*Chemistry of Heterocyclic Compounds* **2024**, *60*(9/10), 524–528

# Selective reduction of 8-hydroxy-5,7-dinitroquinoline and synthesis of 2-substituted 5-nitrooxazolo[4,5-*h*]quinolines

**Il'ya I. Ustinov[1]*, Nikolai V. Khlytin[2]**

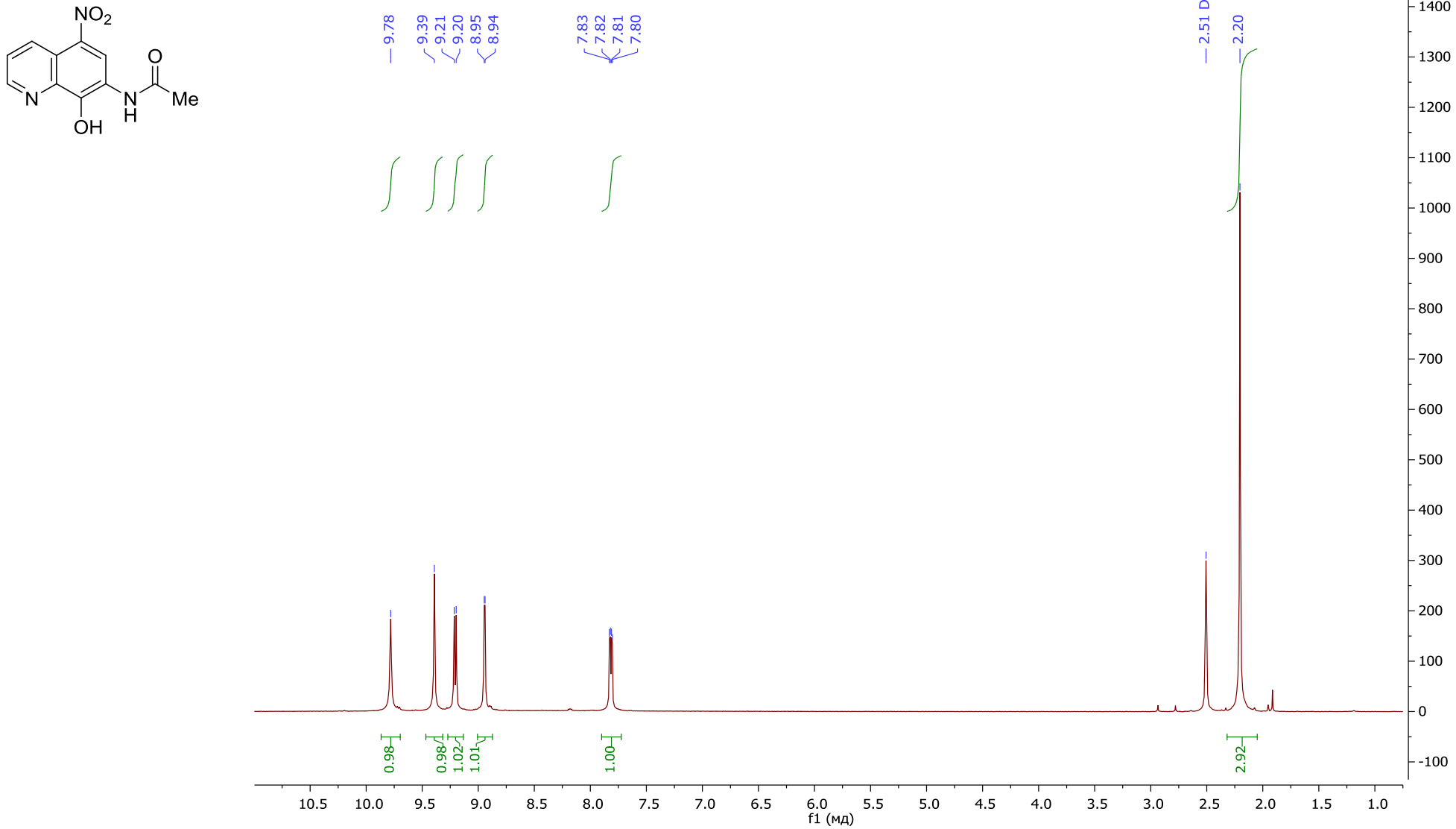
[1] *Tula State Lev Tolstoy Pedagogical University,*
*125 Lenina Ave., Tula 300026, Russia; e-mail: bai2688@yandex.ru*

[2] *Tula Pharmaceutical Factory,*
*10 Torkhovsky Proyezd, Tula 300004, Russia*

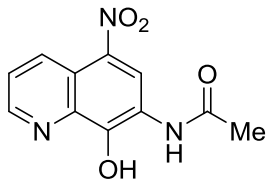
# SUPPLEMENTARY INFORMATION

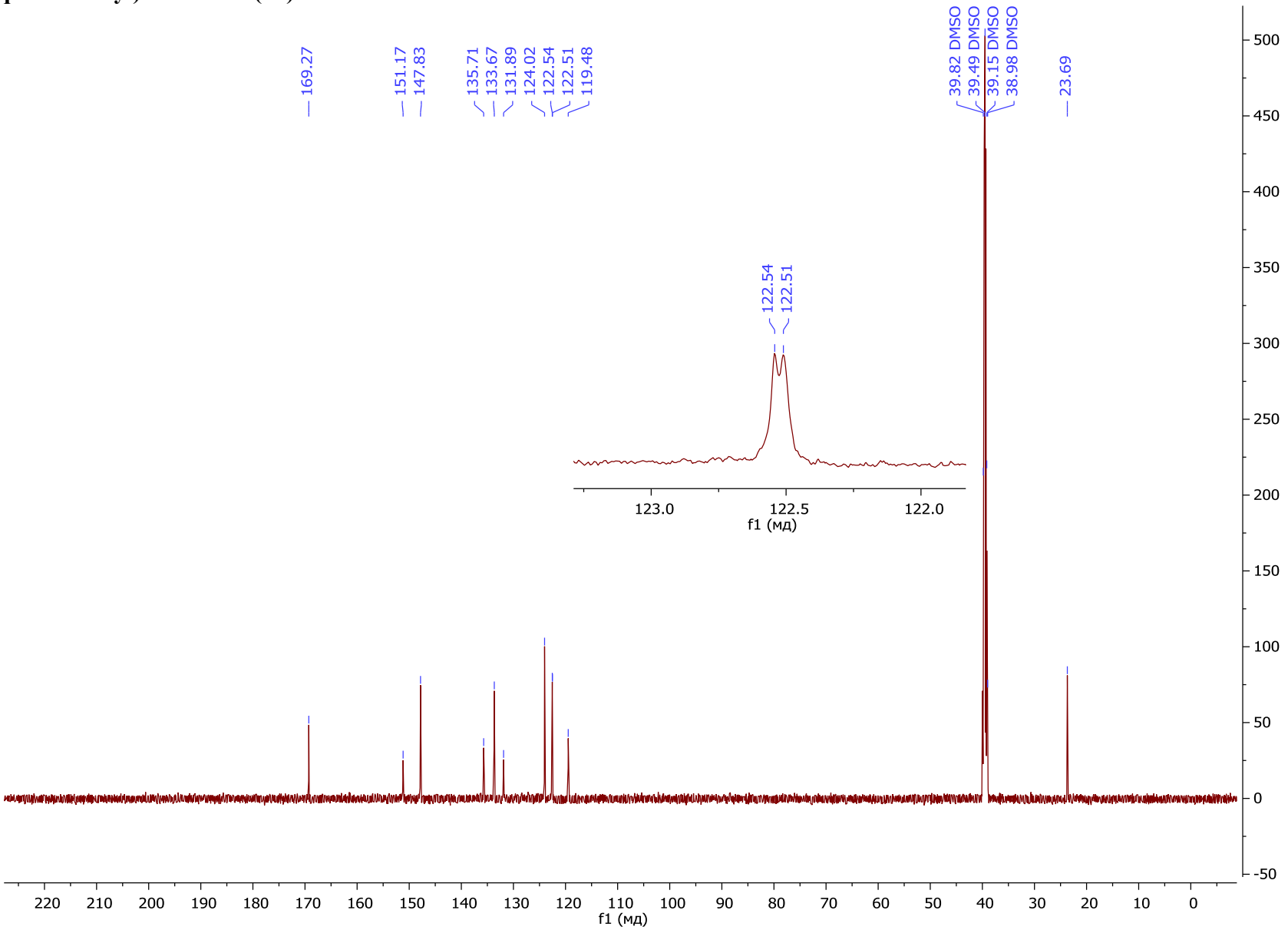
## Copies of NMR, HRMS and FT-IR spectra

**_N_-(8-hydroxy-5-nitroquinolin-7-yl)acetamide (2a) $^{1}$H NMR**

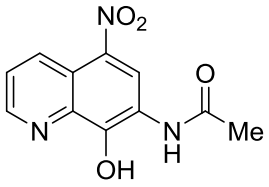


**_N_-(8-hydroxy-5-nitroquinolin-7-yl)acetamide (2a) $^{13}$C NMR**



## *N*-(8-hydroxy-5-nitroquinolin-7-yl)acetamide (2a) HRMS



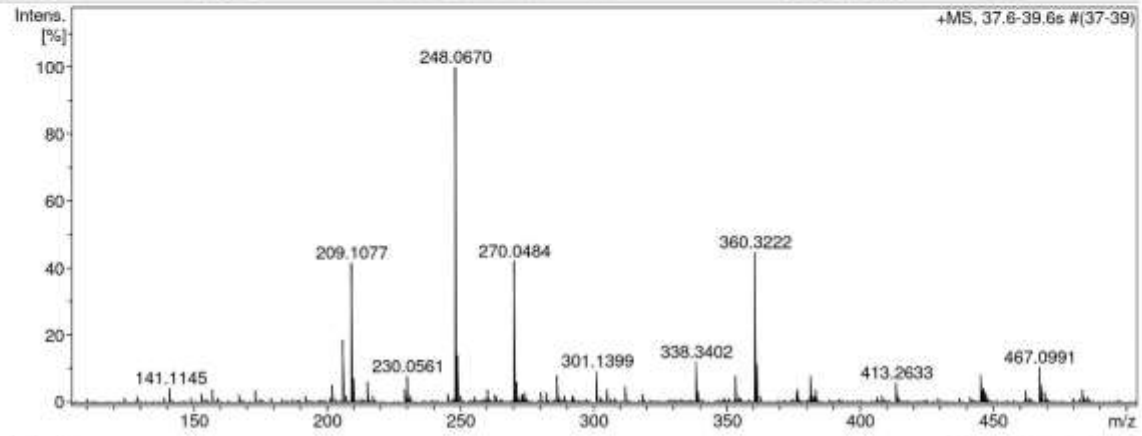

# Display Report

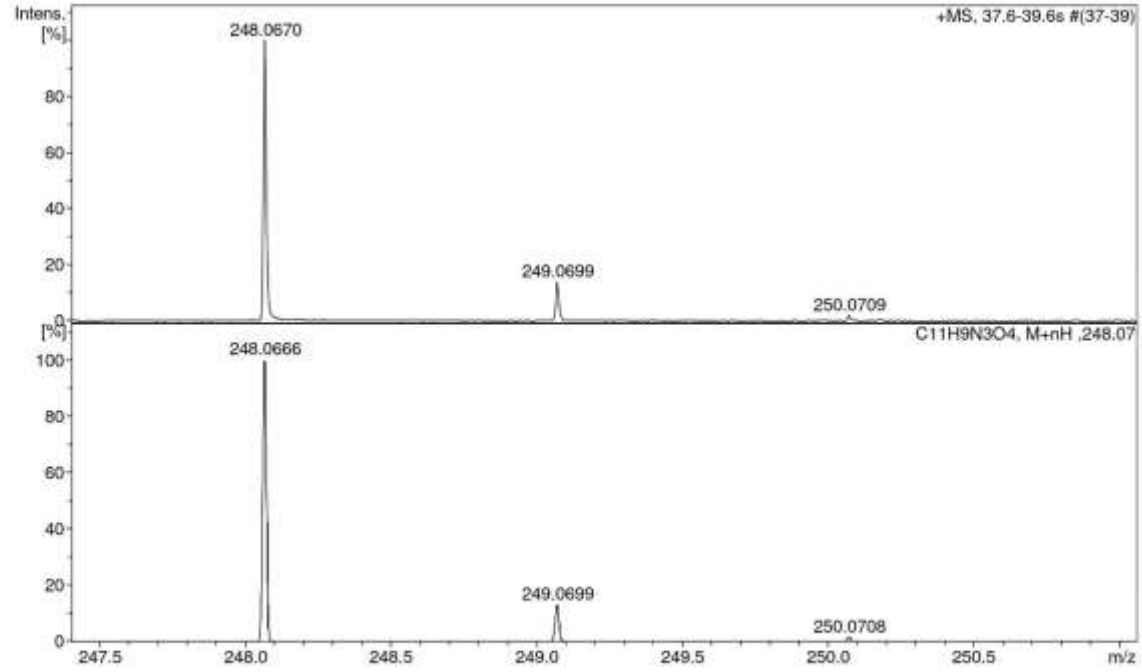
**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\Ilyushenkova\ULI-192.d | Acquisition Date | 26.09.2023 19:19:50 |
| Method | tune_100-1200.m | Operator | BDAL@DE |
| Sample Name | | Instrument / Ser# | maXis 43 |
| Comment | | | |

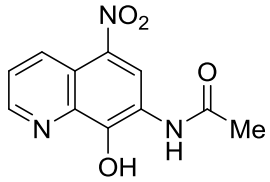
**Acquisition Parameter**

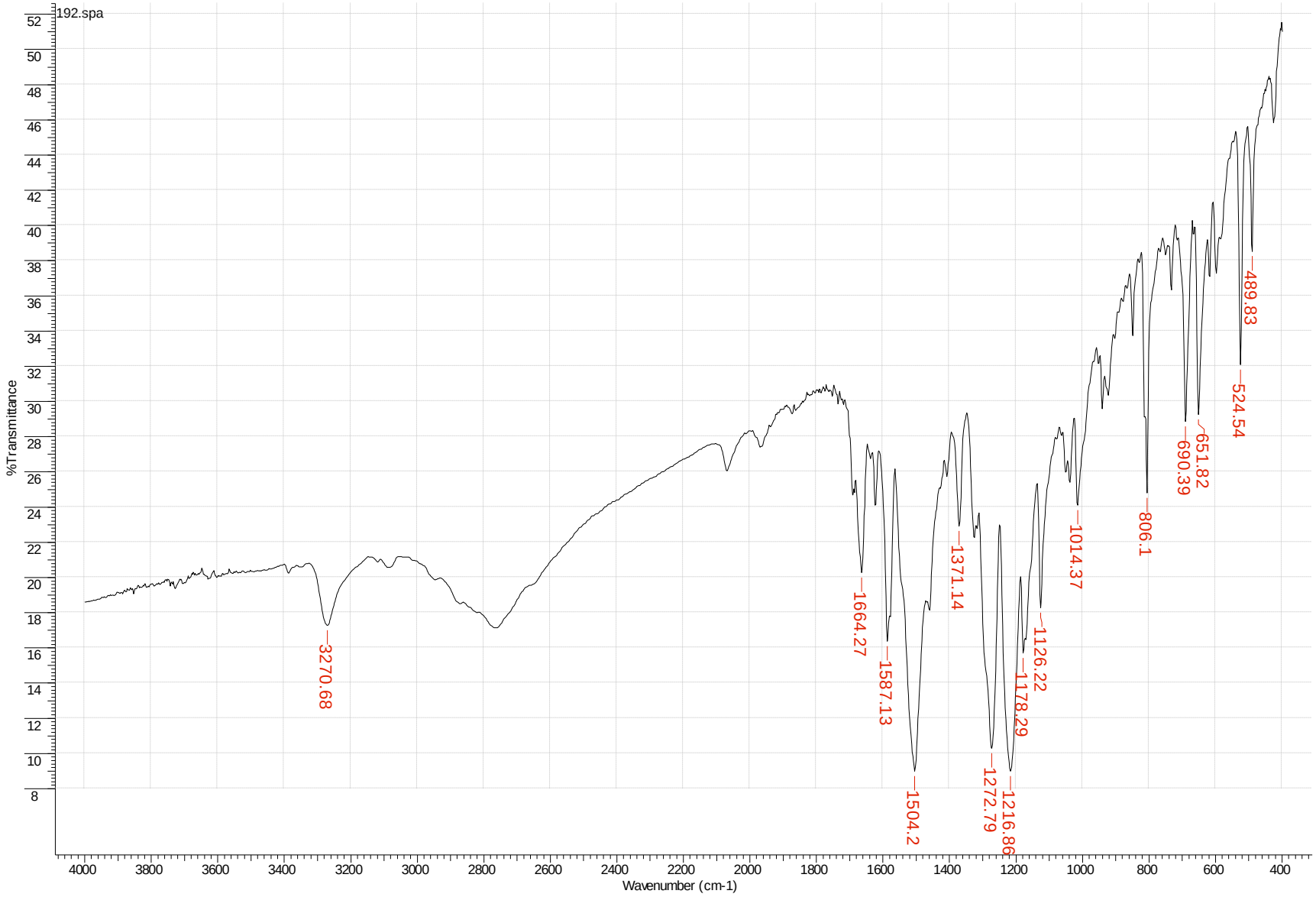
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.0 Bar |
| Focus | Active | | | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set Capillary | 4500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 1800 m/z | Set End Plate Offset | -500 V | Set Divert Valve | Waste |



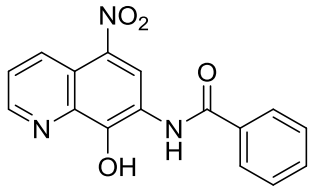



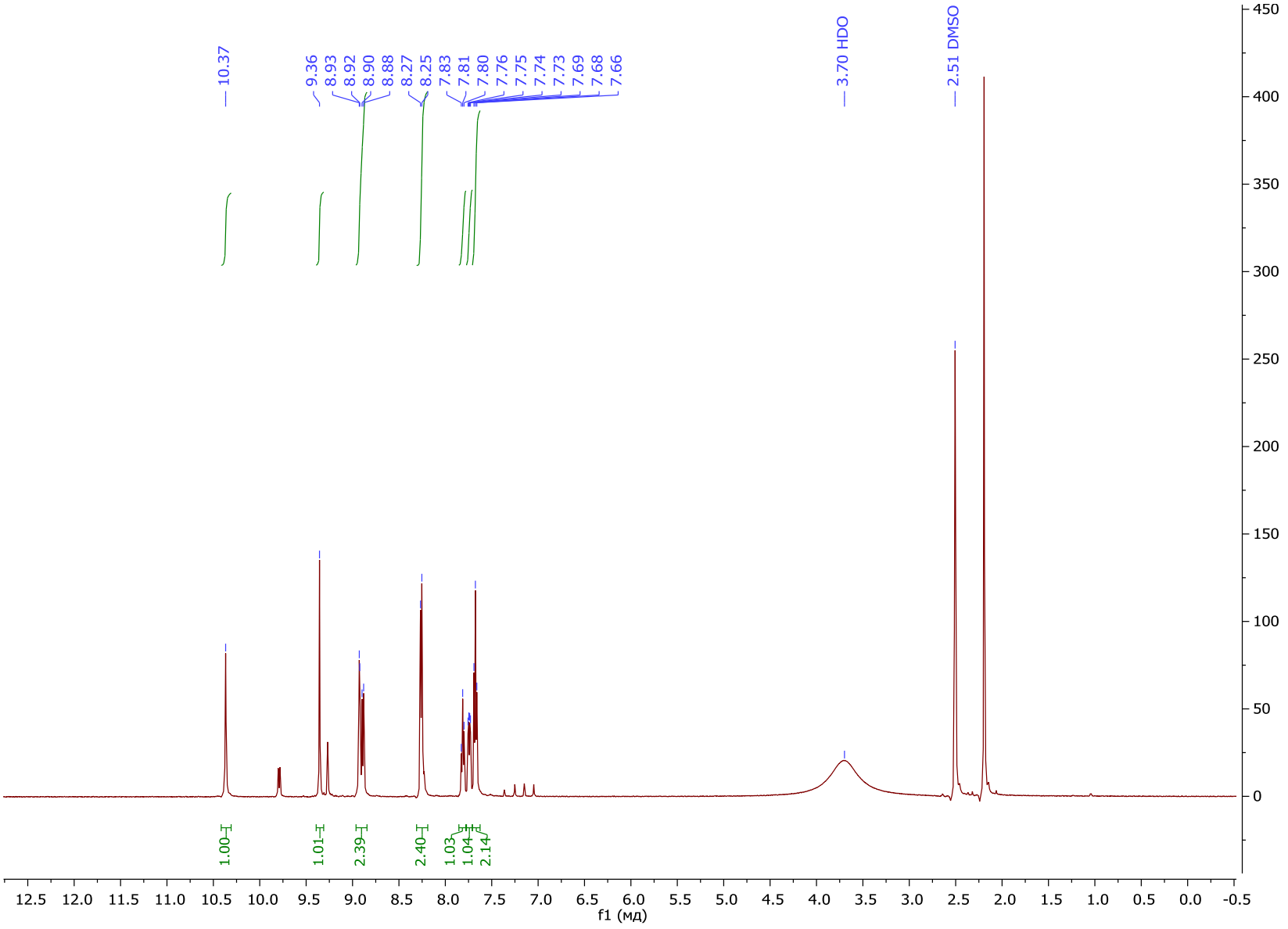
**_N_-(8-hydroxy-5-nitroquinolin-7-yl)acetamide (2a) FT-IR**



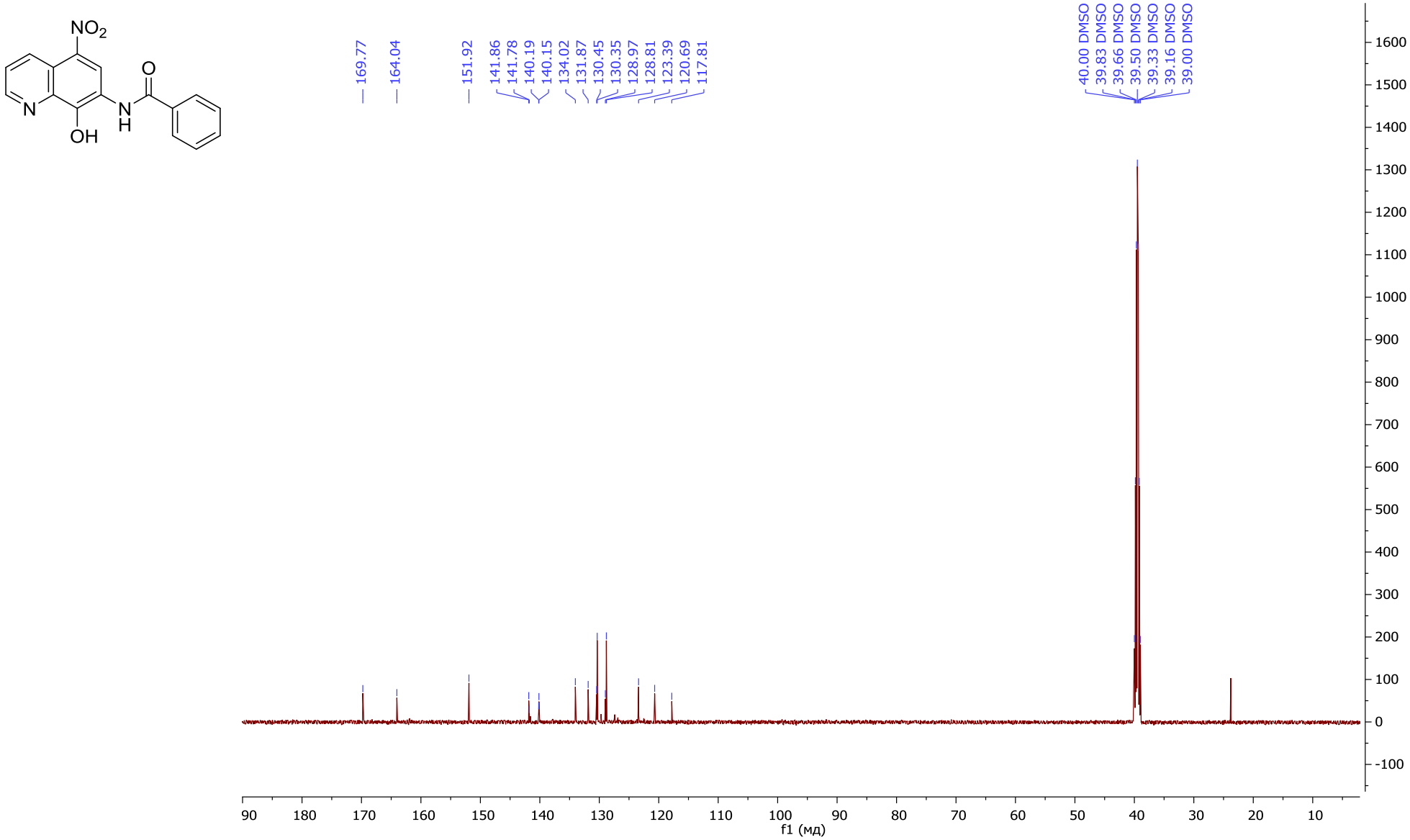



**_N_-(8-hydroxy-5-nitroquinolin-7-yl)benzamide (2b) $^{1}$H NMR**

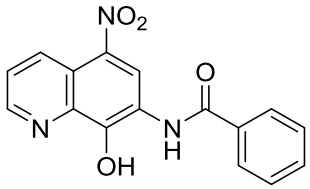


**_N_-(8-hydroxy-5-nitroquinolin-7-yl)benzamide (2b) $^{13}$C NMR**



## *N*-(8-hydroxy-5-nitroquinolin-7-yl)benzamide (2b) HRMS



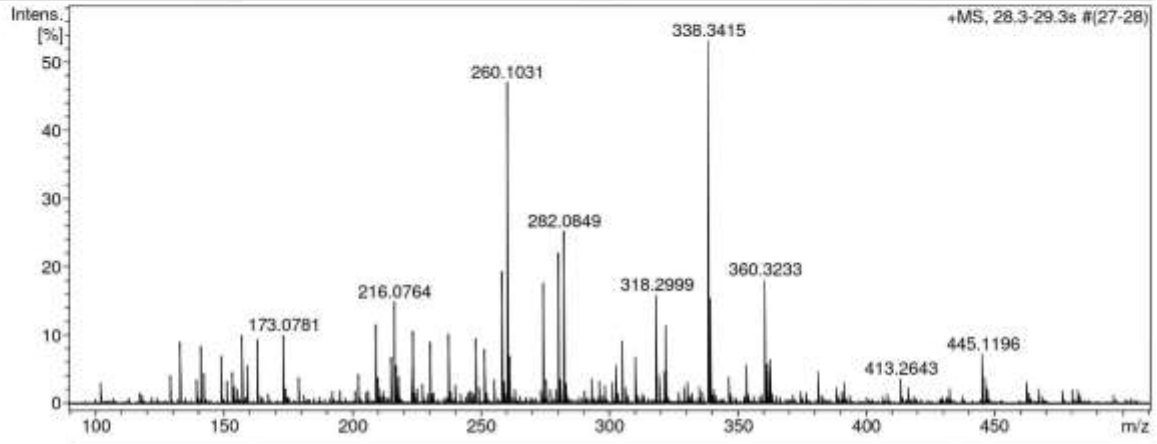

# Display Report

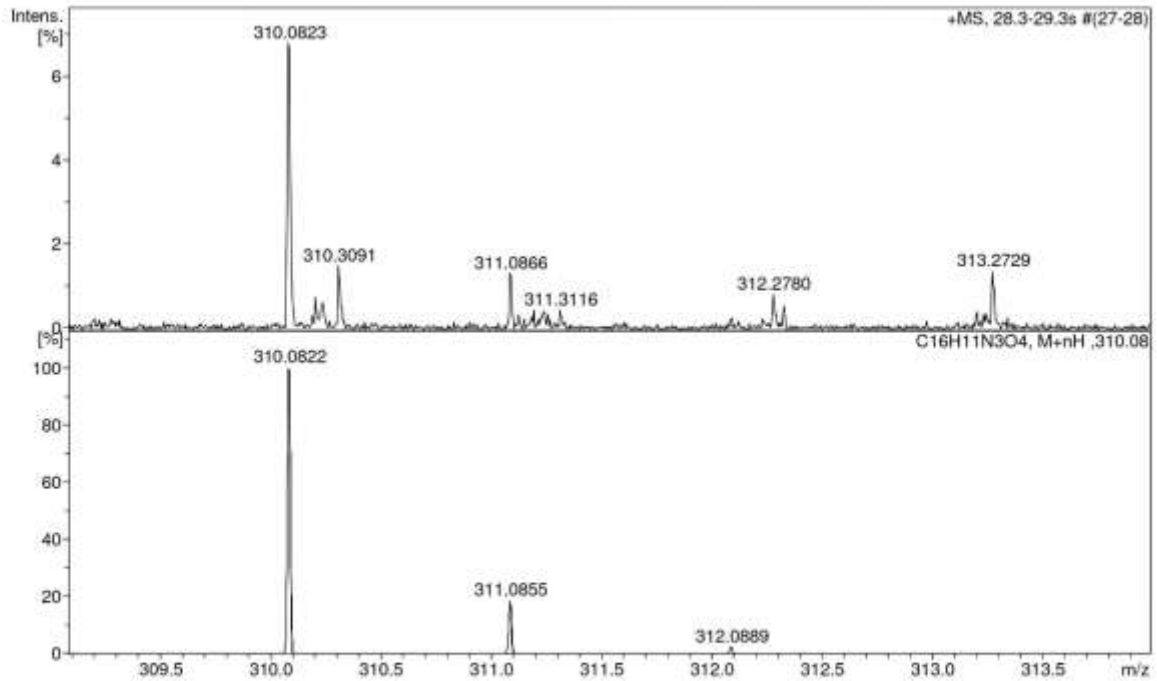
**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\Ilyushenkova\XNV-277.d | Acquisition Date | 26.09.2023 19:50:25 |
| Method | tune_100-1200.m | Operator | BDAL@DE |
| Sample Name | | Instrument / Ser# | maXis 43 |
| Comment | | | |

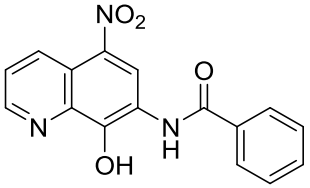
**Acquisition Parameter**

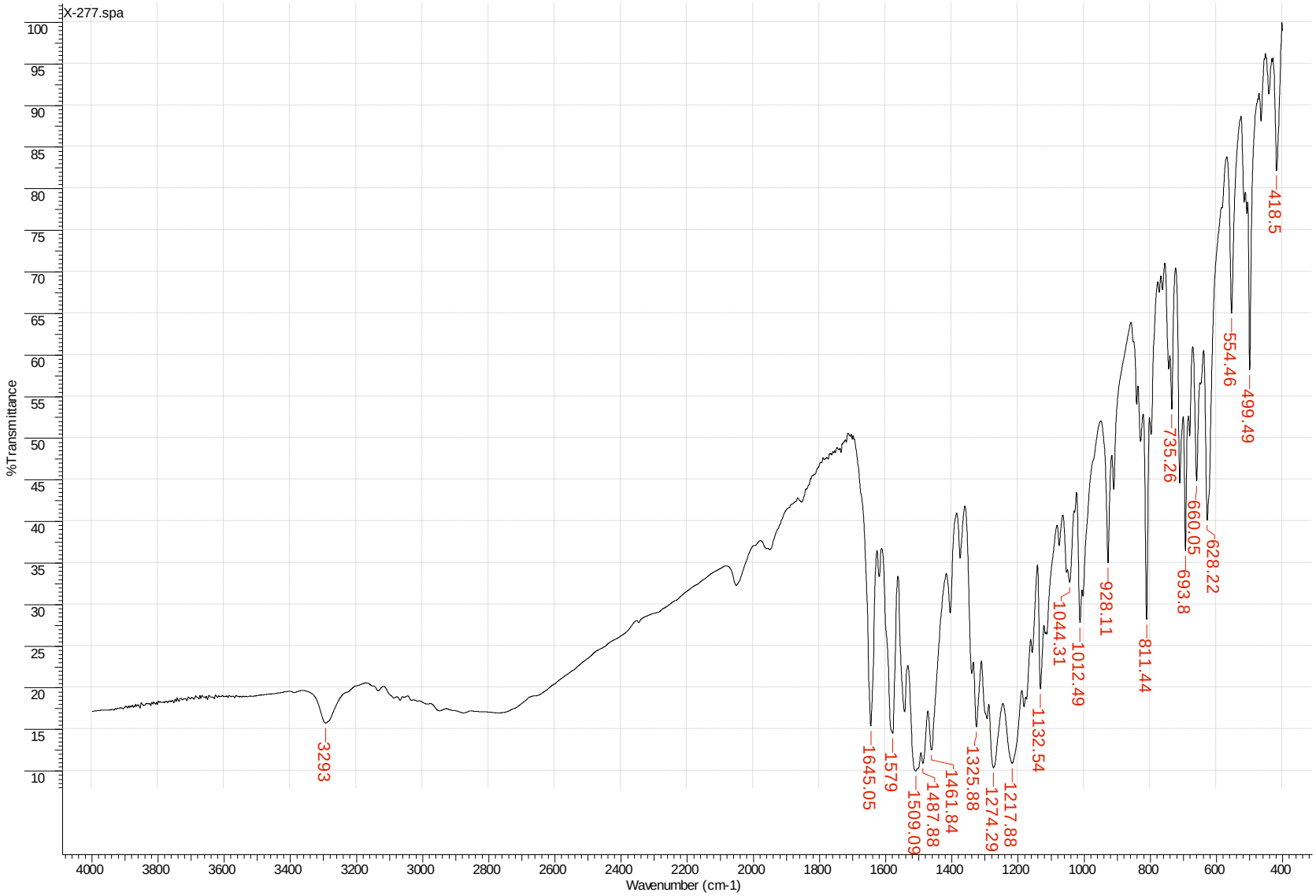
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.0 Bar |
| Focus | Active | | | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set Capillary | 4500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 1800 m/z | Set End Plate Offset | -500 V | Set Divert Valve | Waste |



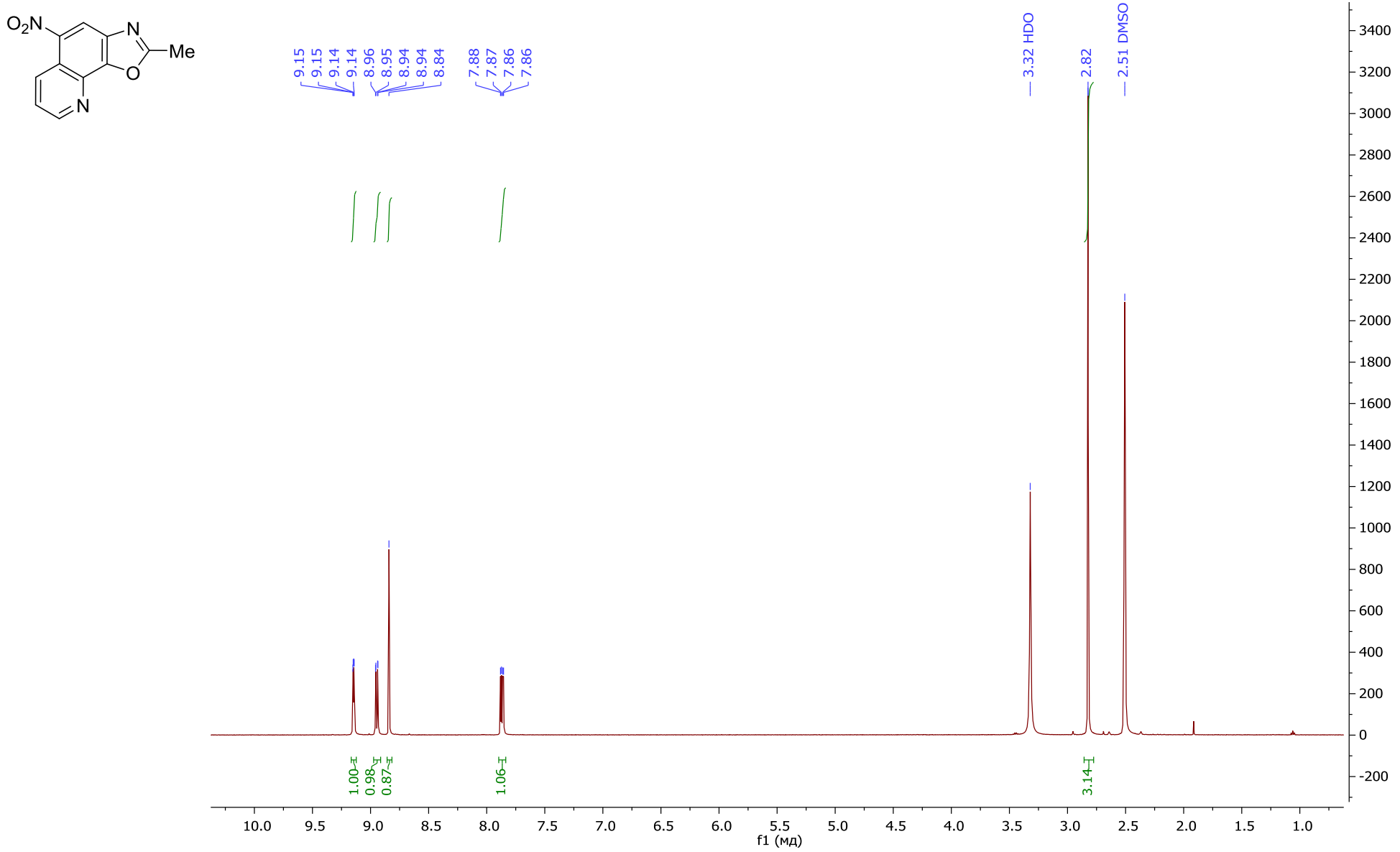



**_N_-(8-hydroxy-5-nitroquinolin-7-yl)benzamide (2b) FT-IR**

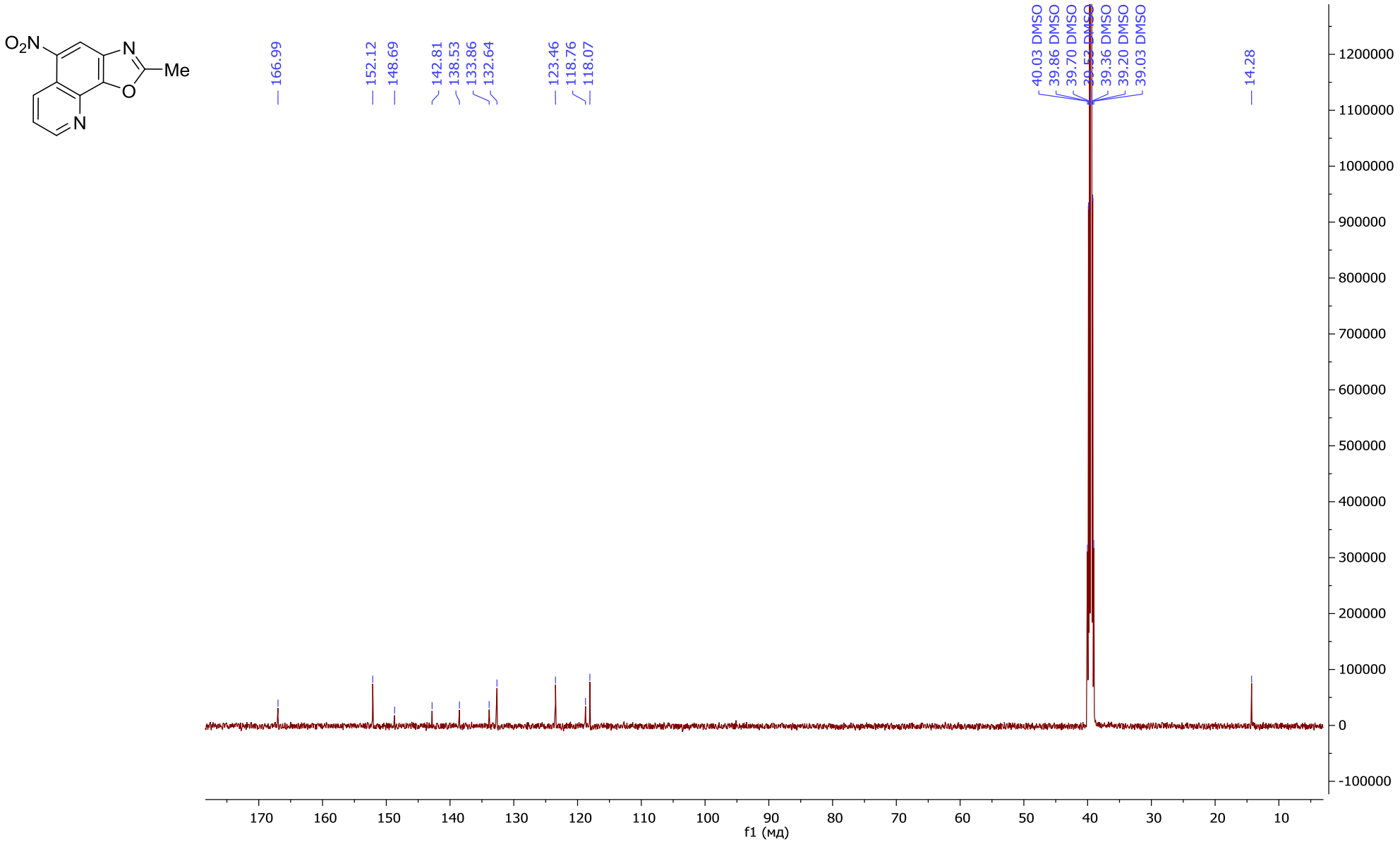


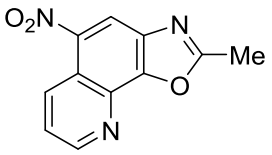
**2-Methyl-5-nitrooxazolo[4,5-*h*]quinoline (3a) $^{1}$H NMR**



**2-Methyl-5-nitrooxazolo[4,5-*h*]quinoline (3a) $^{13}$C NMR**



**2-Methyl-5-nitrooxazolo[4,5-*h*]quinoline (3a) HRMS**



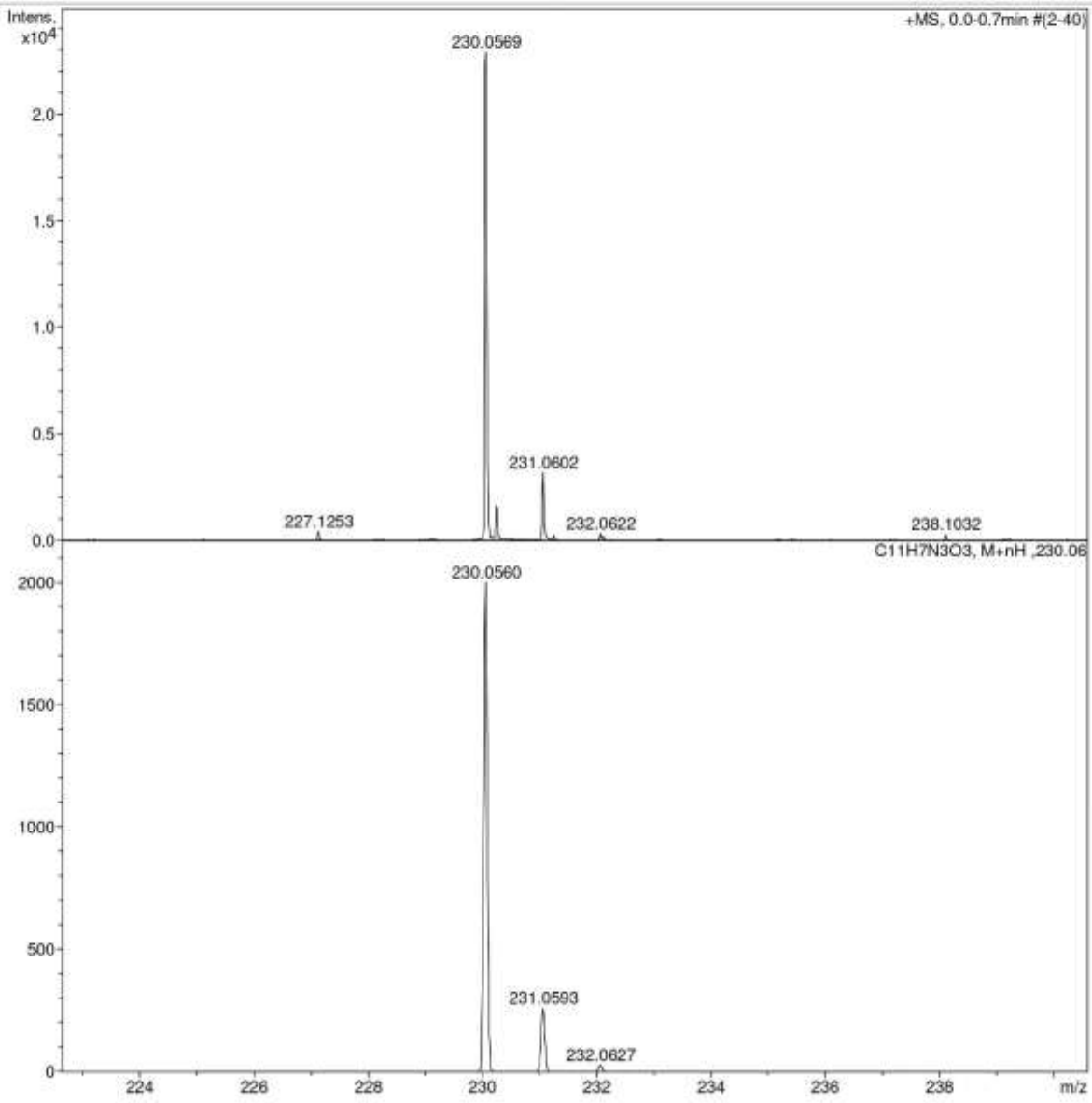

# Display Report

**Analysis Info**

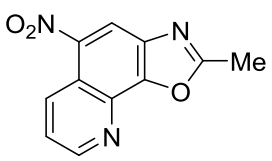
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\Kolotyrkina\2015\Dutov\0609005.d | Acquisition Date | 09.06.2015 13:12:43 |
| Method | tune_low.m | Operator | BDAL@DE |
| Sample Name | /LPIK ULI-193 | Instrument / Ser# | micrOTOF 10248 |
| Comment | C11H7N3O3 mw 229 calibrant added | | |

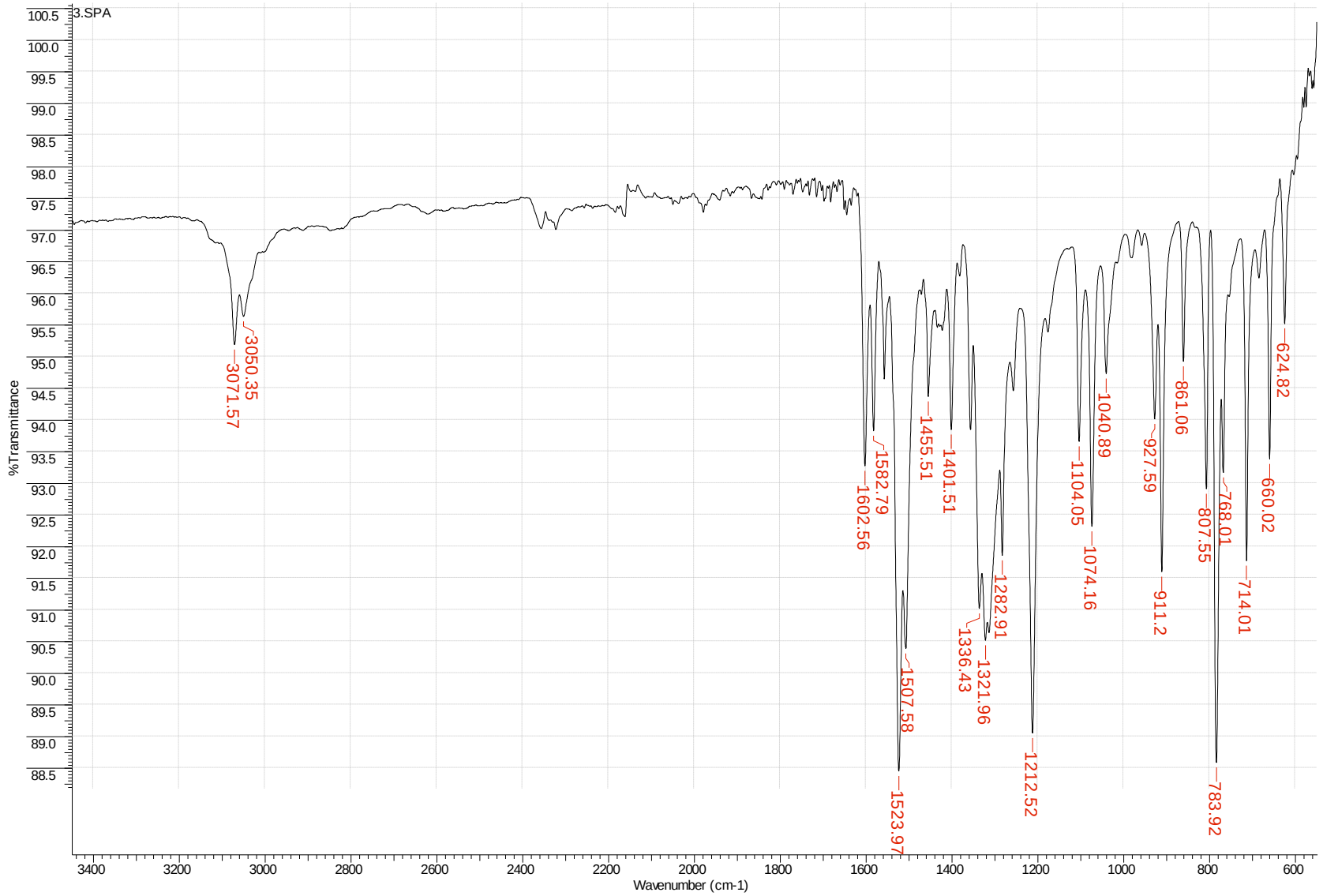
**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 0.4 Bar |
| Focus | Not active | | | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set Capillary | 4500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 3000 m/z | Set End Plate Offset | -500 V | Set Divert Valve | Waste |

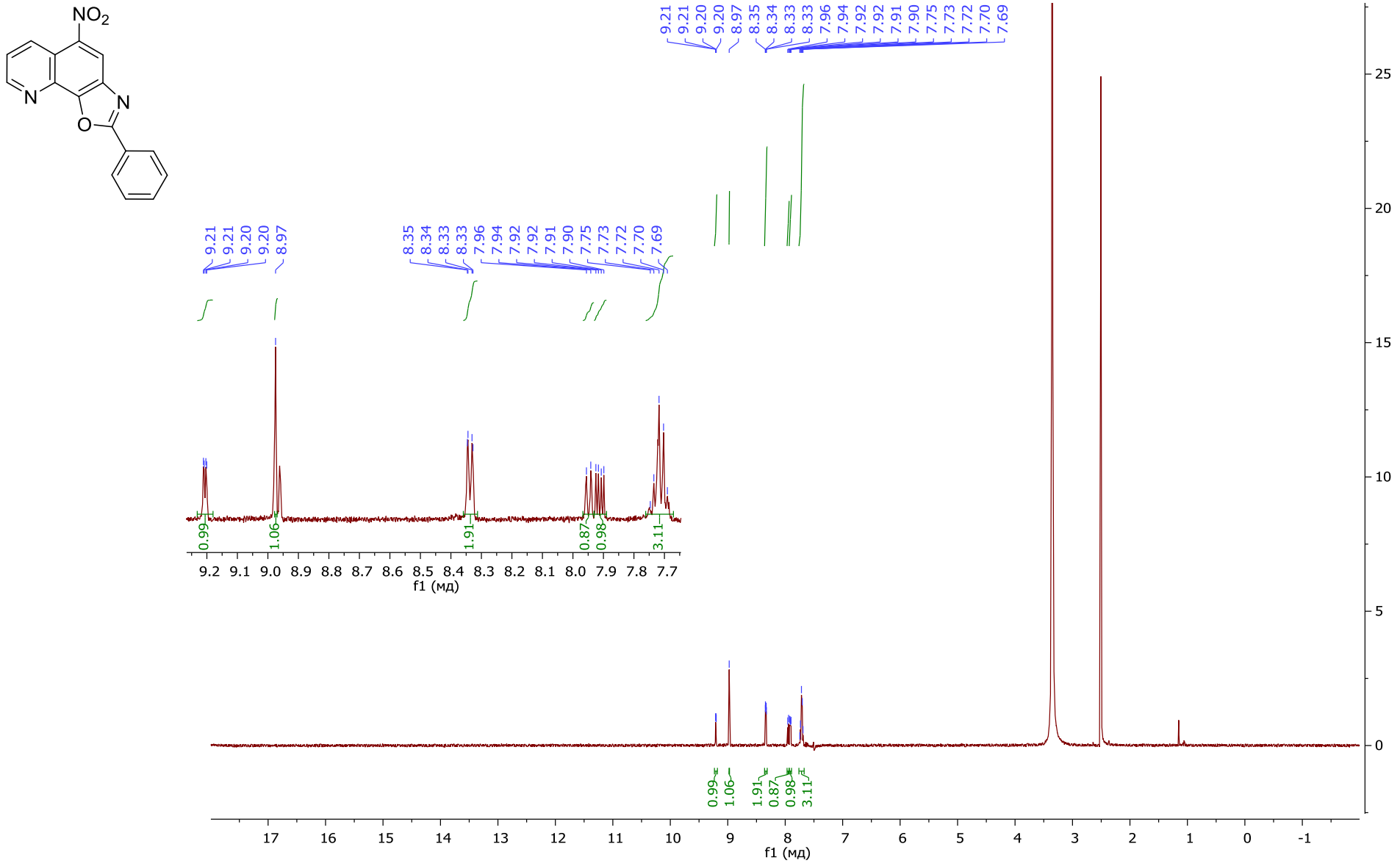


## 2-Methyl-5-nitrooxazolo[4,5-*h*]quinoline (3a) FT-IR

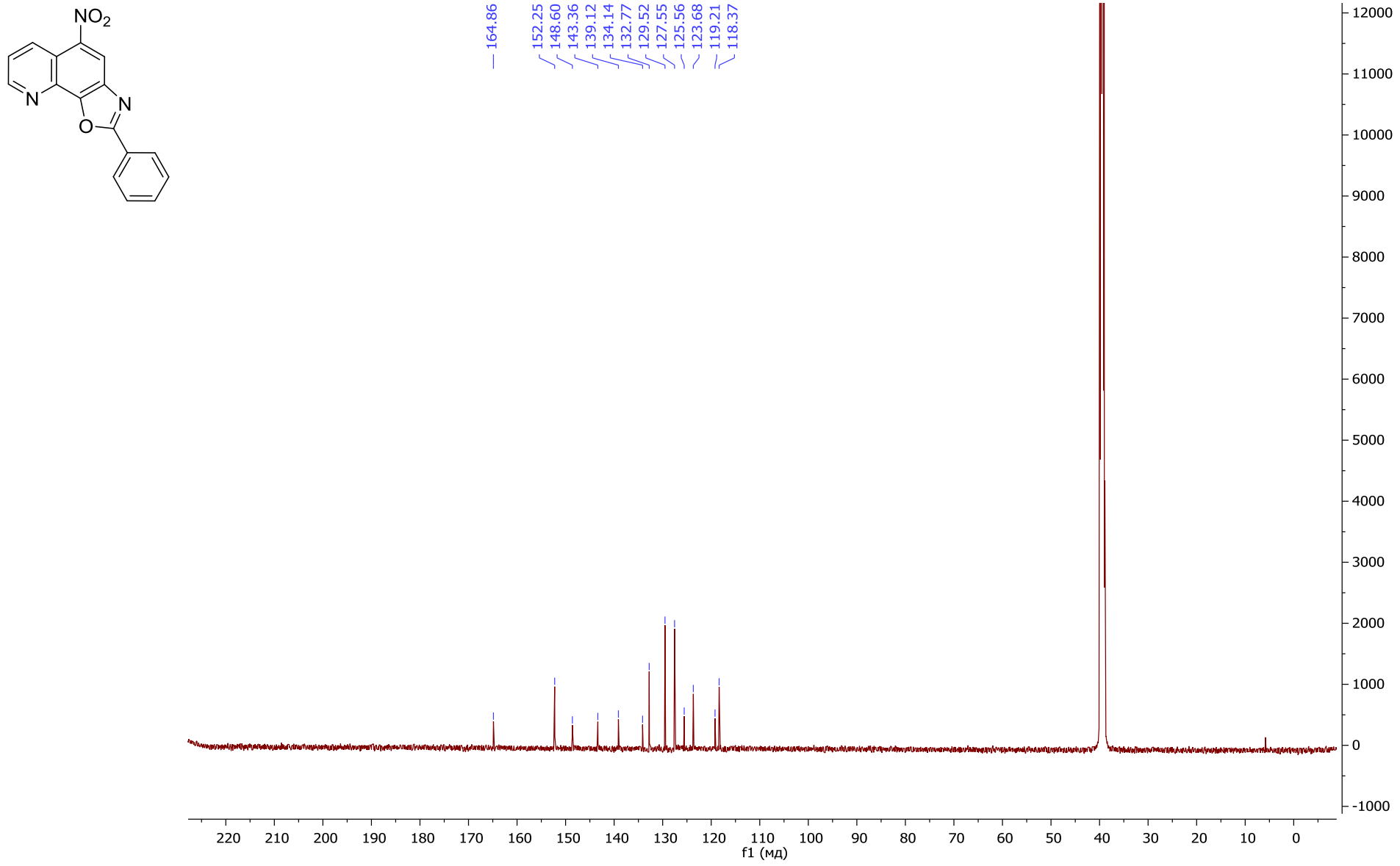


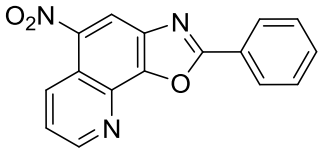
**5-Nitro-2-phenyloxazolo[4,5-*h*]quinoline (3b) $^{1}$H NMR**



**5-Nitro-2-phenyloxazolo[4,5-*h*]quinoline (3b) $^{13}$C NMR**



**5-Nitro-2-phenyloxazolo[4,5-*h*]quinoline (3b) HRMS**



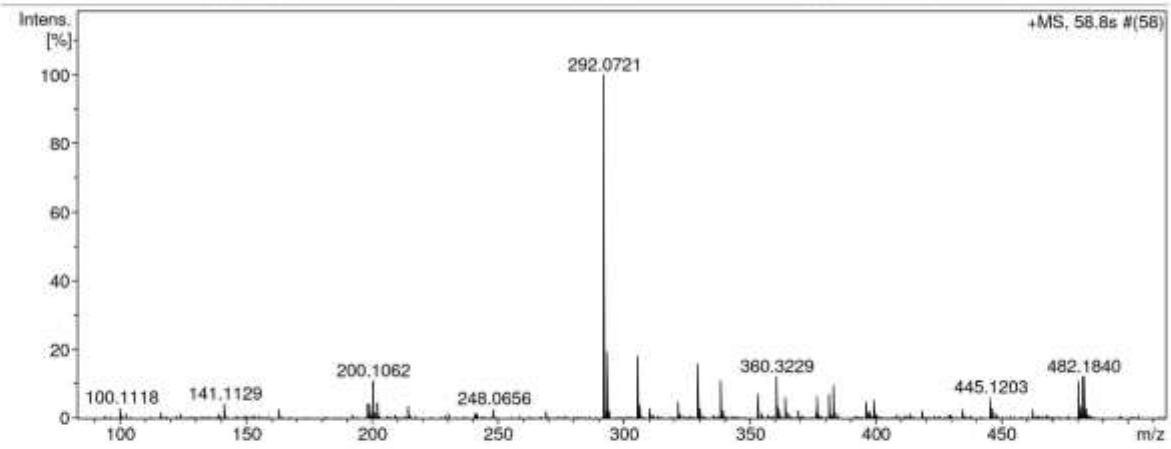

# Display Report

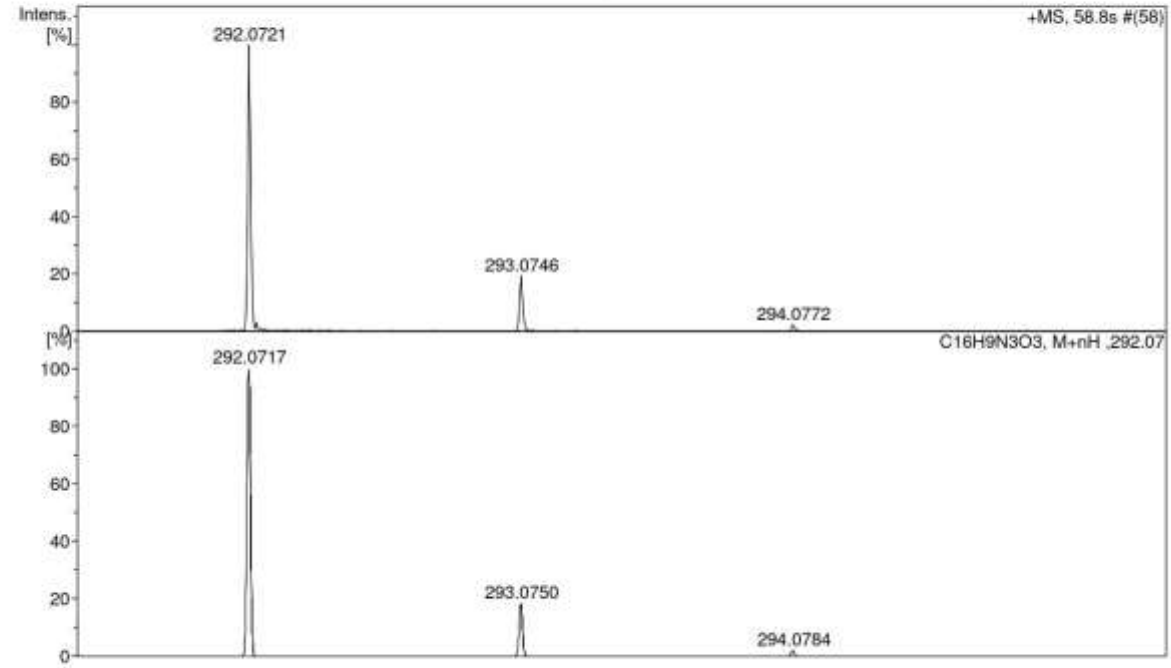
**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\Ilyushenkova\ULI-267.d | Acquisition Date | 26.09.2023 19:24:33 |
| Method | tune_100-1200.m | Operator | BDAL@DE |
| Sample Name | | Instrument / Ser# | maXis 43 |
| Comment | | | |

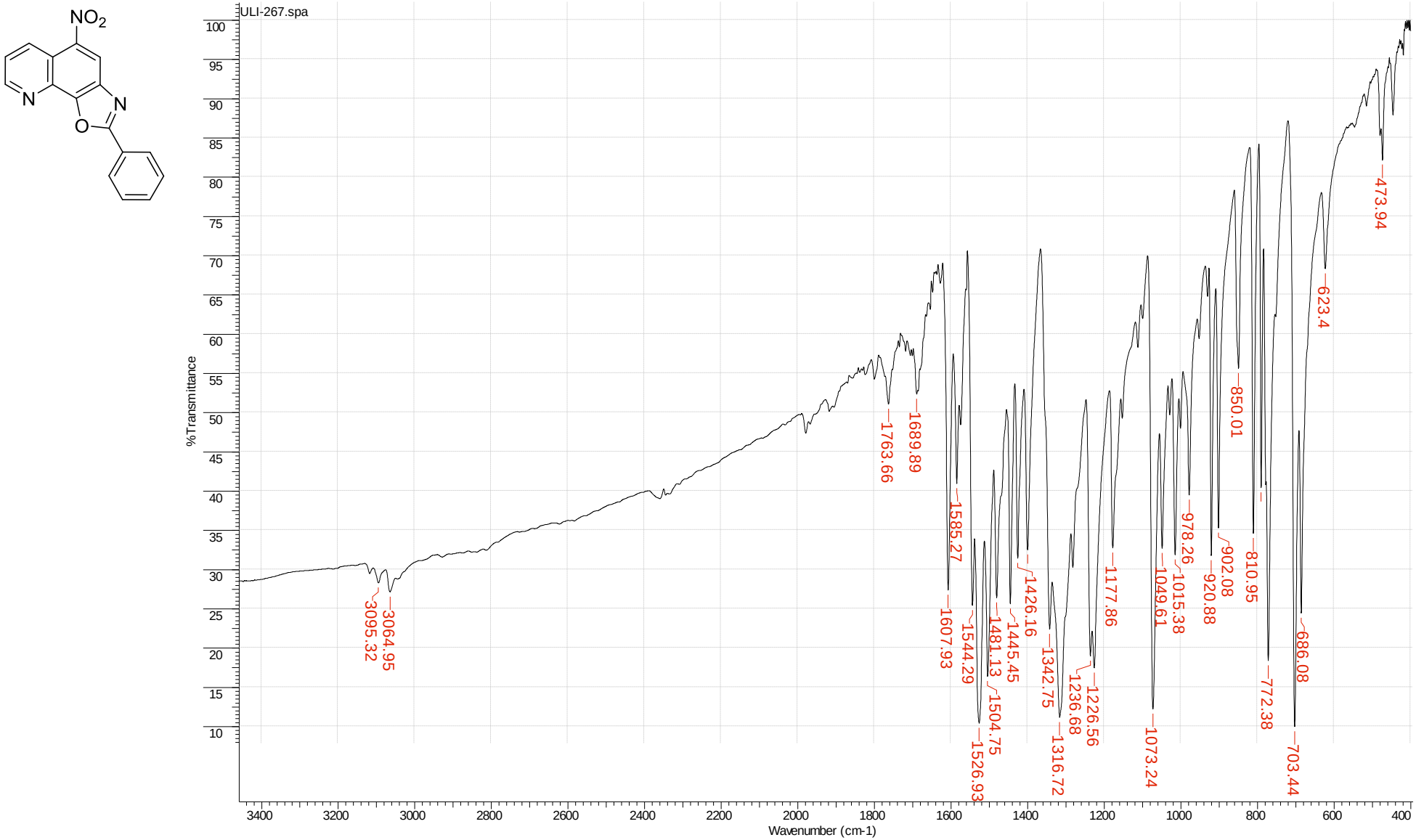
**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.0 Bar |
| Focus | Active | | | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set Capillary | 4500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 1800 m/z | Set End Plate Offset | -500 V | Set Divert Valve | Waste |

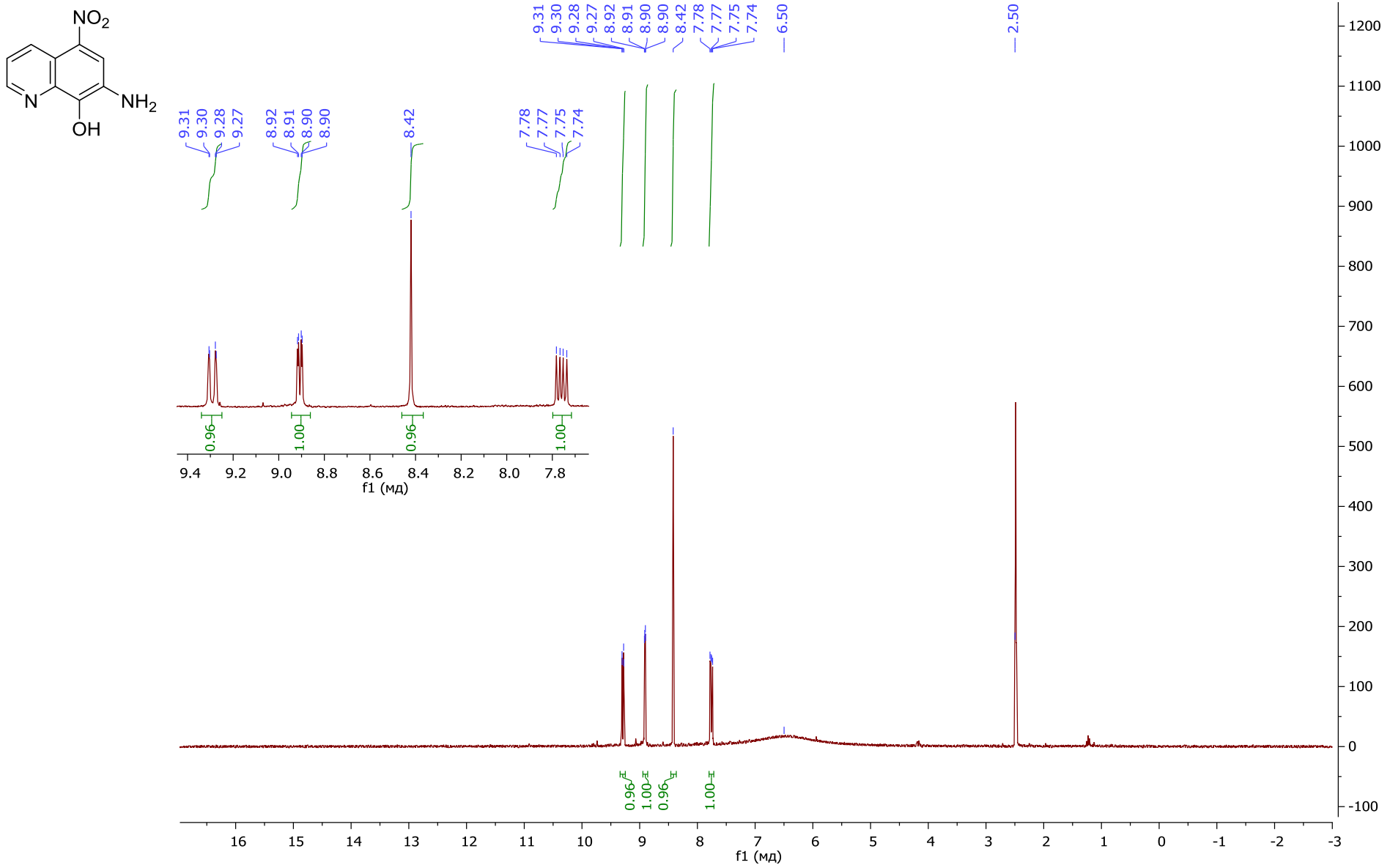


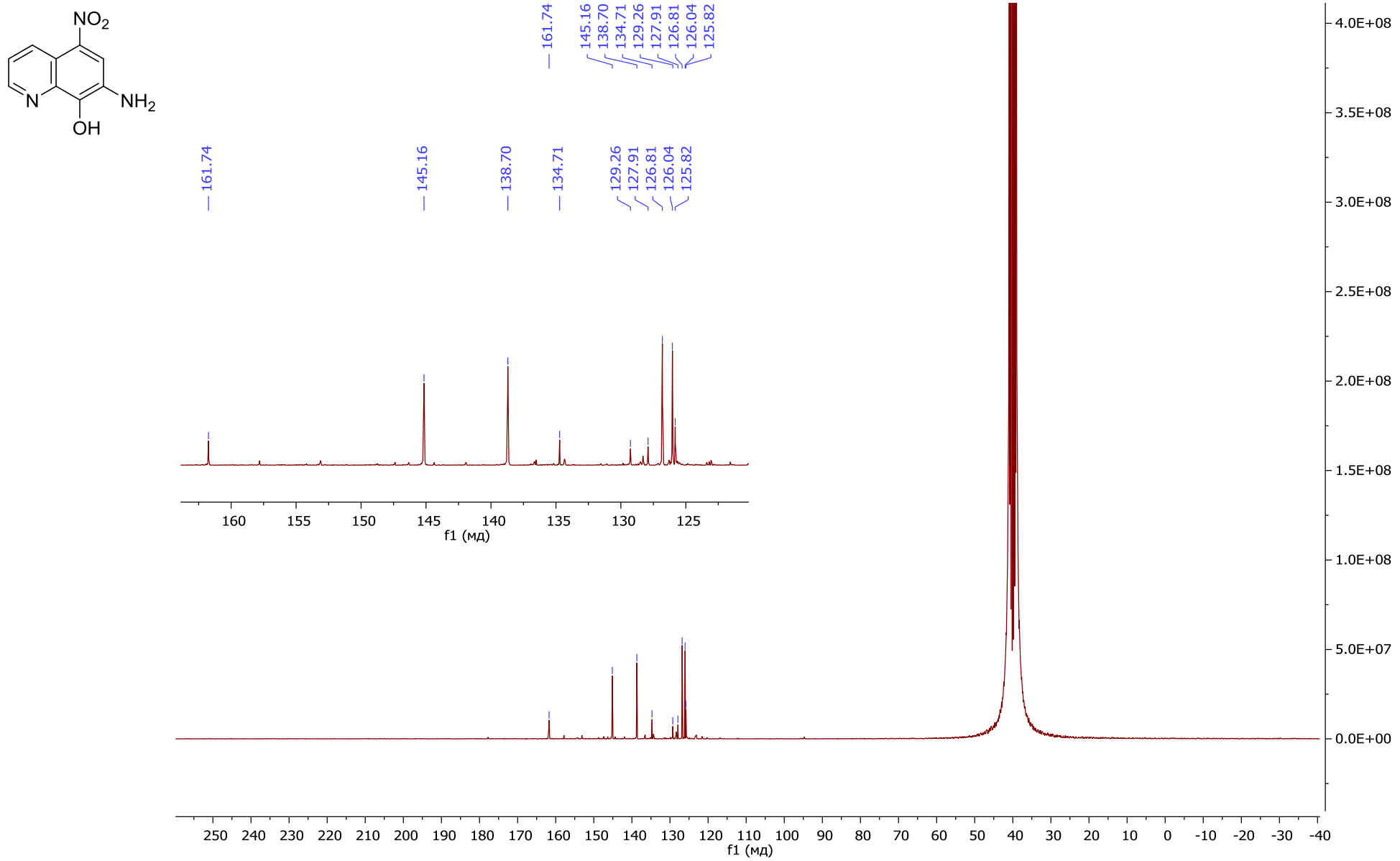
**5-Nitro-2-phenyloxazolo[4,5-*h*]quinoline (3b) FT-IR**



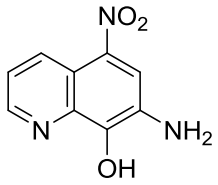
**7-Amino-5-nitroquinolin-8-ol (4) $^{1}$H NMR**



**7-Amino-5-nitroquinolin-8-ol (4) $^{13}$C NMR**



# 7-Amino-5-nitroquinolin-8-ol (4) HRMS



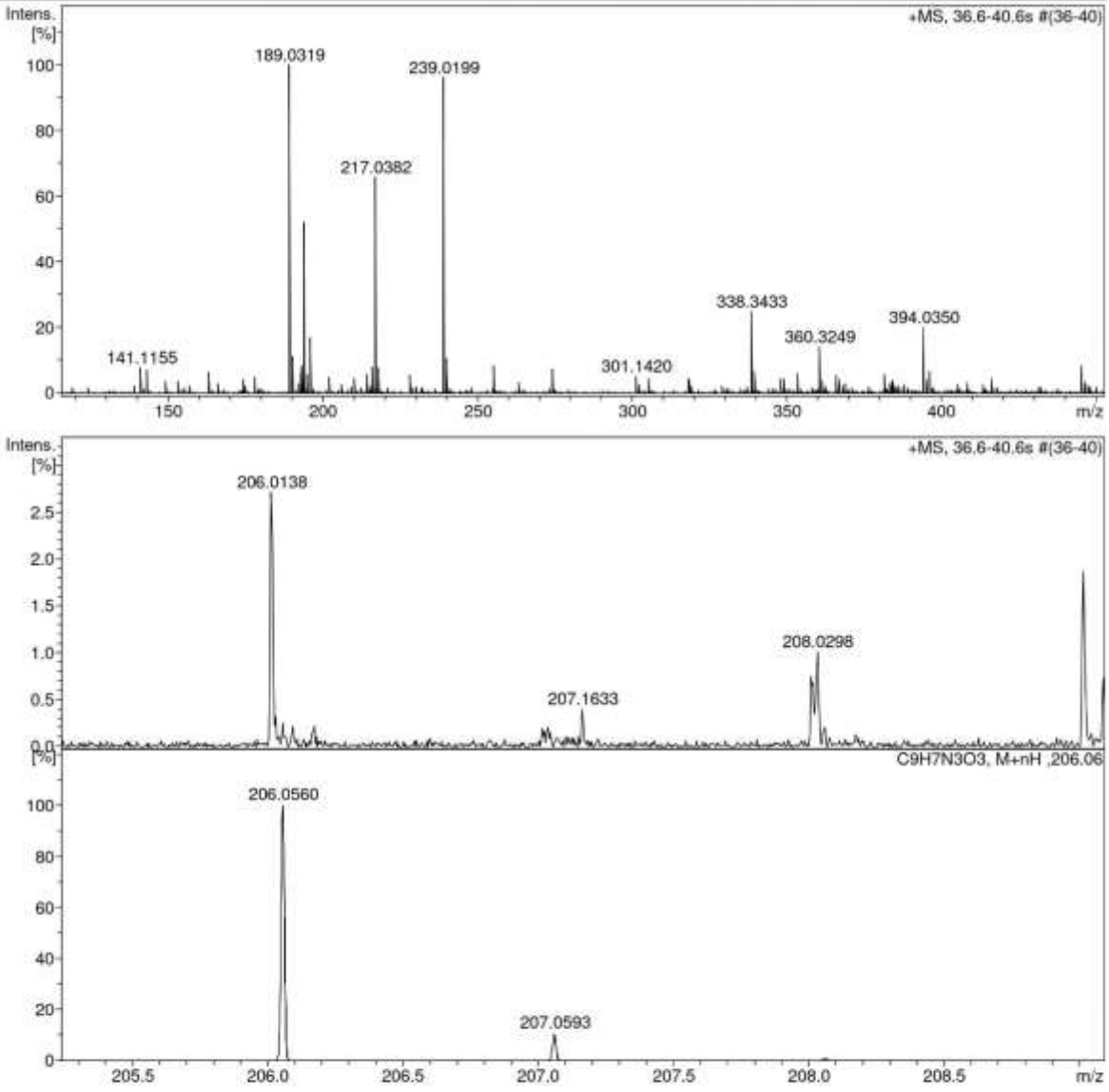

## Display Report

**Analysis Info**

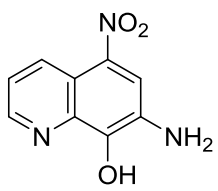
| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\Ilyushenkova\2023-09_Tula\ULI-457.d | Acquisition Date | 26.09.2023 19:39:49 |
| Method | tune_100-1200.m | Operator | BDAL@DE |
| Sample Name | | Instrument / Ser# | maXis 43 |
| Comment | | | |

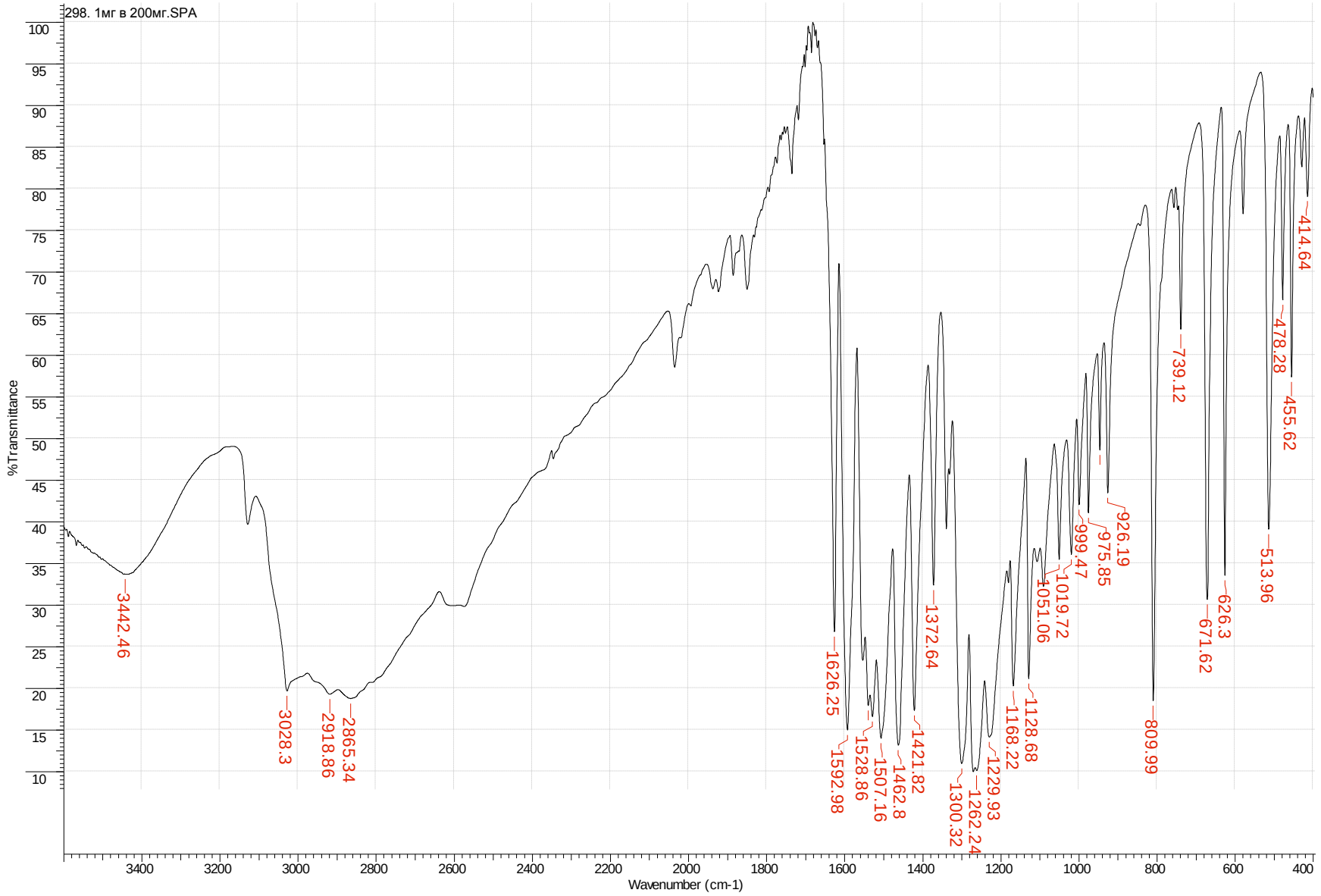
**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.0 Bar |
| Focus | Active | | | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set Capillary | 4500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 1800 m/z | Set End Plate Offset | -500 V | Set Divert Valve | Waste |

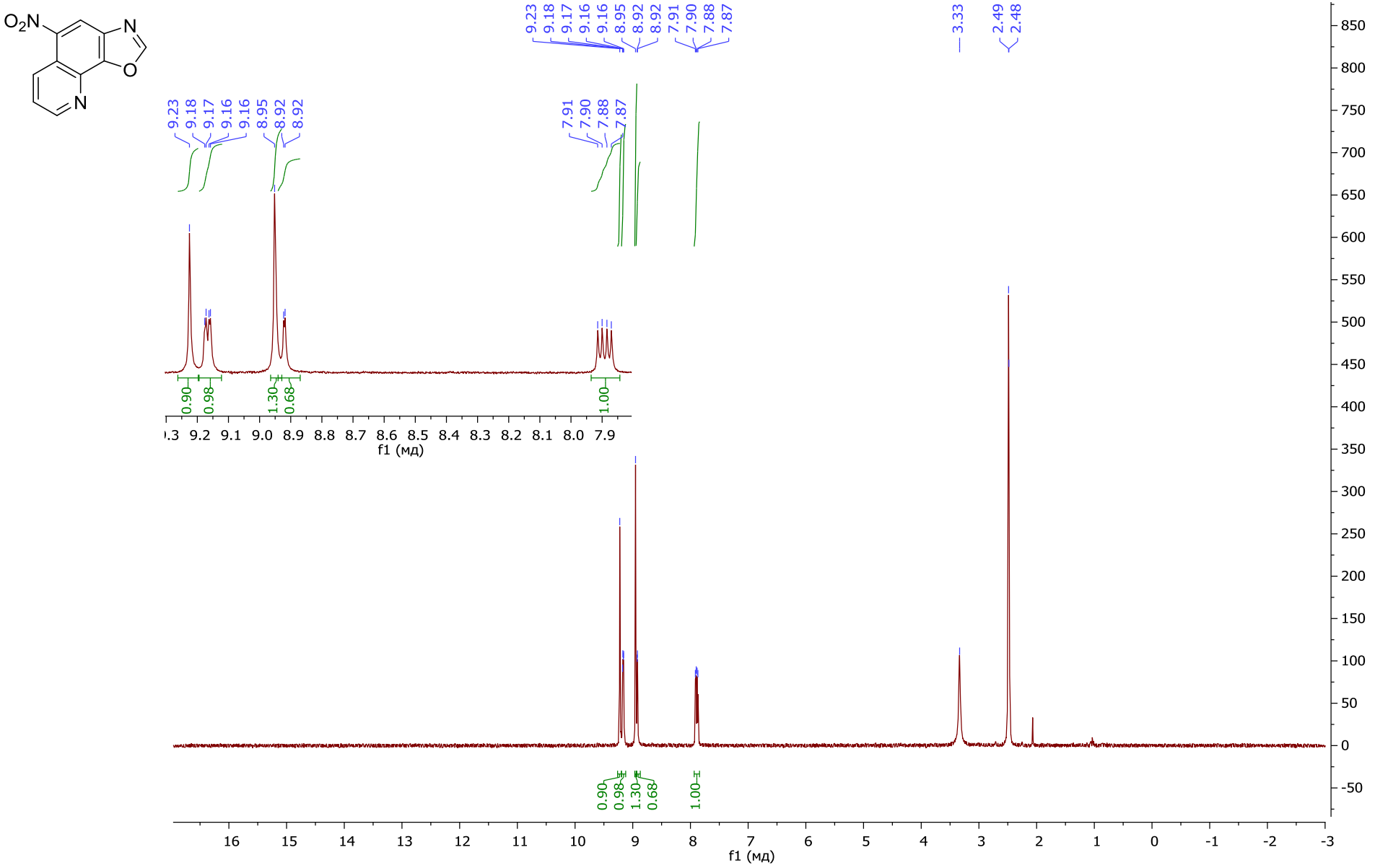


## 7-Amino-5-nitroquinolin-8-ol (4) FT-IR

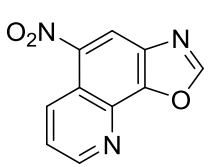


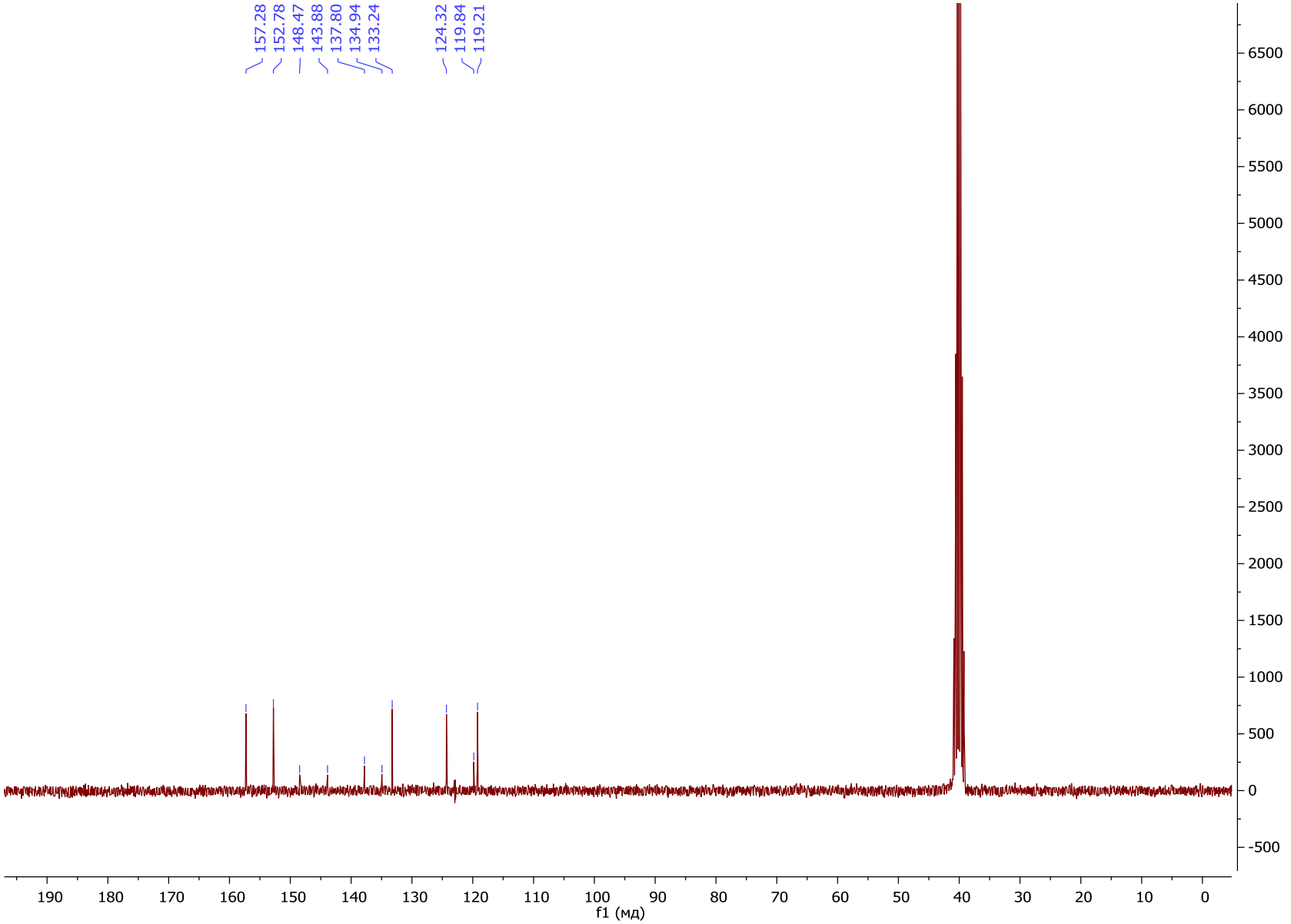



**5-Nitrooxazolo[4,5-*h*]quinoline (5) $^{1}$H NMR**

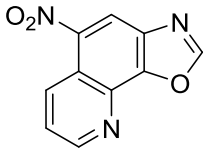


**5-Nitrooxazolo[4,5-*h*]quinoline (5) $^{13}$C NMR**



**5-Nitrooxazolo[4,5-*h*]quinoline (5) HRMS**



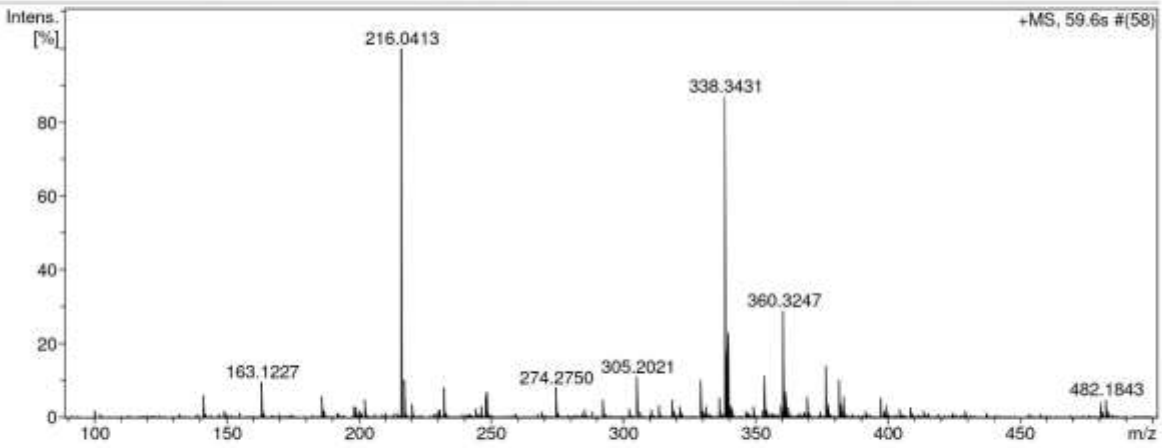
# Display Report

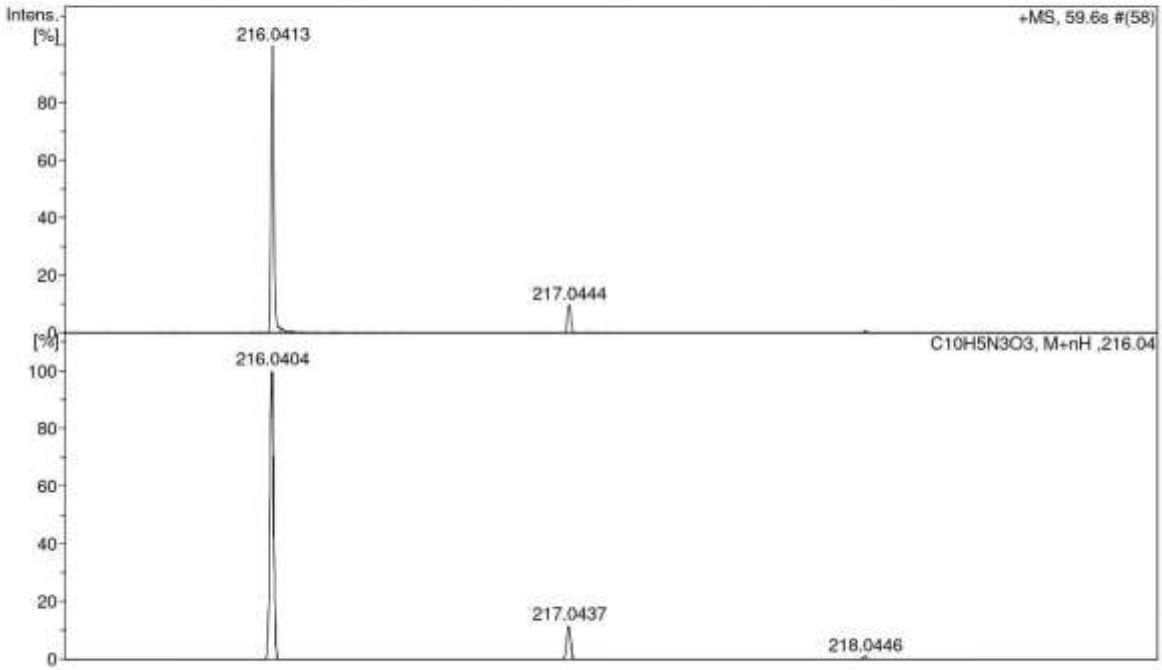
**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\Ilyushenkova\ULI-491.d | Acquisition Date | 26.09.2023 19:37:06 |
| Method | tune_100-1200.m | Operator | BDAL@DE |
| Sample Name | | Instrument / Ser# | maXis 43 |
| Comment | | | |

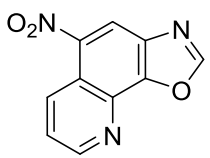
**Acquisition Parameter**

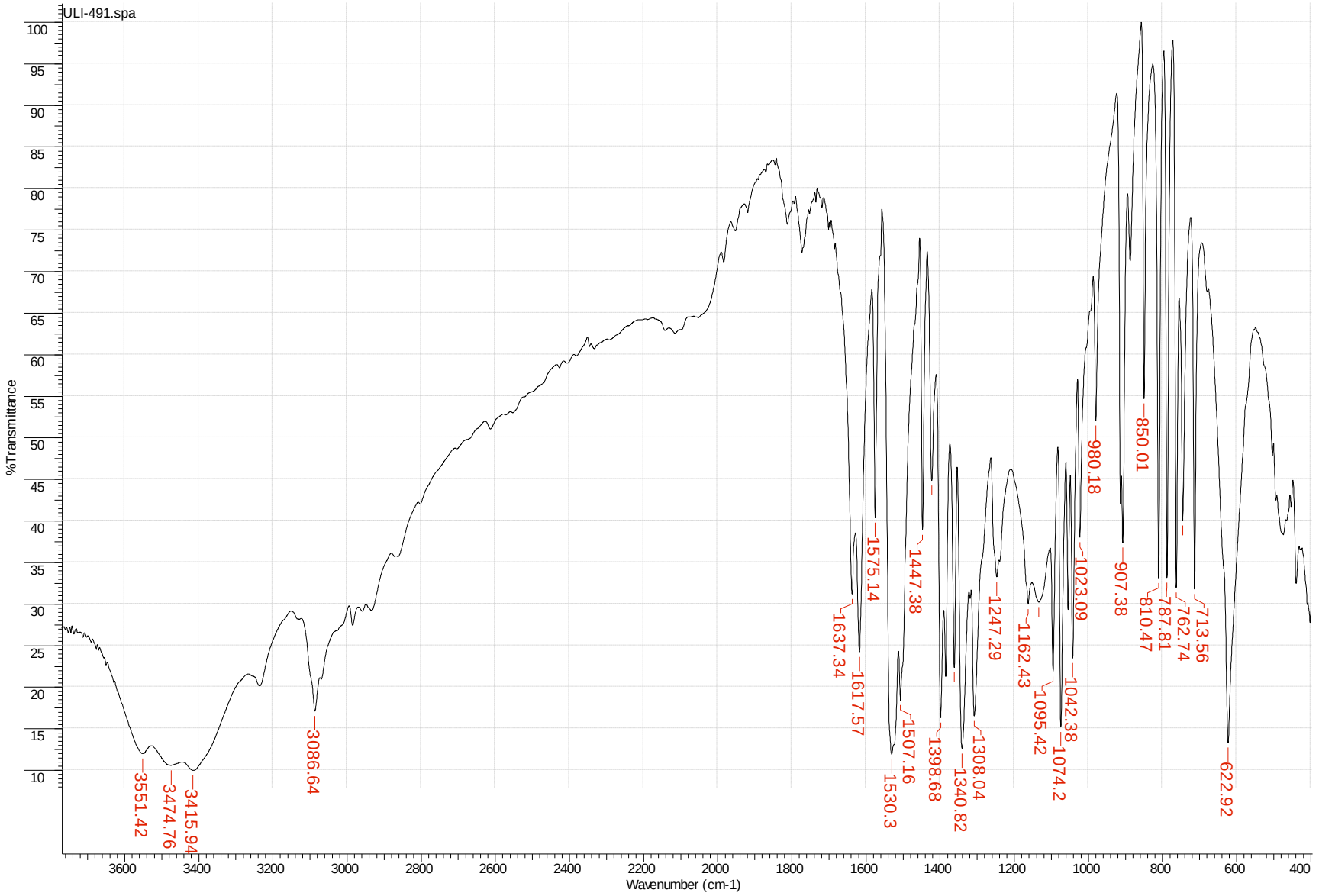
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.0 Bar |
| Focus | Active | | | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set Capillary | 4500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 1800 m/z | Set End Plate Offset | -500 V | Set Divert Valve | Waste |



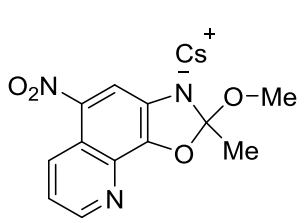



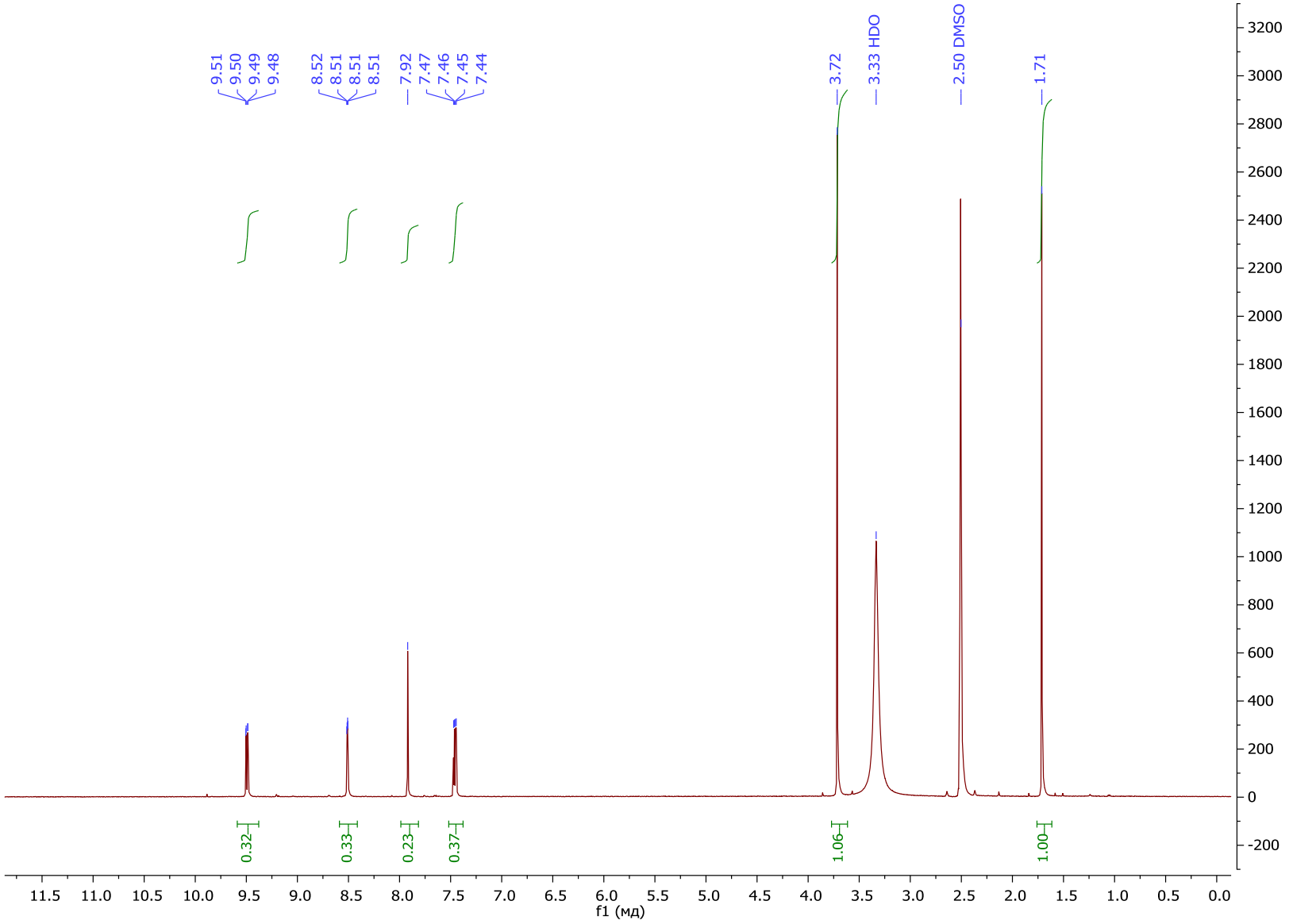
**5-Nitrooxazolo[4,5-*h*]quinoline (5) FT-IR**



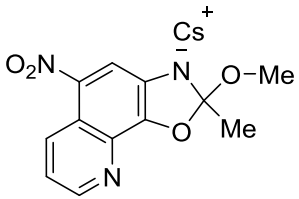



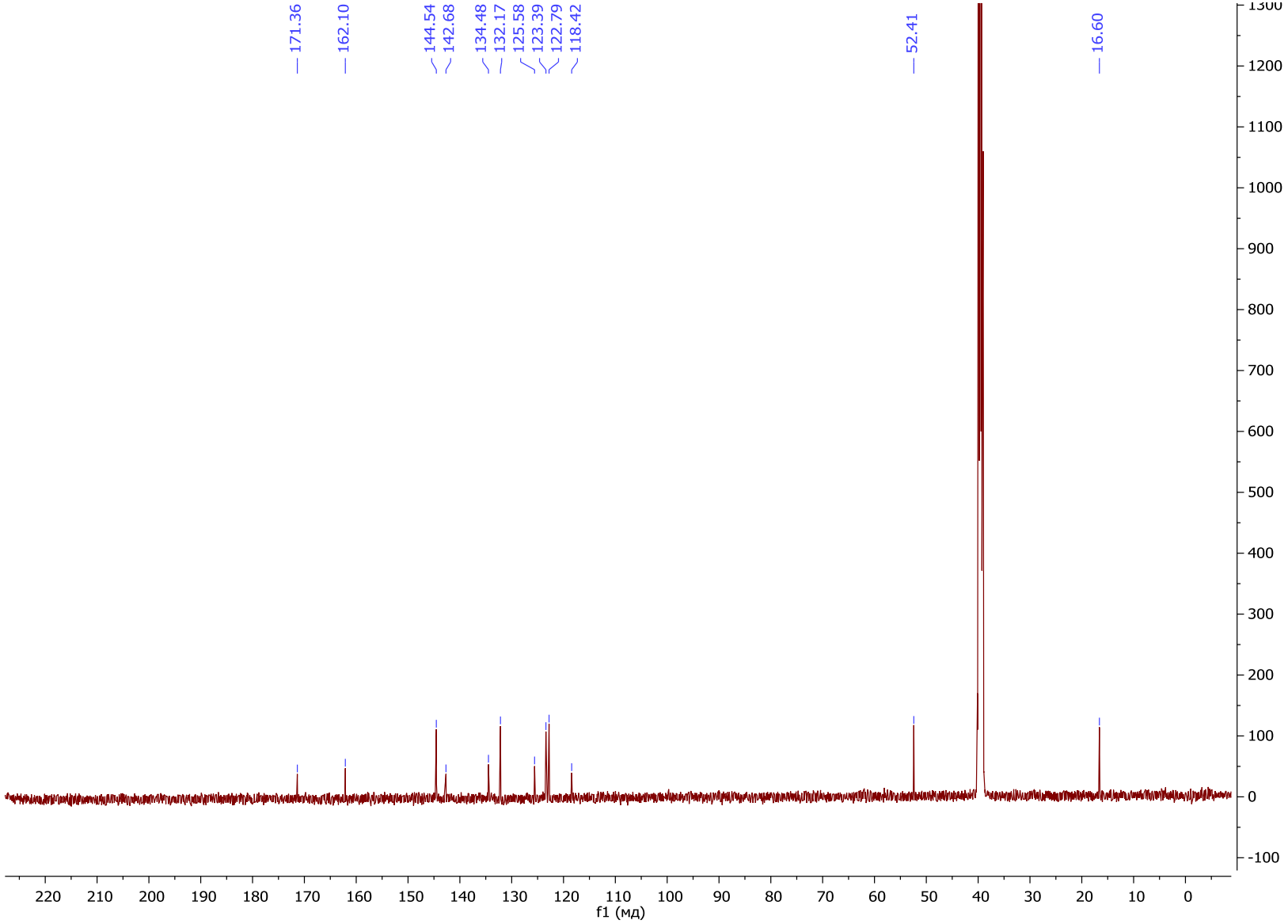
**Caesium 2-methoxy-2-methyl-5-nitrooxazolo[4,5-*h*]quinolin-3-ide (6) $^{1}$H NMR**



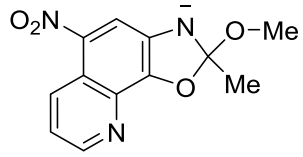
**Caesium 2-methoxy-2-methyl-5-nitrooxazolo[4,5-*h*]quinolin-3-ide (6) $^{13}$C NMR**



**Caesium 2-methoxy-2-methyl-5-nitrooxazolo[4,5-*h*]quinolin-3-ide (6) HRMS**

**For anion $C_{12}H_{10}N_3O_4^-$**



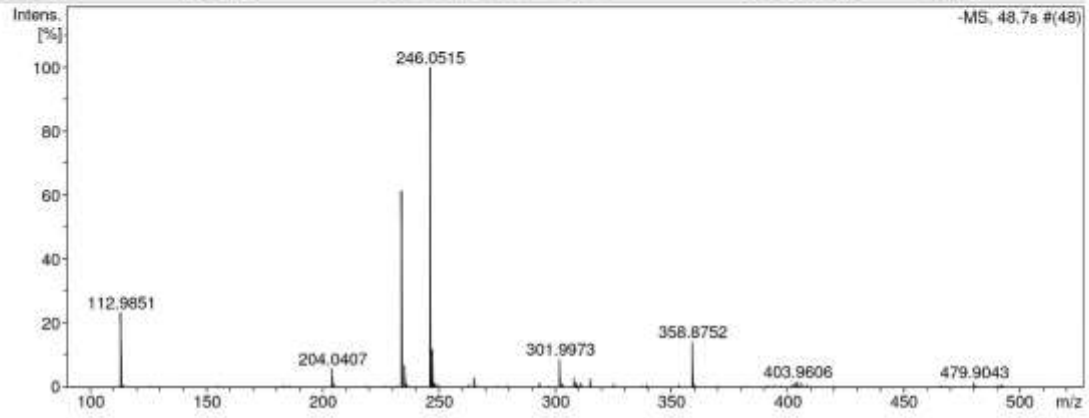

## Display Report

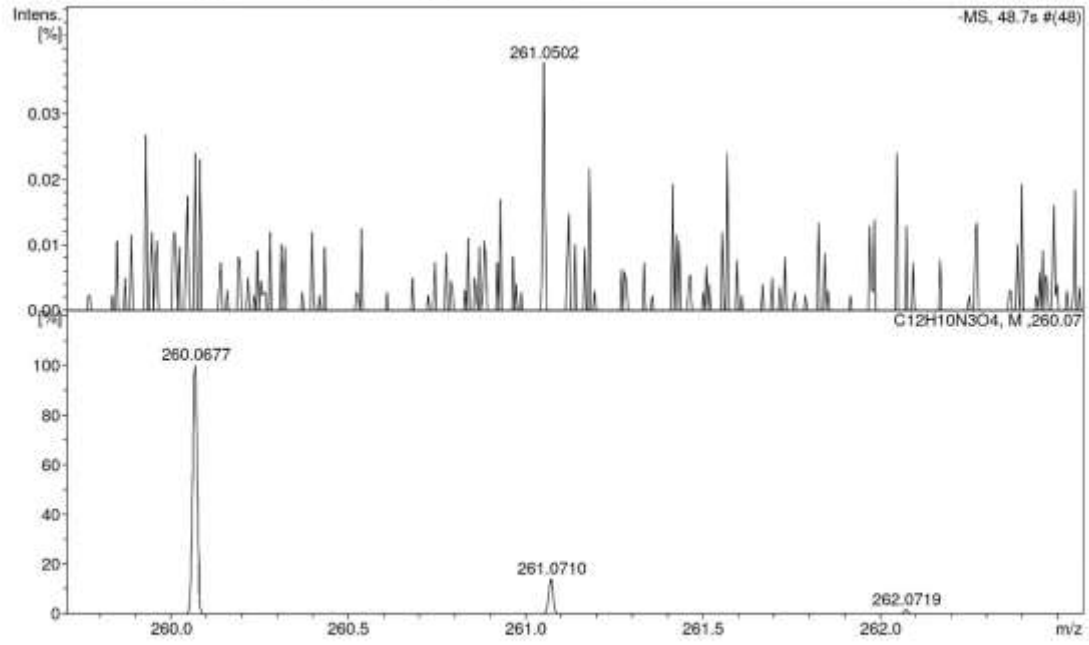
**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\Ilyushenkova\ULI-340_neg.d | Acquisition Date | 27.09.2023 19:37:04 |
| Method | tune_100-1200.m | Operator | BDAL@DE |
| Sample Name | | Instrument / Ser# | maXis 43 |
| Comment | | | |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Negative | Set Nebulizer | 1.0 Bar |
| Focus | Active | | | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set Capillary | 4000 V | Set Dry Gas | 4.0 l/min |
| Scan End | 1800 m/z | Set End Plate Offset | -500 V | Set Divert Valve | Waste |



**For cation $Cs^+$**

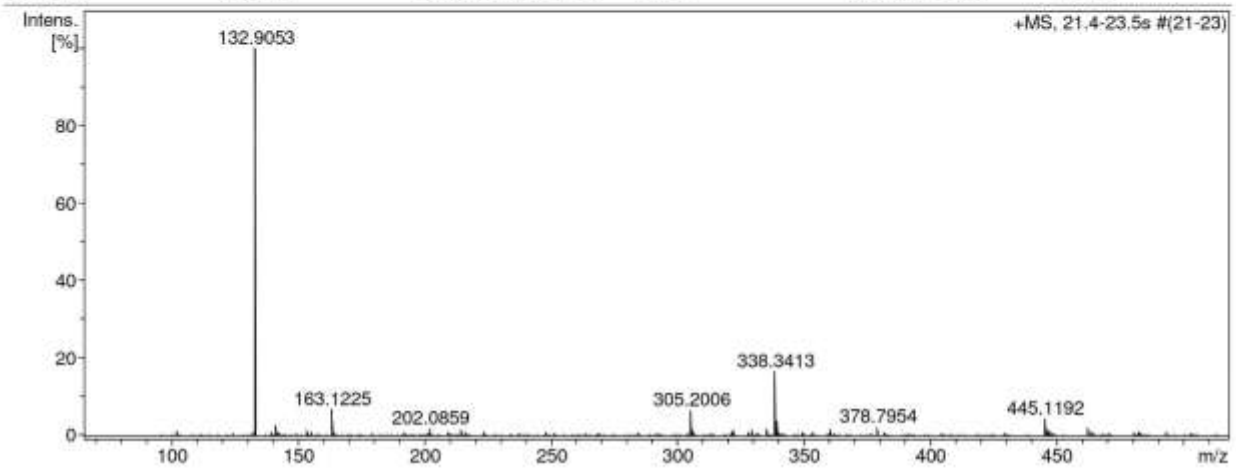
# Display Report

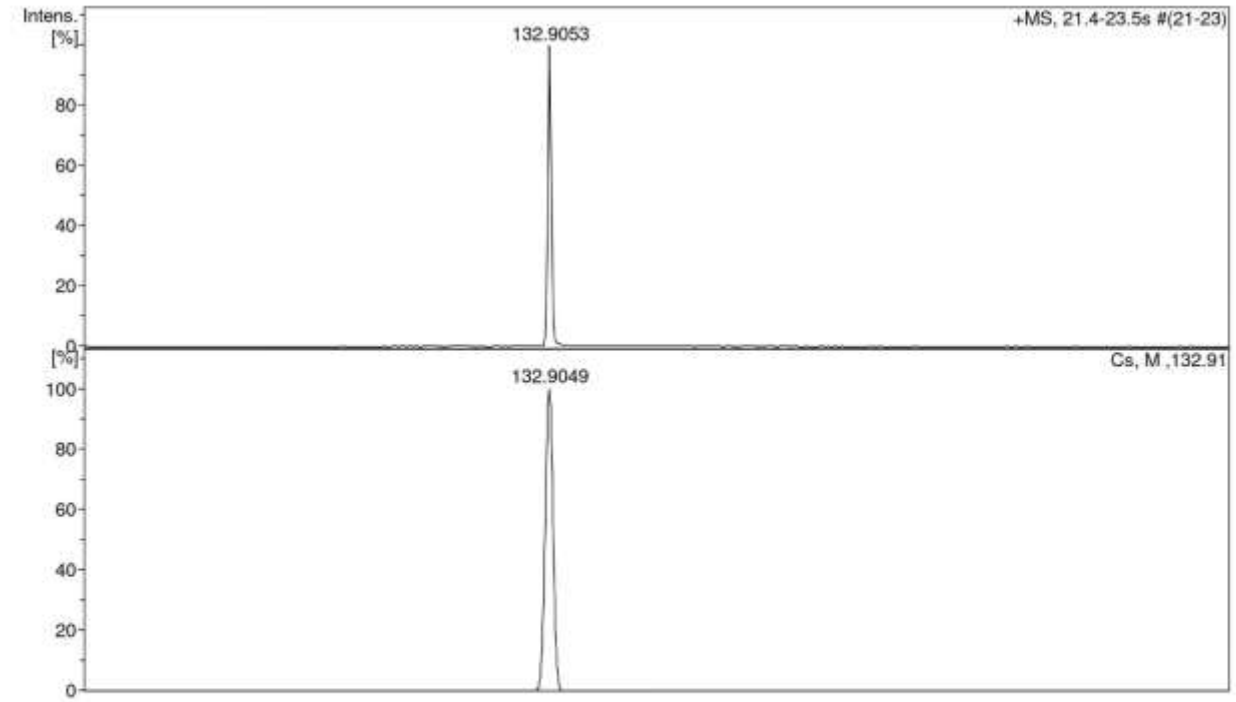
**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\Ilyushenkova\ULI-340.d | Acquisition Date | 26.09.2023 19:42:33 |
| Method | tune_100-1200.m | Operator | BDAL@DE |
| Sample Name | | Instrument / Ser# | maXis 43 |
| Comment | | | |

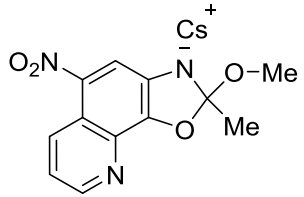
**Acquisition Parameter**

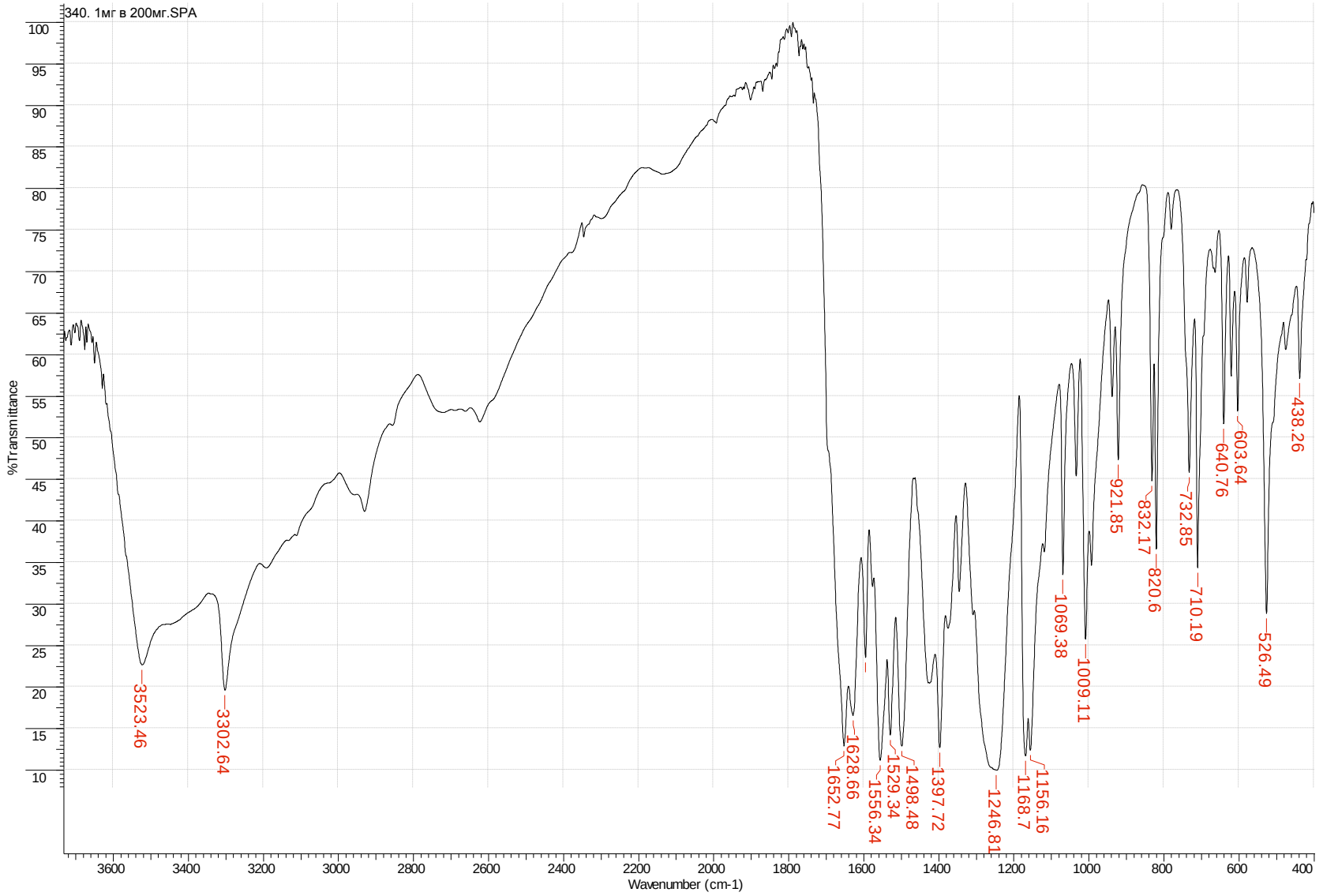
| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.0 Bar |
| Focus | Active | | | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set Capillary | 4500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 1800 m/z | Set End Plate Offset | -500 V | Set Divert Valve | Waste |



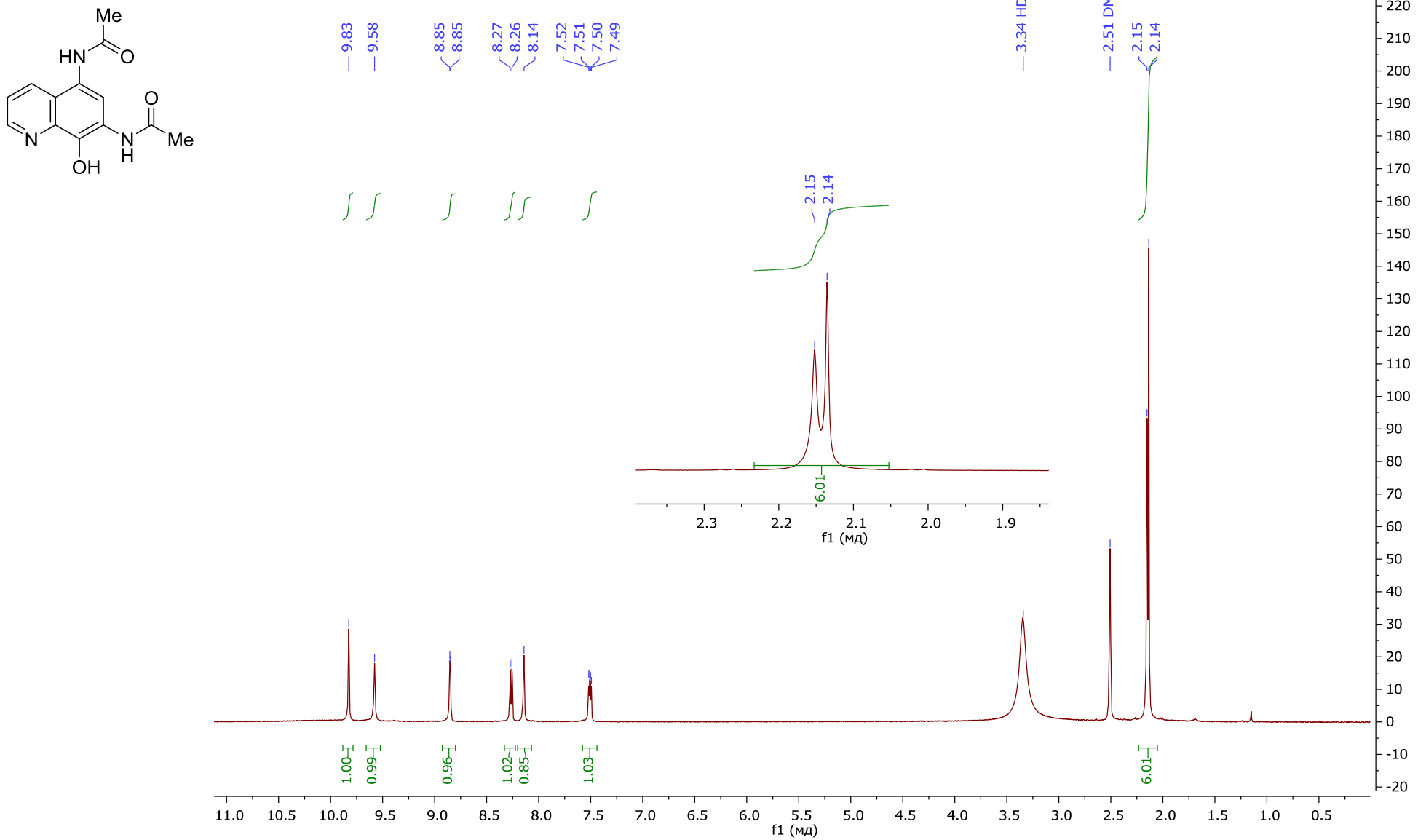



**Caesium 2-methoxy-2-methyl-5-nitrooxazolo[4,5-*h*]quinolin-3-ide (6) FT-IR**

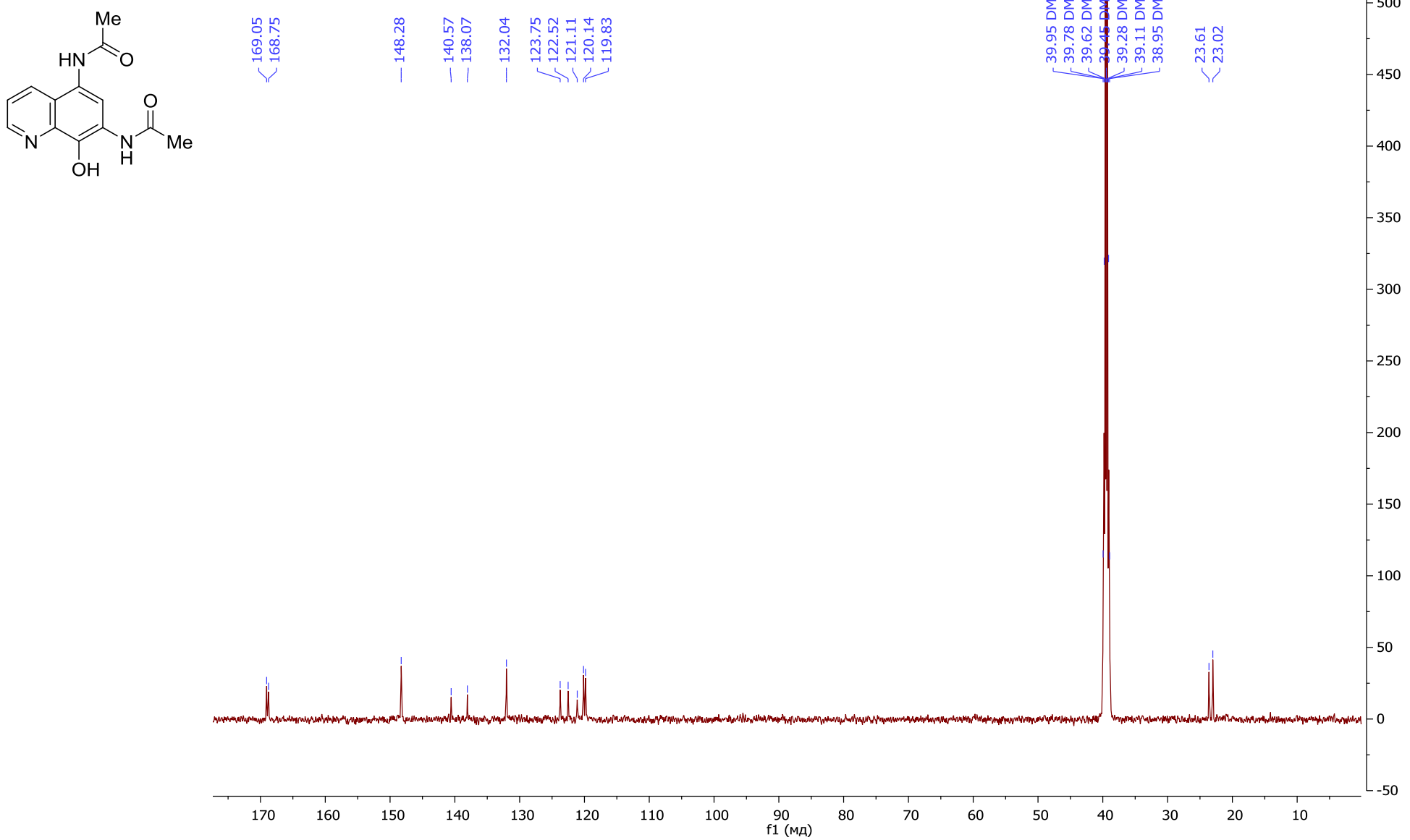


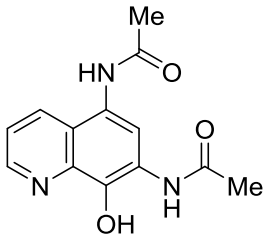
**_N_,_N_'-(8-hydroxyquinoline-5,7-diyl)diacetamide (7) $^{1}$H NMR**



**_N_,_N_'-(8-hydroxyquinoline-5,7-diyl)diacetamide (7) $^{13}$C NMR**



## *N*,*N*'-(8-hydroxyquinoline-5,7-diyl)diacetamide (7) HRMS



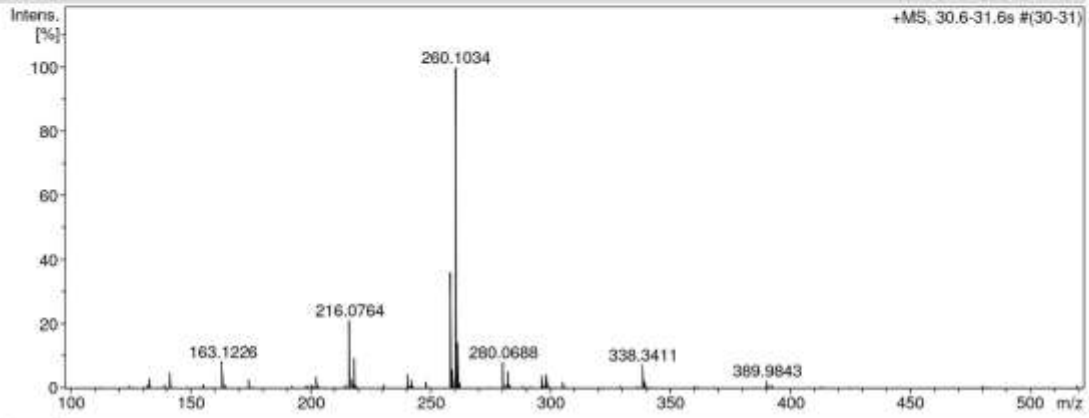

# Display Report

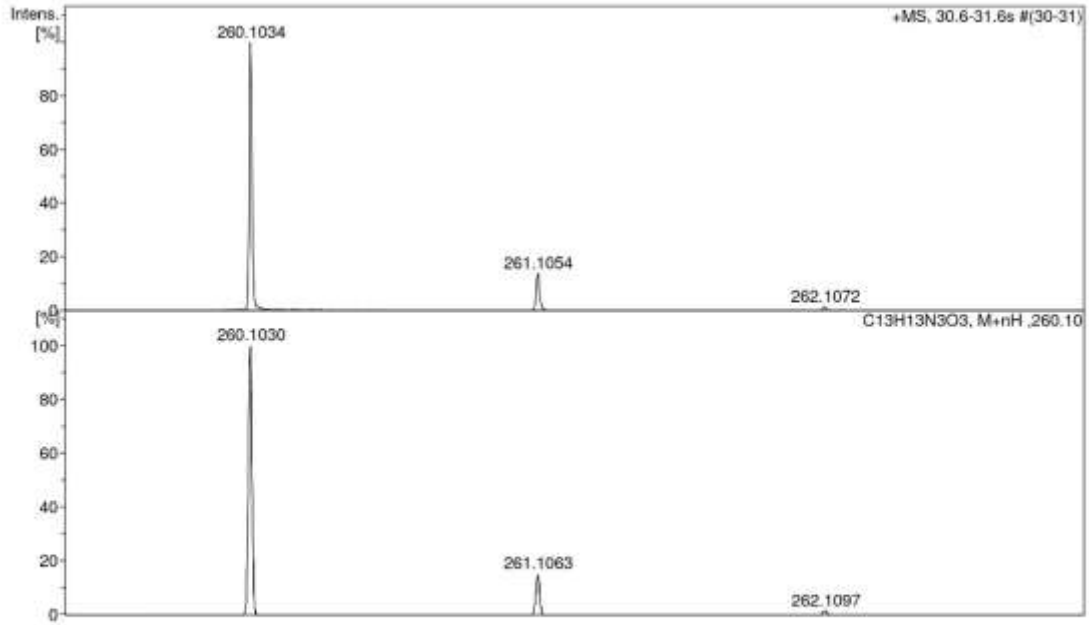
**Analysis Info**

| | | | |
|---|---|---|---|
| Analysis Name | D:\Data\Ilyushenkova\ULI-279.d | Acquisition Date | 26.09.2023 19:45:57 |
| Method | tune_100-1200.m | Operator | BDAL@DE |
| Sample Name | | Instrument / Ser# | maXis 43 |
| Comment | | | |

**Acquisition Parameter**

| | | | | | |
|---|---|---|---|---|---|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.0 Bar |
| Focus | Active | | | Set Dry Heater | 200 °C |
| Scan Begin | 50 m/z | Set Capillary | 4500 V | Set Dry Gas | 4.0 l/min |
| Scan End | 1800 m/z | Set End Plate Offset | -500 V | Set Divert Valve | Waste |



**_N_,_N_'-(8-hydroxyquinoline-5,7-diyl)diacetamide (7) FT-IR**