

## *Supplementary Materials*

### **A New Approach for the Synthesis of 2.3.4a,6.7.8a,9.10–Octaaza-4.8-Dioxo-3.4.4a,7.8.8a,9.9a,10.10a-Decahydroanthracene and High-energy Performance Characterization of Its Dinitramide Salt**

Vera S. Glukhacheva<sup>1,\*</sup>, Sergey G. Il'yasov,<sup>1</sup> Dmitri S. Il'yasov<sup>1</sup>, Egor E. Zhukov<sup>1</sup>,  
Ilia V. Eltsov<sup>2</sup>, Andrey A. Nefedov<sup>2,3</sup>

<sup>1</sup> *Institute for Problems of Chemical and Energetic Technologies, Siberian Branch of the Russian Academy of Sciences (IPCET SB RAS), Biysk 659322, Russia*

<sup>2</sup> *Department of Natural Sciences, Novosibirsk State University, Novosibirsk 630090, Russia*

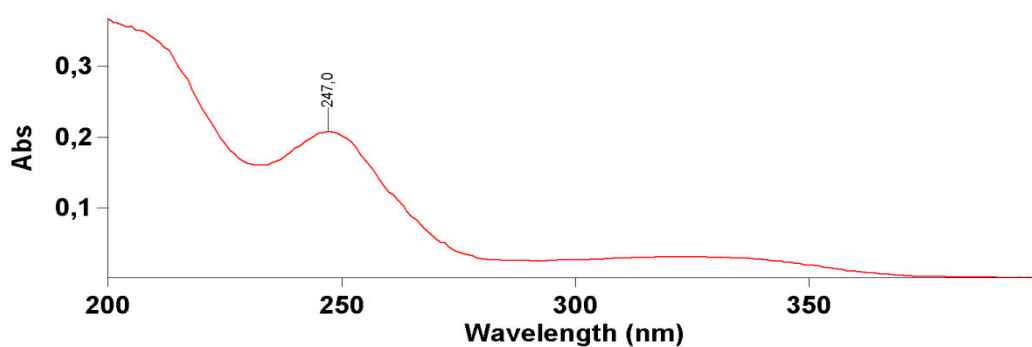
<sup>3</sup> *Novosibirsk Institute of Organic Chemistry, Siberian Branch of the Russian Academy of Sciences (NIOCh SB RAS), Novosibirsk 630090, Russia*

*\*Correspondence. E-mail: vera2878@mail.ru*

#### **Table of Contents**

|   |     |
|---|-----|
| UV spectra of <b>2</b> , <b>3</b> and <b>4</b>  | S2  |
| IR spectra of <b>2</b> , <b>3</b> , <b>4</b> and ADN  | S3  |
| <sup>1</sup> H and <sup>13</sup> C NMR spectra of <b>3</b> , <b>4</b> and ADN   | S5  |
| DSC/TGA of <b>3</b> and <b>4</b>  | S11 |
| MS analysis of <b>2</b> and <b>4</b>  | S13 |
| Effect of compound <b>4</b> on the burning rate of the Zr/KNO <sub>3</sub> and KClO <sub>4</sub> /Al pyrotechnic compositions | S19 |

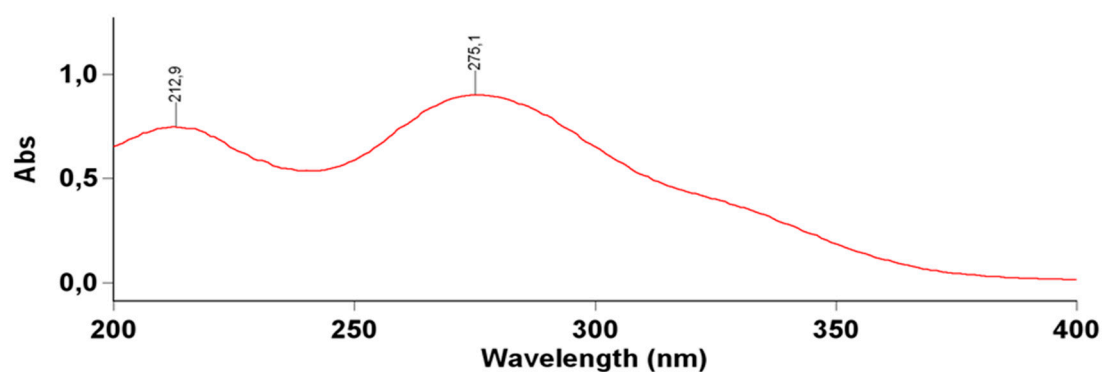
**Figure S1.** UV-vis spectra of **2** in H<sub>2</sub>O at room temperature



| Wavelength (nm) | Abs |
|-----------------|-----|
|-----------------|-----|

|       |       |
|-------|-------|
| 247.0 | 0.219 |
|-------|-------|

**Figure S2.** UV-vis spectra of **3** in H<sub>2</sub>O at room temperature

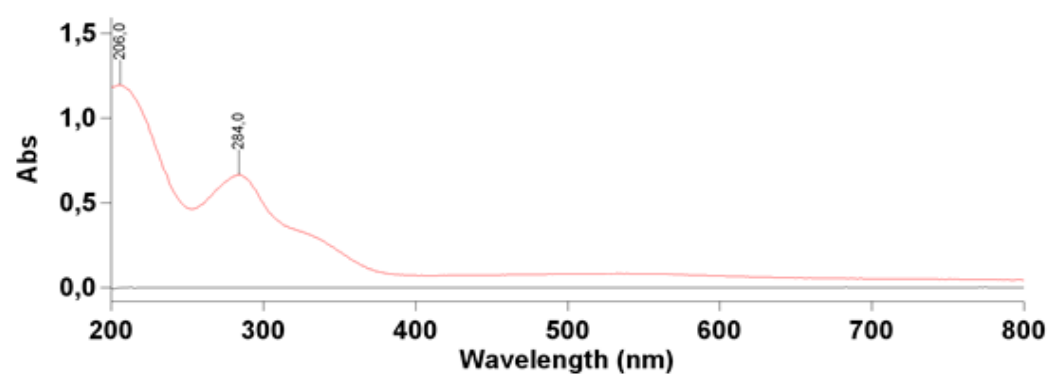


| Wavelength (nm) | Abs |
|-----------------|-----|
|-----------------|-----|

|       |       |
|-------|-------|
| 275.1 | 0.900 |
|-------|-------|

|       |       |
|-------|-------|
| 212.9 | 0.748 |
|-------|-------|

**Figure S3.** UV-vis spectra of **4** in H<sub>2</sub>O at room temperature



| Wavelength (nm) | Abs |
|-----------------|-----|
|-----------------|-----|

|       |       |
|-------|-------|
| 284.0 | 0.664 |
|-------|-------|

|       |       |
|-------|-------|
| 206.0 | 1.195 |
|-------|-------|

**Figure S4.** IR spectrum of **2**

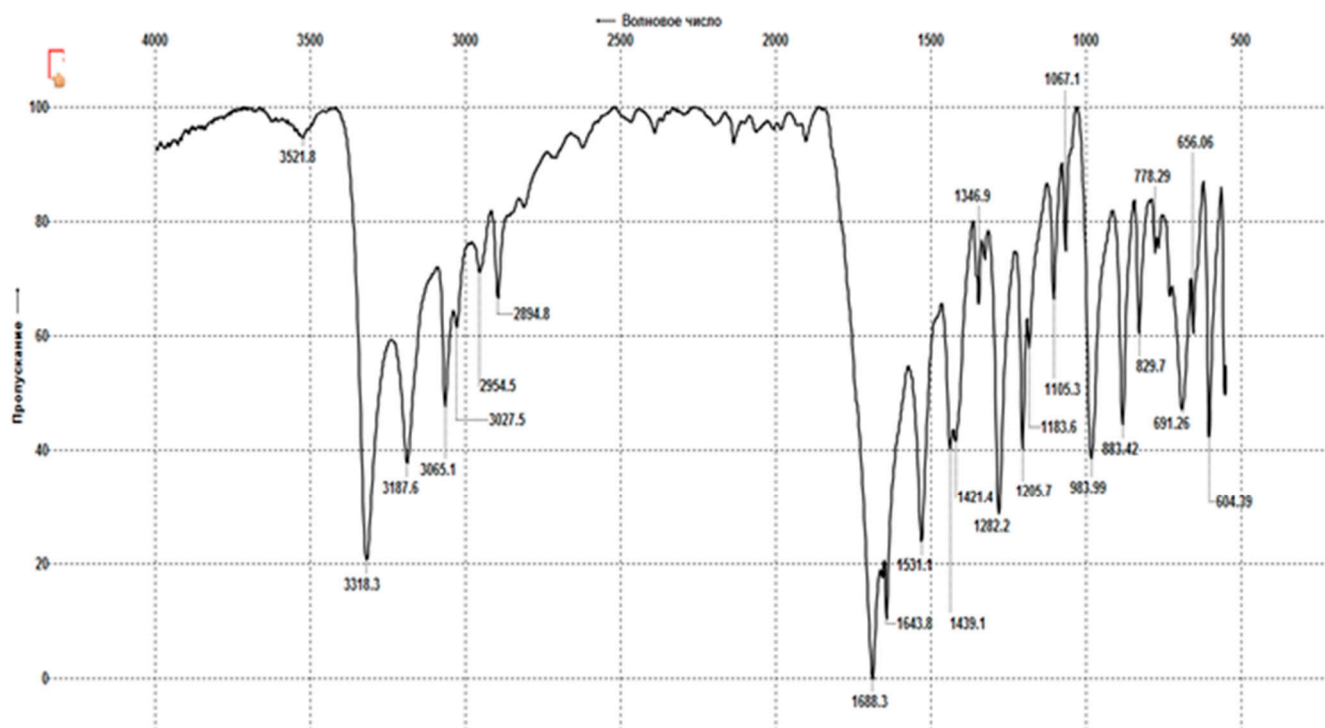


Figure S5. IR spectrum of 3

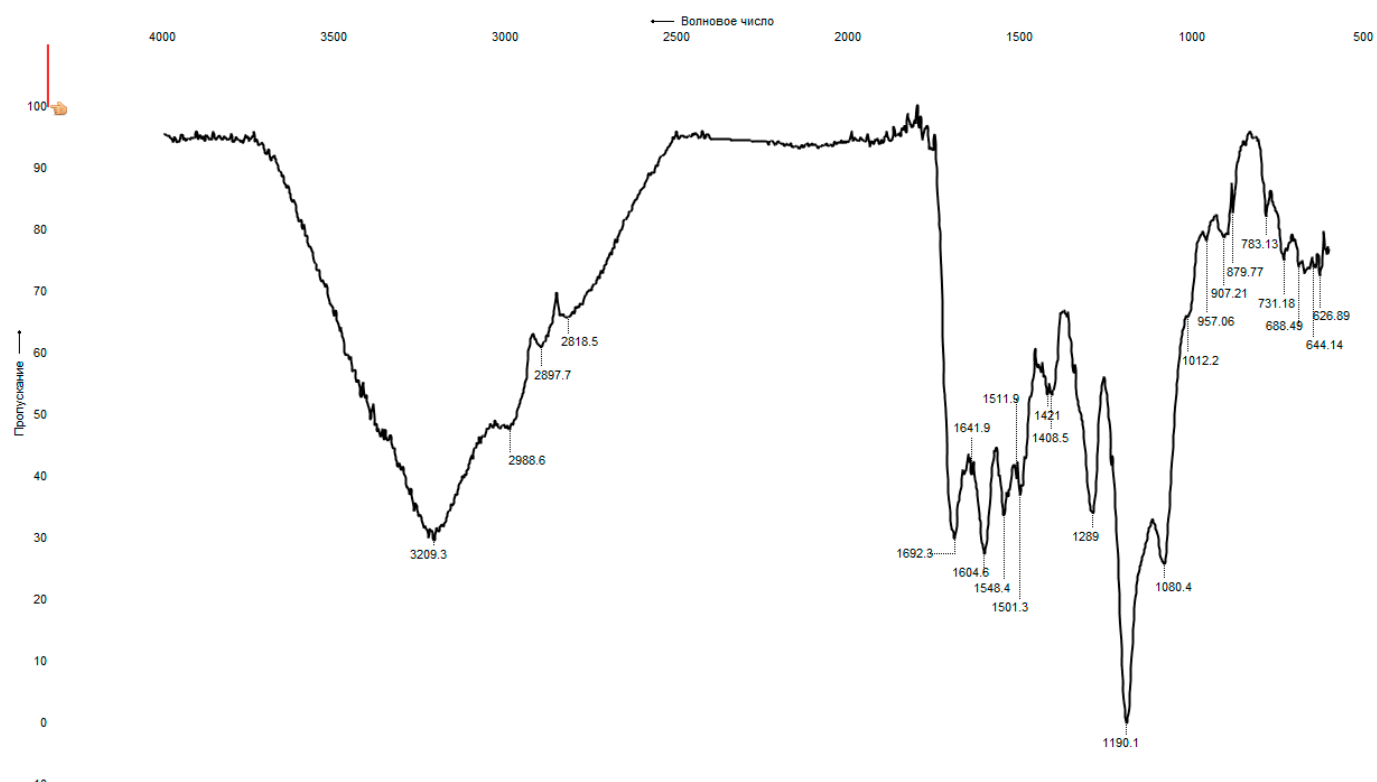
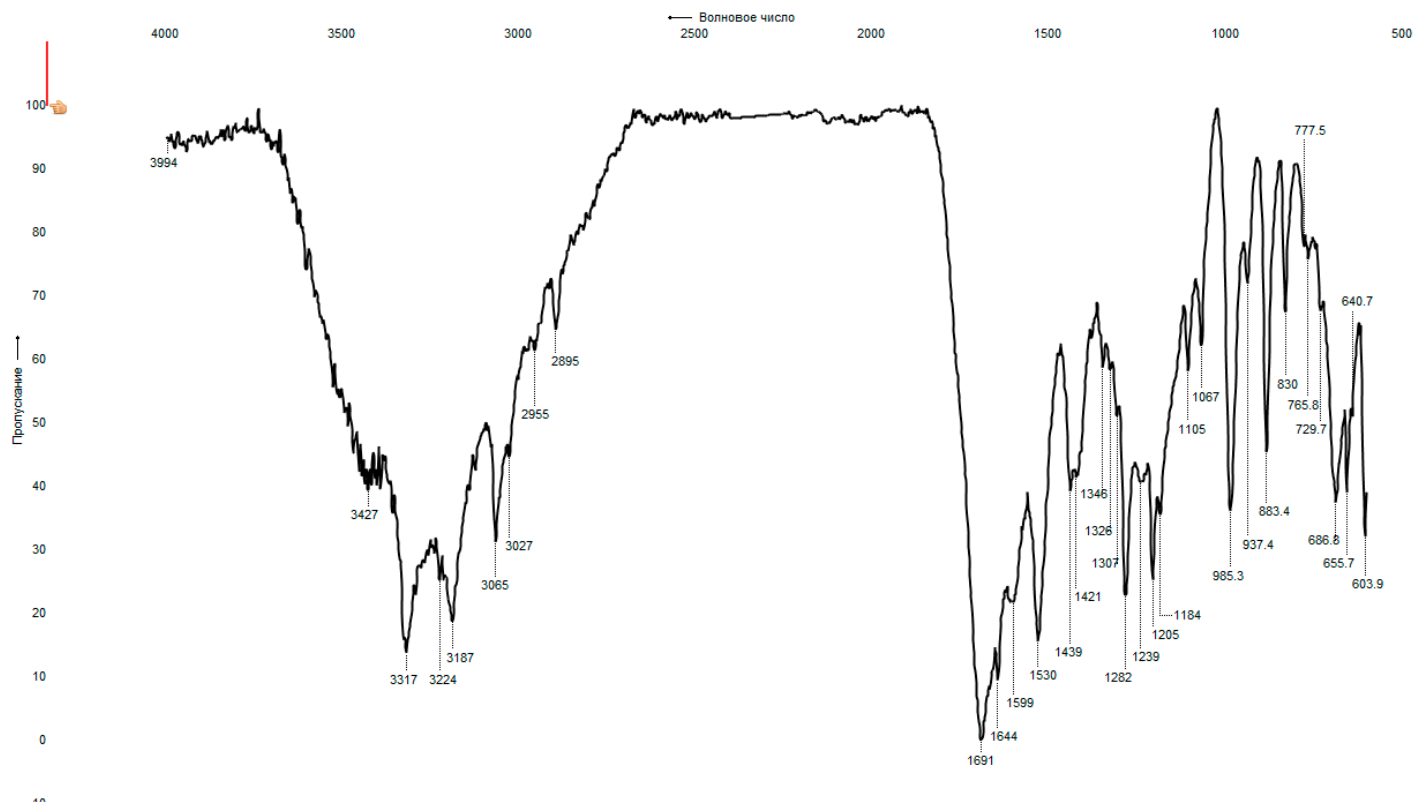
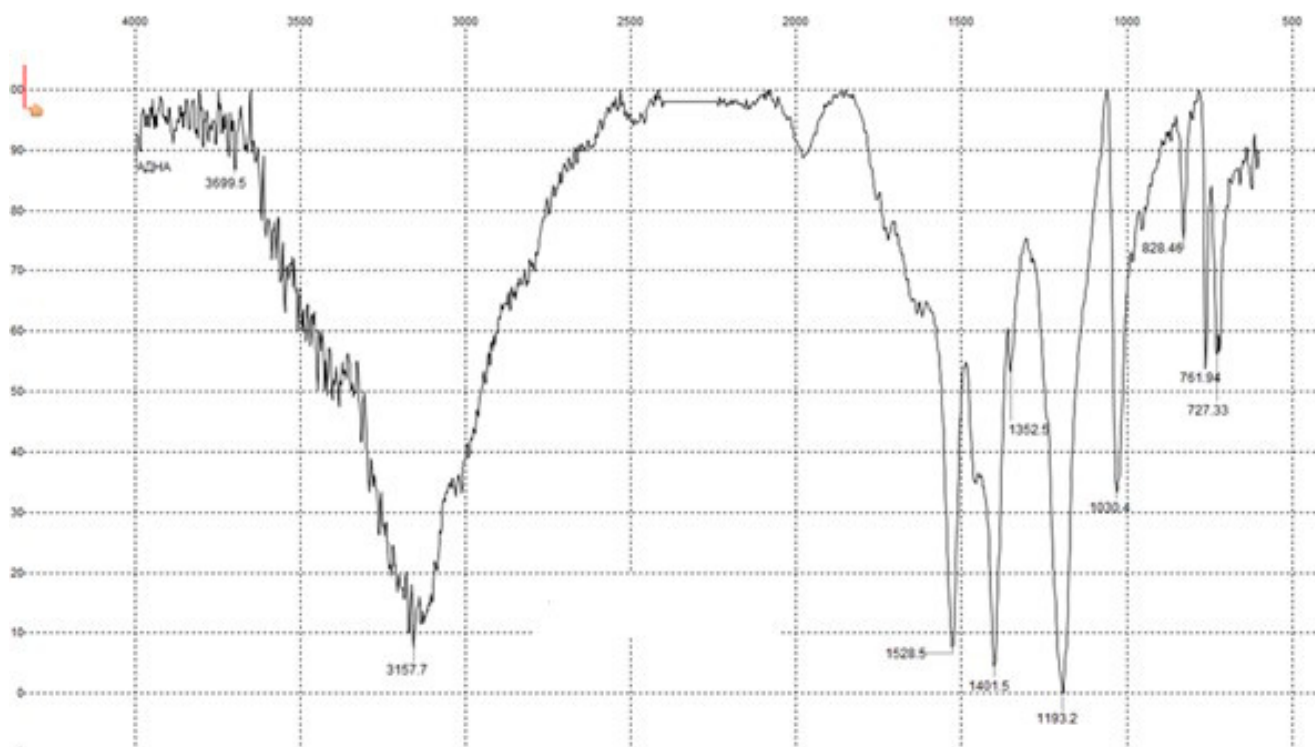


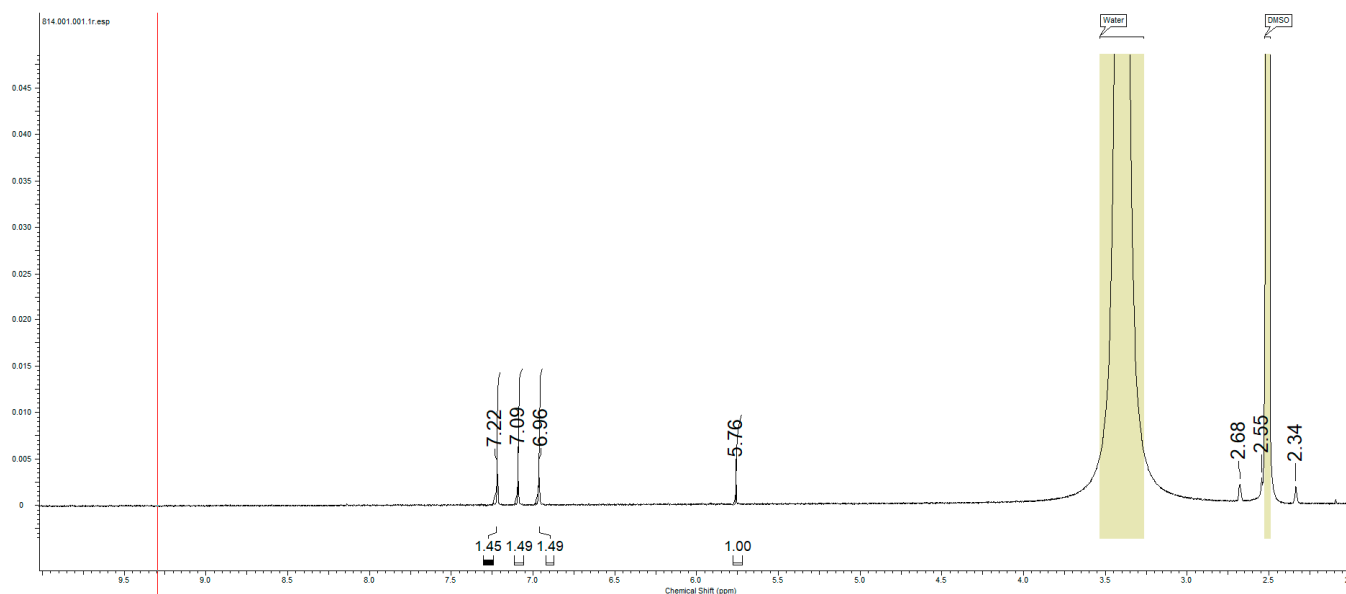
Figure S6. IR spectrum of 4



**Figure S7.** IR spectrum of ADN

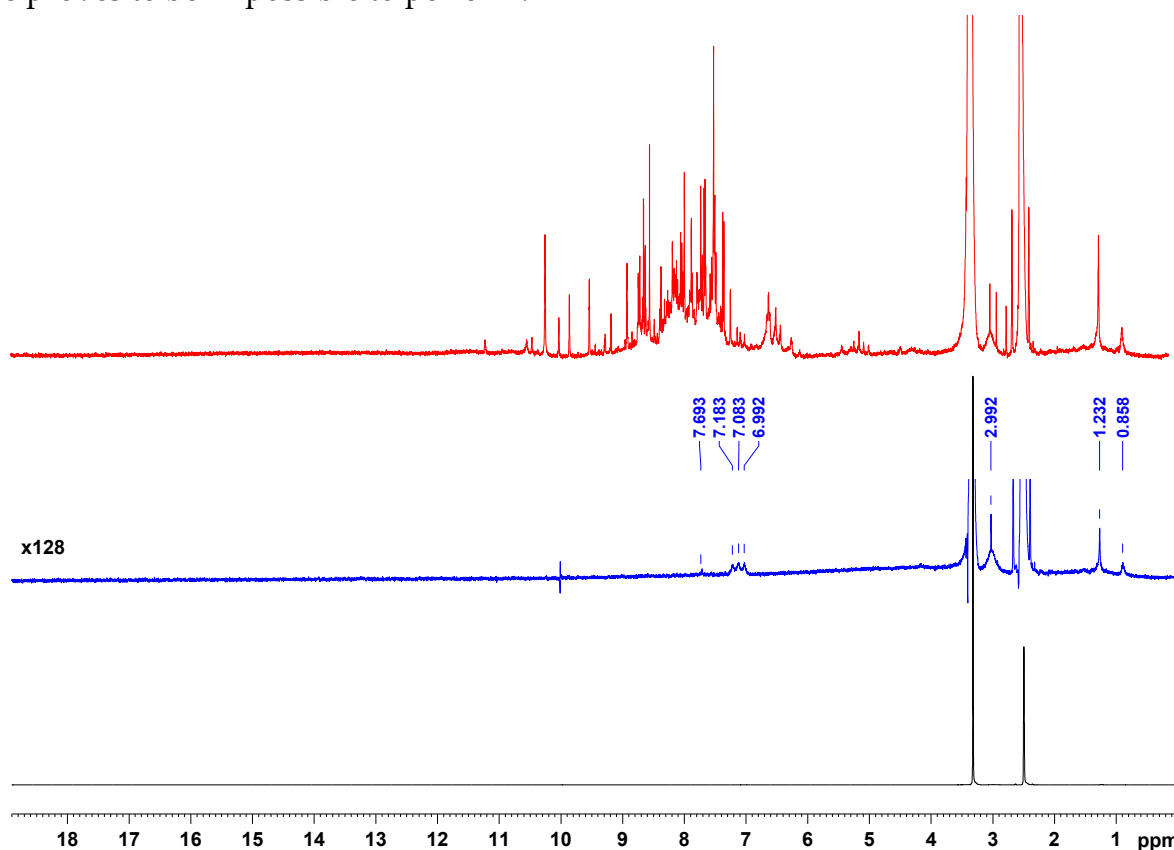


**Figure S8.**  $^1\text{H}$  NMR spectrum of **3** in DMSO, 400.13 MHz (accumulation for 24 h)



**Figure S9.**  $^1\text{H}$  NMR spectrum of **3** in DMSO, 500.03 MHz

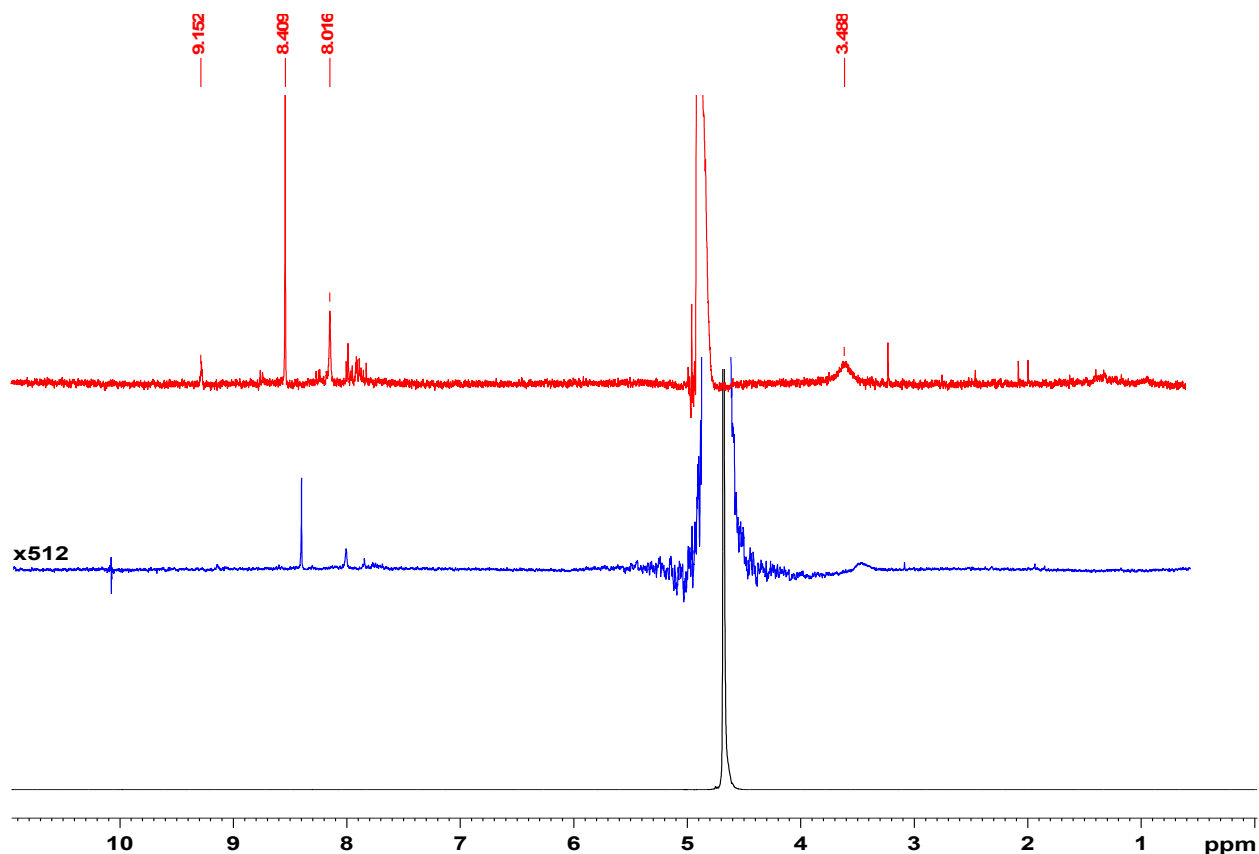
An attempt to heat up the sample for 30 min on a water bath at 80–90°C, followed by recording a  $^1\text{H}$  NMR spectrum under the same conditions, led to the appearance of different signals of 6–11 ppm, which can be assigned both to hydrogen atoms of unsaturated moieties and to hydrogen atoms of NH and  $\text{NH}_2$  groups, and so on. A detailed analysis of the resultant mixture proves to be impossible to perform.



The blue insertion is a spectrum increased by 128 times, and the red insertion is the same sample once heated on a water bath.

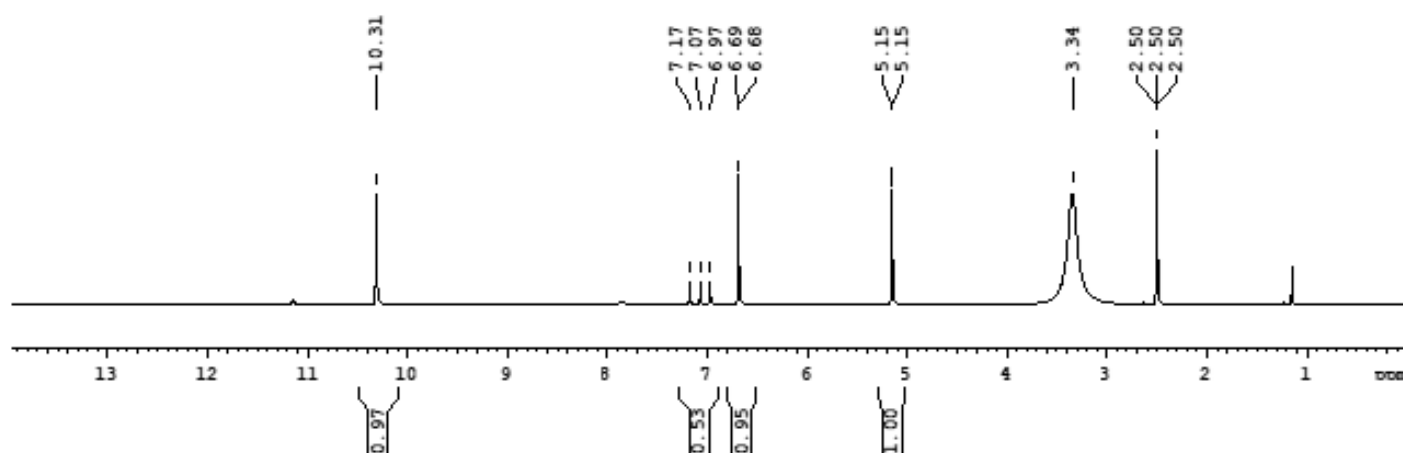
**Figure S10.**  $^1\text{H}$  NMR spectrum of **3** in  $\text{D}_2\text{O}$ .

Due to the low solubility of the sample in DMSO, an attempt was made to take a spectrum in D<sub>2</sub>O and DMF-d<sub>7</sub>. The <sup>1</sup>H NMR spectrum had quite a small quantity of signals, among which the most intense is a singlet at 8.41 ppm; however, it cannot reliably be asserted that it refers to the azomethine CH=N group: we failed to obtain a correlation of CH at this solubility.

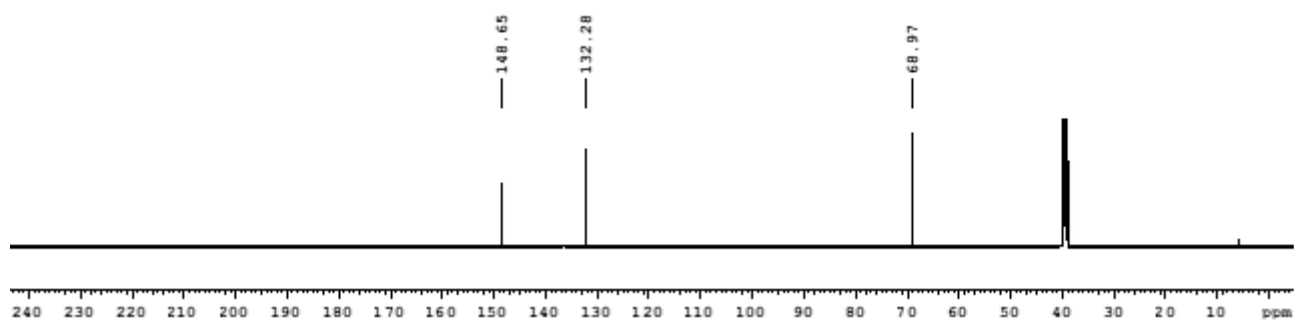


The blue insertion is a spectrum increased by 512 times, and the red insertion is a spectrum obtained via pre-saturation of the water signal.

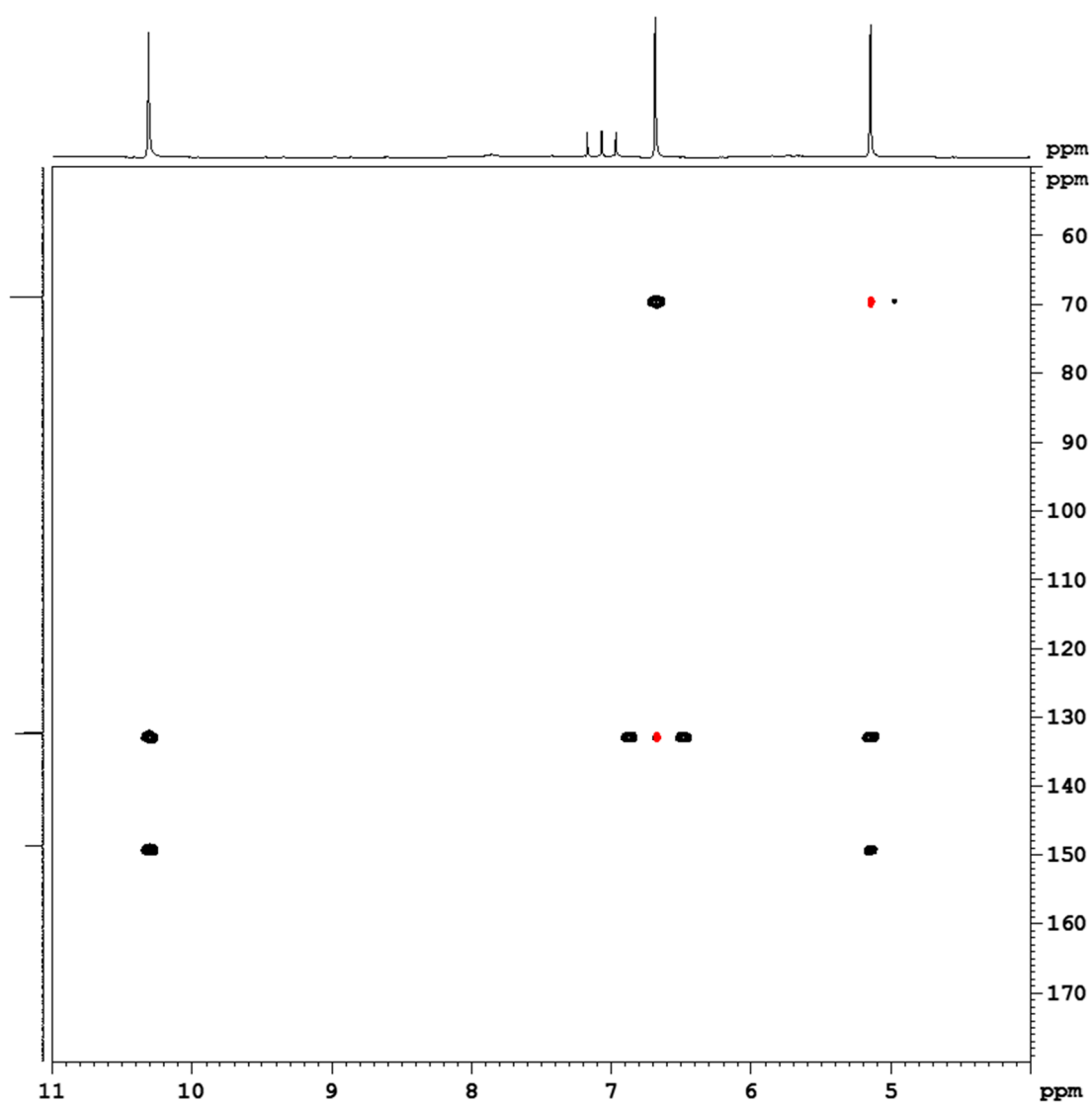
**Figure S11.** <sup>1</sup>H NMR spectrum of **4** in DMSO, 500.03 MHz



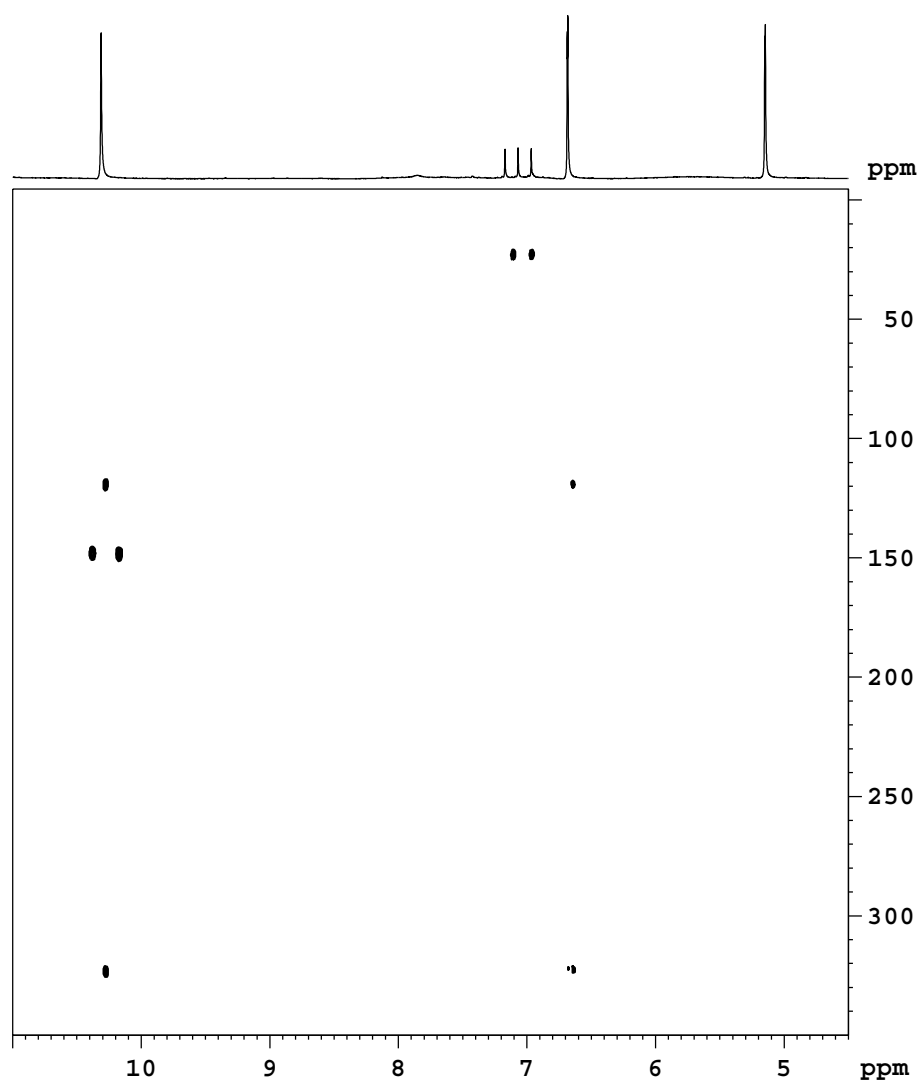
**Figure S 12.** <sup>13</sup>C NMR spectrum of **4** in DMSO



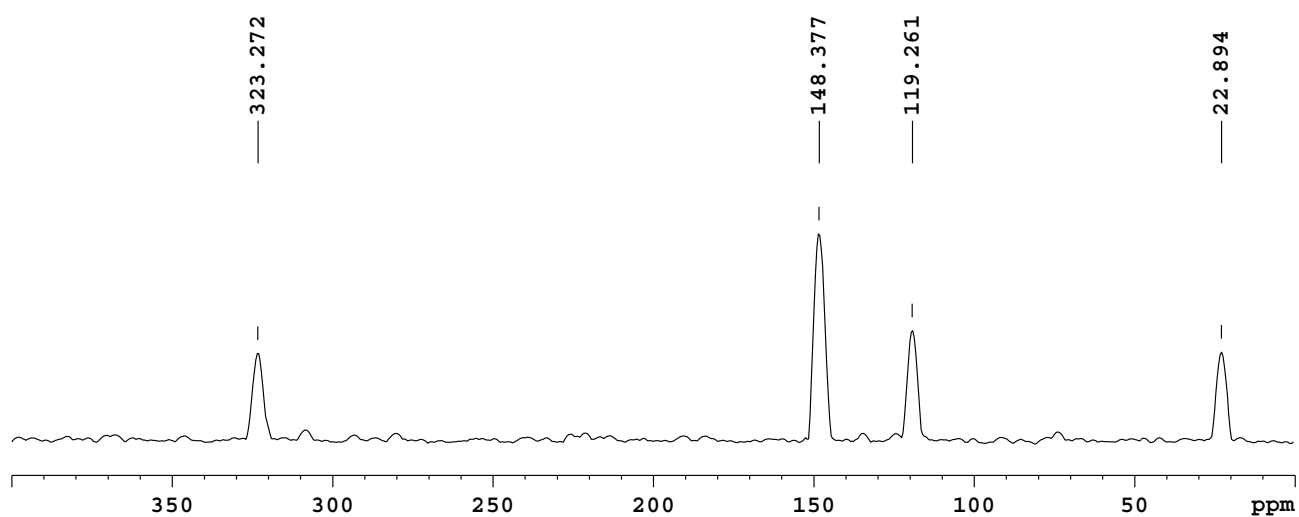
**Figure S13.**  $^1\text{H}$ ,  $^{13}\text{C}$  HSQC (red) and  $^1\text{H}$ ,  $^{13}\text{C}$  HMBC (black) spectrum of **4** in DMSO



**Figure S14.**  $^1\text{H}$ ,  $^{15}\text{N}$  HMBC spectrum of **4** in DMSO

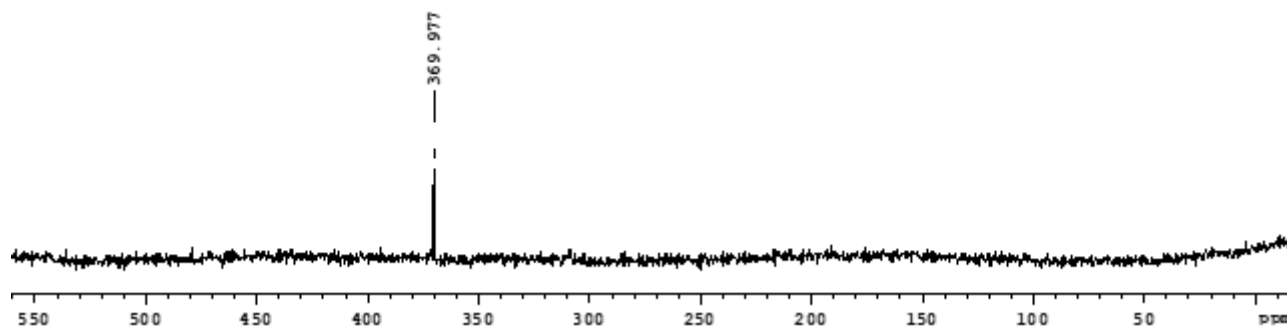


**Figure S15.**  $^1\text{H}$ ,  $^{15}\text{N}$  HMBC spectrum of **4** in DMSO



**Figure S16.**  $^{14}\text{N}$  NMR spectrum of **4** in DMSO recorded relative to formamide as reference standard ( $\delta$ ) = 112.5 ppm



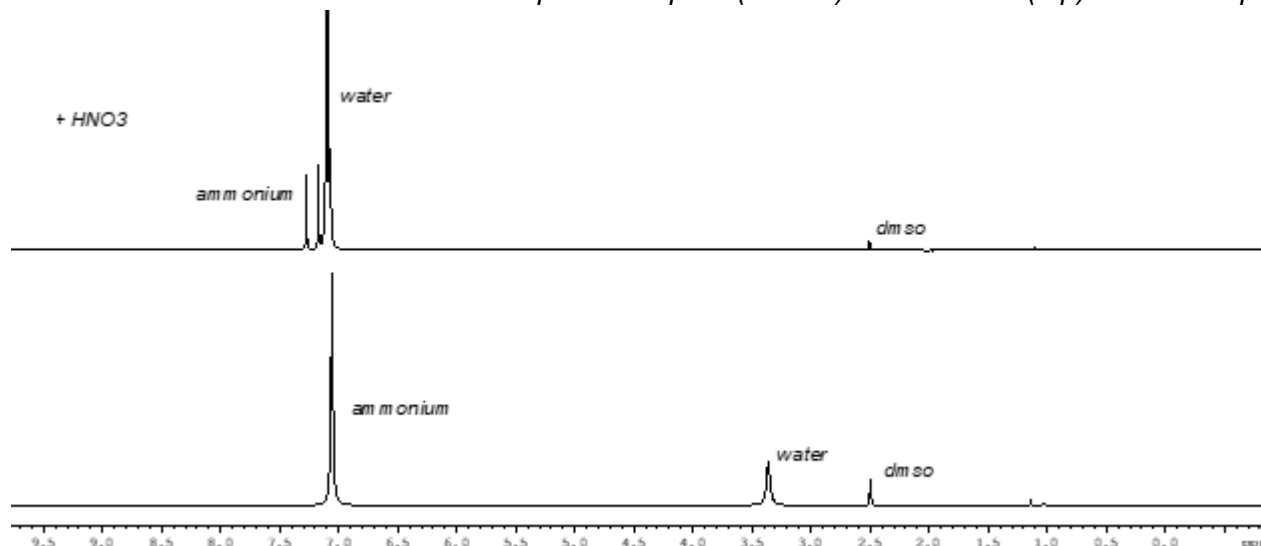


**Figure S17.** NMR spectrum of ADN

ADN was characterized via one-dimensional spectroscopy on  $^1\text{H}$  and  $^{14}\text{N}$  nuclei. The absence of hydrogen atoms in the anion makes analysis via two-dimensional spectroscopy meaningless, and any attempt to obtain a one-dimensional spectrum of  $^{15}\text{N}$  using the natural content of the isotope, taking into account the structure of the molecule, is futile. The sample was removed in neat DMSO (19 mg in 0.6 ml) and under acidification conditions using nitric acid (2 drops of concentrated nitric acid).

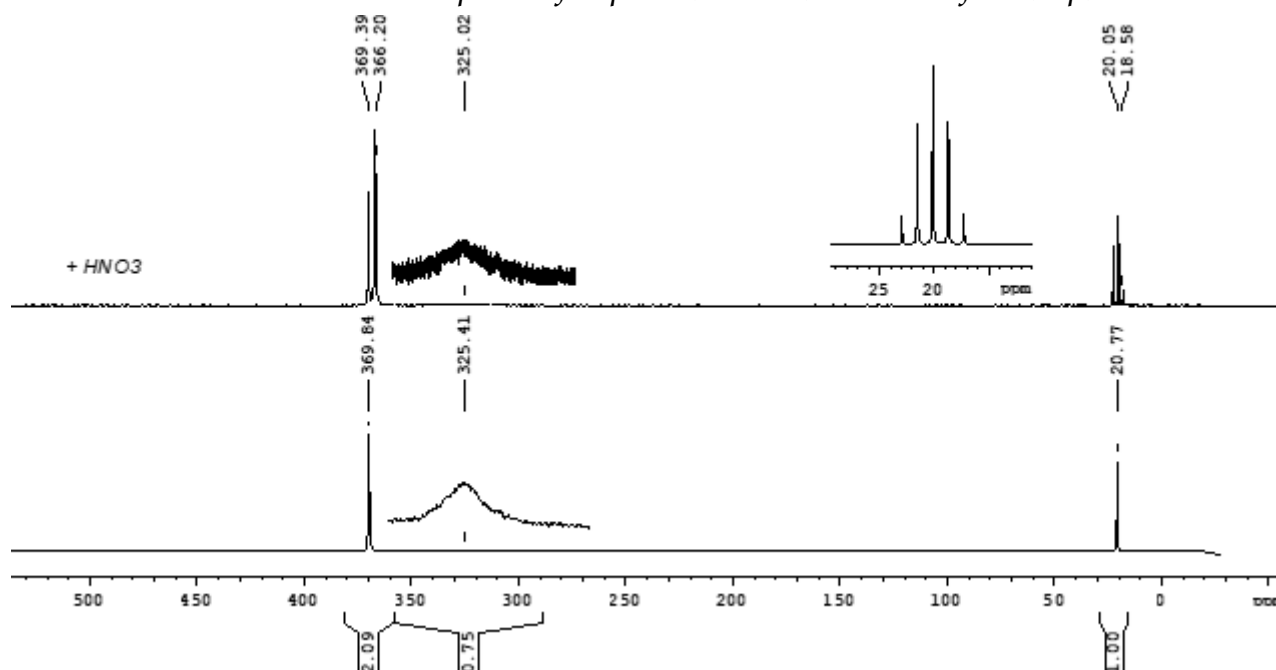
In the proton spectrum of the sample, a slight impurity signal at 1 ppm, a solvent signal (DMSO, 2.5 ppm), water (3.36 ppm), and a signal from the hydrogen atoms of the ammonium cation were observed. The presence of water in the sample indicates the fact that hydrogen atoms are involved in chemical exchange and appear in the form of a broadened singlet. Acidification of the environment shifts the equilibrium of this process towards stabilization of the  $\text{NH}_4^+$  particle, which in turn leads to the manifestation of interaction between the magnetic moments of the  $^1\text{H}$  and  $^{14}\text{N}$  nuclei and transformation of the signal into a triplet with a line ratio of 1:1:1 and a constant  $^1\text{J}_{\text{NH}} = 51.2$  Hz.

<sup>1</sup>H-NMR spectra for pure (bottom) and acidified (top) "ADN" sample.

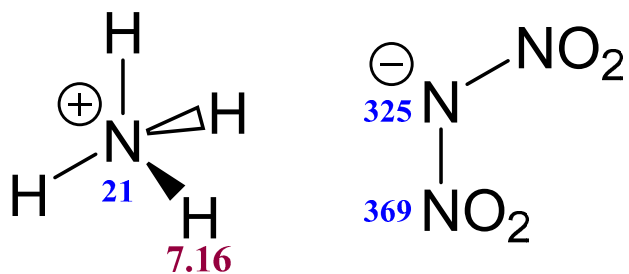


In the spectrum of the <sup>14</sup>N nucleus, there are three signals at an integral ratio of 1: 1: 2. The signal at 20.77 ppm corresponds to the nitrogen atom of the ammonium cation and, upon acidification, also exhibits splitting in the form of a quintet with a constant of  $^1J_{NH} = 51.2$  Hz. The broad signal at 325 ppm corresponds to the central nitrogen atom of the N(NO<sub>2</sub>)<sub>2</sub> anion. The broadening occurs due to quadrupole interactions with neighboring <sup>14</sup>N nuclei. The signal from nitro groups, doubled in intensity, appears at 369 ppm. Acidification of the solution has no effect on the last two signals.

<sup>14</sup>N-NMR spectra for pure (bottom) and acidified (top) "ADN" sample.



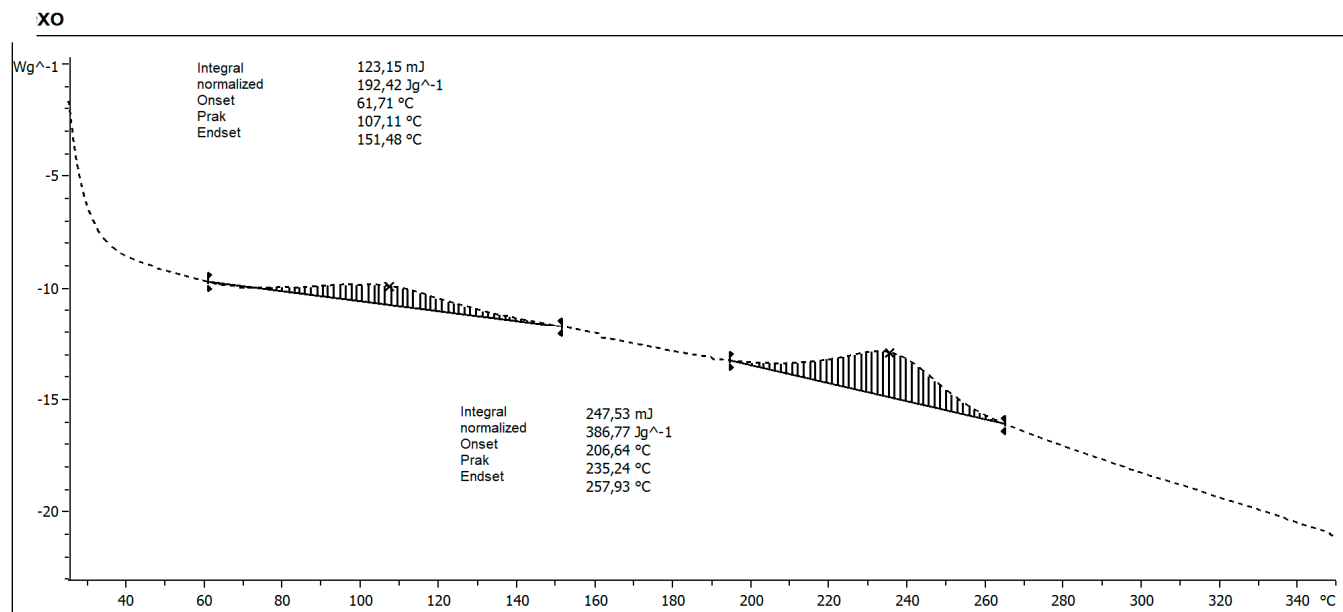
Thus, the following assignment of signals to the connection structure can be performed:



$^1\text{H}$  NMR (500 MHz, DMSO)  $\delta$  7.16 ( $\text{NH}_4^+$ ).

$^{14}\text{N}$  NMR (36 MHz,  $\text{HCONH}_2$ )  $\delta$  21 ( $\text{NH}_4^+$ ), 325 ( $\underline{\text{N}}(\text{NO}_2)_2$ ), 369 ( $\text{N}(\underline{\text{NO}}_2)_2$ ).

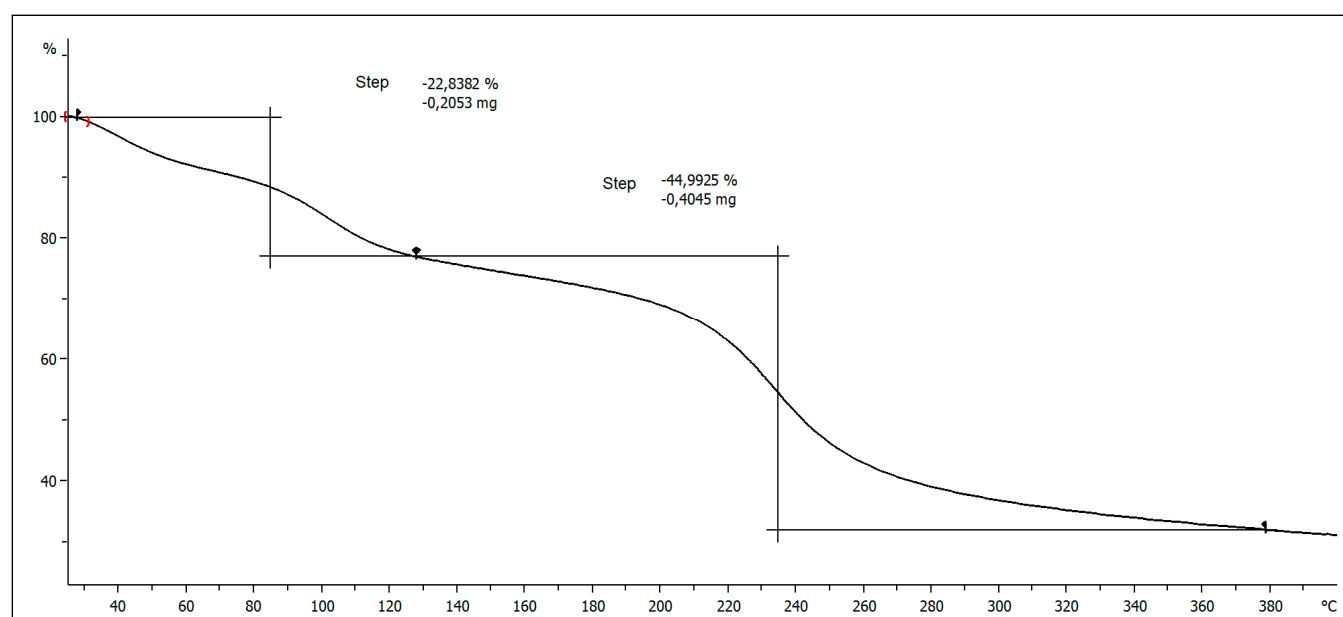
Figure S18. DSC of 3



Lab: METTLER

STAR<sup>e</sup> SW 14.00

Figure S19. TGA of 3



Lab: METTLER

STAR<sup>e</sup> SW 14.00

Figure S20. DSC of 4

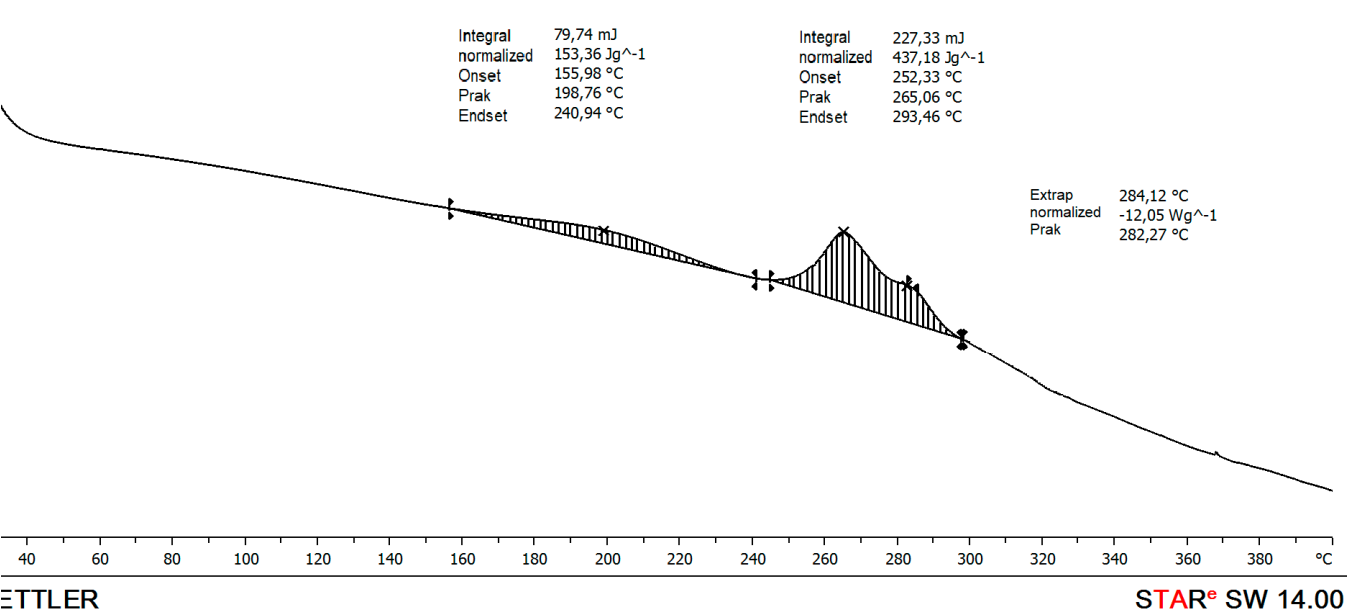


Figure S21. TGA of 4

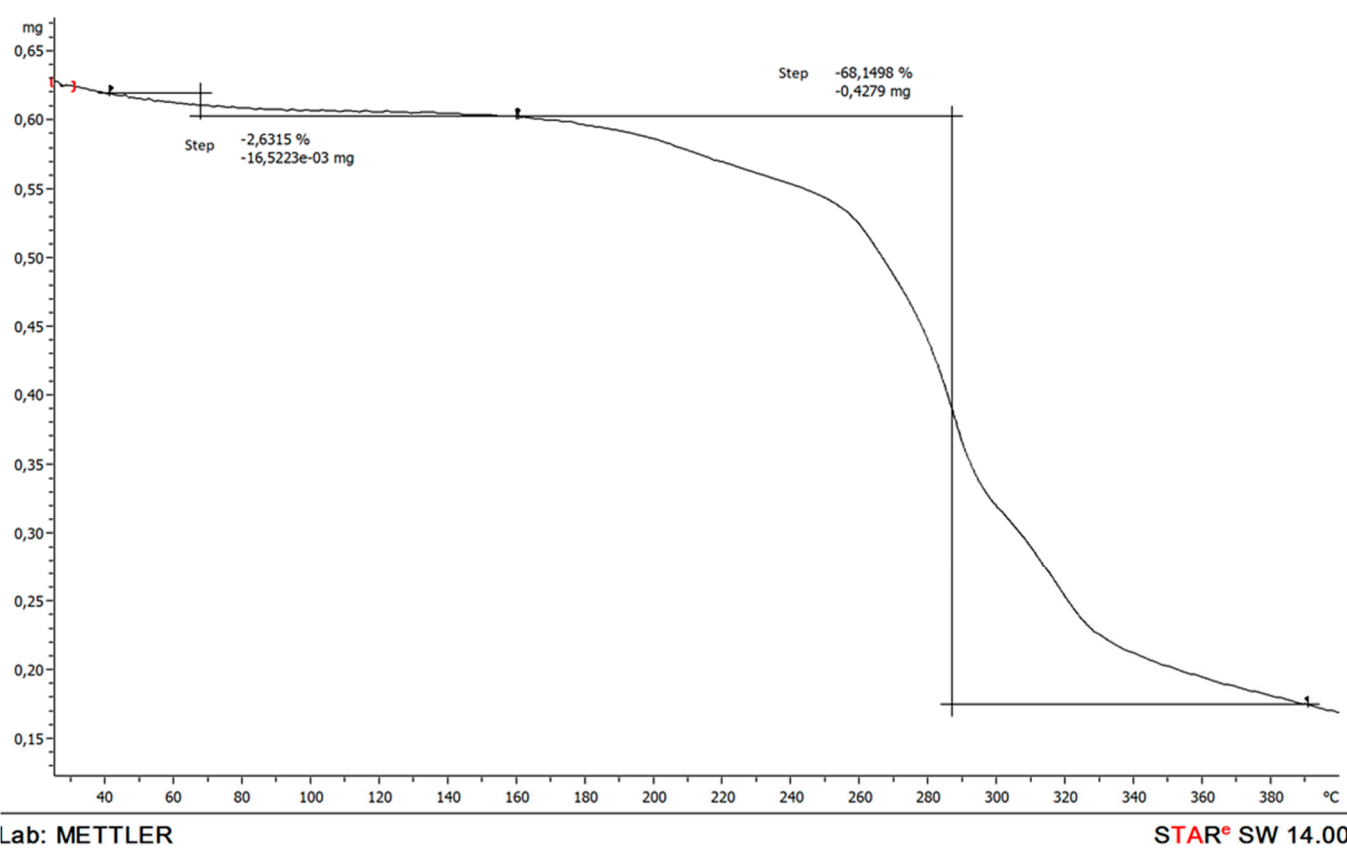


Figure S22. MS analysis of 2  
DFS high-resolution mass-spectrometer

**Injection conditions: a direct injection of the sample by using a heated plunger.**

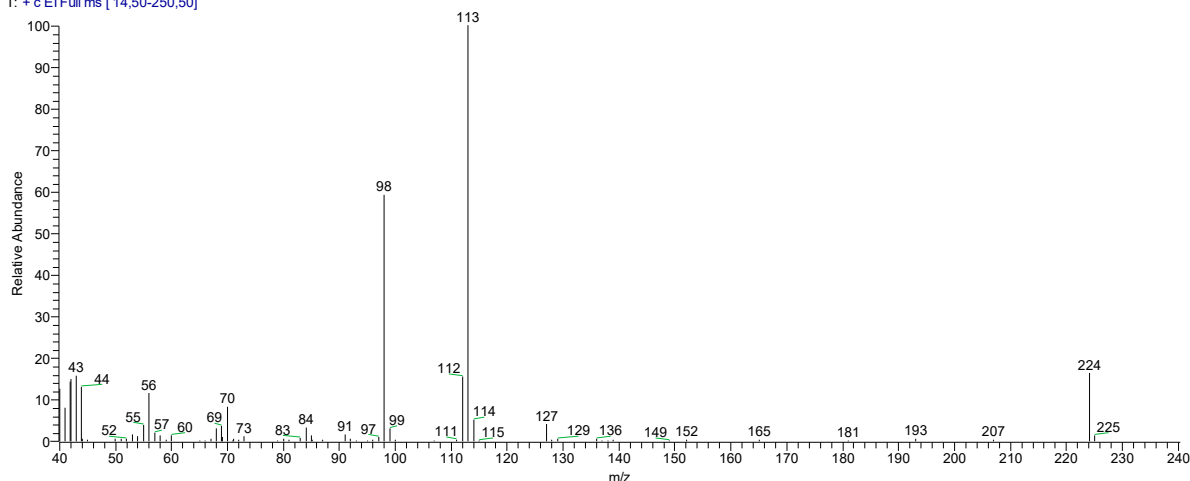
**T<sub>source</sub> = 200 °C**

**T<sub>probe</sub> = 300 °C**

**Electronic Ionization Mass-Spectrometry:** HR-MS: Calcd for C<sub>6</sub>H<sub>8</sub>N<sub>8</sub>O<sub>2</sub> [M]<sup>+</sup> 224.0765; found *m/z* 224.0767. LR-MS, *m/z*, %: 224 (M<sup>+</sup>, 16), 113 (100), 112 (16), 98 (59), 56 (12), 44 (13), 43 (17), 42 (15), 40 (13).

### Graphic representation of the mass-spectrum:

910-2 #76 RT: 4.90 AV: 1 NL: 7.58E5  
T: + c EI Full ms [14,50-250,50]



### Numerical representation of the mass spectrum:

| <i>m/z</i> | Intensity | Relative, % |
|------------|-----------|-------------|
| 40         | 95838.0   | 12.65       |
| 41         | 60373.0   | 7.97        |
| 42         | 113617.0  | 14.99       |
| 43         | 118568.0  | 15.65       |
| 44         | 98391.0   | 12.98       |
| 45         | 2490.0    | 0.33        |
| 50         | 3425.0    | 0.45        |
| 51         | 4483.0    | 0.59        |
| 52         | 4639.0    | 0.61        |
| 53         | 11925.0   | 1.57        |
| 54         | 7939.0    | 1.05        |
| 55         | 29299.0   | 3.87        |
| 56         | 88241.0   | 11.64       |
| 57         | 14385.0   | 1.90        |
| 58         | 10088.0   | 1.33        |
| 59         | 2771.0    | 0.37        |
| 60         | 10586.0   | 1.40        |
| 65         | 1494.0    | 0.20        |
| 66         | 1276.0    | 0.17        |
| 67         | 4421.0    | 0.58        |

|            |                 |               |
|------------|-----------------|---------------|
| 68         | 22978.0         | 3.03          |
| 69         | 27524.0         | 3.63          |
| 70         | 63020.0         | 8.32          |
| 71         | 3829.0          | 0.51          |
| 72         | 1681.0          | 0.22          |
| 73         | 8469.0          | 1.12          |
| 79         | 1152.0          | 0.15          |
| 80         | 4110.0          | 0.54          |
| 81         | 2428.0          | 0.32          |
| 83         | 6165.0          | 0.81          |
| 84         | 25096.0         | 3.31          |
| 85         | 9776.0          | 1.29          |
| 87         | 1774.0          | 0.23          |
| 91         | 11115.0         | 1.47          |
| 92         | 4452.0          | 0.59          |
| 93         | 934.0           | 0.12          |
| 95         | 1556.0          | 0.21          |
| 96         | 2584.0          | 0.34          |
| 97         | 6974.0          | 0.92          |
| <b>98</b>  | <b>449612.0</b> | <b>59.33</b>  |
| 99         | 23165.0         | 3.06          |
| 100        | 2802.0          | 0.37          |
| 107        | 560.0           | 0.07          |
| 111        | 2459.0          | 0.32          |
| <b>112</b> | <b>117852.0</b> | <b>15.55</b>  |
| <b>113</b> | <b>757865.0</b> | <b>100.00</b> |
| 114        | 38422.0         | 5.07          |
| 115        | 1463.0          | 0.19          |
| 127        | 31292.0         | 4.13          |
| 128        | 2241.0          | 0.30          |
| 129        | 4172.0          | 0.55          |
| 136        | 3580.0          | 0.47          |
| 137        | 1120.0          | 0.15          |
| 138        | 1338.0          | 0.18          |
| 139        | 2210.0          | 0.29          |
| 149        | 1401.0          | 0.18          |
| 152        | 1805.0          | 0.24          |
| 165        | 2553.0          | 0.34          |
| 181        | 1401.0          | 0.18          |
| 193        | 3487.0          | 0.46          |
| 207        | 2086.0          | 0.28          |
| <b>224</b> | <b>123799.0</b> | <b>16.34</b>  |
| 225        | 10150.0         | 1.34          |

### Calculation of elemental compositions

Calculated:  $m/z=224.0765$  ( $\text{C}_6\text{H}_8\text{N}_8\text{O}_2$ )<sup>+</sup>

Measured:  $m/z=224.0767$

| Elemental composition |   | RDB | Error (ppm) |
|-----------------------|---|-----|-------------|
| 1                     | C <sub>6</sub> H <sub>8</sub> O <sub>2</sub> N <sub>8</sub> | 7.0 | 1.012       |

### Range of number values of atoms in elements:

| Isotope | min | max |
|---------|-----|-----|
| 12 C    | 4   | 10  |
| 1 H     | 4   | 20  |
| 16 O    | 0   | 6   |
| 14 N    | 0   | 10  |

(M)<sup>+</sup> — a positively charged odd-electron molecular radical ion (radical cation).

### Figure S23. MS analysis of 4

DFS high-resolution mass-spectrometer

Injection conditions: a direct injection of the sample by using a heated plunger.

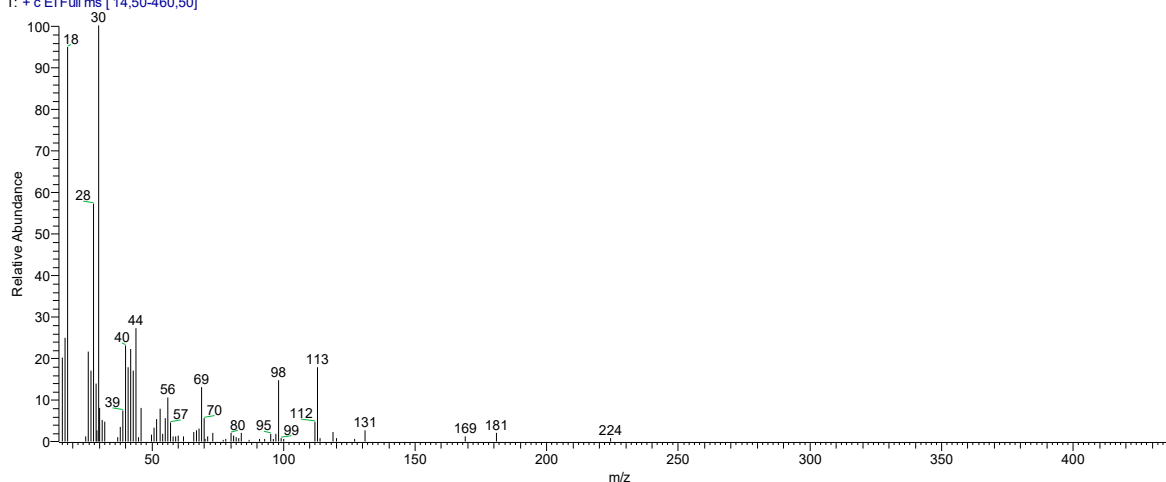
T<sub>source</sub> = 200 °C

T<sub>probe</sub> = 280 °C

**Electronic Ionization Mass-Spectrometry:** HR-MS: Calculated for  $\text{C}_6\text{H}_8\text{N}_8\text{O}_2$  [M]<sup>+</sup> 224.0765; found  $m/z$  224.0763.

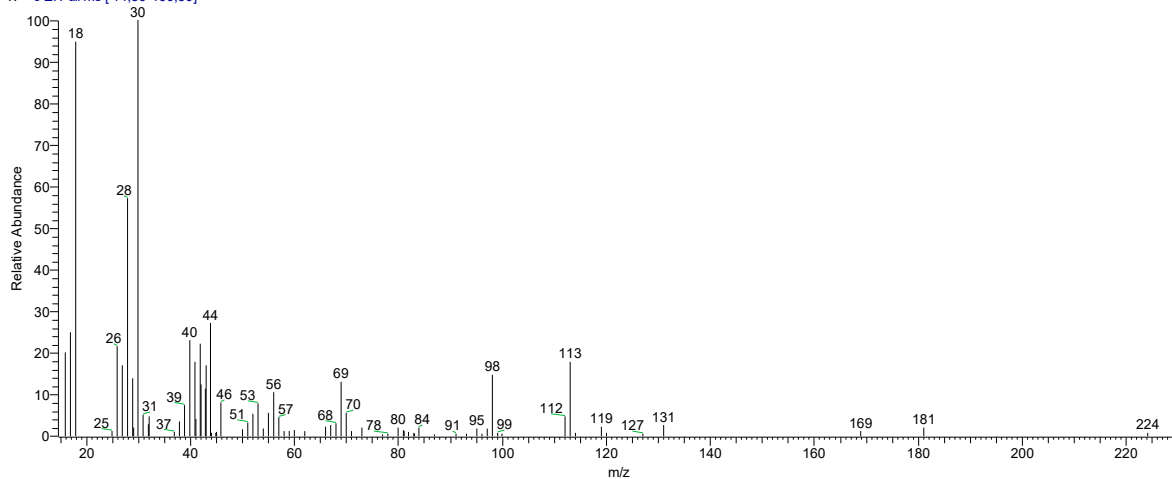
### Graphical representation of mass-spectrum:

1-64\_#2 RT: 0,08 AV: 1 NL: 2,75E5  
T: + cEI Full ms [14,50-460,50]



### Graphical representation of mass-spectrum (enlarged)

1-64\_#2 RT: 0.08 AV: 1 NL: 2.75E5  
T: + c EI Full ms [14,50-460,50]



There are ions from nitro groups with  $m/z = 46$  ( $\text{NO}_2$ ) and  $m/z = 30$  ( $\text{NO}$ ) and intramolecular redox products with  $m/z = 18$  ( $\text{H}_2\text{O}$ ) and  $m/z = 28$  ( $\text{N}_2$ ).

### Numerical representation of mass-spectrum:

| $m/z$ | Intensity | Relative, % |
|-------|-----------|-------------|
| 16    | 54971.0   | 20.00       |
| 17    | 68299.0   | 24.85       |
| 18    | 260700.0  | 94.86       |
| 25    | 3150.0    | 1.15        |
| 26    | 59402.0   | 21.61       |
| 27    | 46871.0   | 17.05       |
| 28    | 157438.0  | 57.29       |
| 29    | 38044.0   | 13.84       |
| 30    | 274824.0  | 100.00      |
| 31    | 13985.0   | 5.09        |
| 32    | 12946.0   | 4.71        |
| 37    | 2388.0    | 0.87        |
| 38    | 9381.0    | 3.41        |
| 39    | 20389.0   | 7.42        |
| 40    | 63314.0   | 23.04       |
| 41    | 48740.0   | 17.73       |
| 42    | 61237.0   | 22.28       |
| 43    | 46767.0   | 17.02       |
| 44    | 74807.0   | 27.22       |
| 45    | 2734.0    | 0.99        |
| 46    | 22154.0   | 8.06        |
| 50    | 4500.0    | 1.64        |
| 51    | 9138.0    | 3.33        |
| 52    | 14642.0   | 5.33        |
| 53    | 21254.0   | 7.73        |
| 54    | 4846.0    | 1.76        |



|     |         |       |
|-----|---------|-------|
| 55  | 15127.0 | 5.50  |
| 56  | 29009.0 | 10.56 |
| 57  | 12185.0 | 4.43  |
| 58  | 3288.0  | 1.20  |
| 59  | 3219.0  | 1.17  |
| 60  | 3877.0  | 1.41  |
| 62  | 3011.0  | 1.10  |
| 66  | 5815.0  | 2.12  |
| 67  | 7096.0  | 2.58  |
| 68  | 8031.0  | 2.92  |
| 69  | 35932.0 | 13.07 |
| 70  | 15335.0 | 5.58  |
| 71  | 2977.0  | 1.08  |
| 73  | 5261.0  | 1.91  |
| 77  | 761.0   | 0.28  |
| 78  | 1176.0  | 0.43  |
| 80  | 5469.0  | 1.99  |
| 81  | 3634.0  | 1.32  |
| 82  | 2561.0  | 0.93  |
| 83  | 1938.0  | 0.71  |
| 84  | 5296.0  | 1.93  |
| 87  | 1038.0  | 0.38  |
| 91  | 1661.0  | 0.60  |
| 93  | 1523.0  | 0.55  |
| 95  | 4880.0  | 1.78  |
| 96  | 1661.0  | 0.60  |
| 97  | 4811.0  | 1.75  |
| 98  | 40571.0 | 14.76 |
| 99  | 1869.0  | 0.68  |
| 100 | 1315.0  | 0.48  |
| 112 | 12635.0 | 4.60  |
| 113 | 49086.0 | 17.86 |
| 114 | 2111.0  | 0.77  |
| 119 | 5919.0  | 2.15  |
| 120 | 2215.0  | 0.81  |
| 127 | 1592.0  | 0.58  |
| 131 | 7442.0  | 2.71  |
| 169 | 3253.0  | 1.18  |
| 181 | 5504.0  | 2.00  |
| 224 | 2007.0  | 0.73  |

**Calculation of elemental compositions:**

Calculated:  $m/z=438.0699$  ( $C_6H_{10}N_{14}O_{10}$ )<sup>+</sup> — not detected.

Measured:  $m/z=224.0763$

| Elemental composition |  | RDB | Error (ppm) |
|-----------------------|--|-----|-------------|
| 1                     | C <sub>6</sub> H <sub>8</sub> O <sub>2</sub> N <sub>8</sub>  | 7.0 | -0.773      |
| 2                     | C <sub>7</sub> H <sub>14</sub> O <sub>7</sub> N <sub>1</sub> | 1.5 | -0.797      |

Calcd for (C<sub>6</sub>H<sub>8</sub>N<sub>8</sub>O<sub>2</sub>)<sup>+</sup>  $m/z=224.0765$

**Range of number values of atoms in elements:**

| Isotope | min | max |
|---------|-----|-----|
| 12 C    | 4   | 10  |
| 1 H     | 4   | 20  |
| 16 O    | 0   | 12  |
| 14 N    | 0   | 20  |

(M)<sup>+</sup> — a positively charged odd-electron molecular radical ion (radical cation).

**Figure S24.** Effect of compound **4** on the burning rate of the KClO<sub>4</sub>/Al pyrotechnic composition

The following formulation was used as the model pyrotechnic composition: 70 % potassium perchlorate (ACS grade, technical specifications No. 6-09-3801-76) and 30 % aluminum (ASD-4 brand, technical specifications No. 48-5-226-87).

Compound **4** was used as the modifier.

| no additive |      |         |      |       |      |      |               |
|-------------|------|---------|------|-------|------|------|---------------|
| l, mm       | t, s | u, mm/s | avr. | disp. | SD   | CI   | half-interval |
| 4.94        | 1.42 | 3.47    | 3.27 | 0.11  | 0.33 | 0.22 | 0.11          |
| 5.14        | 1.53 | 3.35    |      |       |      |      |               |
| 5.00        | 1.65 | 3.04    |      |       |      |      |               |
| 5.14        | 1.52 | 3.38    |      |       |      |      |               |
| 5.20        | 1.64 | 3.17    |      |       |      |      |               |
| 5.10        | 1.45 | 3.51    |      |       |      |      |               |
| 5.00        | 1.85 | 2.71    |      |       |      |      |               |

|      |      |      |  |  |  |  |  |
|------|------|------|--|--|--|--|--|
| 5.00 | 1.67 | 2.99 |  |  |  |  |  |
| 5.14 | 1.34 | 3.83 |  |  |  |  |  |

| 0.5% 4 additive |      |         |      |       |      |      |               |
|-----------------|------|---------|------|-------|------|------|---------------|
| l, mm           | t, s | u, mm/s | avr. | disp. | SD   | CI   | half-interval |
| 5.00            | 1.74 | 2.87    | 2.98 | 0.11  | 0.34 | 0.22 | 0.11          |
| 5.26            | 1.58 | 3.33    |      |       |      |      |               |
| 5.00            | 1.84 | 2.71    |      |       |      |      |               |
| 5.10            | 1.97 | 2.59    |      |       |      |      |               |
| 5.22            | 1.68 | 3.10    |      |       |      |      |               |
| 5.24            | 1.82 | 2.87    |      |       |      |      |               |
| 5.00            | 1.75 | 2.86    |      |       |      |      |               |
| 4.98            | 1.36 | 3.67    |      |       |      |      |               |
| 4.98            | 1.78 | 2.79    |      |       |      |      |               |

| 1% 4 additive |      |         |      |       |      |      |               |
|---------------|------|---------|------|-------|------|------|---------------|
| l, mm         | t, s | u, mm/s | avr. | disp. | SD   | CI   | half-interval |
| 5.12          | 1.81 | 2.83    | 2.87 | 0.22  | 0.47 | 0.34 | 0.17          |
| 5.18          | 1.95 | 2.65    |      |       |      |      |               |
| 5.16          | 1.85 | 2.80    |      |       |      |      |               |
| 5.10          | 1.79 | 2.86    |      |       |      |      |               |
| 5.22          | 1.42 | 3.67    |      |       |      |      |               |
| 5.20          | 2.00 | 2.60    |      |       |      |      |               |
| 5.10          | 1.32 | 3.86    |      |       |      |      |               |
| 5.14          | 1.89 | 2.72    |      |       |      |      |               |
| 5.24          | 2.21 | 2.37    |      |       |      |      |               |

| 1.5% 4 additive |      |         |      |       |      |      |               |
|-----------------|------|---------|------|-------|------|------|---------------|
| l, mm           | t, s | u, mm/s | avr. | disp. | SD   | CI   | half-interval |
| 5.00            | 2.11 | 2.37    | 2.49 | 0.12  | 0.35 | 0.23 | 0.12          |

|      |      |      |  |  |  |  |  |
|------|------|------|--|--|--|--|--|
| 5.10 | 2.21 | 2.30 |  |  |  |  |  |
| 5.10 | 1.51 | 3.37 |  |  |  |  |  |
| 5.12 | 2.15 | 2.38 |  |  |  |  |  |
| 5.22 | 2.00 | 2.62 |  |  |  |  |  |
| 5.00 | 2.02 | 2.48 |  |  |  |  |  |
| 5.24 | 2.38 | 2.20 |  |  |  |  |  |
| 5.22 | 2.32 | 2.25 |  |  |  |  |  |
| 5.10 | 2.08 | 2.45 |  |  |  |  |  |

**Figure S25.** Effect of compound **4** on the burning rate of the Zr/KNO<sub>3</sub> pyrotechnic composition

The following formulation was used as the model pyrotechnic composition: 52 % zirconium (PCZr-1 brand (powdered calciothermic Zr, technical specifications No. 48-4-234-84) and 48 % potassium nitrate (ACS grade, GOST R 4217-77).

Compound **4** was used as the modifier.

| no additive |         |         |       |        |        |               |
|-------------|---------|---------|-------|--------|--------|---------------|
| l, mm       | t, s    | u, mm/s | avr.  | disp.  | SD     | half-interval |
| 4.42        | 0.11864 | 37.26   | 34.10 | 4.0282 | 2.0070 | 1.3908        |
| 4.36        | 0.11967 | 36.43   |       |        |        |               |
| 4.22        | 0.12108 | 34.85   |       |        |        |               |
| 4.38        | 0.12851 | 34.08   |       |        |        |               |
| 4.18        | 0.12901 | 32.40   |       |        |        |               |
| 4.24        | 0.13071 | 32.44   |       |        |        |               |
| 4.42        | 0.14007 | 31.56   |       |        |        |               |
| 4.36        | 0.12910 | 33.77   |       |        |        |               |
| 4.44        | 0.12745 | 34.84   |       |        |        |               |

| 0.5% <b>4</b> additive |         |         |       |        |        |        |               |
|------------------------|---------|---------|-------|--------|--------|--------|---------------|
| l, mm                  | t, s    | u, mm/s | avr.  | disp.  | SD     | CI     | half-interval |
| 4.42                   | 0.14213 | 31.10   | 29.76 | 3.6703 | 1.9158 | 1.4192 | 0.7096        |

|      |         |       |  |  |  |  |  |
|------|---------|-------|--|--|--|--|--|
| 4.36 | 0.14892 | 29.28 |  |  |  |  |  |
| 4.4  | 0.15031 | 29.27 |  |  |  |  |  |
| 4.32 | 0.14517 | 29.76 |  |  |  |  |  |
| 4.42 | 0.14852 | 29.76 |  |  |  |  |  |
| 4.4  | 0.14552 | 30.24 |  |  |  |  |  |
| 4.32 | 0.16523 | 26.15 |  |  |  |  |  |
| 4.28 | 0.14813 | 28.89 |  |  |  |  |  |
| 4.42 | 0.13244 | 33.37 |  |  |  |  |  |

| 1% 4 additive |         |         |       |        |        |        |               |
|---------------|---------|---------|-------|--------|--------|--------|---------------|
| l, mm         | t, s    | u, mm/s | avr.  | disp.  | SD     | CI     | half-interval |
| 4.46          | 0.16520 | 27.00   | 27.30 | 2.6521 | 1.6285 | 1.0640 | 0.5320        |
| 4.24          | 0.15479 | 27.39   |       |        |        |        |               |
| 4.22          | 0.16289 | 25.91   |       |        |        |        |               |
| 4.2           | 0.16851 | 24.92   |       |        |        |        |               |
| 4.32          | 0.14751 | 29.29   |       |        |        |        |               |
| 4.24          | 0.15849 | 26.75   |       |        |        |        |               |
| 4.2           | 0.14223 | 29.53   |       |        |        |        |               |
| 4.4           | 0.15221 | 28.91   |       |        |        |        |               |
| 4.28          | 0.16472 | 25.98   |       |        |        |        |               |

| 1.5% 4 additive |         |         |       |        |        |        |               |
|-----------------|---------|---------|-------|--------|--------|--------|---------------|
| l, mm           | t, d    | u, mm/s | avr.  | disp.  | SD     | CI     | half-interval |
| 4.42            | 0.16952 | 26.07   | 23.79 | 3.9259 | 1.9814 | 1.5854 | 0.7927        |
| 4.24            | 0.18315 | 23.15   |       |        |        |        |               |
| 4.2             | 0.20642 | 20.35   |       |        |        |        |               |
| 4.22            | 0.17852 | 23.64   |       |        |        |        |               |
| 4.32            | 0.17211 | 25.10   |       |        |        |        |               |
| 4.4             | 0.18010 | 24.43   |       |        |        |        |               |
| 4.22            | 0.16958 | 24.89   |       |        |        |        |               |
| 4.44            | 0.16848 | 26.35   |       |        |        |        |               |
| 4.2             | 0.18112 | 23.19   |       |        |        |        |               |