

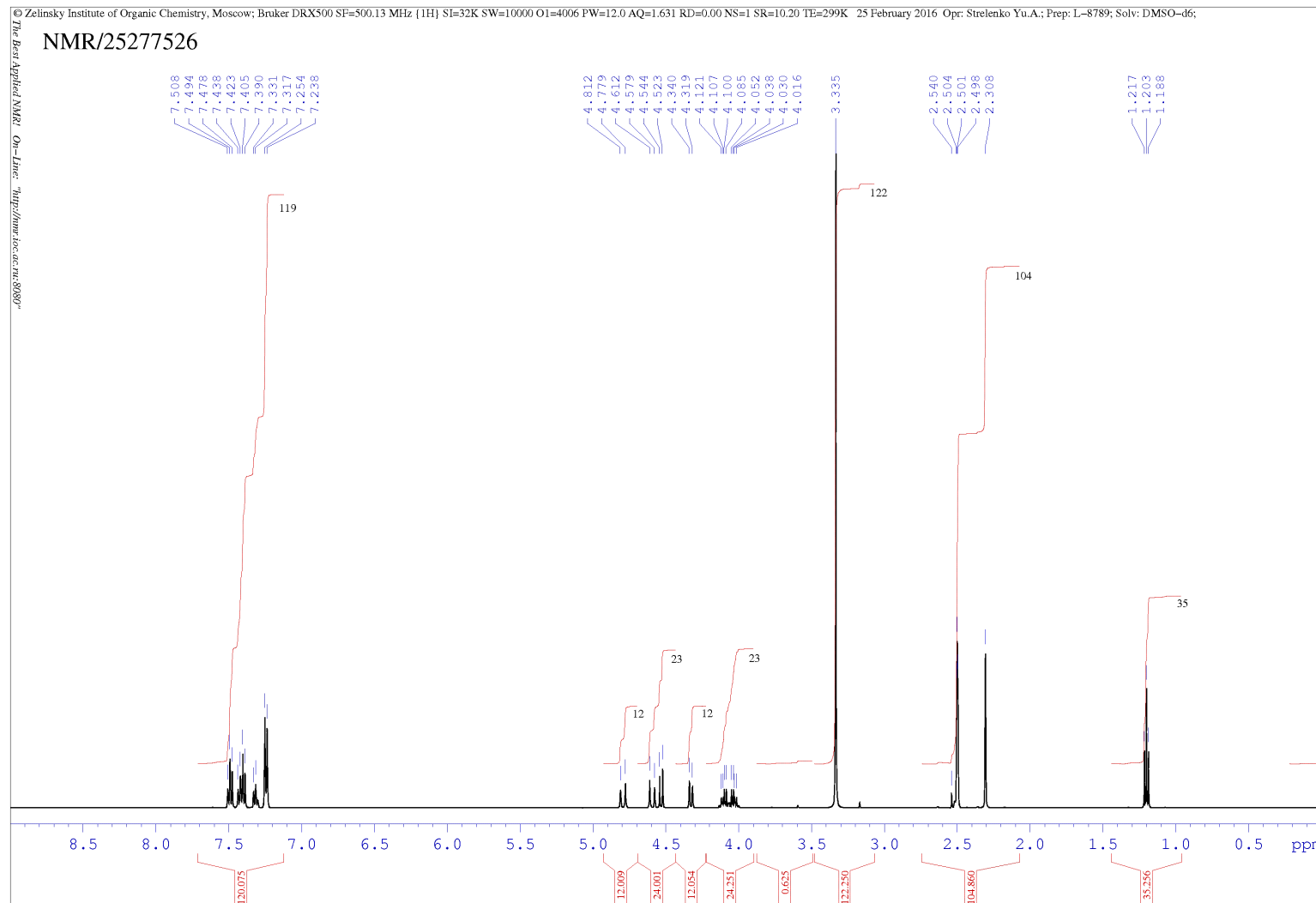
**Supplementary Materials:**

**A new synthetic route to polyhydrogenated pyrrolo[3,4-*b*]pyrroles by the domino reaction of 3-bromopyrrole-2,5-diones with aminocrotonic acid esters**

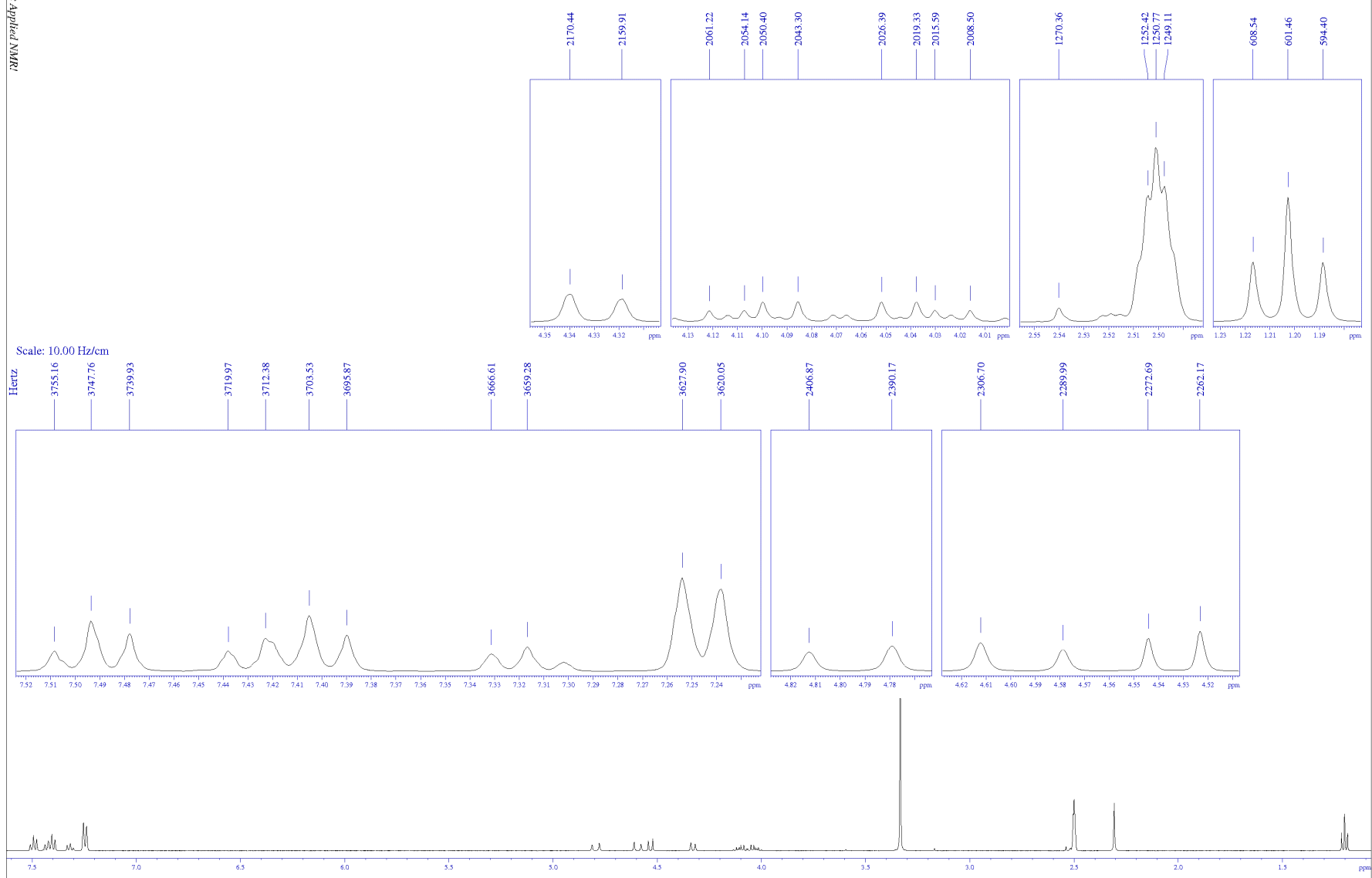
Khidmet Shikhaliev, Artem Sabynin, Valeri Sekirin, Michael Krysin, Fedor Zubkov and Kristina Yankina

## $^1\text{H}$ , $^{13}\text{C}$ NMR spectra and data of HPLC-MS-ESI analysis of pyrrolopyrroles 8

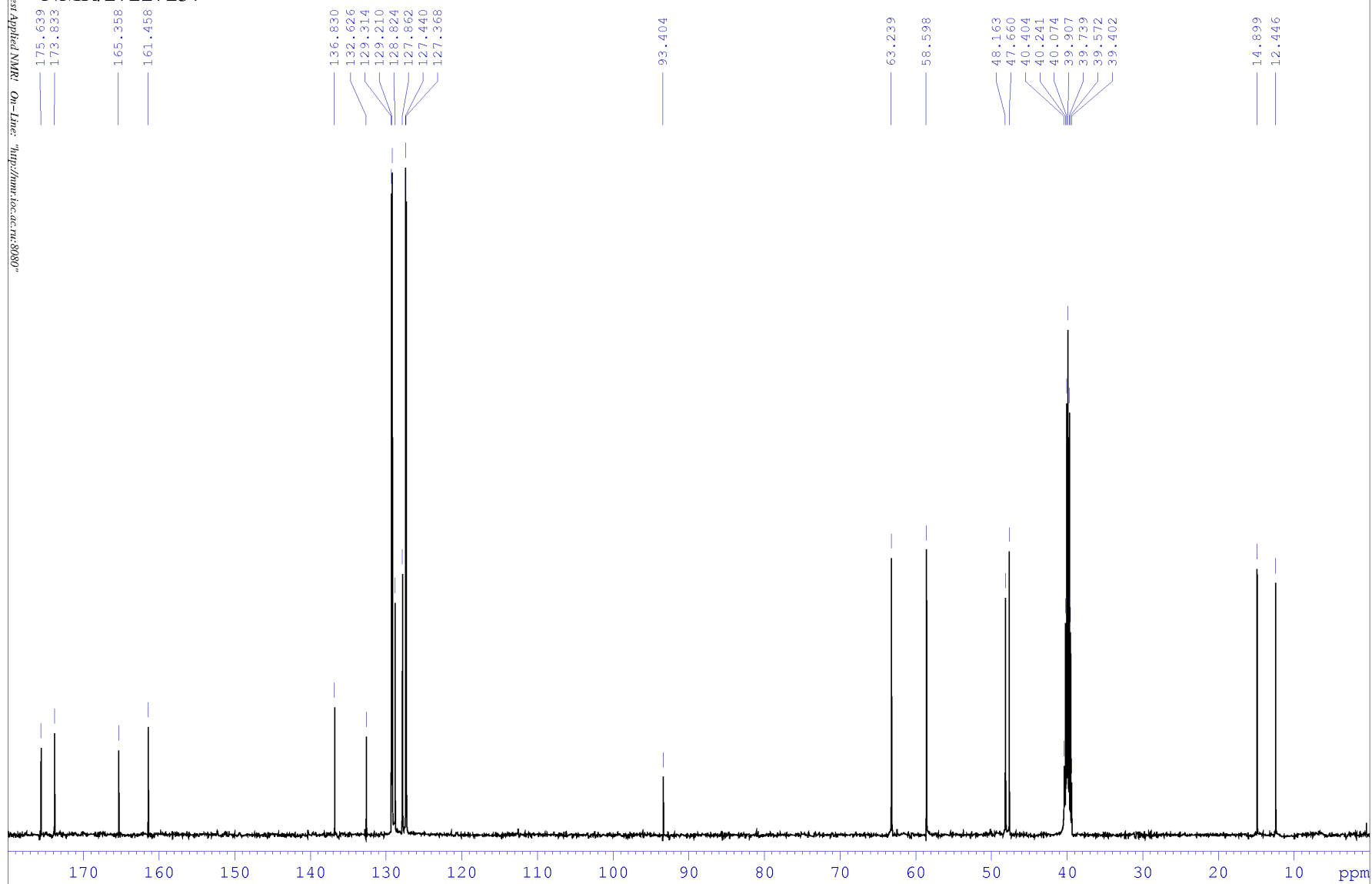
### (3a*S*,6a*R*)-Ethyl 1-benzyl-2-methyl-4,6-dioxo-5-phenyl-1,3a,4,5,6,6a-hexahydropyrrolo[3,4-*b*]pyrrole-3-carboxylate 8a

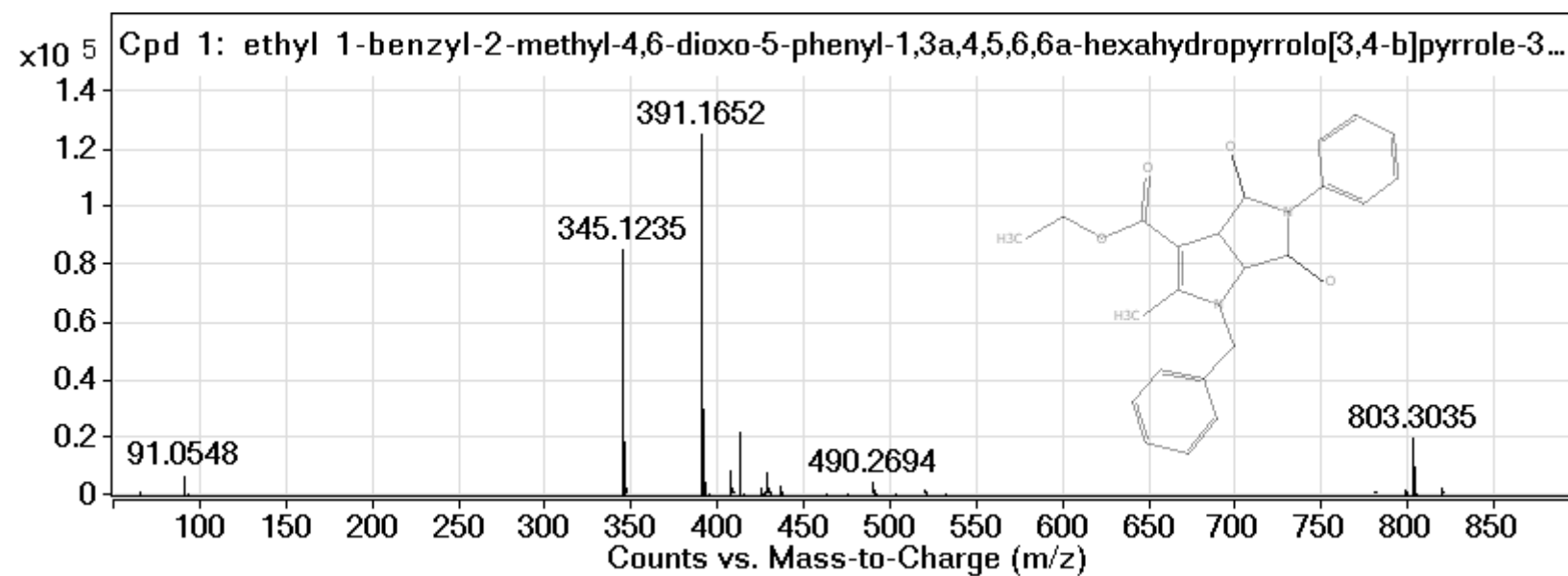
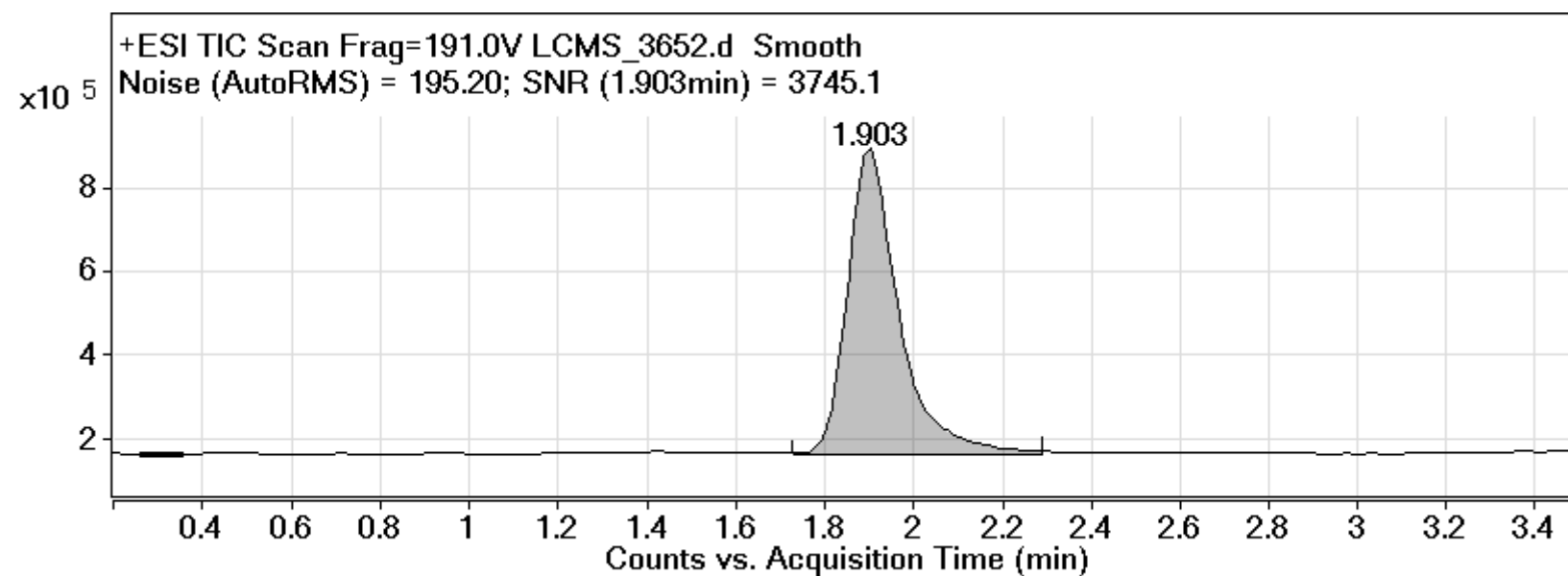


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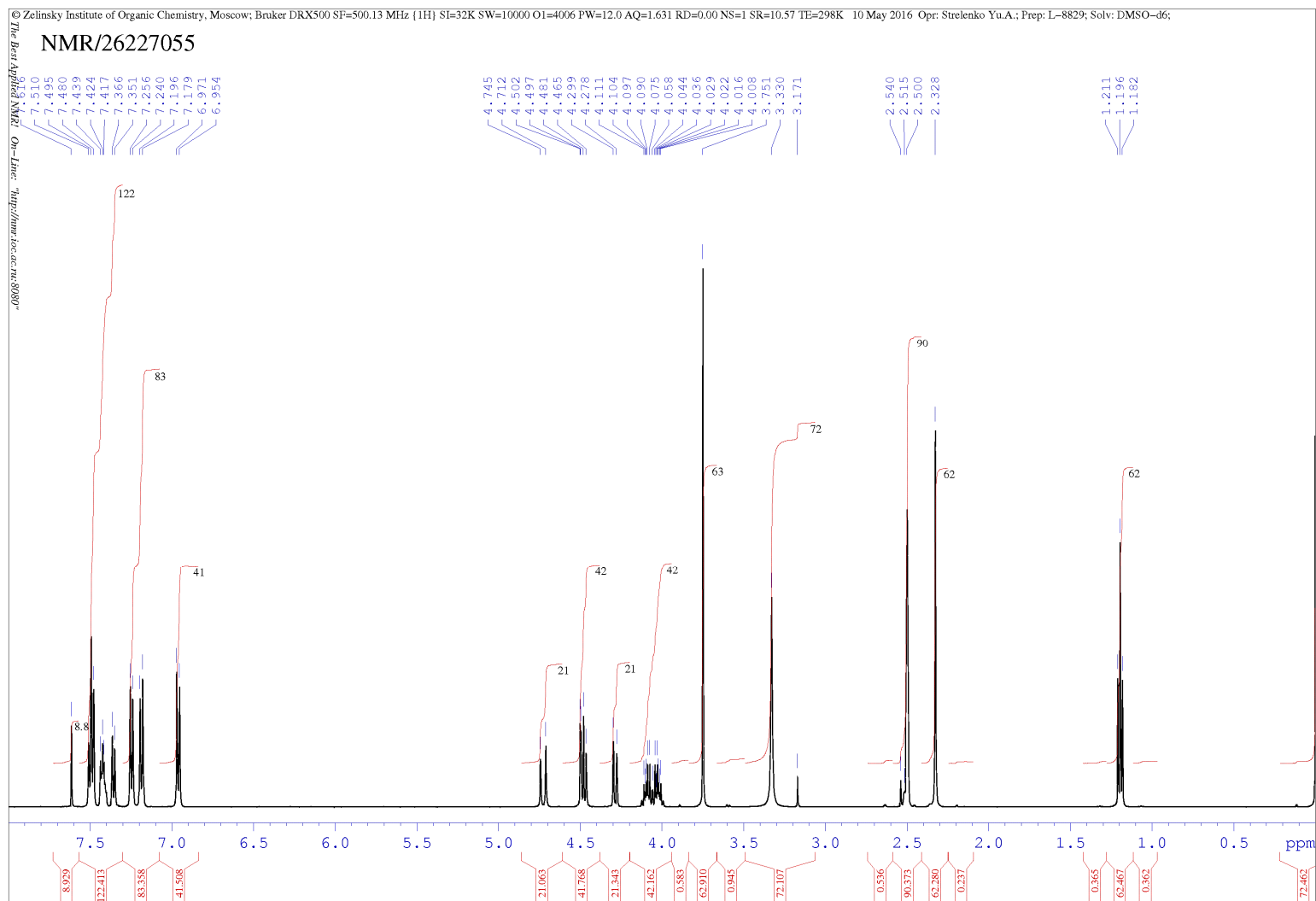


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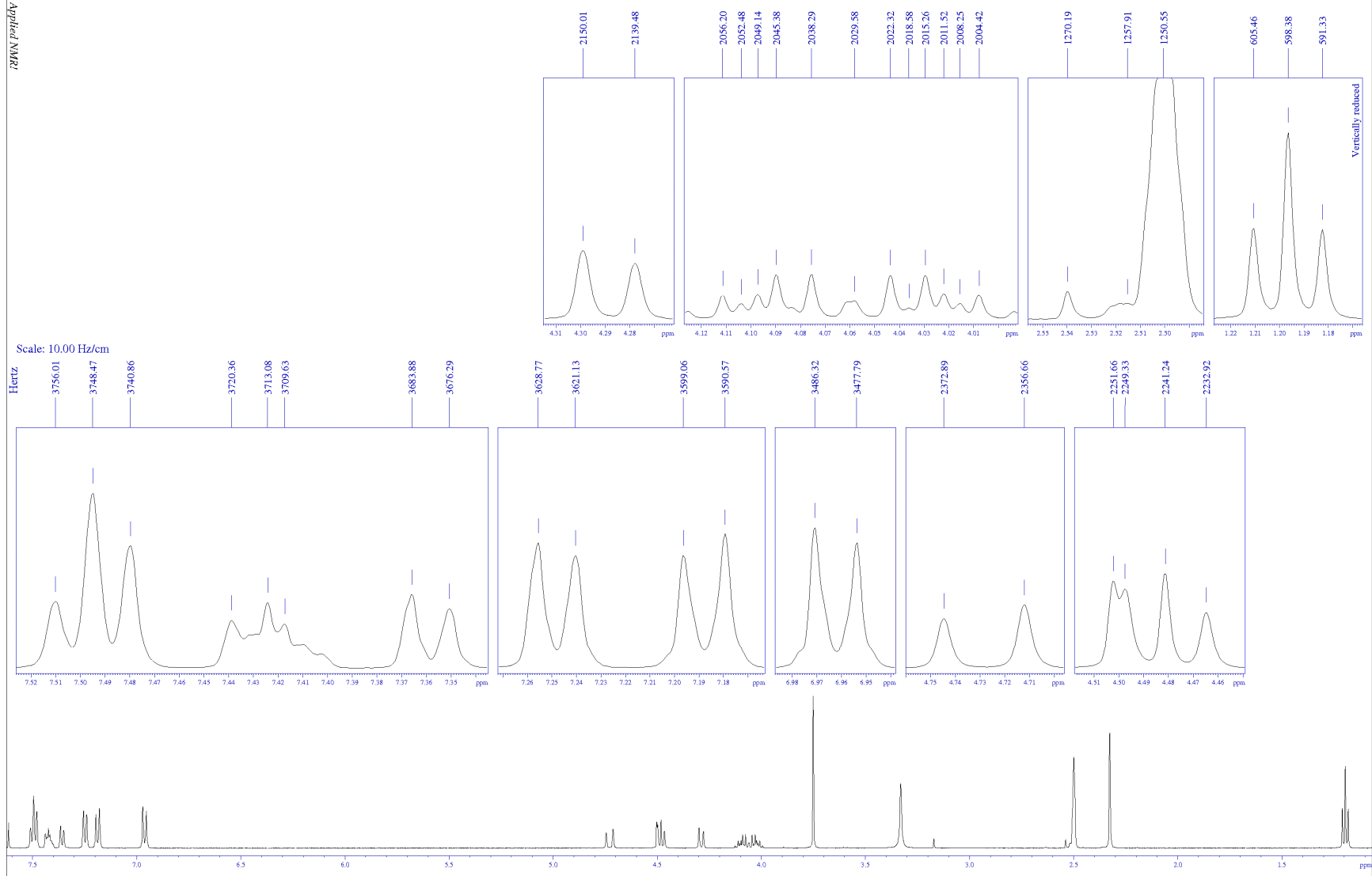




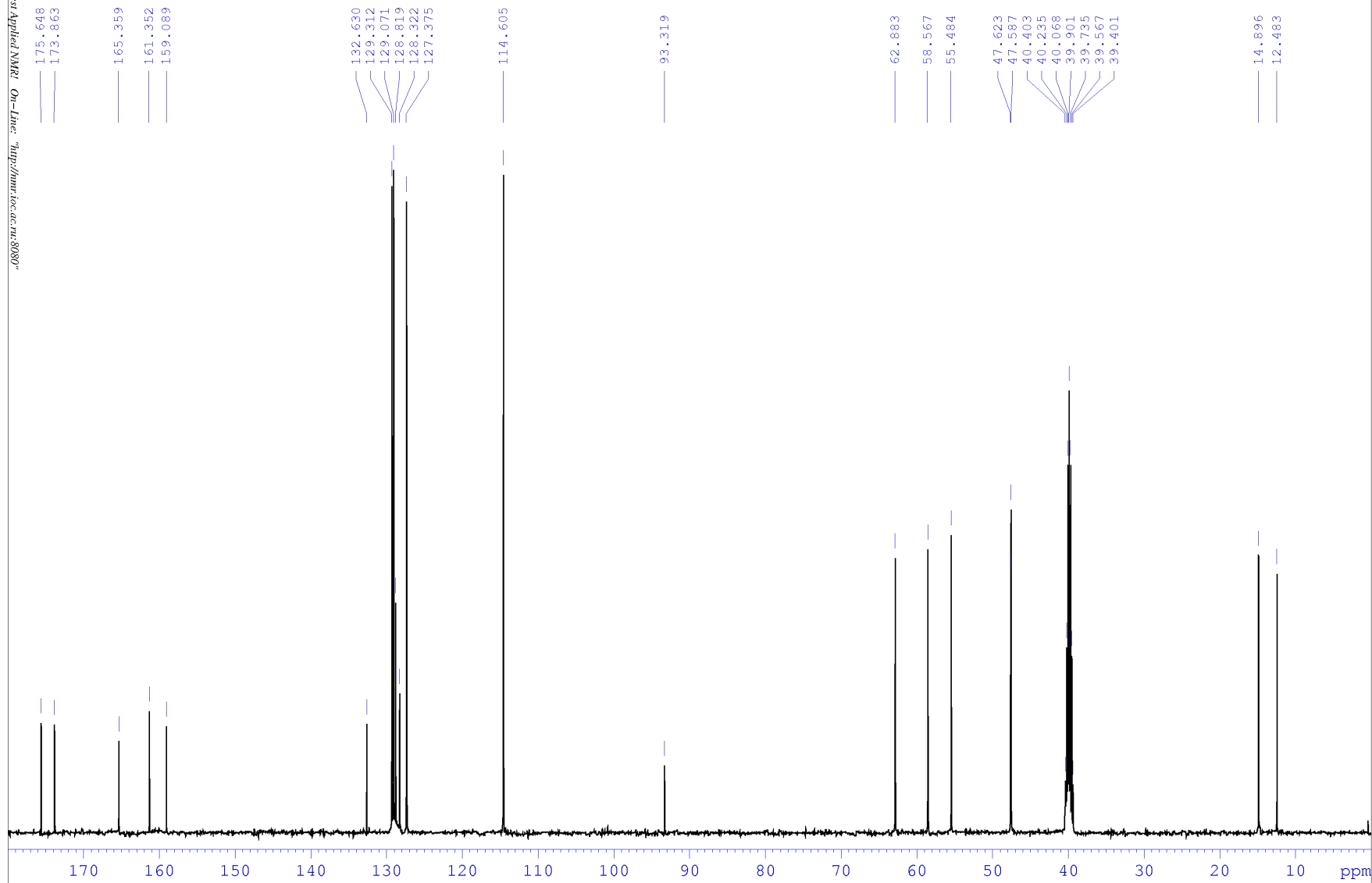
**(3a*S*,6a*R*)-Ethyl 1-methoxybenzyl-2-methyl-4,6-dioxo-5-phenyl-1,3a,4,5,6,6a-hexahydropyrrolo[3,4-*b*]pyrrole-3-carboxylate 8b**



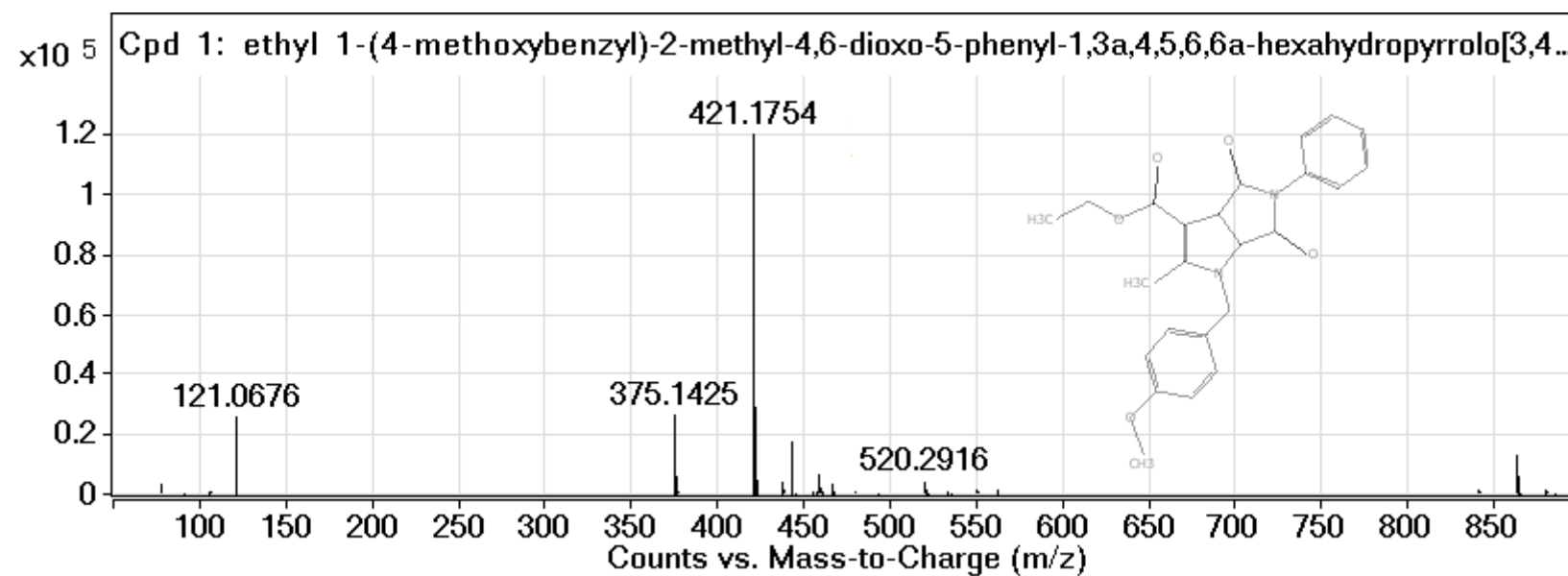
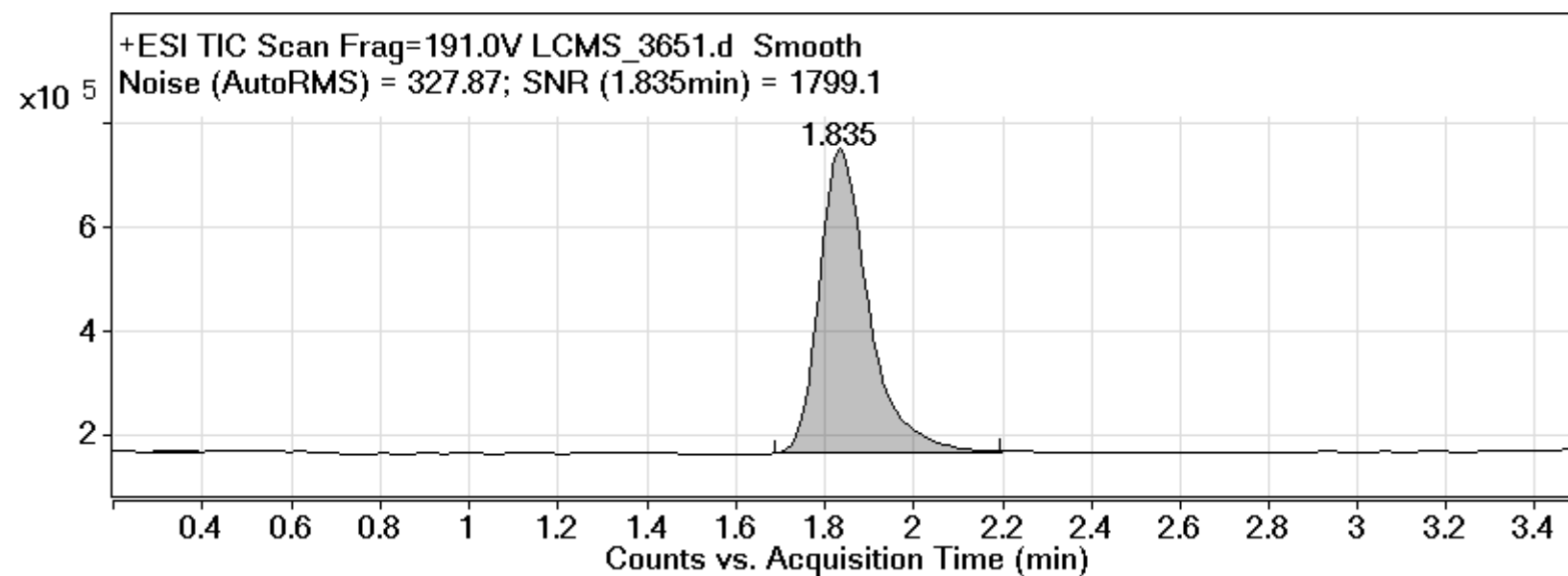
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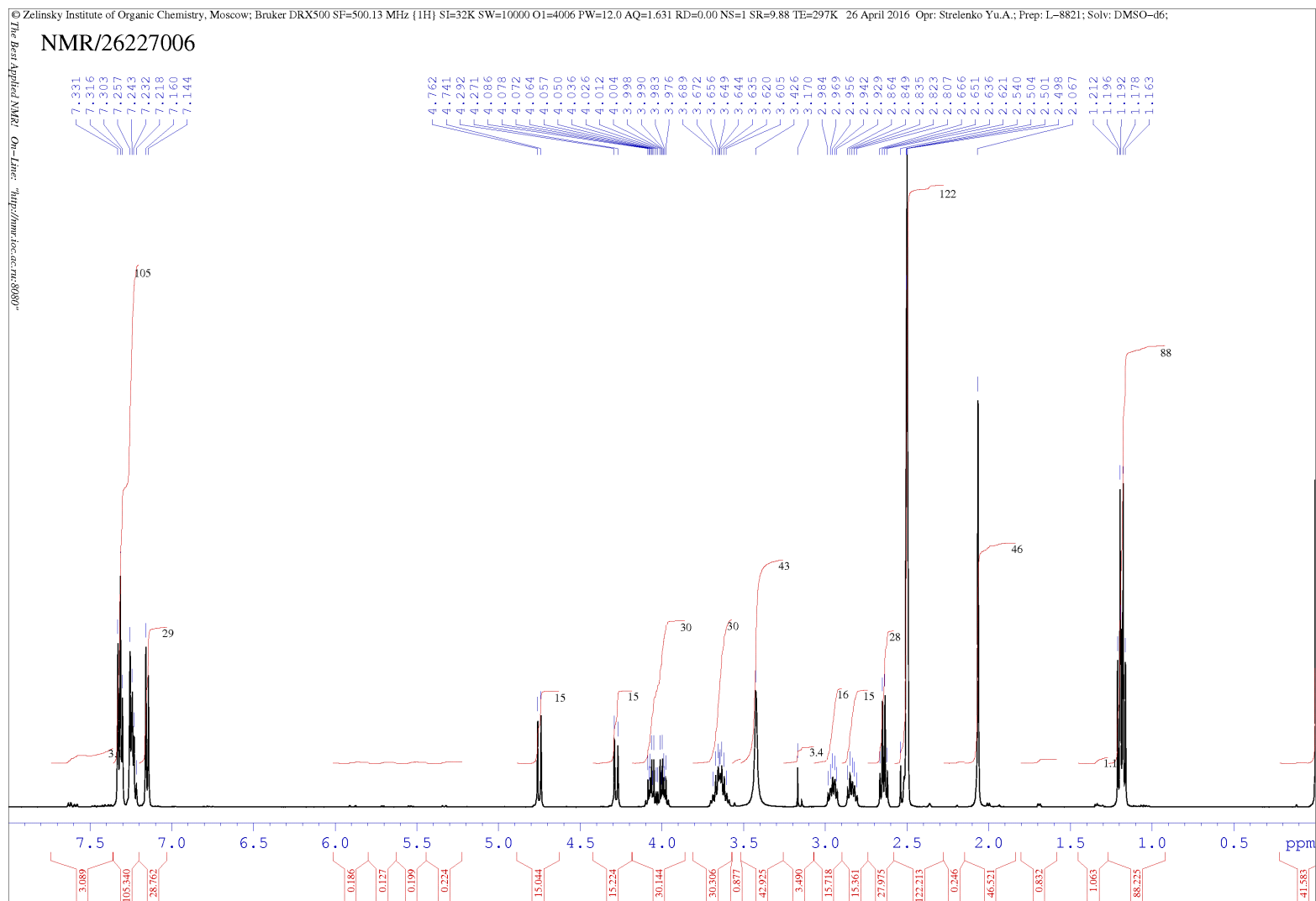
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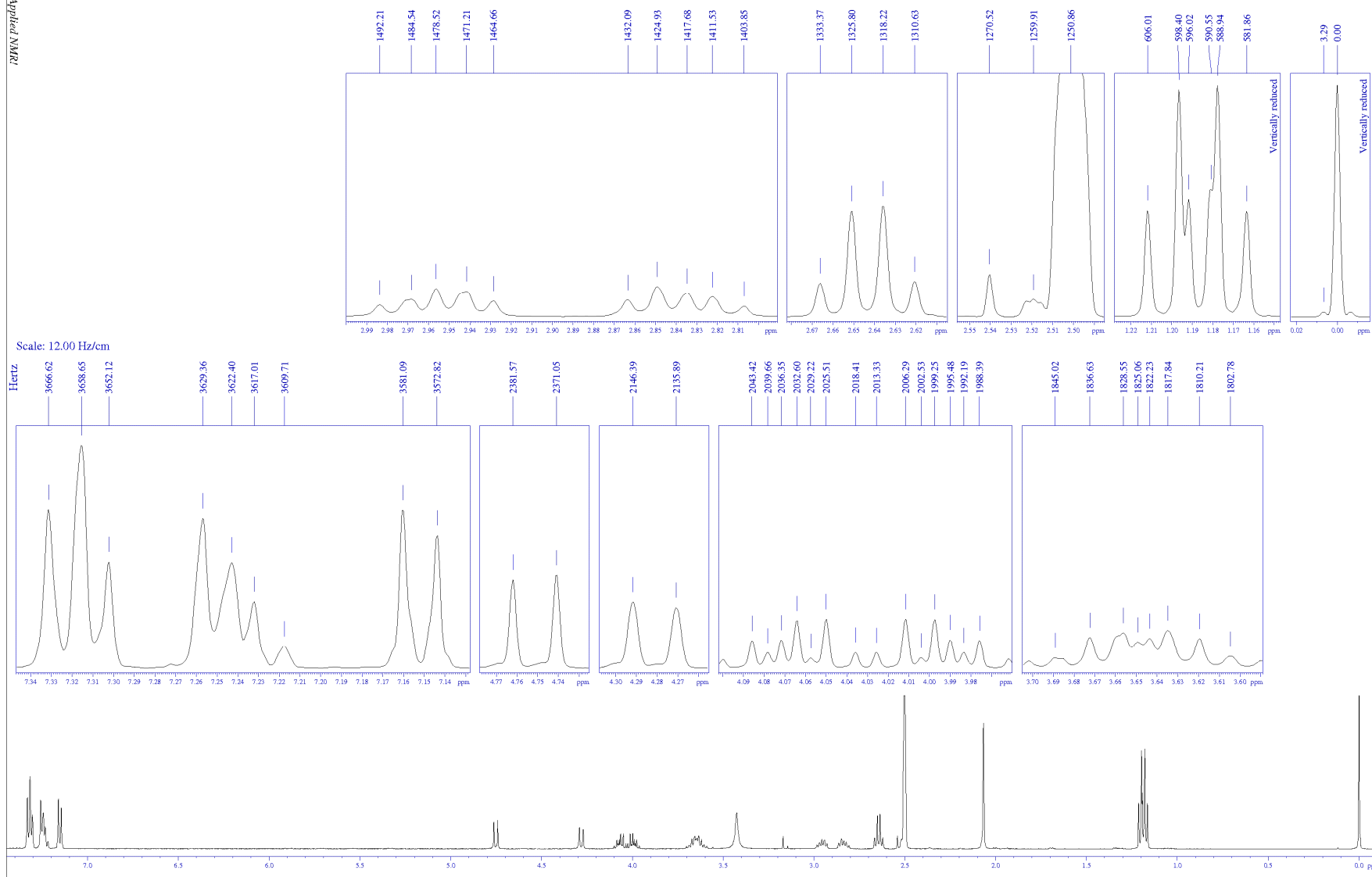


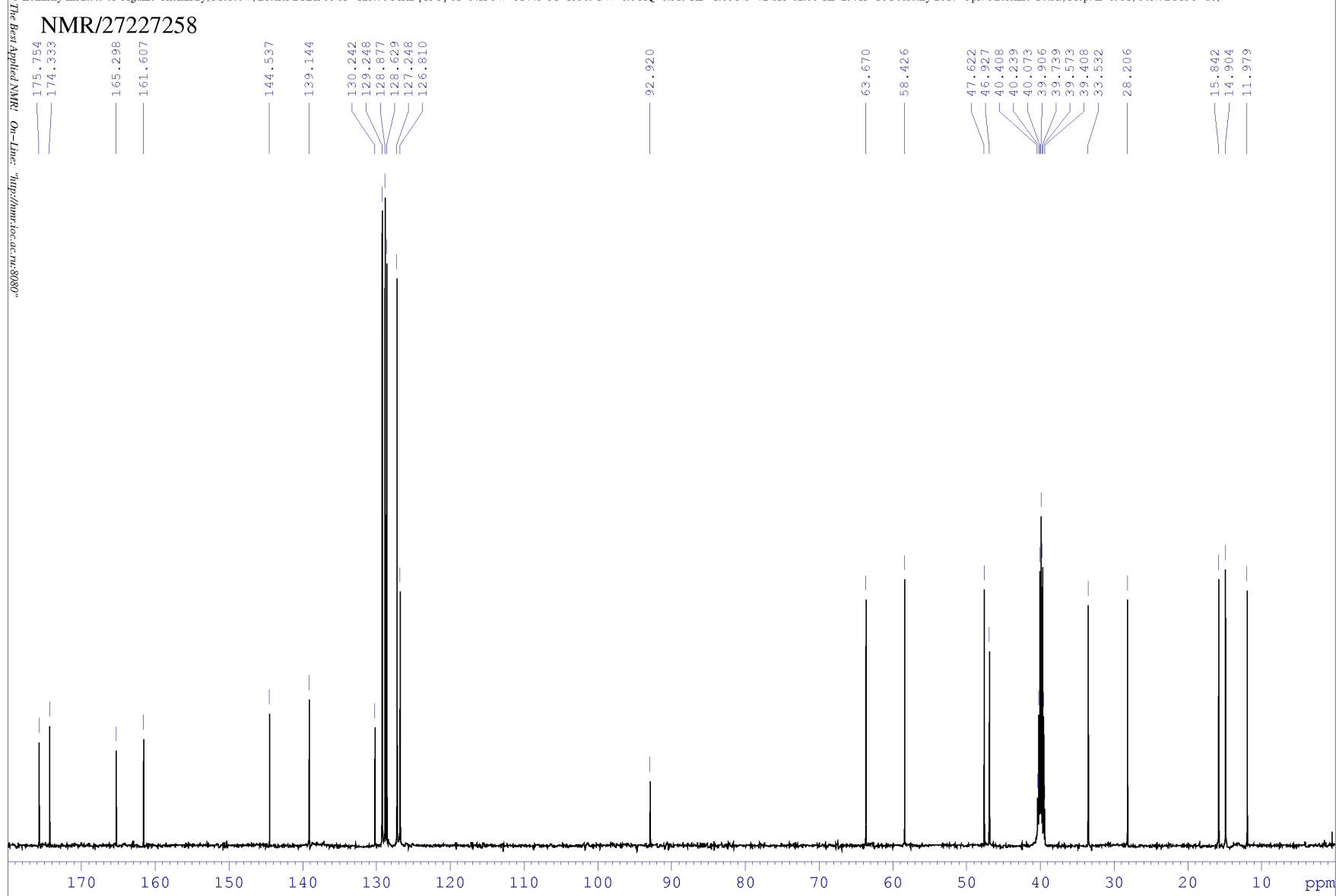


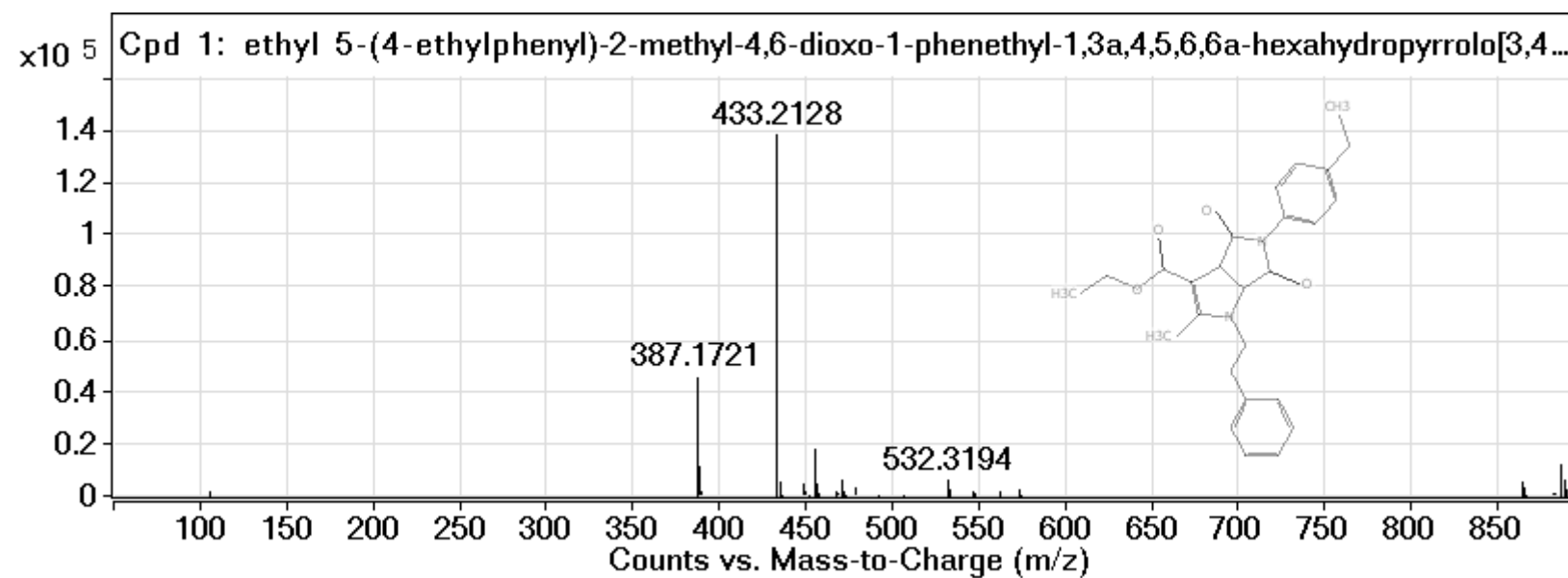
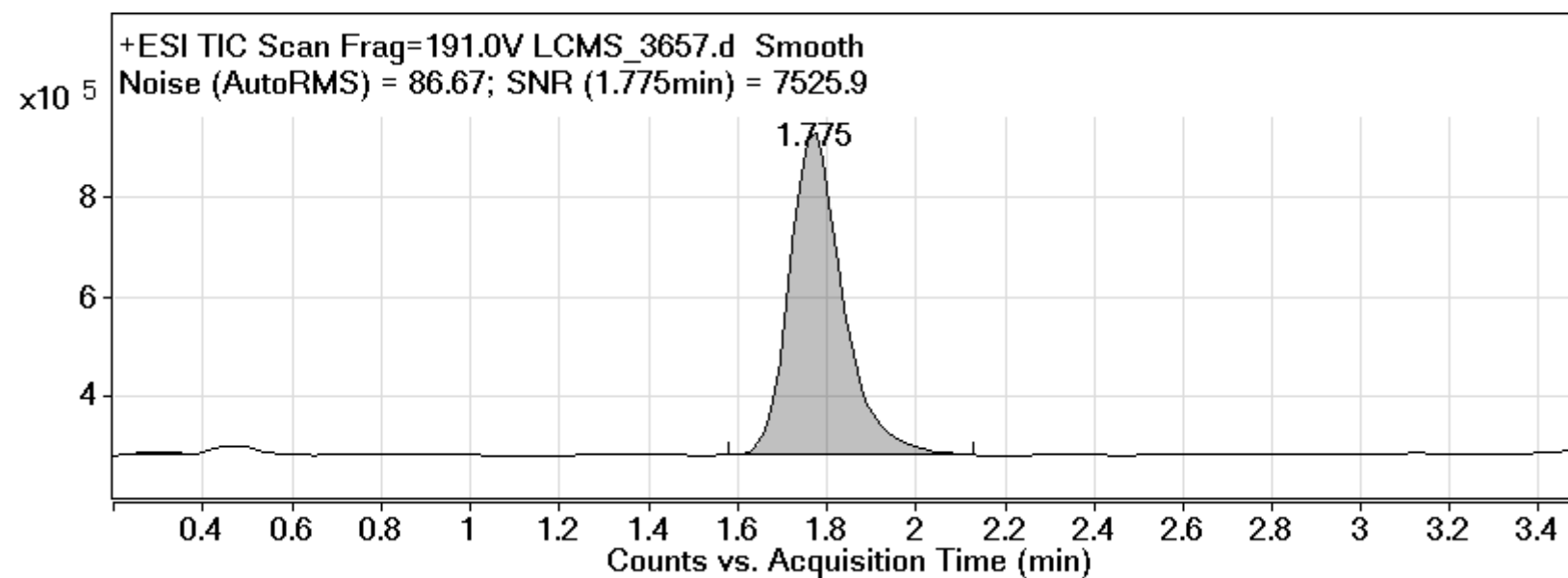
**(3a*S*,6a*R*)-Ethyl 5-(4-ethylphenyl)-2-methyl-4,6-dioxo-1-phenethyl-1,3a,4,5,6,6a-hexahydropyrrolo[3,4-*b*]pyrrole-3-carboxylate 8c**



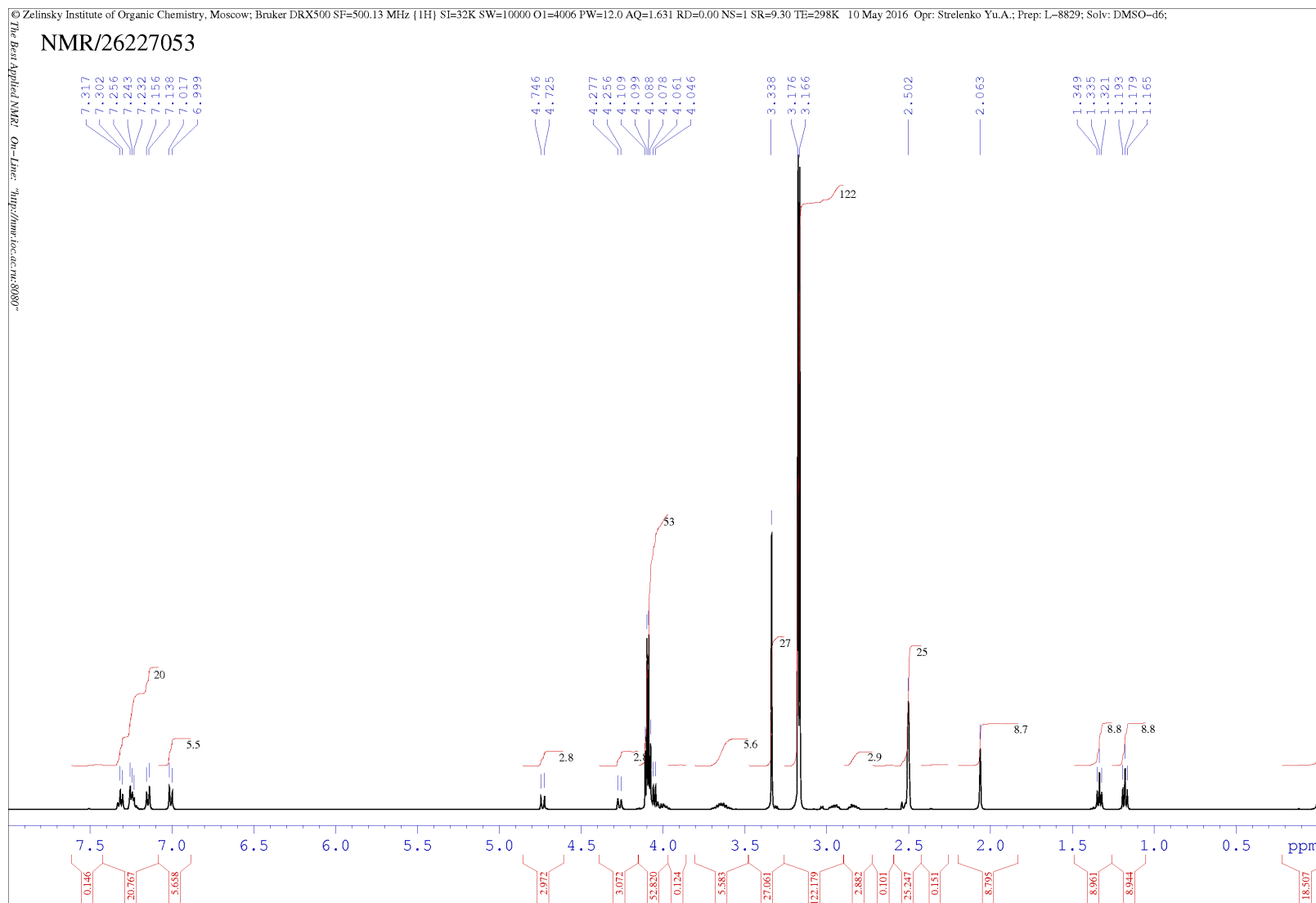
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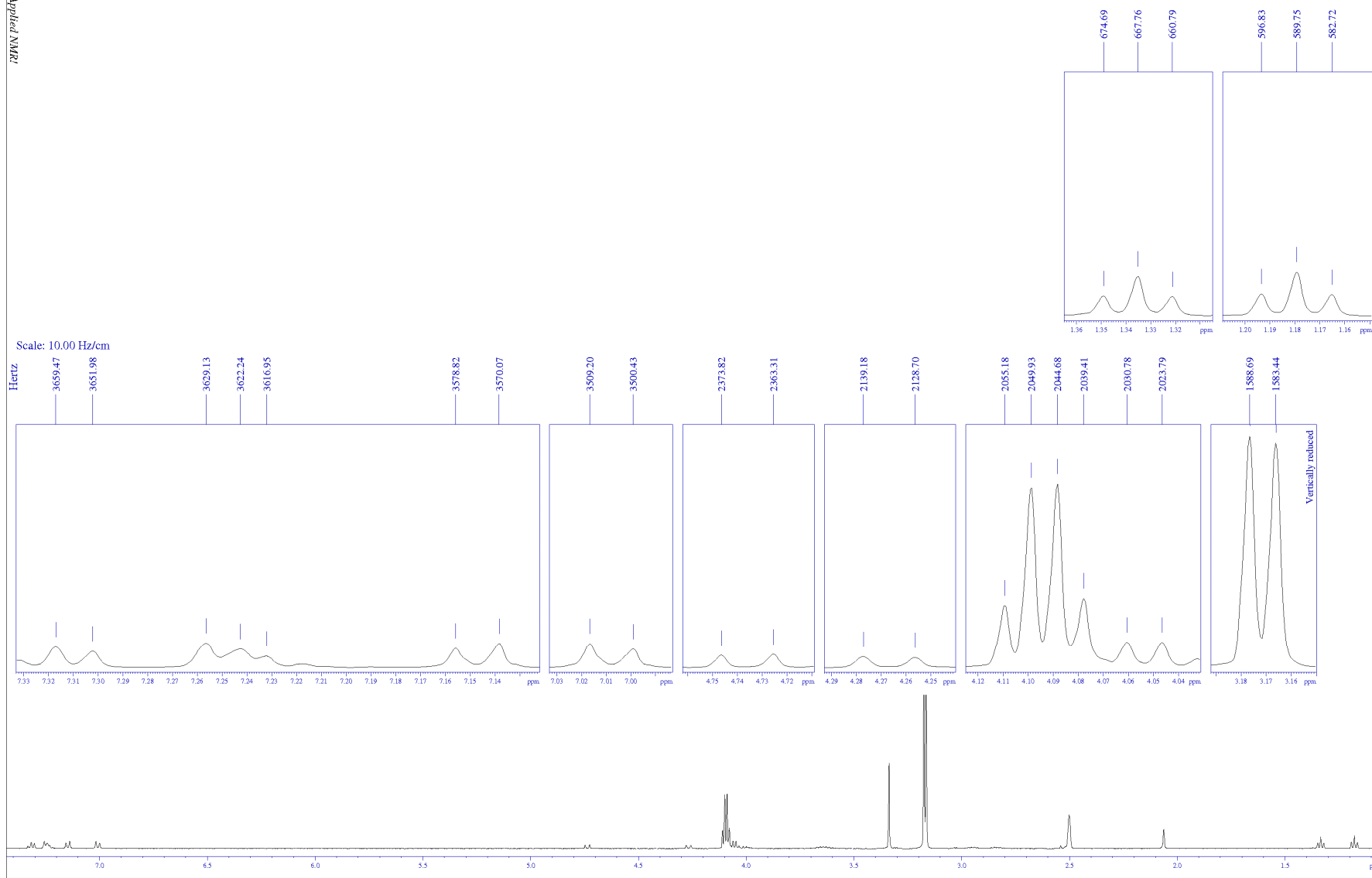


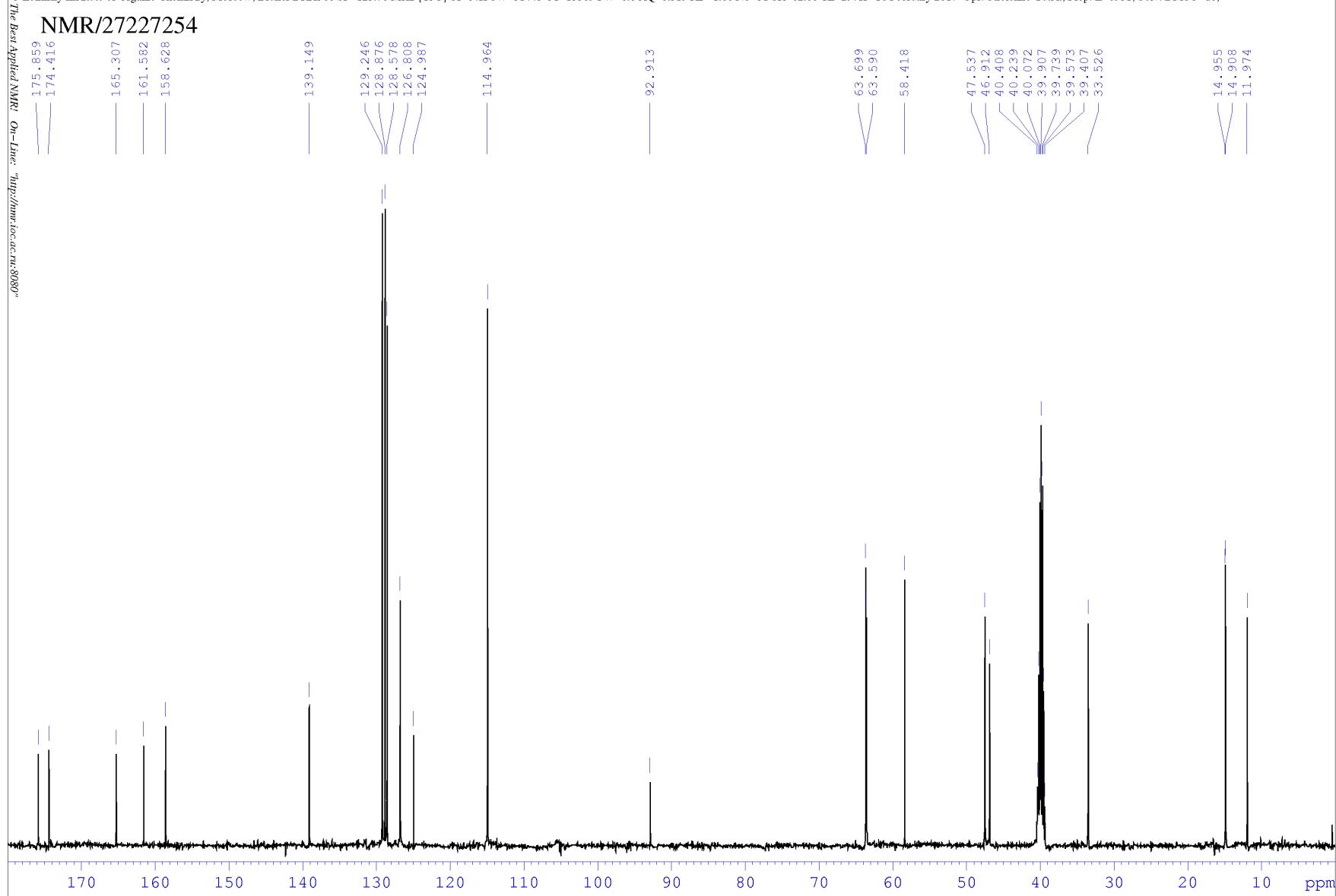


**(3a*S*,6a*R*)-Ethyl 5-(4-ethoxyphenyl)-2-methyl-4,6-dioxo-1-phenetyl-1,3a,4,5,6,6a-hexahydropyrrolo[3,4-*b*]pyrrole-3-carboxylate 8d**

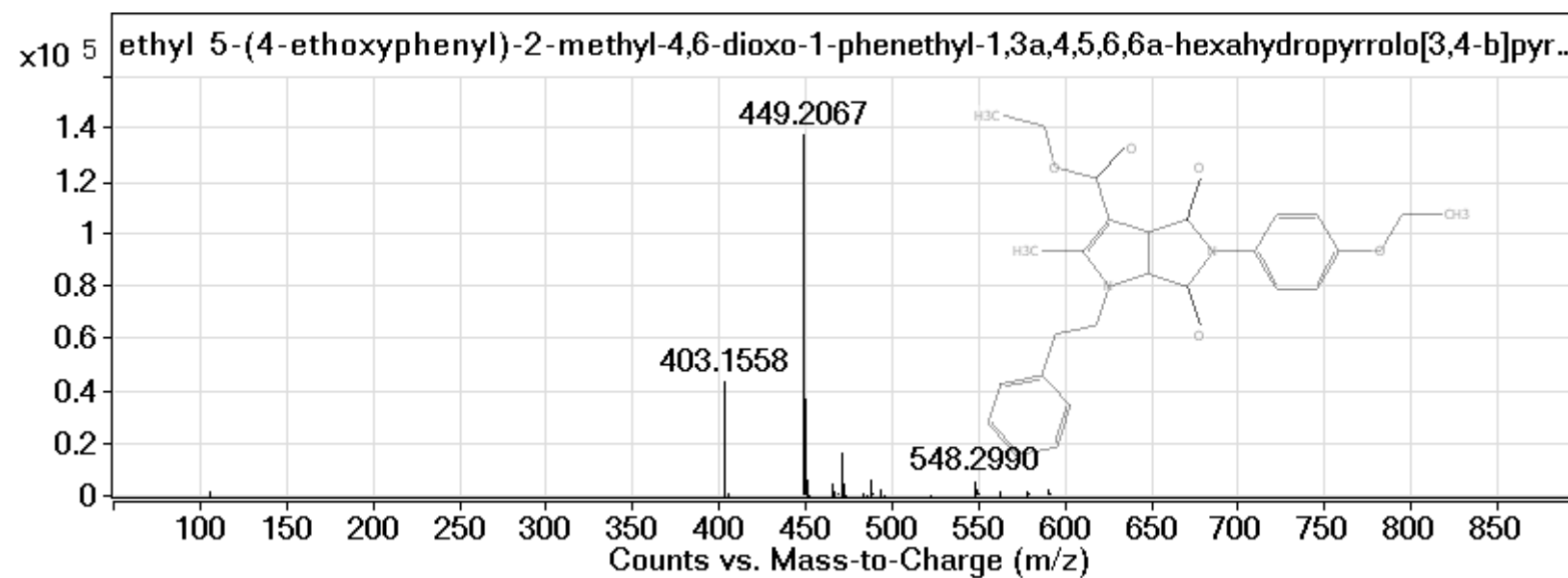
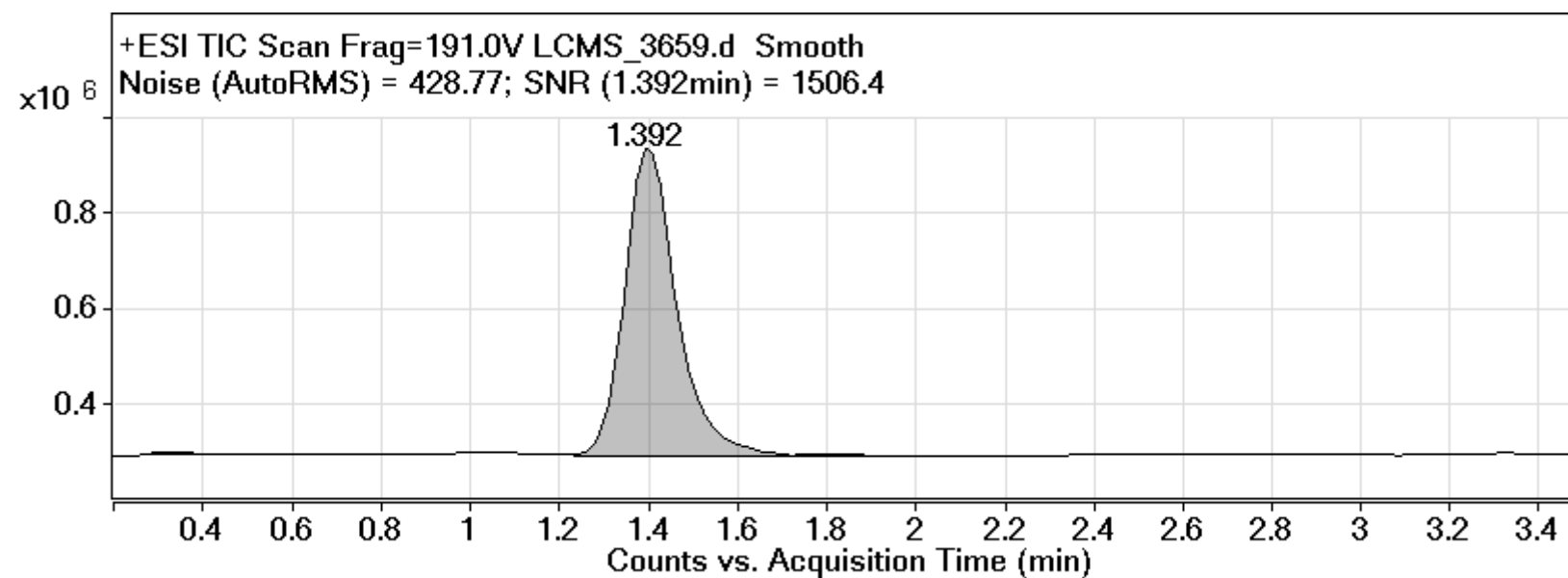


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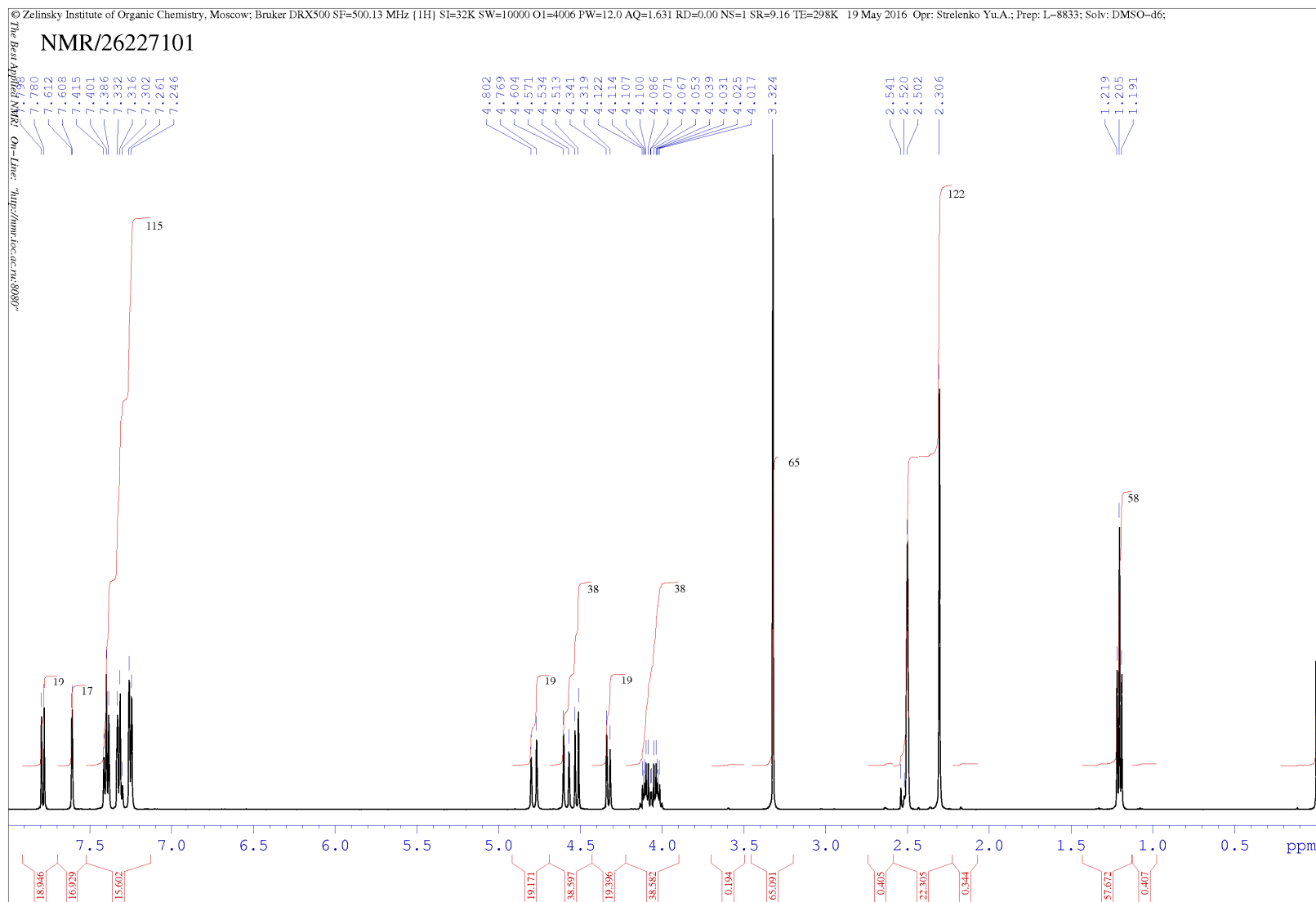




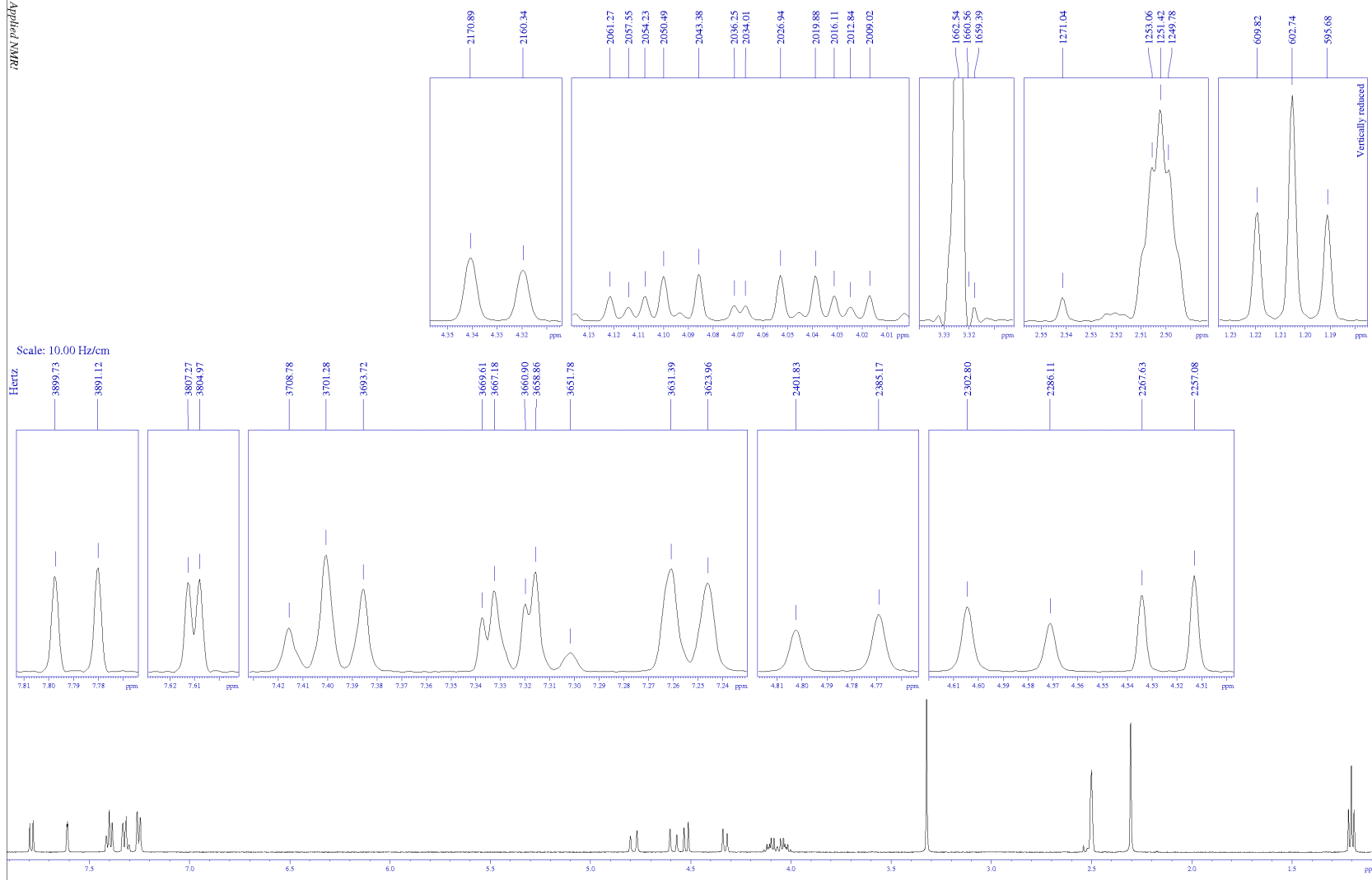


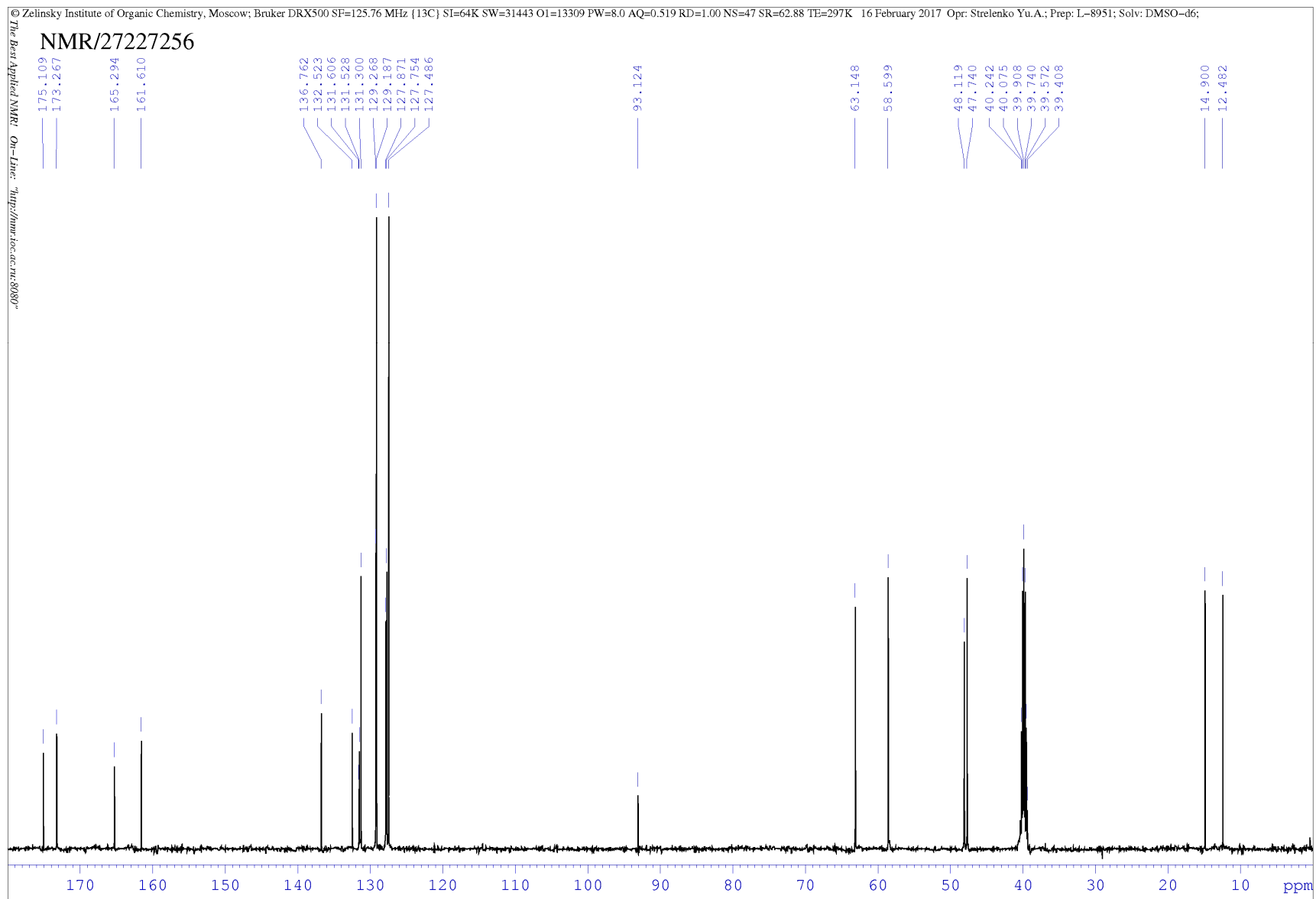


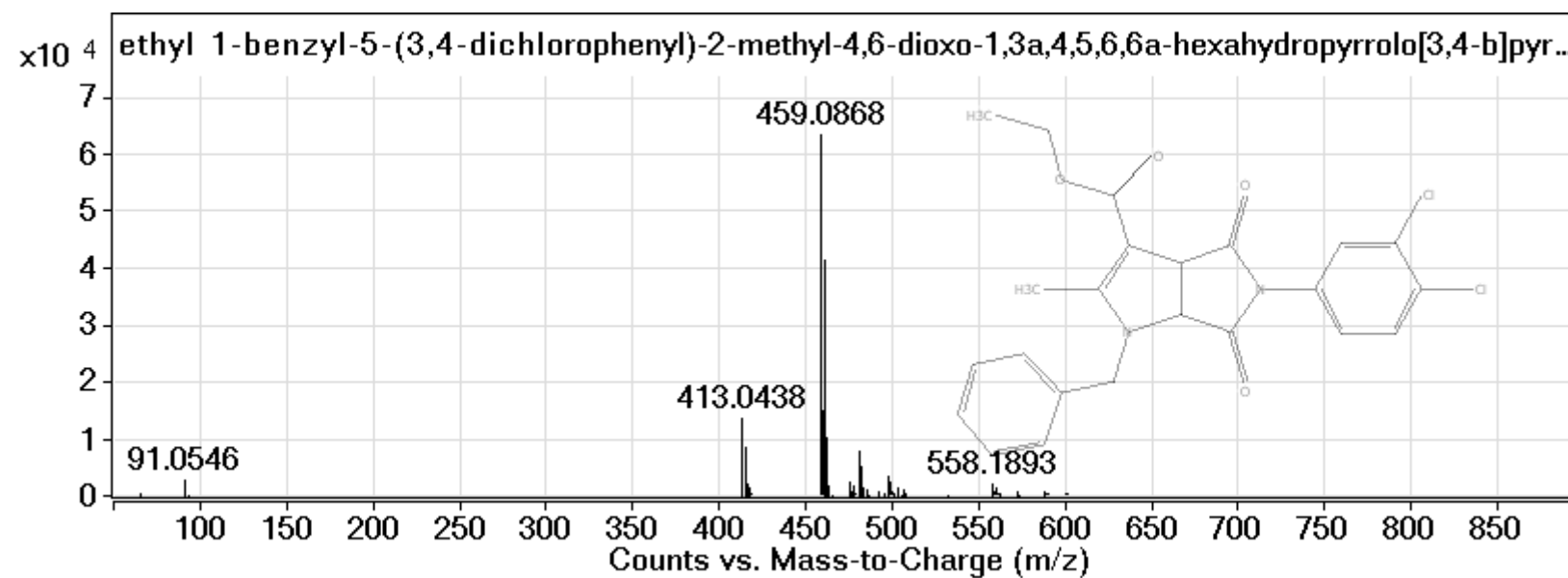
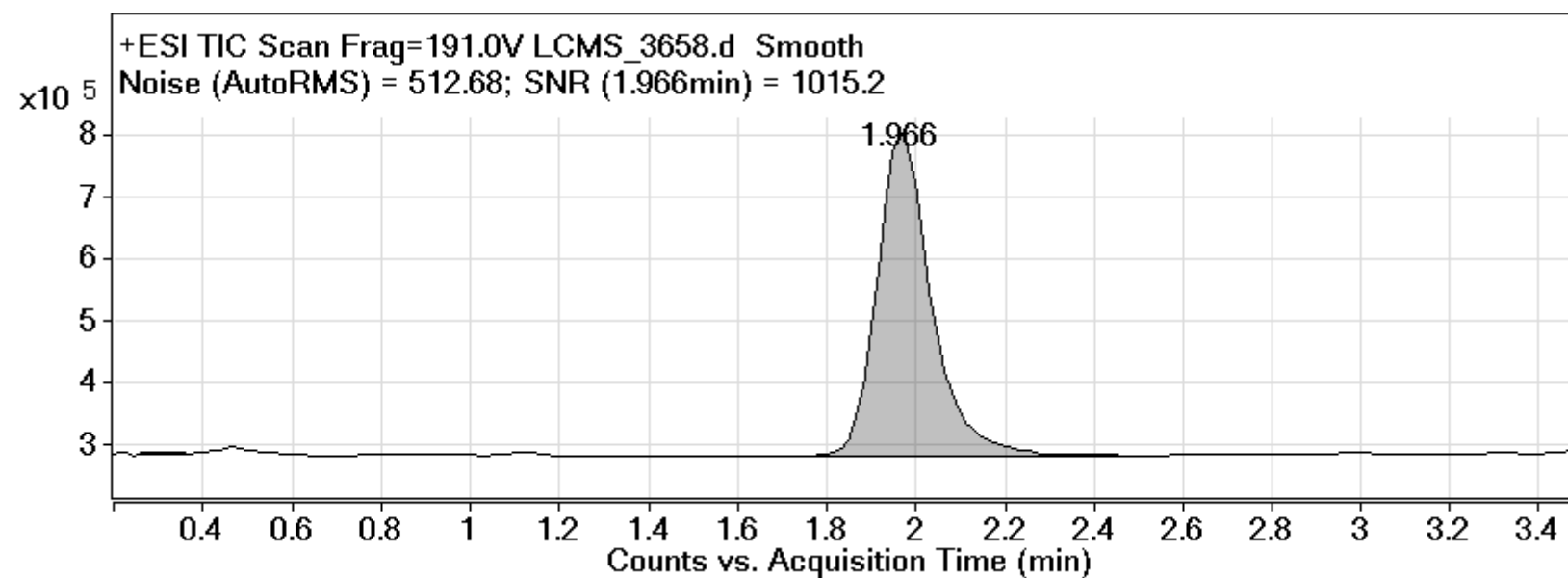
**(3a*S*,6a*R*)-Ethyl 1-benzyl-2-methyl-4,6-dioxo-5-(3,4-dichlorophenyl)-1,3a,4,5,6a-hexahydropyrrolo[3,4-*b*]pyrrole-3-carboxylate 8e**



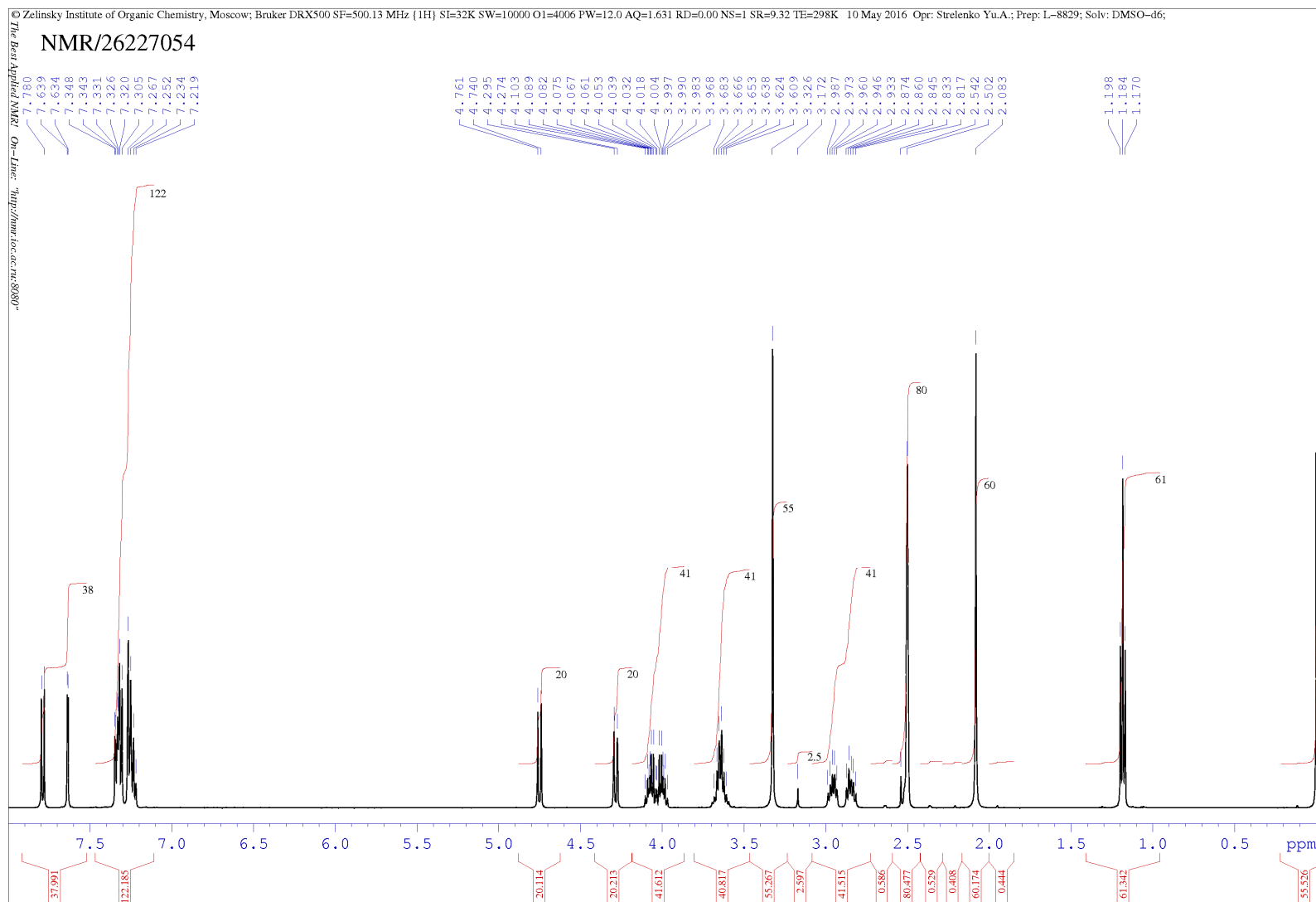
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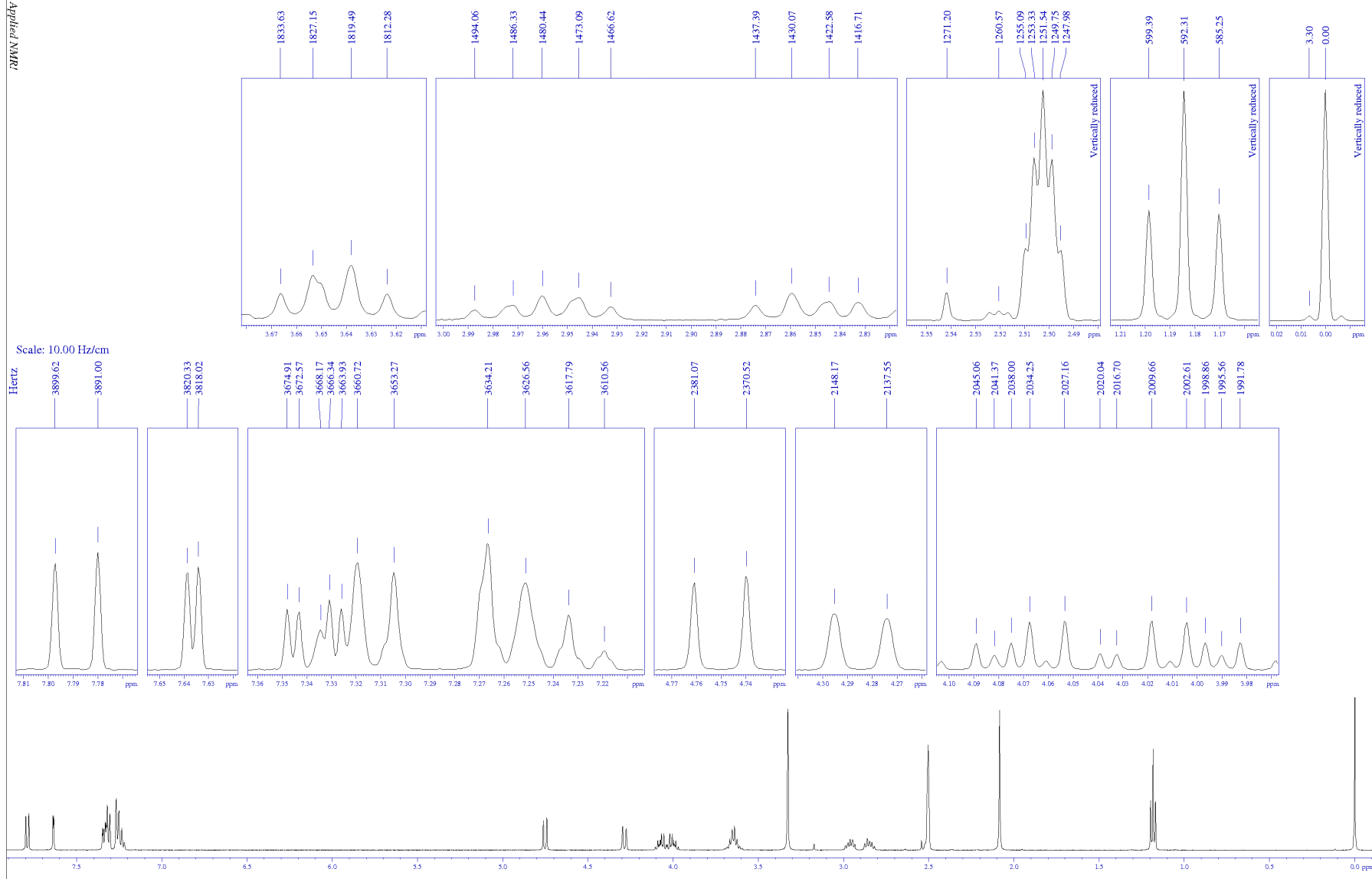


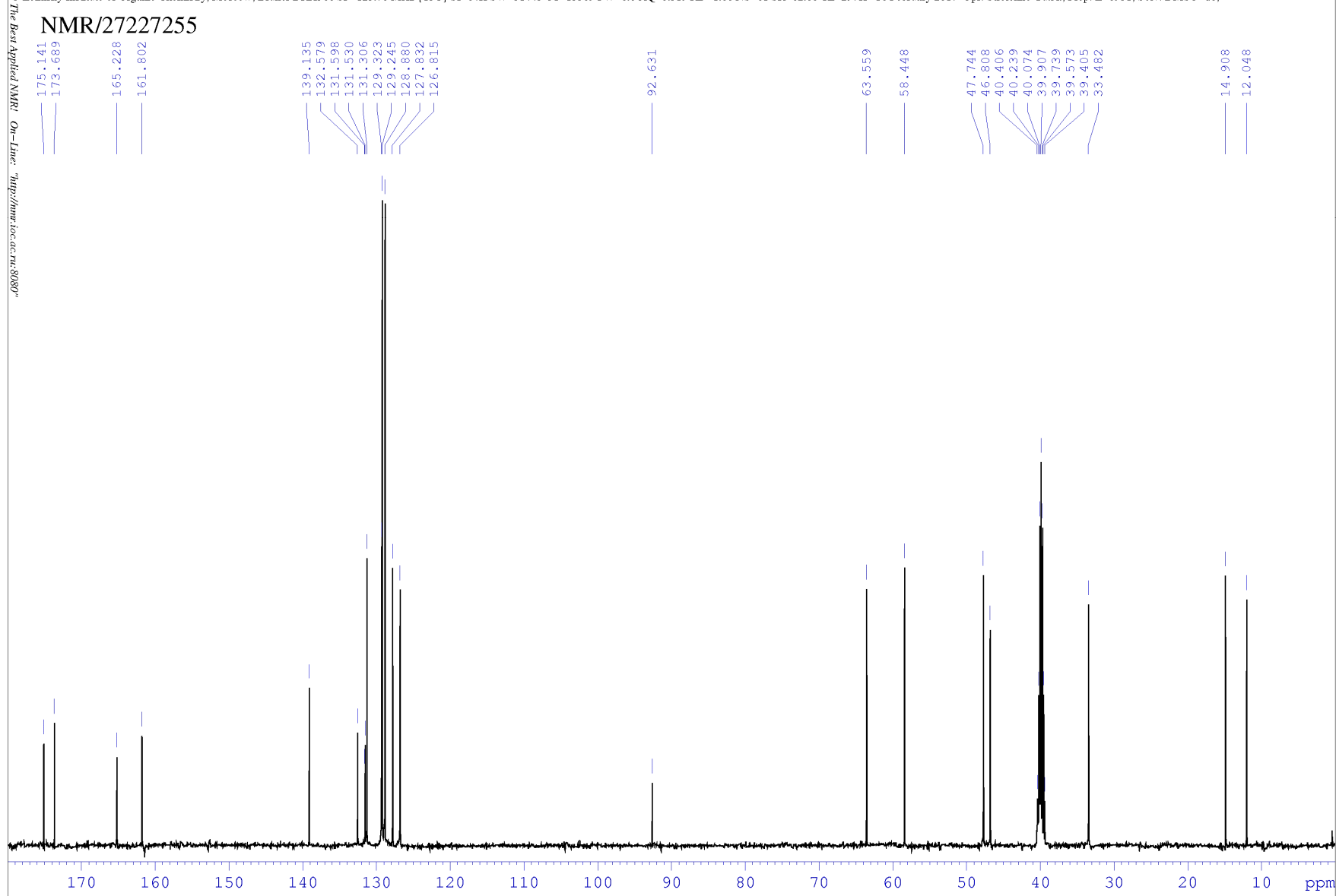


**(3a*S*,6a*R*)-Ethyl 5-(3,4-dichlorophenyl)-2-methyl-4,6-dioxo-1-phenetyl-1,3a,4,5,6a-hexahydropyrrolo[3,4-*b*]pyrrole-3-carboxylate 8f**

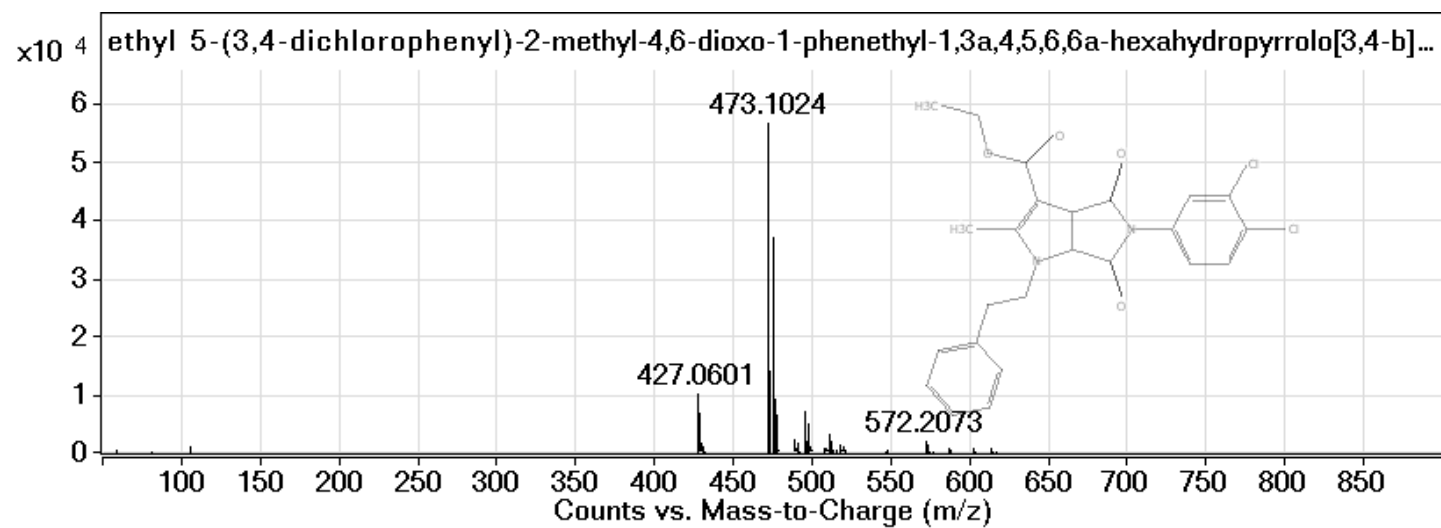
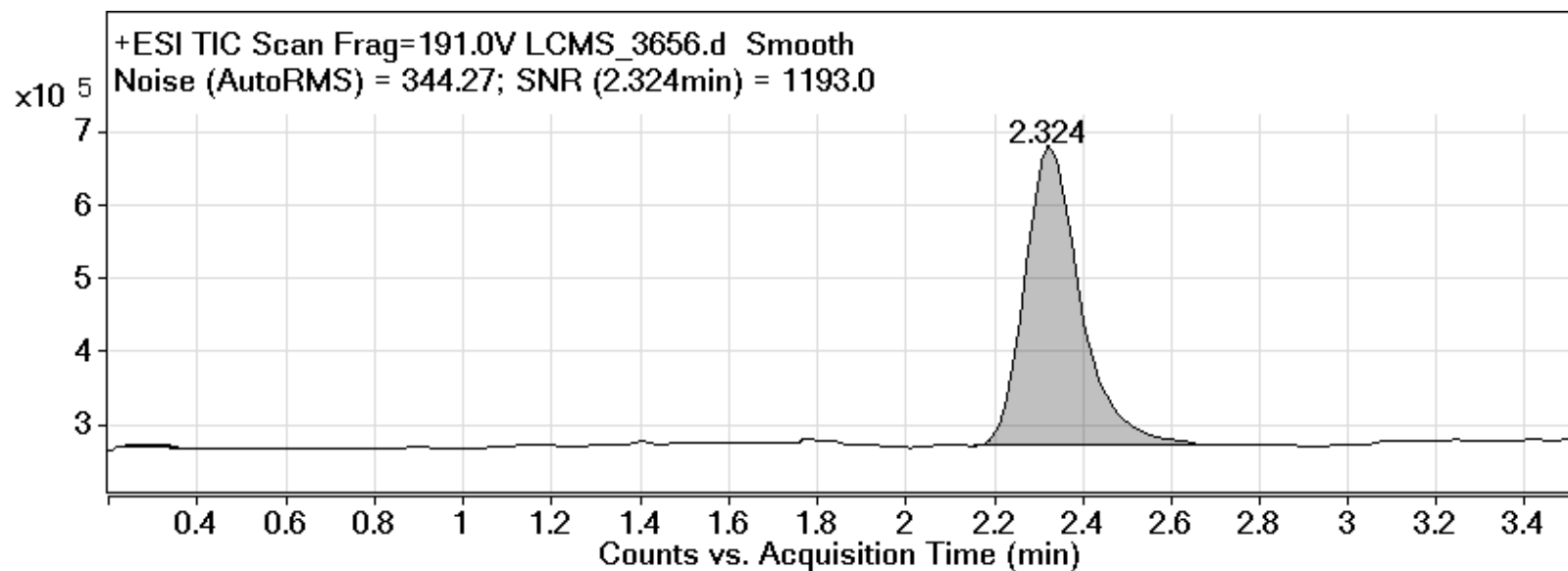


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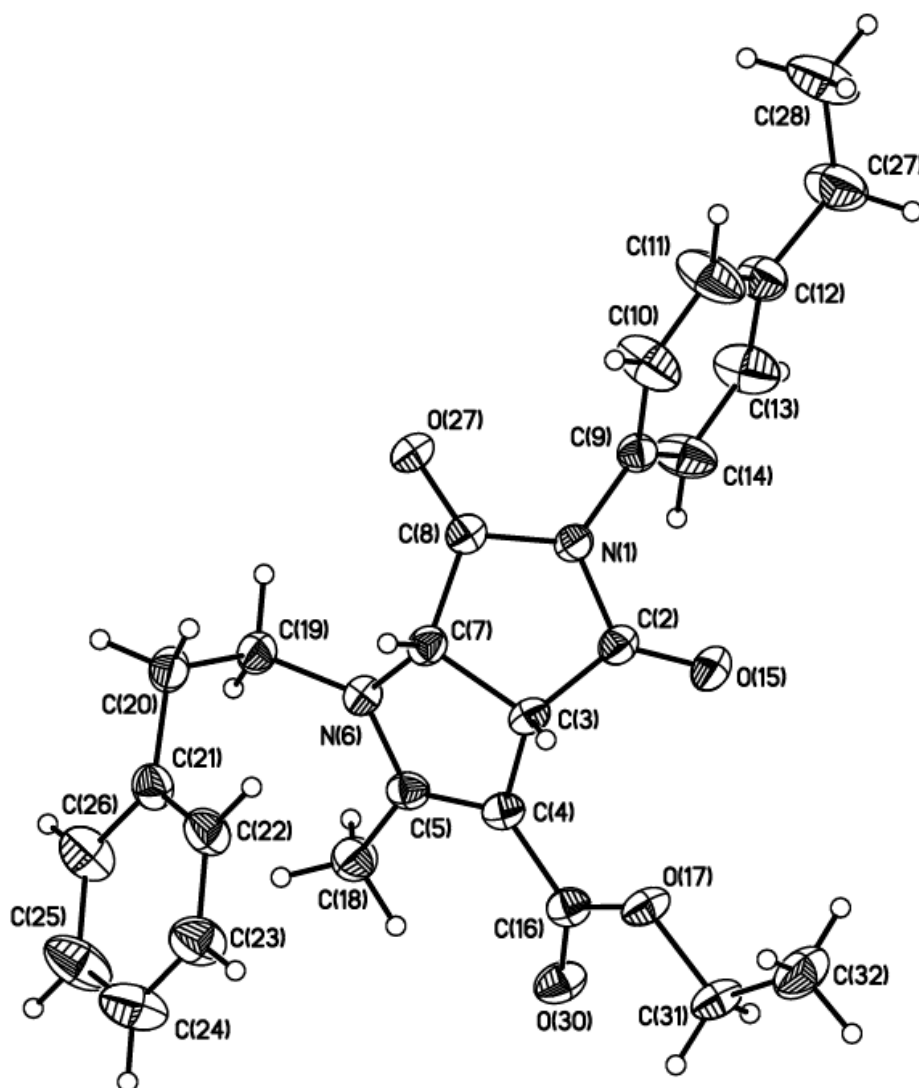




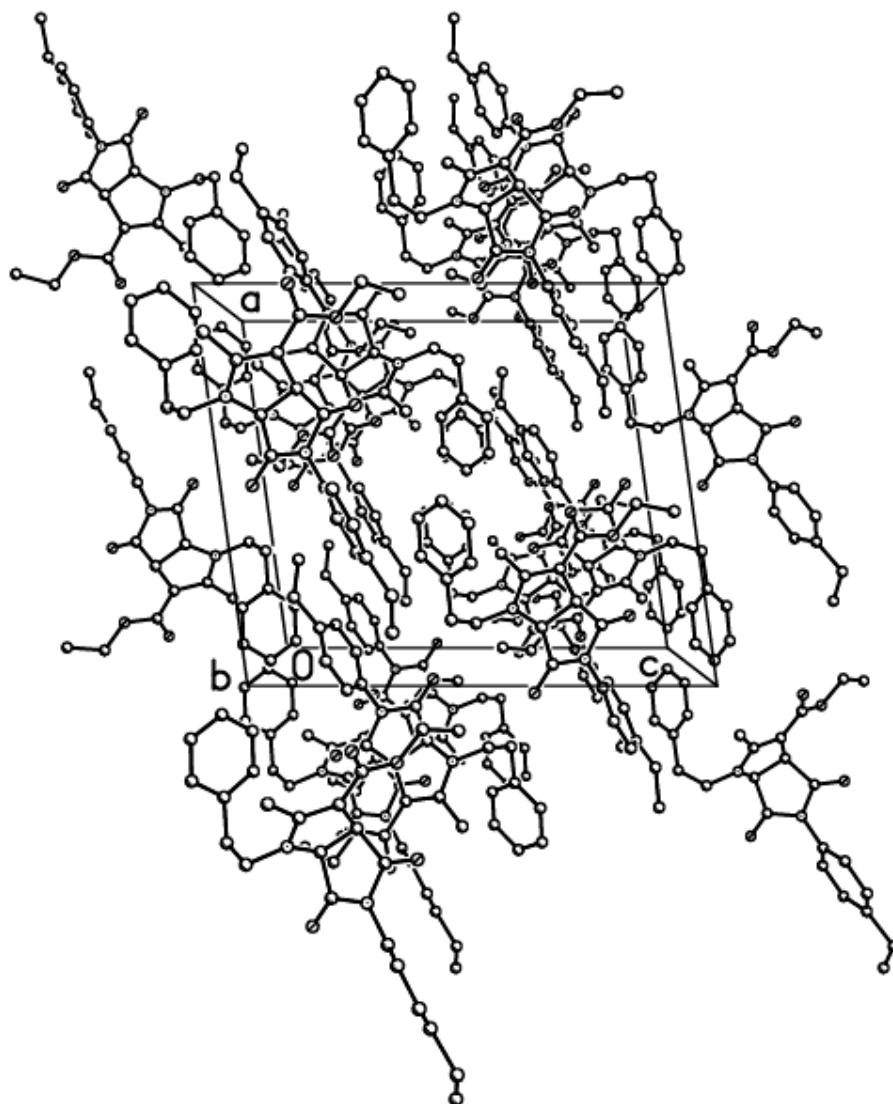


**X-Ray crystallographic data for (3*a*S,6*a*R)-ethyl 5-(4-ethylphenyl)-2-methyl-4,6-dioxo-1-phenetyl-1,3*a*,4,5,6,6*a*-hexahydropyrrolo[3,4-*b*]pyrrole-3-carboxylate 8c.**

CCDC 1574386 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html> (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336033; E-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk)). Crystal data for C<sub>26</sub>H<sub>28</sub>N<sub>2</sub>O<sub>4</sub> (M = 432.50 g/mol): monoclinic, space group P 2<sub>1</sub>/n (no. 14), *a* = 15.0252(9) Å, *b* = 8.6768(5) Å, *c* = 17.3436(10) Å,  $\alpha$  = 90°,  $\beta$  = 97.7760(10)°,  $\gamma$  = 90° *V* = 2240.3(2) Å<sup>3</sup>, *Z* = 4, *T* = 120(2) K,  $\mu$ (CuK $\alpha$ ) = 0.087 mm<sup>-1</sup>, *D*<sub>calc</sub> = 1.282 g/cm<sup>3</sup>. 20916 reflections measured (4.74° ≤ 2 $\theta$  ≤ 51.992°), 4387 unique (*R*<sub>int</sub> = 0.0380, *R*<sub>sigma</sub> = 0.0411) which were used in all calculations. The final *R*1 was 0.0483 (*I* > 2 $\sigma$ (*I*)) and *wR*2 was 0.0817 (all data).



**Figure 1.** The general form of the molecule **8c** in the crystal from X-ray diffraction studies. Non-hydrogen atoms are represented by probabilistic ellipsoids of atomic displacements (*p* = 0.5).



**Figure 2.** Fragment of the crystalline package **8c** along the crystallographic axis *b*.

An X-ray diffraction study of compound **8c** was carried out on an APEX II CCD diffractometer (MoK $\alpha$  radiation, graphite monochromator,  $\omega$  scan). The structure is deciphered by a direct method and refined by the least squares in the anisotropic full-matrix approximation by F2hkl. The positions of all hydrogen atoms are calculated from geometric considerations. All hydrogen atoms are refined in the isotropic approximation in the rider model. All calculations were carried out using the SHELXTL PLUS program complex [1].

## References

1. Sheldrick, G.M. A short history of SHELX. *Acta Cryst.* **2008**, *A64*, 112-122, <https://doi.org/10.1107/S0108767307043930>

Table 1. Crystal data and structure refinement for **8c**.

|                                   |   |                 |
|-----------------------------------|---|-----------------|
| Identification code               | x   |                 |
| Empirical formula                 | C <sub>26</sub> H <sub>28</sub> N <sub>2</sub> O <sub>4</sub> |                 |
| Formula weight                    | 432.50  |                 |
| Temperature                       | 120(2) K  |                 |
| Wavelength                        | 0.71073 Å   |                 |
| Crystal system                    | Monoclinic  |                 |
| Space group                       | P2 <sub>1</sub> /n  |                 |
| Unit cell dimensions              | a = 15.0252(9) Å  | = 90°.          |
|                                   | b = 8.6768(5) Å   | = 97.7760(10)°. |
|                                   | c = 17.3436(10) Å   | = 90°.          |
| Volume                            | 2240.3(2) Å <sup>3</sup>                                      |                 |
| Z                                 | 4   |                 |
| Density (calculated)              | 1.282 Mg/m <sup>3</sup>                                       |                 |
| Absorption coefficient            | 0.087 mm <sup>-1</sup>  |                 |
| F(000)                            | 920   |                 |
| Crystal size                      | 0.270 × 0.210 × 0.160 mm <sup>3</sup>                         |                 |
| Theta range for data collection   | 2.370 to 25.996°.   |                 |
| Index ranges                      | -18 ≤ h ≤ 18, -10 ≤ k ≤ 10, -21 ≤ l ≤ 21                      |                 |
| Reflections collected             | 20916   |                 |
| Independent reflections           | 4387 [R(int) = 0.0380]  |                 |
| Completeness to theta = 25.242°   | 99.3 %  |                 |
| Absorption correction             | Semi-empirical from equivalents                               |                 |
| Max. and min. transmission        | 0.995 and 0.962   |                 |
| Refinement method                 | Full-matrix least-squares on F <sup>2</sup>                   |                 |
| Data / restraints / parameters    | 4387 / 0 / 293  |                 |
| Goodness-of-fit on F <sup>2</sup> | 1.335   |                 |
| Final R indices [I > 2σ(I)]       | R1 = 0.0483, wR2 = 0.0817                                     |                 |
| R indices (all data)              | R1 = 0.0704, wR2 = 0.0879                                     |                 |
| Extinction coefficient            | n/a   |                 |
| Largest diff. peak and hole       | 0.794 and -0.338 e. Å <sup>-3</sup>                           |                 |

Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for **8c**.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^{ij}$  tensor.

|       | x        | y        | z       | $U(\text{eq})$ |
|-------|----------|----------|---------|----------------|
| O(15) | 8248(1)  | 9871(2)  | 1671(1) | 38(1)          |
| O(17) | 6242(1)  | 9111(2)  | 1846(1) | 30(1)          |
| O(27) | 10021(1) | 8704(2)  | 3940(1) | 33(1)          |
| O(30) | 5706(1)  | 10990(2) | 2555(1) | 34(1)          |
| N(1)  | 9290(1)  | 9531(2)  | 2757(1) | 23(1)          |
| N(6)  | 8044(1)  | 9508(2)  | 4134(1) | 26(1)          |
| C(2)  | 8432(1)  | 9401(2)  | 2324(1) | 25(1)          |
| C(3)  | 7813(1)  | 8615(2)  | 2829(1) | 23(1)          |
| C(4)  | 7074(1)  | 9678(2)  | 3019(1) | 24(1)          |
| C(5)  | 7277(1)  | 10172(2) | 3771(1) | 25(1)          |
| C(7)  | 8400(1)  | 8389(2)  | 3618(1) | 25(1)          |
| C(8)  | 9344(1)  | 8849(2)  | 3484(1) | 25(1)          |
| C(9)  | 10060(1) | 10113(2) | 2436(1) | 24(1)          |
| C(10) | 10761(2) | 9150(3)  | 2356(2) | 49(1)          |
| C(11) | 11497(2) | 9701(3)  | 2039(2) | 56(1)          |
| C(12) | 11545(1) | 11208(2) | 1798(1) | 36(1)          |
| C(13) | 10838(1) | 12155(3) | 1903(1) | 46(1)          |
| C(14) | 10095(1) | 11624(2) | 2216(1) | 41(1)          |
| C(16) | 6286(1)  | 10028(2) | 2482(1) | 26(1)          |
| C(18) | 6765(1)  | 11291(2) | 4191(1) | 34(1)          |
| C(19) | 8416(1)  | 9642(2)  | 4952(1) | 29(1)          |
| C(20) | 8222(1)  | 8249(2)  | 5451(1) | 32(1)          |
| C(21) | 7233(1)  | 7929(2)  | 5426(1) | 31(1)          |
| C(22) | 6806(1)  | 6881(2)  | 4897(1) | 35(1)          |
| C(23) | 5890(1)  | 6638(3)  | 4839(1) | 44(1)          |
| C(24) | 5386(2)  | 7448(3)  | 5309(2) | 52(1)          |
| C(25) | 5805(2)  | 8488(3)  | 5846(2) | 54(1)          |
| C(26) | 6720(2)  | 8719(2)  | 5906(1) | 42(1)          |
| C(31) | 5471(1)  | 9302(2)  | 1258(1) | 34(1)          |
| C(32) | 5639(2)  | 8325(3)  | 587(1)  | 48(1)          |
| C(27) | 12324(2) | 11814(3) | 1404(2) | 53(1)          |
| C(28) | 13173(2) | 10843(3) | 1521(2) | 56(1)          |

Table 3. Bond lengths [ $\text{\AA}$ ] and angles [ $^\circ$ ] for **8c**.

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|                   |            |
|-------------------|------------|
| O(15)-C(2)        | 1.200(2)   |
| O(17)-C(16)       | 1.355(2)   |
| O(17)-C(31)       | 1.445(2)   |
| O(27)-C(8)        | 1.208(2)   |
| O(30)-C(16)       | 1.225(2)   |
| N(1)-C(8)         | 1.384(2)   |
| N(1)-C(2)         | 1.407(2)   |
| N(1)-C(9)         | 1.441(2)   |
| N(6)-C(5)         | 1.364(2)   |
| N(6)-C(19)        | 1.458(2)   |
| N(6)-C(7)         | 1.470(2)   |
| C(2)-C(3)         | 1.523(2)   |
| C(3)-C(4)         | 1.513(2)   |
| C(3)-C(7)         | 1.536(2)   |
| C(4)-C(5)         | 1.368(3)   |
| C(4)-C(16)        | 1.437(2)   |
| C(5)-C(18)        | 1.488(3)   |
| C(7)-C(8)         | 1.521(2)   |
| C(9)-C(10)        | 1.367(3)   |
| C(9)-C(14)        | 1.368(3)   |
| C(10)-C(11)       | 1.384(3)   |
| C(11)-C(12)       | 1.378(3)   |
| C(12)-C(13)       | 1.373(3)   |
| C(12)-C(27)       | 1.528(3)   |
| C(13)-C(14)       | 1.385(3)   |
| C(19)-C(20)       | 1.536(3)   |
| C(20)-C(21)       | 1.506(3)   |
| C(21)-C(22)       | 1.386(3)   |
| C(21)-C(26)       | 1.389(3)   |
| C(22)-C(23)       | 1.382(3)   |
| C(23)-C(24)       | 1.379(3)   |
| C(24)-C(25)       | 1.385(3)   |
| C(25)-C(26)       | 1.380(3)   |
| C(31)-C(32)       | 1.489(3)   |
| C(27)-C(28)       | 1.518(3)   |
| <br>              |            |
| C(16)-O(17)-C(31) | 116.96(14) |
| C(8)-N(1)-C(2)    | 112.90(15) |

|                   |            |
|-------------------|------------|
| C(8)-N(1)-C(9)    | 123.38(14) |
| C(2)-N(1)-C(9)    | 123.15(15) |
| C(5)-N(6)-C(19)   | 127.17(16) |
| C(5)-N(6)-C(7)    | 110.62(14) |
| C(19)-N(6)-C(7)   | 121.53(15) |
| O(15)-C(2)-N(1)   | 123.70(17) |
| O(15)-C(2)-C(3)   | 127.94(17) |
| N(1)-C(2)-C(3)    | 108.35(15) |
| C(4)-C(3)-C(2)    | 112.03(15) |
| C(4)-C(3)-C(7)    | 103.52(14) |
| C(2)-C(3)-C(7)    | 104.36(14) |
| C(5)-C(4)-C(16)   | 127.51(17) |
| C(5)-C(4)-C(3)    | 108.87(15) |
| C(16)-C(4)-C(3)   | 123.58(16) |
| N(6)-C(5)-C(4)    | 111.90(16) |
| N(6)-C(5)-C(18)   | 120.68(16) |
| C(4)-C(5)-C(18)   | 127.42(17) |
| N(6)-C(7)-C(8)    | 110.11(15) |
| N(6)-C(7)-C(3)    | 104.23(14) |
| C(8)-C(7)-C(3)    | 105.40(14) |
| O(27)-C(8)-N(1)   | 125.57(17) |
| O(27)-C(8)-C(7)   | 126.33(17) |
| N(1)-C(8)-C(7)    | 108.05(14) |
| C(10)-C(9)-C(14)  | 119.87(18) |
| C(10)-C(9)-N(1)   | 119.67(17) |
| C(14)-C(9)-N(1)   | 120.45(17) |
| C(9)-C(10)-C(11)  | 119.8(2)   |
| C(12)-C(11)-C(10) | 121.7(2)   |
| C(13)-C(12)-C(11) | 117.03(19) |
| C(13)-C(12)-C(27) | 120.4(2)   |
| C(11)-C(12)-C(27) | 122.50(19) |
| C(12)-C(13)-C(14) | 122.1(2)   |

Table 4. Anisotropic displacement parameters ( $E^2 \times 10^3$ ) for **8c**. The anisotropic displacement factor exponent takes the form:  $-2 \text{ }^2 [ h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12} ]$

| $U^{11}$   | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|------------|----------|----------|----------|----------|----------|
| O(15)32(1) | 52(1)    | 27(1)    | 11(1)    | -2(1)    | 0(1)     |
| O(17)24(1) | 30(1)    | 32(1)    | -6(1)    | -7(1)    | 3(1)     |
| O(27)24(1) | 46(1)    | 28(1)    | 4(1)     | -4(1)    | 1(1)     |
| O(30)29(1) | 31(1)    | 40(1)    | -4(1)    | -4(1)    | 7(1)     |
| N(1)22(1)  | 24(1)    | 24(1)    | 2(1)     | 1(1)     | 0(1)     |
| N(6)25(1)  | 30(1)    | 23(1)    | 1(1)     | 1(1)     | 0(1)     |
| C(2) 25(1) | 25(1)    | 24(1)    | 0(1)     | 0(1)     | 3(1)     |
| C(3) 21(1) | 21(1)    | 26(1)    | 1(1)     | -2(1)    | -1(1)    |
| C(4) 22(1) | 20(1)    | 28(1)    | 0(1)     | 1(1)     | -1(1)    |
| C(5) 22(1) | 23(1)    | 30(1)    | 2(1)     | 4(1)     | -3(1)    |
| C(7) 25(1) | 24(1)    | 26(1)    | 3(1)     | 1(1)     | 0(1)     |
| C(8) 25(1) | 24(1)    | 26(1)    | -1(1)    | 1(1)     | 1(1)     |
| C(9) 23(1) | 24(1)    | 25(1)    | -1(1)    | 2(1)     | -1(1)    |
| C(10)51(1) | 26(1)    | 76(2)    | 14(1)    | 34(1)    | 8(1)     |
| C(11)48(1) | 40(1)    | 88(2)    | 14(1)    | 38(1)    | 16(1)    |
| C(12)31(1) | 35(1)    | 43(1)    | 4(1)     | 9(1)     | 0(1)     |
| C(13)39(1) | 25(1)    | 77(2)    | 9(1)     | 18(1)    | -2(1)    |
| C(14)31(1) | 26(1)    | 68(2)    | 4(1)     | 16(1)    | 6(1)     |
| C(16)23(1) | 22(1)    | 31(1)    | 0(1)     | 1(1)     | -3(1)    |
| C(18)32(1) | 37(1)    | 32(1)    | -4(1)    | 4(1)     | 4(1)     |
| C(19)28(1) | 34(1)    | 24(1)    | 1(1)     | 1(1)     | -4(1)    |
| C(20)33(1) | 36(1)    | 25(1)    | 4(1)     | 1(1)     | -3(1)    |
| C(21)34(1) | 30(1)    | 29(1)    | 3(1)     | 7(1)     | -3(1)    |
| C(22)35(1) | 37(1)    | 34(1)    | -6(1)    | 11(1)    | -2(1)    |
| C(23)37(1) | 46(1)    | 49(1)    | -14(1)   | 9(1)     | -9(1)    |
| C(24)34(1) | 53(2)    | 74(2)    | -16(1)   | 17(1)    | -7(1)    |
| C(25)49(1) | 47(2)    | 72(2)    | -22(1)   | 31(1)    | -7(1)    |
| C(26)46(1) | 36(1)    | 47(1)    | -14(1)   | 17(1)    | -12(1)   |
| C(31)25(1) | 35(1)    | 37(1)    | -3(1)    | -9(1)    | 1(1)     |
| C(32)44(1) | 56(2)    | 39(1)    | -13(1)   | -12(1)   | 10(1)    |
| C(27)36(1) | 55(2)    | 72(2)    | 16(1)    | 18(1)    | -2(1)    |
| C(28)36(1) | 53(2)    | 84(2)    | -20(1)   | 28(1)    | -10(1)   |

Table 5. Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $E^2 \times 10^3$ ) for **8c**.



|        | x     | y     | z    | U(eq) |
|--------|-------|-------|------|-------|
| H(3A)  | 7571  | 7617  | 2600 | 28    |
| H(7A)  | 8374  | 7309  | 3813 | 30    |
| H(10A) | 10744 | 8106  | 2518 | 59    |
| H(11A) | 11981 | 9022  | 1987 | 67    |
| H(13A) | 10861 | 13207 | 1757 | 55    |
| H(14A) | 9613  | 12303 | 2278 | 49    |
| H(18A) | 6221  | 11609 | 3851 | 50    |
| H(18B) | 7139  | 12196 | 4337 | 50    |
| H(18C) | 6596  | 10804 | 4660 | 50    |
| H(19A) | 8166  | 10578 | 5170 | 35    |
| H(19B) | 9074  | 9778  | 4990 | 35    |
| H(20A) | 8517  | 7327  | 5265 | 38    |
| H(20B) | 8487  | 8438  | 5996 | 38    |
| H(22A) | 7148  | 6321  | 4569 | 42    |
| H(23A) | 5608  | 5913  | 4474 | 53    |
| H(24A) | 4755  | 7293  | 5266 | 63    |
| H(25A) | 5461  | 9044  | 6173 | 65    |
| H(26A) | 7003  | 9426  | 6279 | 51    |
| H(31A) | 5403  | 10396 | 1099 | 40    |
| H(31B) | 4916  | 8968  | 1460 | 40    |
| H(32A) | 5129  | 8409  | 173  | 72    |
| H(32B) | 5711  | 7249  | 755  | 72    |
| H(32C) | 6187  | 8675  | 392  | 72    |
| H(27A) | 12120 | 11900 | 838  | 64    |
| H(27B) | 12476 | 12865 | 1602 | 64    |
| H(28A) | 13652 | 11379 | 1298 | 84    |
| H(28B) | 13056 | 9846  | 1261 | 84    |
| H(28C) | 13357 | 10678 | 2078 | 84    |

Table 6. Torsion angles [°] for **8c**.

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|                       |             |
|-----------------------|-------------|
| C(8)-N(1)-C(2)-O(15)  | -177.61(18) |
| C(9)-N(1)-C(2)-O(15)  | -6.0(3)     |
| C(8)-N(1)-C(2)-C(3)   | 3.4(2)      |
| C(9)-N(1)-C(2)-C(3)   | 175.02(15)  |
| O(15)-C(2)-C(3)-C(4)  | -64.5(3)    |
| N(1)-C(2)-C(3)-C(4)   | 114.37(16)  |
| O(15)-C(2)-C(3)-C(7)  | -175.87(19) |
| N(1)-C(2)-C(3)-C(7)   | 3.02(19)    |
| C(2)-C(3)-C(4)-C(5)   | -104.09(17) |
| C(7)-C(3)-C(4)-C(5)   | 7.78(19)    |
| C(2)-C(3)-C(4)-C(16)  | 78.0(2)     |
| C(7)-C(3)-C(4)-C(16)  | -170.12(16) |
| C(19)-N(6)-C(5)-C(4)  | -173.61(17) |
| C(7)-N(6)-C(5)-C(4)   | -3.1(2)     |
| C(19)-N(6)-C(5)-C(18) | 6.4(3)      |
| C(7)-N(6)-C(5)-C(18)  | 177.00(16)  |
| C(16)-C(4)-C(5)-N(6)  | 174.52(17)  |
| C(3)-C(4)-C(5)-N(6)   | -3.3(2)     |
| C(16)-C(4)-C(5)-C(18) | -5.5(3)     |
| C(3)-C(4)-C(5)-C(18)  | 176.67(17)  |
| C(5)-N(6)-C(7)-C(8)   | 120.41(16)  |
| C(19)-N(6)-C(7)-C(8)  | -68.4(2)    |
| C(5)-N(6)-C(7)-C(3)   | 7.79(19)    |
| C(19)-N(6)-C(7)-C(3)  | 178.97(15)  |
| C(4)-C(3)-C(7)-N(6)   | -9.05(17)   |
| C(2)-C(3)-C(7)-N(6)   | 108.33(15)  |
| C(4)-C(3)-C(7)-C(8)   | -125.01(15) |
| C(2)-C(3)-C(7)-C(8)   | -7.63(18)   |
| C(2)-N(1)-C(8)-O(27)  | 174.08(18)  |
| C(9)-N(1)-C(8)-O(27)  | 2.5(3)      |
| C(2)-N(1)-C(8)-C(7)   | -8.5(2)     |
| C(9)-N(1)-C(8)-C(7)   | 179.90(15)  |
| N(6)-C(7)-C(8)-O(27)  | 75.4(2)     |
| C(3)-C(7)-C(8)-O(27)  | -172.71(18) |
| N(6)-C(7)-C(8)-N(1)   | -101.92(16) |
| C(3)-C(7)-C(8)-N(1)   | 9.94(19)    |
| C(8)-N(1)-C(9)-C(10)  | 57.2(3)     |
| C(2)-N(1)-C(9)-C(10)  | -113.5(2)   |

|                         |             |
|-------------------------|-------------|
| C(8)-N(1)-C(9)-C(14)    | -122.3(2)   |
| C(2)-N(1)-C(9)-C(14)    | 67.0(3)     |
| C(14)-C(9)-C(10)-C(11)  | -1.2(4)     |
| N(1)-C(9)-C(10)-C(11)   | 179.2(2)    |
| C(9)-C(10)-C(11)-C(12)  | -0.1(4)     |
| C(10)-C(11)-C(12)-C(13) | 1.6(4)      |
| C(10)-C(11)-C(12)-C(27) | -176.4(2)   |
| C(11)-C(12)-C(13)-C(14) | -1.8(4)     |
| C(27)-C(12)-C(13)-C(14) | 176.1(2)    |
| C(10)-C(9)-C(14)-C(13)  | 1.0(3)      |
| N(1)-C(9)-C(14)-C(13)   | -179.49(19) |
| C(12)-C(13)-C(14)-C(9)  | 0.6(4)      |
| C(31)-O(17)-C(16)-O(30) | -0.8(3)     |
| C(31)-O(17)-C(16)-C(4)  | 178.64(16)  |
| C(5)-C(4)-C(16)-O(30)   | 10.9(3)     |
| C(3)-C(4)-C(16)-O(30)   | -171.64(18) |
| C(5)-C(4)-C(16)-O(17)   | -168.54(17) |
| C(3)-C(4)-C(16)-O(17)   | 9.0(2)      |
| C(5)-N(6)-C(19)-C(20)   | 100.5(2)    |
| C(7)-N(6)-C(19)-C(20)   | -69.1(2)    |
| N(6)-C(19)-C(20)-C(21)  | -57.8(2)    |
| C(19)-C(20)-C(21)-C(22) | 93.7(2)     |
| C(19)-C(20)-C(21)-C(26) | -83.4(2)    |
| C(26)-C(21)-C(22)-C(23) | 0.8(3)      |
| C(20)-C(21)-C(22)-C(23) | -176.4(2)   |
| C(21)-C(22)-C(23)-C(24) | 0.2(4)      |
| C(22)-C(23)-C(24)-C(25) | -0.8(4)     |
| C(23)-C(24)-C(25)-C(26) | 0.4(4)      |
| C(24)-C(25)-C(26)-C(21) | 0.7(4)      |
| C(22)-C(21)-C(26)-C(25) | -1.3(3)     |
| C(20)-C(21)-C(26)-C(25) | 175.9(2)    |
| C(16)-O(17)-C(31)-C(32) | 172.89(17)  |
| C(13)-C(12)-C(27)-C(28) | 163.4(2)    |
| C(11)-C(12)-C(27)-C(28) | -18.7(4)    |