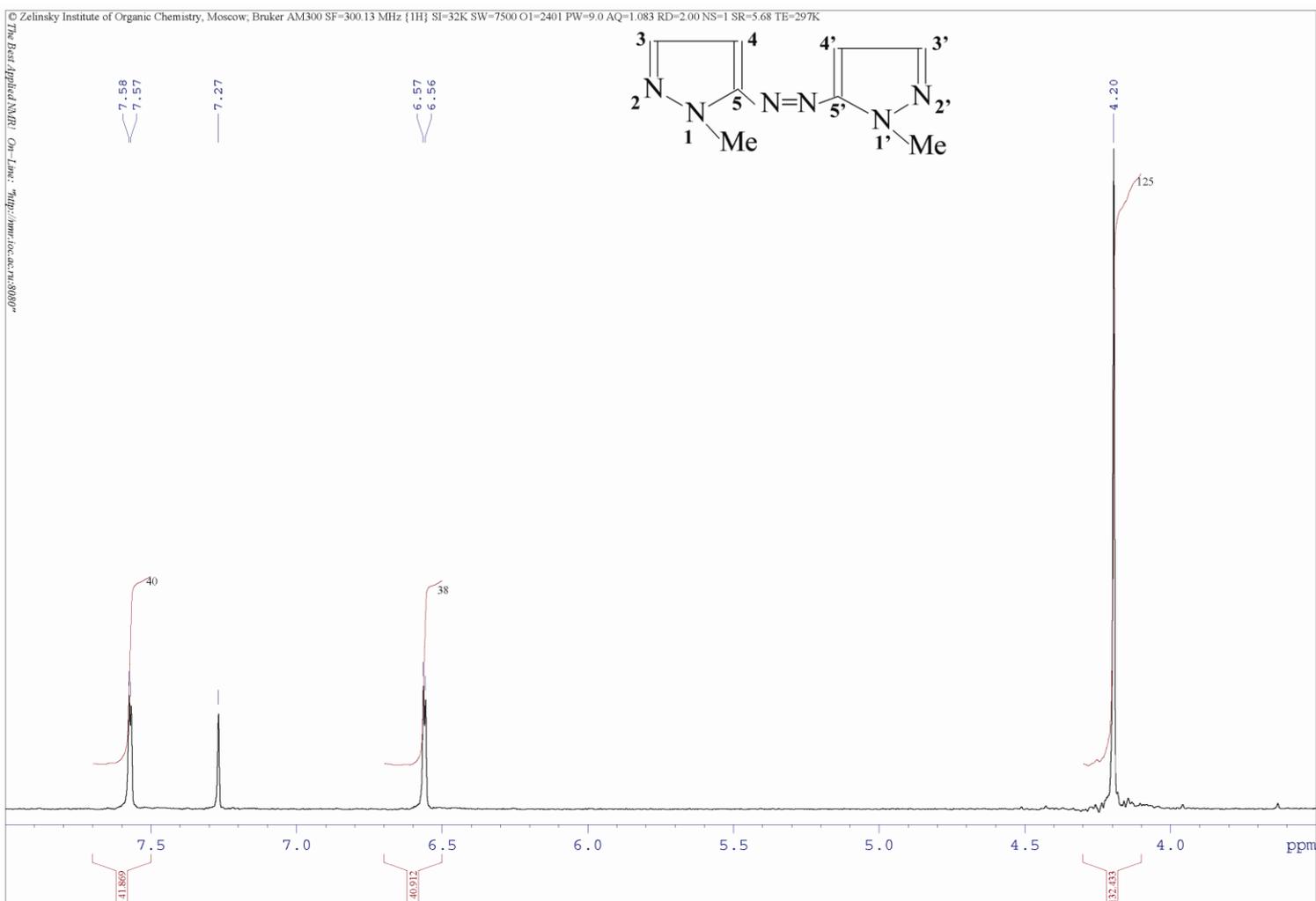


## A new synthesis of azopyrazoles by oxidation of *C*-aminopyrazoles on a NiO(OH) electrode

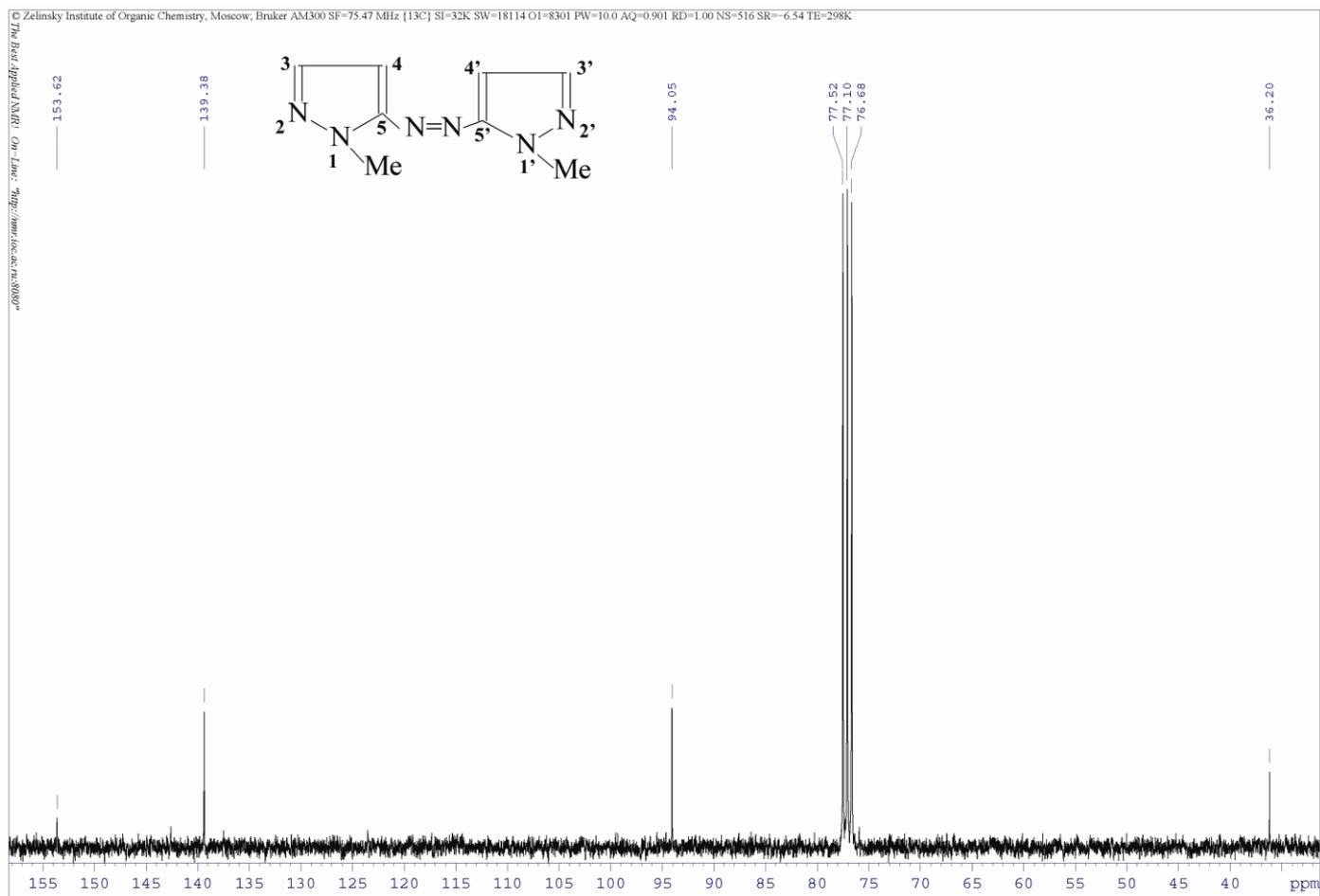
Boris V. Lyalin, Vera L. Sigacheva, Vladimir A. Kokorekin and Vladimir A. Petrosyan

Spectral data for (*E*)-1,2-bis(1-methyl-1H-pyrazol-5-yl)diazene **2a**

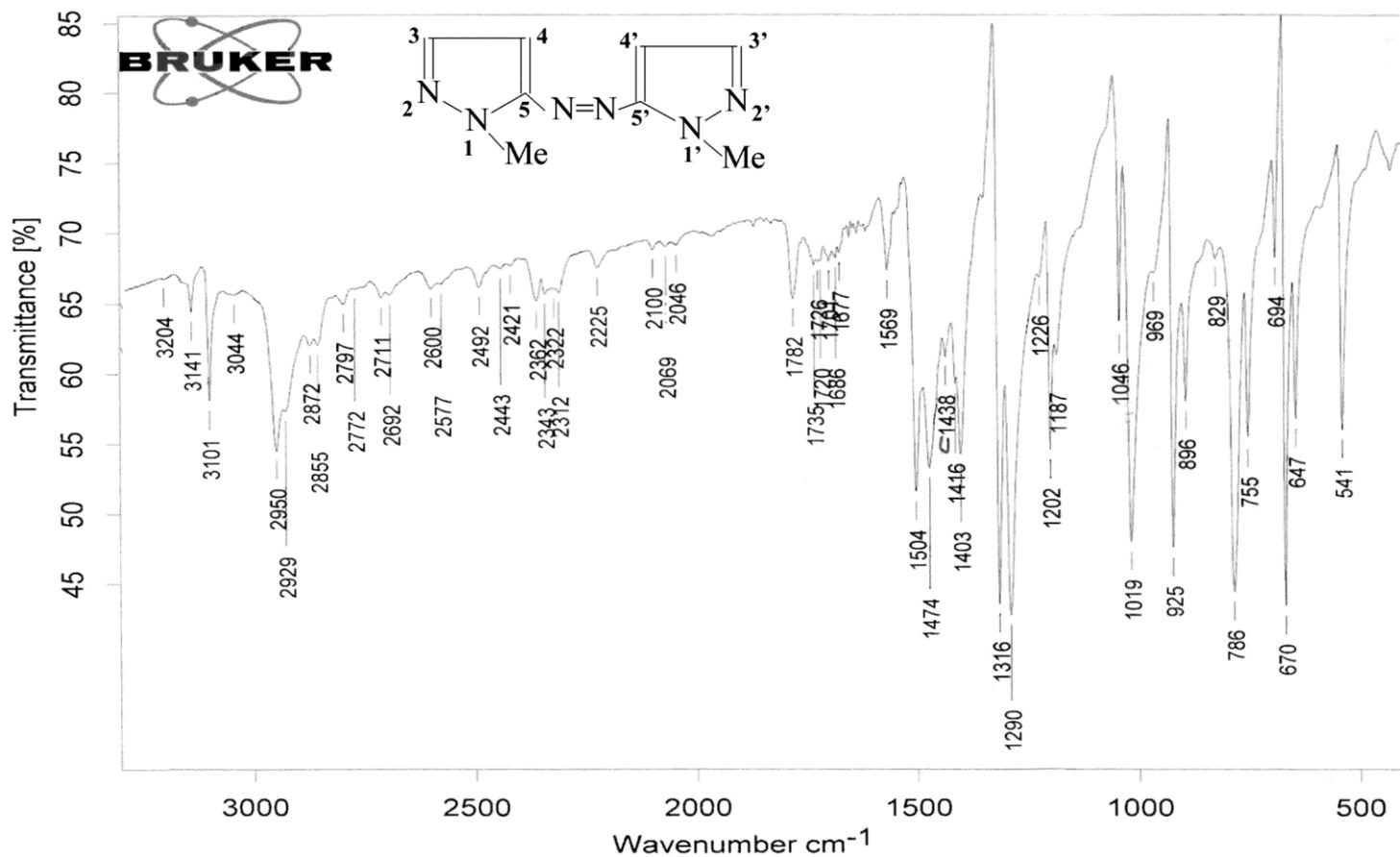
$^1\text{H}$  NMR (300.13 MHz,  $\text{CDCl}_3$ )  $\delta$ : 4.20 (s, 6H,  $\text{N}^1\text{CH}_3$ ,  $\text{N}^{1'}\text{CH}_3$ ), 6.56 (d, 2H,  $\text{C}^4\text{H}$ ,  $\text{C}^{4'}\text{H}$ ,  $J_{4-3}$  2.1 Hz), 7.57 (d, 2H,  $\text{C}^3\text{H}$ ,  $\text{C}^{3'}\text{H}$ ,  $J_{3-4}$  2.1 Hz)



$^{13}\text{C}$  NMR (75.47 MHz,  $\text{CDCl}_3$ )  $\delta$ : 36.20 ( $\text{N}^1\text{CH}_3$ ,  $\text{N}^{1'}\text{CH}_3$ ), 94.05 ( $\text{C}^4\text{H}$ ,  $\text{C}^{4'}\text{H}$ ), 139.38 ( $\text{C}^3\text{H}$ ,  $\text{C}^{3'}\text{H}$ ), 153.62 ( $\text{C}^5$ ,  $\text{C}^{5'}$ )



IR (KBr,  $\nu$ ,  $\text{cm}^{-1}$ ): 3101, 2955, 2492, 2362, 1782, 1735, 1569, 1504, 1474, 1403, 1316, 1290, 1202, 1046, 925, 896, 829, 786, 755, 694, 670, 541

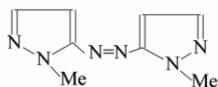


HRMS (ESI) m/z calcd for C<sub>8</sub>H<sub>11</sub>N<sub>6</sub> 191.1040 [M + H]<sup>+</sup>, found 191.1042.

## Display Report

### Analysis Info

Analysis Name D:\Data\Kolotyrkina\2015\Sigacheva\0528036.d  
Method tune\_low.m  
Sample Name /VAPP SIGmass013  
Comment C8H10N6 mw 190 clb added

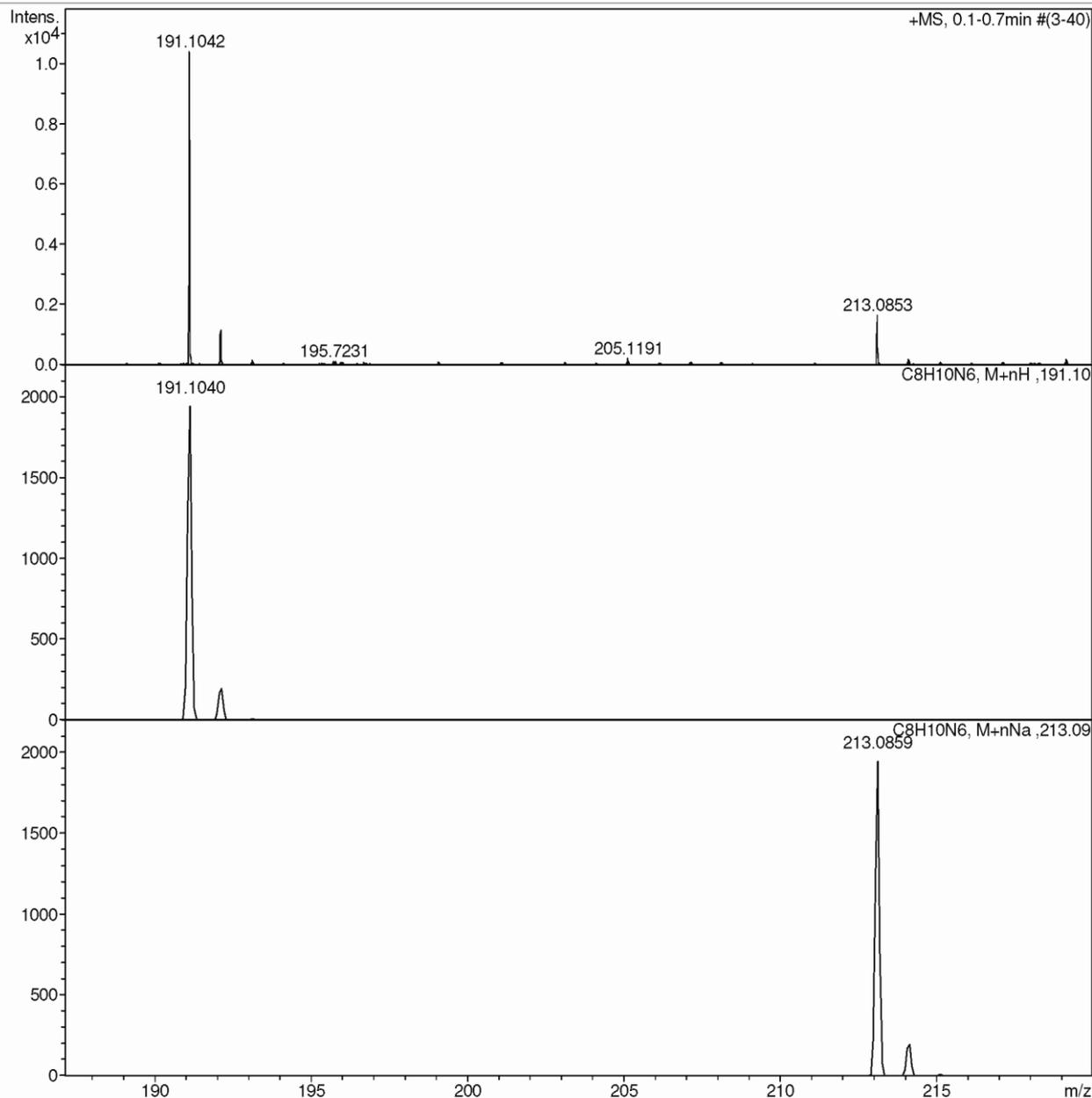


Acquisition Date 28.05.2015 16:42:09

Operator BDAL@DE  
Instrument / Ser# micrOTOF 10248

### 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



Elemental analysis:

Calc. for C<sub>8</sub>H<sub>10</sub>N<sub>6</sub> (%): C, 50.57; H, 5.26; N, 44.20;

Found (%): C, 50.97; H, 5.43; N, 43.45.