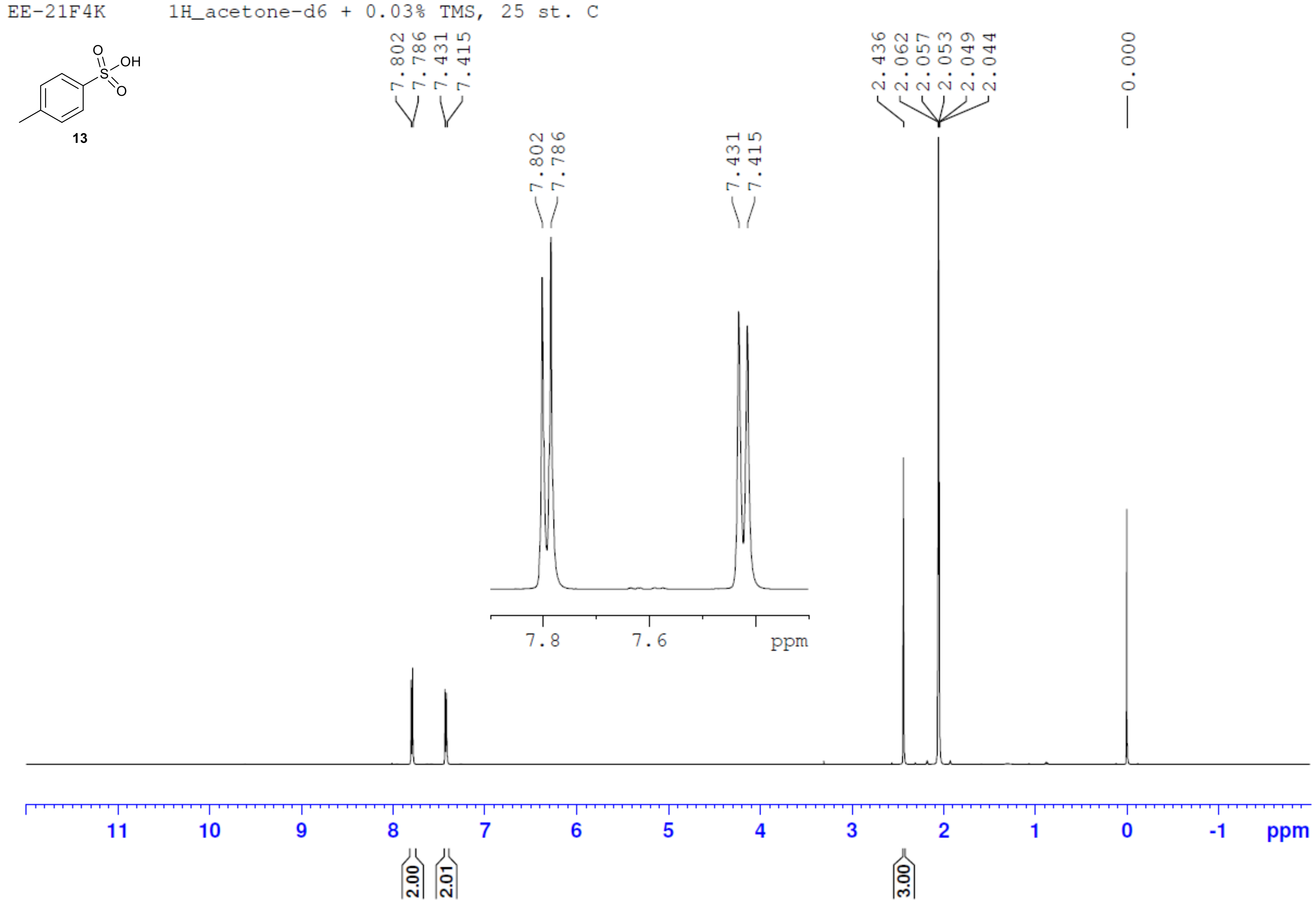
G. Groszek
WF5.2 F3F3
pr_gg0789h 347 (13.235) Cm (343:359)

AUTOSPEC

13-Apr-2023 16:14:41
Operator: Marian Olejnik
Voltage EI+
40



| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
|---|---|---|---|---|---|---|
| Minimum: | | | | -1.5 | | |
| Maximum: | | 5.0 | 100.0 | 150.0 | | |
| 416.0463 | 416.0464 | -0.1 | -0.2 | 2.0 | 0.5 | C15 H28 O S6 |


EE-21F4K 1H_acetone-d6 + 0.03% TMS, 25 st. C
13
7.802
7.786
7.431
7.415
2.436
2.062
2.057
2.053
2.049
2.044
0.000
7.8
7.6
ppm
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.01
3.00