

Supporting Materials

Inhibitors of Fumarylacetoacetate Hydrolase Domain Containing Protein 1 (FAHD1)

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- **Compounds described in the manuscript:**

- 2-(2-methoxy-2-oxoacetamido)benzoic acid (**1a**) (RN: 174575-83-8)
2-(carboxyformamido)benzoic acid (**1b**) (RN: 5651-01-4)
2-(2-(2-methoxy-2-oxoacetamido)benzamido)benzoic acid (**1d**)
2-(2-(carboxyformamido)benzamido)benzoic acid (**1e**)
3-(2-methoxy-2-oxoacetamido)benzoic acid (**2a**)
3-(Carboxyformamido)benzoic acid (**2b**) (RN: 58465-46-6)
4-(2-methoxy-2-oxoacetamido)benzoic acid (**3a**) (RN: 1086273-37-1)
4-(carboxyformamido)benzoic acid (**3b**) (RN: 14121-56-3)
Methyl 2-((2-nitrophenyl)amino)-2-oxoacetate (**4a**)
2-((2-nitrophenyl)amino)-2-oxoacetic acid (**4b**) (RN: 77901-51-0)
Methyl 2-((3-nitrophenyl)amino)-2-oxoacetate (**5a**) (RN: 1237427-49-4)
2-((3-nitrophenyl)amino)-2-oxoacetic acid(**5b**) (RN: 6274-26-6)
Methyl 2-((4-nitrophenyl)amino)-2-oxoacetate (**6a**) (RN: 82633-22-5)
2-((4-nitrophenyl)amino)-2-oxoacetic acid (**6b**) (RN: 103-94-6)
Methyl 2-(naphthalen-2-ylamino)-2-oxoacetate (**7a**) (RN: 20518-40-5)
2-(naphthalen-2-ylamino)-2-oxoacetic acid (**7b**) RN: 81682-60-2
Methyl 2-(naphthalen-1-ylamino)-2-oxoacetate (**8a**) (RN: 254751-07-0)
2-(naphthalen-1-ylamino)-2-oxoacetic acid (**8b**) (RN: 21660-76-4)
Methyl 2-oxo-2-(pyridin-3-ylamino)acetate (**9a**) (RN: 480452-68-4)
3-(2-Methoxy-2-oxoacetamido)pyridine 1-oxide (**11a**)

3-(Carboxyformamido)pyridine 1-oxide (**11b**)
Methyl 2-oxo-2-(pyridin-2-ylamino)acetate (**10a**)
2-(2-Methoxy-2-oxoacetamido)pyridine 1-oxide (**12a**)
2-(Carboxyformamido)pyridine 1-oxide (**12b**)
Methyl 2-oxo-2-(quinolin-3-ylamino)acetate (**13a**)
Methyl 2-oxo-2-(quinolin-2-ylamino)acetate (**14a**)
3-(2-Methoxy-2-oxoacetamido)quinoline 1-oxide (**15a**)
3-(Carboxyformamido)quinoline 1-oxide (**15b**)
2-(2-Methoxy-2-oxoacetamido)quinoline 1-oxide (**16a**)
2-(Carboxyformamido)quinoline 1-oxide (**16b**)
Methyl 4-oxo-4*H*-benzo[d][1,3]oxazine-2-carboxylate (**17a**) (RN: 3603-20-1)
Methyl 2-((2-carbamoylphenyl)amino)-2-oxoacetate (**18a**) (RN: 69065-88-9)
Methyl 4-oxo-1,4-dihydroquinazoline-2-carboxylate (**19a**) (RN: 63569-82-4)
4-oxo-1,4-dihydroquinazoline-2-carboxylic acid (**19b**) (RN: 29113-34-6)
Methyl 2-((2-methoxyphenyl)amino)-2-thioxoacetate (**20a**)
2-((2-methoxyphenyl)amino)-2-thioxoacetic acid (**20b**) (RN: 7267-58-5)
Methyl 2-((3-methoxyphenyl)amino)-2-thioxoacetate (**21a**)
2-((3-methoxyphenyl)amino)-2-thioxoacetic acid (**21b**) (RN: 276693-17-5)
Methyl 2-((4-methoxyphenyl)amino)-2-thioxoacetate (**22a**)
2-((4-methoxyphenyl)amino)-2-thioxoacetic acid (**22b**) (RN: 946-61-2)
*N*¹-(pyridin-2-yl)oxalamide (**23**) (RN: 52781-00-7)
*N*¹-(naphthalen-2-yl)oxalamide (**24**) (RN: 21775-72-4)
N-(6-methylpyridin-2-yl)-2-oxo-2-(pyrrolidin-1-yl)acetamide (**25**) (RN: 1210298-59-1)
2-(2-*tert*-Butoxy-2-oxoacetamido)benzoic acid (**26**)
3-(2-*tert*-Butoxy-2-oxoacetamido)benzoic acid (**27**)
4-(2-*tert*-Butoxy-2-oxoacetamido)benzoic acid (**28**) (RN: 614760-53-1)
*N*¹,*N*²-bis(5-methylpyridin-2-yl)oxalamide (**29**) (RN: 349401-68-9)
*N*¹,*N*²-di(pyridin-2-yl)oxalamide (**30**) (RN: 20172-97-8)
N,N'-Di-pyridin-3-yl-oxalamide (**31**) (RN: 39642-61-0)
N-Pyridin-2-yl-*N*'-p-tolyl-oxalamide (**32**) (RN: 301344-55-8)
N-Pyridin-2-yl-*N*'-m-tolyl-oxalamide (**33**)
N-Pyridin-3-yl-*N*'-pyridin-2-yl-oxalamide (**34**) (RN: 1796089-38-7)
N-(1-Oxy-pyridin-2-yl)-*N*'-m-tolyl-oxalamide (**35**)
N-(1-Oxy-pyridin-2-yl)-*N*'-p-tolyl-oxalamide (**36**)
N-(1-Oxy-pyridin-3-yl)-*N*'-m-tolyl-oxalamide (**37**)

• Starting materials and intermediates used in synthesis:

Methyl 2-((6-methylpyridin-2-yl)amino)-2-oxoacetate (**S1**) (RN: 1566684-50-1)
Methyl 2-((2-methoxyphenyl)amino)-2-oxoacetate (**S2**) (RN: 113449-16-4)
Methyl 2-((3-methoxyphenyl)amino)-2-oxoacetate (**S3**) (RN: 103448-86-8)
Methyl 2-((4-methoxyphenyl)amino)-2-oxoacetate (**S4**) (RN: 24439-54-1)

- Additional compounds from SAR:

- 2-oxo-2-(*o*-tolylamino)acetic acid (**S5**) (RN: 406190-09-8)
- 2-(((1*R*,2*S*)-2-hydroxy-2,3-dihydro-1*H*-inden-1-yl)amino)-2-oxoacetic acid (**S6**) (RN: 1849465-46-8)
- Methyl 2-(*tert*-butylamino)-2-oxoacetate (**S7**) (RN: 1450740-20-1)
- 2-(benzylamino)-2-oxoacetic acid (**S8**) (RN: 6345-08-0)
- Methyl 2-(((1*S*,2*R*)-1-hydroxy-1-phenylpropan-2-yl)amino)-2-oxoacetate (**S9**) (RN: 1218912-11-8)
- 2-((4-fluorophenyl)amino)-2-oxoacetic acid (**S10**) (RN: 69066-43-9)
- 2-((2-fluorophenyl)amino)-2-oxoacetic acid (**S11**) (RN: 84944-15-0)
- 2-((2-bromophenyl)amino)-2-oxoacetic acid (**S12**) (RN: 868565-59-7)
- 2-((3-bromophenyl)amino)-2-oxoacetic acid (**S13**) (RN: 946744-52-1)
- 2-((2,4-dichlorophenyl)amino)-2-oxoacetic acid (**S14**) (RN: 17772-30-4)
- 2-((3-hydroxyphenyl)amino)-2-oxoacetic acid (**S15**) (RN: 38188-60-2)
- 2-((2-hydroxyphenyl)amino)-2-oxoacetic acid (**S16**) (RN: 89942-67-6)
- 2-((2-methoxyphenyl)amino)-2-oxoacetic acid (**S17**) (RN: 57727-23-8)
- 2-((4-methoxyphenyl)amino)-2-oxoacetic acid (**S18**) (RN: 41374-62-3)
- 2-(2-*(tert*-butoxy)-2-oxoacetamido)-5-methylpyridine 1-oxide (**S19**)
- tert*-butyl 2-((5-methylpyridin-2-yl)amino)-2-oxoacetate (**S20**)
- 5-bromo-2-(2-*(tert*-butoxy)-2-oxoacetamido)pyridine 1-oxide (**S21**)
- 2-(2-*(tert*-butoxy)-2-oxoacetamido)-6-(methoxycarbonyl)pyridine 1-oxide (**S22**)

- A comprehensive discussion of the “many faces” of FAHD1 substrate oxaloacetate (OAA)

The structural status of oxaloacetate is mainly dependent on the environmental pH value. OAA may add water to the 2-keto group and form a hydrate (only at low pH). The methylene protons in OAA are acidic due to the flanking 1,3-keto-groups. The molecule can tautomerize into enol forms, which may exist on their part in *E*- and *Z*-configuration. Each of the different OAA-forms will show an individual chemical reactivity. FAHD1 is operative in mitochondria where OAA will be fully deprotonated to the di-anion at the prevailing pH of 7.9 in the mitochondrial matrix (Figure S1)[1]. The pH-dependent protonation states of possible OAA-species in aqueous solution can be calculated (<https://chemicalize.com/#/calculation>). At physiologocal pH the doubly deprotonated OAA^{2-} species predominates. At lower pH, the C¹-carboxylate is more easily deprotonated than the C⁴-carboxylate which reflects the binding affinity of OAA^- towards the Mg^{2+} ion in the catalytic cavity of FAHD1.

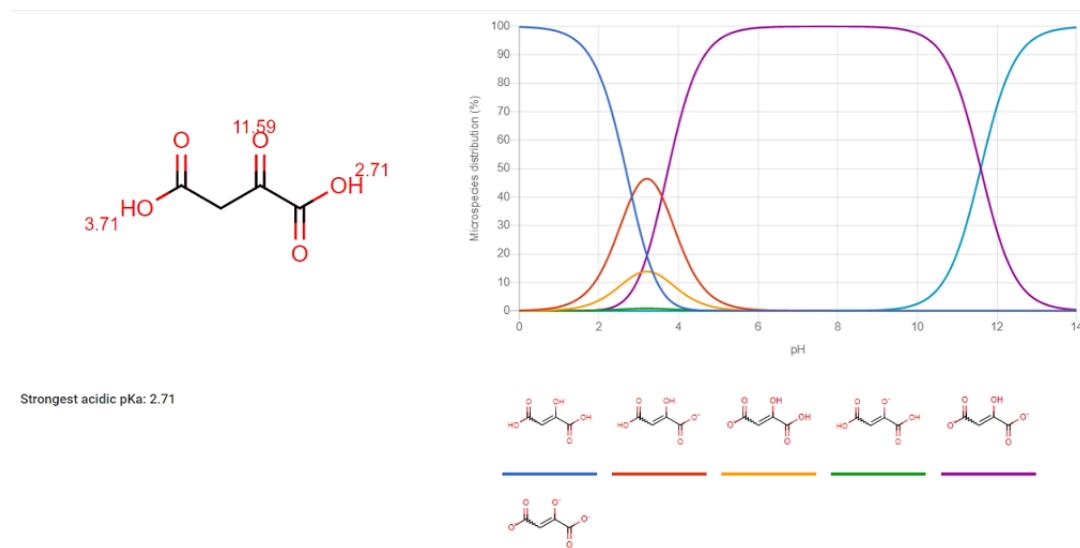


Figure S1. Calculated pH-dependent protonation states of OAA-species.

It has been shown that OAA exists in solution and in solid state in (*Z*)-enol form[2] OAA is unstable in solution and starts slowly to decarboxylate following a first-order kinetics[3]. In organic solvents such as ether OAA is also mainly in the enol form (~90 %)[4]. In a 0.1 M tris buffer at physiological pH value of 7.4 the keto-OAA is predominant (74.3 %), but the enolic species (B') contributes significantly (17.8 %) while the hydrate (D') is a minor component (7.8 %). Decreasing the pH to 2.0 leads to an increase of the hydrate (54.9 %) in expense of the keto form[5]. The three titration dissociations constants of OAA have been determined: 2.22 (pKa 1-COOH), 3.89 (pKa 4-COOH), and 12.18 (pKa enolic OH), respectively. The pKa(1) and pKa (2) are slightly higher in the enol form[6,7]. A striking observation is reported for the dissociation of the third proton from enolic oxaloacetic acid. Upon complexation to magnesium the dissociation constant increases more than ten-thousand-fold[6], enabling the deprotonation of the enol function (e.g. by an amino residue). For comparison: without Mg pKa of enol-OH = 12.18 but complexed to Mg pKa = 8.11. Additionally, the enol form is stabilized by the magnesium. Investigations of complexes between magnesium and enolate OAA^{3-} revealed for a 1:1 complex a stability constant of $\log K = 6.27$ which is also ten-thousand-fold greater than any reported $\log K$ for Mg^{2+} ions with simple organic acid ligands[8], such as the acid ligands complexing magnesium in the catalytic center of FAHD1.

Another important result was provided by investigation of metal-ion catalyzed decarboxylation of dimethyl-oxaloacetic acid (Figure S2.A), a molecule which cannot adopt an enol form. Enolization was found to be not a prerequisite for metal catalyzed decarboxylation and that the first product was the enol form of α -ketoisovaleric acid[9] (**B**). Furthermore, Steinberger and Westheimer demonstrated that decarboxylation of **C** to **D** was not catalyzed by metals[9] Similar to mono anion, thermal decarboxylation of mono ethyl ester (**C**) yields the enol form of ethyl α -ketoisovalerate (**D**)[10]

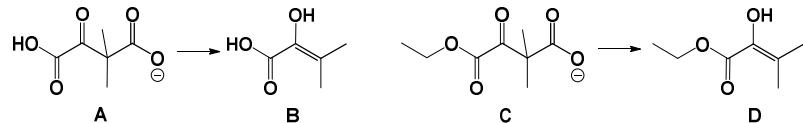


Figure S2. Decarboxylation of dimethyl-oxaloacetic acid mono anion **A** and corresponding mono ethyl ester **C**: primary products are enol forms **B** and **D**

From the Steinberger and Westheimer experiments it could be concluded that the 4-carboxylate group is the source of carbon dioxide. Furthermore, UV/Vis spectroscopy revealed that when OAA is exposed to divalent metal ions a rapid, strong increase of absorption is recorded, at a wavelength where α -keto acids have little absorption. The authors assigned the new absorbing species to an enolic metal complex of structure **B** shown in Figure S2. The presence of an enol was verified by iodine titration experiments. But was chelated **B** the structure from which carbon dioxide originates? Two possible Mg^{2+} - OAA²⁻ chelates are depicted in Figure 4.

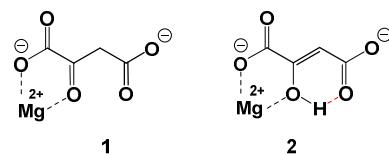


Figure S3. Metal chelated oxaloacetate di-anion: 2-keto form (**1**), enol form (**2**); hydrogen bonding: dashed red bond.

Considering that 3,3-dimethyl oxaloacetic acid (see Figure S2, **A**) cannot form an enolic complex of type **2** (Figure S3, **2**) but nevertheless decarboxylates metal catalyzed. This suggests complex **1** as the most likely active species in the metal catalyzed decarboxylation of OAA. Additionally, complex **1** has rotational freedom around two C-C bonds (C²-C³ and C³-C⁴). But the enol-complex **2** is a flat molecule with resonance stabilized intramolecular hydrogen bond between enolic hydrogen and oxygen of the 4-carboxylate (red bond in Figure S3, **2**). The C³-C⁴ σ -bond has no orbital overlap with the π -bond present in the enol. Decarboxylation of complex **2** would produce a non-stabilized sp²-centered carbanion. Gelles and Hay provided experimental evidence that ketonic chelate compounds are a kinetically active species in decarboxylation. They demonstrated in spectrophotometric studies also the presence of enolic chelates which did not decarboxylate[11]. Therefore complex **2** is unlikely the source of carbon dioxide. But the conformationally flexible oxaloacetate in complex **1** can circumvent the obstacle of missing orbital overlap through rotational freedom around the C²-C³ bond such positioning the C³-C⁴ σ -bond in parallel alignment with the π^* -orbital of the 2-keto group (see Figure 1 in manuscript). The demanded conformation for decarboxylation of complex **1** is adjusted by FAHD1.

Summary and working hypothesis: OAA binds initially in the enol-form to the Mg^{2+} co-factor followed by slow ketonization of the substrate. The necessary deprotonation of the enol is supported by the K123 residue of FAHD1. The driving force is the highly increased acidity of the Mg^{2+} bound enolic hydroxyle group. The conformation of the such formed 2-keto OOA Mg^{2+} complex gets under control of FAHD1 residues R106-Q109 which finally ensures parallel alignment of the orbitals involved in C-C bond cleavage.

- Additional compounds investigated for FAHD1 inhibition (Figure S4)

ID	Structure	CAS RN	IC ₅₀ (μM)		ID	Structure	CAS RN	IC ₅₀ (μM)
<i>N</i> -alkyl, <i>N</i> -arylalkyl								
S5		406190-09-8	108		S15		38188-60-2	35
S6		1849465-46-8	250		S16		89942-67-6	212
S7		1450740-20-1	inactive		S17		57727-23-8	121
S8		6345-08-0	552		S18		41374-62-3	155
S9		1218912-11-8	inactive					
ID	Structure	CAS RN	IC ₅₀ (μM)		ID	Structure	CAS RN	IC ₅₀ (μM)
Halogen substitution								
S10		69066-43-9	140		S19		none	3
S11		84944-15-0	inactive		S20		none	5
S12		868565-59-7	34		S21		none	13
S13		946744-52-1	75		S22		none	14
S14		17772-30-4	134					

- Analytical data of synthesized compounds**

NMR assignment, HRMS and melting points

2-(2-methoxy-2-oxoacetamido)benzoic acid (1a)

(RN: 174575-83-8); Mp: 184–185 °C; HRMS: calc. [C₁₀H₉NO₅]: 223.05; found: 246.0365 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.87 (s, 3H, OCH₃), 7.23–7.28 (m[dt], 1H, H-4 aniline), 7.64–7.69 (m[dt], 1H, H-5 aniline), 8.05 (dd, ³J = 7.9, ⁴J = 1.6, 1H, H-3 aniline), 8.59 (dd, ³J = 8.5, ⁴J = 0.9, 1H, H-6 aniline), 12.58 (s, 1H, NH), 13.84 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 53.62 (OCH₃), 117.03 (C-2 aniline), 119.67 (C-6 aniline), 124.08 (C-4 aniline), 131.44 (C-3 aniline), 134.41 (C-5 aniline), 139.29 (C-1 aniline), 154.22 (NHCO), 160.53 (COOMe), 169.24 (COOH).

2-(carboxyformamido)benzoic acid (1b)

(RN: 5651-01-4); Mp: 206–208 °C; HRMS: calc. [C₉H₇NO₅]: 209.0324; found: 210.034 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.0–5.0 (NHCOOCOOH in exchange with water), 7.24 (t[ddd], ³J₁ = ³J₂ = 7.7, ⁴J = 1.1, 1H, H-5), 7.66 (t[ddd], ³J₁ = 7.2, ³J₂ = 8.0, ⁴J = 1.6, 1H, H-4), 8.04 (dd, ³J₂ = 7.9, ⁴J = 1.6, H-6), 8.62 (dd, ³J₂ = 8.3, ⁴J = 0.8, 1H, H-3), 12.50 (s, sh, 1H, NHCOOCOOH), 12.8–15.0 (br, 1H, 1-COOH). ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 117.04 (C_q-1), 119.61 (C-3), 123.94 (C-5), 131.46 (C-6), 134.36 (C-4), 139.43 (C_q-2), 156.02 (NHCOOCOOH), 161.45 (NHCOOCOOH), 169.08 (1-COOH).

2-(2-(2-methoxy-2-oxoacetamido)benzamido)benzoic acid (1d)

(RN: none); Mp: 200–202 °C (MeOH); HRMS: calc. [C₁₇H₁₄N₂O₆]: 342.09; found: 343.0925 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.85 (s, 3H, OCH₃), 7.27 (dt, ³J = 7.6, ⁴J = 1.1, 1H, H-5 aryl B), 7.38 (dt, ³J = 7.6, ⁴J = 1.1, 1H, H-5 aryl A), 7.66 (m[t], 1H, H-4 aryl A), 7.69 (m[t], 1H, H-4 aryl B), 7.94 (dd, ³J = 7.9, ⁴J = 1.3, 1H, H-6 aryl A), 8.05 (dd, ³J = 8.0, ⁴J = 1.6, 1H, H-6 aryl B), 8.44 (dd, ³J = 8.4, ⁴J = 0.7, 1H, H-3 aryl B), 8.54 (dd, ³J = 8.4, ⁴J = 0.8, 1H, H-3 aryl A), 12.06 (s, 1H, NHCO), 12.14 (s, 1H, NHCOOCOME), (COOH in exchange with water in DMSO). ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 53.57 (OCH₃), 117.89 (C-1 aryl B), 120.85 (C-3 aryl B), 121.34 (C-3 aryl A), 122.83 (C-1 aryl A), 123.79 (C-5 aryl B), 124.79 (C-5 aryl A), 127.96 (C-6 aryl A), 131.25 (C-6 aryl B), 132.92 (C-4 aryl A), 134.18 (C-4 aryl B), 137.34 (C-2 aryl A), 140.14 (C-2 aryl B), 154.27 (NHCOOCOME), 160.62 (COOME), 166.41 (NHCO), 169.72 (COOH); aryl A: aryl bearing the oxaryl head; aryl B: aryl bearing the COOH function.

2-(2-(carboxyformamido)benzamido)benzoic acid (1e)

(RN: none); Mp: 215–217 °C; HRMS: calc. [C₁₆H₁₂N₂O₆]: 328.07; found: 351.0691[M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.27 (d, ³J = 7.7, 1H, H-5 aryl B), 7.37 (d, ³J = 7.7, 1H, H-5 aryl A), 7.67 (m[t], 1H, H-4 aryl A), 7.70 (m[t], 1H, H-4 aryl B), 7.94 (dd, ³J = 7.9, ⁴J = 1.3, 1H, H-6

aryl A), 8.05 (dd, $^3J = 7.9$, $^4J = 1.6$, 1H, H-6 aryl B), 8.51 (d, $^3J = 8.2$, 1H, H-3 aryl B), 8.53 (d, $^3J = 8.4$, 1H, H-3 aryl A), 12.06 (s, 1H, NHCO), 12.13 (s, 1H, NH A). $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 117.92 (C-1 aryl B), 120.87 (C-3 aryl B), 121.04 (C-3 aryl A), 122.55 (C-1 aryl A), 123.77 (C-5 aryl B), 124.57 (C-5 aryl A), 127.92 (C-6 aryl A), 131.25 (C-6 aryl B), 132.94 (C-4 aryl A), 134.18 (C-4 aryl B), 137.65 (C-2 aryl A), 140.15 (C-2 aryl B), 155.90 (NHCO B), 161.49 (COOH A), 166.47 (NHCO), 169.71 (COOH B); aryl A: aryl bearing the oxalyl part; aryl B: aryl bearing the COOH function.

3-(2-methoxy-2-oxoacetamido)benzoic acid (2a)

(RN: none); Mp: 192–193 °C.

^1H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 3.86 (s, 3H, OCH₃), 7.48 (t, $^3J = 7.9$, 1H, H-5 aryl), 7.72 (m[d], $^3J = 7.8$, 1H, H-6 aryl), 7.94 (m[d], $^3J = 8.2$, 1H, H-4 aryl), 8.41 (t, $^4J = 1.9$, 1H, H-2 aryl), 10.98 (s, 1H, NH); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 53.27 (OCH₃), 121.28 (C-2 aryl), 124.71 (C-4 aryl), 125.57 (C-6 aryl), 129.10 (C-5 aryl), 131.44 (C-1 aryl), 137.78 (C-3 aryl), 155.47 (NHCO), 160.86 (COOMe), 167.02 (COOH).

3-(Carboxyformamido)benzoic acid (2b)

(RN: 58465-46-6); Mp: 210–211 °C; HRMS: calc. [C₉H₇NO₅]: 209.0324; found: 232.0216 [M+Na]⁺.

^1H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.47 (t, $^3J_1 = ^3J_2 = 8.0$, 1H, H-5), 7.70 (dt, $^3J = 7.7$, $^4J_1 = ^4J_2 = 1.0$, 1H, H-6), 7.95 (ddd, $^3J = 8.0$, $^4J_1 = 0.7$, $^4J_2 = 1.8$, 1H, H-5), 8.44 (t, $^4J_1 = ^4J_2 = 1.7$, 1H, H-2), 10.91 (s, 1H, NHCOCOOH), 13.09 (br, COOH); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 121.13 (C-2), 124.59 (C-4), 125.38 (C-6), 129.02 (C-5), 131.39 (C-1), 138.01 (C-3), 157.11 (NHCOCOOH), 161.98 (NHCOCOOH), 167.08 (1-COOH)

4-(2-methoxy-2-oxoacetamido)benzoic acid (3a)

(RN: 1086273-37-1); Mp: 251–253 °C; HRMS: calc. [C₁₀H₉NO₅]: 223.05; found: 224.0561[M+H]⁺.

^1H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 3.86 (s, 3H, OCH₃), 7.88 (m[d], 2H, H-2, H-6 aniline), 7.93 (m[d], 2H, H-3, H-5 aniline), 11.06 (s, 1H, NH), 12.84 (br, s, 1H, COOH); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 53.30 (OCH₃), 119.90 (C-2, C-6 aniline), 126.66 (C-4 aniline), 130.28 (C-3, C-5 aniline), 141.51 (C-1 aniline), 155.59 (NHCO), 160.76 (COOMe), 166.82 (COOH).

4-(carboxyformamido)benzoic acid (3b)

(RN: 14121-56-3); Mp: 87–92 °C; HRMS: calc. [C₉H₇NO₅]: 209.03; found: 232.0209 [M+Na]⁺.

^1H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 7.88 (m, 2H, H-2, H-6 aniline), 7.92 (m, 2H, H-3, H-5 aniline), 10.99 (s, 1H, NH), COOH in rapid exchange; $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 119.75 (C-2, C-6 aniline), 126.48 (C-4 aniline), 130.27 (C-3, C-5 aniline), 141.75 (C-1 aniline), 157.38 (NHCO), 161.87 (COOH), 166.85 (Ar-COOH).

Methyl 2-((2-nitrophenyl)amino)-2-oxoacetate (4a)

(RN: none); Mp: 162–167°C; HRMS: calc. [C₉H₈N₂O₅]: 224.04; found: 247.0324 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 3.89 (s, 3H, OCH₃), 7.45 (dt, ³J = 7.9, ⁴J = 1.2, 1H, H-4 aniline), 7.80 (dt, ³J = 7.9, ⁴J = 1.4, 1H, H-5 aniline), 8.06 (dd, ³J = 8.2, ⁴J = 1.2, 1H, H-6 aniline), 8.12 (dd, ³J = 8.2, ⁴J = 1.4, 1H, H-3 aniline), 11.39 (s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 53.67 (OCH₃), 124.47 (C-6 aniline), 125.51 (C-3 aniline), 126.08 (C-4 aniline), 130.79 (C-1 aniline), 134.95 (C-5 aniline), 140.76 (C-2 aniline), 154.90 (NHCO), 160.24 (COOMe).

2-((2-nitrophenyl)amino)-2-oxoacetic acid (4b)

(RN: 77901-51-0); Mp: 148–151°C; HRMS: calc. [C₉H₆N₂O₅]: 210.03; found: 211.0394 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 7.42 (t, ³J = 7.7, 1H, H-4 aniline), 7.80 (dt, ³J = 7.6, 1H, H-5 aniline), 8.13 (d, ³J = 8.2, 1H, H-6 aniline), 8.17 (d, ³J = 8.2, 1H, H-3 aniline), 11.40 (s, 1H, NH), COOH in rapid exchange; ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 123.79 (C-3 aniline), 125.56 (C-6 aniline), 125.62 (C-4 aniline), 131.40 (C-1 aniline), 135.09 (C-5 aniline), 140.07 (C-2 aniline), 156.63 (NHCO), 161.19 (COOH).

Methyl 2-((3-nitrophenyl)amino)-2-oxoacetate (5a)

(RN: 1237427-49-4); Mp: 169–172°C; HRMS: calc. [C₉H₈N₂O₅]: 224.04; found: 247.0326 [M+Na]⁺

¹H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 3.87 (s, 3H, OCH₃), 7.65 (t, ³J = 8.1, 1H, H-5 aniline), 7.99 (m, 1H, H-4 aniline), 8.16 (m, 1H, H-6 aniline), 8.74 (m, 1H, H-2 aniline), 11.27 (s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 53.39 (OCH₃), 114.71 (C-2 aniline), 119.24 (C-4 aniline), 126.49 (C-6 aniline), 130.24 (C-5 aniline), 138.74 (C-1 aniline), 147.88 (C-3 aniline), 155.56 (NHCO), 160.43 (COOMe).

2-((3-nitrophenyl)amino)-2-oxoacetic acid (5b)

(RN: 6274-26-6); Mp: 155–162°C; HRMS: calc. [C₉H₆N₂O₅]: 210.03; found: 211.0394 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 7.66 (t, ³J = 8.1, 1H, H-5 aniline), 8.00 (m, 1H, H-4 aniline), 8.17 (m, 1H, H-6 aniline), 8.78 (m, 1H, H-2 aniline), 11.20 (s, 1H, NH), 14.41 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 114.54 (C-2 aniline), 119.06 (C-4 aniline), 126.38 (C-6 aniline), 130.21 (C-5 aniline), 138.96 (C-1 aniline), 147.91 (C-3 aniline), 157.34 (NHCO), 161.55 (COOH).

Methyl 2-((4-nitrophenyl)amino)-2-oxoacetate (6a)

(RN: 82633-22-5); Mp: 232–234°C; HRMS: calc. [C₉H₈N₂O₅]: 224.04; found: 247.0322[M+Na]⁺

¹H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 3.87 (s, 3H, OCH₃), 8.03 (m, 2H, H-2, H-6 aniline), 8.25 (m, 2H, H-3, H-5 aniline), 11.33 (s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 53.41 (OCH₃), 120.41 (C-2, C-6 aniline), 124.77 (C-3, C-5 aniline), 143.33 (C-4 aniline), 143.66 (C-1 aniline), 155.75 (NHCO), 160.37 (COOMe).

2-((4-nitrophenyl)amino)-2-oxoacetic acid (6b)

(RN: 103-94-6); Mp: 205–212 °C; HRMS: calc. [C₉H₆N₂O₅]: 210.03; found: 211.0394 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 8.05 (d, ³J = 9.2, 2H, H-2, H-6 aniline), 8.26 (d, ³J = 9.2, 2H, H-3, H-5 aniline), 11.26 (s, 1H, NH), 14.49 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 120.24 (C-2, C-6 aniline), 124.78 (C-3, C-5 aniline), 143.20 (C-4 aniline), 143.90 (C-1 aniline), 157.61 (NHCO), 161.53 (COOH).

Methyl 2-(naphthalen-2-ylamino)-2-oxoacetate (7a)

(RN: 20518-40-5) Mp: 133–136 °C; HRMS: calc. [C₁₃H₁₁NO₃]: 229.0739; found: 252.0642 [M+Na]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 3.99 (s, 3H, OCH₃), 7.45 (t, ³J = 7.5, 1H, H-6 naphthyl), 7.49 (t, ³J = 7.5, 1H, H-7 naphthyl), 7.57 (dd, ³J = 8.8, ⁴J = 2.1, 1H, H-3 naphthyl), 7.80 (d, ³J = 8.0, 1H, H-4 naphthyl), 7.84 (d, ³J = 8.5, 2H, H-5, H-8 naphthyl), 8.33 (d, ⁴J = 1.8, 1H, H-1 naphthyl), 9.04 (s, 1H, NH); ¹³CNMR (150.93 MHz, CDCl₃, 25 °C): δ = 54.25 (OCH₃), 117.33 (C-1 naphthyl), 119.37 (C-3 naphthyl), 125.84 (C-6 naphthyl), 126.95 (C-7 naphthyl), 127.77 (C-4 naphthyl), 128.01 (C-8 naphthyl), 129.27 (C-5 naphthyl), 131.29 (C-4a naphthyl), 133.70 (C-2 or C-8a naphthyl), 133.74 (C-2 or C-8a naphthyl), 153.81 (NHCO), 161.61 (COOMe).

2-(naphthalen-2-ylamino)-2-oxoacetic acid (7b)

RN: 81682-60-2); Mp: 150–155 °C; HRMS: calc. [C₁₂H₉NO₃]: 215.06; found: 238.0639 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.44 (t, ³J = 7.5, 1H, H-6 naphthyl), 7.49 (t, ³J = 7.0, 1H H-7 naphthyl), 7.81 (dd, ³J = 8.8, J = 1.9, 1H, H-3 naphthyl), 7.85 (dd[t], ³J = 7.7, 2H, H-5, H-8 naphthyl), 7.88 (d, ³J = 8.8, 1H, H-4 naphthyl), 8.43 (s, 1H, H-1 naphthyl), 10.90 (s, 1H, NH), 14.29 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 116.87 (C-1 naphthyl), 120.56 (C-3 naphthyl), 125.25 (C-6 naphthyl), 126.57 (C-7 naphthyl), 127.52 (C-5 naphthyl), 127.59 (C-8 naphthyl), 128.40 (C-4 naphthyl), 130.34 (C-4a naphthyl), 133.13 (C-8a naphthyl), 135.38 (C-2 naphthyl), 157.32 (NHCO), 162.21 (COOH).

Methyl 2-(naphthalen-1-ylamino)-2-oxoacetate (8a)

(RN: 254751-07-0); Mp: 75–76 °C.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.87 (s, 3H, OCH₃), 7.48–7.55 (m, 1H, *), 7.83–7.87 (m, 2H, *), 7.91–7.95 (m, 4H, *), 10.90 (s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 54.19 (OCH₃), 123.42 (*), 124.33 (*), 126.41 (*), 127.29 (*), 127.35 (*), 128.12 (*), 129.02 (*), 129.15 (*), 132.44 (*), 134.46 (*), 157.67 (NHCO), 161.82 (COOMe); *Assignment not possible due to mixture of conformers.

2-(naphthalen-1-ylamino)-2-oxoacetic acid (8b)

(RN: 21660-76-4); Mp: 176–177 °C.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.48–7.56 (m, 4H, *), 7.81–7.86 (m, 2H, *), 7.91–7.95 (m, 1H, *) 10.73 (s, 1H, NH), COOH in rapid exchange; ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 123.27 (*), 123.94 (*), 126.44 (*), 127.26 (*), 127.31 (*), 127.83 (*), 129.01 (*), 129.05 (C_q*), 132.72 (C_q*),

134.46 (C_q^*), 159.46 (NHCO), 163.06 (COOH); *Assignment not possible due to mixture of conformers.

Methyl 2-oxo-2-(pyridin-3-ylamino)acetate (9a)

(RN: 480452-68-4); Mp: 118–121 °C; HRMS: calc. [C₈H₈N₂O₃]: 180.0535; found: 203.0427 [M+Na]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 3.92 (s, 3H, NHCOCOOCH₃), 7.28 (dd, 3J_1 = 8.3, 3J_2 = 4.8, 1H, H-5), 8.20 (ddd, 3J = 8.3, 4J_1 = 2.2, 4J_2 = 1.2, 1H, H-4), 8.38 (dd, 3J = 4.8, 4J = 1.2, 1H, H-6), 8.67 (d, 4J = 2.4, 1H, H-2), 9.04 (s, br, 1H, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 54.39 (NHCOCOOCH₃), 123.98 (C-5), 127.22 (C-4), 133.30 (C_q-3), 141.49 (C-2), 146.72 (C-6), 154.29 (NHCOCOOCH₃), 161.03 (NHCOCOOCH₃).

3-(2-Methoxy-2-oxoacetamido)pyridine 1-oxide (11a)

Mp: 184–191 °C; HRMS: calc. [C₈H₈N₂O₄]: 196.0484; found: 219.0376 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.86 (s, 3H, NHCOCOOCH₃), 7.41 (dd, 3J_1 = 8.5, 3J_2 = 6.5, 1H, H-5), 7.72 (ddd, 3J = 8.5, 4J_1 = 1.7, 4J_2 = 0.7, 1H, H-4), 8.04 (ddd, 3J = 6.5, 4J_1 = 1.7, 4J_2 = 0.7, 1H, H-6), 8.73 (dd[t], 4J_1 = 1.7, 4J_2 = 1.7, 1H, H-2), 11.21 (s, sh, 1H, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 53.50 (NHCOCOOCH₃), 117.04 (C-4), 126.31 (C-5), 131.12 (C-2), 134.98 (C-6), 137.07 (C_q-3), 155.57 (NHCOCOOCH₃), 160.09 (NHCOCOOCH₃).

3-(Carboxyformamido)pyridine 1-oxide (11b)

Mp: 236 °C; HRMS: calc. [C₇H₆N₂O₄]: 182.0328; found: 183.0400 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.41 (dd, 3J_1 = 8.5, 3J_2 = 6.5, 1H, H-5), 7.74 (ddd, 3J = 8.5, 4J_1 = 1.6, 4J_2 = 0.6, 1H, H-4), 8.04 (ddd, 3J = 6.5, 4J_1 = 1.5, 4J_2 = 0.7, 1H, H-6), 8.76 (m[t], br, 2x 4J = 1.6, 1H, H-2), 11.13 (s, sh, 1H, NHCOCOOH), 14.6 (s, vbr, 1H, NHCOCOOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 117.13 (C-4), 126.31 (C-5), 130.98 (C-2), 134.82 (C-6), 137.27 (C_q-3), 157.39 (NHCOCOOH), 161.21 (NHCOCOOH).

Methyl 2-oxo-2-(pyridin-2-ylamino)acetate (10a)

(RN: 54166-60-8); Mp: 103–104 °C.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 3J = 8.4, 4J = 1.8, 1H, H-4), 3.98 (s, 3H, NHCOCOOCH₃), 7.13 (ddd, 3J_1 = 4.9, 3J_2 = 7.4, 4J = 0.8, 1H, H-5), 7.76 (ddd, 3J_1 = 7.5, 3J_2 = 7.8, 4J = 1.8, 1H, H-4), 8.24 (d, br, 3J = 8.3, 1H, H-3), 8.35 (d, br, 3J = 4.8, 1H, H-6), 9.41 (s, br, 1H, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 54.26 (NHCOCOOCH₃), 114.40 (C-3), 121.17 (C-5), 138.73 (C-4), 148.51 (C-6), 149.93 (C_q-2), 154.10 (NHCOCOOCH₃), 160.78 (NHCOCOOCH₃).

2-(2-Methoxy-2-oxoacetamido)pyridine 1-oxide (12a)

Mp: 209–210 °C;

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.89 (NHCOCOOCH₃), 7.26 (ddd, 3J_1 = 3J_2 = 7.5, 4J = 1.8, 1H, H-5), 7.52 (ddd, 3J_1 = 3J_2 = 7.5, 4J = 1.4, 1H, H-4), 8.27 (dd, 3J = 8.3, 4J = 1.8, 1H, H-3), 8.47 (m[dd], 3J = 6.5, 4J = 1.3, 1H, H-6), 11.13 (s, br, 1H, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 53.81 (NHCOCOOCH₃), 114.25 (C-3), 120.74 (C-5), 127.81 (C-4), 137.57 (C-6), 142.29 (C_q-2), 154.43 (NHCOCOOCH₃), 159.47 (NHCOCOOCH₃).

2-(Carboxyformamido)pyridine 1-oxide (12b)

(RN: none); Mp: 198 °C (sharp, dec.); HRMS: calc. [C₇H₆N₂O₄]: 182.0328; found: 183.0400 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.24 (ddd[t], 1H, ³J₁ = 8.1, ³J₁ = 6.5, ⁴J = 1.7, 1H, H-5), 7.50 (ddd[t], 1H, ³J₁ = 8.0, ³J₁ = 8.0, ⁴J = 1.1, 1H, H-4), 8.27 (dd, ³J = 8.3, ⁴J = 1.7, 1H, H-3), 8.46 (dd, ³J = 6.5, ⁴J = 0.7, 1H, H-6), 11.15 (s, vbr, 1H, NHCOCOOH), (NHCOCOOH in exchange with H₂O-DMSO-d₆); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 114.03 (C-3), 120.57 (C-5), 127.81 (C-4), 137.57 (C-6), 142.46 (C_q-2), 155.94 (NHCOCOOH), 160.45 (NHCOCOOH).

Methyl 2-oxo-2-(quinolin-3-ylamino)acetate (13a)

(RN: 1710262-07-9); Mp: 181-182 °C (ethyl acetate); HRMS: calc. [C₁₂H₁₀N₂O₃]: 230.0691; found: 231.0764 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.89 (s, 3H, NHCOCOOCH₃), 7.59 (ddd, ³J₁ = 8.0, ³J₂ = 6.9, ⁴J = 1.1, 1H, H-6), 7.69 (ddd, ³J₁ = 8.3, ³J₂ = 6.7, ⁴J = 1.1, 1H, H-7), 7.96, 7.98 (2xd [t], br, 2H, H-5, H-8), 8.78 (d, ⁴J = 2.4, 1H, H-4), 9.14 (d, ⁴J = 2.4, 1H, H-2), 11.29 (s, br, 1H, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 53.38 (NHCOCOOCH₃), 124.17 (C-4), 127.24 (C-6), 127.46 (C_q-4a), 128.00 (C-5), 128.63 (C-8), 128.64 (C-7), 131.36 (C_q-3), 144.81 (C_q-8a), 145.25 (C-2), 155.82 (NHCOCOOCH₃), 160.60 (NHCOCOOCH₃).

Methyl 2-oxo-2-(quinolin-2-ylamino)acetate (14a)

(RN: none); Mp: 131-134 °C; HRMS: calc. [C₁₂H₁₀N₂O₃], 230.0691; found: 253.0584 [M+Na]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 4.00 (s, 3H, NHCOCOOCH₃), 7.50 (m[t], br, ³J_{app} = 7.5, 1H, H-6), 7.70 (m[t; ddd], br, ³J_{app} = 7.6, 1H, H-7), 7.81 (d, br, ³J = 8.0, 1H, H-5), 7.88 (d, ³J = 8.5, 1H, H-8), 8.23 (d, ³J = 9.0, 1H, H-4), 8.42 (d, ³J = 9.0, 1H, H-3), 9.59 (s, br, 1H, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 54.33 (NHCOCOOCH₃), 113.90 (C-3), 126.05 (C-6), 126.88 (C_q-4a), 127.75 (C-8), 127.95 (C-5), 130.48 (C-7), 139.13 (C-4), 146.80 (C_q-8a), 149.29 (C_q-2), 154.43 (NHCOCOOCH₃), 160.80 (NHCOCOOCH₃).

3-(2-Methoxy-2-oxoacetamido)quinoline 1-oxide (15a)

(RN: none); Mp: 219-220 °C (methanol); HRMS: calc. [C₁₂H₁₀N₂O₄]: 246.0641; found: 269.0533 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.89 (s, 3H, NHCOCOOCH₃), 7.69 (m[ddd], 1H, H-6), 7.72 (m[ddd], 1H, H-7), 8.05 (dd, ³J = 8.0, ⁴J = 1.2, 1H, H-5), 8.39 (s, br, 1H, H-4), 8.44 (m[d], 1H, H-8), 8.97 (s, br, 1H, H-2), 11.27 (s, br, 1H, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 53.49 (NHCOCOOCH₃), 114.39 (C-4), 118.67 (C-8), 128.64 (C-5), 129.32 (C-6), 129.35 (C-7), 129.55 (C_q-4a), 129.90 (C-2), 132.18 (C_q-3), 138.30 (C_q-8a), 155.66 (NHCOCOOCH₃), 160.24 (NHCOCOOCH₃).

3-(Carboxyformamido)quinoline 1-oxide (15b)

(RN: none); Mp: 236-237 °C (disintegration)

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.70 (ddd[t], 1H, H-6), 7.73 (ddd[t], 1H, H-7), 8.06 (dd, ³J = 7.9, ⁴J = 1.4, 1H, H-5), 8.43 (d, ⁴J = 1.3, 1H, H-4), 8.45 (d, br, ³J = 8.4, 1H, H-8), 9.00 (d, ⁴J = 1.5, 1H, H-2), 11.23 (s, sh, 1H, NHCOCOOH), (NHCOCOOH in exchange with H₂O in DMSO-d₆);

$^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 114.42 (C-4), 118.70 (C-8), 128.67 (C-5), 129.34 (C-6), 129.40 (C-7), 129.64 (C_q-4a), 130.01 (C-2), 132.43 (C_q-3), 138.19 (C_q-8a), 157.46 (NHCOCOOH), 161.38 (NHCOCOOH).

2-(2-Methoxy-2-oxoacetamido)quinoline 1-oxide (16a)

(RN: none)

^1H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 4.05 (s, 3H, NHCOCOOCH₃), 7.64 (m[t], 3J = 7.4, 1H, H-6), 7.84 (m[t], 3J = 7.7, 1H, H-7), 7.88 (d, 3J = 9.4, 1H, H-4), 7.90 (d, 3J = 8.6, 1H, H-5), 8.60 (d, 3J = 9.22, 1H, H-3), 8.69 (d, 3J = 8.72, 1H, H-8), 11.60 (s, br, 1H, NHCOCOOCH₃); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, CDCl₃, 25 °C): δ = 54.46 (NHCOCOOCH₃), 112.90 (C-3), 119.14 (C-8), 126.59 (C_q-4a), 127.81 (C-6), 128.22 (C-4), 128.39 (C-5), 131.63 (C-7), 139.50 (C_q-8a), 140.65 (C_q-2), 154.76 (NHCOCOOCH₃), 159.45 (NHCOCOOCH₃).

2-(Carboxyformamido)quinoline 1-oxide (16b)

(RN: none); Mp: 119.5–125 °C; HRMS: calc. [C₁₁H₈N₂O₄]: 232.0484; found: 255.0376 [M+Na]⁺.

^1H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.69 (ddd[t], 3J = 7.8, 1H, H-6), 7.88 (ddd[t], 3J = 7.5, 1H, H-7), 8.09 (d, 3J = 8.3, 1H, H-4), 8.11 (d, 3J = 9.3, 1H, H-5), 8.46 (d, 3J = 9.1, 1H, H-3), 8.49 (d, 3J = 8.6, 1H, H-8), 10.8–12.4 (s, br, 1H, NHCOCOOH), (NHCOCOOH in exchange with H₂O in DMSO-d₆); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 112.24 (C-3), 118.27 (C-8), 126.02 (C_q-4a), 127.44 (C-6), 127.48 (C-4),

128.83 (C-5), 131.44 (C-7), 138.75 (C_q-8a), 140.25 (C_q-2), 156.41 (NHCOCOOH), 160.52 (NHCOCOOH).

Methyl 4-oxo-4*H*-benzo[d][1,3]oxazine-2-carboxylate (17a)

(RN: 3603-20-1); Mp: 173–175 °C; HRMS: calc. [C₁₀H₇NO₄]: 205.04; found: 206.0447 [M+H]⁺.

^1H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.94 (s, 3H, OCH₃), 7.77 (dt, 3J = 7.6, 4J = 1.0, H-5 benzoxazine), 7.83 (dd, 3J = 8.0, 4J = 0.7, H-8 benzoxazine), 8.02 (dt, 3J = 7.8, 4J = 1.4, H-7 benzoxazine), 8.20 (dd, 3J = 7.8, 4J = 1.2, H-5 benzoxazine); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 53.58 (OCH₃), 118.37 (C-4a benzoxazine), 128.05 (C-8 benzoxazine), 128.27 (C-5 benzoxazine), 130.81 (C-6 benzoxazine), 137.12 (C-7 benzoxazine), 144.68 (C-8a benzoxazine), 146.99 (C-2 benzoxazine), 158.08 (COOMe), 158.15 (C-4 benzoxazine).

Methyl 2-((2-carbamoylphenyl)amino)-2-oxoacetate (18a)

(RN: 69065-88-9); Mp: 164–166 °C.

HRMS: calc. [C₁₀H₁₀N₂O₄]: 222.06; found: 223.0614 [M+H]⁺.

^1H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.85 (s, 3H, OCH₃), 7.24 (dt, 3J = 7.7, 4J = 1.1, 1H, H-4 aryl), 7.57 (dt, 3J = 7.8, 4J = 1.3, 1H, H-5 aryl), 7.82 (s, 1H, NH₂), 7.87 (dd, 3J = 8.0, 4J = 1.3, 1H, H-3 aryl), 8.36 (s, 1H, NH), 8.52 (dd, 3J = 8.4, 4J = 0.9, 1H, H-6 aryl), 13.04 (s, 1H, NH₂); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 53.66 (OCH₃), 120.20 (C-6 aryl), 120.47 (C-2 aryl), 124.07 (C-4 aryl), 128.91 (C-3 aryl), 132.67 (C-5 aryl), 138.14 (C-1 aryl), 154.24 (NHCO), 160.71 (COOMe), 170.51 (COONH₂).

Methyl 4-oxo-1,4-dihydroquinazoline-2-carboxylate (19a)

(RN: 63569-82-4); Mp: 201–204 °C; Lit.: 202–203 °C; HRMS: calc. [C₁₀H₈N₂O₃]: 204.05; found: 205.0584 [M+H]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 3.95 (s, 3H, OCH₃), 7.22 (dt, ³J = 7.7, ⁴J = 1.0, H-6 quinazoline), 7.59 (m[d], 1H, H-5 quinazoline), 7.60 (m[dt], 1H, H-7 quinazoline), 8.39 (dd, ³J = 8.9, ⁴J = 1.0, 1H, H-8 quinazoline), 9.32 (br, s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 54.54 (OCH₃), 103.18 (C-4a quinazoline), 115.68 (C-8a quinazoline), 121.14 (C-8 quinazoline), 125.67 (C-6 quinazoline), 132.75 (C-5 quinazoline), 134.48 (C-7 quinazoline), 138.71 (C-4 quinazoline), 154.04 (C-2 quinazoline), 160.44 (COOMe).

4-oxo-1,4-dihydroquinazoline-2-carboxylic acid (19b)

(RN: 29113-34-6); Mp: 213–215 °C; HRMS: calc. [C₉H₆N₂O₃]: 190.04; found: 213.0379.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.43 (dt, ³J = 7.7, ⁴J = 0.9, 1H, H-6 quinazoline), 7.64 (d, ³J = 8.2, 1H, H-8 quinazoline), 7.74 (dt, ³J = 7.9, ⁴J = 1.5, 1H, H-7 quinazoline), 7.87 (dd, ³J = 7.9, ⁴J = 1.3, 1H, H-5 quinazoline), 10.89 (s, 1H, NH), COOH in rapid exchange; ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 108.23 (C-4a quinazoline), 116.57 (C-4 quinazoline), 125.89 (C-8 quinazoline), 126.85 (C-6 quinazoline), 133.29 (C-5 quinazoline), 134.03 (C-7 quinazoline), 138.93 (C-8a quinazoline), 157.30 (COOH), 161.46 (C-2 quinazoline).

Methyl 2-((2-methoxyphenyl)amino)-2-thioxoacetate (20a)

(RN: none); Mp: 94–96 °C; HRMS: calc. [C₁₀H₁₁NO₃S]: 225.05; found: 248.0353 [M+Na]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 3.95 (s, 3H, Ar-OCH₃), 3.99 (s, 3H, OCH₃), 6.97 (dd, ³J = 8.2, ⁴J = 1.0, 1H, H-3 aniline), 7.02 (dt, ³J = 7.7, ⁴J = 1.0, 1H, H-5 aniline), 7.22–7.27 (m[dt], ³J = 8.0, ⁴J = 1.4, 1H, H-4 aniline, CDCl₃), 9.26 (dd, ³J = 8.2, ⁴J = 1.4, 1H, H-6 aniline), 11.29 (br, s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 54.99 (OCH₃), 56.16 (Ar-OCH₃), 110.44 (C-3 aniline), 120.39 (C-6 aniline), 120.54 (C-5 aniline), 127.60 (C-4, C-1 aniline[127.54]), 149.83 (C-2 aniline), 160.42 (COOMe), 176.95 (NHCS).

2-((2-methoxyphenyl)amino)-2-thioxoacetic acid (20b)

(RN: 7267-58-5); Mp: 133–135 °C; HRMS: calc. [C₉H₉NO₃S]: 211.03; found: 234.0196 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.84 (s, 3H, OCH₃), 7.00 (dt, ³J = 7.7, ⁴J = 1.2, 1H, H-5 aniline), 7.16 (dd, ³J = 8.4, ⁴J = 1.0, 1H, H-3 aniline), 7.31 (dt, ³J = 7.9, ⁴J = 1.6, 1H, H-4 aniline), 8.07 (dd, ³J = 8.0, ⁴J = 1.4, 1H, H-6 aniline), 11.73 (s, 1H, NH), COOH in rapid exchange; ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 55.90 (OCH₃), 111.97 (C-6 aniline), 120.07 (C-5 aniline), 124.35 (C-3 aniline), 126.60 (C-1 aniline), 128.26 (C-4 aniline), 151.97 (C-2 aniline), 162.88 (COOH), 186.40 (NHCS).

Methyl 2-((3-methoxyphenyl)amino)-2-thioxoacetate (21a)

(RN: none) Mp: 86–89 °C; HRMS: calc. [C₁₀H₁₁NO₃S]: 225.05; found: 248.0353 [M+Na]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 3.83 (s, 3H, OCH₃), 3.99 (s, 3H, OCH₃), 6.86 (dd, ³J = 8.1, ⁴J = 2.0, 1H, H-4 aniline), 7.33 (t, ³J = 8.0, 1H, H-5 aniline), 7.36 (d, ³J = 8.2, 1H, H-6 aniline), 7.89 (t, ⁴J = 2.1, 1H, H-2 aniline), 10.57 (s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 55.18 (OCH₃),

55.62 (OCH₃), 107.26 (C-2 aniline), 113.52 (C-4 aniline), 114.29 (C-6 aniline), 129.99 (C-5 aniline), 138.88 (C-1 aniline), 160.13 (C-3 aniline), 160.24 (COOMe), 178.50 (NHCS).

2-((3-methoxyphenyl)amino)-2-thioxoacetic acid (21b)

(RN: 276693-17-5); Mp: 97–98°C; HRMS: calc. [C₉H₉NO₃S]: 211.03; found: 234.0196 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 3.75 (s, 3H, OCH₃), 6.87 (dd, ³J = 8.3, ⁴J = 2.5, 1H, H-4 aniline), 7.34 (t, ³J = 8.1, 1H, H-5 aniline), 7.47 (dd, ³J = 8.0, ⁴J = 2.0, 1H, H-6 aniline), 7.68 (t, ⁴J = 2.1, 1H, H-2 aniline), 12.27 (s, 1H, NH), COOH in rapid exchange; ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 55.24 (OCH₃), 108.37 (C-2 aniline), 112.24 (C-4 aniline), 114.94 (C-6 aniline), 129.59 (C-5 aniline), 139.45 (C-1 aniline), 159.22 (C-3 aniline), 163.91 (COOH), 187.53 (NHCS).

Methyl 2-((4-methoxyphenyl)amino)-2-thioxoacetate (22a)

(RN: none); Mp: 134–143°C; HRMS: calc. [C₁₀H₁₁NO₃S]: 225.05; found: 248.0353 [M+Na]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25°C): δ = 3.83 (s, 3H, OCH₃), 3.98 (s, 3H, Ar-OCH₃), 6.94 (m, 2H, H-3, H-5 aniline), 7.90 (m, 2H, H-2, H-6 aniline), 10.51 (s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 23°C): δ = 55.07 (Ar-OCH₃), 55.64 (OCH₃), 114.27 (C-3, C-5 aniline), 123.74 (C-2, C-6 aniline), 130.90 (C-1 aniline), 158.53 (C-4 aniline), 160.52 (COOMe), 177.73 (NHCS).

2-((4-methoxyphenyl)amino)-2-thioxoacetic acid (22b)

(RN: 946-61-2); Mp: 139–140°C; HRMS: calc. [C₉H₉NO₃S]: 211.03; found: 234.0196 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 3.77 (s, 3H, OCH₃), 6.98 (d, ³J = 8.8, 2H, H-3, H-5 aniline), 7.83 (d, ³J = 8.4, 2H, H-2, H-6 aniline), 12.19 (s, 1H, NH), COOH in rapid exchange; ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25°C): δ = 55.35 (OCH₃), 113.78 (C-3, C-5 aniline), 124.41 (C-2, C-6 aniline), 131.37 (C-1 aniline), 157.51 (C-4 aniline), 164.00 (COOH), 186.19 (NHCS).

N1-(pyridin-2-yl)oxalamide (23)

(RN: 52781-00-7); Mp: 156–157 °C (chloroform)

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 6.11 (s, br, 1H, NHCOCONH_aH_b), 7.12 (dd, ³J₁ = 7.2, ³J₂ = 5.2, 1H, H-5), 7.44 (s, br, 1H, NHCOCONH_aH_b), 7.76 (ddd, ³J₁ = 7.2, ³J₂ = 7.2, ⁴J = 1.5, 1H, H-4), 8.20 (m[d]), 1H, H-3), 8.37 (m[dd]), 1H, H-6), 9.72 (s, br, 1H, NHCOCONH_aH_b); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 114.10 (C-3), 121.01 (C-5), 138.55 (C-4), 148.63 (C-6), 149.94 (C_q-2), 157.58 (NHCOCONH₂), 161.53 NHCOCONH₂).

N¹-(naphthalen-2-yl)oxalamide (24)

(RN: 21775-72-4); Mp: 143–147°C; HRMS: calc. [C₁₀H₈NO₃]: 214.07; found: [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25°C): δ = 7.44 (dt, ³J = 7.4, ⁴J = 1.1, 1H, H-6 naphthyl), 7.49 (dt, ³J = 7.4, ⁴J = 1.1, 1H, H-7 naphthyl), 7.83 (d, ³J = 8.2, 1H, H-8 naphthyl), 7.86 (d, ³J = 8.1, 1H, H-5 naphthyl), 7.88 (m[d], 2H, H-3, H-4 naphthyl), 8.04 (s, 1H, NH₂), 8.34 (s, 1H, NH₂), 8.47 (s, 1H, H-1 naphthyl), 10.77 (br, s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, DMSO- d₆, 25°C): δ = 116.76 (C-1 naphthyl), 120.65 (C-3 naphthyl), 125.19 (C-6 naphthyl), 126.53 (C-7 naphthyl), 127.52 (C-5 naphthyl), 127.56 (C-8 naphthyl), 128.35 (C-4 naphthyl), 130.31 (C-4a naphthyl), 133.14 (C-8a naphthyl), 135.37 (C-2 naphthyl), 159.15 (COONH₂), 162.17 (NHCO).

N-(6-methylpyridin-2-yl)-2-oxo-2-(pyrrolidin-1-yl)acetamide (25)

(RN: 1210298-59-1; no references); Mp: 111–112.5 °C; HRMS: calc. [C₁₂H₁₅N₃O₂]: 233.1164; found: 234.1237 [M+H]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 1.87 (m[q], 2H, ³J_{1,4} = 6.8, 2H, H-4'), 1.98 (m[q], 2H, ³J_{1,4} = 6.8, 2H, H-3'), 2.47 (s, 3H, 6-CH₃), 3.61 (t, ³J₁ = ³J₂ = 7.0, 2H, H-5'), 4.04 (t, ³J₁ = ³J₂ = 6.8, 2H, H-2'), 6.93 (d, ³J = 7.4, 1H, H-5), 7.60 (t, ³J₁ = ³J₂ = 7.7, 1H, H-4), 7.99 (d, ³J = 8.1, 1H, H-3), 9.80 (s, 1H, NHCOCCON); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 23.55 (CH₂-4'), 24.24 (6-CH₃), 27.03 (CH₂-3'), 48.43 (CH₂-5'), 49.00 (CH₂-2'), 110.69 (C-3), 120.01 (C-5), 138.58 (C-4), 149.77 (C_q-6), 157.56 (C_q-2), 158.63 (NHCOCCON), 158.73 (NHCOCCON).

2-(2-*tert*-Butoxy-2-oxoacetamido)benzoic acid (26)

(RN: none); Mp: 139–142 °C (at 142 °C dec.); HRMS: calc. [C₁₃H₁₅NO₅]: 265.0950; found: 288.0842 [M+H]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 1.63 (2, 9H, NHCOCOOC(CH₃)₃), 7.23 (m[ddd], ³J₁ = ³J₂ = 7.7, ⁴J = 0.8, 1H, H-5), 7.67 (m[ddd], ³J₁ = ³J₂ = 7.9, ⁴J = 1.4, 1H, H-4), 8.20 (dd, ³J = 8.0, ⁴J = 1.4, 1H, H-6), 8.81 (m[d], ³J = 8.4, 1H, H-3), 9.2–11.0 (br, 1H, COOH), 12.32 (s, 1H, NHCOCOOC(CH₃)₃); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 27.87 (NHCOCOOC(CH₃)₃), 85.32 (NHCOCOOC(CH₃)₃), 115.20 (C_q-1), 120.74 (C-3), 124.22 (C-5), 32.24 (C-6), 136.00 (C-4), 140.45 (C_q-2), 155.86 (NHCOCOOC(CH₃)₃), 158.85 (NHCOCOOC(CH₃)₃).

3-(2-*tert*-Butoxy-2-oxoacetamido)benzoic acid (27)

(RN: none); Mp: 148 °C (dec.); HRMS: calculated [C₁₃H₁₅NO₅]: 265.0950; found: 288.0842 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 1.53 (s, 9H, NHCOCOOC(CH₃)₃), 7.45 (t, ³J₁ = ³J₂ = 7.7, 1H, H-5), 7.71 (d, br, ³J = 7.8, 1H, H-6), 7.91 (d, br, ³J = 7.9, 1H, H-4), 8.34 (s[t], br, 1H, H-2), 10.81 (s, br, 1H, NHCOCOOC(CH₃)₃), (COOH in exchange with water in DMSO-d₆); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 27.45 (NHCOCOOC(CH₃)₃), 83.81 (NHCOCOOC(CH₃)₃), 121.28 (C-2), 124.19 (C-4), 125.45 (C-6), 128.90 (C-5), 132.64 (C_q-1), 137.70 (C_q-3), 156.59 (NHCOCOOC(CH₃)₃), 159.82 (NHCOCOOC(CH₃)₃), 167.38 (COOH)

4-(2-*tert*-Butoxy-2-oxoacetamido)benzoic acid (28)

(RN: 614760-53-1); Mp: oil; HRMS: calc. [C₁₃H₁₅NO₅]: 265.0950; found: 288.0842 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 1.53 (s, 9H, NHCOCOOC(CH₃)₃); 7.84, 7.85, (m, AA', 2H, H-3,5 phenyl), 7.92, 7.93 (m, BB', 2H, H-2,6 phenyl), 10.94 (NHCOCOOC(CH₃)₃), (COOH in exchange with water in DMSO-d₆); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 27.46 (NHCOCOOC(CH₃)₃), 84.00 (NHCOCOOC(CH₃)₃), 119.86 (C-3,5 phenyl), 126.57 (C_q-1), 130.33 (C-2,6 phenyl), 141.62 (C_q-4), 156.75 (NHCOCOOC(CH₃)₃), 159.73 (NHCOCOOC(CH₃)₃), 166.88 (phenyl-1-COOH).

N¹,N²-bis(5-methylpyridin-2-yl)oxalamide (29)

(RN: 349401-68-9); Mp: 230–235 °C; HRMS: calc. [C₁₄H₁₄N₄O₂]: 270.1117; found: 271.1190 [M+H]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 2.33 (s, 6H, 2x 5-CH₃), 7.58 (dd, ³J = 8.5, ⁴J = 2.1, 2H, 2x H-3), 8.15 (d, ³J = 8.5, 2H, 2x H-4), 8.20 (s, br, 2H, 2x H-6), 9.73 (s, br, 2H, 2x NHCOCOCONH);

$^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, CDCl_3 , 25 °C): δ = 18.12 (5-CH₃), 113.68 (C-3), 130.64 (C_q-5), 139.02 (C-6), 147.74 (C-4), 148.54 (C_q-2), 157.42 (NHCOCOCONH).

N¹,N²-di(pyridin-2-yl)oxalamide (30)

(RN: 20172-97-8); Mp: 163–164 °C.

^1H NMR (600.25 MHz, CDCl_3 , 25 °C): δ = 7.14 (ddd, 3J_1 = 7.3, 3J_2 = 5.0, 4J = 1.8, 1H, 2x H-5), 7.78 (ddd, 3J_1 = 3J_2 = 7.8, 4J = 1.8, 1H, 2x H-4), 8.27 (d, 3J = 7.8, 1H, 2x H-3), 8.39 (m[d], 3J = 4.9, 2H, 2x H-6); 9.81 (s, 2H, NHCOCOCONH); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, CDCl_3 , 25 °C): δ = 114.27 (C-3), 121.13 (C-5), 138.65 (C-4), 148.64 (C-6), 149.93 (C_q-2), 157.51 (NHCOCOCONH).

N,N'-Di-pyridin-3-yl-oxalamide (31)

(RN: 39642-61-0); Mp: 250–255 °C; HRMS: calc. [C₁₂H₁₀N₄O₂]: 242.24; found: 243.0876 [M+H⁺]⁺.

^1H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.44 (m, 4J = 4.3, 1H, 2x H-5), 8.24 (ddd, 3J = 8.4, 4J_1 = 3.3, 4J_2 = 1.7, 1H, 2x H-4), 8.38 (dd, 3J = 4.8, 4J = 0.9, 1H, 2x H-6), 9.05 (d, 4J = 2.2, 1H, 2x H-2), 11.16 (s, 2H, 2x NHCOCOONH); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, CDCl_3 , 25 °C): δ = 123.55 (C-5), 127.59 (C-4), 134.37 (C_q-3), 142.28 (C-2), 145.52 (C-6), 158.58 (NHCOCOONH).

N-Pyridin-2-yl-N'-p-tolyl-oxalamide (32)

(RN: 301344-55-8); Mp: 176–178 °C; HRMS: calc. [C₁₄H₁₄N₃O₃]: 255.28; found: 256.1083 [M+H⁺]⁺.

^1H NMR (600.25 MHz, CDCl_3 , 25 °C): δ = 2.35 (s, 3H, 4-CH₃), 7.19 (m[d]; AA' – part of AA'BB' – spin system 3J = 8.3, H-2, H-5), 7.56 (m[d]; BB' – part of AA'BB' spin system 3J = 8.2, H-3, H-6), 7.14 ([m]t, 3J_1 = 6.1, 3J_2 = 6.1, 1H, H-5'), 7.77 (t, br, 3J_1 = 7.8, 3J_2 = 7.8, 1H, H-4'), 8.24 (d, br, 3J_1 = 8.3, 2H, H-3'), 8.39 (d, br, 3J_1 = 4.9, 2H, H-6'), 9.26 (s, br, 1H, NHCOCOONH), 9.88 (s, br, 1H, NHCOCOONH); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, CDCl_3 , 25 °C): δ = 21.18 (4-CH₃), 119.97 (C-3), 129.98 (C-5), 133.85 (C_q-4), 135.46 (C_q-1), 114.16 (C-3'), 121.10 (C-5'), 138.53 (C-4'), 148.54 (C-6'), 149.71 (C_q-2'), 156.77 (NHCOCOONH), 158.39 (NHCOCOONH).

N-Pyridin-2-yl-N'-m-tolyl-oxalamide (33)

(RN: 920210-16-8; no reference); Mp: 134–135 °C; HRMS: calc. [C₉H₁₀N₂O₃]: 194.0691; found: 217.0584 [M+Na⁺]⁺

^1H NMR (600.25 MHz, CDCl_3 , 25 °C): δ = 2.35 (s, 3H, 3-CH₃), 7.01 (d, 3J = 7.5, H-4), 7.25 (t, 3J_1 = 7.8, 3J_2 = 7.8, 1H, H-5) 7.47 (d, 3J = 8.2, 1H, H-6), 7.50 (s, 1H, H-2), 9.33 (s, br, 1H, NHCOCOONH), 9.93 (s, br, 1H, NHCOCOONH), 7.12 ([m]ddd, 3J_1 = 6.2, 3J_2 = 4.0, 4J = 1.0, 1H, H-5'), 7.74 (ddd, 3J_1 = 8.0, 3J_2 = 6.0, 4J = 1.9, 1H, H-4'), 8.23 (d, br, 3J = 8.4, 1H, H-3'), 8.38 (ddd, br, $^3J_{10}$ = 4.8, 3J_2 = 1.9, 4J = 0.8, 1H, H-6'); $^{13}\text{C}\{\text{H}\}$ NMR (150.93 MHz, CDCl_3 , 25 °C): δ = 114.12 (C-3'), 120.19 (C-5'), 138.49 (C-4'), 148.66 (C-6'), 149.96 (C_q-2'), 157.06 (NHCOCOOCH₃), 158.19 (NHCOCOOCH₃), 21.63 (3-CH₃), 117.18 (C-2), 121.06 (C-6), 126.55 (C-4), 128.81 (C-5), 136.25 (C_q-3), 139.30 (C_q-1).

N-Pyridin-3-yl-N'-pyridin-2-yl-oxalamide (34)

(RN: 1796089-38-7); Mp. 163–165 °C; HRMS: calc. [C₁₂H₁₀N₄O₂]: 242.24; found: 243.0879 [M+H⁺]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 7.14 (ddd, ³J₁ = 4.9, ³J₂ = 7.0, ⁴J = 0.5, 1H, H-5), 7.78 (ddd, ³J₁ = 7.5, ³J₂ = 7.8, ⁴J = 1.8, 1H, H-4), 8.27 (d, br, ³J = 8.3, 1H, H-3), 8.39 (d, br, ³J = 4.8, 1H, H-6), 9.81 (s, br, 1H, NHCOCOOCH₃), 7.34 (m, ⁴J = 4.6, 1H, H-5'), 8.23 (d, ⁴J = 8.3, 1H, H-4'), 8.46 (dd, ³J = 4.8, ⁴J = 1.3, 1H, H-6'), 8.80 (d, ⁴J = 2.7, 1H, H-2'); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 114.40 (C-3), 121.17 (C-5), 138.73 (C-4), 148.51 (C-6), 149.93 (C_q-2), 157.26 (NHCOCOOCH₃), 158.00 (NHCOCOOCH₃), 124 (C-5'), 127 (C-4'), 133 (C_q-3'), 141 (C-2'), 146 (C-6').

N-(1-Oxy-pyridin-2-yl)-N'-*m*-tolyl-oxalamide (35)

(RN: none); Mp: 253–255 °C; HRMS: calc. [C₁₄H₁₃N₃O₃]: 271.28; found: 294.0850 [M+Na⁺]⁺.

¹H NMR (600.25 MHz, DMSO- d₆, 25 °C): δ = 2.32 (s, 3H, 3-CH₃), 7.01 (d, ³J = 7.6, H-4), 7.27 (t, ³J = 2.7, 1H, H-5), 7.28 (s, 1H, H-2), 7.65 (d, ³J = 8.3, 1H, H-6), 10.95 (s, br, 1H, NHCOCOONH), 11.46 (s, br, 1H, NHCOCOONH), 7.54 (m[ddd], ³J₁ = 8.7, ³J₂ = 8.1, ⁴J = 1.1, 1H, H-5'), 7.71 (s, br, 1H, H-4'), 8.34 (dd, ³J = 8.4, ⁴J = 1.9, 1H, H-6'), 8.48 (dd, ³J = 6.6, ⁴J = 1.2, 1H, H-3'); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 21.19 (3-CH₃), 117.90 (C-6), 121.26 (C-5), 125.57 (C-4), 127.65 (C-2), 137.18 (C_q-3), 137.63 (C_q-1), 156.91 (NHCOCOONH), 158.02 (NHCOCOONH), 120.46 (C-4'), 128.60 (C-5'), 138.01 (C-3'), 113.90 (C-6'), 142.34 (C_q-2').

N-(1-Oxy-pyridin-2-yl)-N'-*p*-tolyl-oxalamide (36)

(RN: none); Mp: nd (contains residual 3-chlorobenzoic acid); HRMS: calc. [C₁₄H₁₃N₃O₃]: 271.2780; found: 272.1029 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO- d₆, 25 °C): δ = 2.31 (s, 3 H, 4-CH₃), 7.00 (d, br, 1 H), 7.26 (t, br, 1 H), 7.54 (d, br, 2 H), 7.65 (d, br, 1H), 7.68 – 7.72 (m, br, 2 H), 7.88 – 7.91 (m, br, 3 H), 8.34 (dd, 1 H), 8.48 (d, br, 1H), 10.95 (s, br, 1H), 11.46 (s, br, 1H), 13.00 – 13.6 (br, 1H). ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 21.20 (4-CH₃), 114.01 (CH), 118.00 (CH), 120.66 (CH), 121.26 (CH), 125.72 (CH), 127.66 (CH), 127.92 (CH), 128.61 (CH), 128.83 (CH), 130.66 (CH), 132.71 (CH), 132.95 (C_q), 133.35 (C_q), 137.20 (C_q), 137.64 (CH), 138.03 (C_q), 142.37 (C_q), 156.92 (C_q), 158.08 (C_q), 166.08 (C_q).

N-(1-Oxy-pyridin-3-yl)-N'-*m*-tolyl-oxalamide (37)

(RN: none); Mp: 231–232 °C; HRMS: calc. [C₁₄H₁₃N₃O₃]: 271.28; found: 272.1026 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO- d₆, 25 °C): δ = 2.31 (s, 3H, 3-CH₃), 6.99 (d, ³J = 7.5, H-4), 7.26 (t, ³J₁ = 7.7, ³J₂ = 7.7, 1H, H-5) 7.66 (d, ³J = 8.8, 1H, H-6), 7.67 (s, 1H, H-2), 10.80 (s, br, 1H, NHCOCOONH), 11.26 (s, br, 1H, NHCOCOONH), 7.43 (m[dd], ³J₁ = 8.5, ³J₂ = 7.7, ⁴J = 1.3, 1H, H-3'), 7.82 (dd, ³J = 8.5, ⁴J = 1.7, 1H, H-6'), 8.05 (dd, ³J = 6.5, ⁴J = 1.6, 1H, H-2'), 8.85 (t, ³J₁ = 1.6, ³J₂ = 1.6, 1H, H-4'); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 21.24 (3-CH₃), 116.80 (C-6), 121.15 (C-2), 125.35 (C-4), 128.58 (C-5), 136.97 (C_q-3), 137.94 (C_q-1), 157.62 (NHCOCOONH), 159.41 (NHCOCOONH), 126.00 (C-3'), 117.60 (C-6'), 131.16 (C-4'), 137.15 (C-2').

Methyl 2-((6-methylpyridin-2-yl)amino)-2-oxoacetate (S1)

(RN: 1566684-50-1; no references); Mp: 68–69 °C; HRMS: calc. [C₉H₁₀N₂O₃]: 194.0691; found: 217.0584 [M+Na]⁺; [FAHD1: IC₅₀ = 33 μM].

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 2.48 (s, 3H, CH₃-6), 3.96 (s, 3H, NHCOCOOCH₃), 6.98 (d, ³J = 7.5, 1H, H-5), 7.64 (t, ³J = 7.8, 1H, H-4), 8.04 (d, ³J = 8.1, 1H, H-3), 9.32 (s, br, 1H, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 24.15 (CH₃-6), 54.22 (COOCH₃), 111.23 (C-3), 120.72 (C-5), 138.96 (C-4), 149.12 (C_q-6), 154.00 (NHCOCOOCH₃), 157.61 (C_q-2), 160.76 (NHCOCOOCH₃).

Methyl 2-((2-methoxyphenyl)amino)-2-oxoacetate (S2)

(RN: 113449-16-4); Mp: 85–87 °C; HRMS: calc. [C₁₀H₁₁NO₄]: 209.07; found: 210.0773 [M+H]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 3.91 (s, 3H, Ar-OCH₃), 3.96 (s, 3H, OCH₃), 6.91 (dd, ³J = 8.3, ⁴J = 1.0, 1H H-3 aniline), 6.99 (dt, ³J = 7.9, ⁴J = 1.0, 1H H-5 aniline), 7.13 (dt, ³J = 7.9, ⁴J = 1.5, 1H H-4 aniline), 8.40 (dd, ³J = 8.3, ⁴J = 1.5, 1H H-6 aniline), 9.47 (s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 54.05 (OCH₃), 55.90 (Ar-OCH₃), 110.22 (C-3 aniline), 120.05 (C-6 aniline), 121.19 (C-5 aniline), 125.47 (C-4 aniline), 126.10 (C-1 aniline), 148.56 (C-2 aniline), 153.51 (NHCO), 161.45 (COOMe).

Methyl 2-((3-methoxyphenyl)amino)-2-oxoacetate (S3)

(RN: 103448-86-8); Mp: 70–72 °C; HRMS: calc. [C₁₀H₁₁NO₄]: 209.07; found: 210.0773 [M+H]⁺.

¹H NMR (600.25 MHz, CDCl₃, 23 °C): δ = 3.81 (s, 3H, Ar-OCH₃), 3.96 (s, 3H, OCH₃), 6.74 (dd, ³J = 8.3, ⁴J = 2.1, 1H H-4 aniline), 7.12 (dd, ³J = 8.3, ⁴J = 2.1, 1H H-6 aniline), 7.26 (t, ³J = 8.3, 1H, H-5 aniline), 7.36 (t, ⁴J = 2.1, 1H, H-2 aniline), 8.86 (s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 23 °C): δ = 54.21 (OCH₃), 55.47 (Ar-OCH₃), 105.72 (C-2 aniline), 111.52 (C-4 aniline), 112.18 (C-6 aniline), 130.08 (C-5 aniline), 137.48 (C-1 aniline), 153.68 (NHCO), 160.33 (C-3 aniline), 161.54 (-COOMe).

Methyl 2-((4-methoxyphenyl)amino)-2-oxoacetate (S4)

(RN: 24439-54-1); Mp: 140–141 °C; HRMS: calc. [C₁₀H₁₁NO₄]: 209.07; found: 210.0773 [M+H]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 3.80 (s, 3H, Ar-OCH₃), 3.95 (s, 3H, OCH₃), 6.89 (d, ³J = 9.0, 2H, H-3, H-5 aniline), 7.56 (d, ³J = 9.0, 2H, H-2, H-6 aniline), 8.81 (s, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 54.12 (OCH₃), 55.59 (Ar-OCH₃), 114.45 (C-3, C-5 aniline), 121.55 (C-2, C-6 aniline), 129.50 (C-1 aniline), 153.47 (NHCO), 157.32 (C-4 aniline), 161.74 (COOMe).

2-oxo-2-(*o*-tolylamino)acetic acid (S5)

(RN: 406190-09-8); Mp: 179–180 °C; HRMS: calc. [C₉H₉NO₃]: 179.0582; found: [M+Na]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 2.35 (s, 3H, 2-CH₃ of tolyl), 7.18 (m[t], 1H, H-5 phenyl), 7.25 (m[d], 1H, H-3 phenyl), 7.27 (m[t], 1H, H-4 phenyl), 7.94 (m[d], 1H, H-3 phenyl), 8.20–8.80 (br, 1H, COOH), 8.99 (s, br, 1H, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 17.54 (2-CH₃-phenyl), 121.83 (C-6 tolyl), 126.85 (C-4 tolyl), 127.27 (C-5 tolyl), 129.01 (C_q-2 tolyl), 131.02 (C-3 tolyl), 133.61 (C_q-1 tolyl), 155.13 (COCOOH), 160.48 (COCOOH).

2-(((1*R*,2*S*)-2-hydroxy-2,3-dihydro-1*H*-inden-1-yl)amino)-2-oxoacetic acid (S6)

(RN: 1849465-46-8); Mp: 204–205 °C; HRMS: calc. [C₁₁H₁₁NO₄]: 221.07; found: 244.0580 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 2.85 (m[dd], 1H, ²J = 16.1, ³J = 1.5, H_{eq} indene), 3.10 (m[dd], 1H, ²J = 16.2, ³J = 5.1, H_{ax} indene), 4.48 (m[dt], 1H H-2 indene), 5.18 (m[q], 1H, H-1 indene), 5.38 (br, s, 1H, OH), 7.16–7.20 (m, 2H, H-5 indene, H-7 indene), 7.20–7.24 (m, 1H, H-6 indene), 7.25 (m[d], 1H H-4 indene), 8.18 (d, 1H, ³J = 8.6, NH), 14.07 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 39.76 (C-3 indene), 56.90 (C-1 indene), 71.63 (C-2 indene), 124.22 (C-4, C-5, C-6, C-7 indene), 125.02 (C-4, C-5, C-6, C-7 indene), 126.53 (C-4, C-5, C-6, C-7 indene), 127.72 (C-4, C-5, C-6, C-7 indene), 140.79 (C-4a indene), 141.15 (C-7a indene), 158.19 (NHCO), 162.02 (COOH).

Methyl 2-(*tert*-butylamino)-2-oxoacetate (S7)

(RN: 1450740-20-1 (patent); Mp: oil; HRMS: calc. [C₇H₁₃NO₃]: 159.0895; found: 182.0788 [M+Na]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 1.39 (s, 9H, (CH₃)₃CNH), 3.86 (s, 3H, (COOCH₃), 6.96 (s, br, 1H, (CH₃)₃CNH); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 28.29 ((CH₃)₃CNH), 52.06 ((CH₃)₃CNH), 53.70 (COOCH₃), 155.35 (NHCOCOOCH₃), 161.96 (NHCOCOOCH₃).

2-(benzylamino)-2-oxoacetic acid (S8)

(RN: 6345-08-0); Mp: 126–127 °C; HRMS: calc. [C₉H₉NO₃]: 179.06; found: 202.1647 [M+Na]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 4.52 (d, ³J = 6.2, 2H, phenyl-CH₂), 7.27–7.30 (m, 2H, H-2,6 benzyl), 7.31–7.35 (m, 1H, H-4 benzyl), 7.34–7.39 (m, 2H, H-3,5 benzyl), 7.68 (s, br, 1H, NHCO), 7.80–8.20 (br, COOH); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 47.02 (phenyl-CH₂), 128.10 (C-2,6 benzyl), 128.44 (C-4 benzyl), 129.15 (C-3,5 benzyl), 135.85 (C-1 benzyl), 157.48 (NHCO), 159.97 (COOH).

Methyl 2-((1*S*,2*R*)-1-hydroxy-1-phenylpropan-2-yl)amino)-2-oxoacetate (S9)

(RN: none); Mp: 112–115 °C; HRMS: calc. [C₁₂H₁₅NO₄]: 237.10; found: 260.0941[M+Na]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 1.04 (d, ³J = 6.8, 3H, CHCH₃), 2.83 (d, ³J = 3.0, 1H, OH), 3.88 (s, 3H, OCH₃), 4.29 (m, 1H, CHCH₃), 4.92 (s, 1H, CHOH), 7.29 (m, 1H, H-4, aryl), 7.35 (m[d], 4H, H-2, 3, 5, 6 aryl), 7.41 (br, d, ³J = 8.3, 1H, NH); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 13.60 (CHCH₃), 51.40 (CHCH₃), 53.76 (OCH₃), 75.43 (CHOH), 126.12 (C-2, 3, 5, 6 aryl), 127.96 (C-4 aryl), 128.54 (C-2, 3, 5, 6 aryl), 140.40 (C-1 aryl), 156.16 (NHCO), 161.21 (COOMe).

2-((4-fluorophenyl)amino)-2-oxoacetic acid (S10)

(RN: 69066-43-9); Mp: 155–159 °C; HRMS: calc. [C₈H₆FNO₃]: 183.03; found: 206.0232 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.13–7.23 (m, br, 2H, H-3, H-5 aniline), 7.75–7.83 (m, br, 2H, H-2, H-6 aniline), 10.79 (s, 1H, NH), 14.24 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 115.40 (d, ²J = 22.3, C-3, C-5 aniline), 122.21 (d, ³J = 7.9, C-2, C-6 aniline), 134.17 (C-1 aniline), 156.88 (NHCO), 158.75 (d, ¹J = 241.1, C-4 aniline), 162.09 (COOH).

2-((2-fluorophenyl)amino)-2-oxoacetic acid (S11)

(RN: 84944-15-0); Mp: 162–165 °C; HRMS: calc. [C₈H₆FNO₃]: 183.03; found: 206.0224 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.18–7.24 (m, 1H, H-6 aniline), 7.24–7.33 (m, 2H, H-4, H-5 aniline), 7.60–7.69 (m, 1H, H-3 aniline), 10.36 (s, 1H, NH), 14.12 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 115.86 (d, ²J = 19.5, C-3 aniline), 124.41 (C-1 aniline), 124.50 (d, ⁴J = 3.7, C-5 aniline), 125.73 (C-6 aniline), 127.28 (d, ³J = 7.7, C-4 aniline), 154.95 (d, ¹J = 248.8, C-2 aniline), 157.31 (NHCO), 161.78 (COOH).

2-((2-bromophenyl)amino)-2-oxoacetic acid (S12)

(RN: 868565-59-7); Mp: 123–125 °C; HRMS: calc. [C₈H₆BrNO₃]: 242.95; found: 265.9431 [M+Na]⁺.

¹H NMR (700.40 MHz, DMSO-d₆, 25 °C): δ = 7.20 (dt, ³J = 7.6, ²J = 1.2, 1H, H-4 aniline), 7.43 (dd, ³J = 8.1, ²J = 1.3, 1H, H-5 aniline), 7.71 (dt, ³J = 7.6, ²J = 1.2, 1H, H-3 aniline), 7.81 (dd, ³J = 8.1, ²J = 1.3, 1H, H-6 aniline), 10.14 (s, 1H, NH), 14.29 (br, s, 1H, COOH); ¹³C{¹H}NMR (176.11 MHz, DMSO-d₆, 25 °C): δ = 117.37 (C-2 aniline), 125.26 (C-6 aniline), 127.65 (C-4 aniline), 128.44 (C-5 aniline), 132.76 (C-3 aniline), 134.99 (C-1 aniline), 156.55 (NHCO), 161.68 (COOH).

2-((3-bromophenyl)amino)-2-oxoacetic acid (S13)

(RN: 946744-52-1); Mp: 146–150 °C; HRMS: calc. [C₈H₆BrNO₃]: 242.95; found: 265.9430 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.31 (m, 2H, H-4, H-5 aniline), 7.76 (m, 1H, H-6 aniline), 8.07 (s, 1H, H-2 aniline), 10.84 (s, 1H, NH), 14.36 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 119.09 (C-6 aniline), 121.46 (C-3 aniline), 122.53 (C-2 aniline), 127.06 (C-4 aniline), 130.74 (C-5 aniline), 139.47 (C-1 aniline), 157.86 (NHCO), 161.83 (COOH).

2-((2,4-dichlorophenyl)amino)-2-oxoacetic acid (S14)

(RN: 17772-30-4); Mp: 125–128 °C; HRMS: calc. [C₈H₅Cl₂NO₃]: 232.96; found: 255.9541 [M+Na]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 7.48 (dd, ³J = 8.7, ⁴J = 2.3, 1H, H-5 aniline), 7.74 (d, ⁴J = 2.3, 1H, H-3 aniline), 7.80 (d, ³J = 8.7, 1H, H-6 aniline), 10.26 (s, 1H, NH), 13.95 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 126.55 (C-6 aniline), 127.96 (C-5 aniline), 128.06 (C-4 aniline), 129.13 (C-3 aniline), 130.58 (C-2 aniline), 132.88 (C-1 aniline), 156.97 (NHCO), 161.50 (COOH).

2-((3-hydroxyphenyl)amino)-2-oxoacetic acid (S15)

(RN: 38188-60-2); Mp: 212–214 °C; HRMS: calc. [C₈H₇NO₄]: 181.04; found: 204.0280 [M+Na]⁺.

¹H NMR (700.40 MHz, DMSO-d₆, 25 °C): δ = 6.53 (d, ³J = 7.8, 1H, H-4 aniline), 7.10 (t, ³J = 7.9, 1H, H-5 aniline), 7.14 (d, ³J = 8.3, 1H H-6 aniline), 7.31 (d, ⁴J = 2.0, 1H, H-2 aniline), 9.48 (s, 1H, NH), 10.54 (br, s, 1H, ArOH), 14.12 (br, s, 1H, COOH); ¹³C{¹H}NMR (176.11 MHz, DMSO-d₆, 25 °C): δ = 107.40 (C-2 aniline), 111.12 (C-6 aniline), 111.71 (C-4 aniline), 129.42 (C-5 aniline), 138.69 (C-1 aniline), 156.86 (NHCO), 157.56 (C-3 aniline), 162.26 (COOH).

2-((2-hydroxyphenyl)amino)-2-oxoacetic acid (S16)

(RN: 89942-67-6); Mp: 250–254 °C; HRMS: calc. [C₈H₇NO₄]: 181.04; found: 204.0275 [M+Na]⁺.

¹H NMR (700.40 MHz, DMSO-d₆, 25 °C): δ = 6.83 (dt, ³J = 7.6, ⁴J = 1.1, 1H, H-5 aniline), 6.92 (dd, ³J = 8.1, ⁴J = 1.2, 1H, H-3 aniline), 7.00 (dt, ³J = 7.7, ⁴J = 1.5, 1H, H-4 aniline), 8.02 (dd, ³J = 7.9, ⁴J = 1.2, 1H, H-6 aniline), 9.59 (s, 1H, NH), 10.26 (br, s, 1H, ArOH), 14.38 (br, s, 1H, COOH); ¹³C{¹H}NMR (176.11 MHz, DMSO-d₆, 25 °C): 115.04 (C-3 aniline), 119.24 (C-5 aniline), 120.22 (C-6 aniline), 124.99 (C-1 aniline), 125.36 (C-4 aniline), 147.20 (C-2 aniline), 155.39 (NHCO), 161.91 (COOH).

2-((2-methoxyphenyl)amino)-2-oxoacetic acid (S17)

(RN: 57727-23-8); Mp: 148–152 °C; HRMS: calc. [C₉H₉NO₄]: 195.05; found: 196.0618 [M+H]⁺.

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.88 (s, 3H, Ar-OCH₃), 6.98 (t, ³J = 7.7, 1H H-5 aniline), 7.11 (d, ³J = 7.1, 1H H-3 aniline), 7.16 (t, ³J = 7.9, 1H H-4 aniline), 8.08 (d, ³J = 7.8, 1H H-6 aniline), 9.63 (s, 1H, NH), 14.48 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 55.98 (Ar-OCH₃), 111.24 (C-3 aniline), 120.15 (C-6 aniline), 120.59 (C-5 aniline), 125.50 (C-4 aniline), 125.79 (C-1 aniline), 149.09 (C-2 aniline), 155.72 (NHCO), 161.90 (COOH).

2-((4-methoxyphenyl)amino)-2-oxoacetic acid (S18)

(RN: 41374-62-3); Mp: 155–160 °C; HRMS: calc. [C₉H₉NO₄]: 195.05; found: 196.0616 [M+H]⁺

¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 3.74 (s, 3H, Ar-OCH₃), 6.92 (d, ³J = 9.0, 2H, H-3, H-5 aniline), 7.69 (d, ³J = 9.0, 2H, H-2, H-6 aniline), 10.61 (s, 1H, NH), 14.14 (br, s, 1H, COOH); ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 55.22 (Ar-OCH₃), 113.87 (C-3, C-5 aniline), 121.80 (C-2, C-6 aniline), 130.82 (C-1 aniline), 156.11 (NHCO), 156.47 (C-4 aniline), 162.34 (COOH).

2-(2-(*tert*-butoxy)-2-oxoacetamido)-5-methylpyridine 1-oxide (S19)

(RN: none; 1:1 clathrate with 3-chlorobenzoic acid); Mp1 (plates): 157–158 °C (thermal *t*-butyl cleavage), formation of needles; Mp2: 200–202 °C; HRMS: calc. [C₁₂H₁₆N₂O₄]: 252.1110; found: 275.1002 [M+Na]⁺.

Parent compound S19: ¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 1.60 (s, 9H, NHCOCOOC(CH₃)₃), 2.34 (s, 3H, CH₃-5), 7.29 (dd, 1H, H-4), 8.31 (s, br, 1H, H-6), 8.36 (d, ³J = 6.7, 1H, H-3), 11.14 (s, br, 1H, NHCOCOOC(CH₃)₃); ¹H resonances of complexed 3-chlorobenzoic acid: 7.38 (t, ³J₁ = ³J₂ = 7.8, 1H, H-5'), 7.53 (ddd, ³J = 7.9, ⁴J₁ = 2.0, ⁴J₂ = 1.0, 1H, H-4'), 7.96 (dt, ³J = 7.9, ⁴J₁ = ⁴J₂ = 1.2, 1H, H-6'), 8.05 (m[t]), ⁴J₁ = 2.0, ⁴J₂ = 1.0, 1H, H-2'), 8.5–10.5 (vbr, 1H, COOH);

Parent compound S19: ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 18.21 (CH₃-5), 27.81 (NHCOCOOC(CH₃)₃), 86.04 (NHCOCOOC(CH₃)₃), 114.86 (C-3), 130.94 (C_q-5), 131.03 (C-4), 137.68 (C-6), 140.86 (C_q-2), 155.42 (NHCOCOOC(CH₃)₃), 157.67 (NHCOCOOC(CH₃)₃); ¹³C resonances of complexed 3-chlorobenzoic acid: 128.23 (C-6'), 129.80 (C-2'), 130.20 (C-5'), 132.09 (C_q-1'), 133.29 (C-4'), 134.59 (C_q-3'), 169.00 (COOH).

tert-butyl 2-((5-methylpyridin-2-yl)amino)-2-oxoacetate (S20)

(RN: none); Mp: < 50 °C; HRMS: calc. [C₁₂H₁₆N₂O₃]: 236.1161; found: 237.1234 [M+H]⁺.

¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 1.58 (s, 9H, COO(CH₃)₃), 2.30 (s, 3H, CH₃-5), 7.54 (dd, ³J = 8.4, ⁴J = 2.1, 1H, H-4), 8.12 (d, ³J = 8.4, 1H, H-3), 8.14 (m[dq]), 1H, H-6), 9.33 (s, sh, 1H, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 18.02 (5-CH₃), 27.81 (COOC(CH₃)₃), 85.29 (COOC(CH₃)₃), 113.71 (C-3), 130.39 (C_q-5), 139.13 (C-4), 147.94 (C_q-2), 148.30 (C-6), 155.15 (NHCOCOOCH₃), 159.12 (NHCOCOOCH₃).

5-bromo-2-(2-(*tert*-butoxy)-2-oxoacetamido)pyridine 1-oxide (S21)

(RN: none; 1:1 clathrate with 3-chlorobenzoic acid); Mp: 166-187 °C; HRMS: calc. [C₁₁H₁₃BrN₂O₄]: 316.0059; found: 338.9951 (M+Na)⁺.

Parent compound S21: ¹H NMR (600.25 MHz, DMSO-d₆, 25 °C): δ = 1.61 (s, 9H, NHCOCOOCH₃), 7.59 (dd, ³J = 9.1, ⁴J = 1.8, 1H, H-4), 8.40 (d, ³J = 9.1, 1H, H-3), 8.58 (d, ⁴J = 1.8, 1H, H-6), 11.09 (s, br, 1H, NHCOCOOCH₃); ¹H resonances of complexed 3-chlorobenzoic acid: δ = 7.41 (t, ³J₁ = ³J₂ = 7.9, 1H, H'-5), 7.56 (m[dt]), ³J = 7.8, ⁴J₁ = 1.8, ⁴J₂ = 0.9, 1H, H'-4), 7.98 (dt, ³J = 7.7, ⁴J₁ = ⁴J₂ = 1.2, 1H, H'-6), 8.07 (m[t]), ⁴J₁ = 2.0, ⁴J₂ = 1.0, 1H, H'-2), 9.2-10.0 (br, 1H, COOH);

Parent compound S21: ¹³C{¹H}NMR (150.93 MHz, DMSO-d₆, 25 °C): δ = 27.80 (NHCOCOOCH₃), 86.47 (NHCOCOOCH₃), 113.51 (C_q-5), 115.41 (C-3), 132.62 (C-4), 139.06 (C-6), 142.40 (C_q-2), 155.46 (NHCOCOOCH₃), 157.40 (NHCOCOOCH₃); ¹³C resonances of complexed 3-chlorobenzoic acid: δ = 128.36 (C-6'), 129.91 (C-2'), 130.31 (C-5'), 131.46 (C_q-1'), 133.71 (C-4'), 134.73 (C_q-3'), 169.87 (COOH).

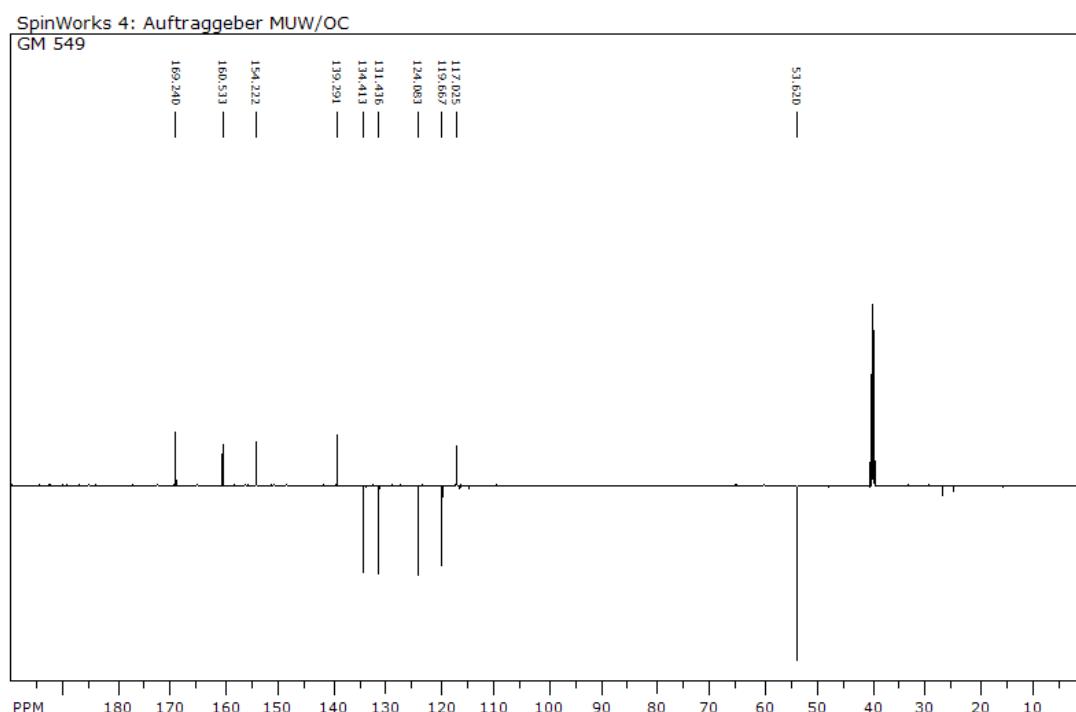
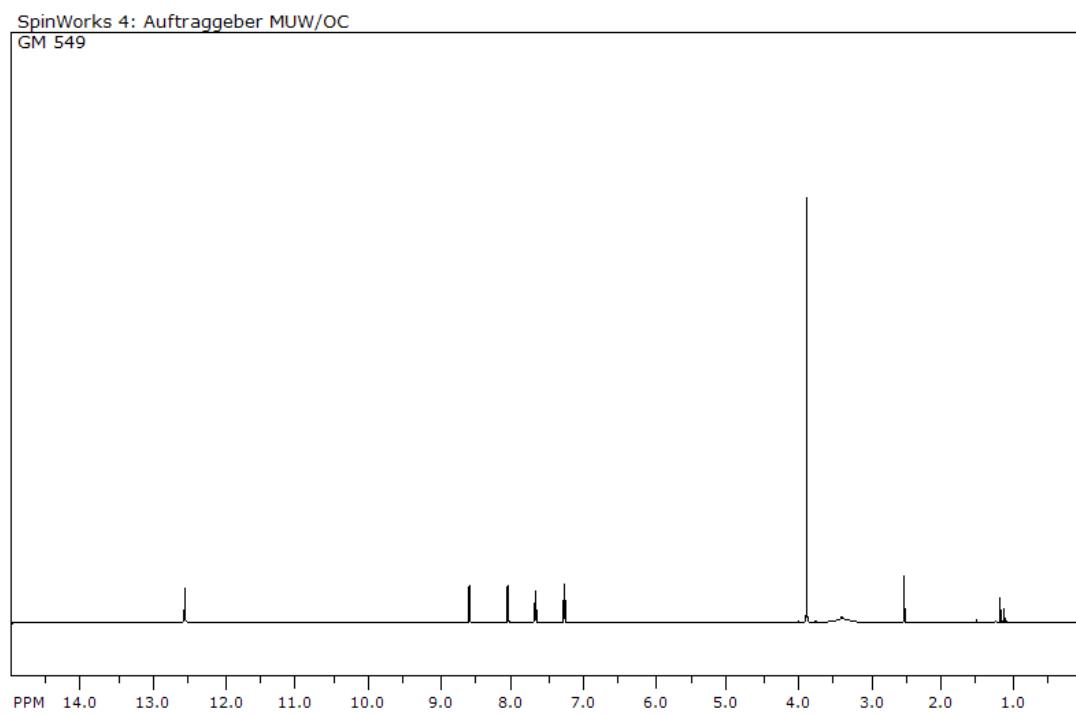
2-(2-(*tert*-butoxy)-2-oxoacetamido)-6-(methoxycarbonyl)pyridine 1-oxide (S22)

(RN: none); Mp: oil; HRMS: calc. [C₁₃H₁₆N₂O₆]: 296.1008; found: 319.0901 (M+Na)⁺.

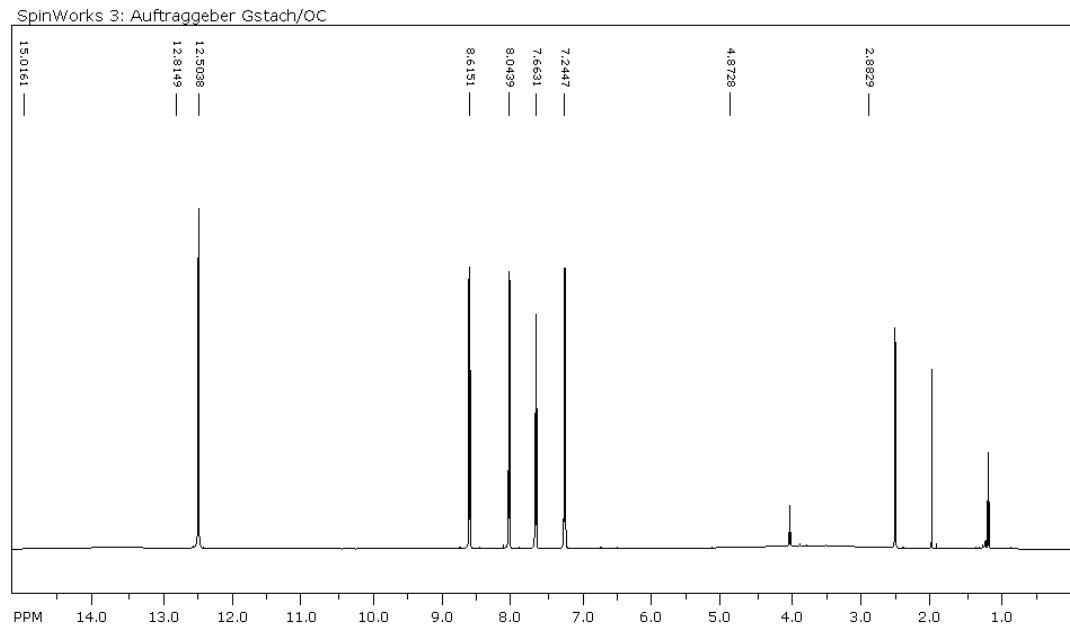
¹H NMR (600.25 MHz, CDCl₃, 25 °C): δ = 1.58 (s, 9H, NHCOCOOCH₃), 3.99 (s, 3H, 6-COOCH₃), 7.38-7.43 (m, 2H, AB-part of ABX, H-4,5), 8.49-8.56 (m, 1H, X-part of ABX, H-3), 11.29 (s, br, NHCOCOOCH₃); ¹³C{¹H}NMR (150.93 MHz, CDCl₃, 25 °C): δ = 27.75 (NHCOCOOCH₃), 53.63 (6-COOCH₃), 86.23 (NHCOCOOCH₃), 116.48 (C-3), 120.79 (C-5), 127.38 (C-4), 139.79 (C_q-6), 144.12 (C_q-2), 155.58 (NHCOCOOCH₃), 157.01 (NHCOCOOCH₃), 161.46 (6-COOCH₃).

- Graphical representation of NMR-spectra

2-(2-methoxy-2-oxoacetamido)benzoic acid (1a)

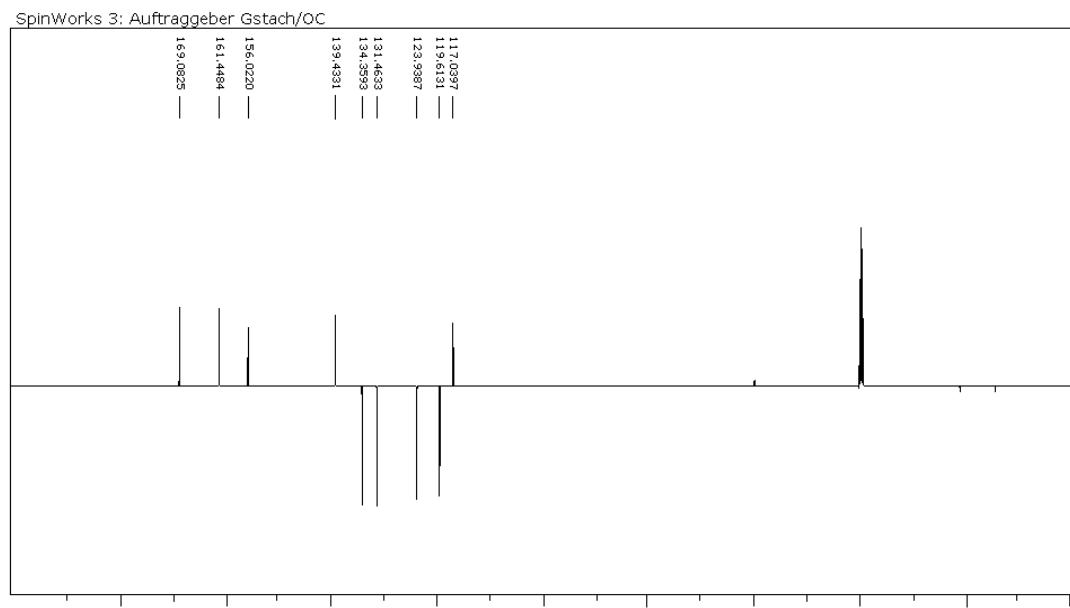


2-(carboxyformamido)benzoic acid (1b)



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number of scans: 16

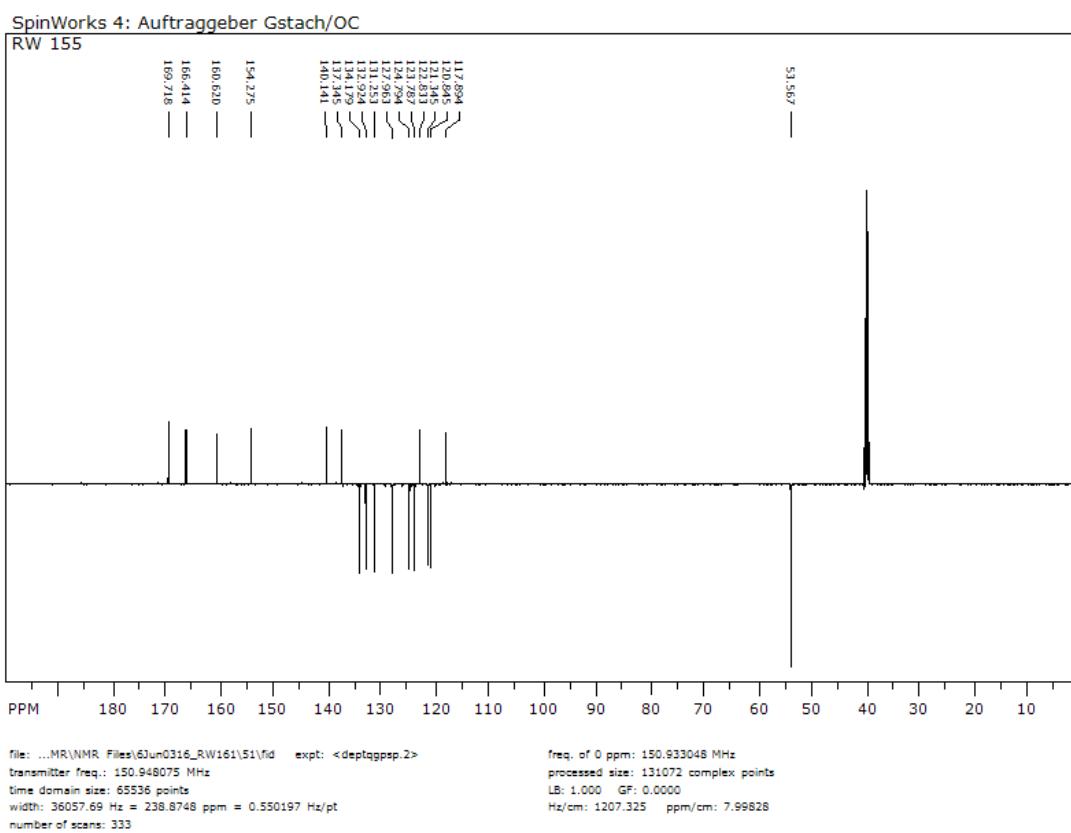
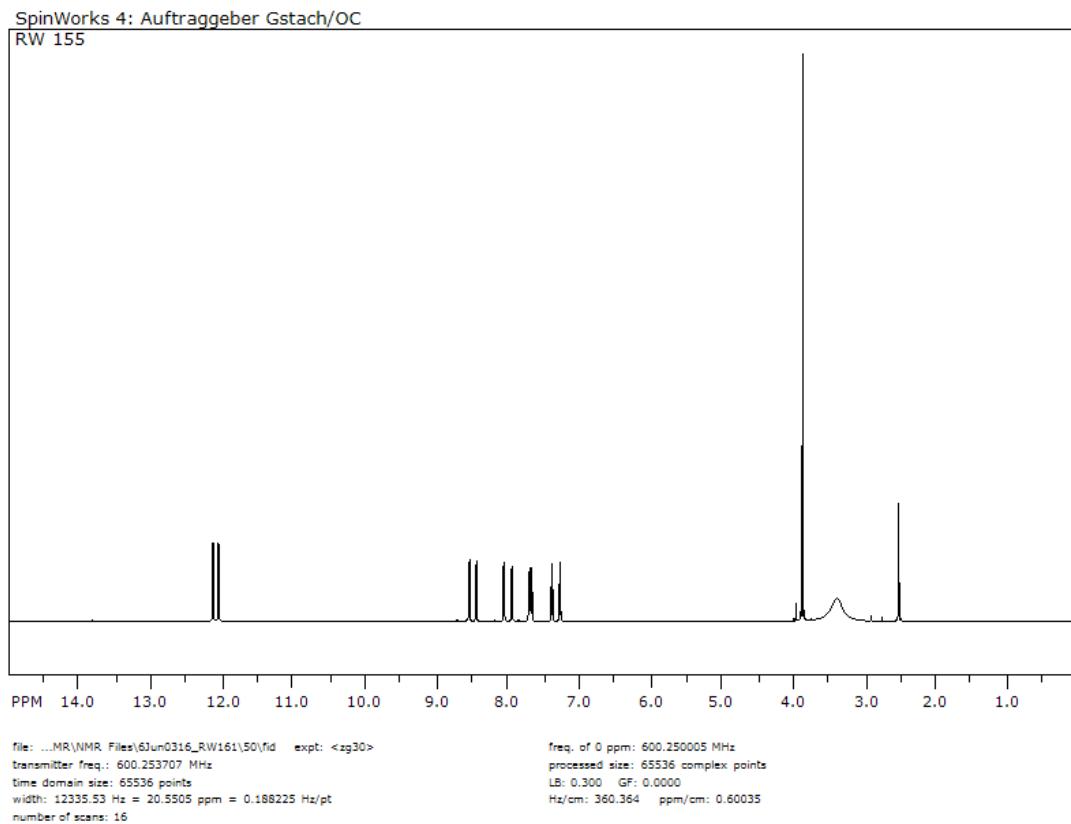
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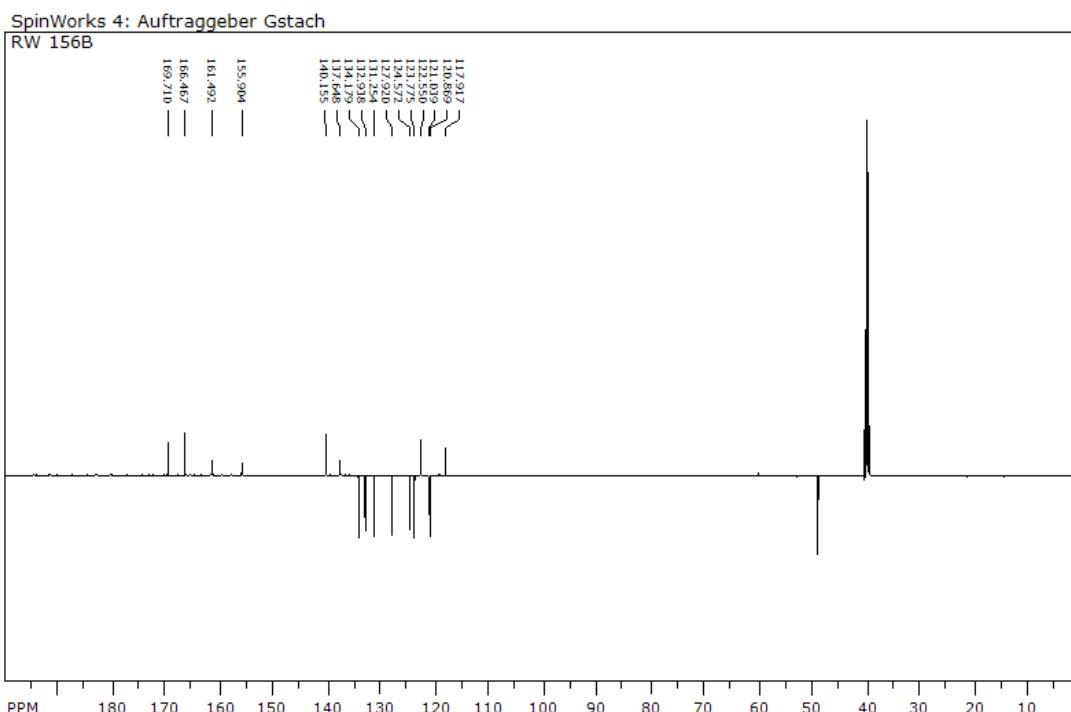
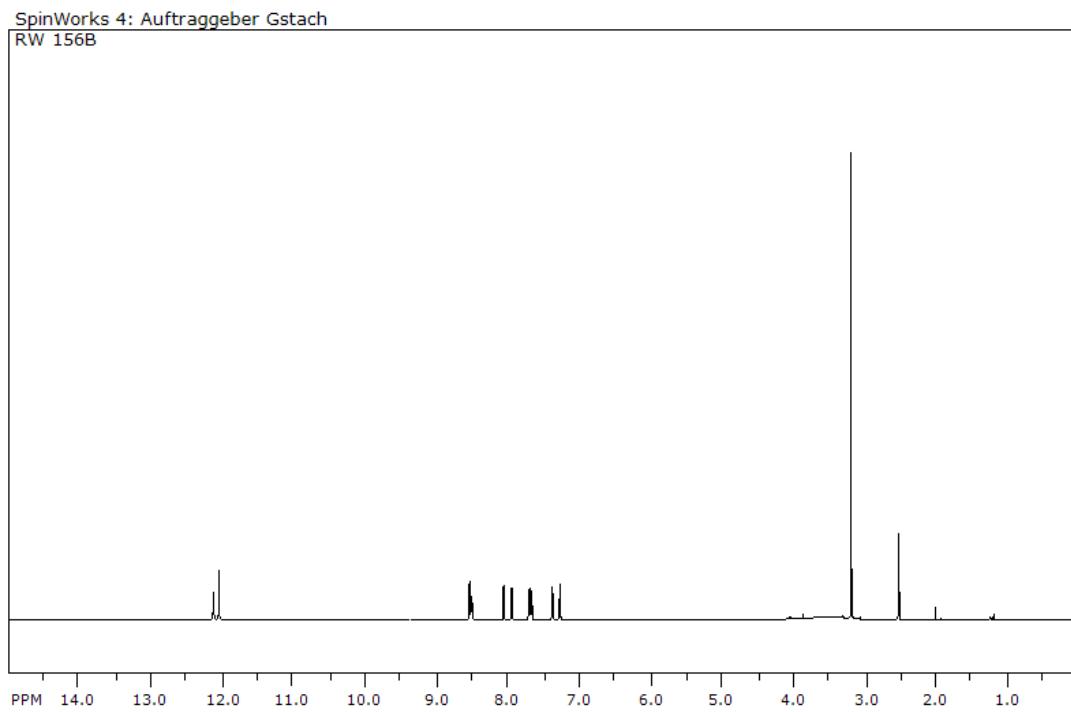
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number of scans: 1024

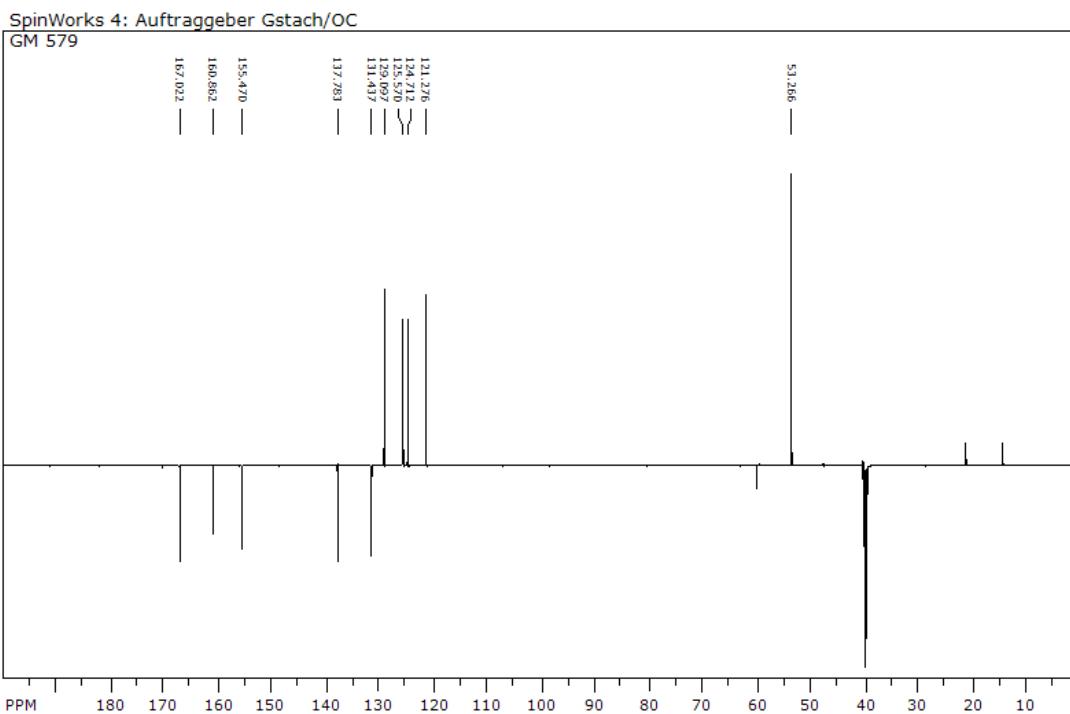
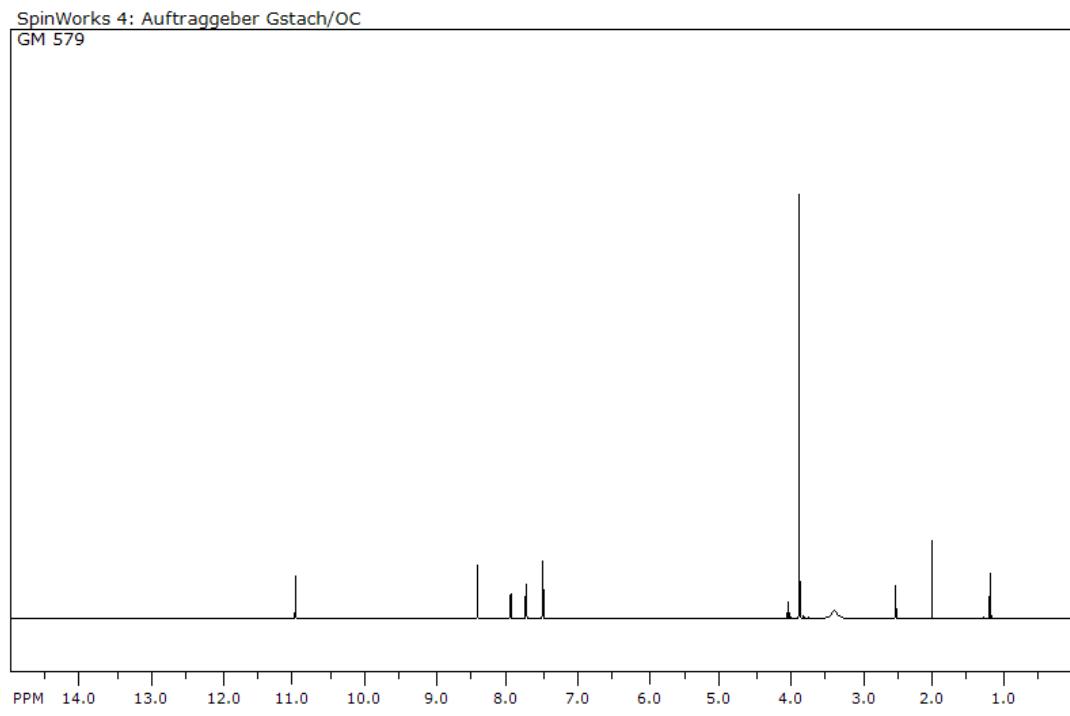
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Hz/cm: 1089.146 ppm/cm: 7.21537

2-(2-(2-methoxy-2-oxoacetamido)benzamido)benzoic acid (1d)

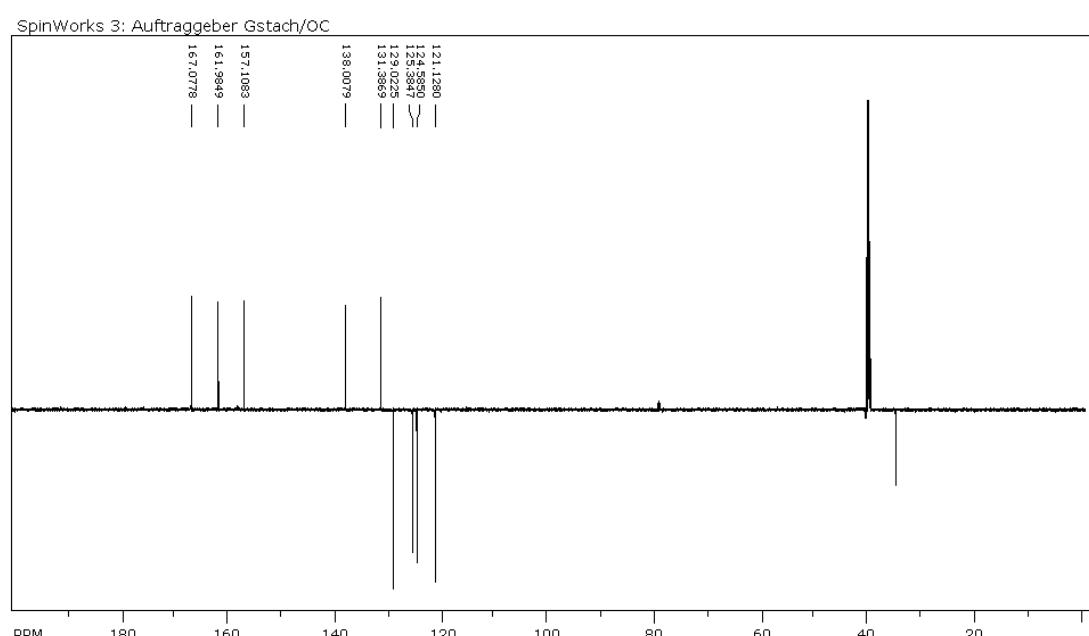
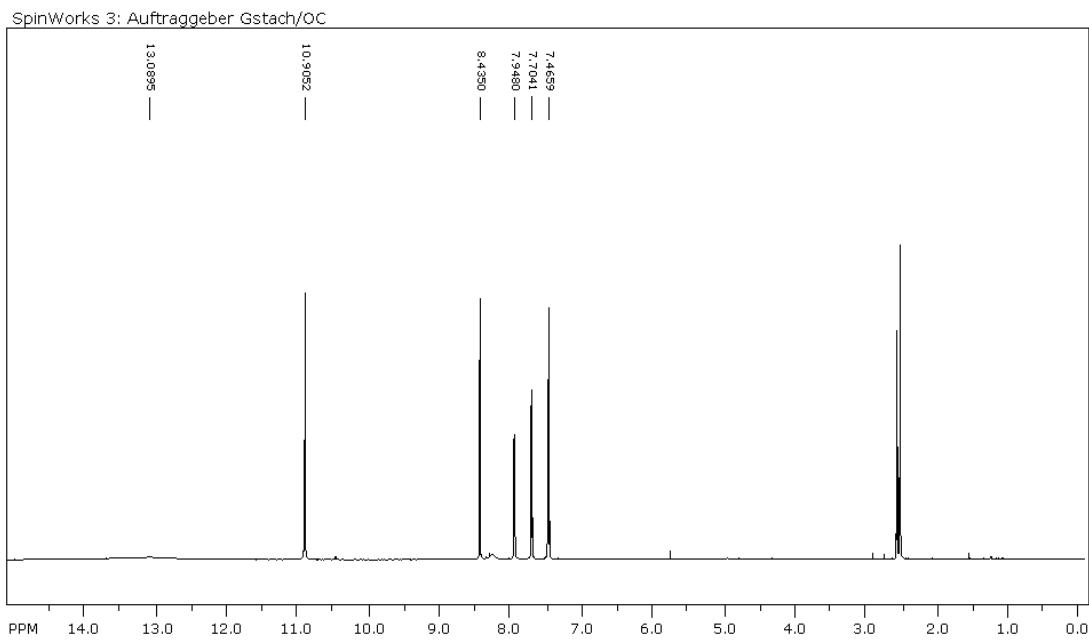


2-(2-(carboxyformamido)benzamido)benzoic acid (1e)

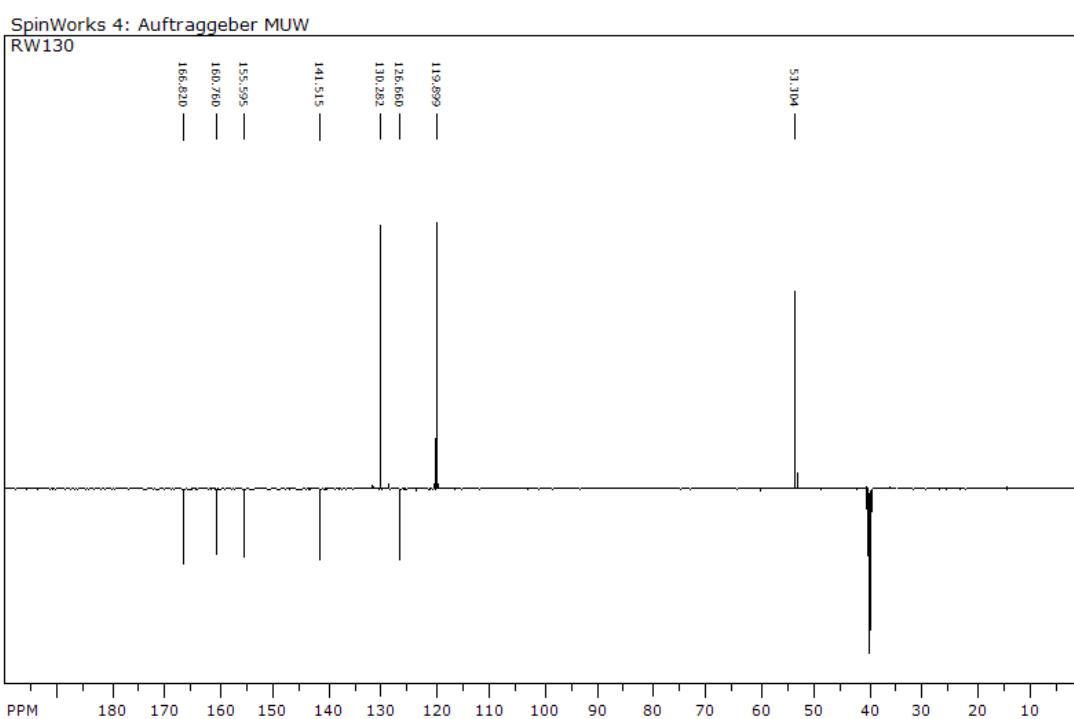
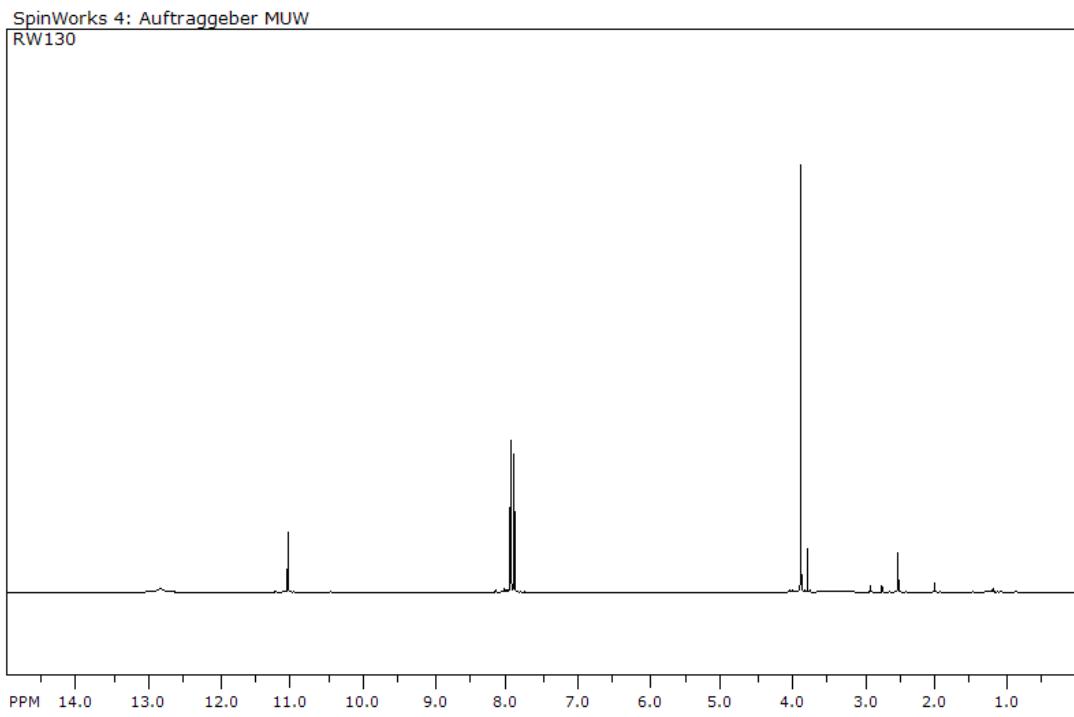


3-(2-methoxy-2-oxoacetamido)benzoic acid (2a)

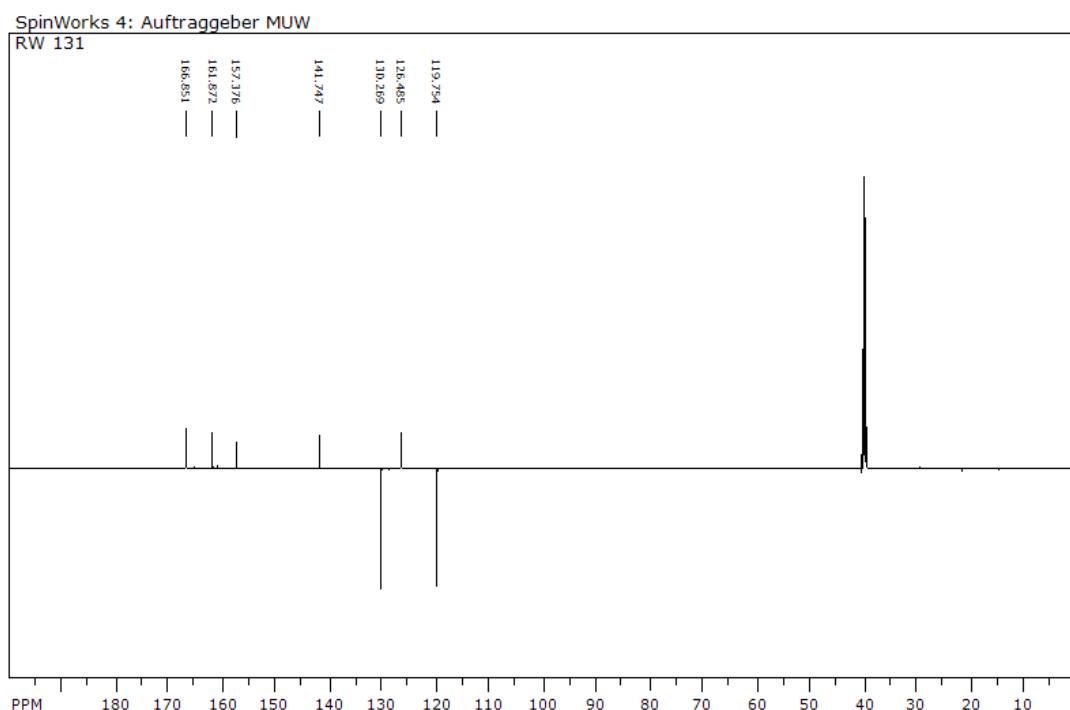
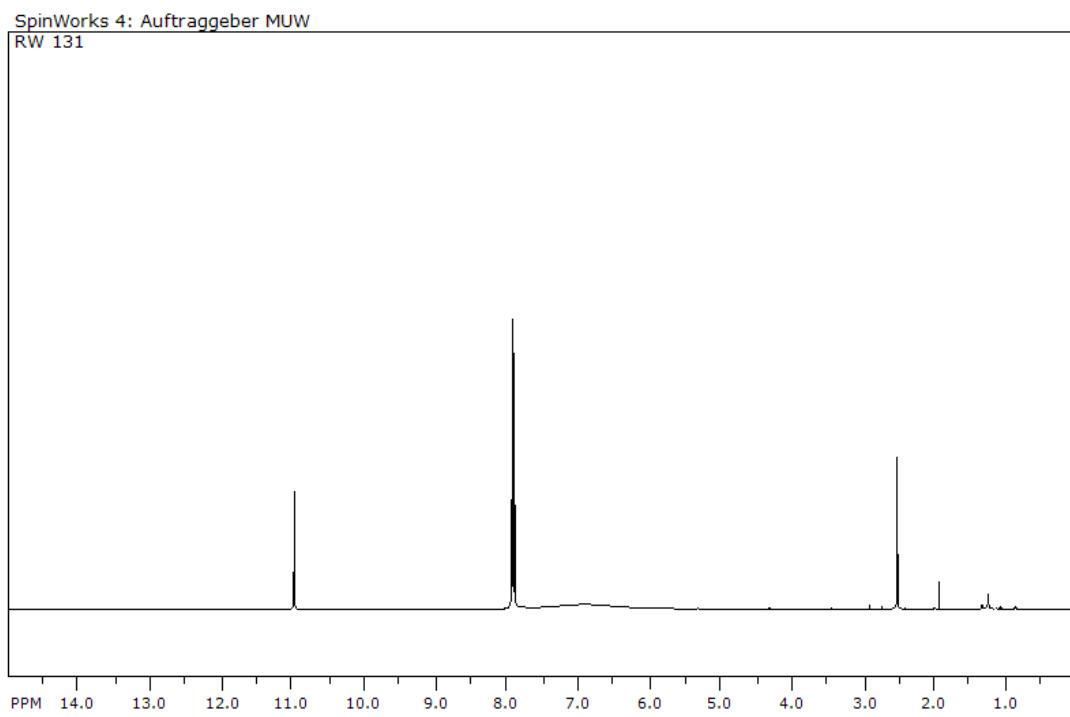
3-(Carboxyformamido)benzoic acid (2b)



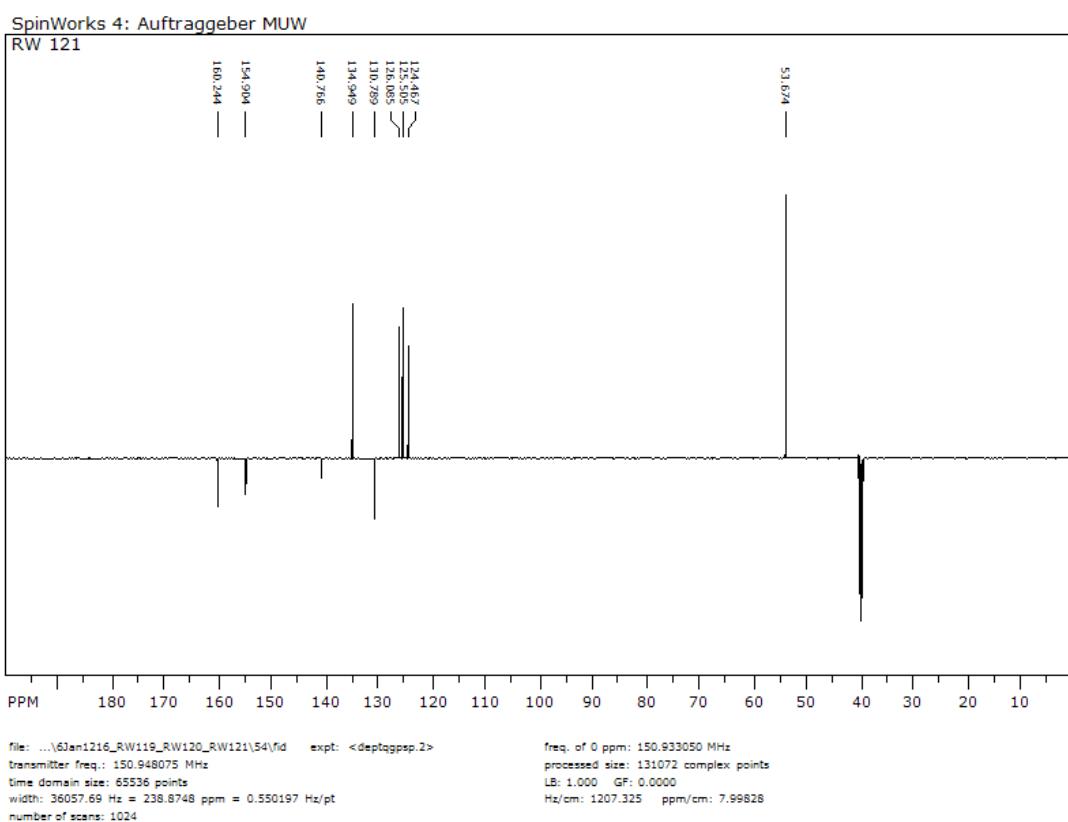
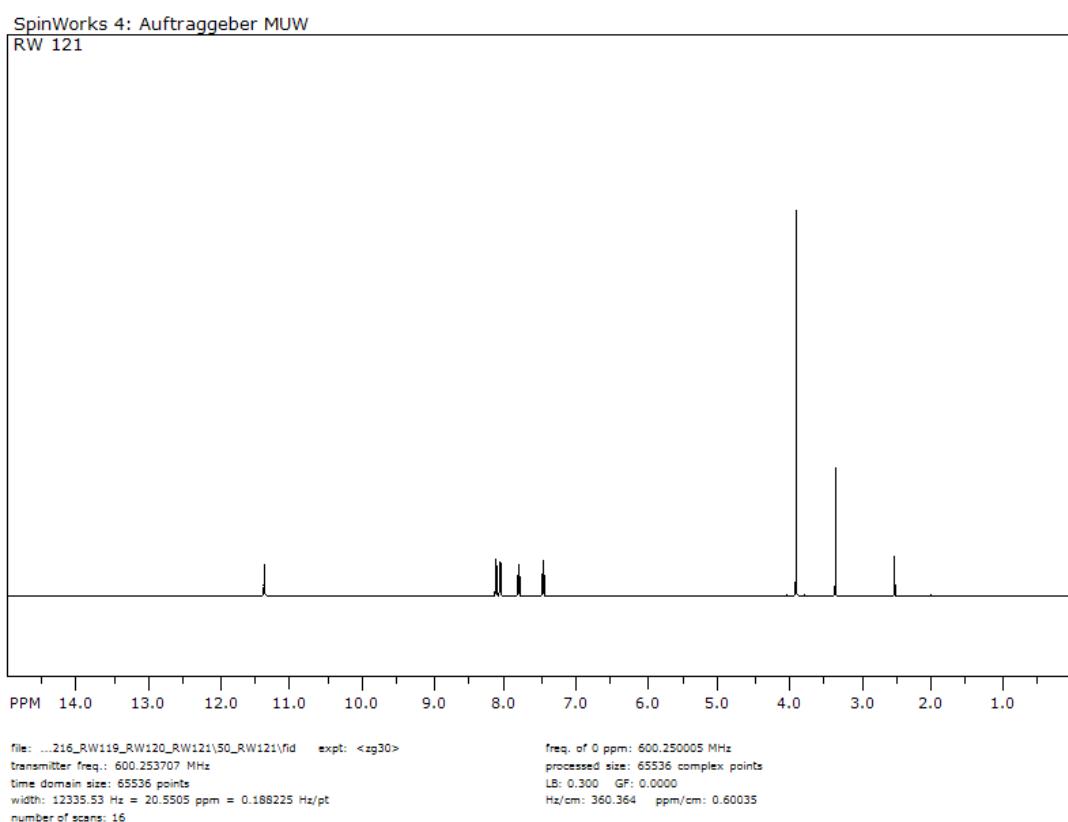
4-(2-methoxy-2-oxoacetamido)benzoic acid (3a)



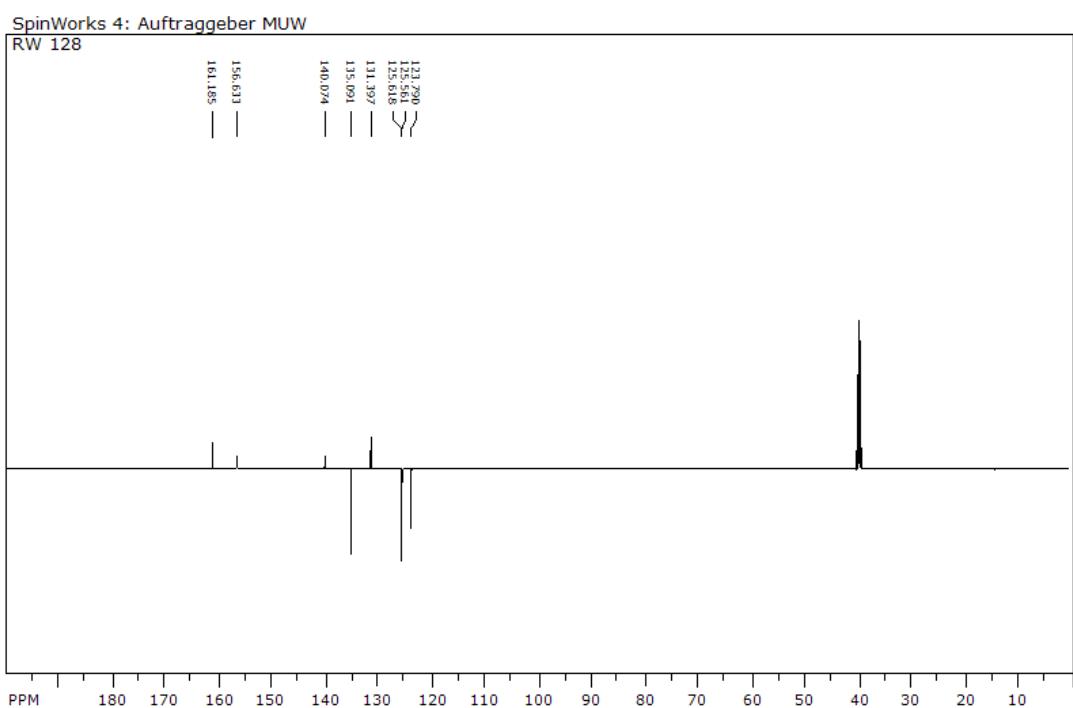
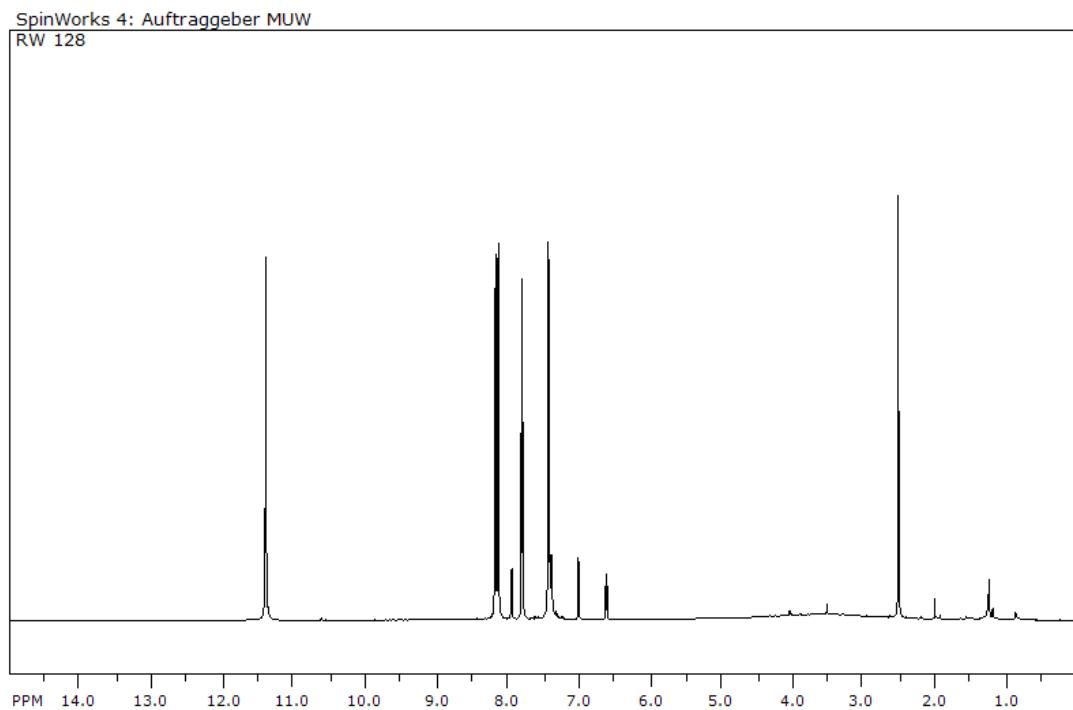
4-(carboxyformamido)benzoic acid (3b)



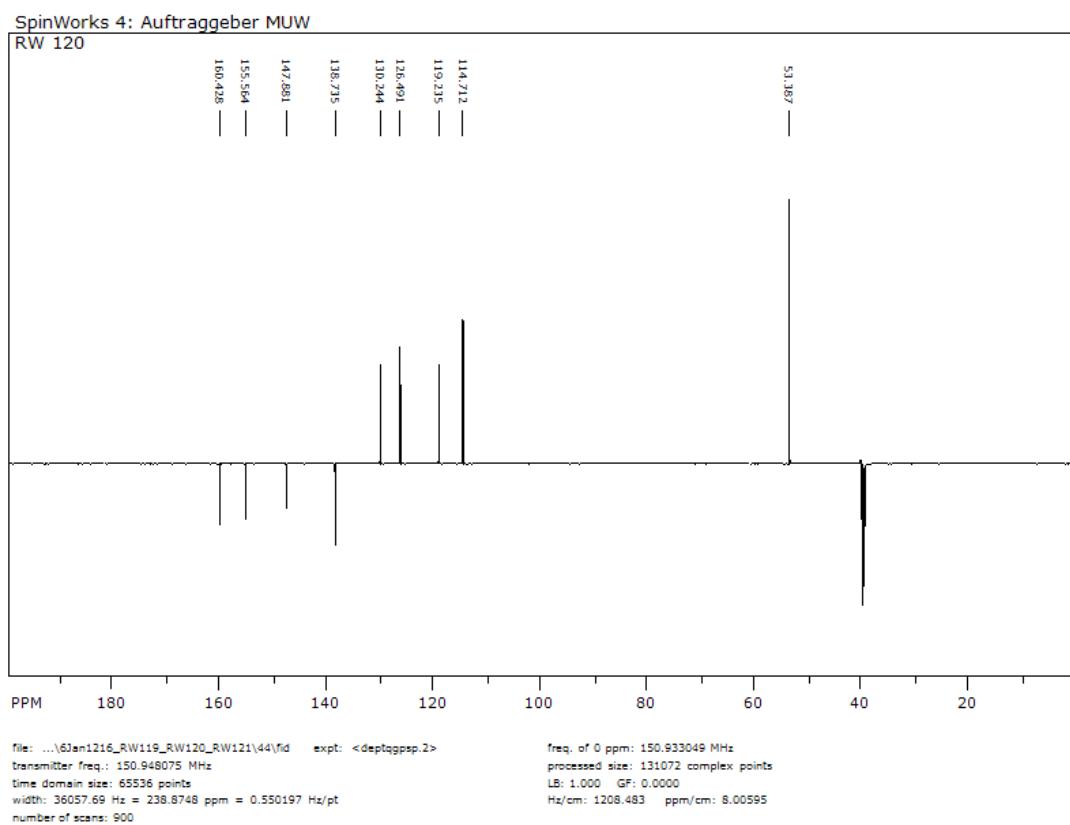
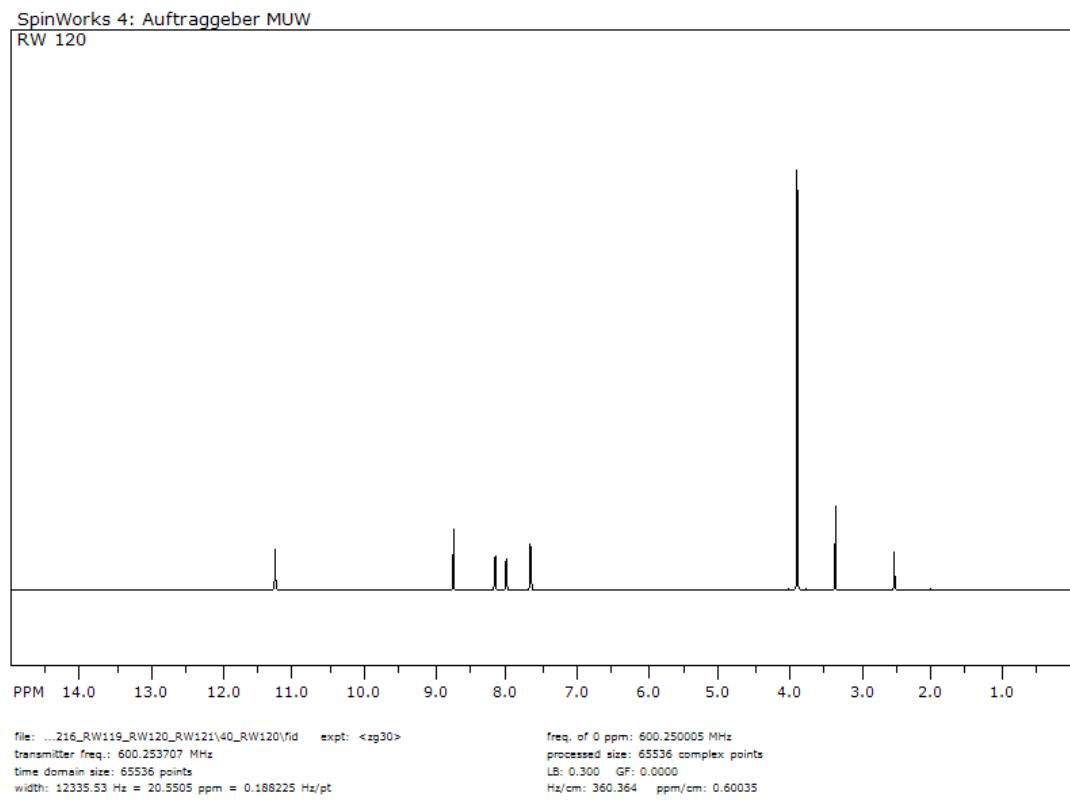
Methyl 2-((2-nitrophenyl)amino)-2-oxoacetate (4a)



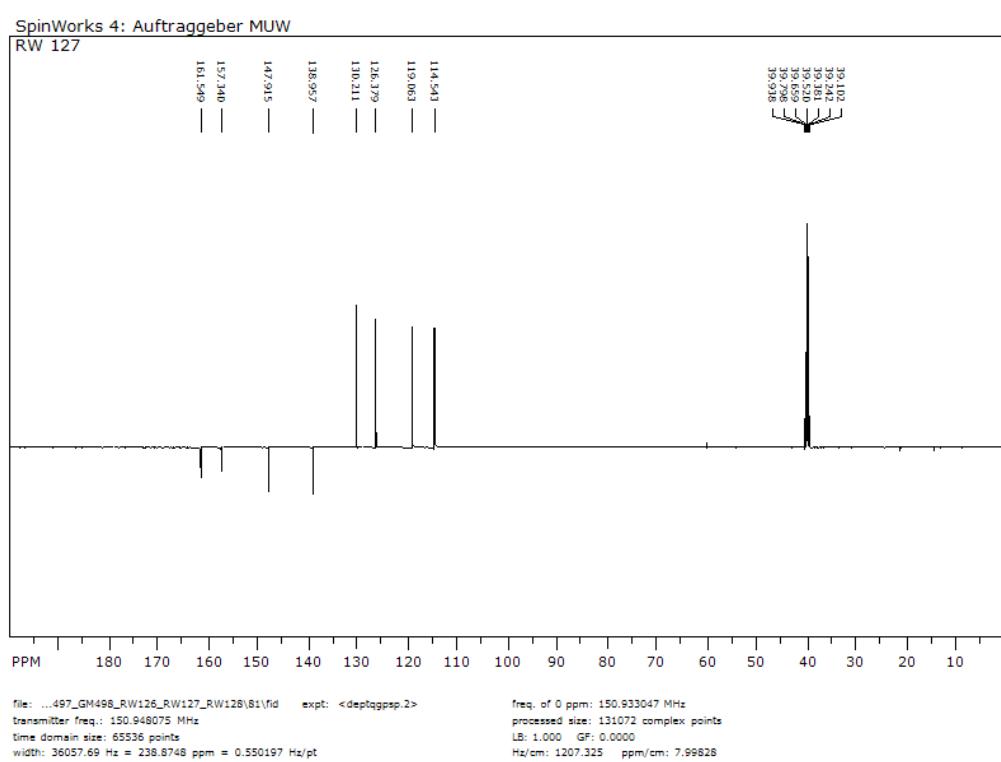
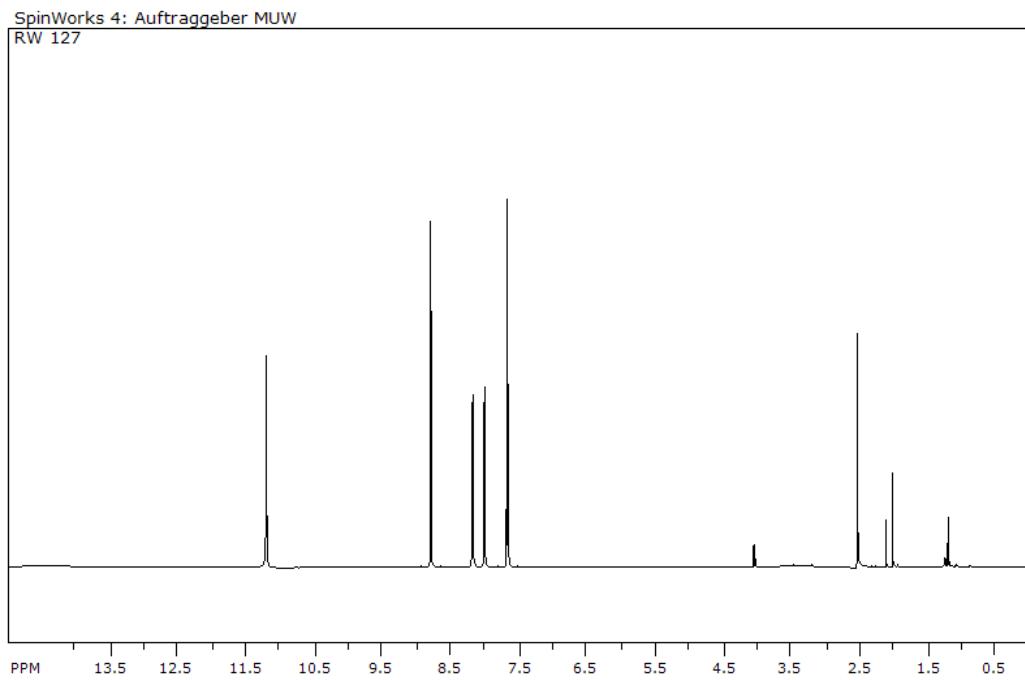
2-((2-nitrophenyl)amino)-2-oxoacetic acid (4b)



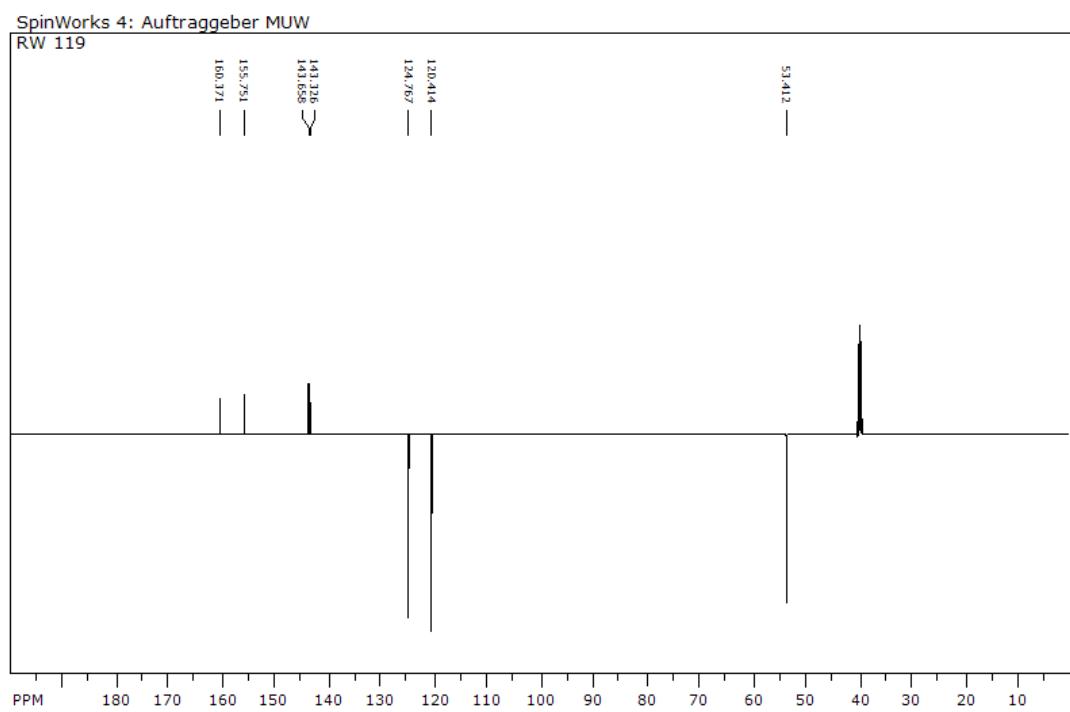
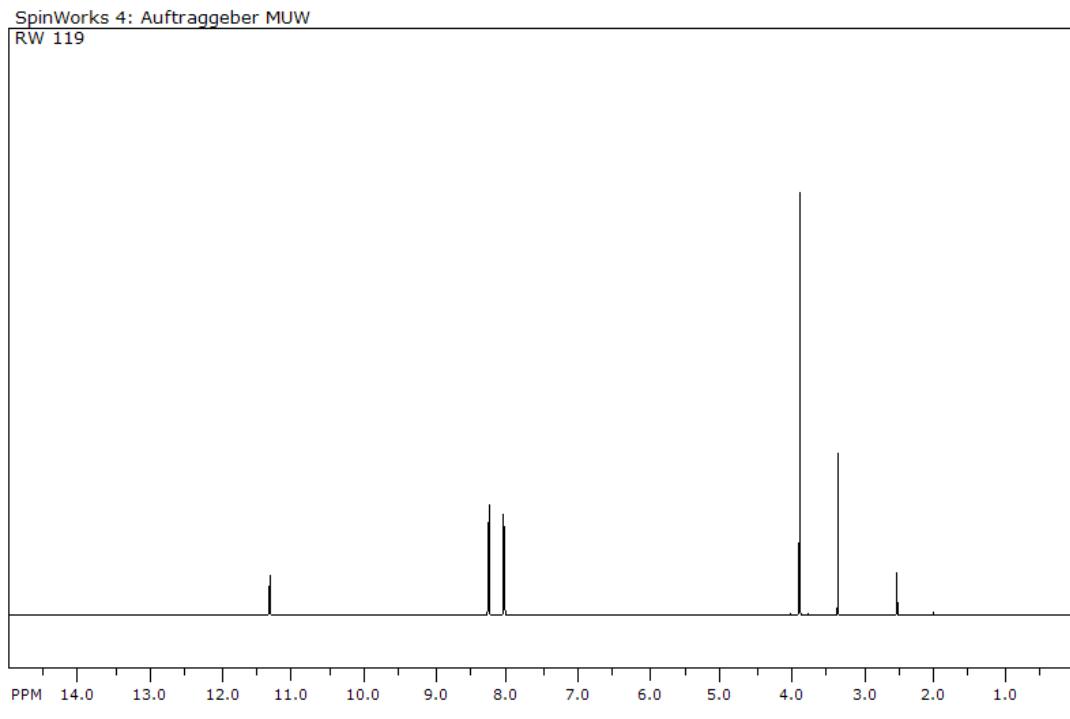
Methyl 2-((3-nitrophenyl)amino)-2-oxoacetate (5a)



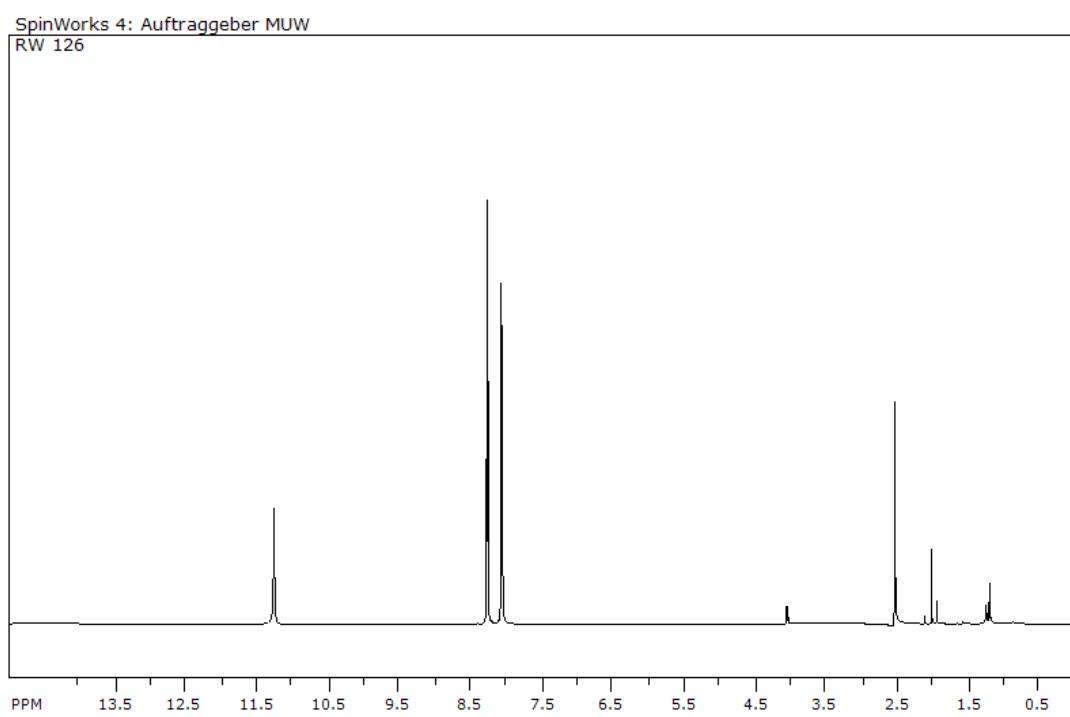
2-((3-nitrophenyl)amino)-2-oxoacetic acid (5b)



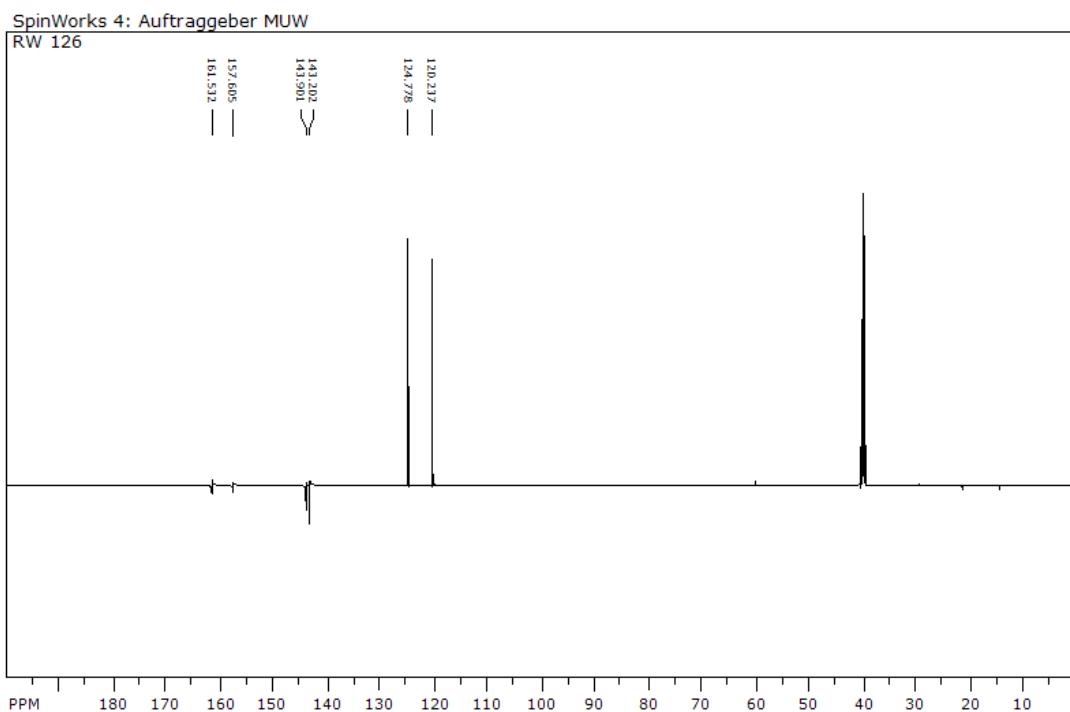
Methyl 2-((4-nitrophenyl)amino)-2-oxoacetate (6a)



2-((4-nitrophenyl)amino)-2-oxoacetic acid (6b)

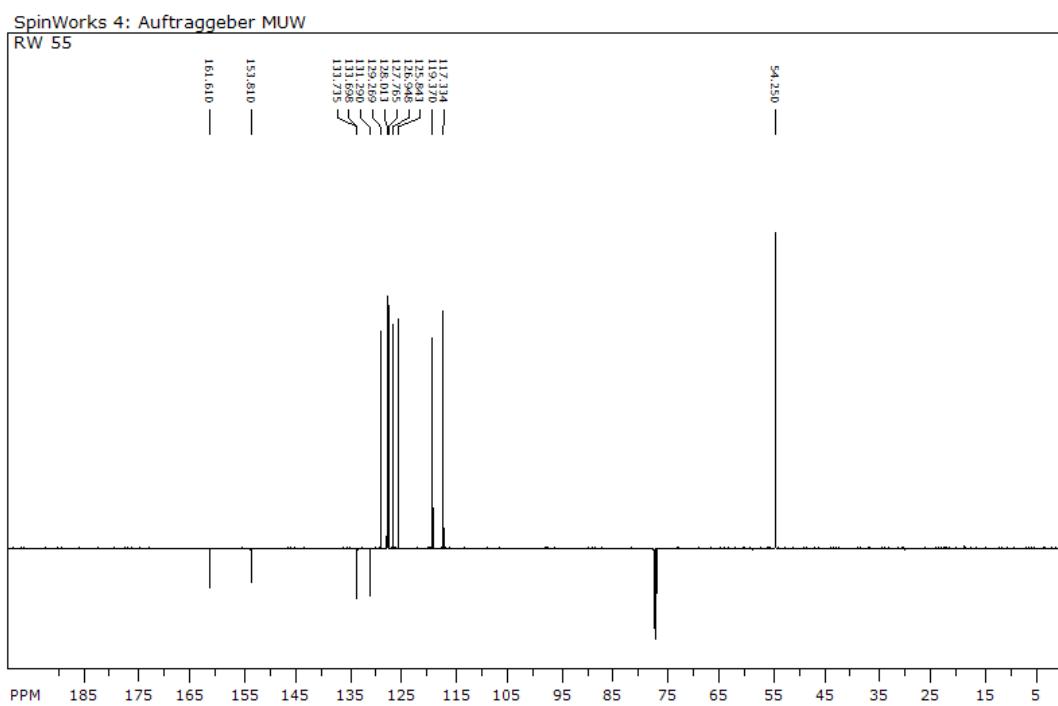
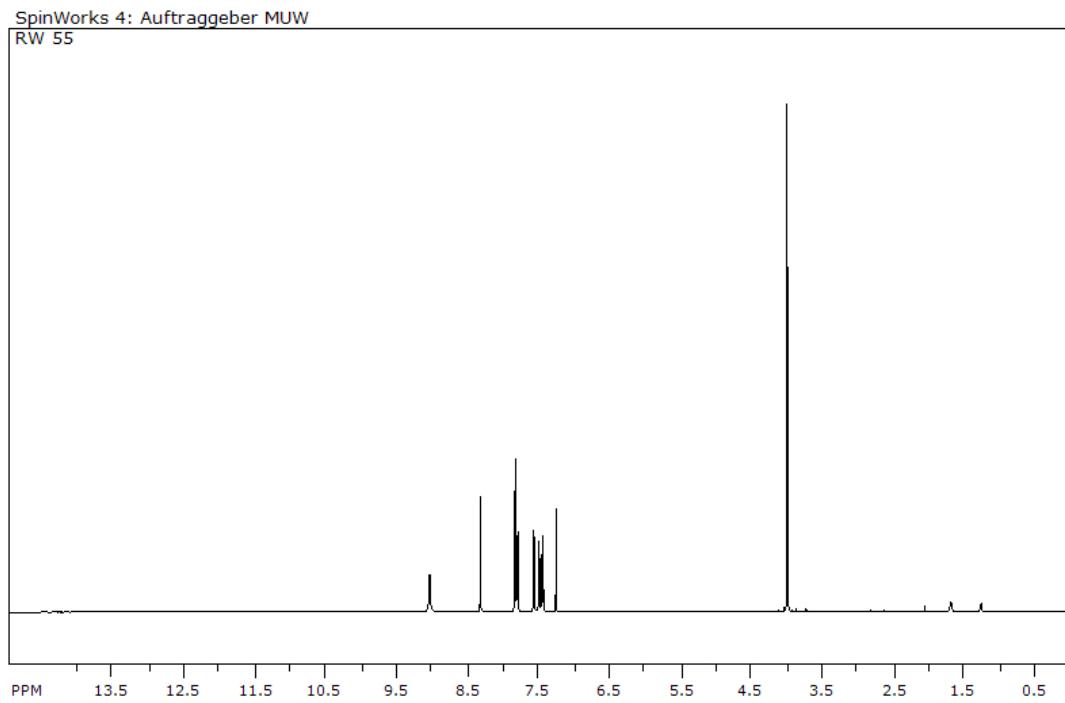


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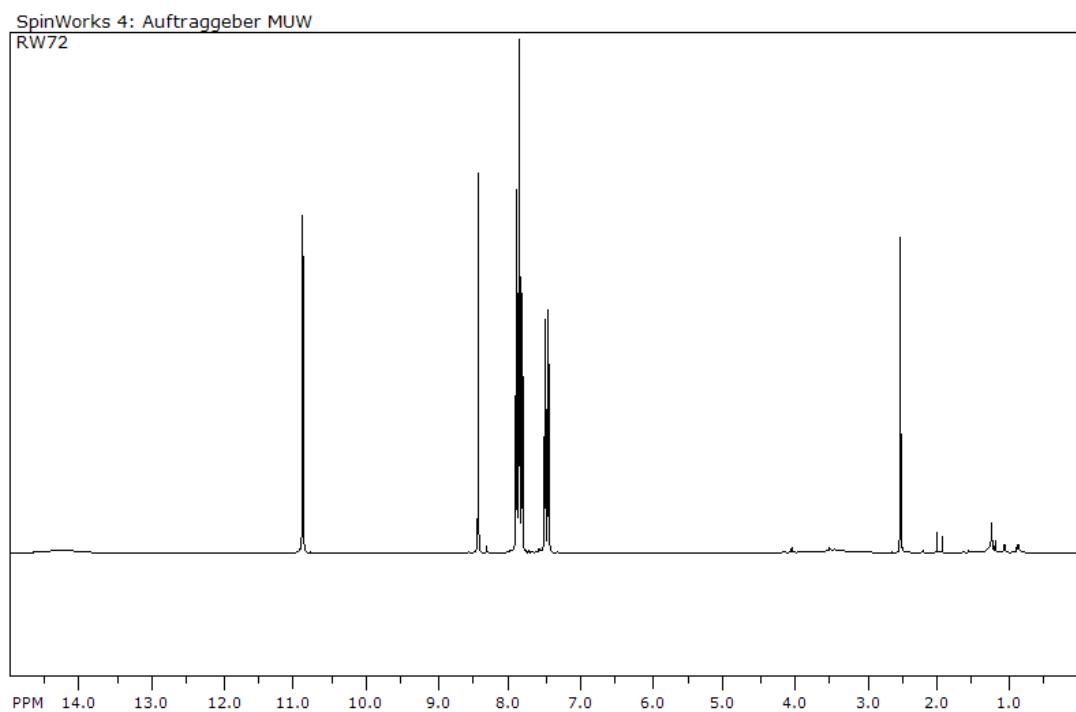


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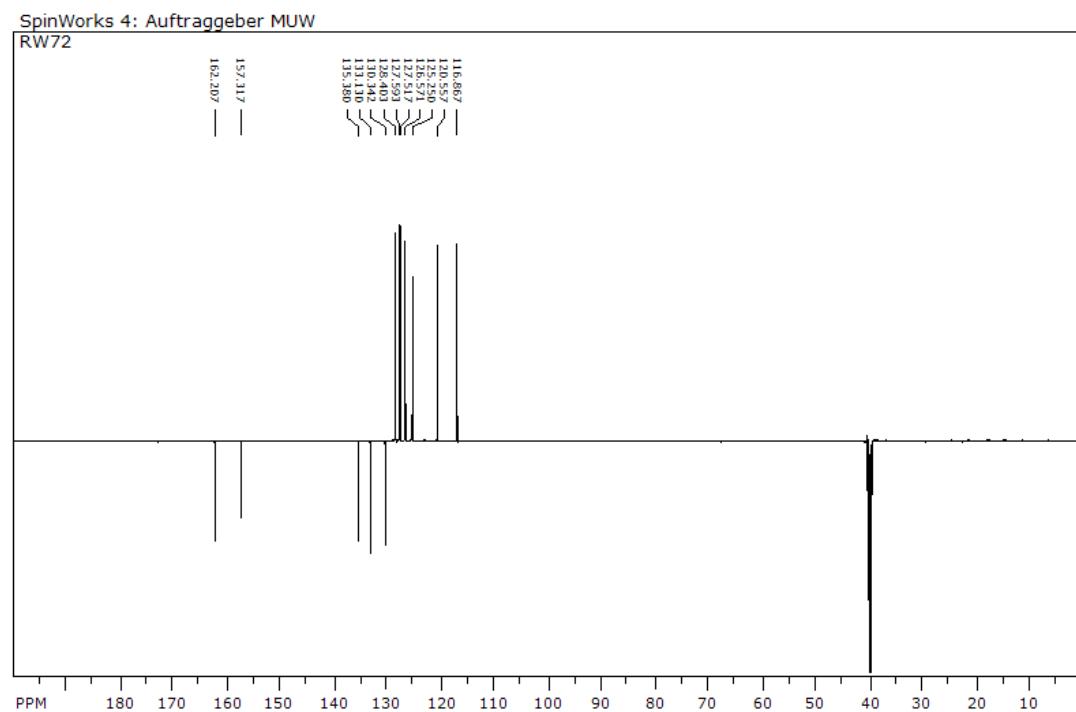
Methyl 2-(naphthalen-2-ylamino)-2-oxoacetate (7a)



2-(naphthalen-2-ylamino)-2-oxoacetic acid (7b)

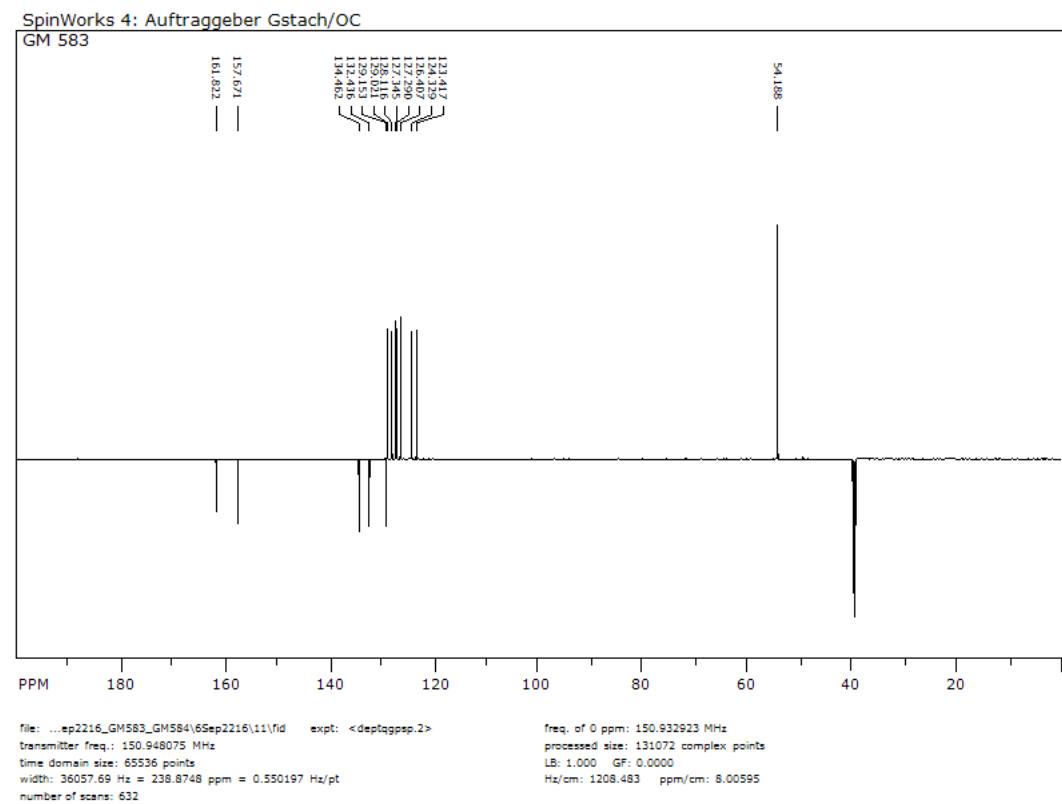
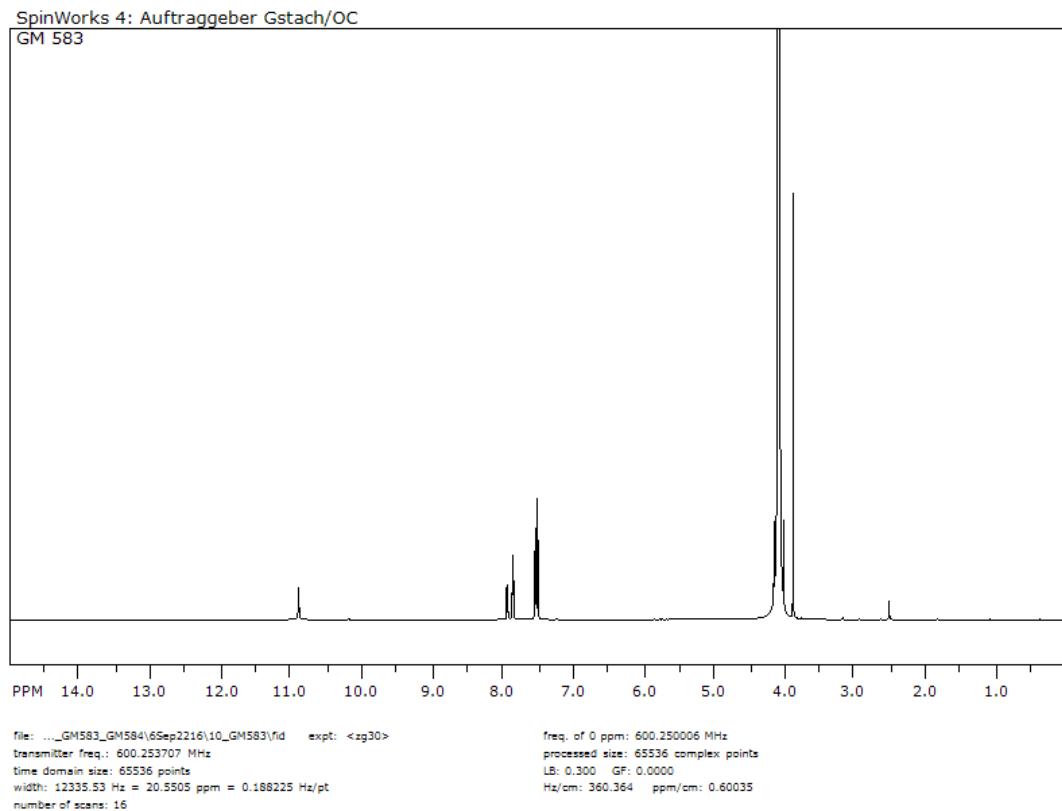


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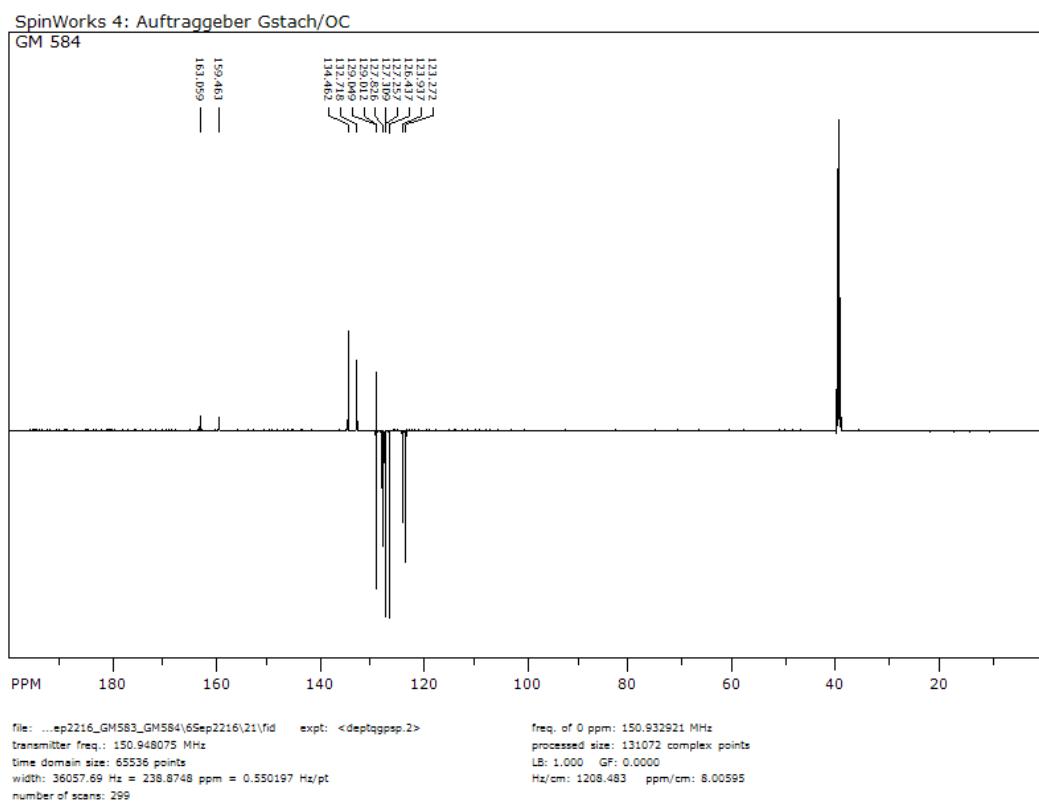
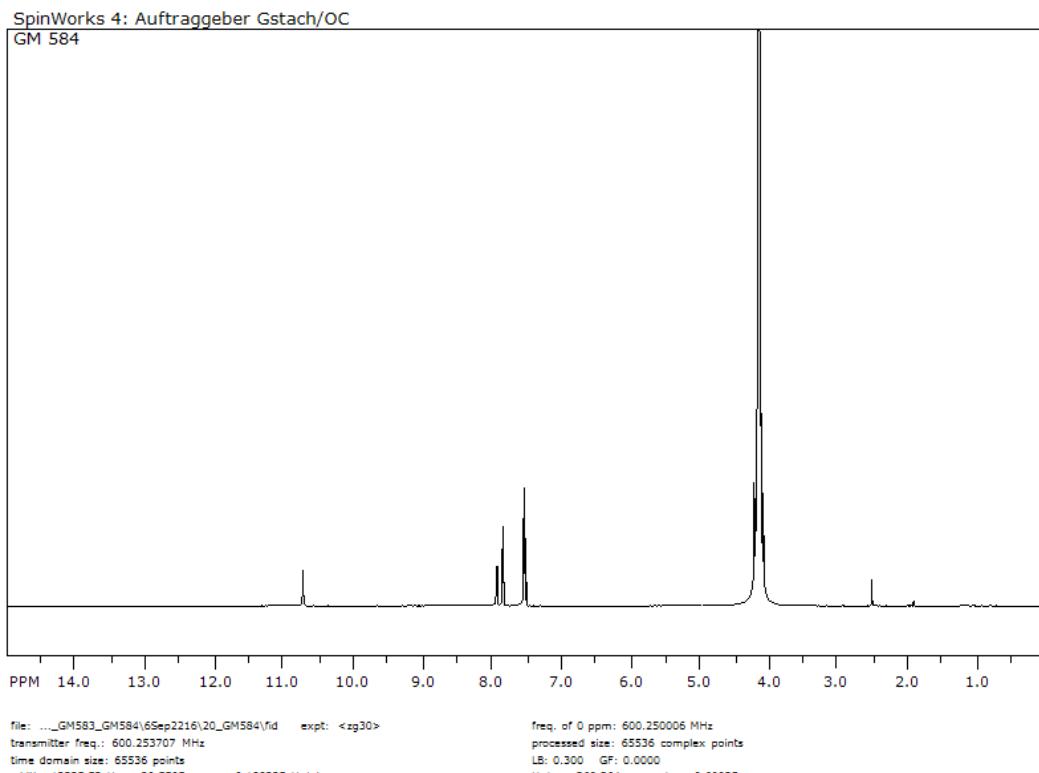


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number of scans: 1024
freq. of 0 ppm: 150.933048 MHz
processed size: 131072 complex points
LB: 1.000 GF: 0.0000
Hz/cm: 1207.325 ppm/cm: 7.99828

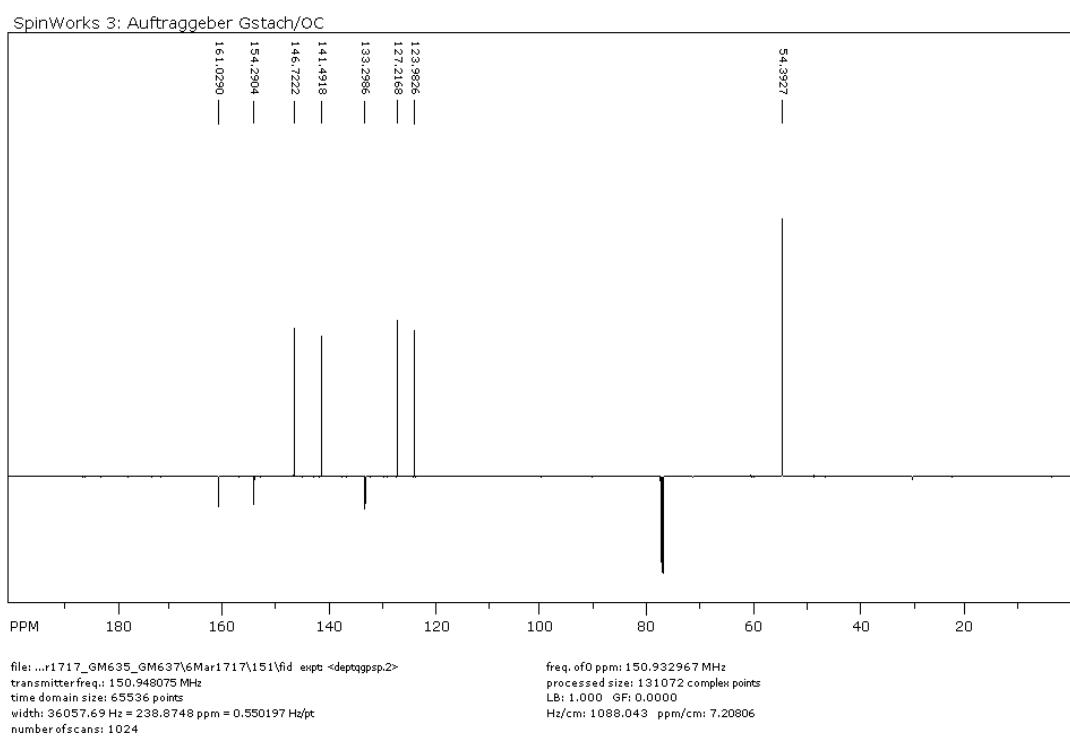
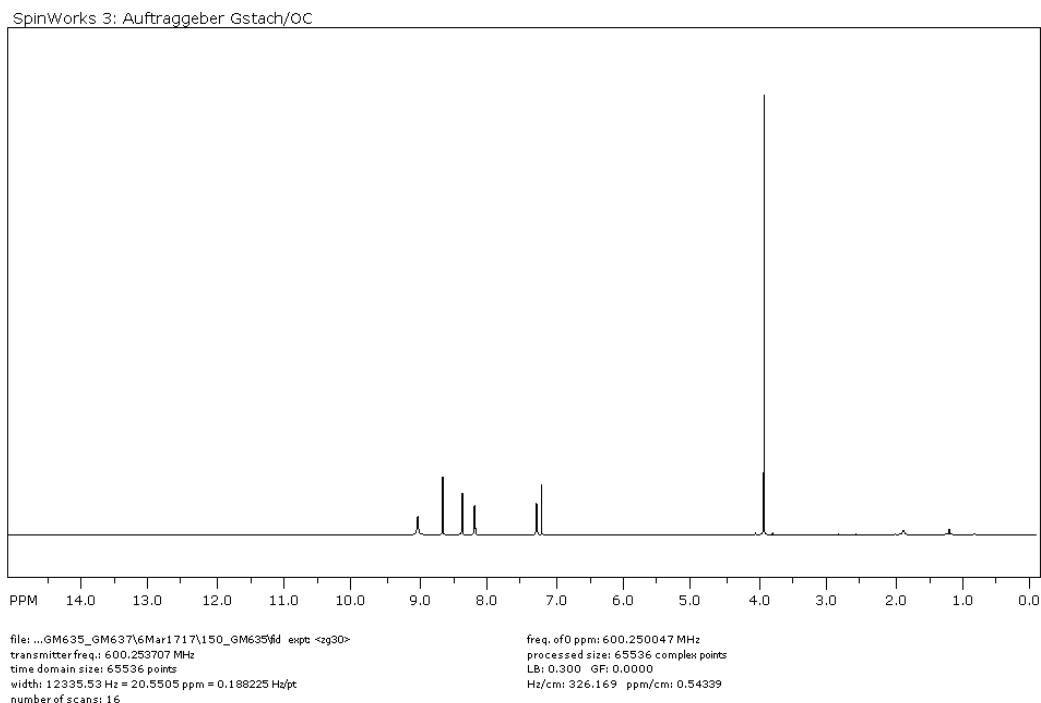
Methyl 2-(naphthalen-1-ylamino)-2-oxoacetate (8a)



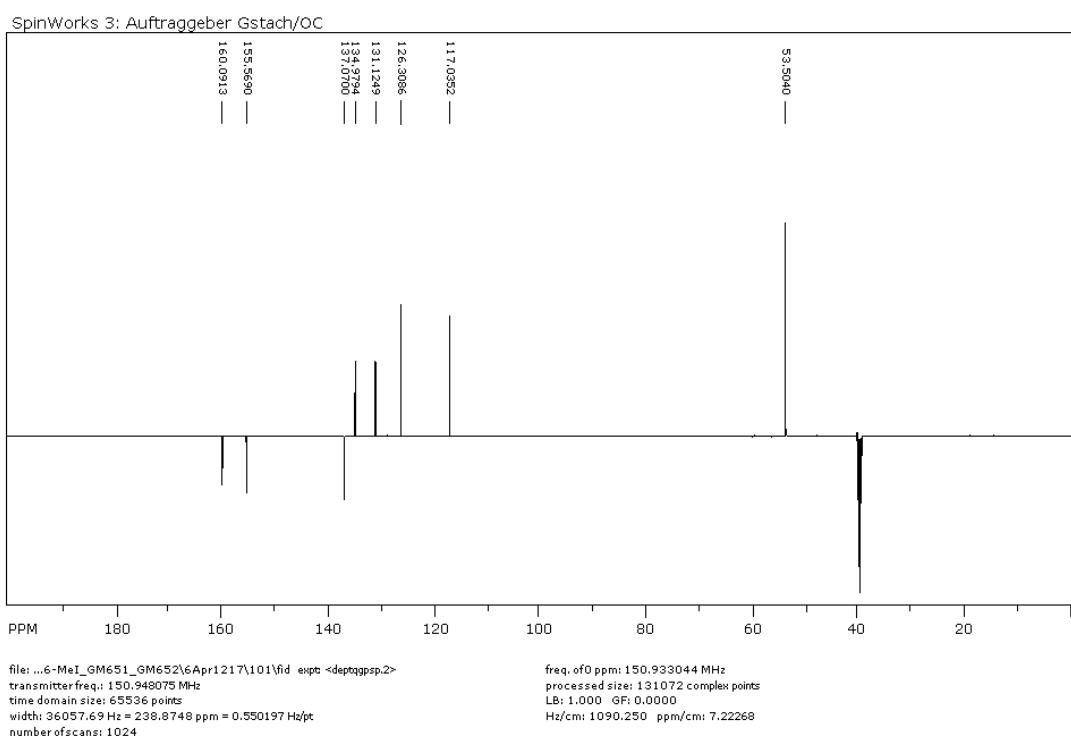
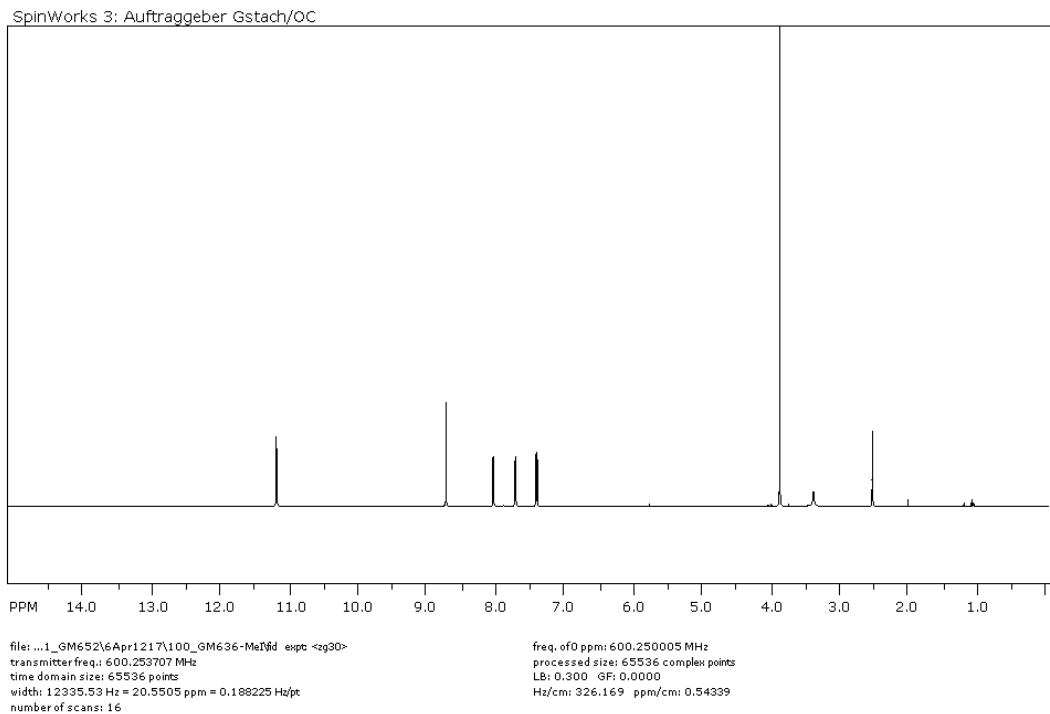
2-(naphthalen-1-ylamino)-2-oxoacetic acid (8b)



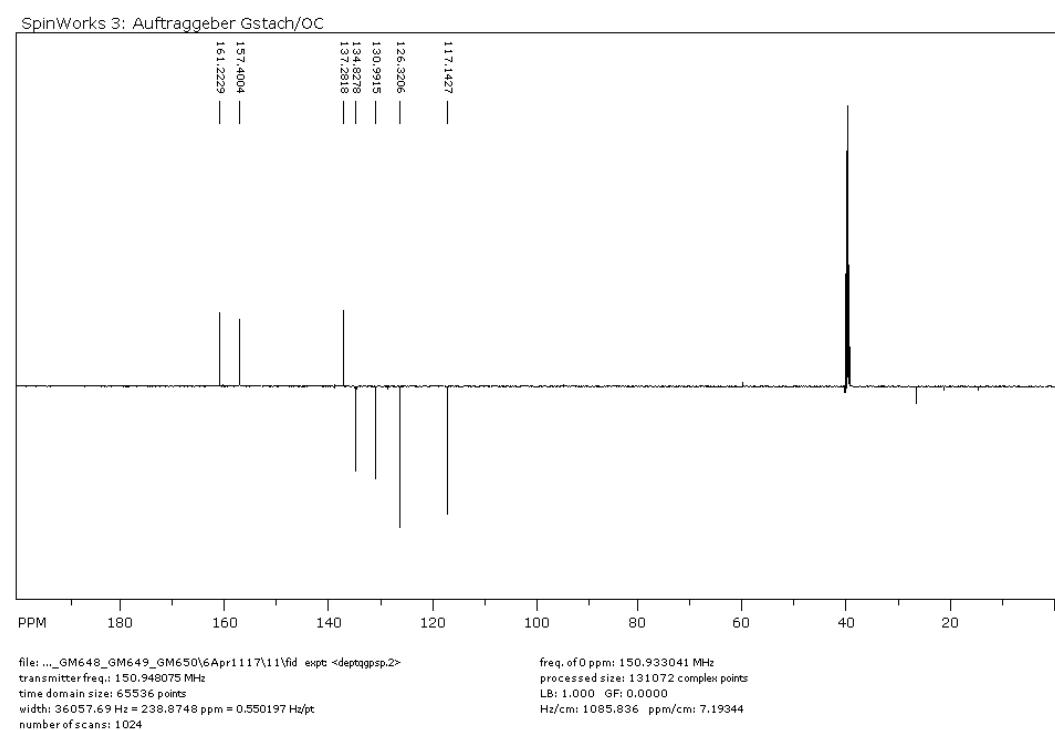
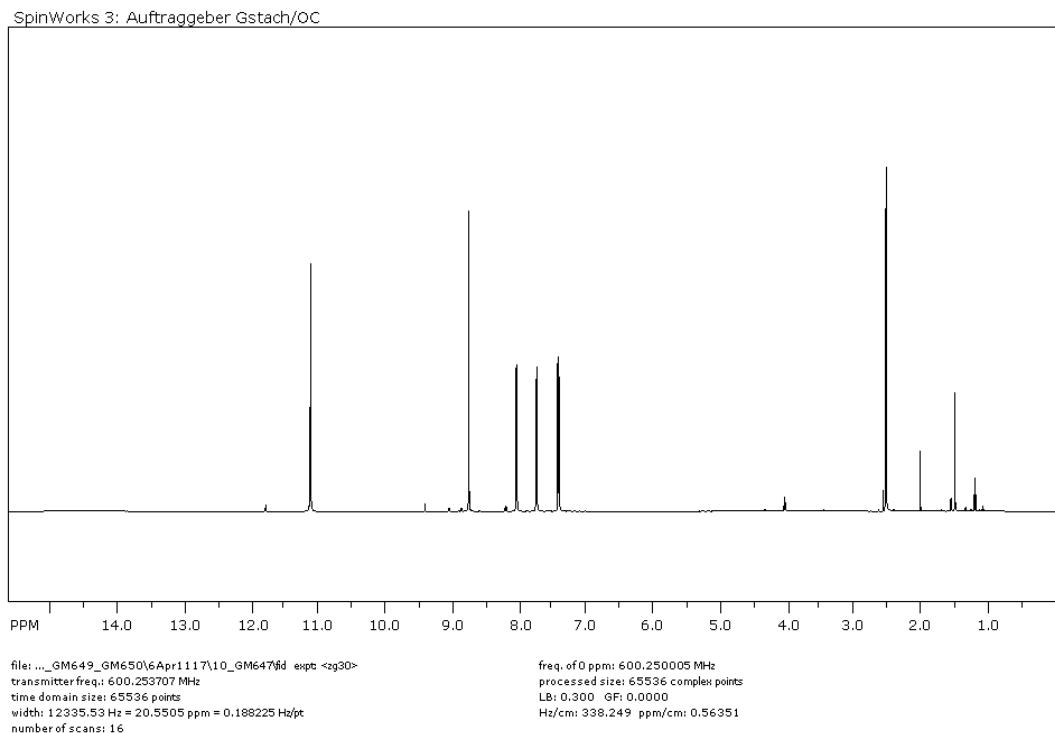
Methyl 2-oxo-2-(pyridin-3-ylamino)acetate (9a)



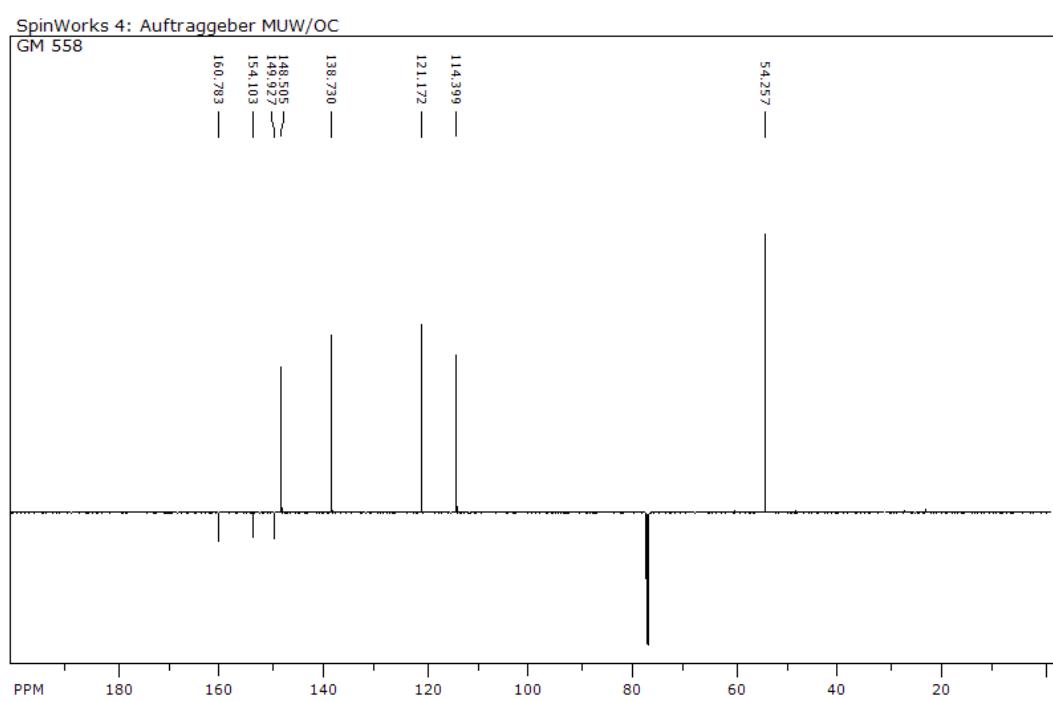
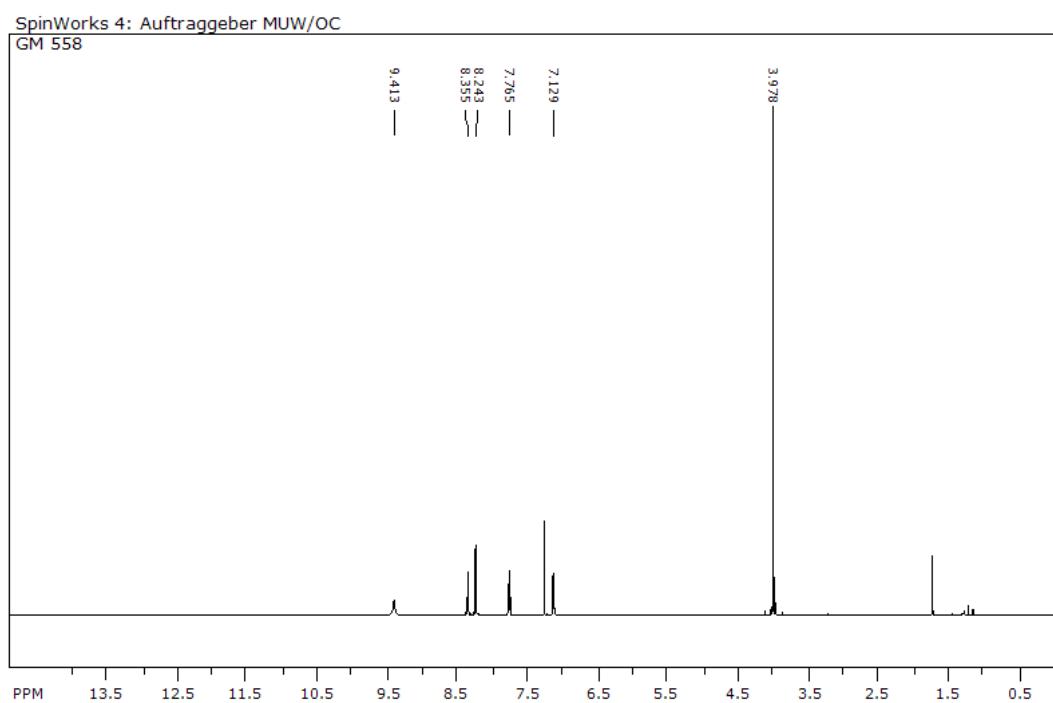
3-(2-Methoxy-2-oxoacetamido)pyridine 1-oxide (11a)



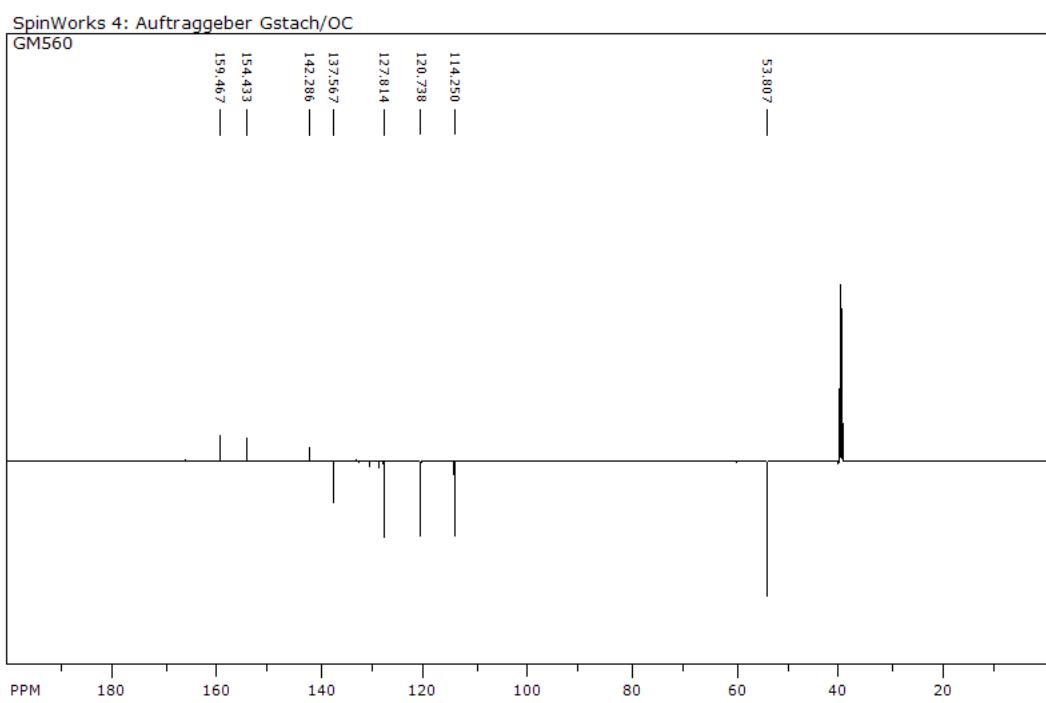
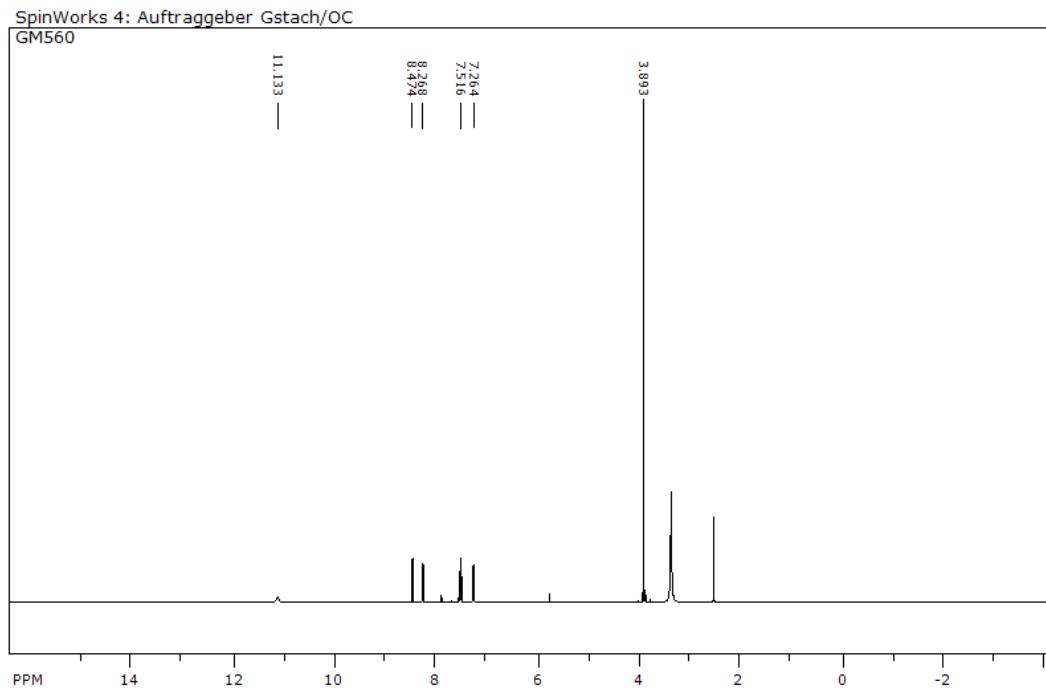
3-(Carboxyformamido)pyridine 1-oxide (11b)



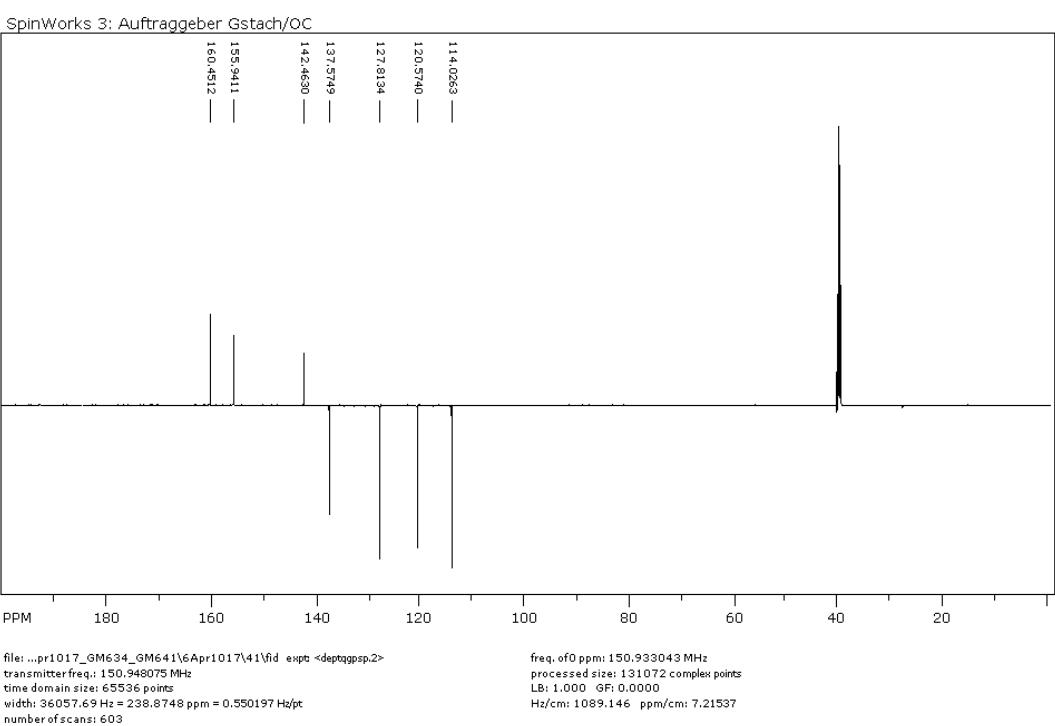
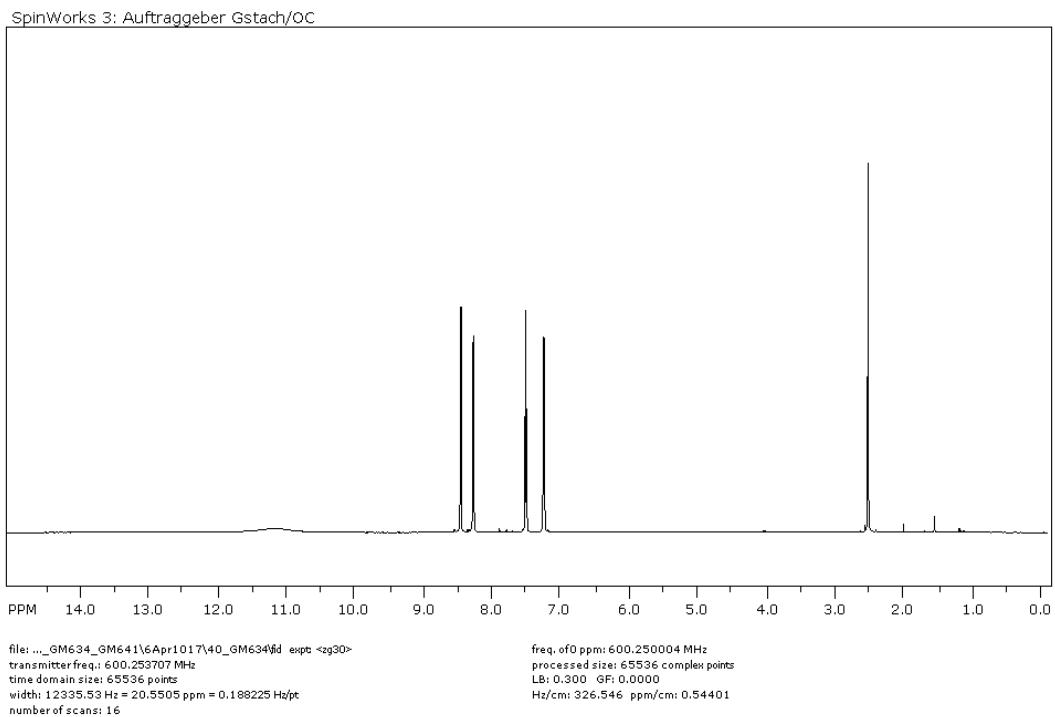
Methyl 2-oxo-2-(pyridin-2-ylamino)acetate (10a)



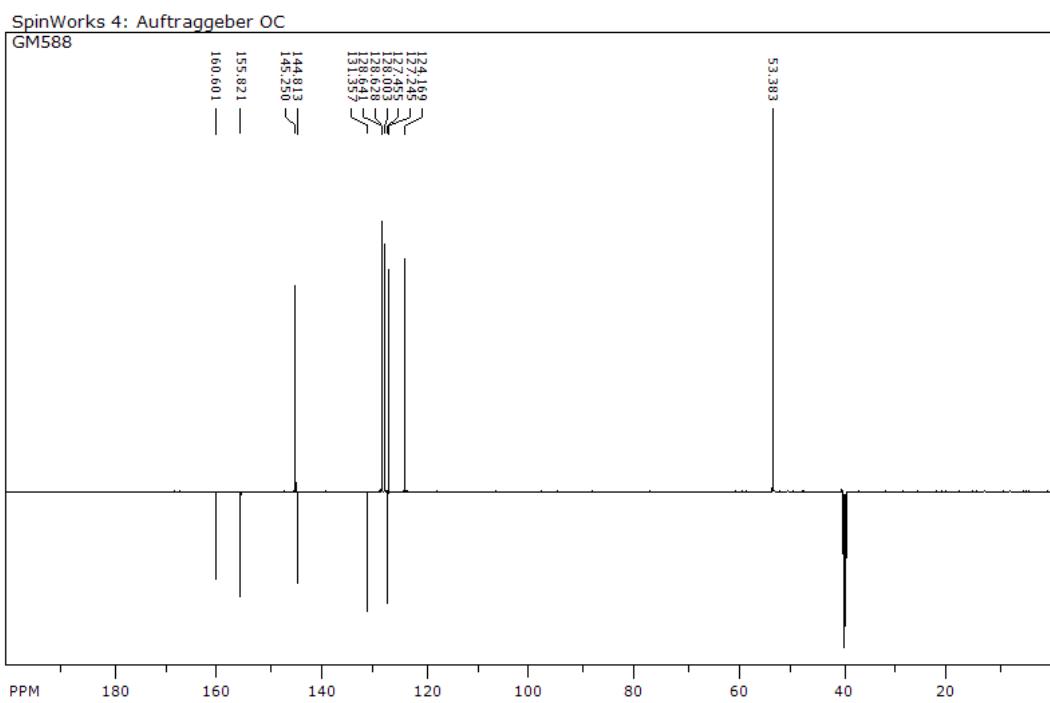
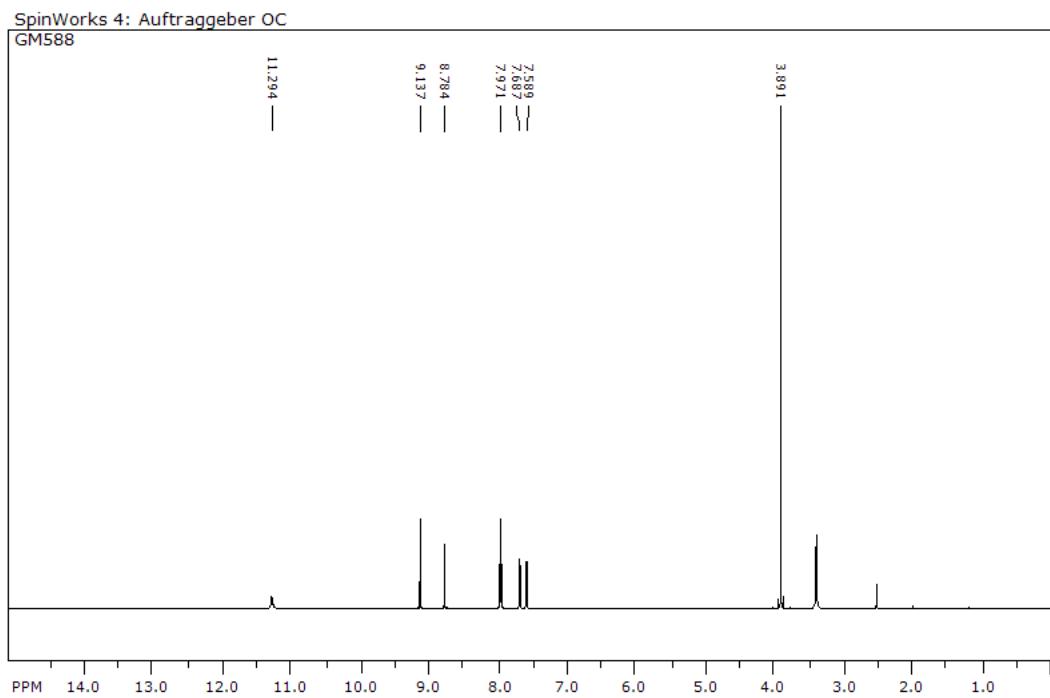
2-(2-Methoxy-2-oxoacetamido)pyridine 1-oxide (12a)



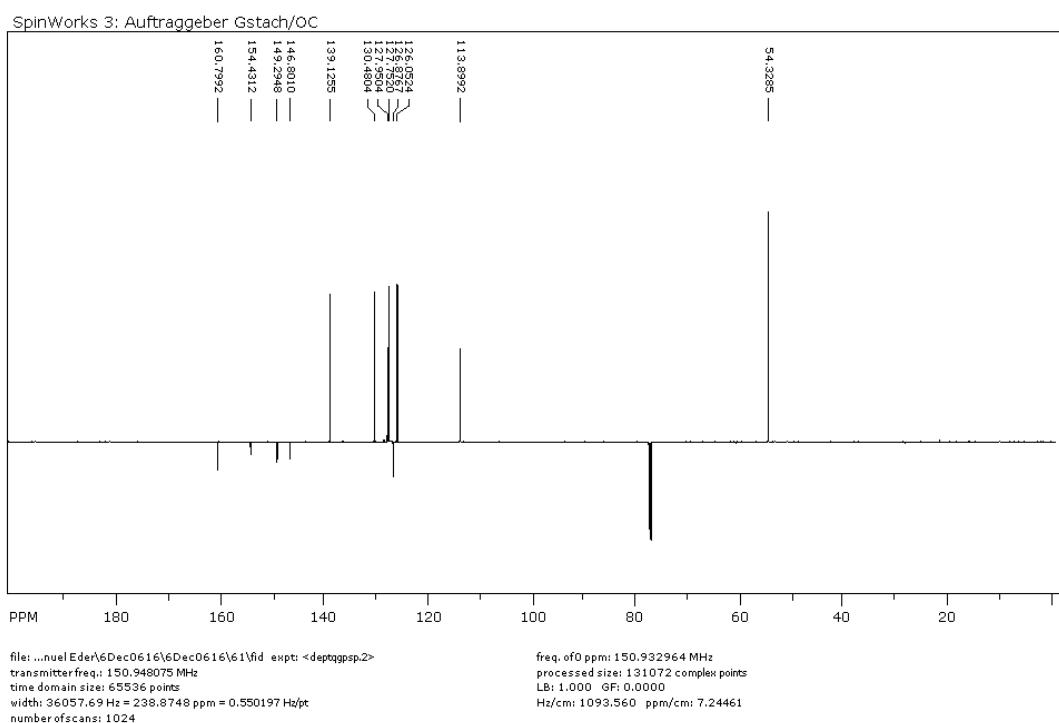
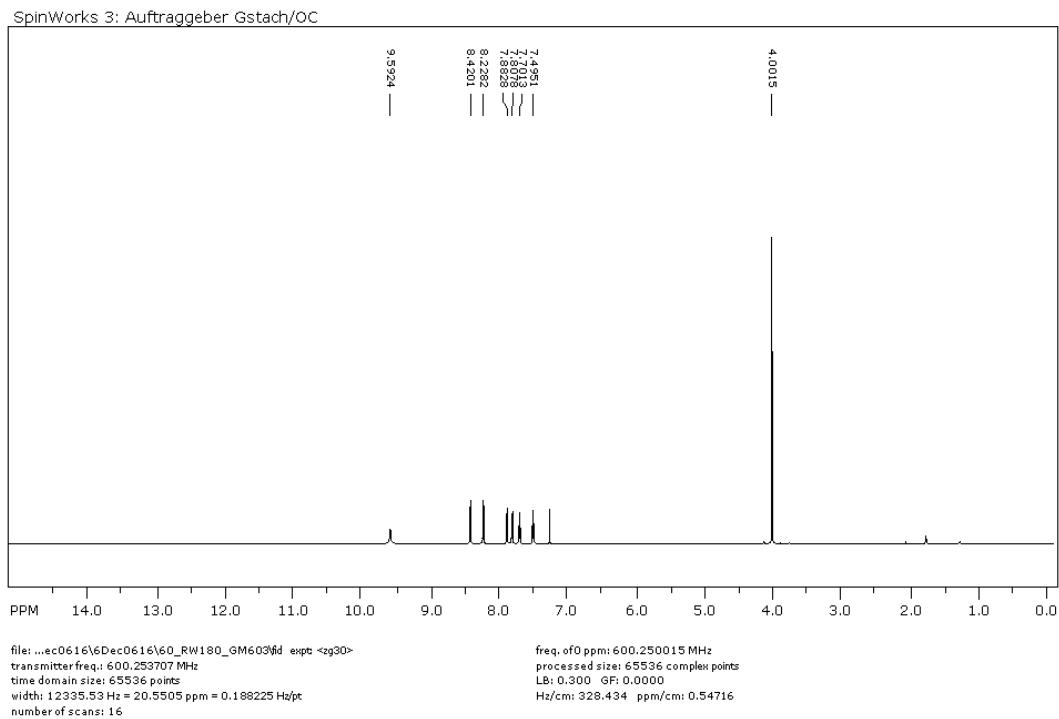
2-(Carboxyformamido)pyridine 1-oxide (12b)

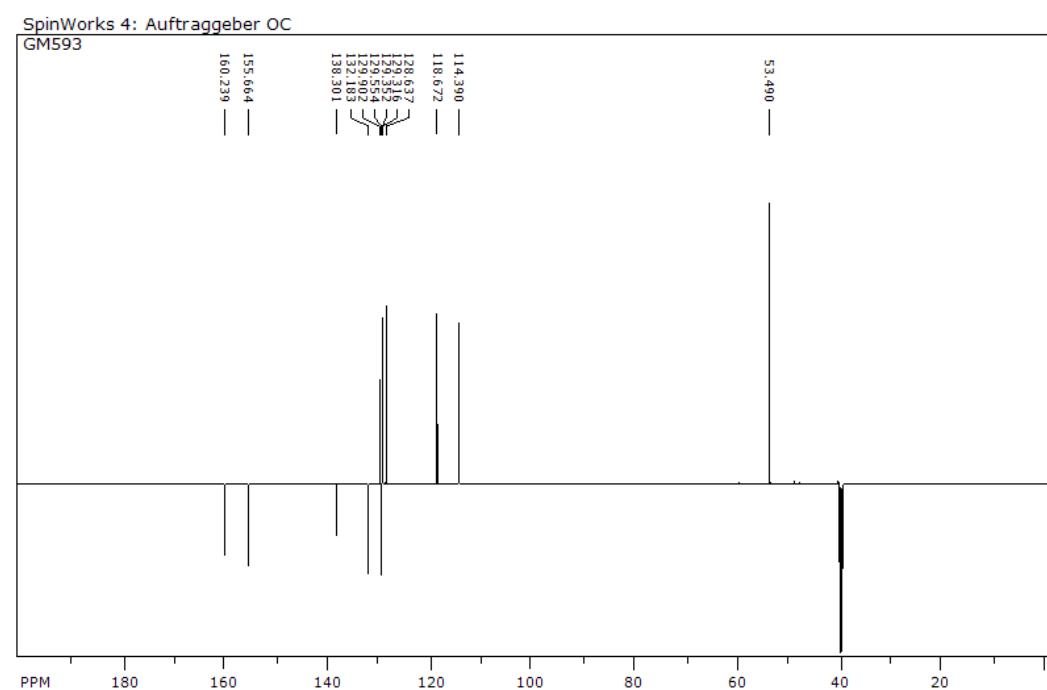
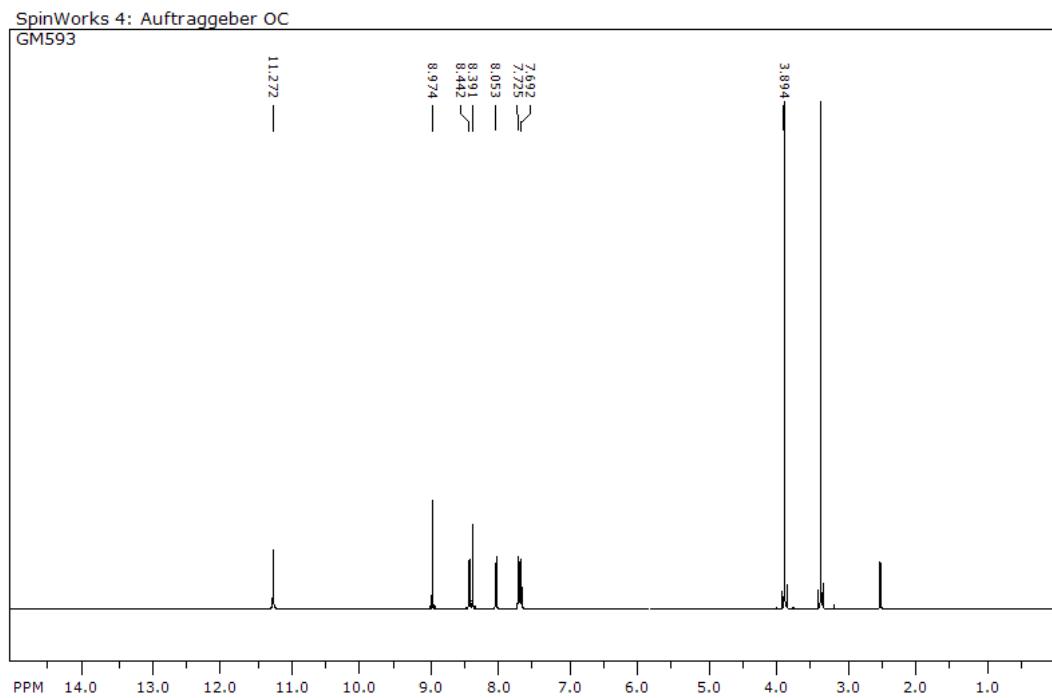


Methyl 2-oxo-2-(quinolin-3-ylamino)acetate (13a)

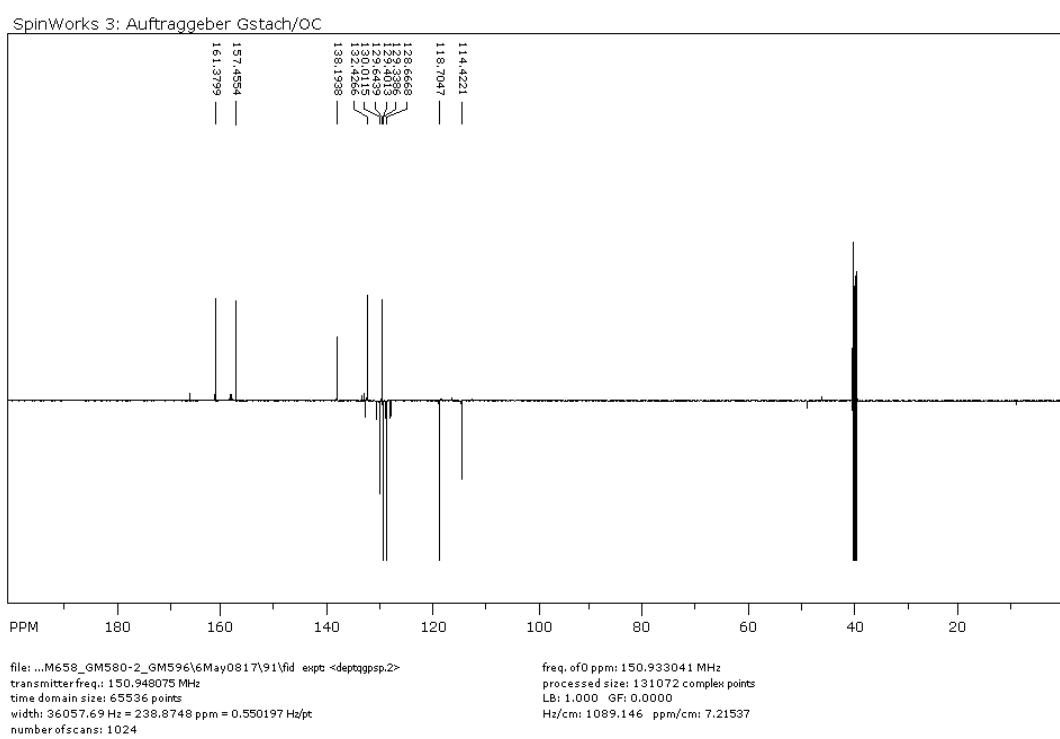
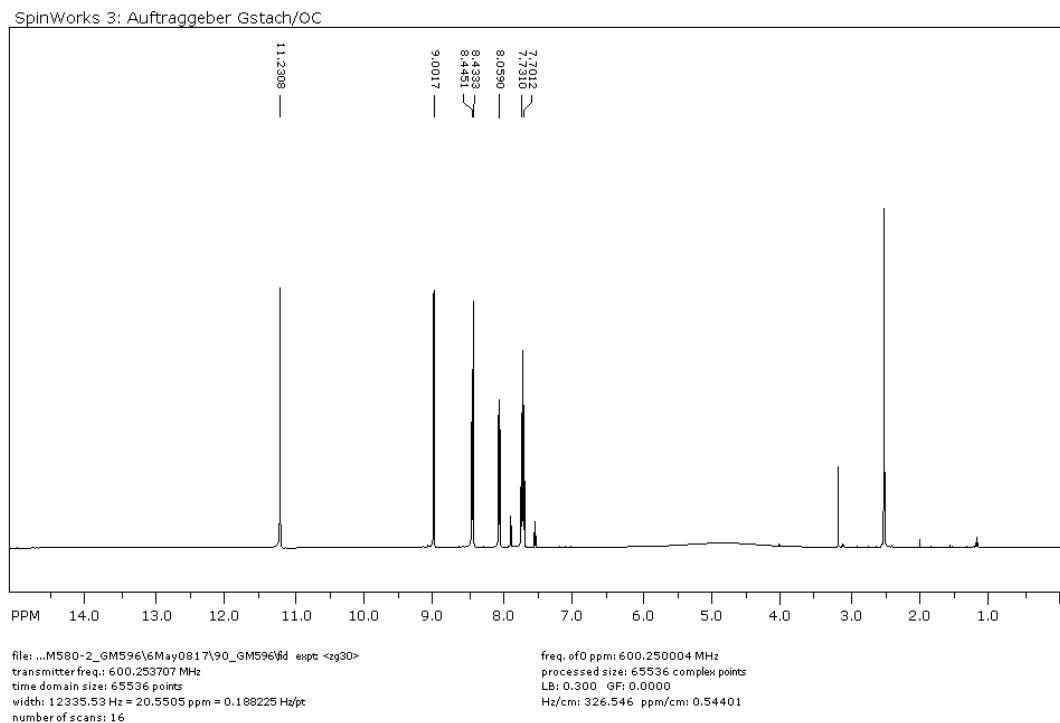


Methyl 2-oxo-2-(quinolin-2-ylamino)acetate (14a)

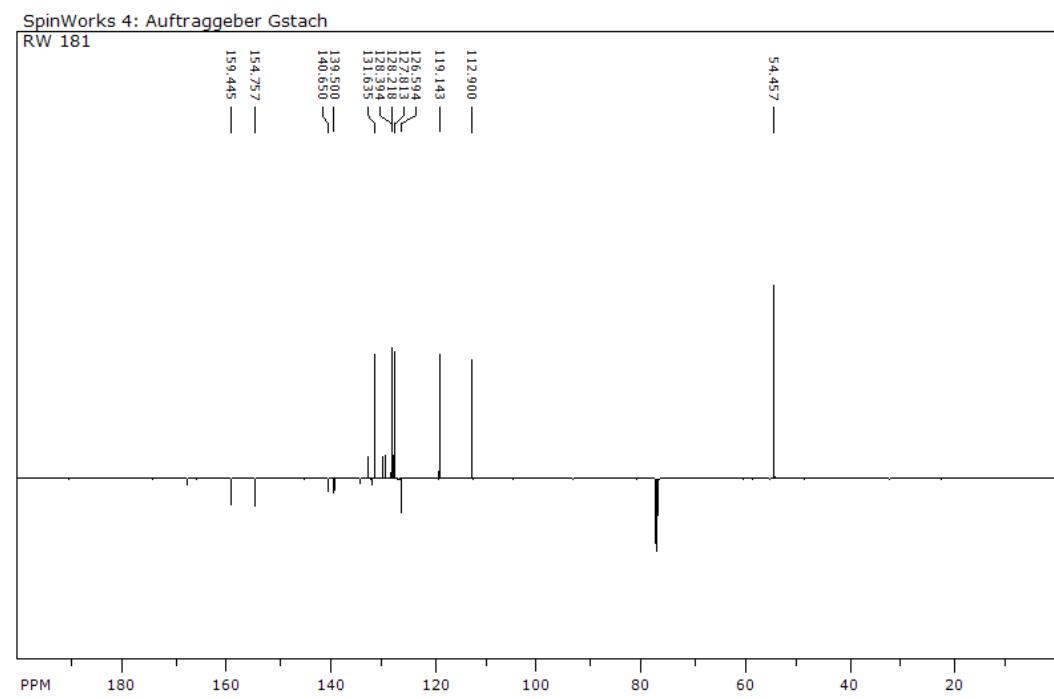
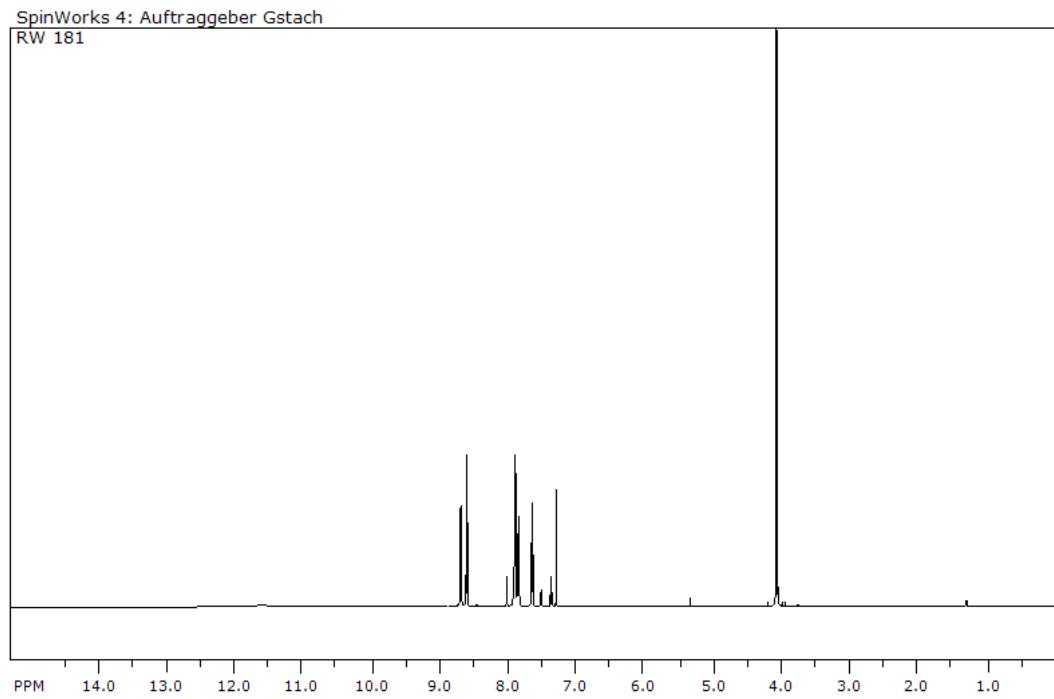


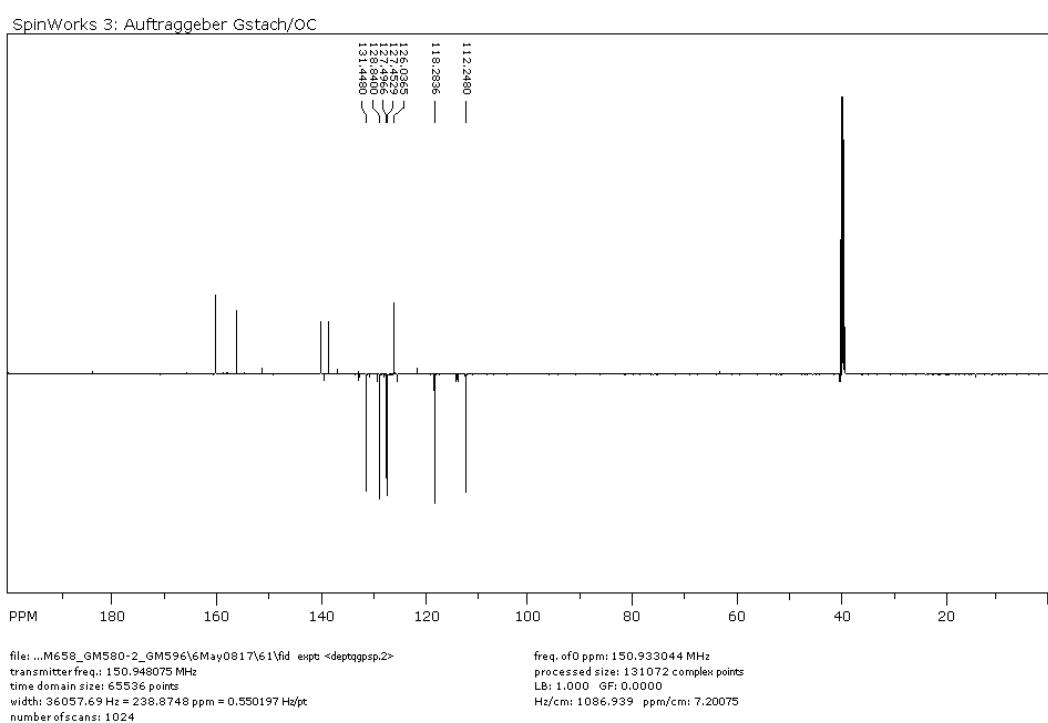
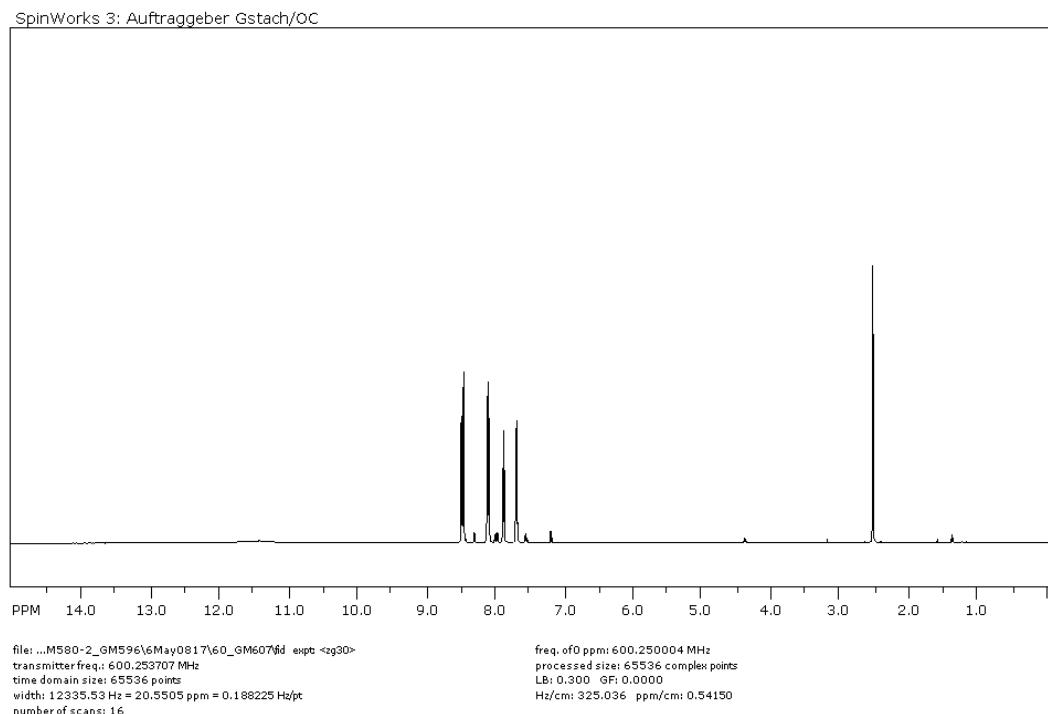
3-(2-Methoxy-2-oxoacetamido)quinoline 1-oxide (15a)

3-(Carboxyformamido)quinoline 1-oxide (15b)

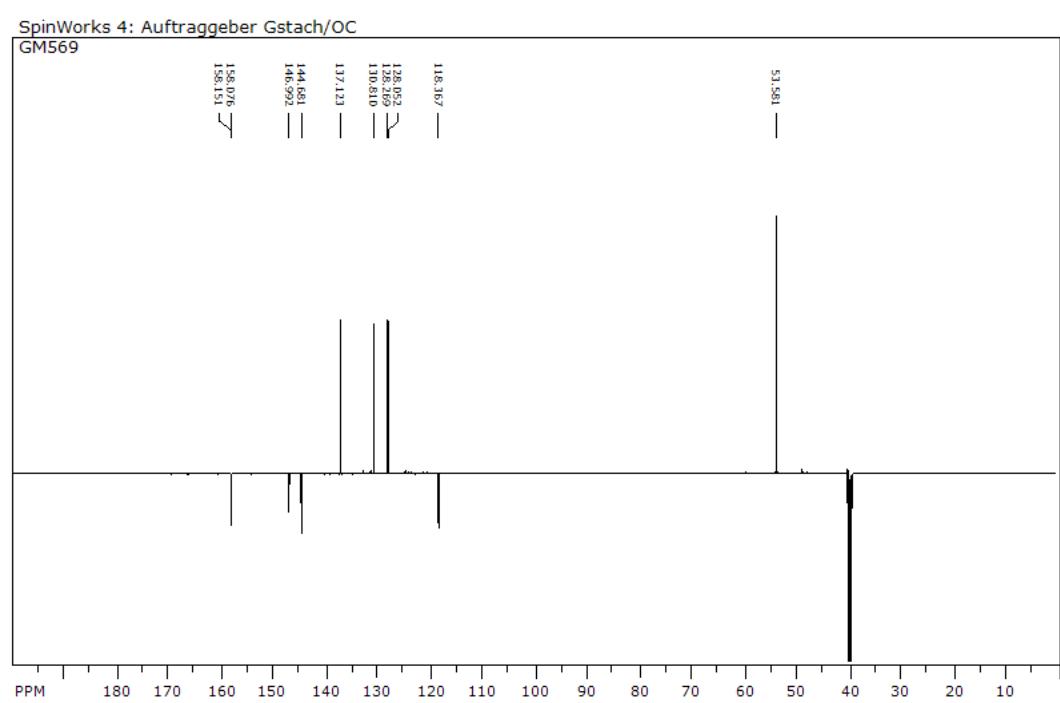
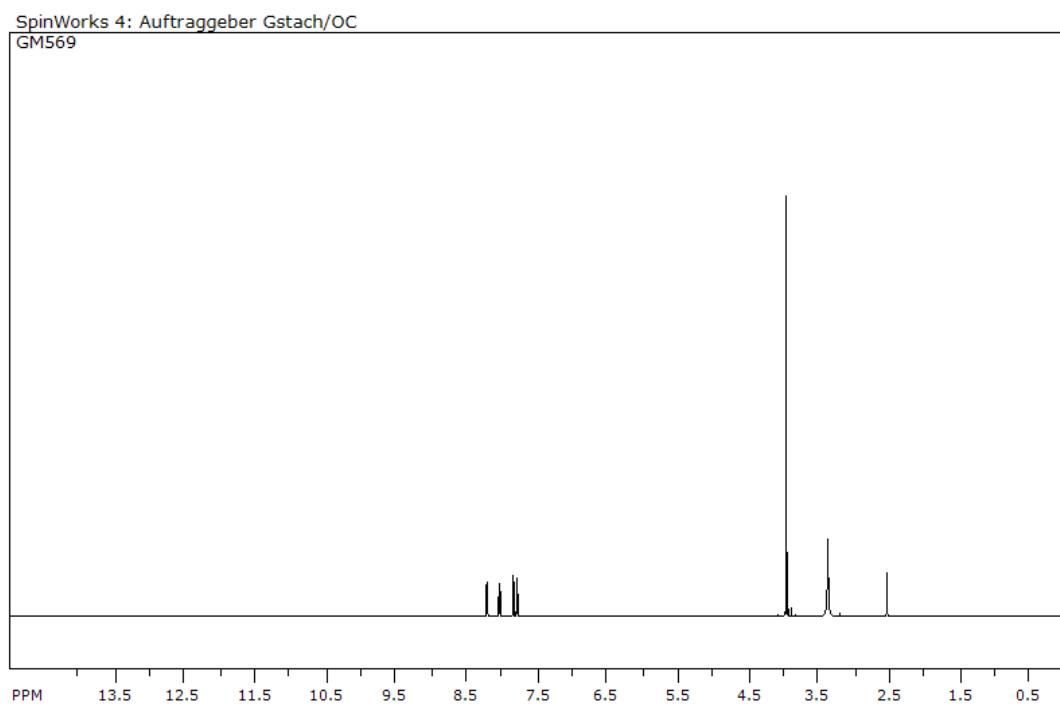


2-(2-Methoxy-2-oxoacetamido)quinoline 1-oxide (16a)

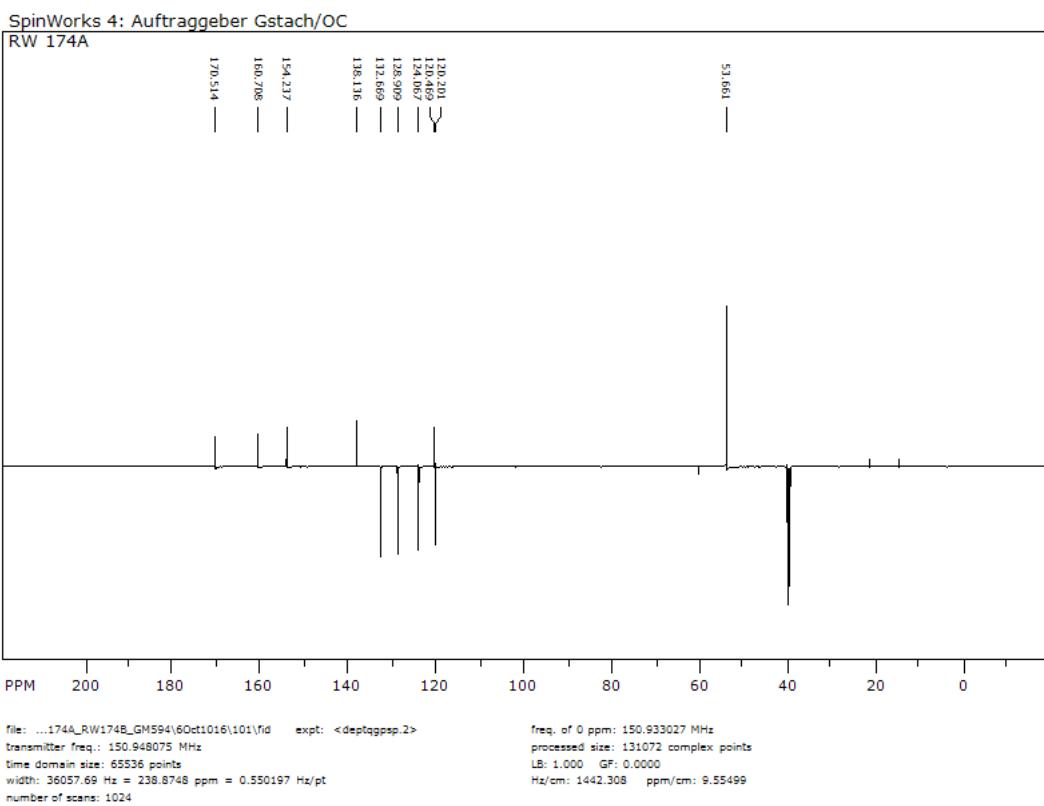
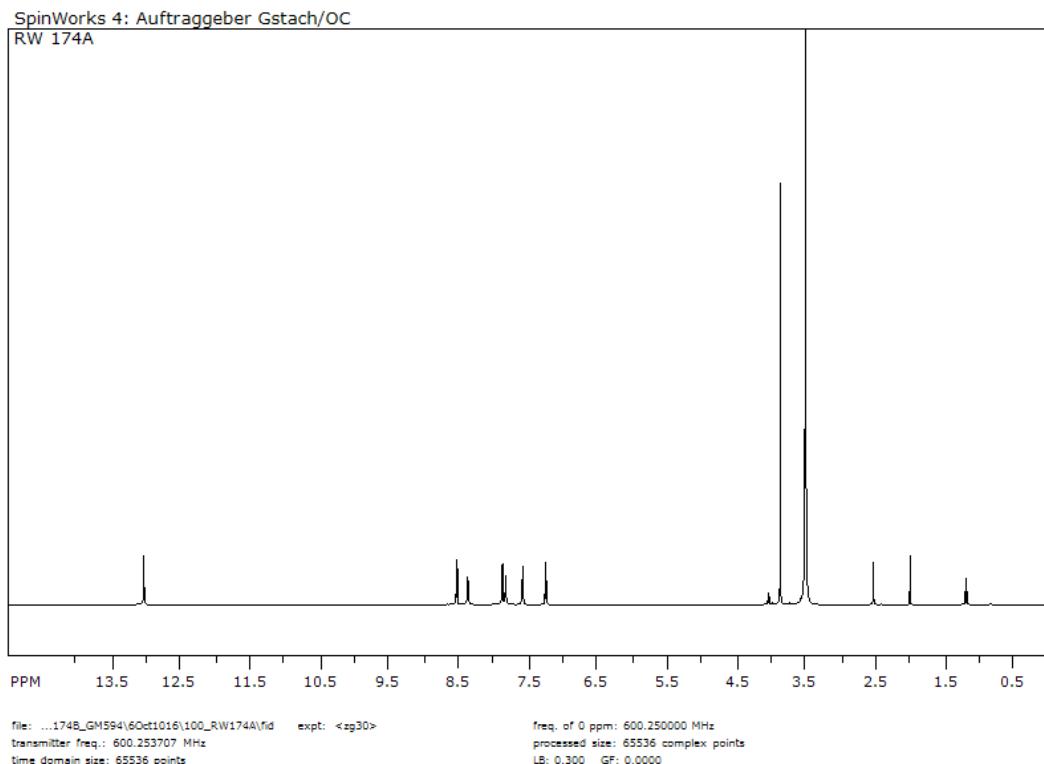


2-(Carboxyformamido)quinoline 1-oxide (16b)

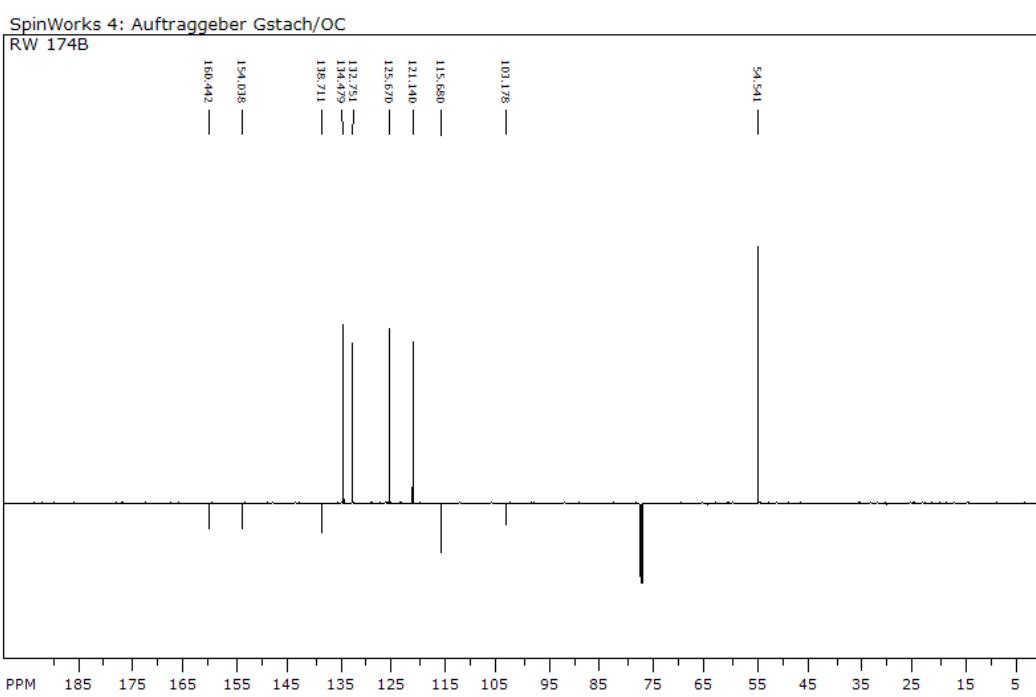
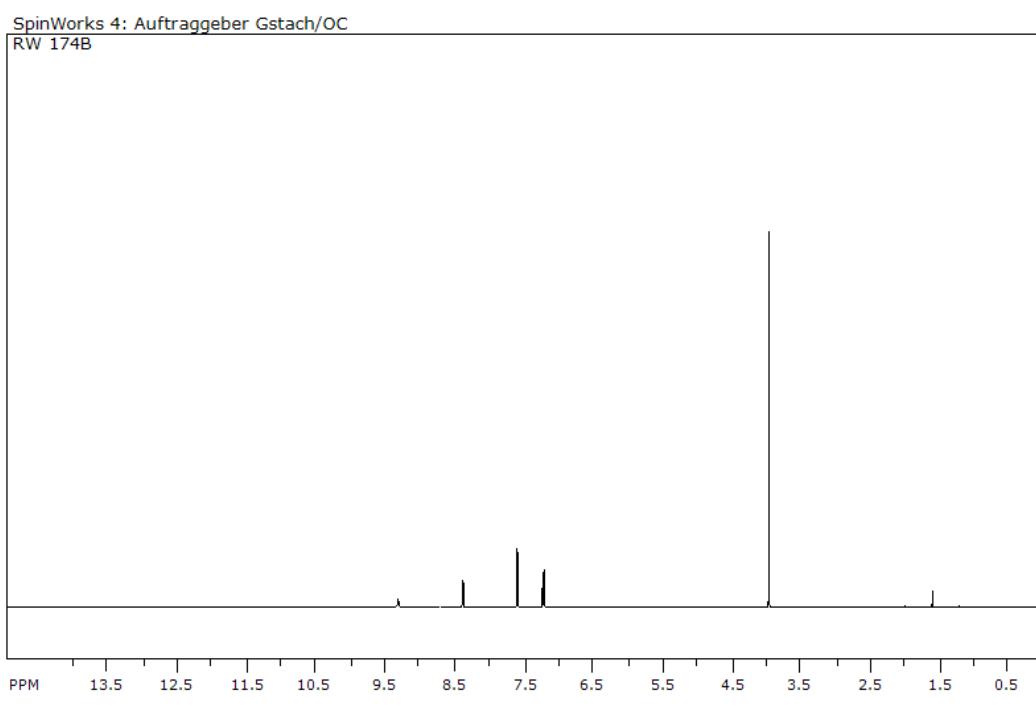
Methyl 4-oxo-4*H*-benzo[*d*][1,3]oxazine-2-carboxylate (17a)



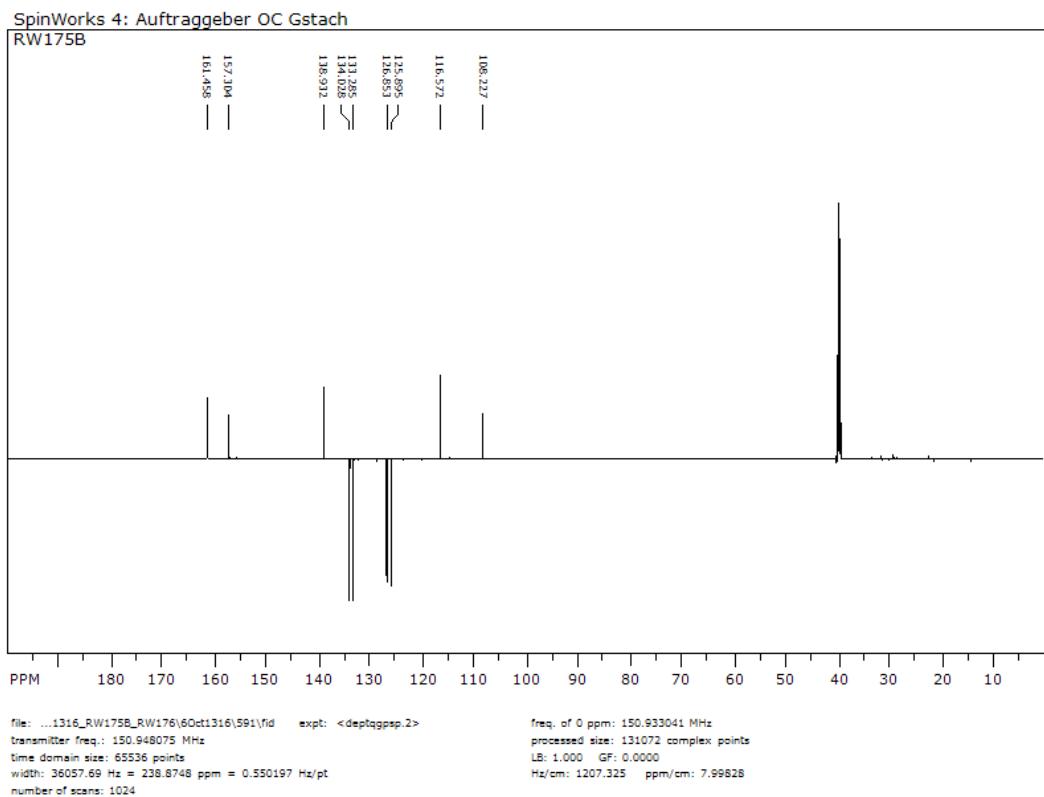
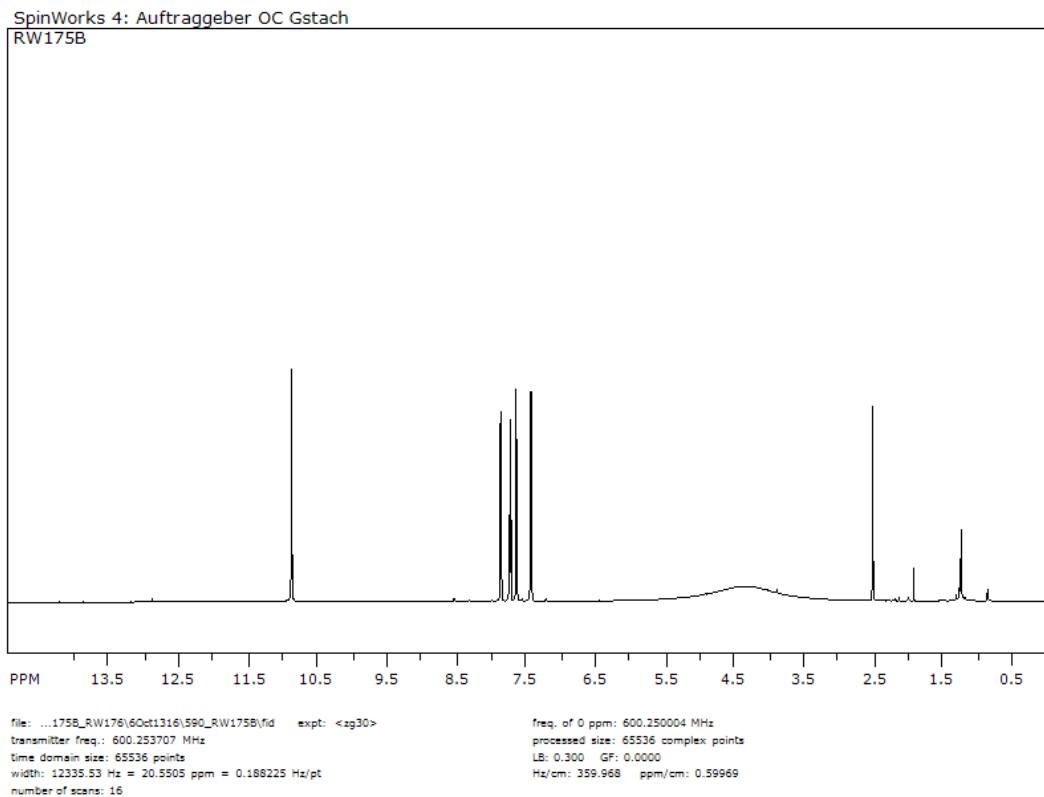
Methyl 2-((2-carbamoylphenyl)amino)-2-oxoacetate (18a)



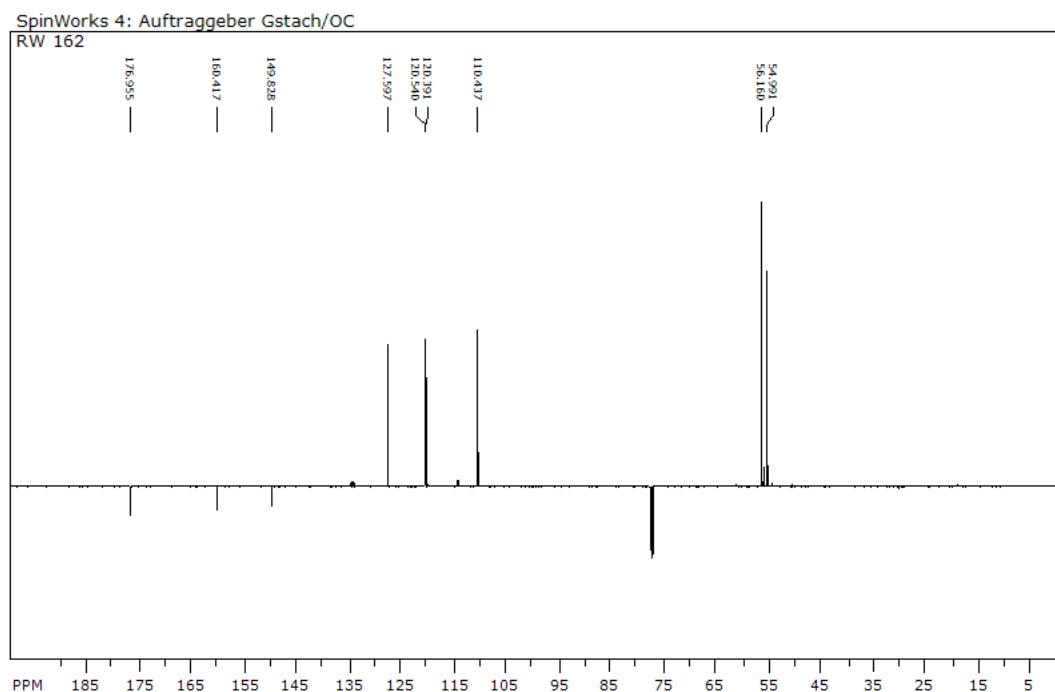
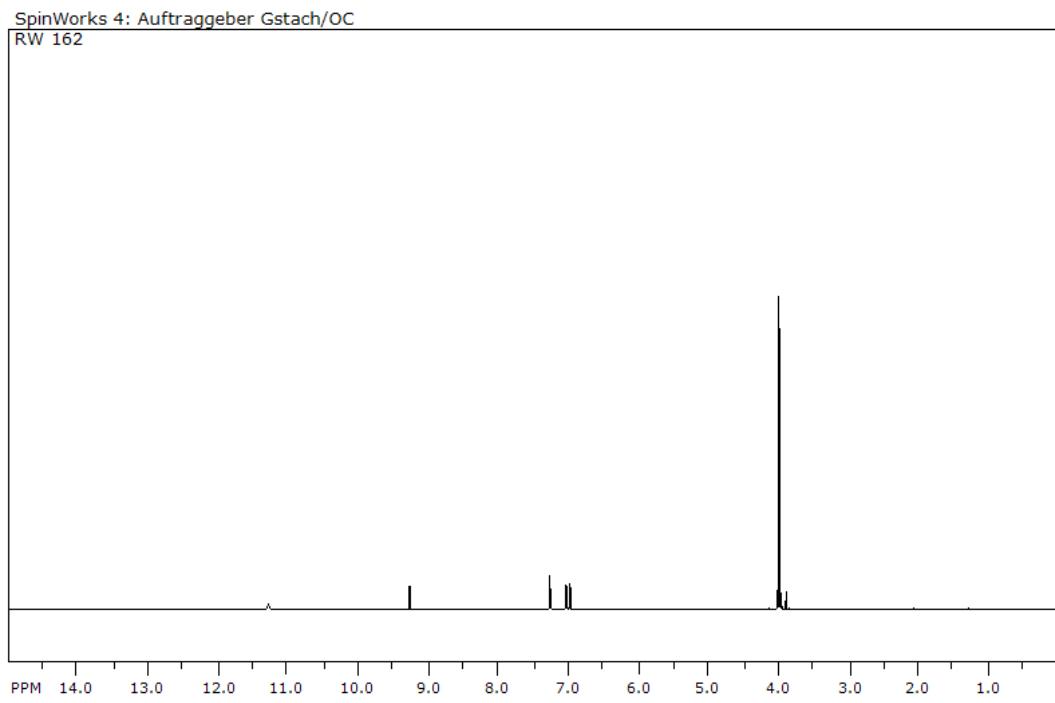
Methyl 4-oxo-1,4-dihydroquinazoline-2-carboxylate (19a)



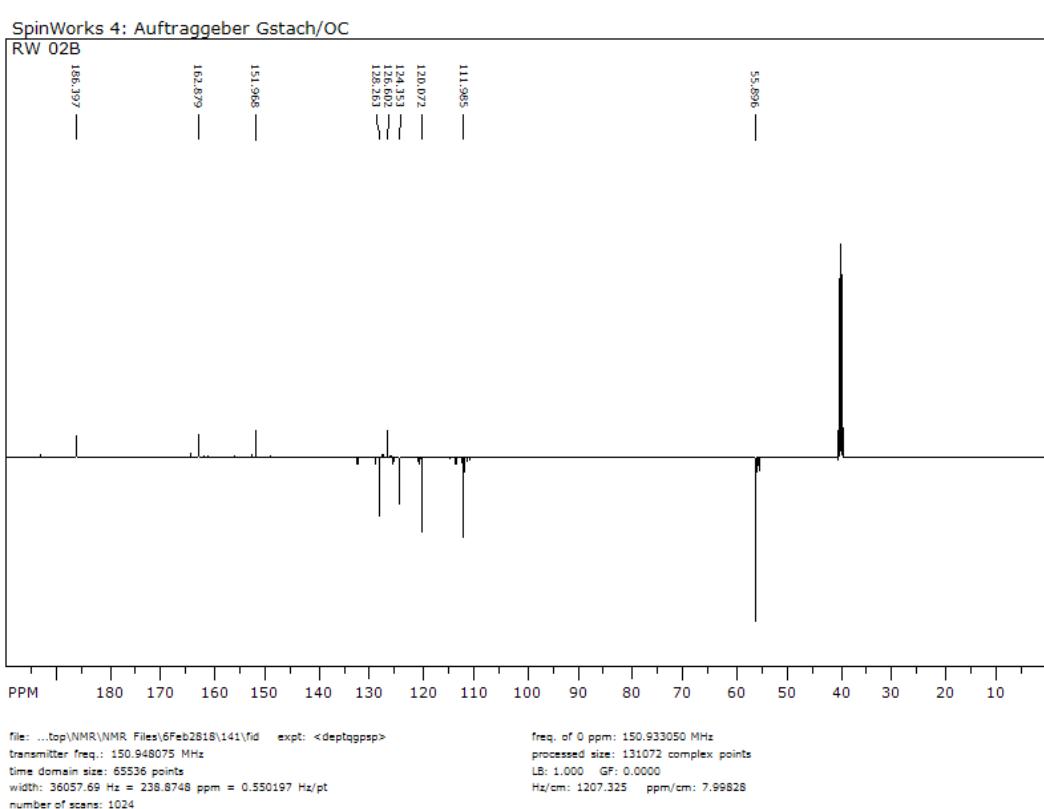
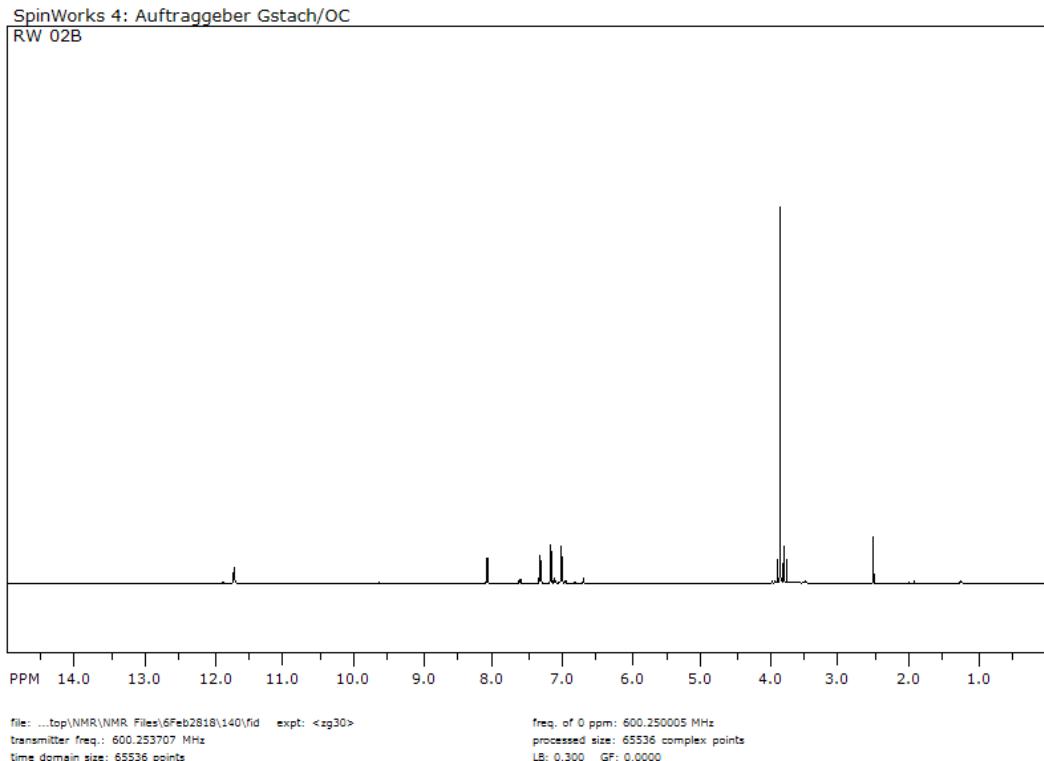
4-oxo-1,4-dihydroquinazoline-2-carboxylic acid (19b)



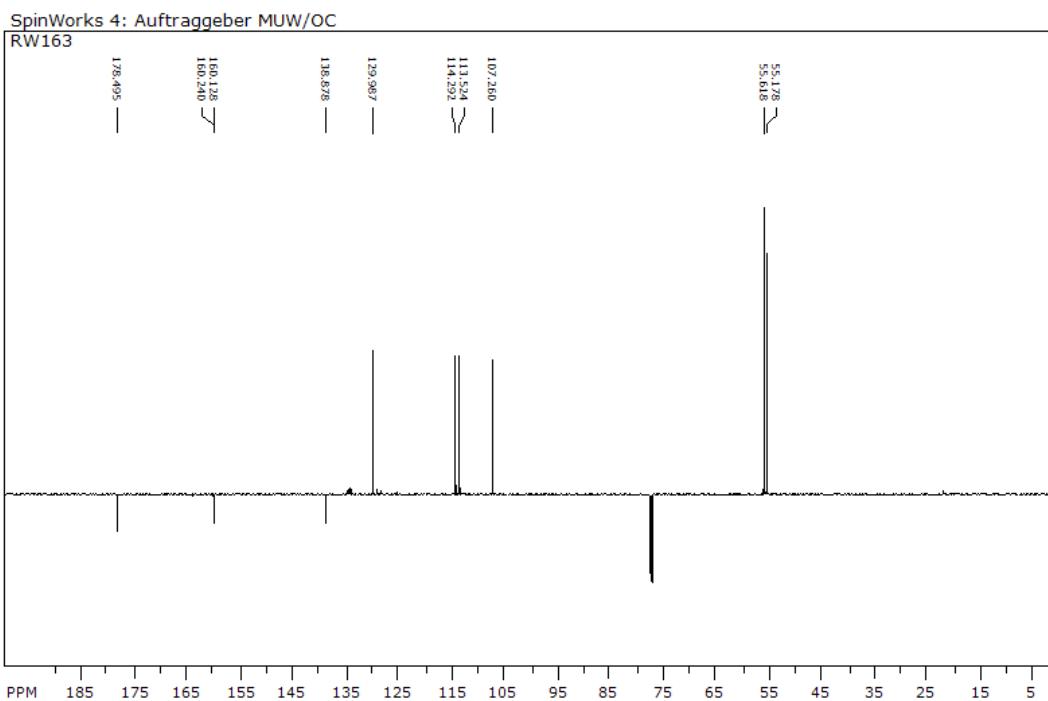
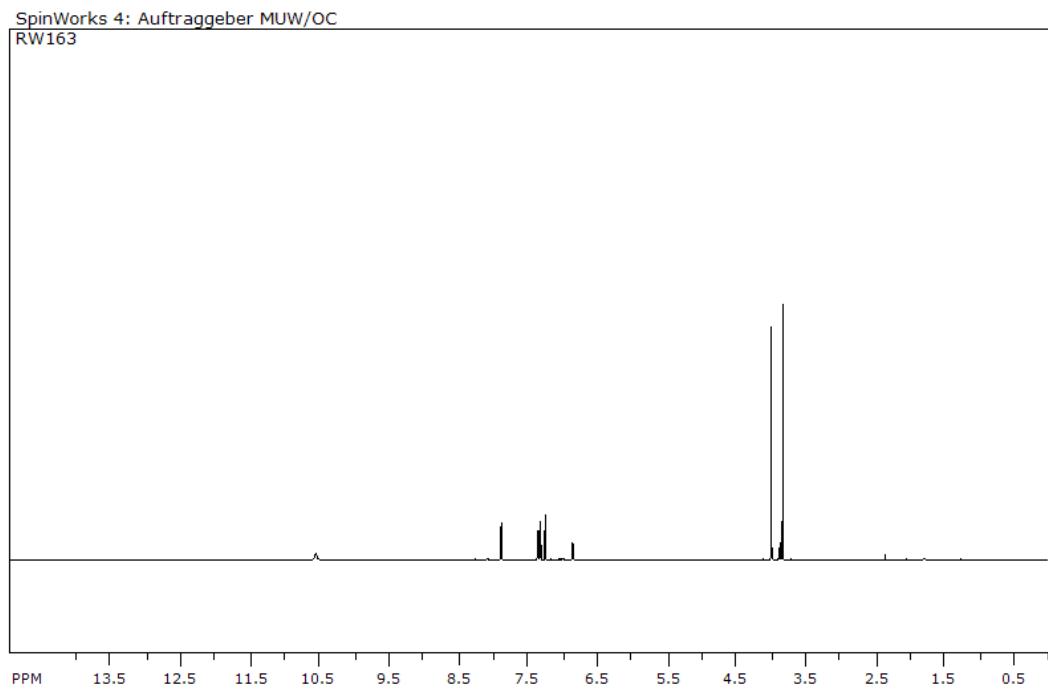
Methyl 2-((2-methoxyphenyl)amino)-2-thioxoacetate (20a)



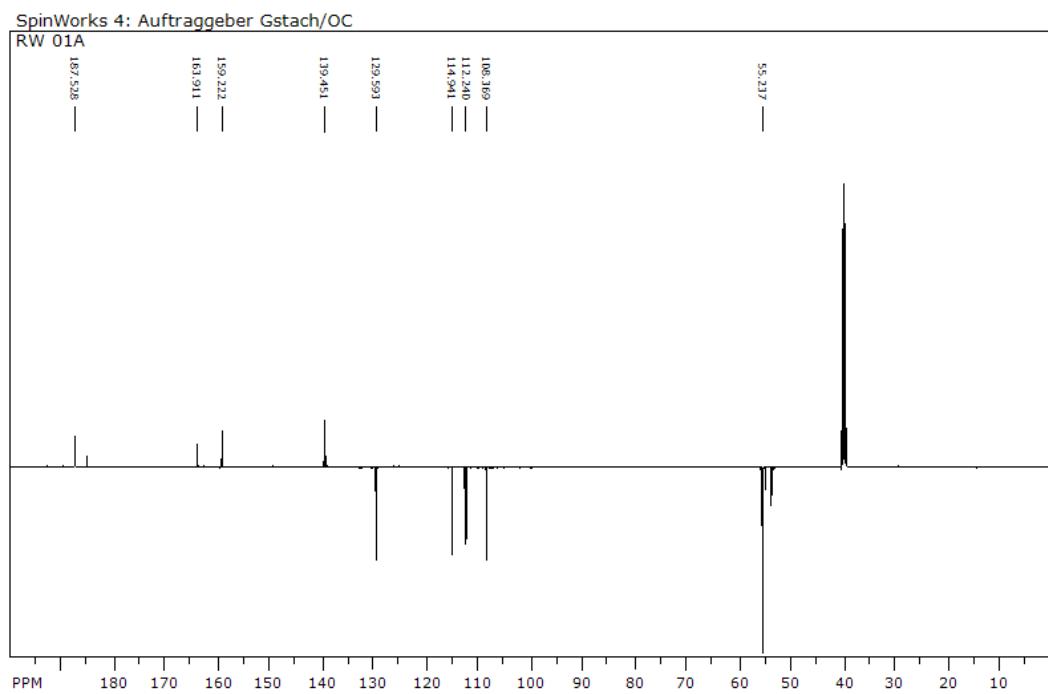
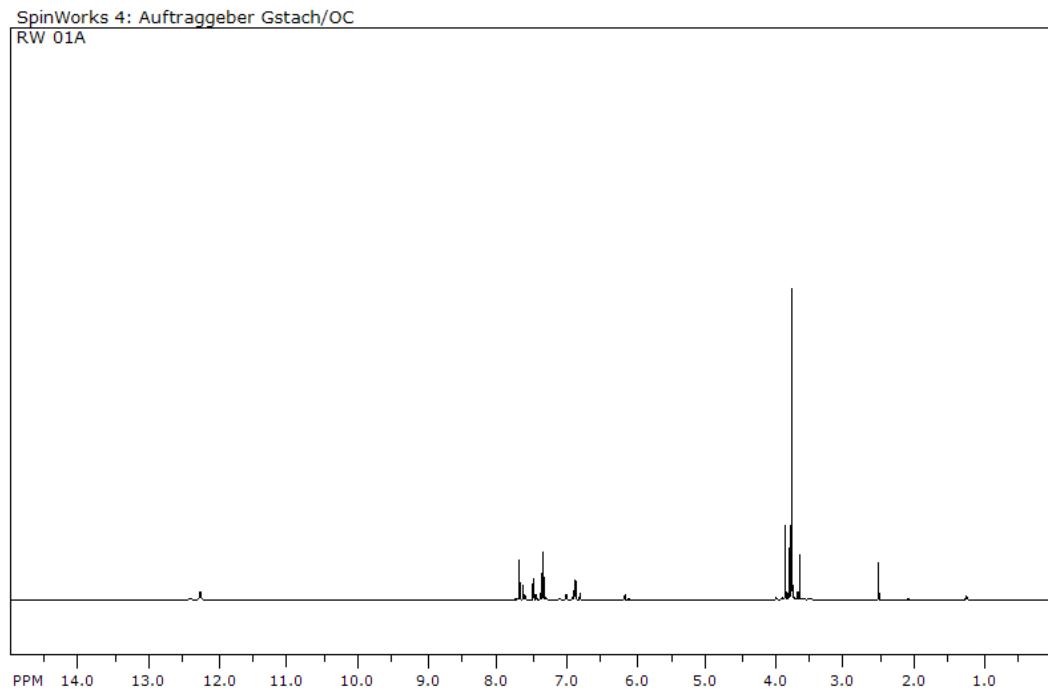
2-((2-methoxyphenyl)amino)-2-thioxoacetic acid (20b)



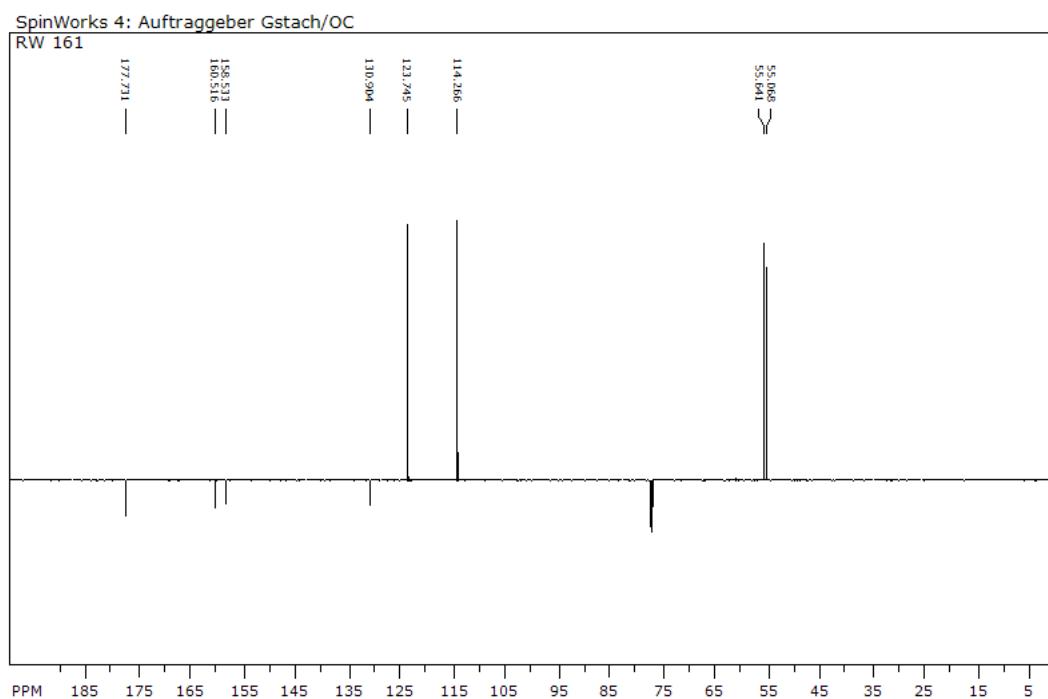
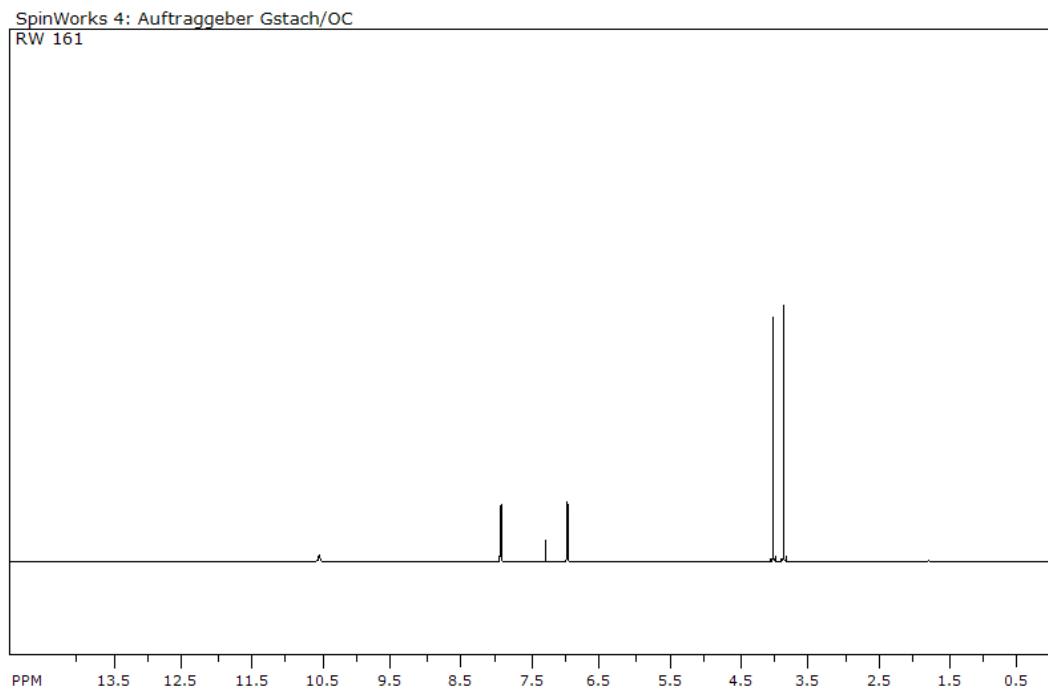
Methyl 2-((3-methoxyphenyl)amino)-2-thioxoacetate (21a)



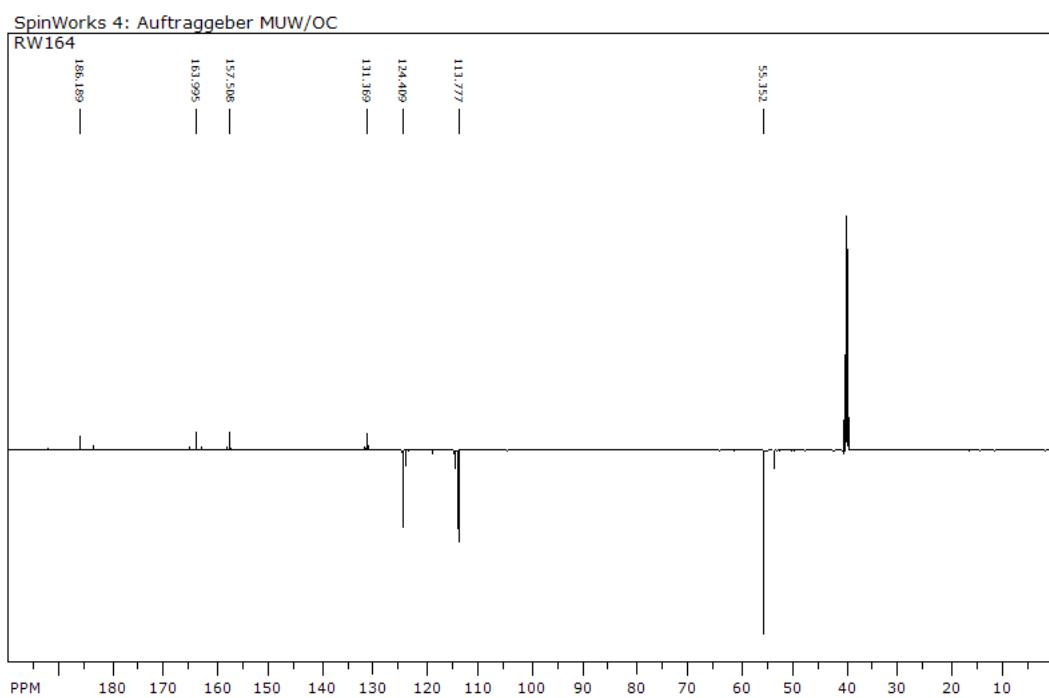
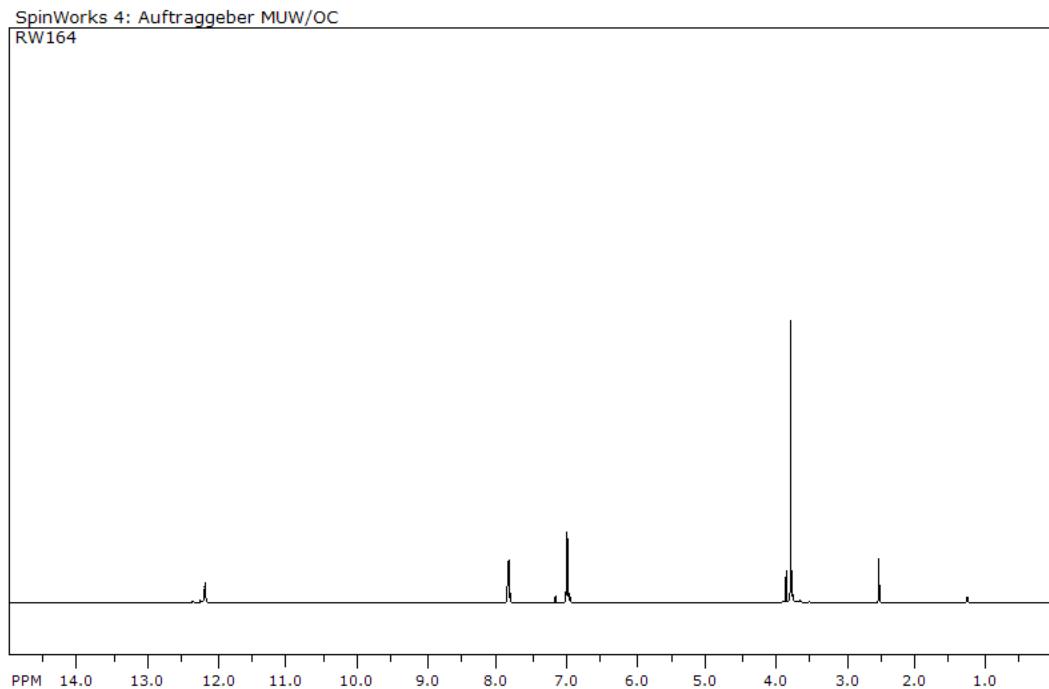
2-((3-methoxyphenyl)amino)-2-thioxoacetic acid (21b)

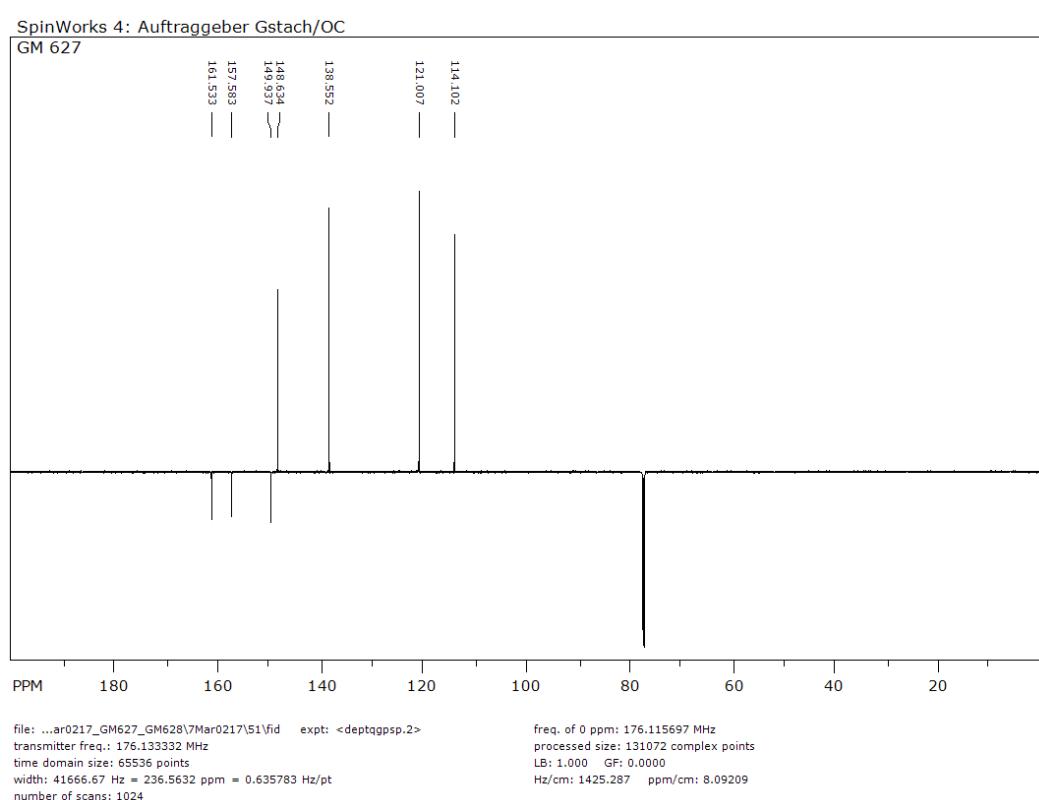
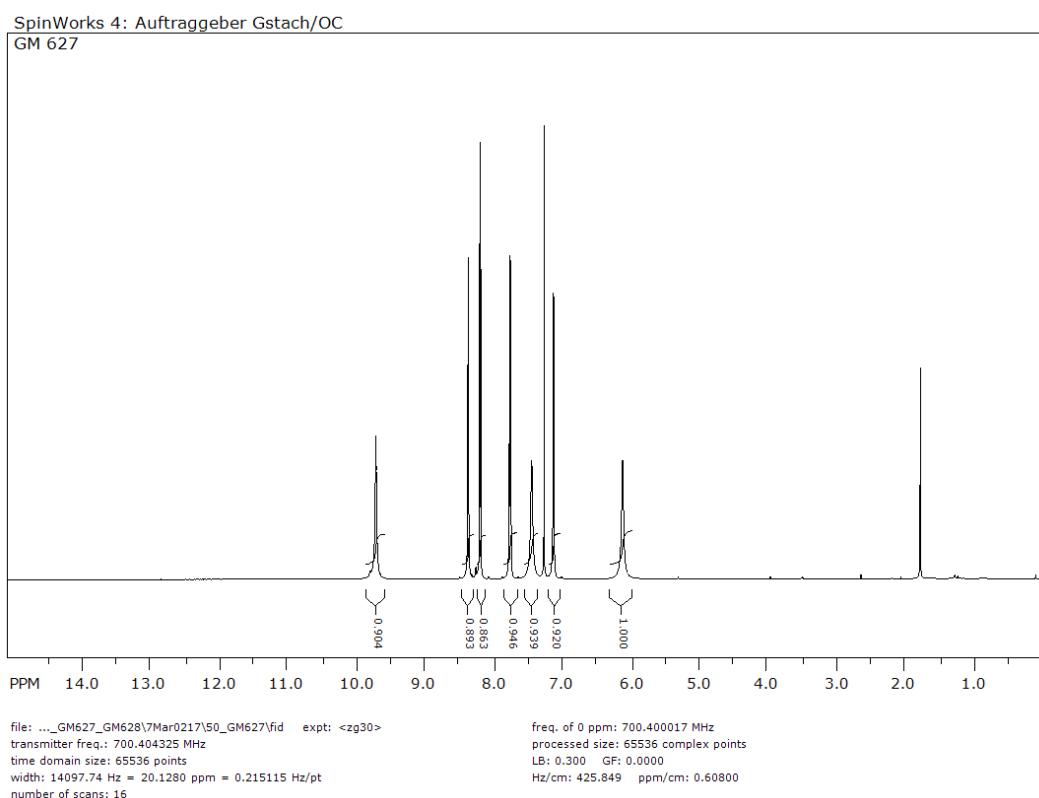


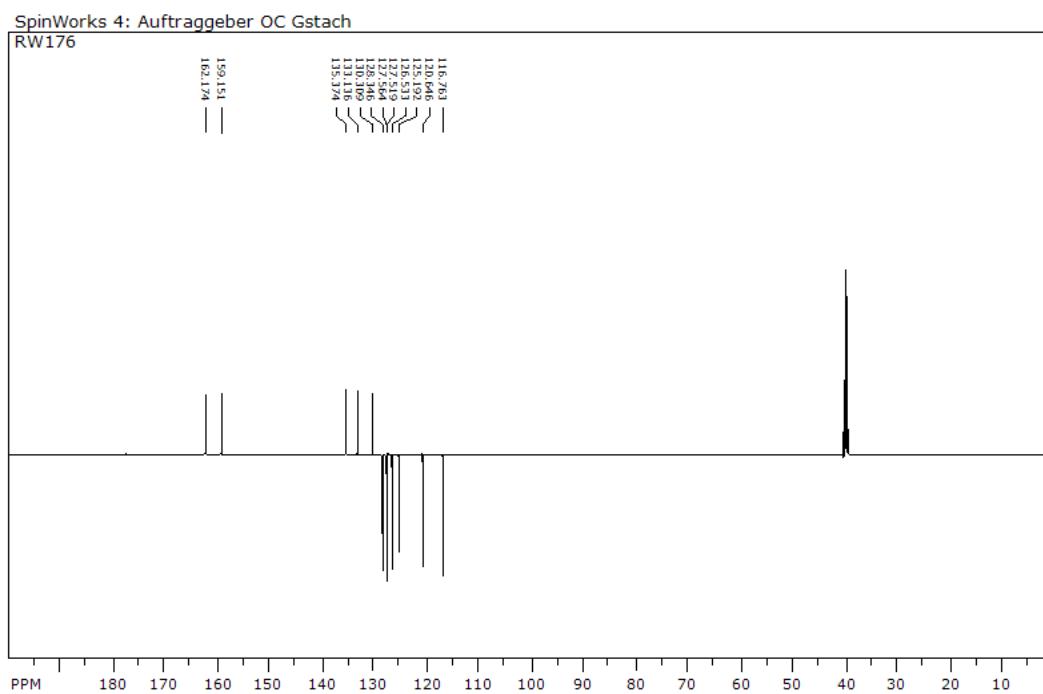
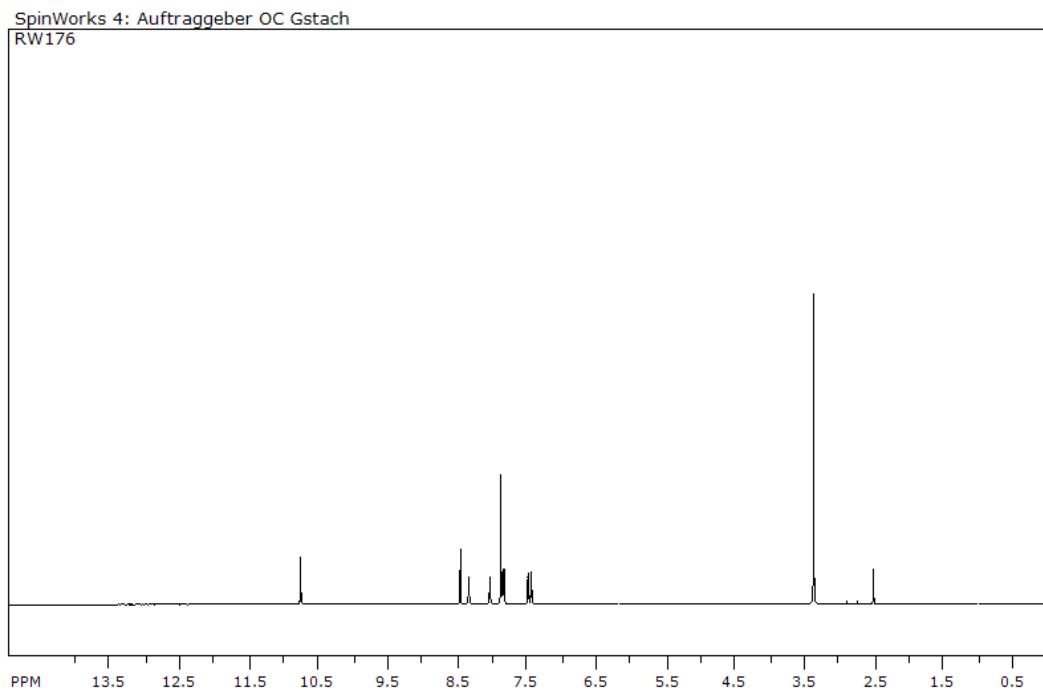
Methyl 2-((4-methoxyphenyl)amino)-2-thioxoacetate (22a)



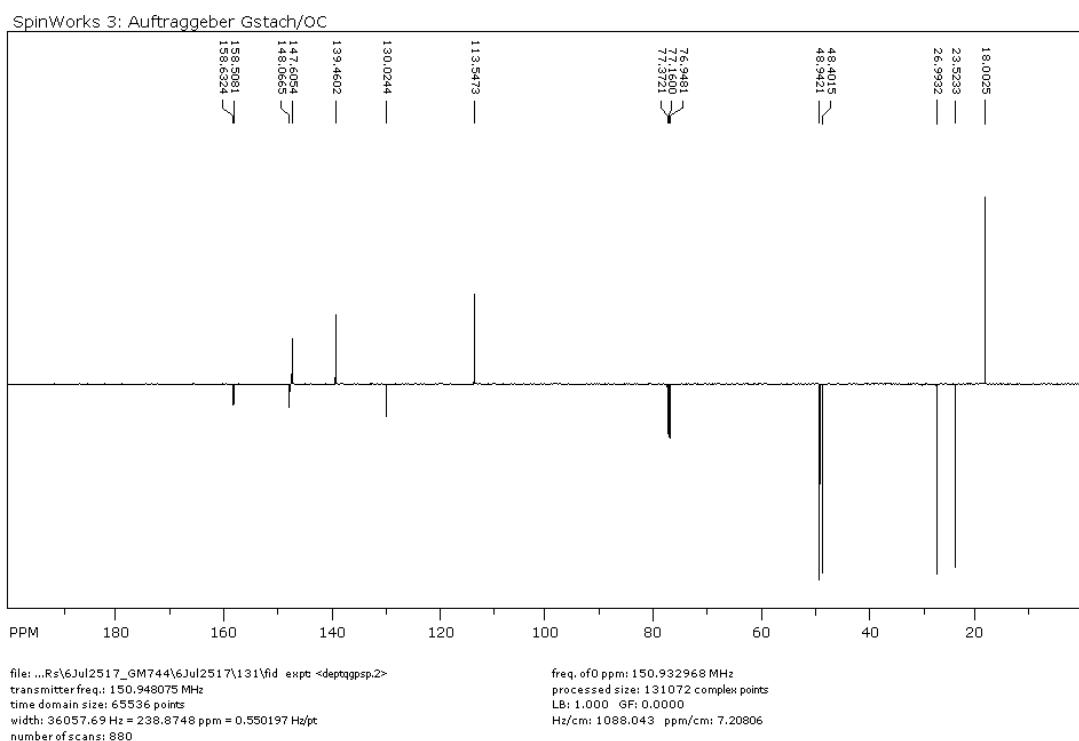
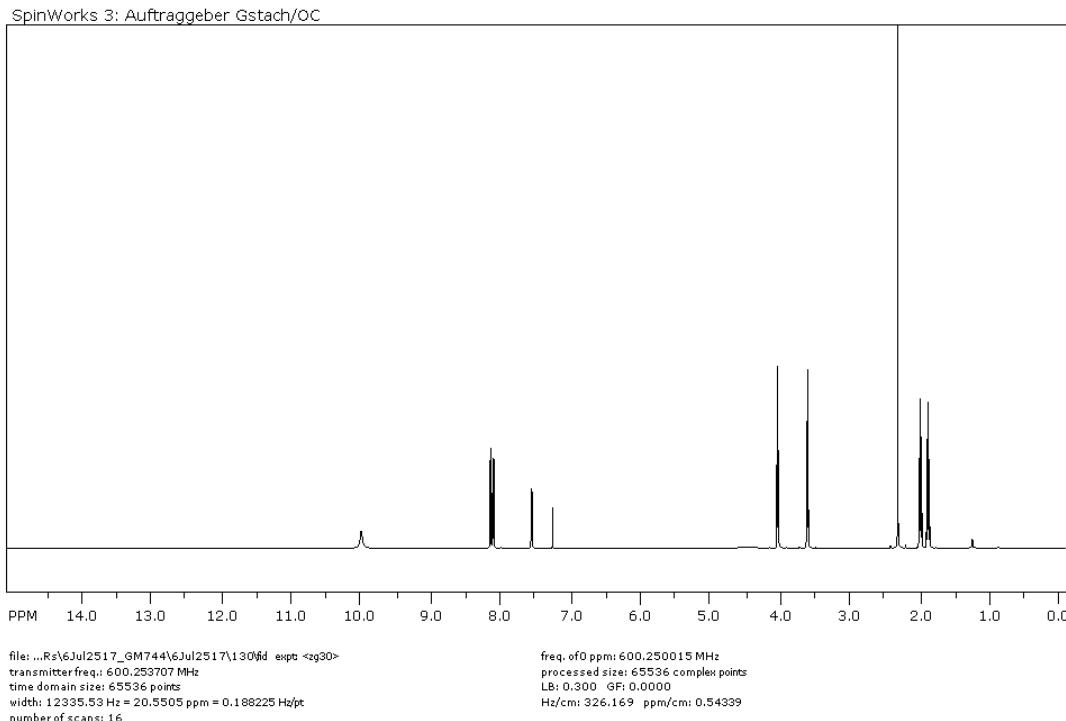
2-((4-methoxyphenyl)amino)-2-thioxoacetic acid (22b)



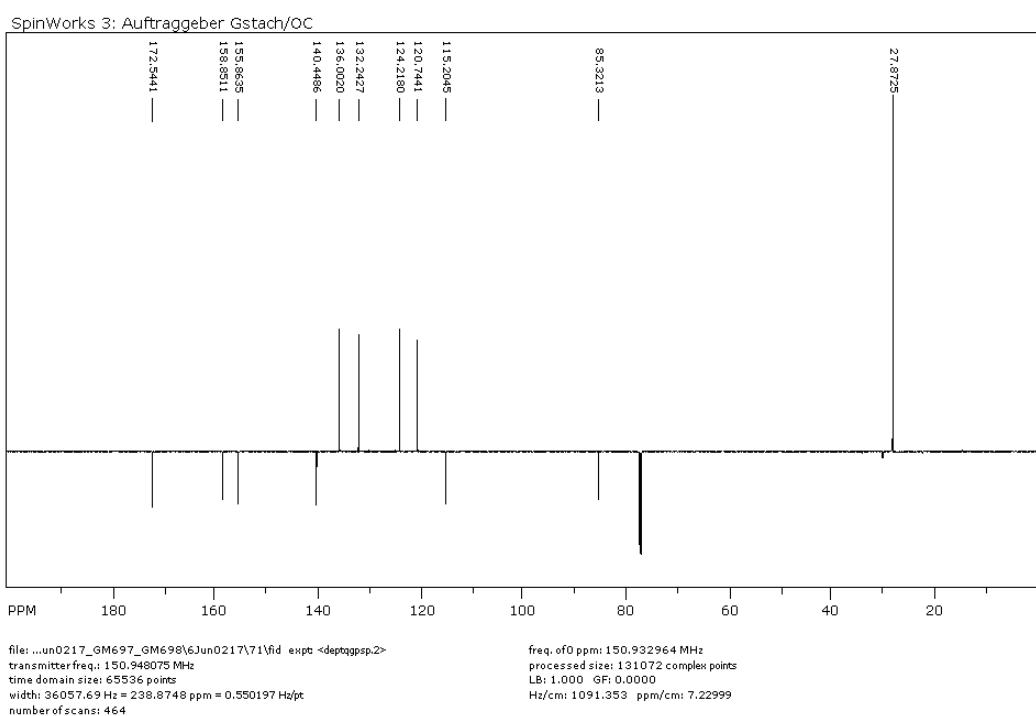
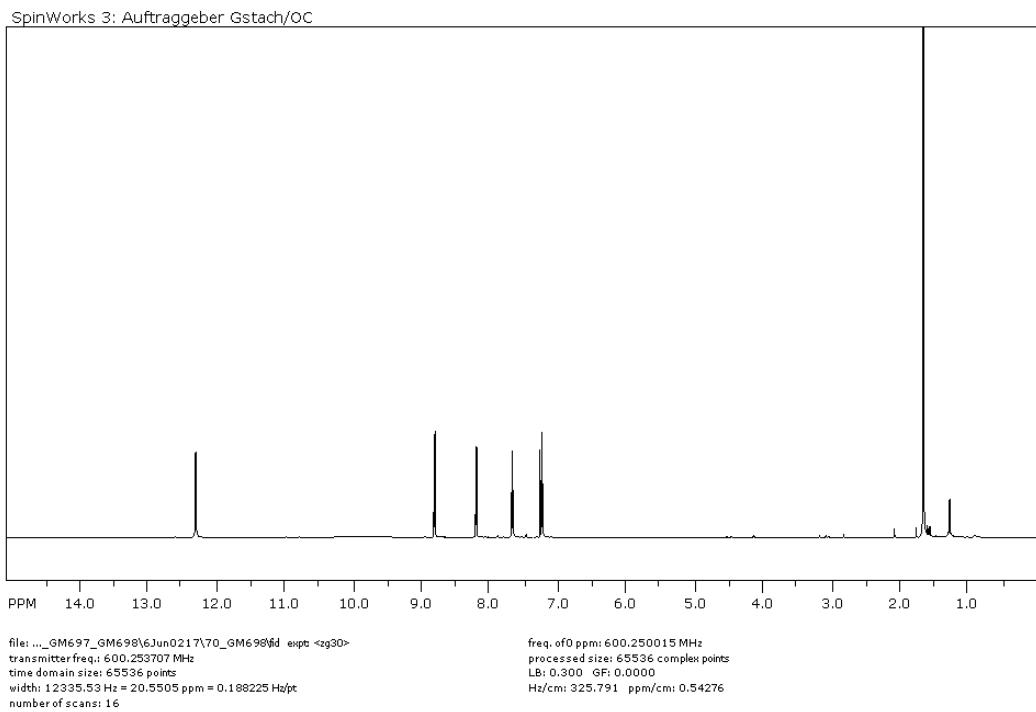
N¹-(pyridin-2-yl)oxalamide (23)

N¹-(naphthalen-2-yl)oxalamide (24)

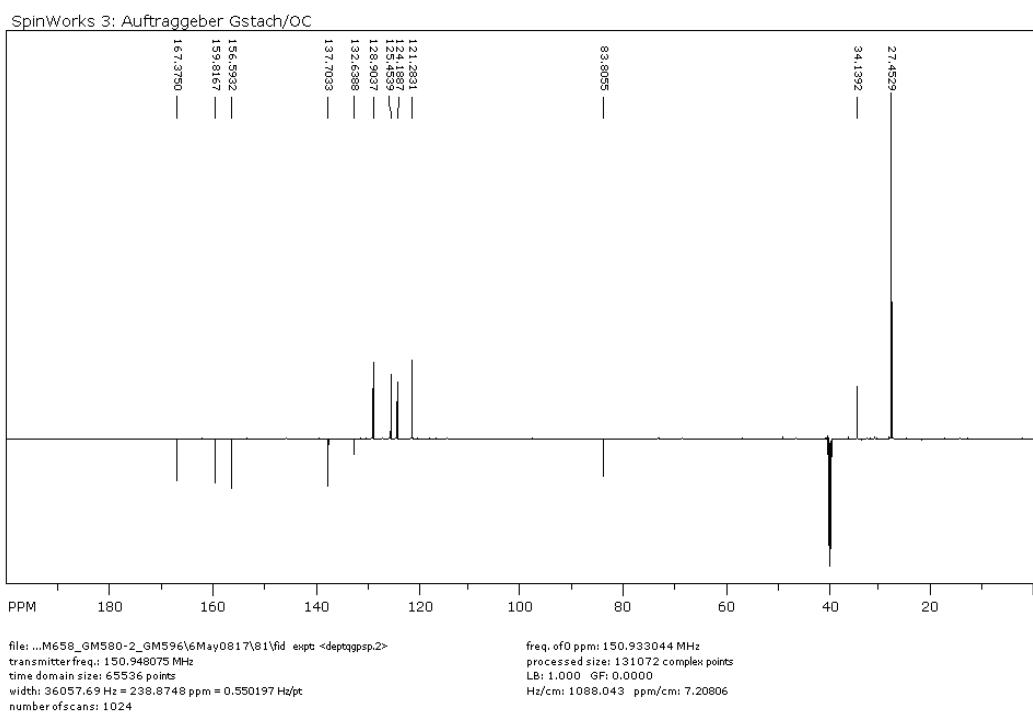
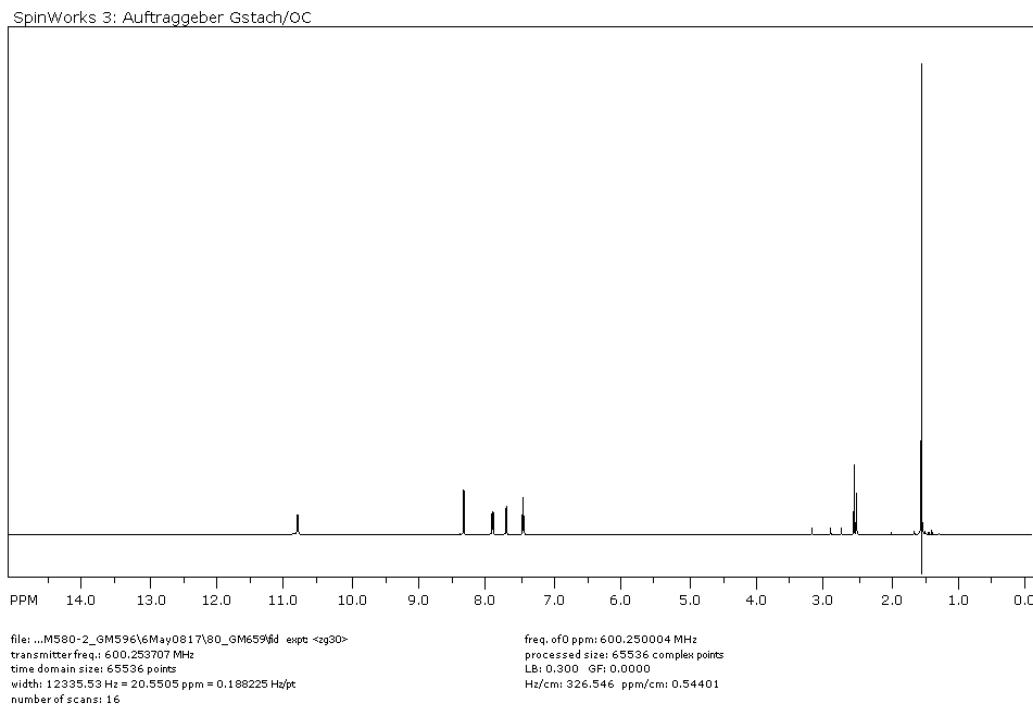
N-(6-methylpyridin-2-yl)-2-oxo-2-(pyrrolidin-1-yl)acetamide (25)



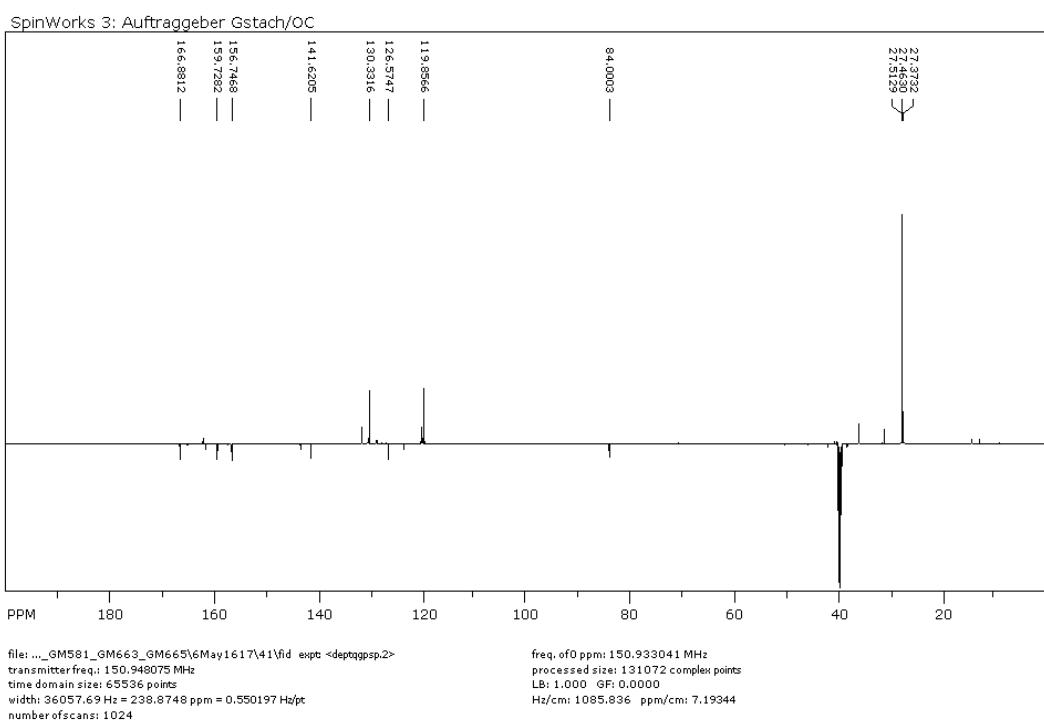
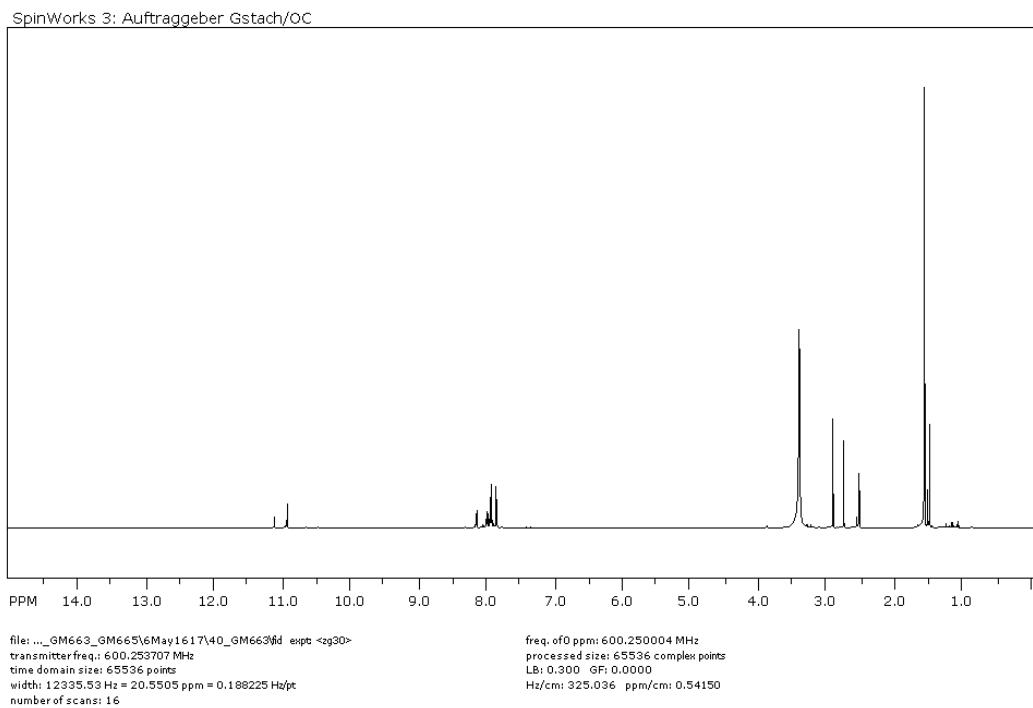
2-(2-*tert*-Butoxy-2-oxoacetamido)benzoic acid (26)

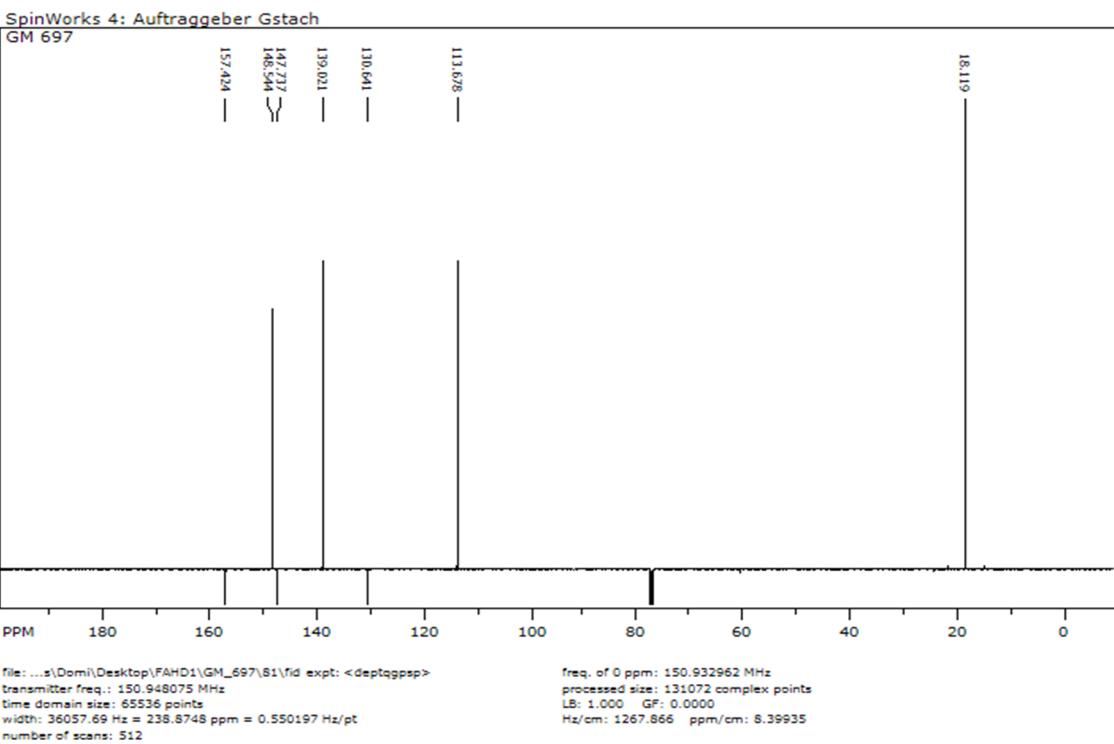
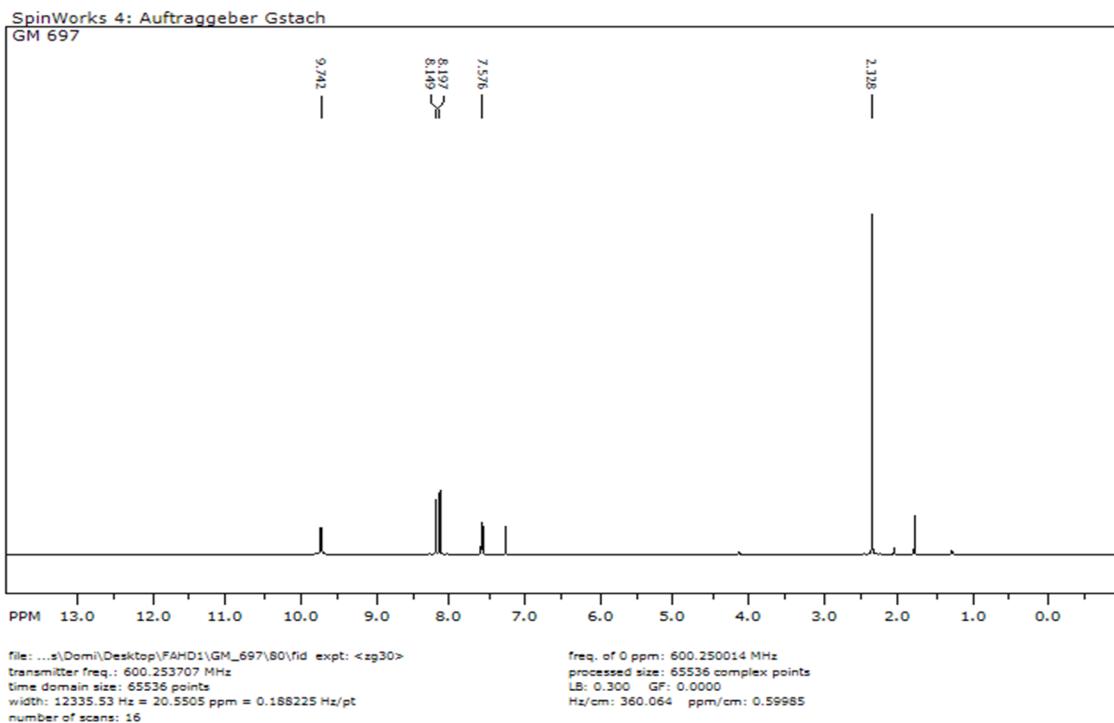


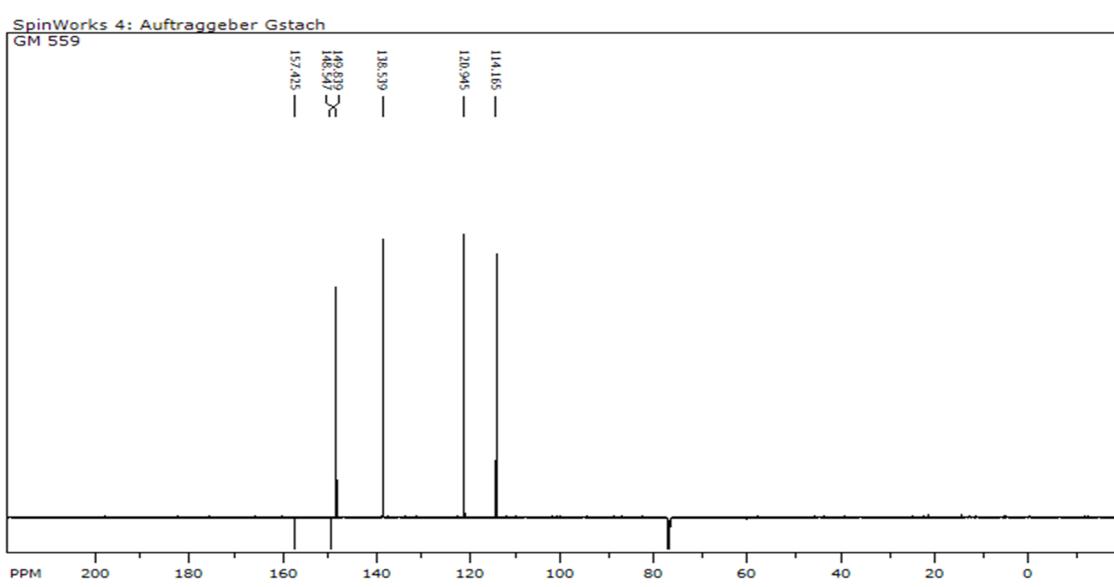
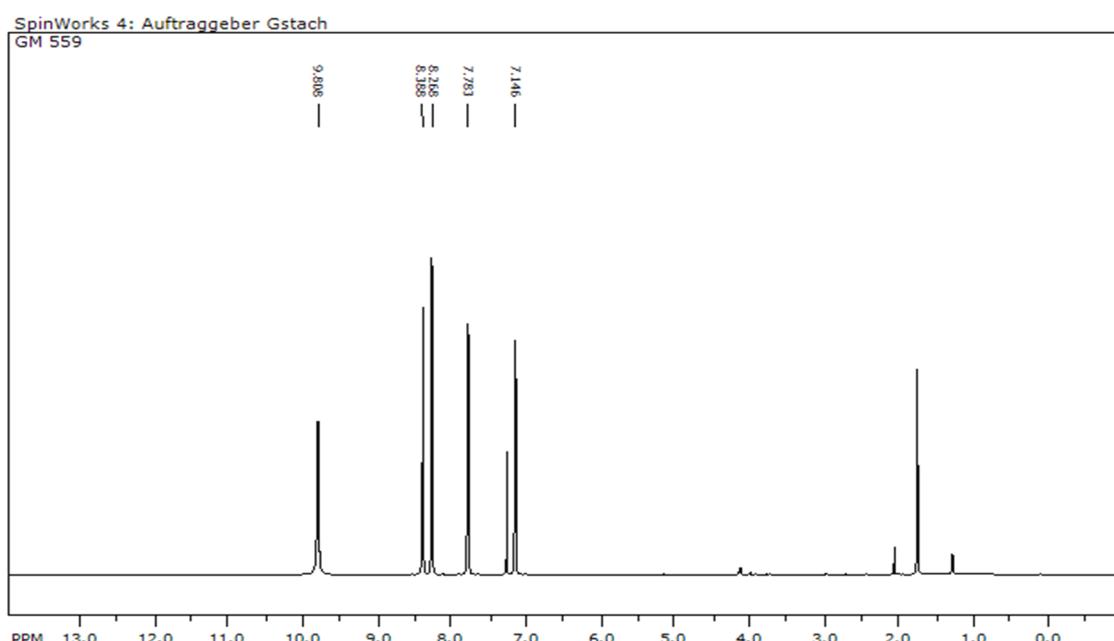
3-(2-*tert*-Butoxy-2-oxoacetamido)benzoic acid (27)

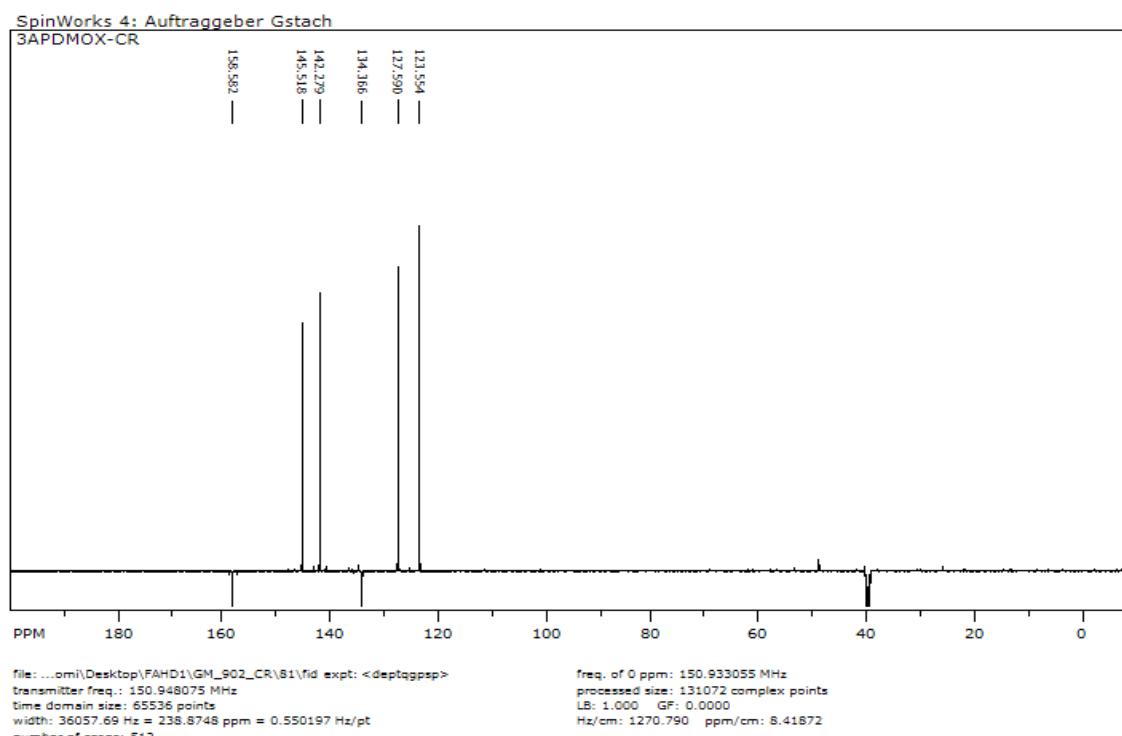
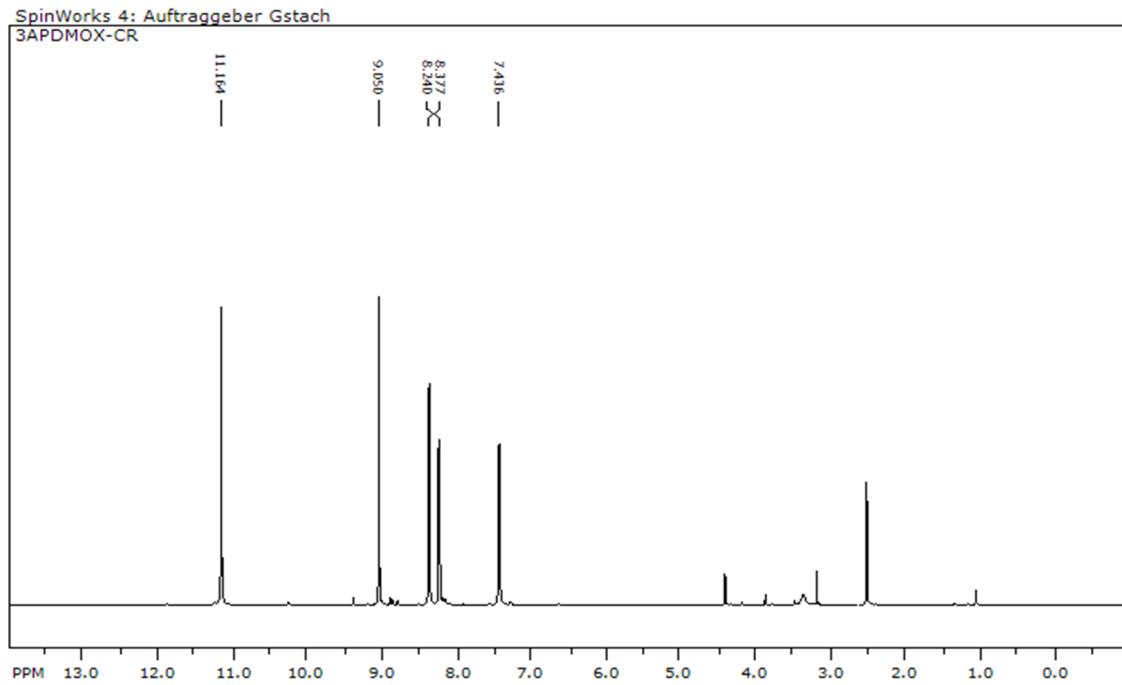


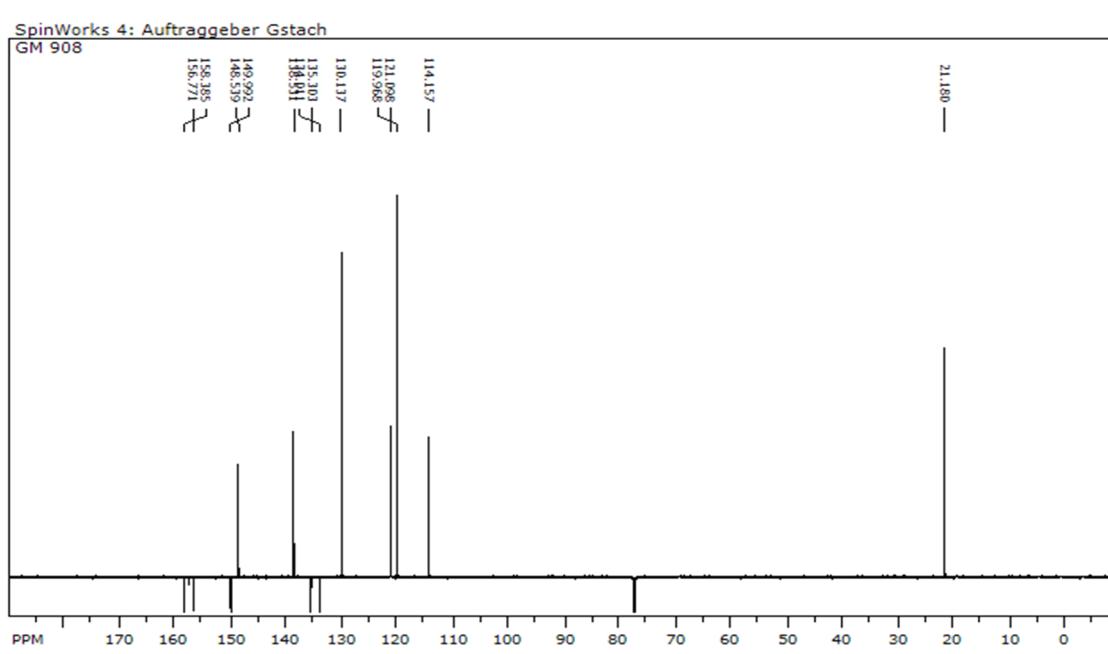
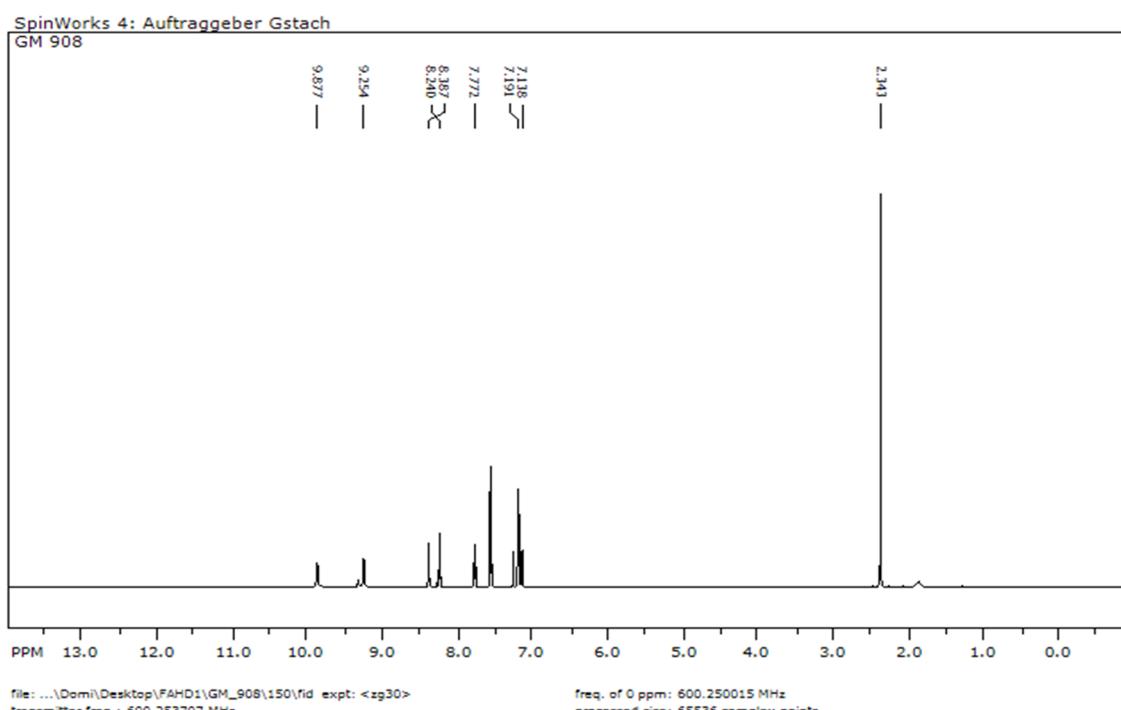
4-(2-*tert*-Butoxy-2-oxoacetamido)benzoic acid (28)

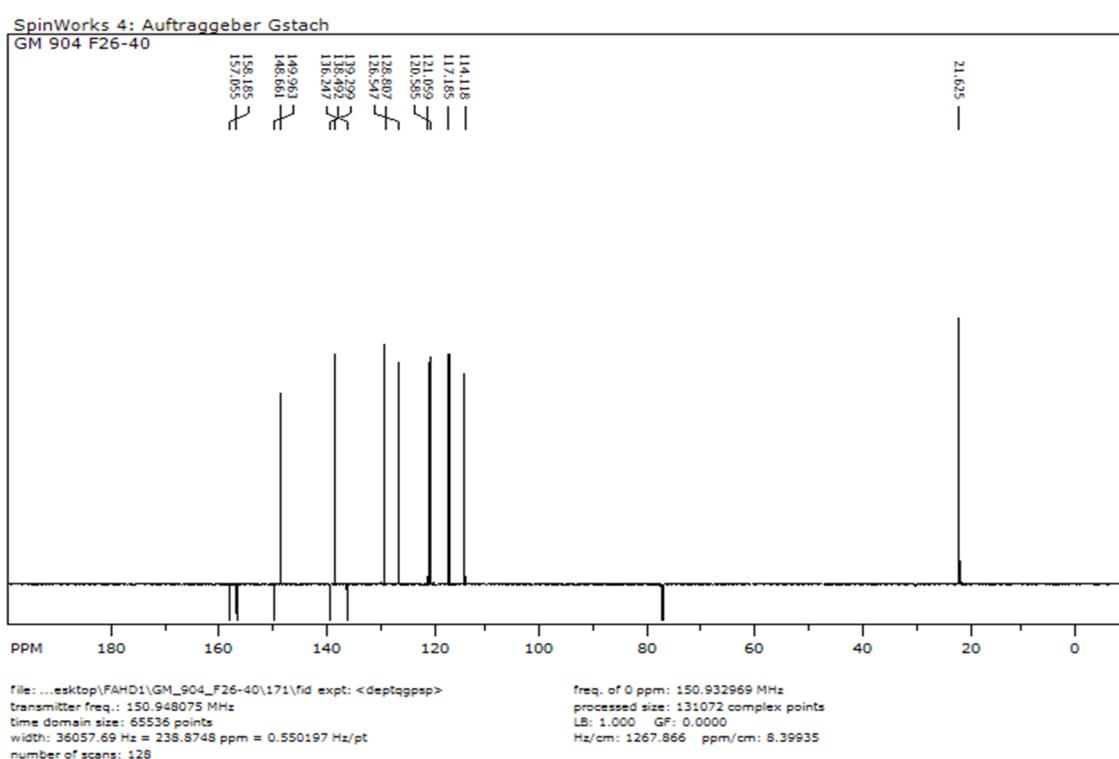
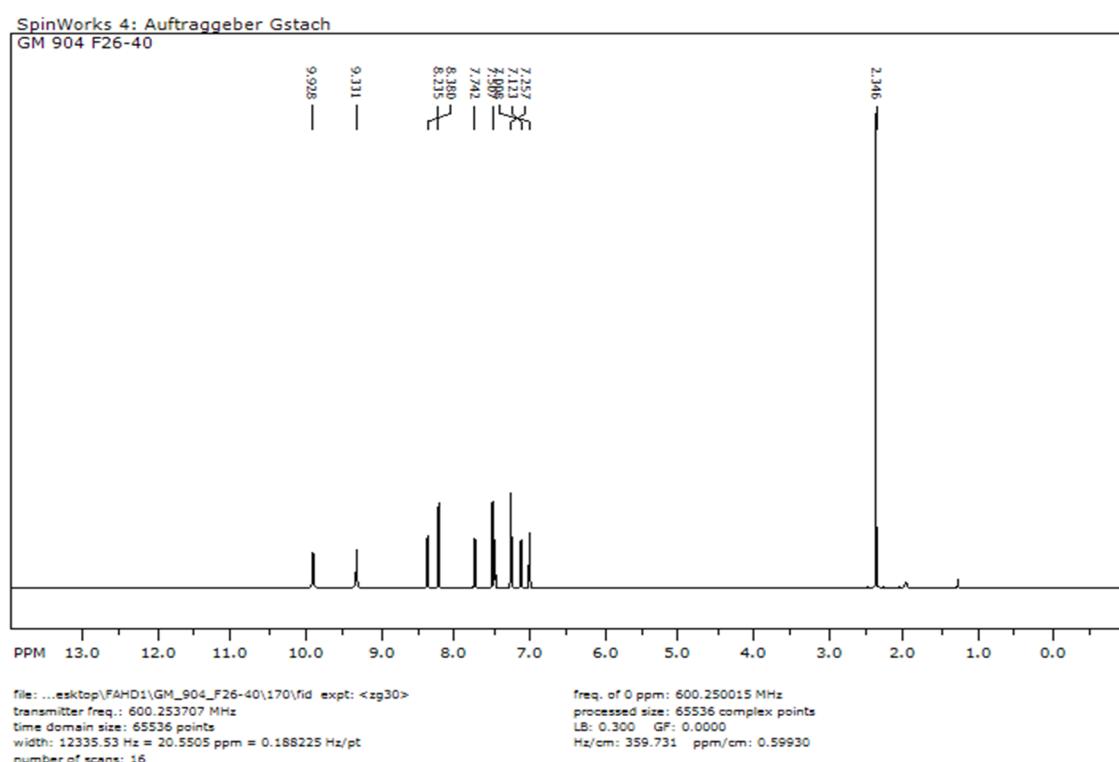


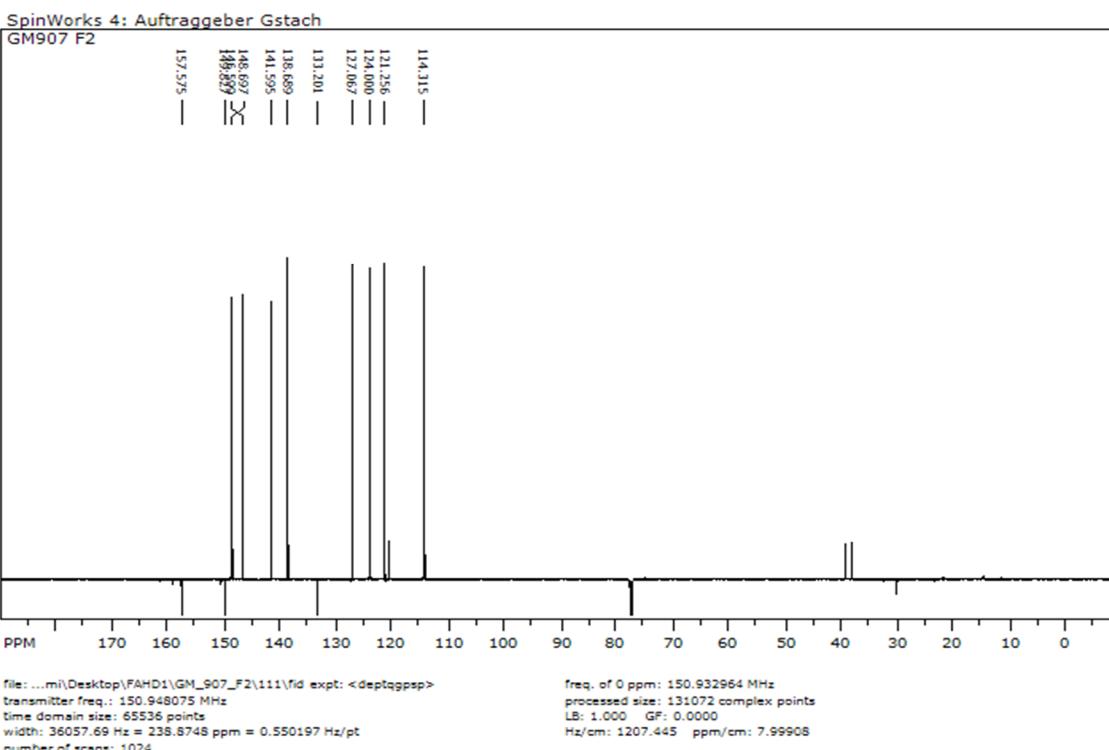
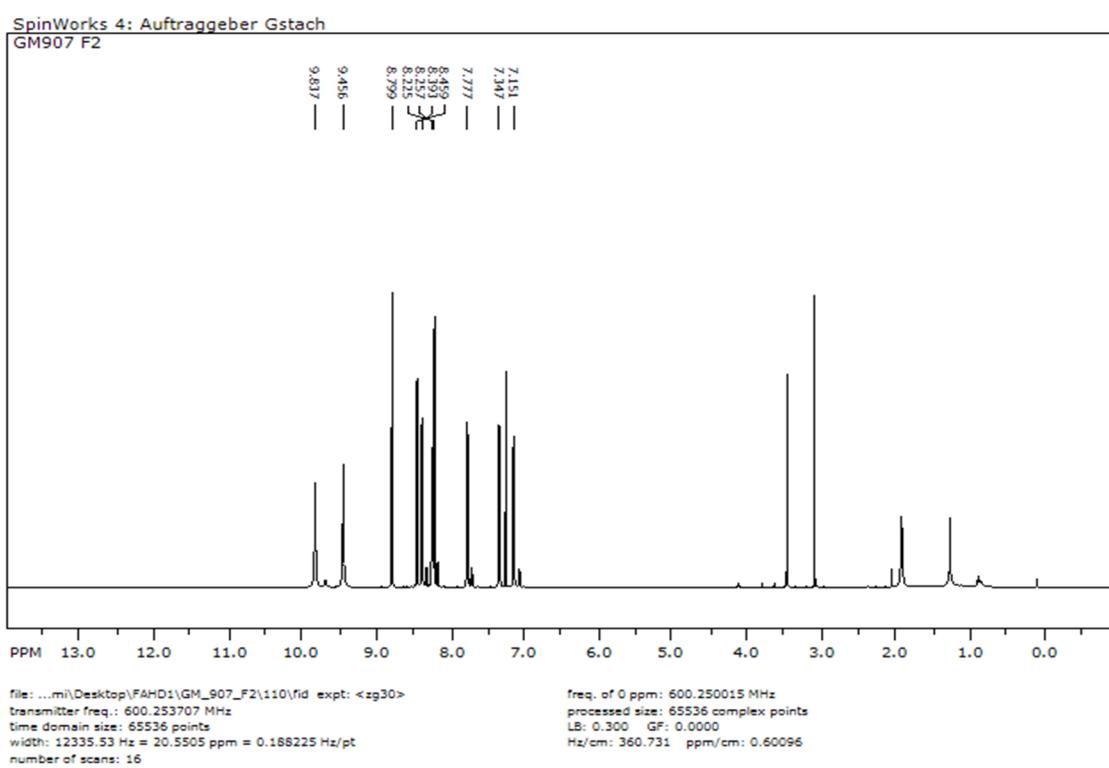
N¹,N²-bis(5-methylpyridin-2-yl)oxalamide (29)

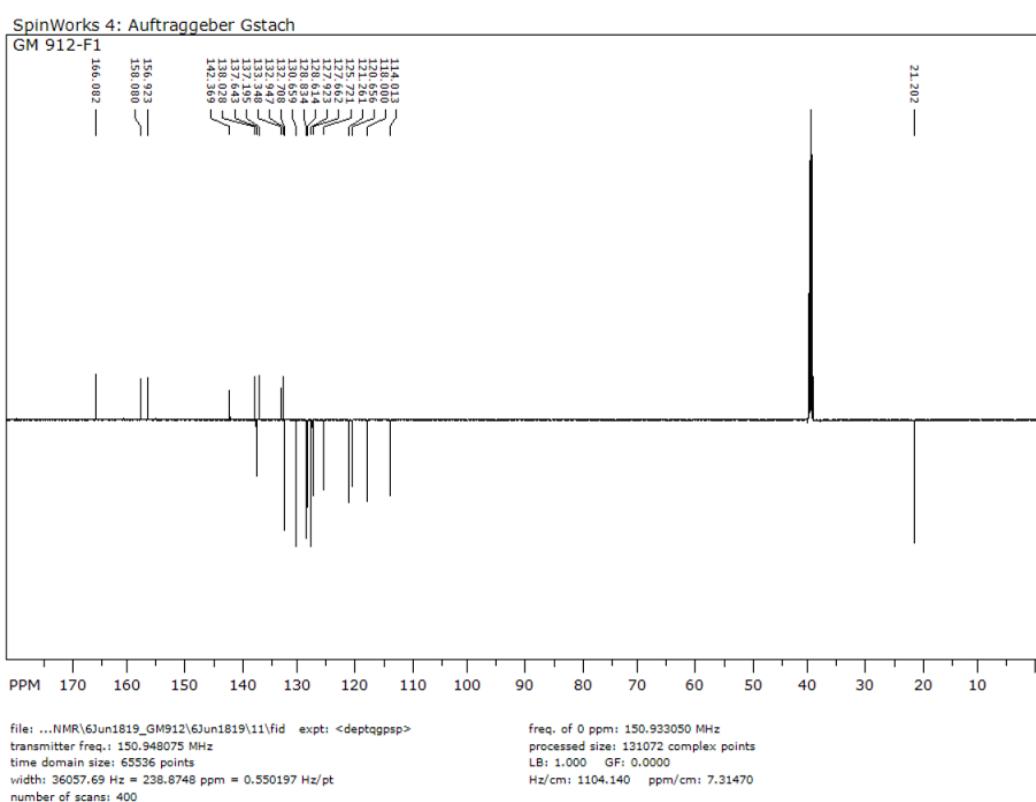
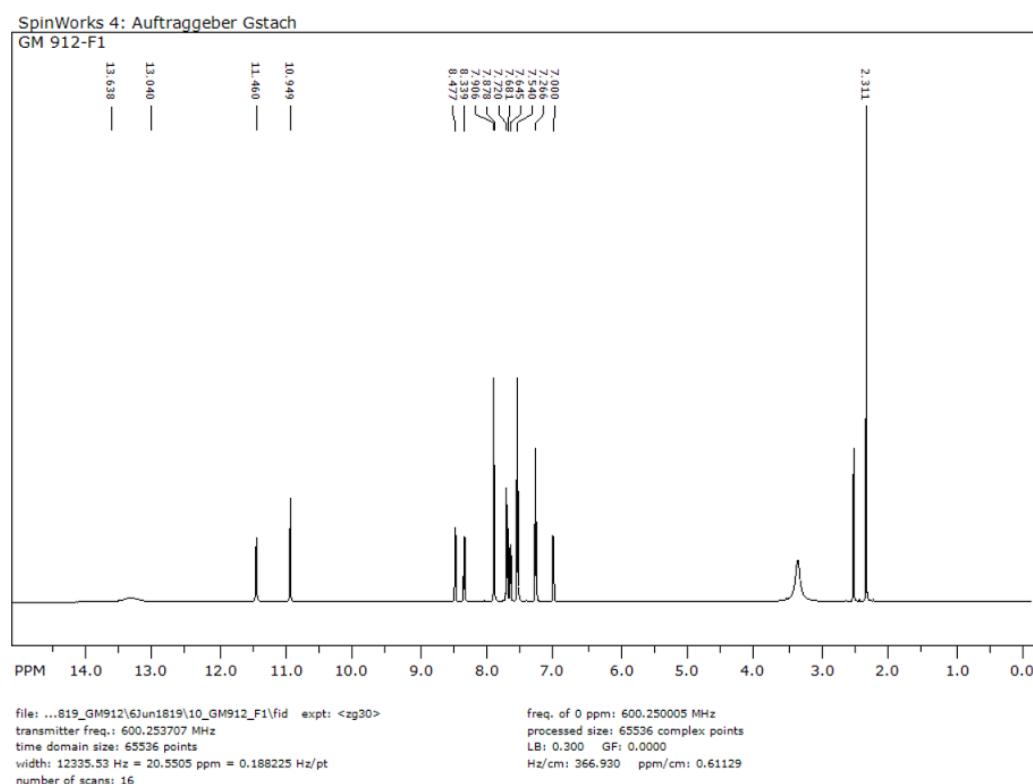
N¹,N²-di(pyridin-2-yl)oxalamide (30)

N,N'-Di-pyridin-3-yl-oxalamide (31)

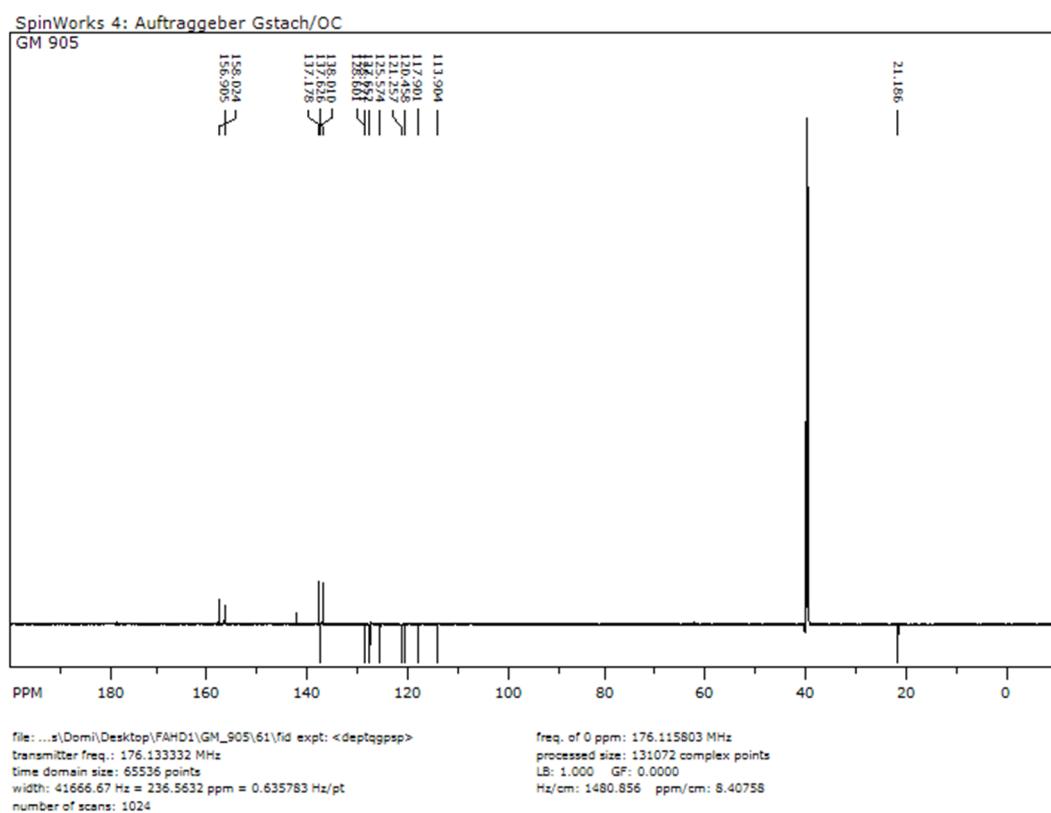
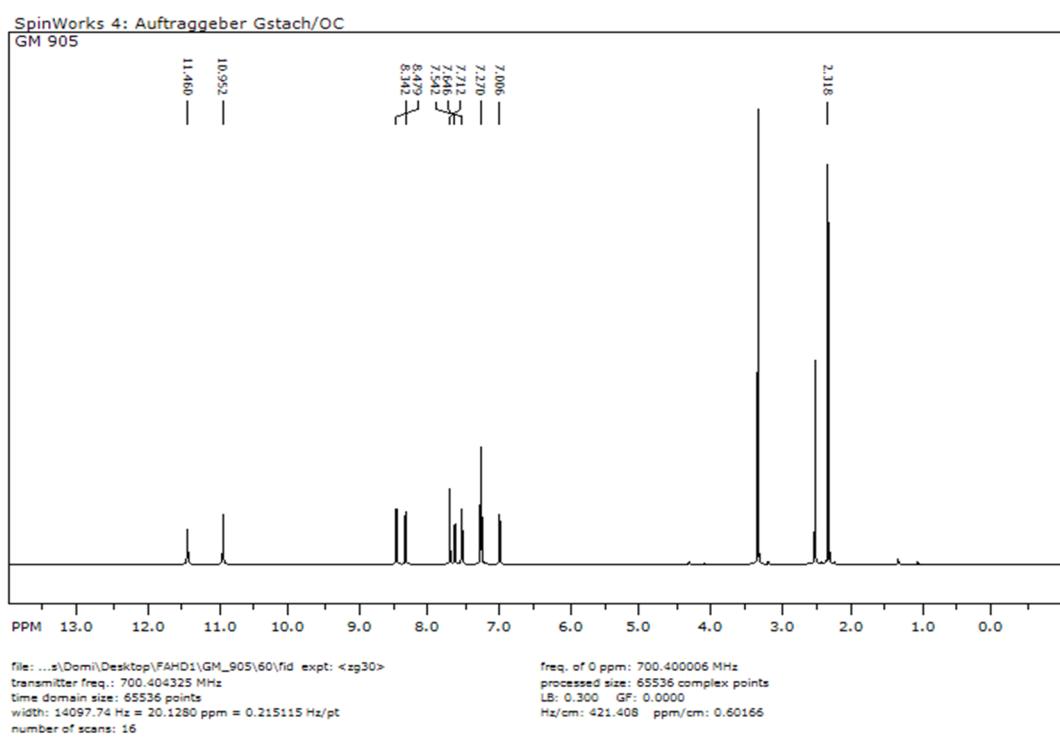
N-Pyridin-2-yl-N'-p-tolyl-oxalamide (32)

N-Pyridin-2-yl-N'-*m*-tolyl-oxalamide (33)

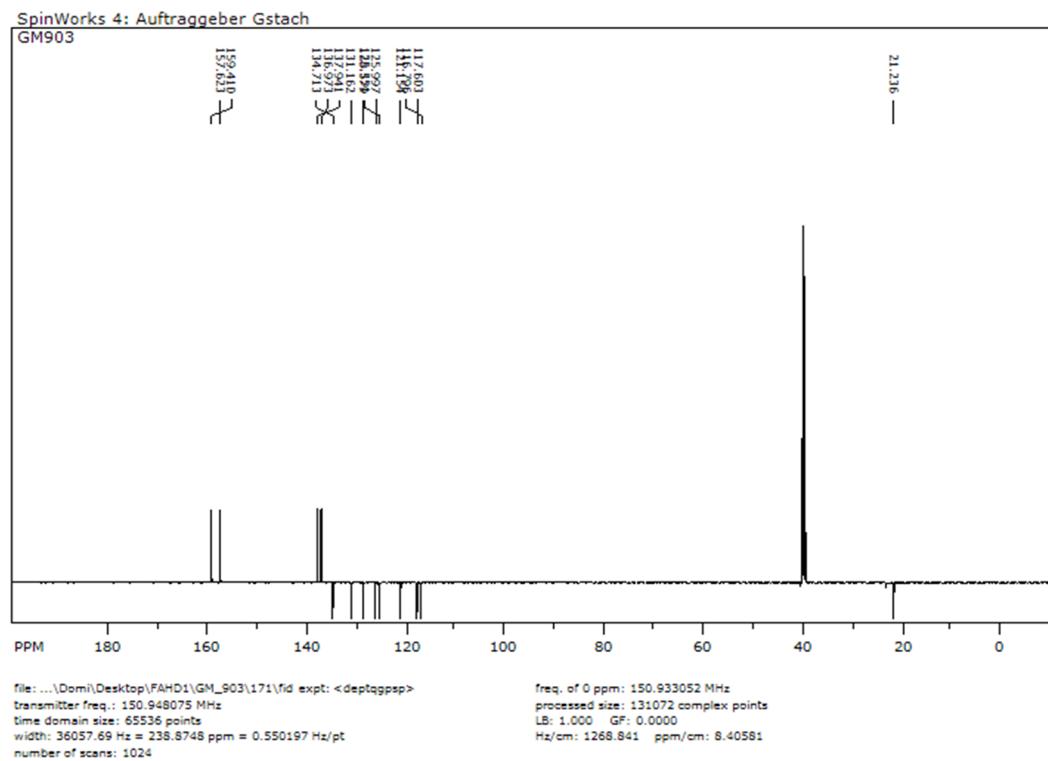
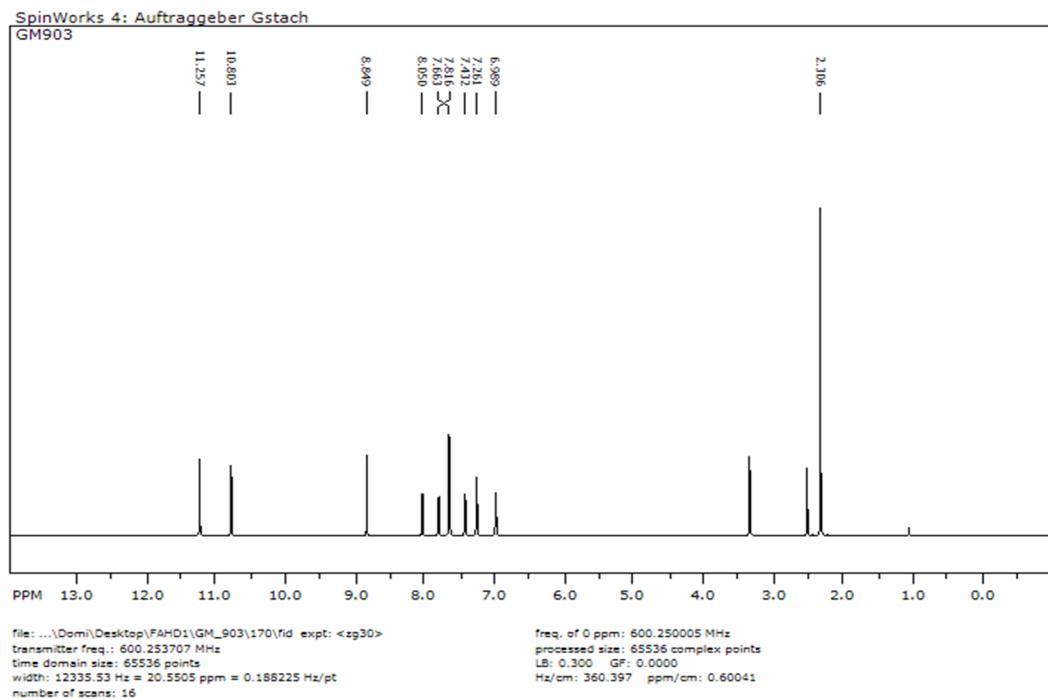
N-Pyridin-3-yl-N'-pyridin-2-yl-oxalamide (34)

N-(1-Oxy-pyridin-2-yl)-N'-*p*-tolyl-oxalamide (35)

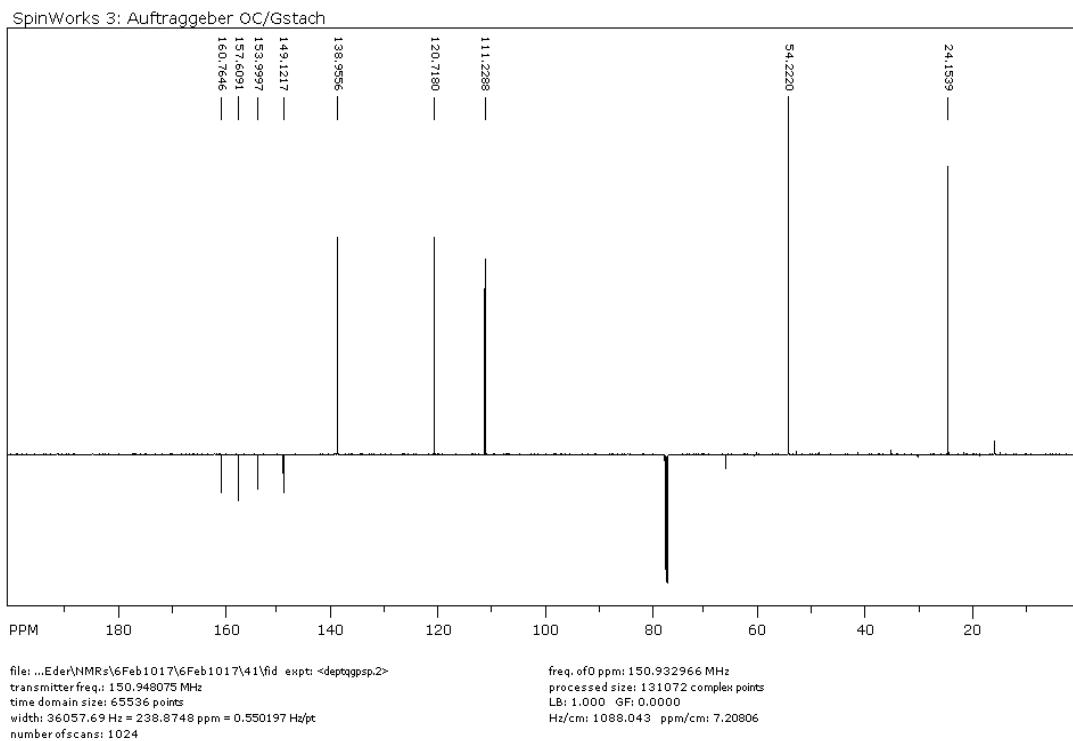
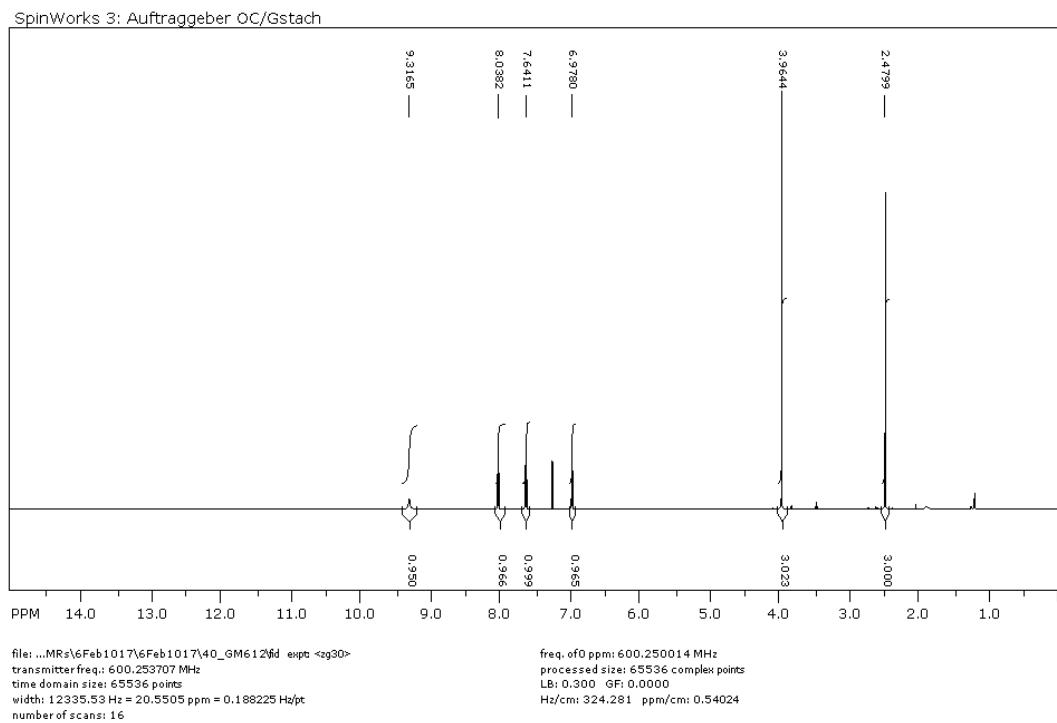
***N*-(1-Oxy-pyridin-2-yl)-*N'*-*m*-tolyl-oxalamide (36)**



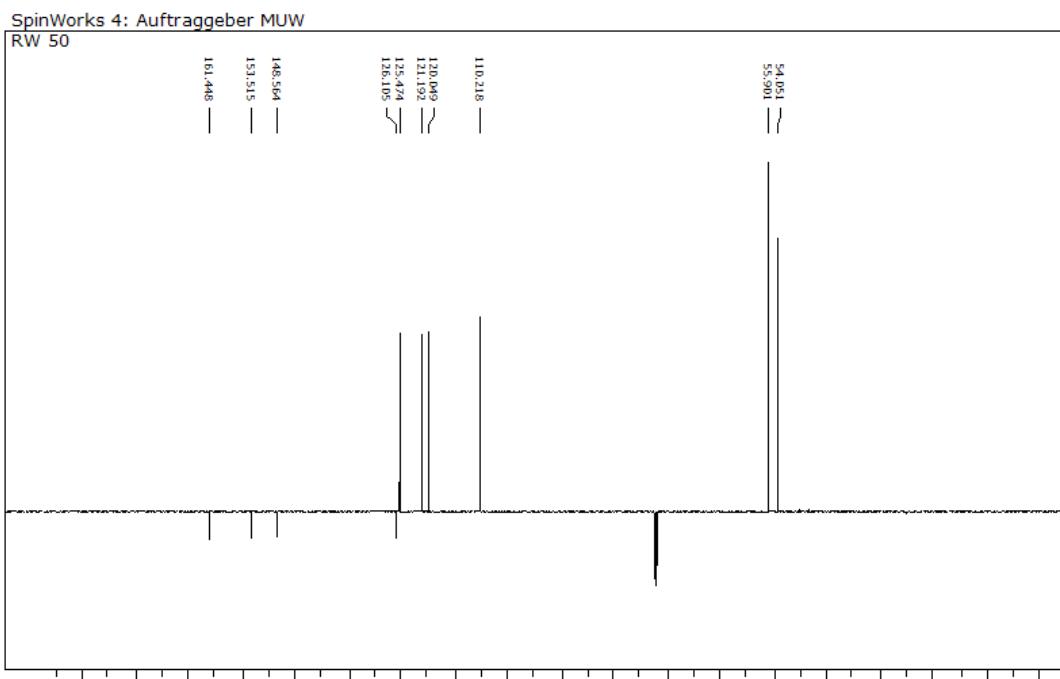
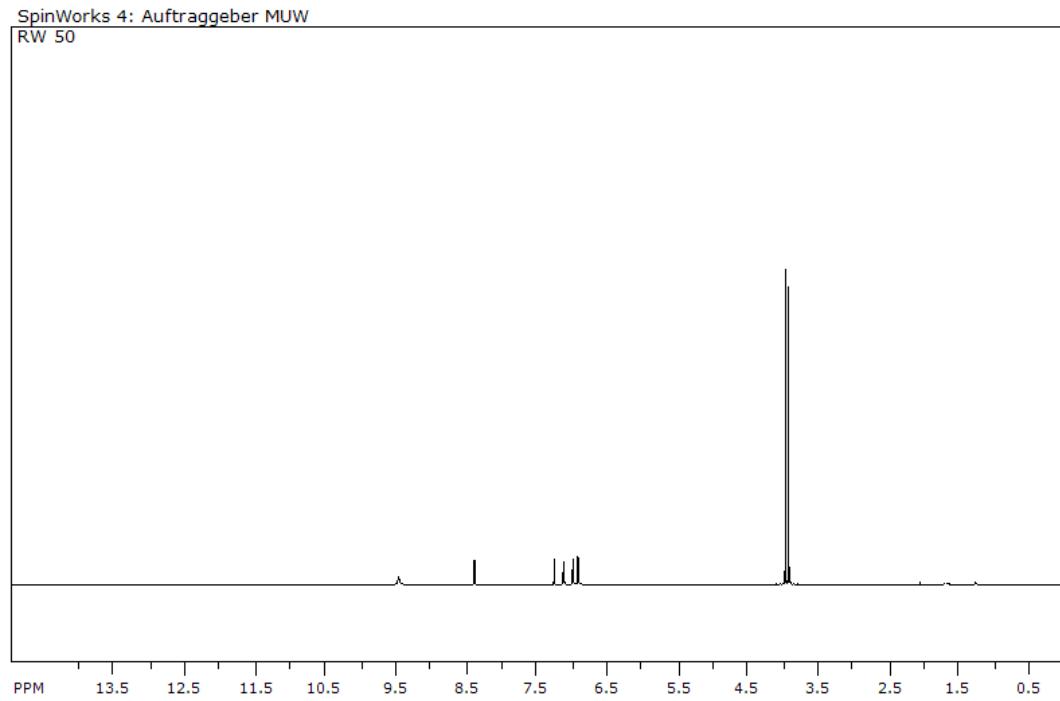
N-(1-Oxy-pyridin-3-yl)-N'-*m*-tolyl-oxalamide (37)



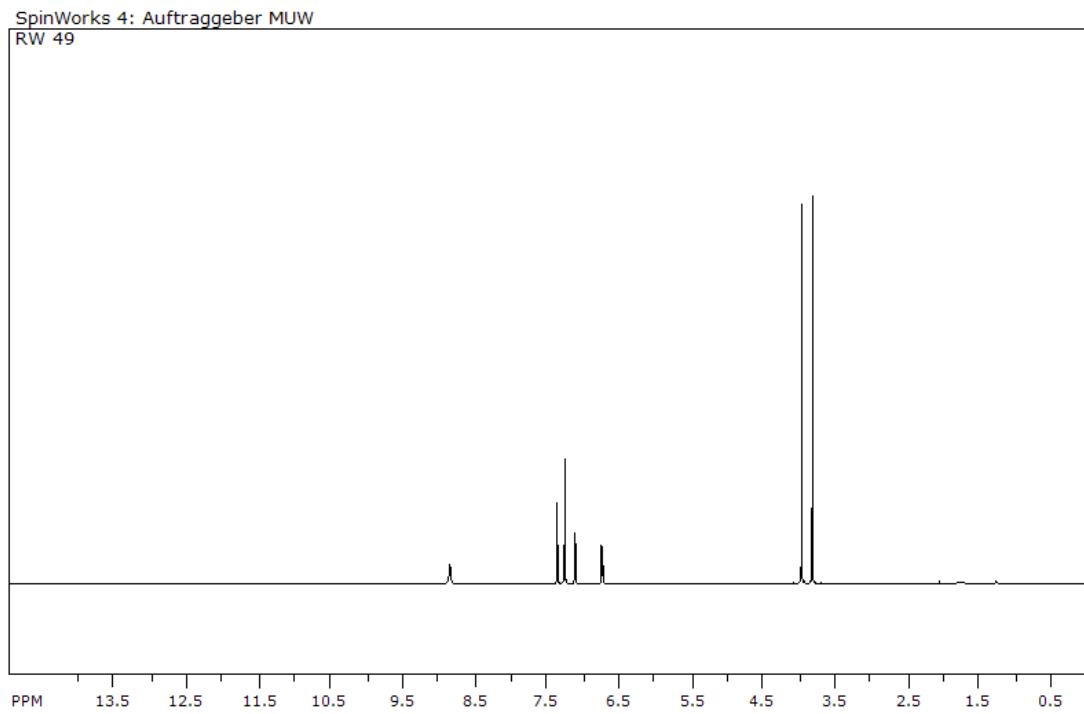
Methyl 2-((6-methylpyridin-2-yl)amino)-2-oxoacetate (S1)



Methyl 2-((2-methoxyphenyl)amino)-2-oxoacetate (S2)

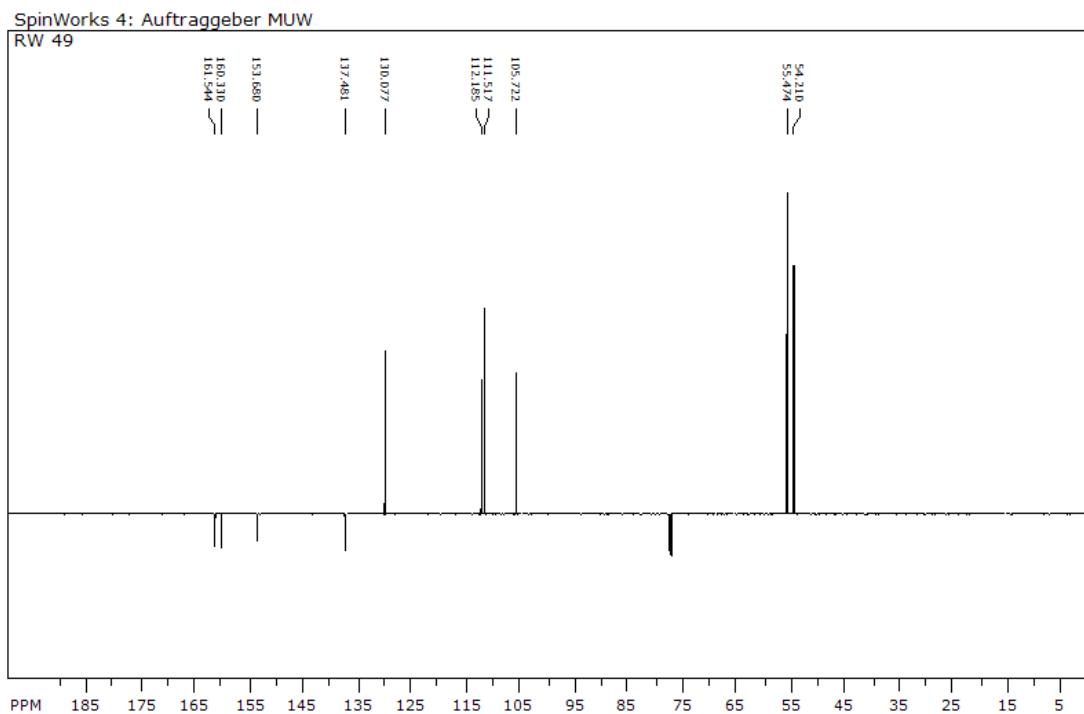


Methyl 2-((3-methoxyphenyl)amino)-2-oxoacetate (S3)



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time domain size: 65536 points
width: 12335.53 Hz = 20.5505 ppm = 0.188225 Hz/pt
number of scans: 16

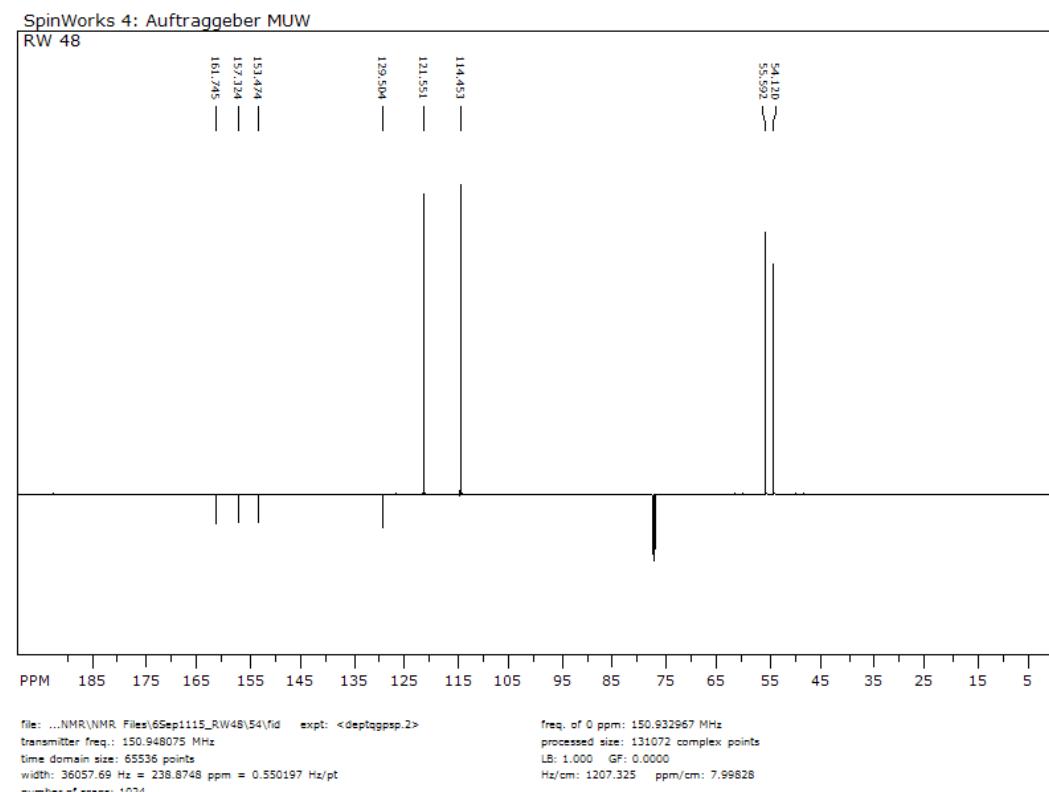
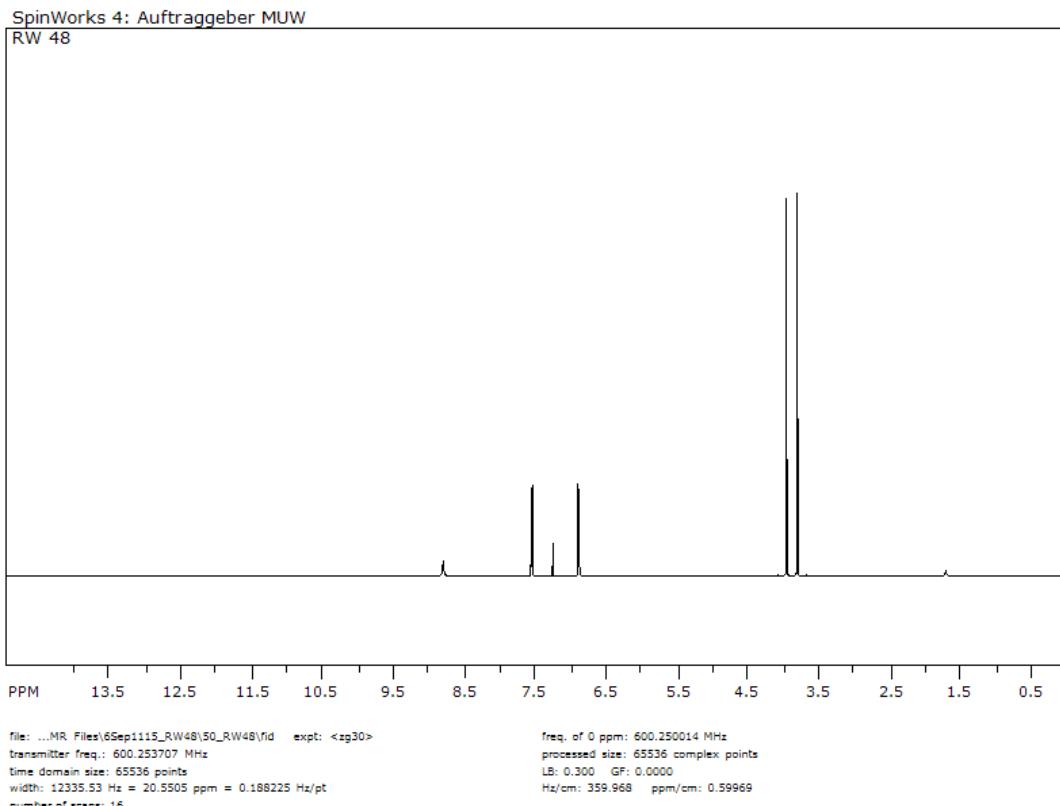
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LB: 0.300 GF: 0.0000
Hz/cm: 359.968 ppm/cm: 0.59969



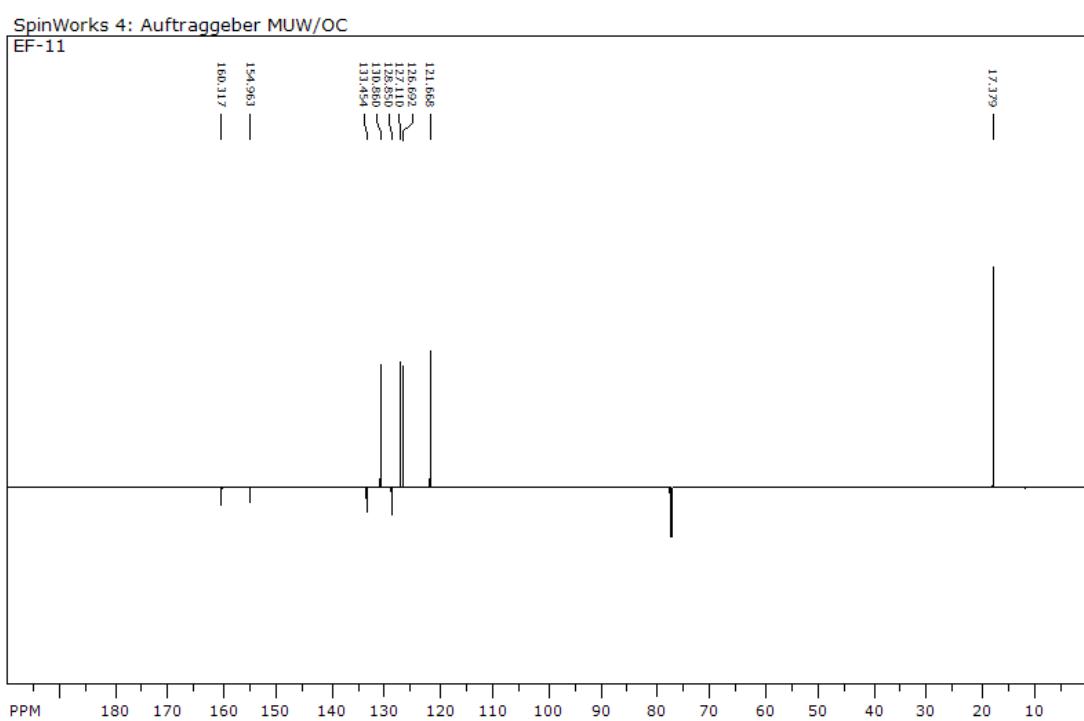
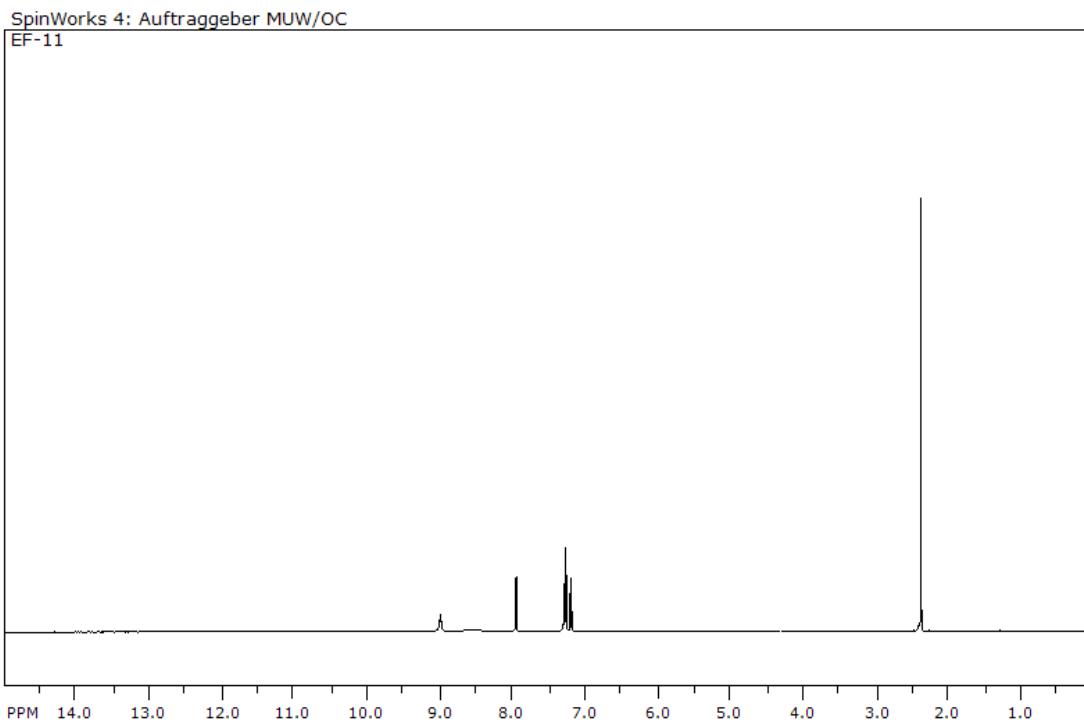
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time domain size: 65536 points
width: 36057.69 Hz = 238.8748 ppm = 0.550197 Hz/pt
number of scans: 1024

freq. of 0 ppm: 150.932966 MHz
processed size: 131072 complex points
LB: 1.000 GF: 0.0000
Hz/cm: 1207.325 ppm/cm: 7.99828

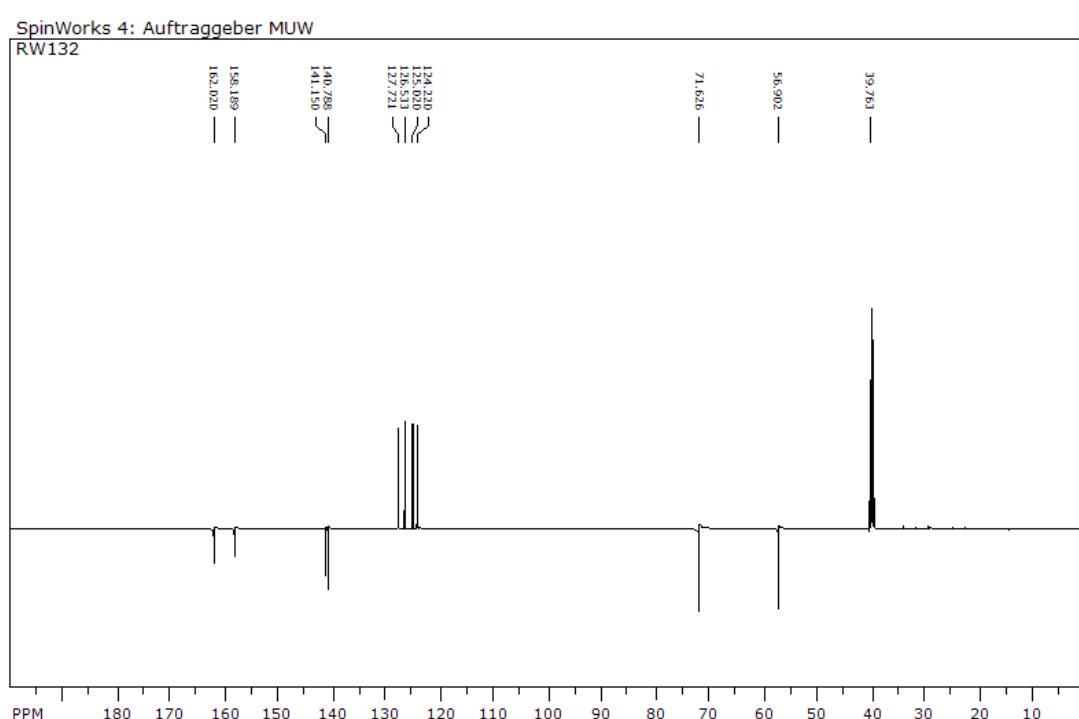
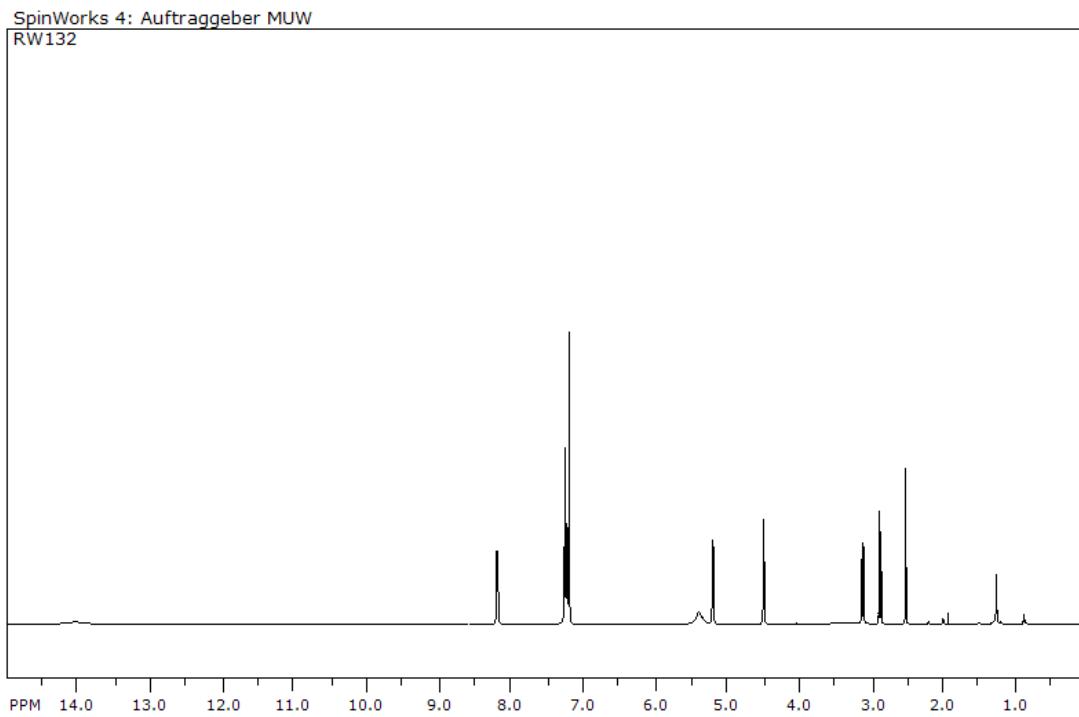
Methyl 2-((4-methoxyphenyl)amino)-2-oxoacetate (S4)



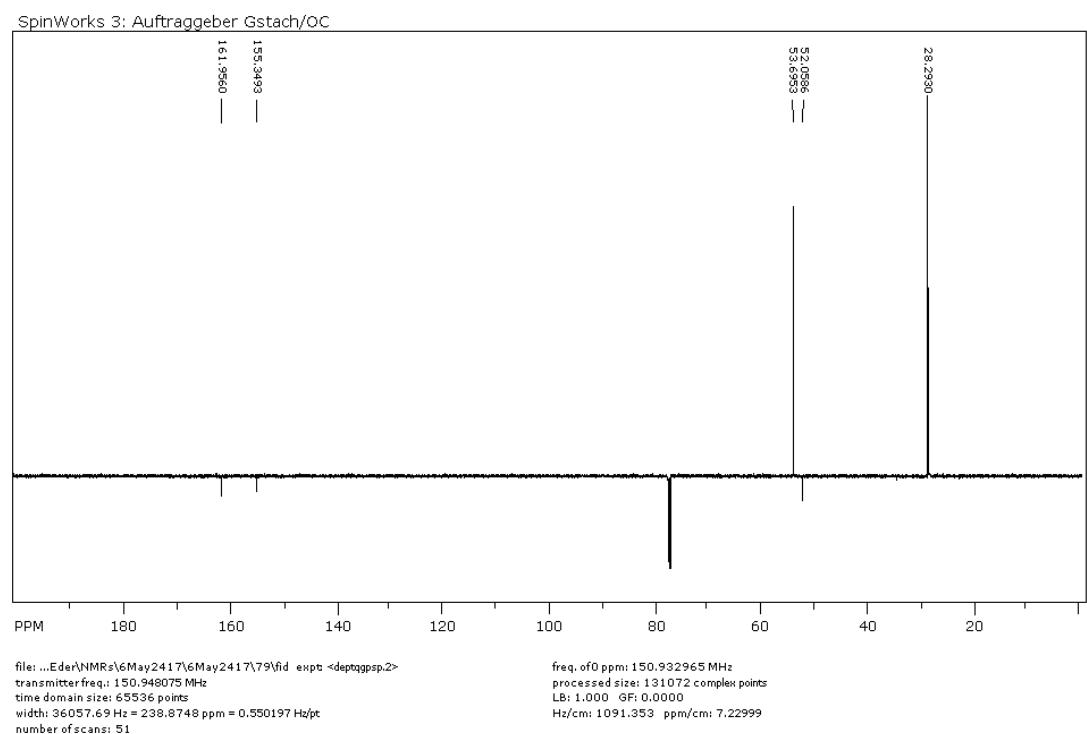
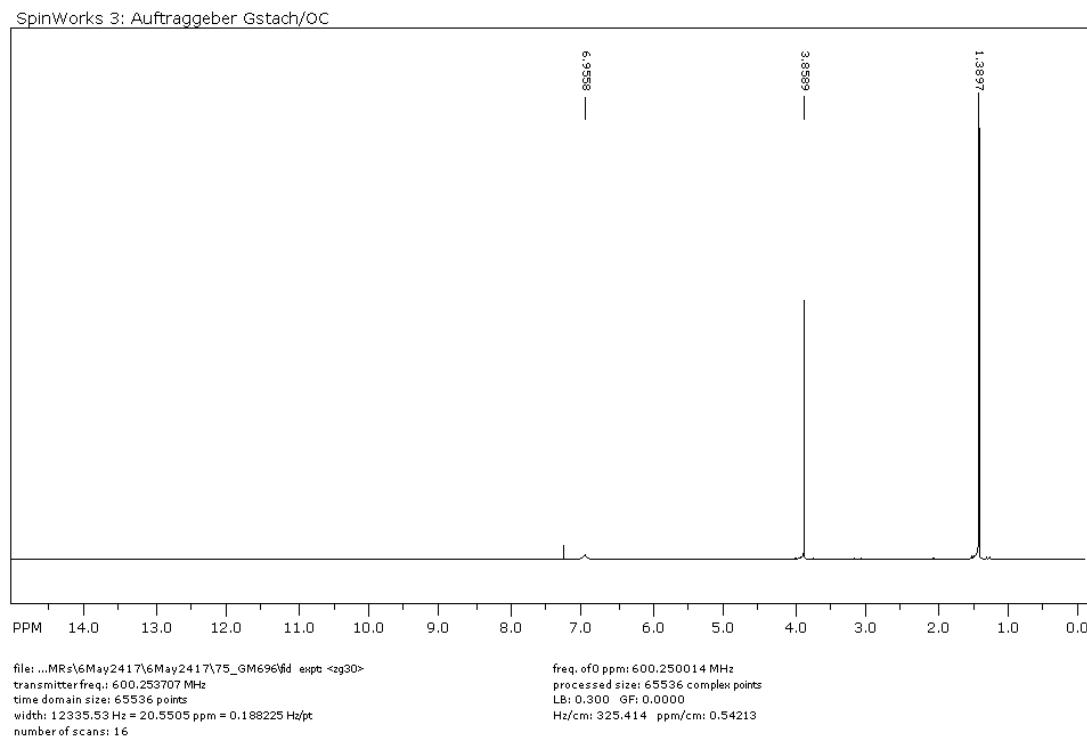
2-oxo-2-(*o*-tolylamino)acetic acid (S5)



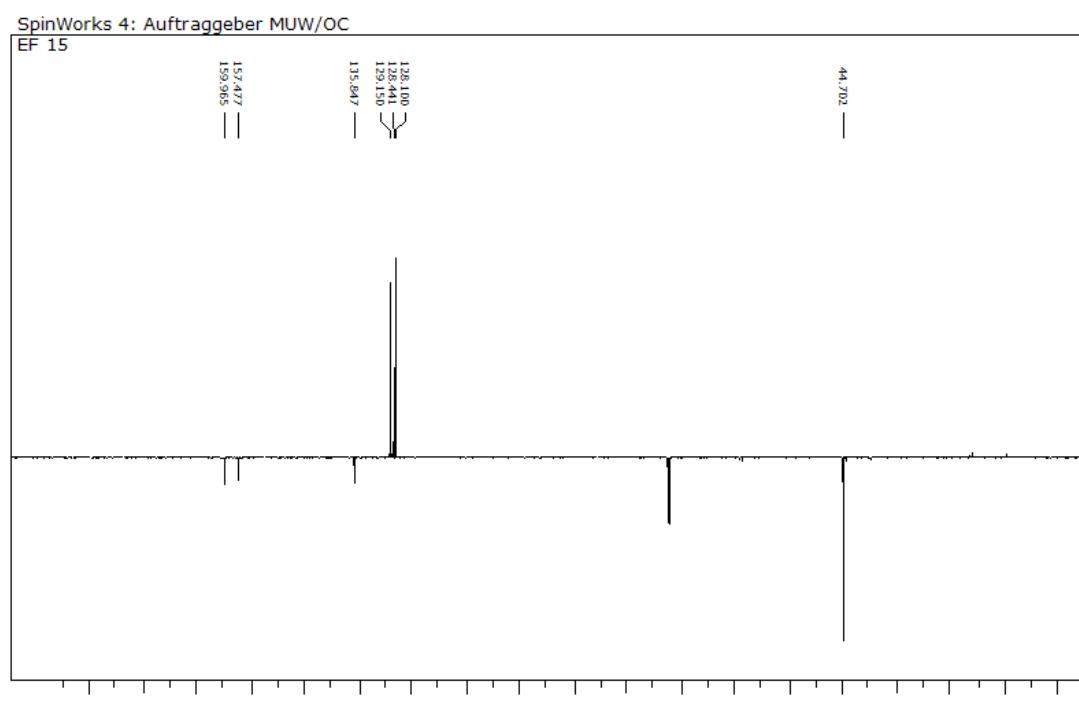
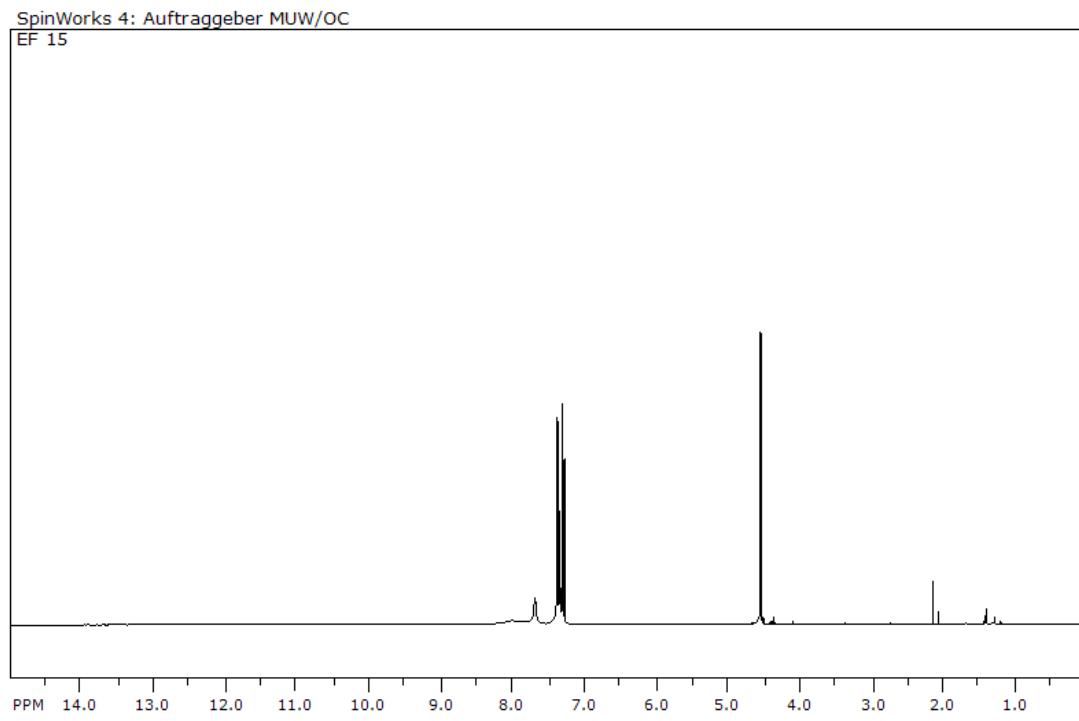
2-(((1R,2S)-2-hydroxy-2,3-dihydro-1H-inden-1-yl)amino)-2-oxoacetic acid (S6)



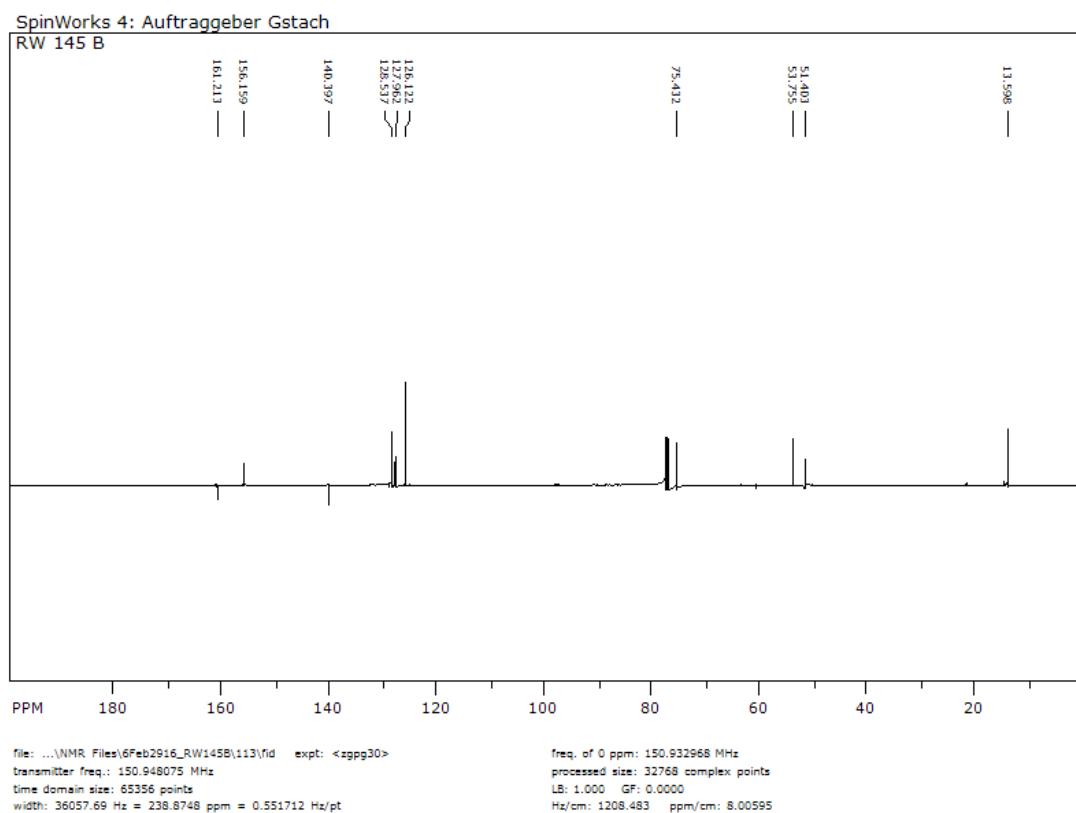
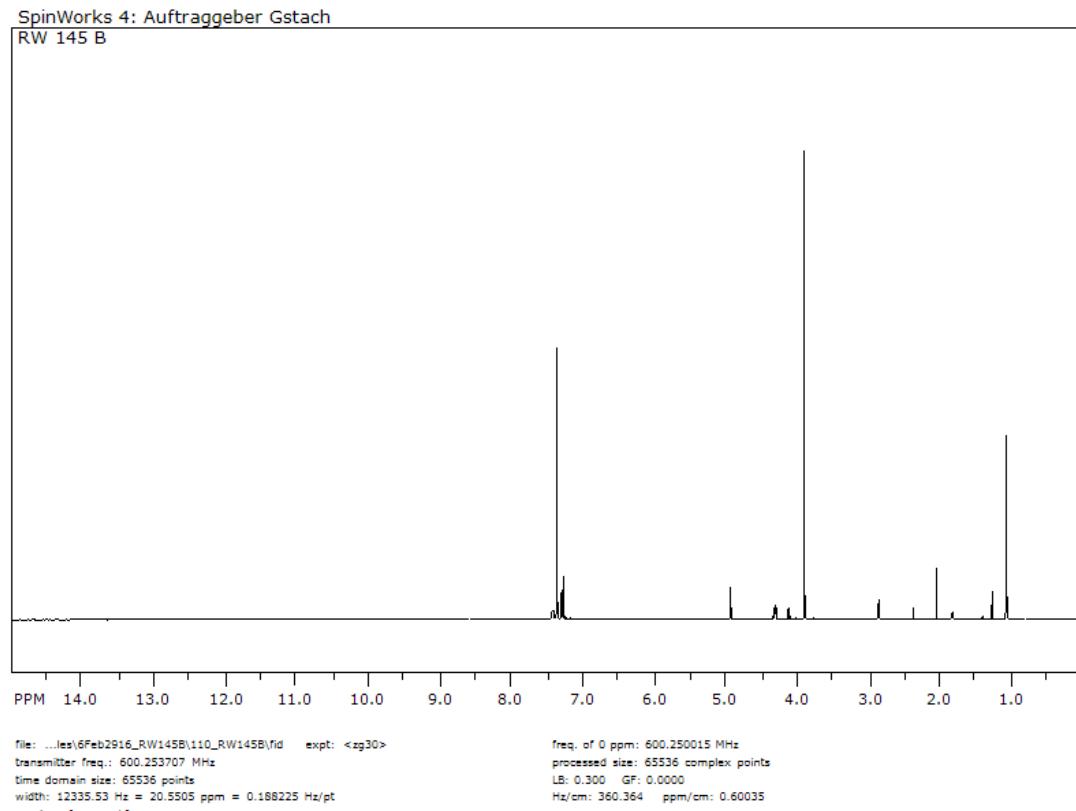
Methyl 2-(*tert*-butylamino)-2-oxoacetate (S7)



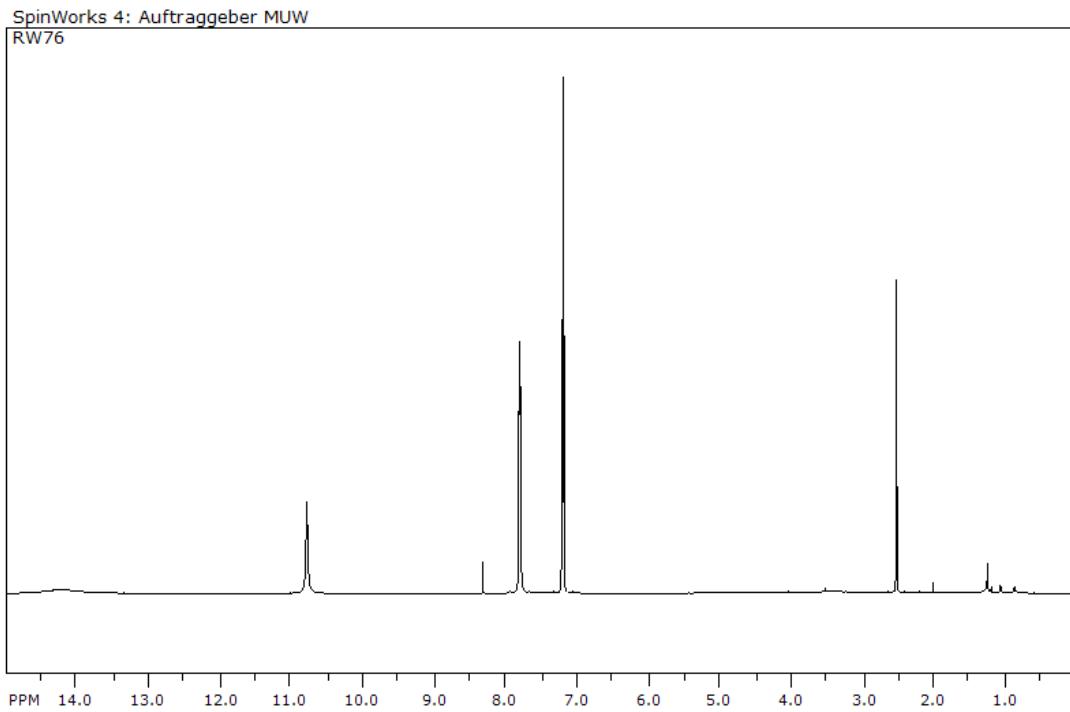
2-(benzylamino)-2-oxoacetic acid (S8)



Methyl 2-(((1*S*,2*R*)-1-hydroxy-1-phenylpropan-2-yl)amino)-2-oxoacetate (S9)

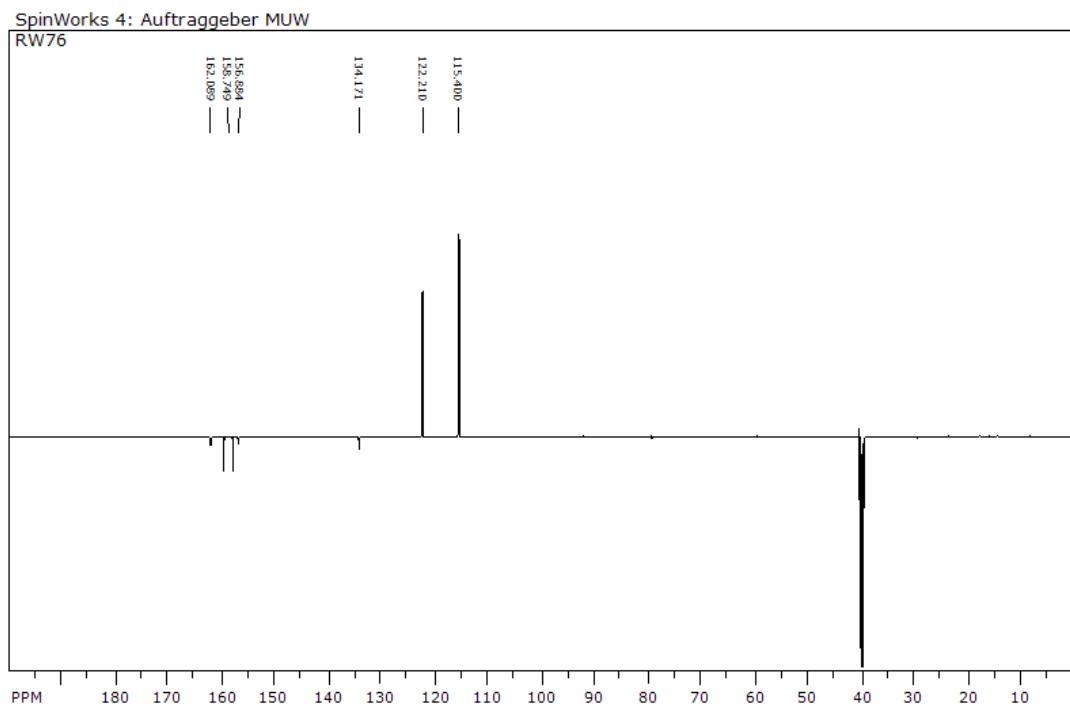


2-((4-fluorophenyl)amino)-2-oxoacetic acid (S10)



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 width: 12335.53 Hz = 20.5505 ppm = 0.188225 Hz/pt
 number of scans: 16

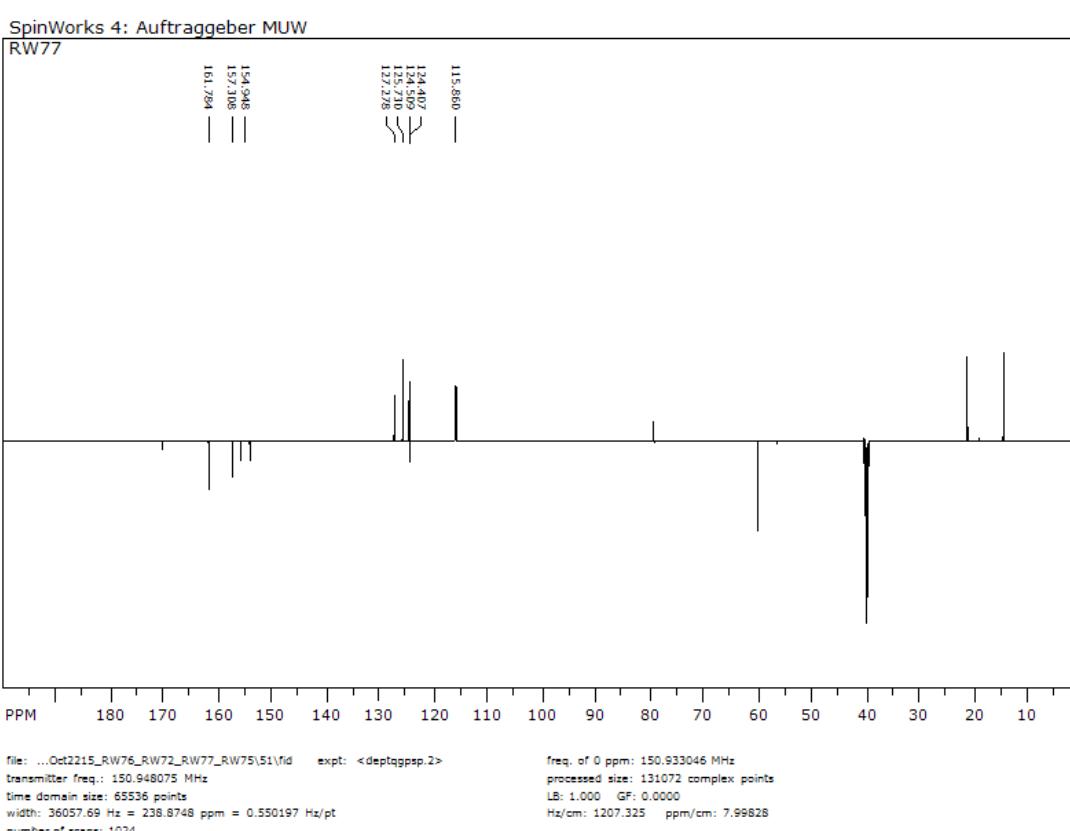
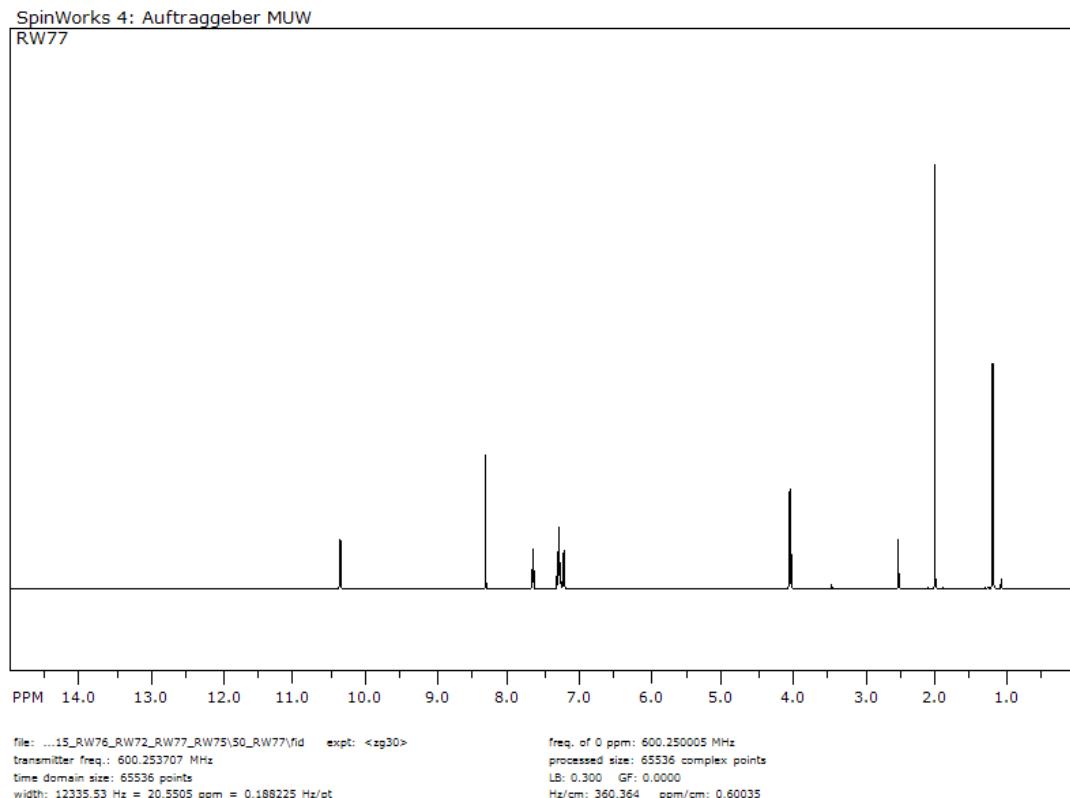
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 processed size: 65536 complex points
 LB: 0.300 GF: 0.0000
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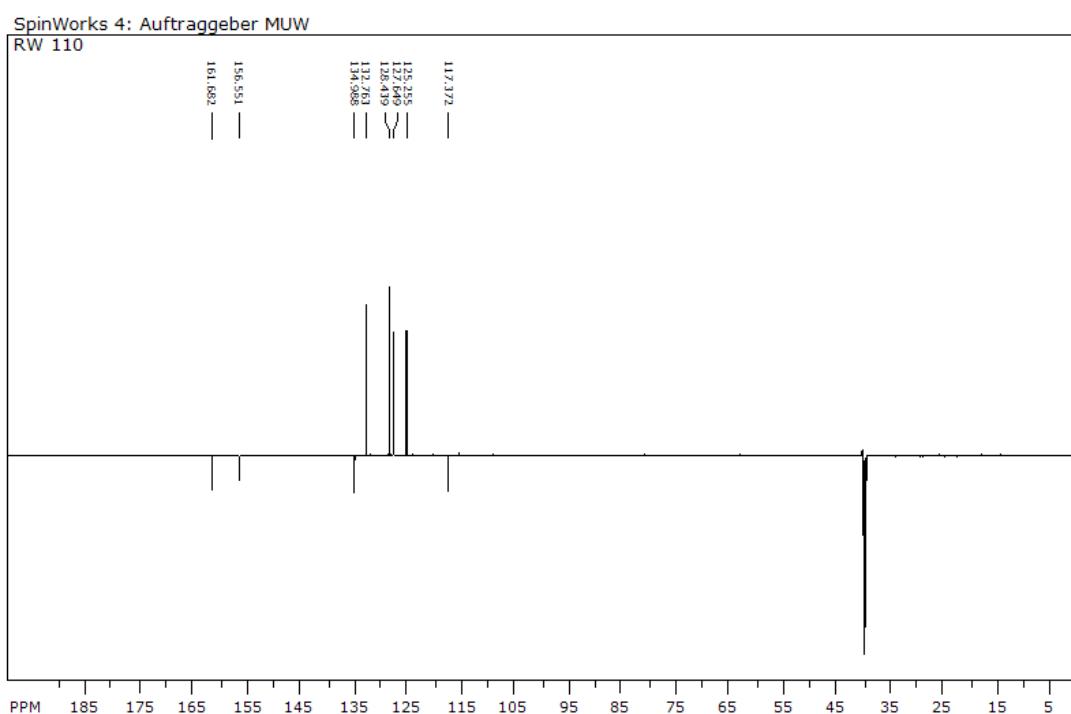
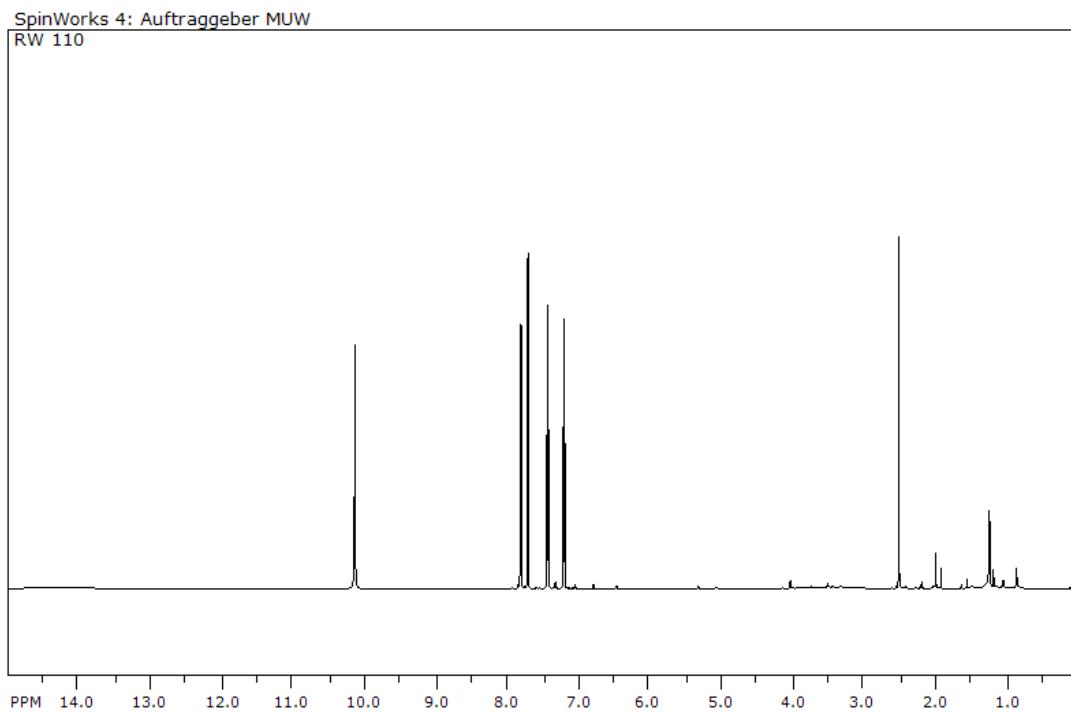
file: ...Oct2215_RW76_RW72_RW77_RW75\41\fid expt: < deplqgppsp,2 >
 transmitter freq.: 150.948075 MHz
 time domain size: 65536 points
 width: 36057.69 Hz = 238.8748 ppm = 0.550197 Hz/pt
 number of scans: 1024

freq. of 0 ppm: 150.933046 MHz
 processed size: 131072 complex points
 LB: 1.000 GF: 0.0000
 Hz/cm: 1207.325 ppm/cm: 7.99828

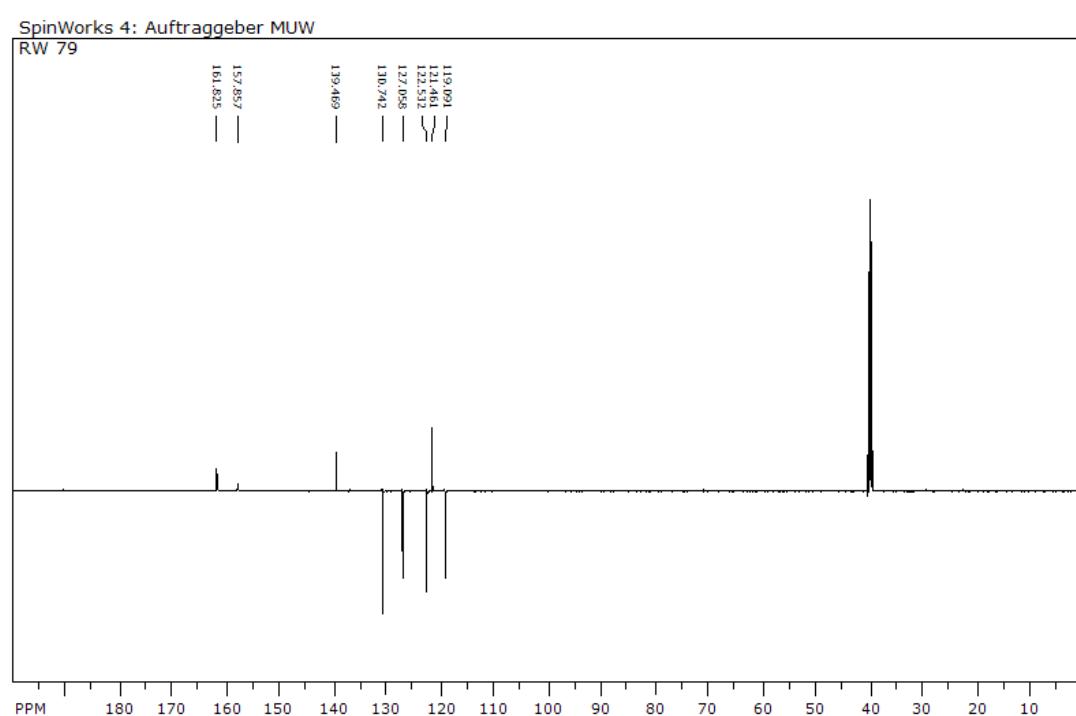
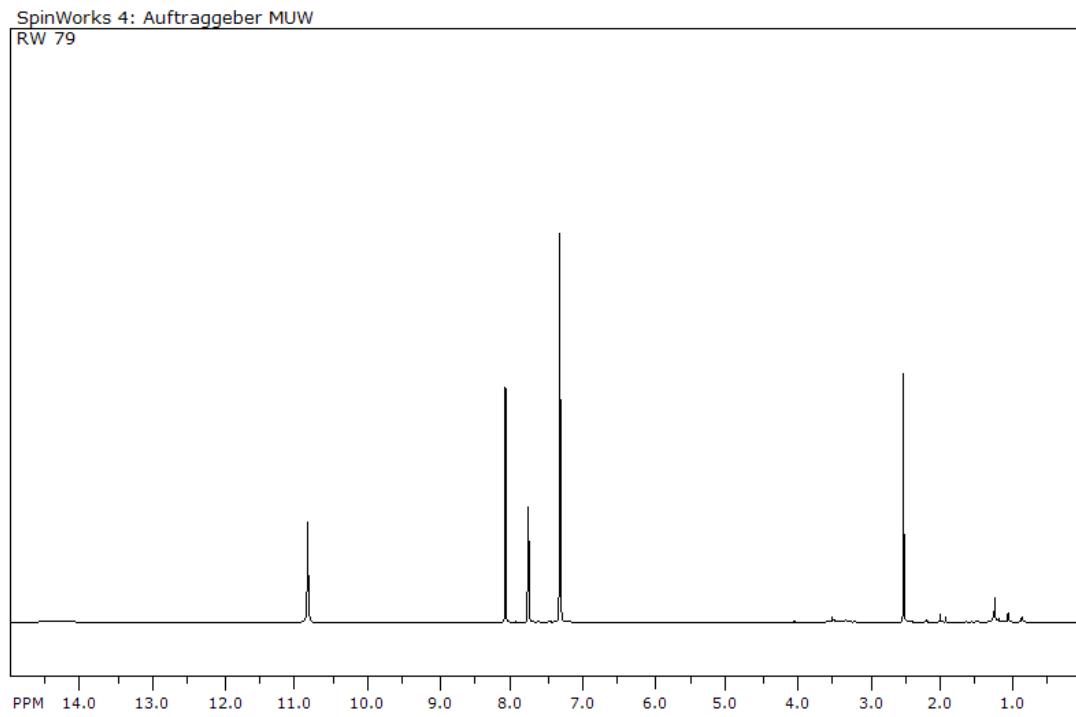
2-((2-fluorophenyl)amino)-2-oxoacetic acid (S11)



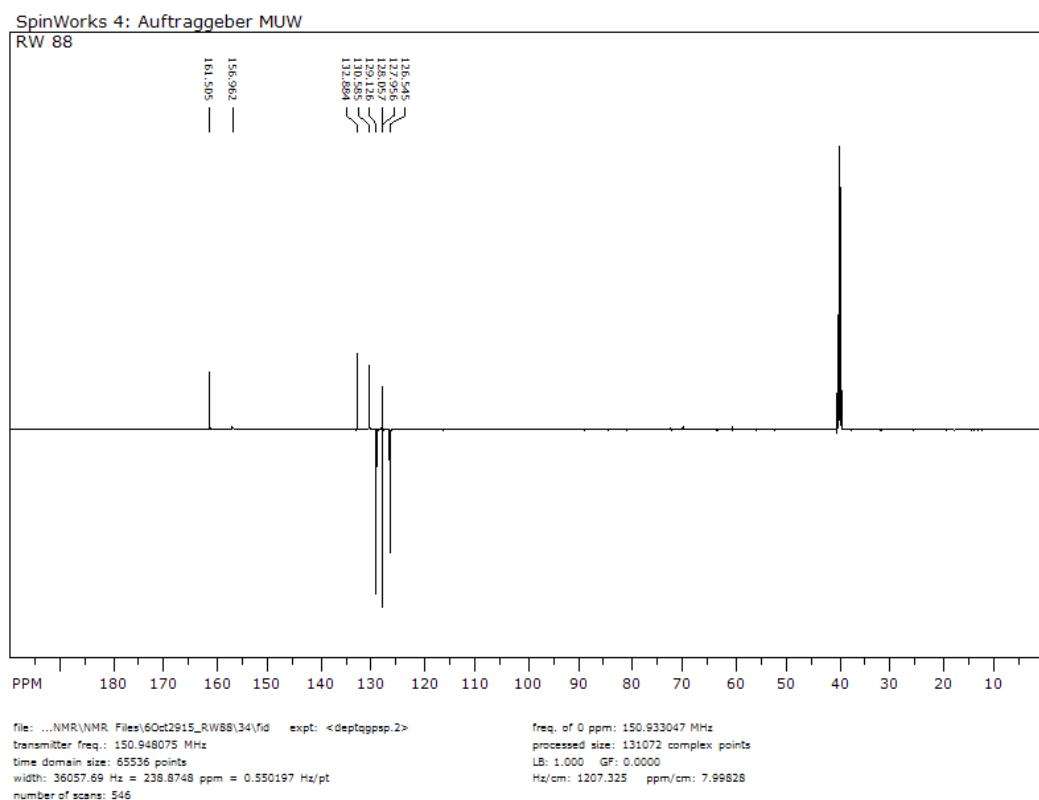
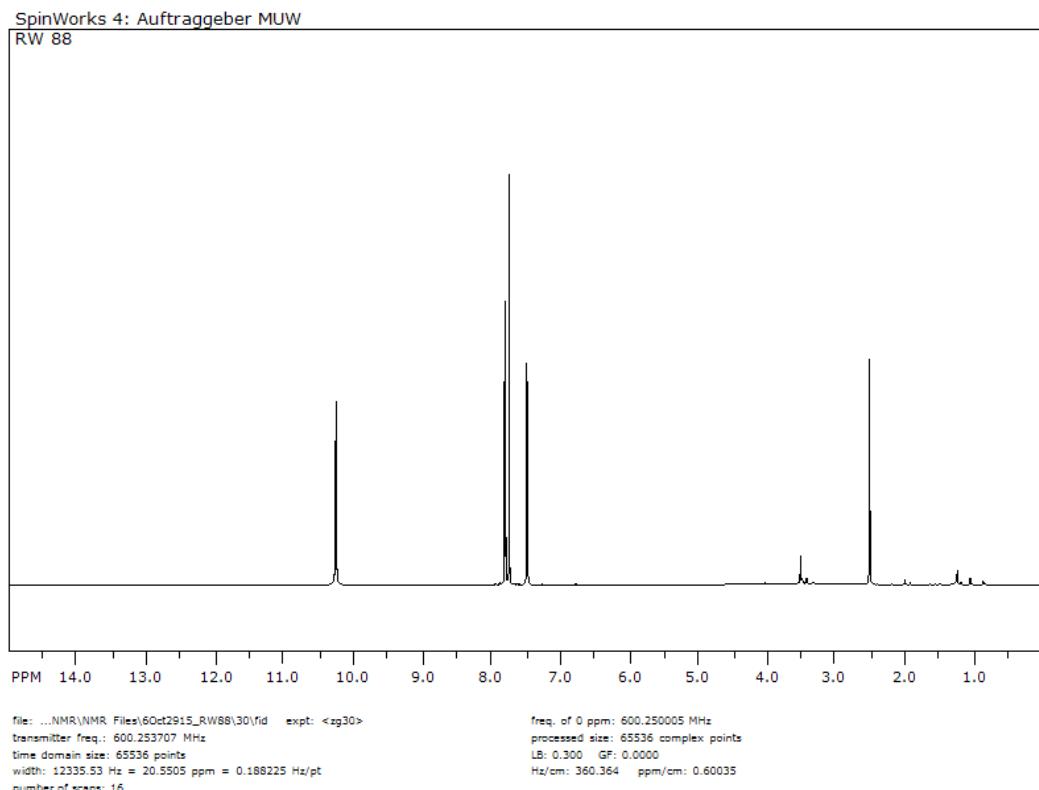
2-((2-bromophenyl)amino)-2-oxoacetic acid (S12)



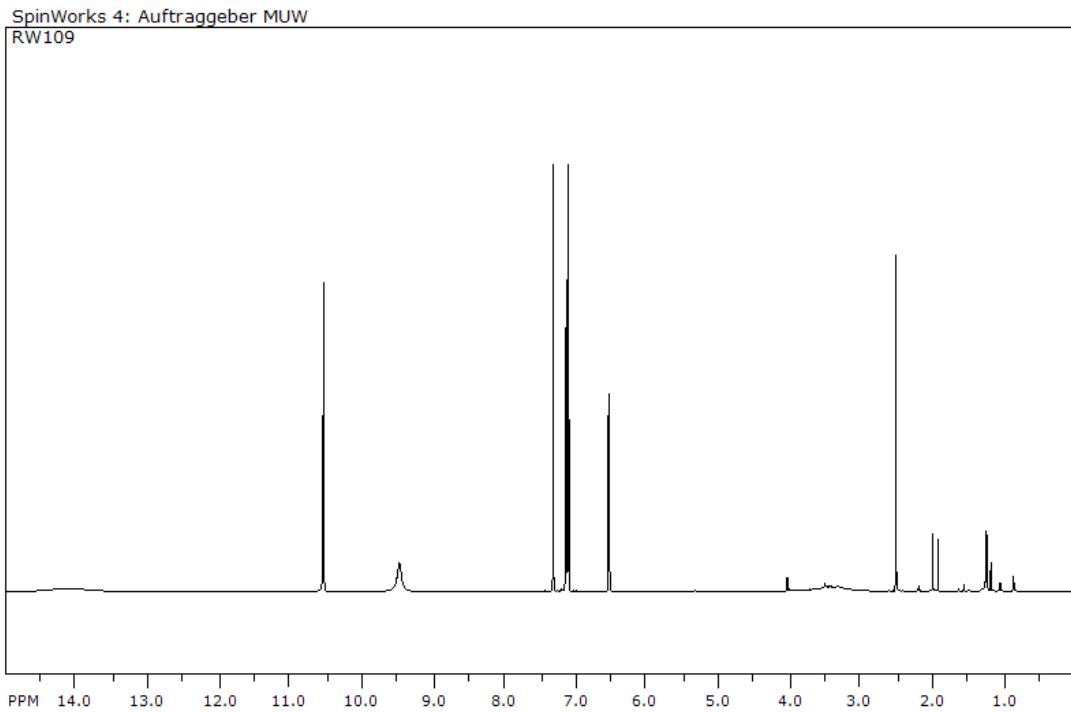
2-((3-bromophenyl)amino)-2-oxoacetic acid (S13)



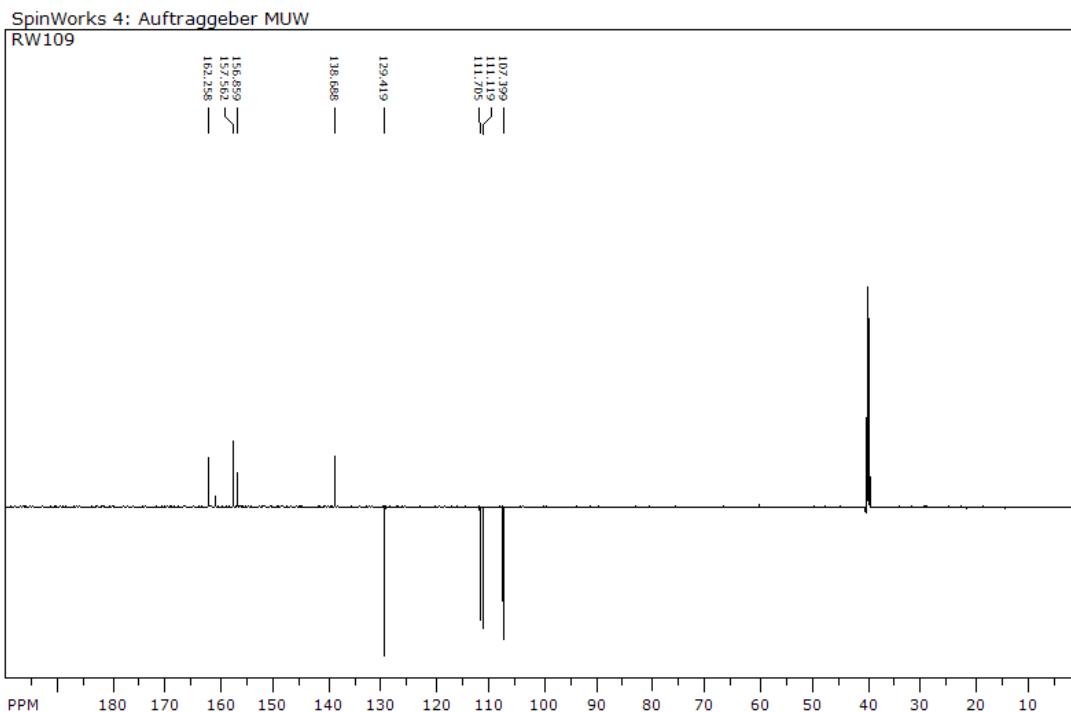
2-((2,4-dichlorophenyl)amino)-2-oxoacetic acid (S14)



2-((3-hydroxyphenyl)amino)-2-oxoacetic acid (S15)

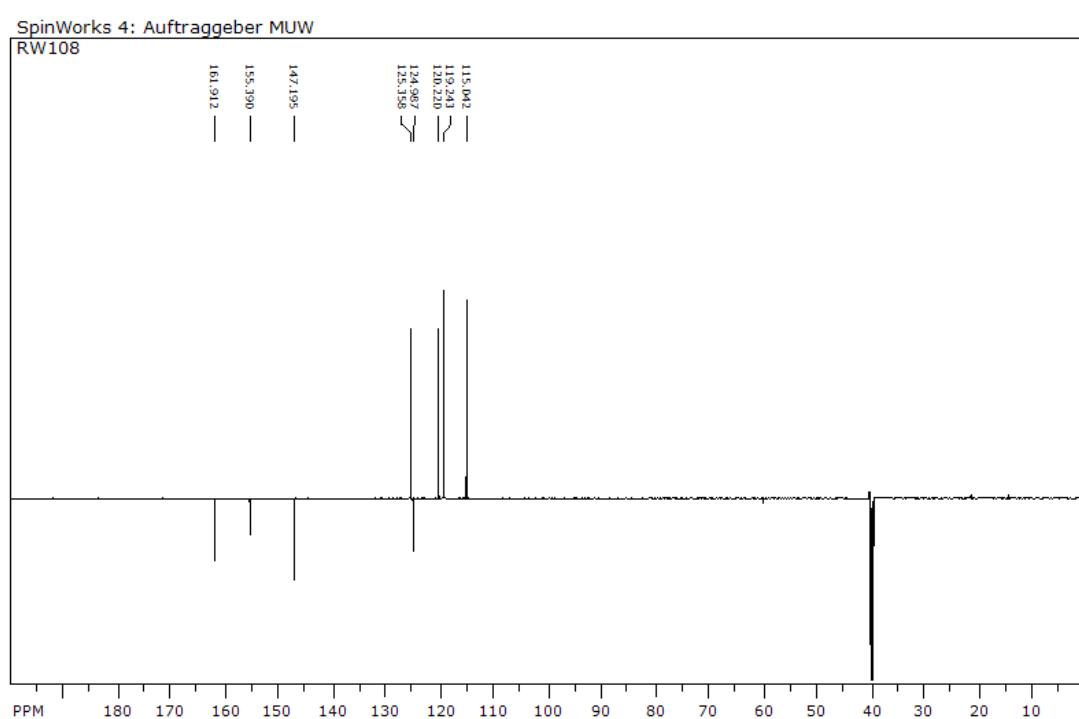
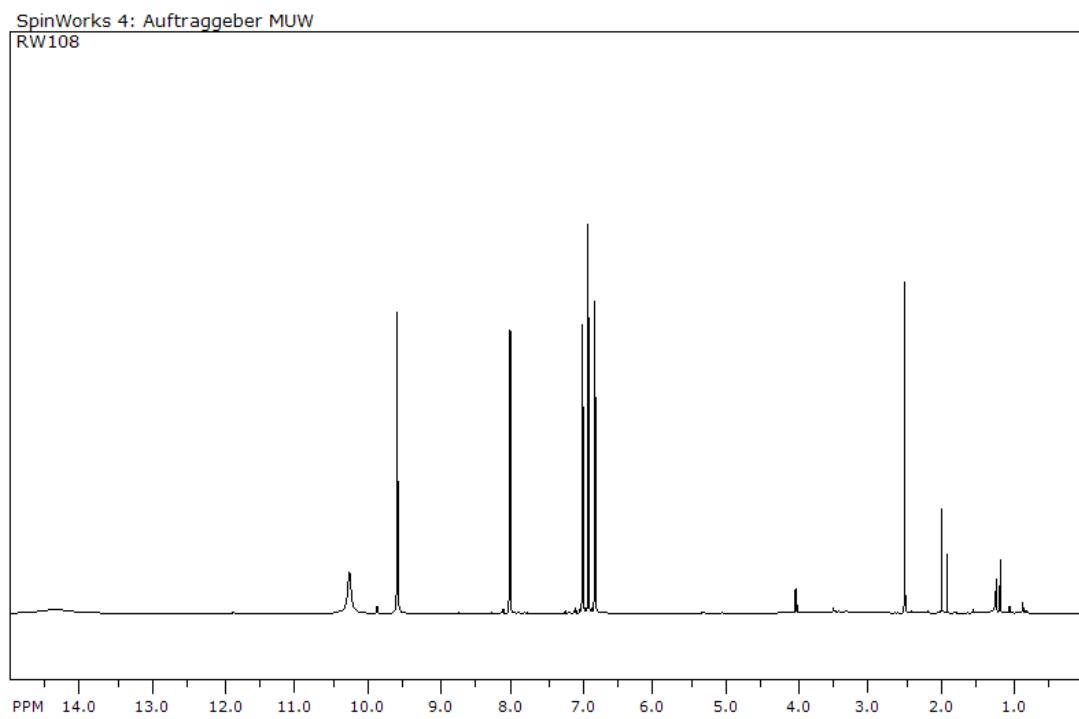


file: ...\\7Nov3015_RW107_RW108_RW109\\30\\fid expt: < zg30>
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 width: 14097.74 Hz = 20.1280 ppm = 0.215115 Hz/pt LB: 0.300 GF: 0.0000
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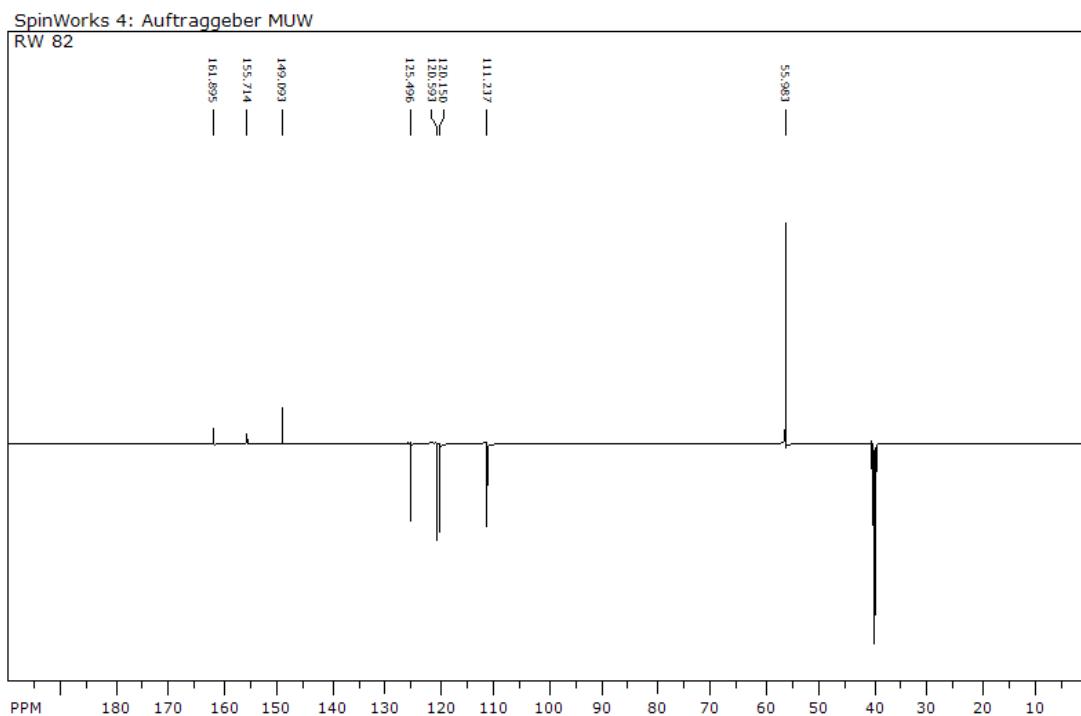
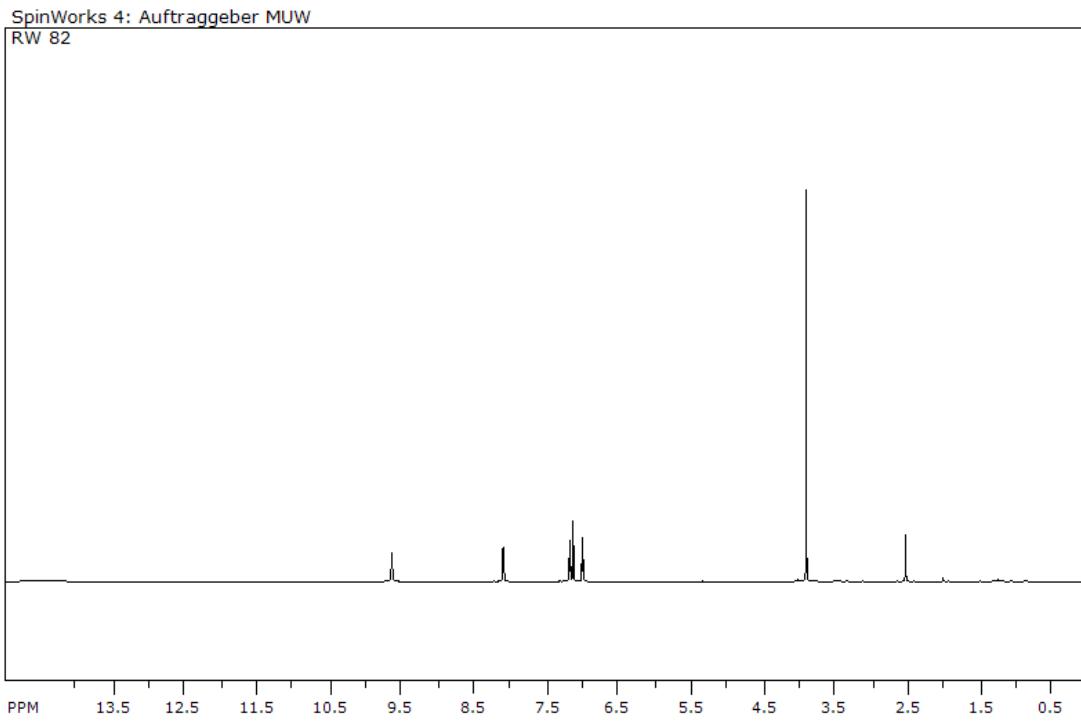


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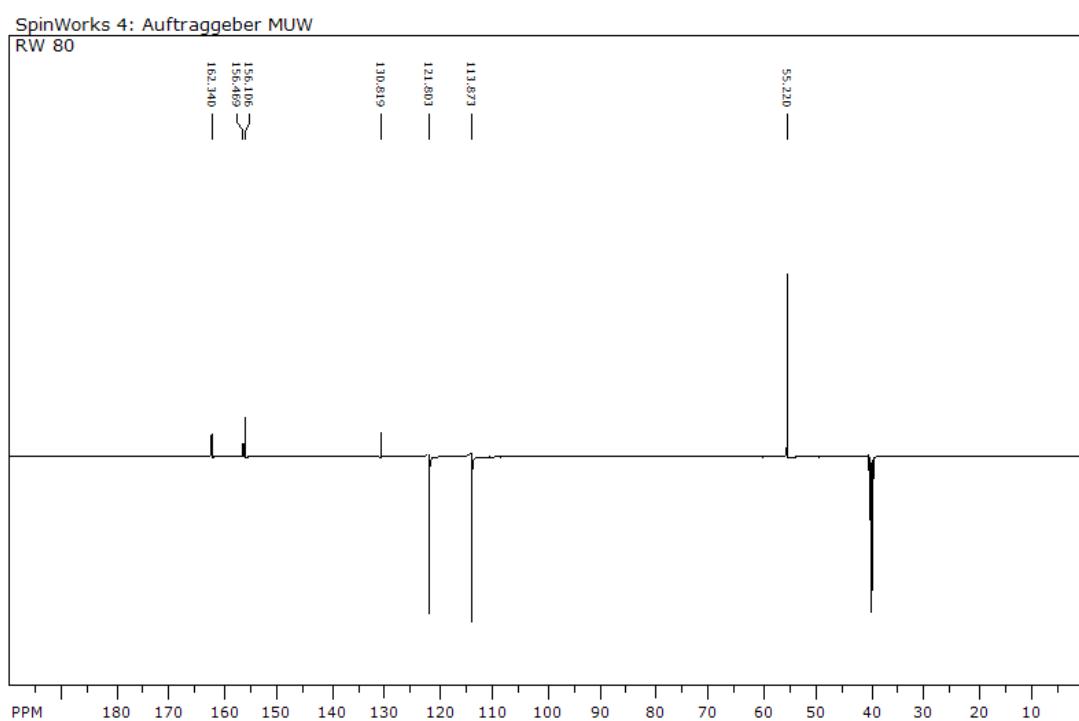
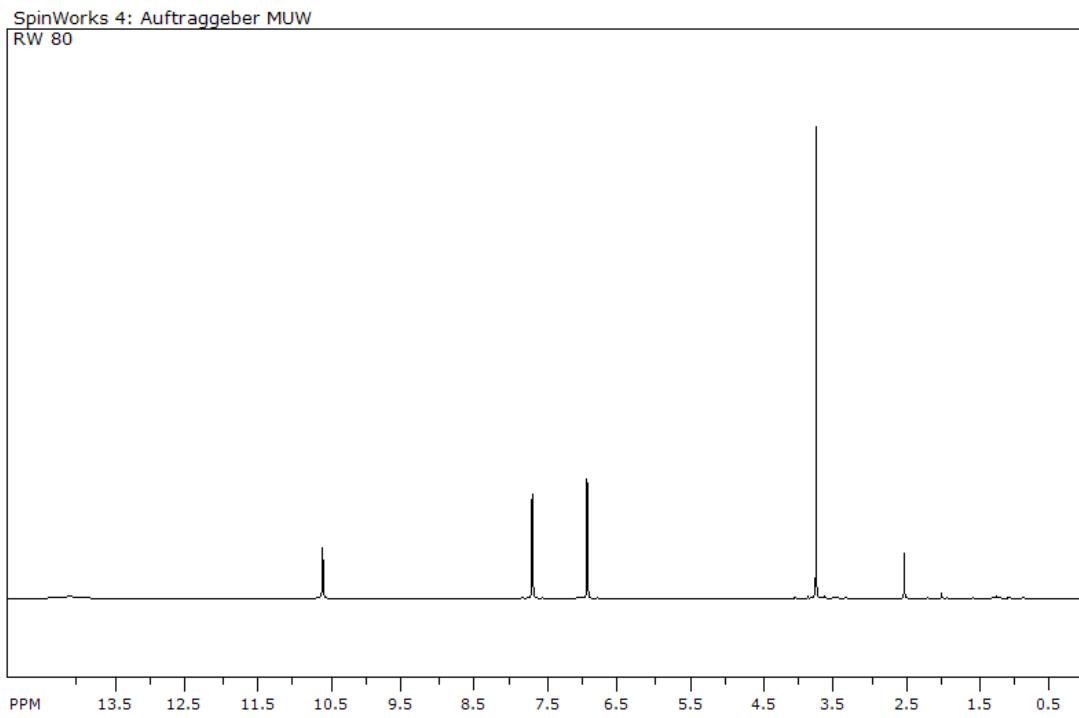
2-((2-hydroxyphenyl)amino)-2-oxoacetic acid (S16)



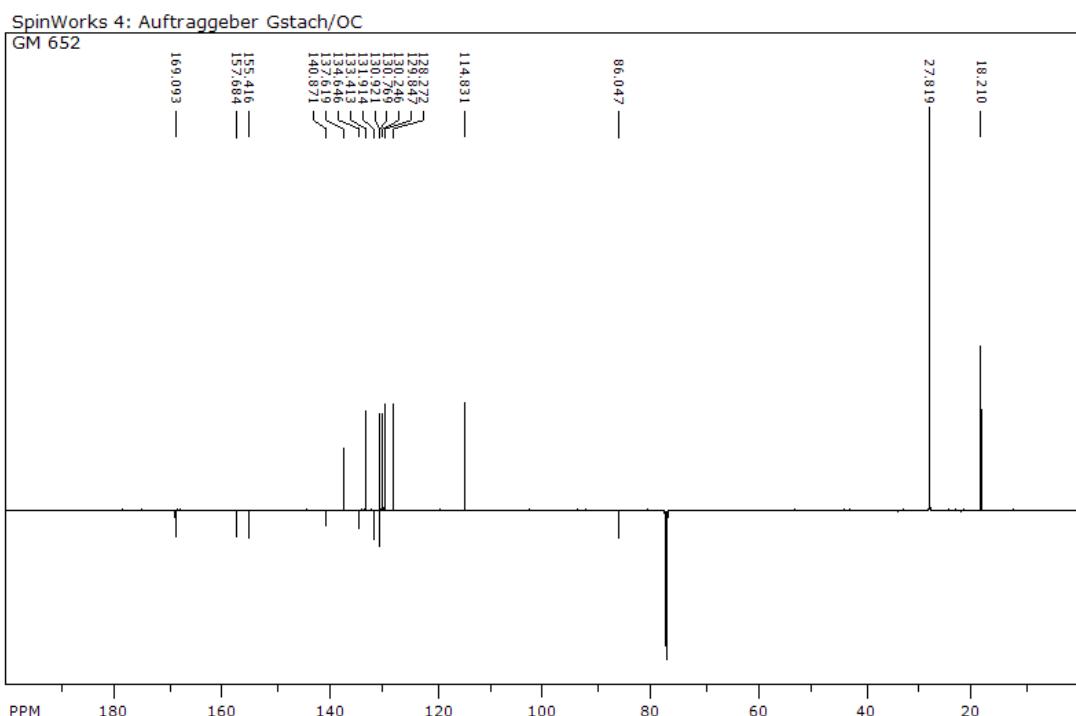
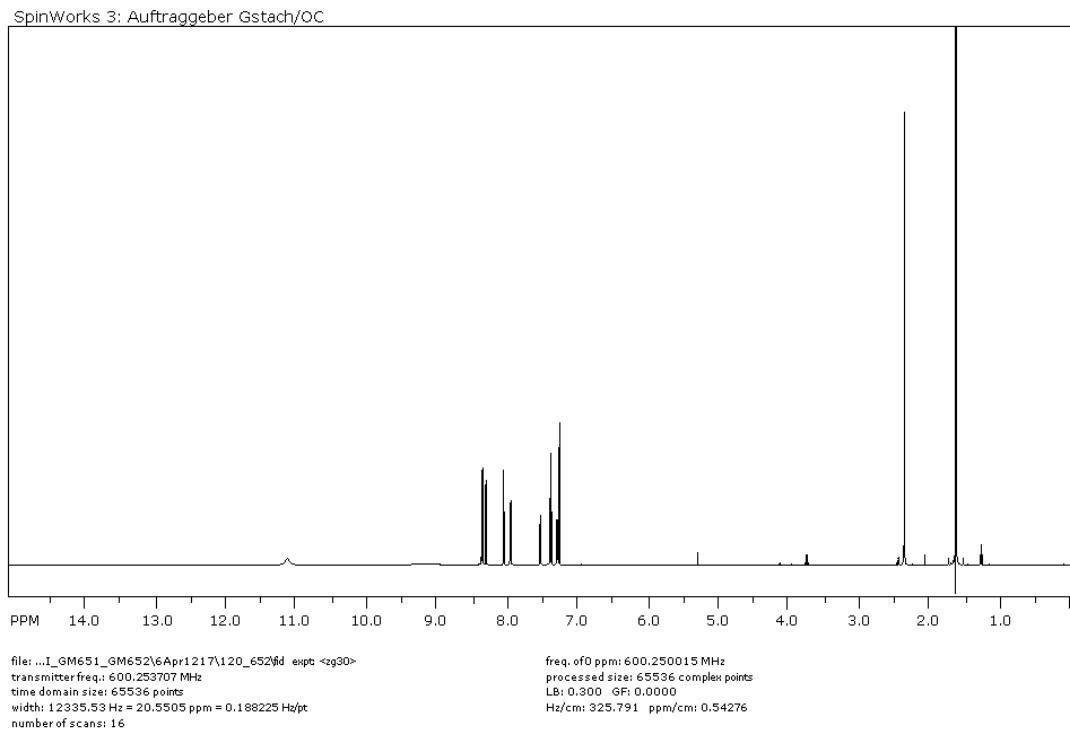
2-((2-methoxyphenyl)amino)-2-oxoacetic acid (S17)

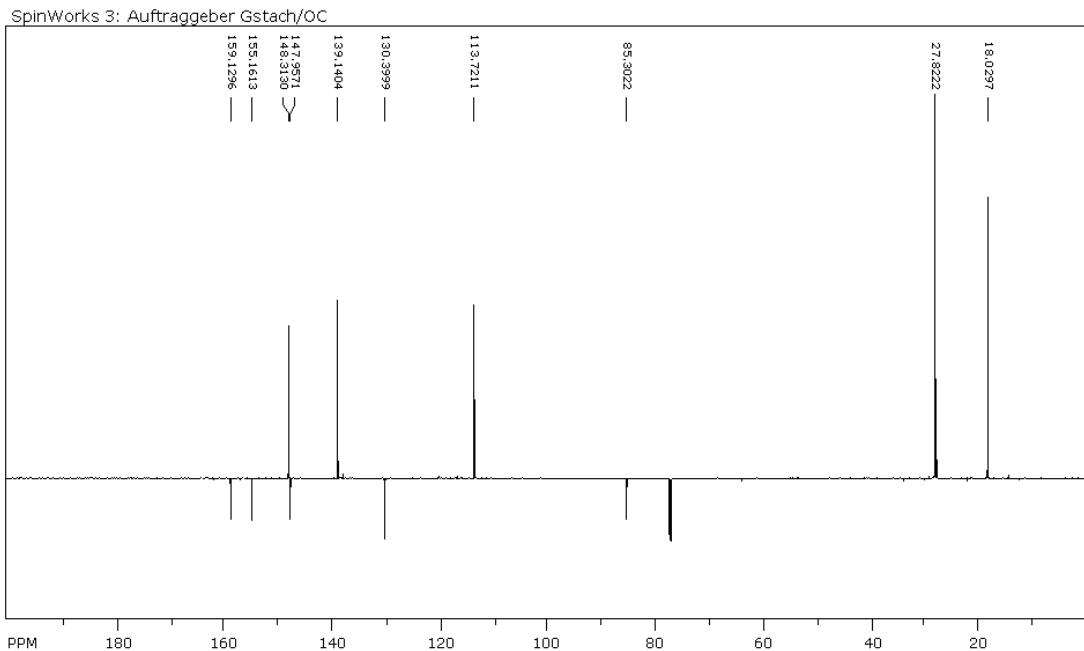
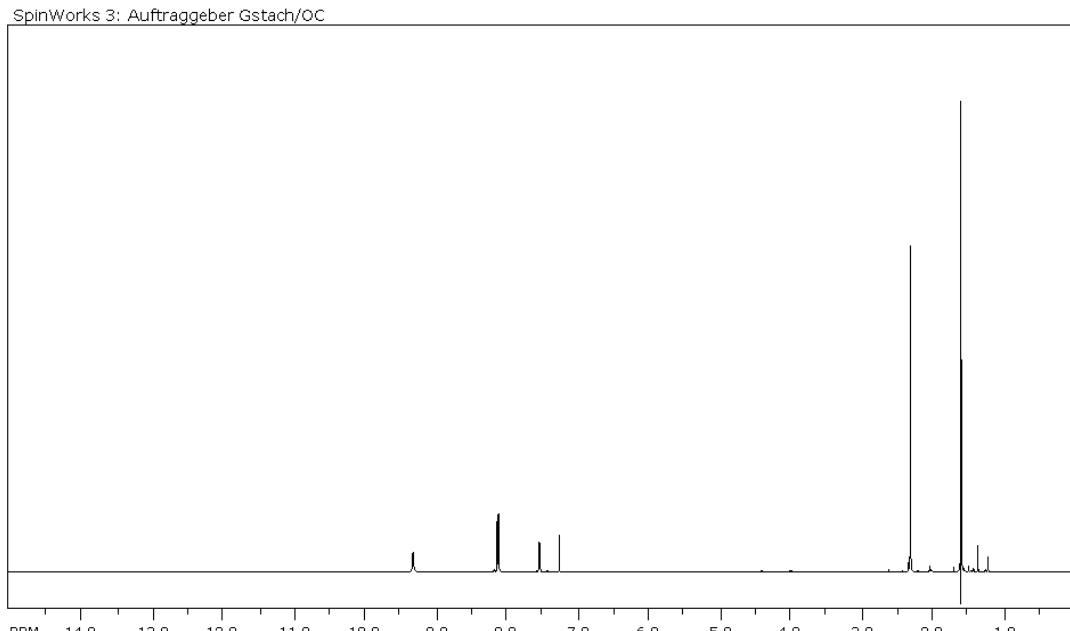


2-((4-methoxyphenyl)amino)-2-oxoacetic acid (S18)

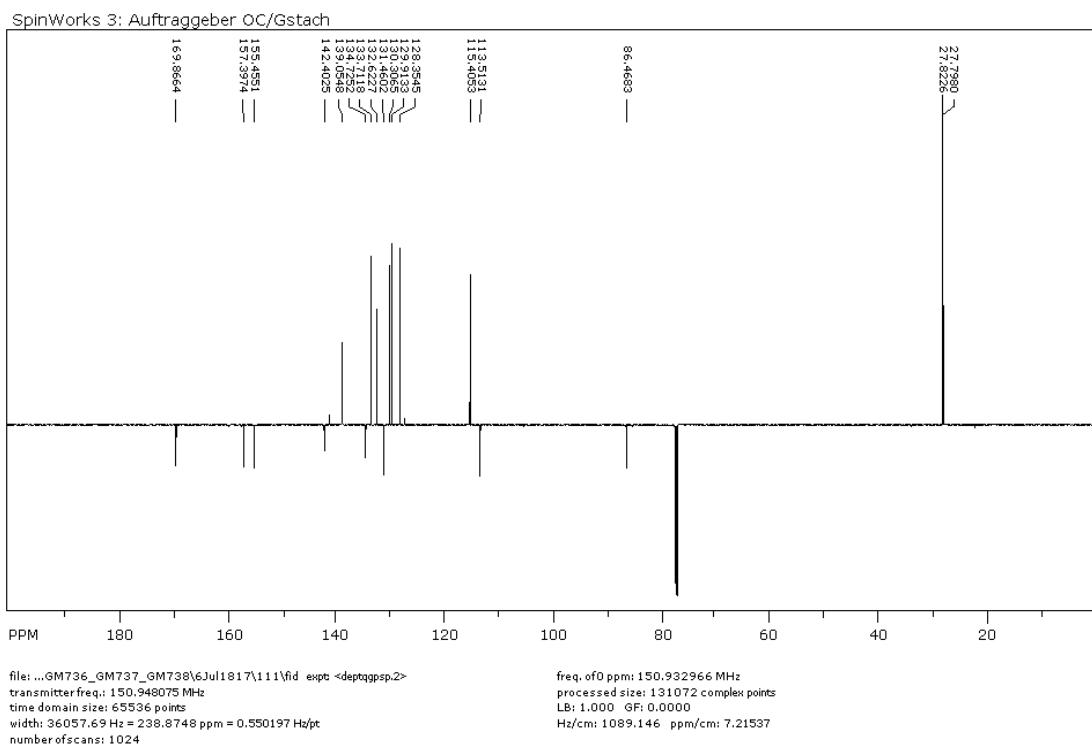
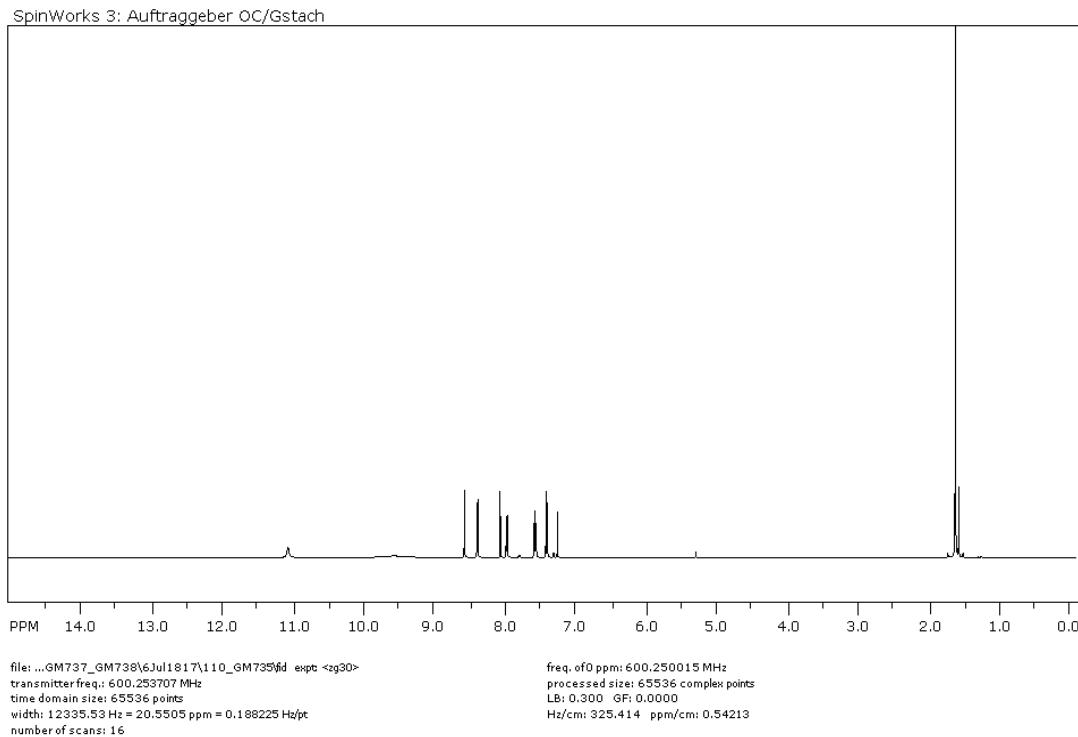


2-(2-(*tert*-butoxy)-2-oxoacetamido)-5-methylpyridine 1-oxide (S19)

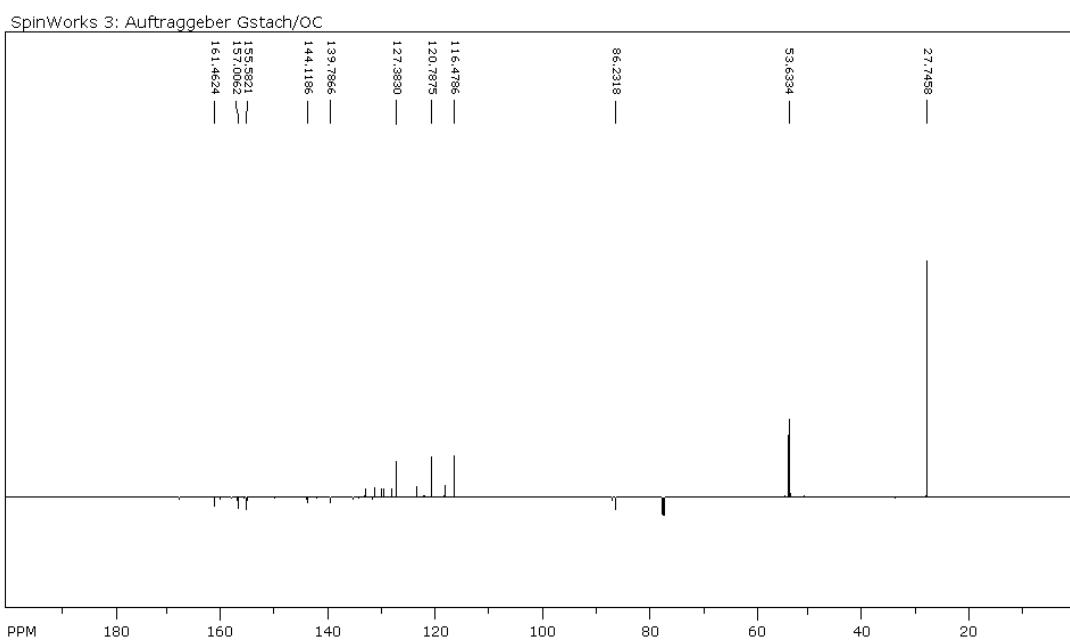
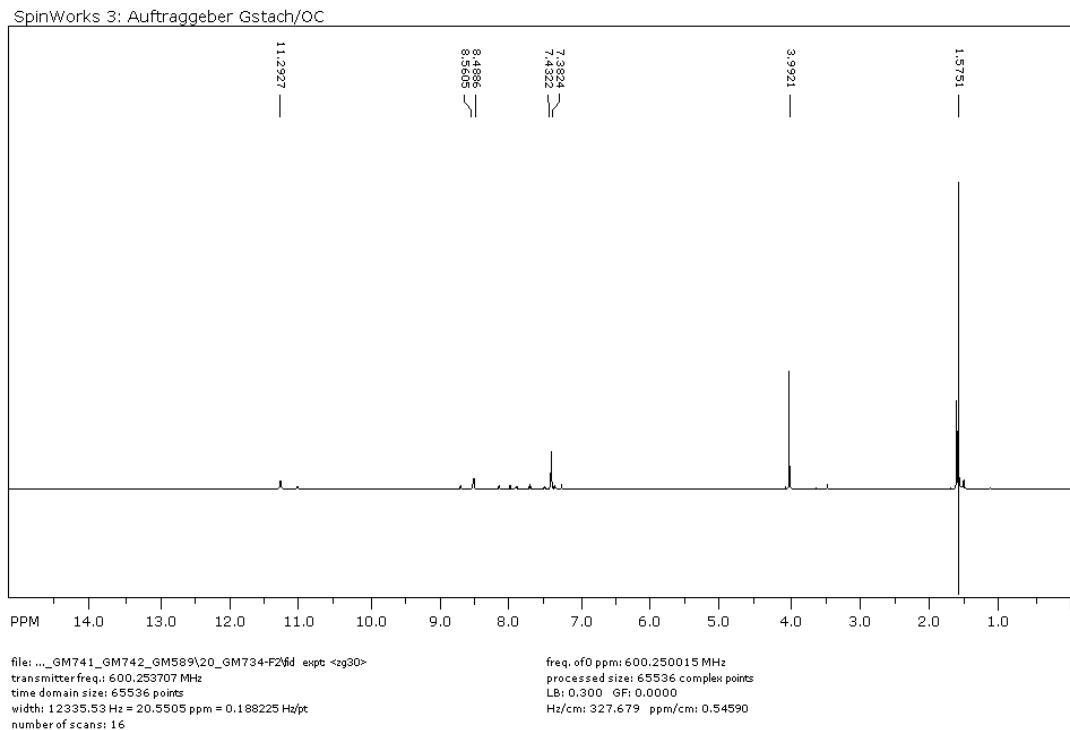


tert-butyl 2-((5-methylpyridin-2-yl)amino)-2-oxoacetate (S20)


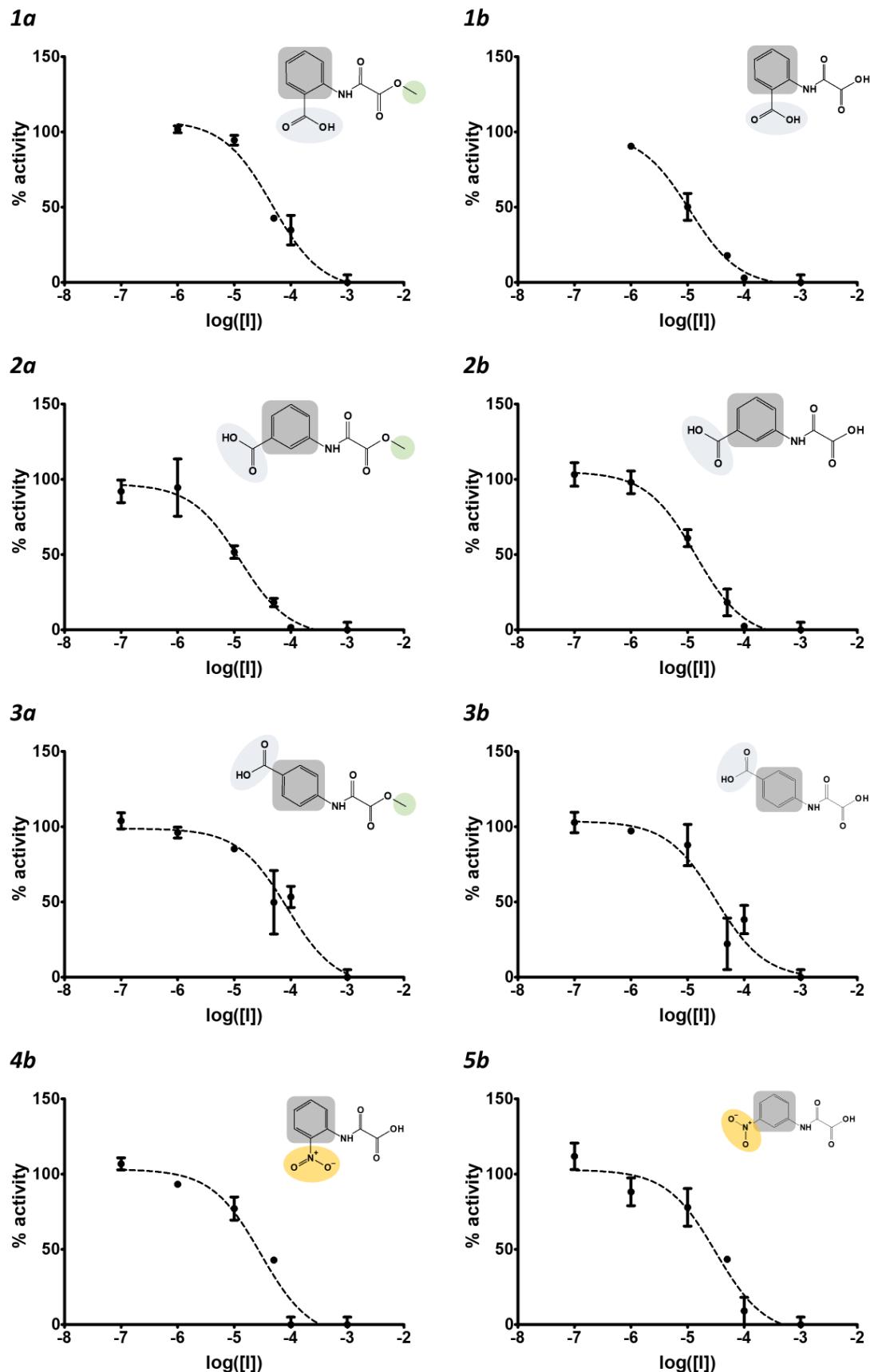
5-bromo-2-(2-(*tert*-butoxy)-2-oxoacetamido)pyridine 1-oxide (S21)

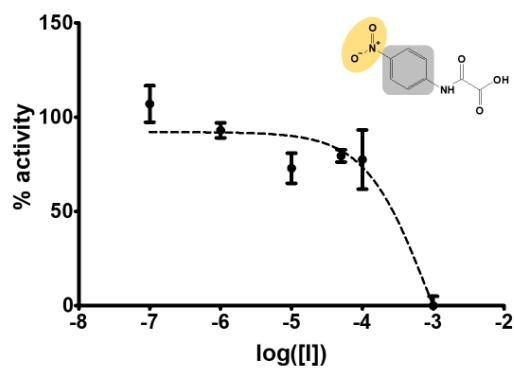
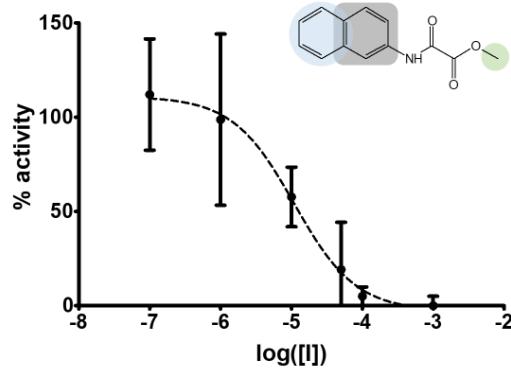
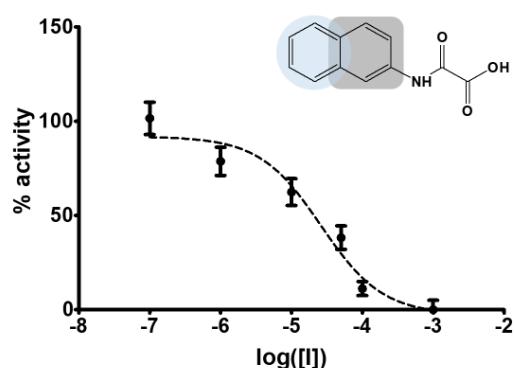
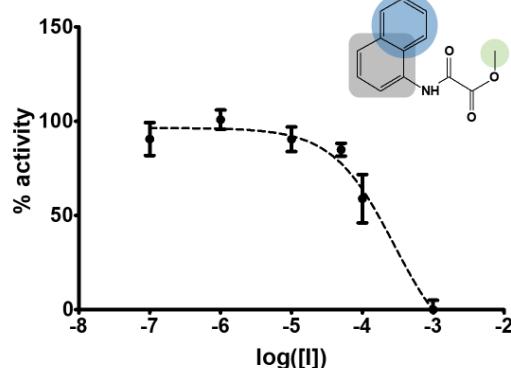
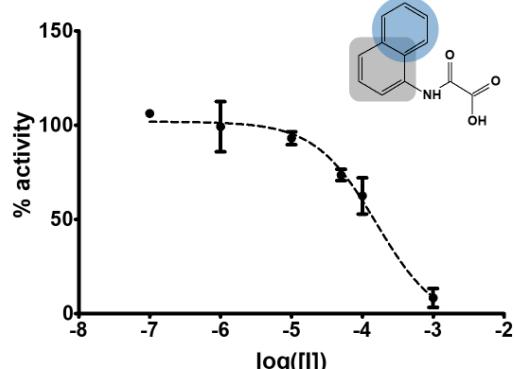
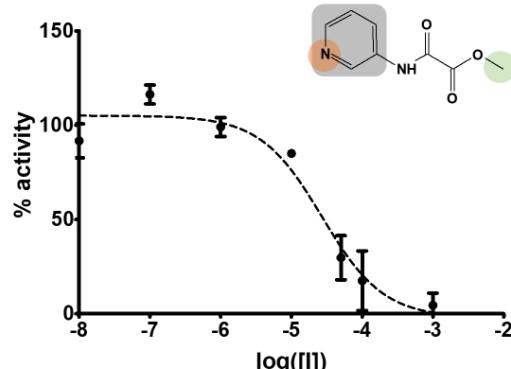
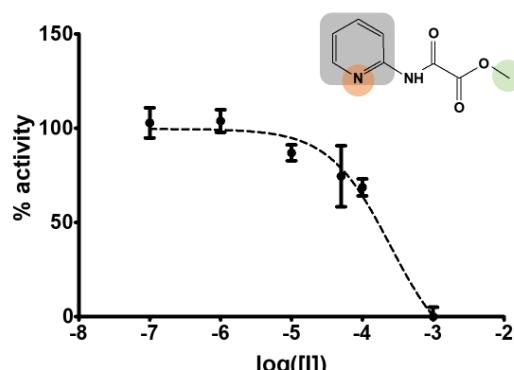
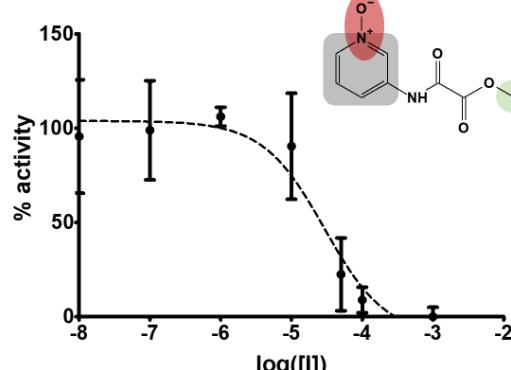


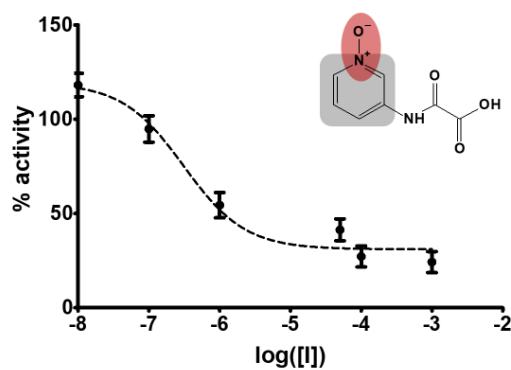
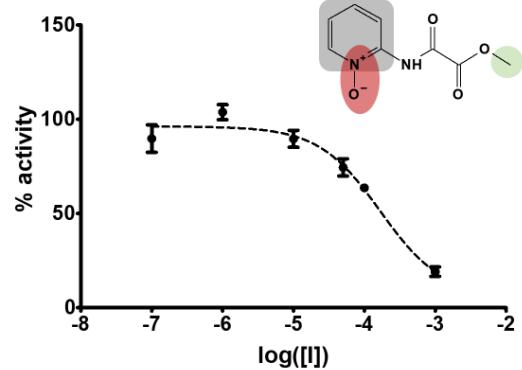
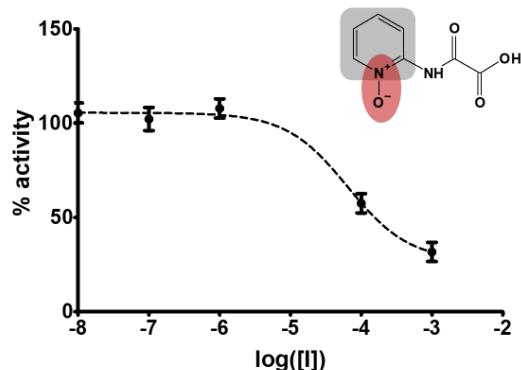
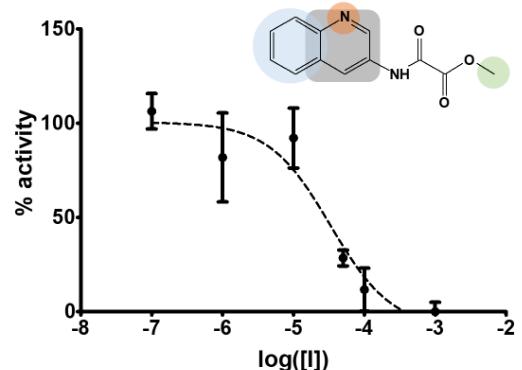
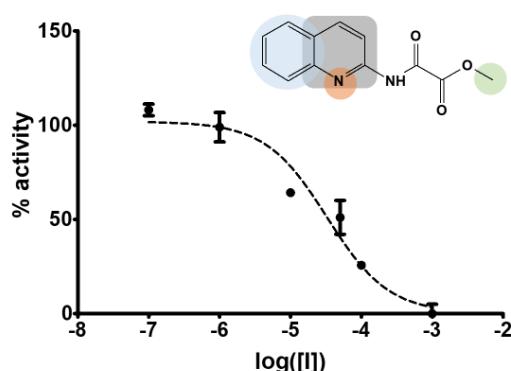
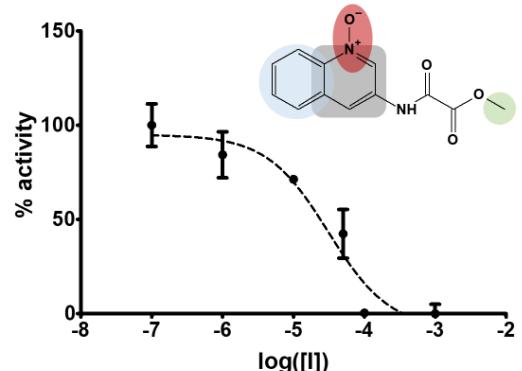
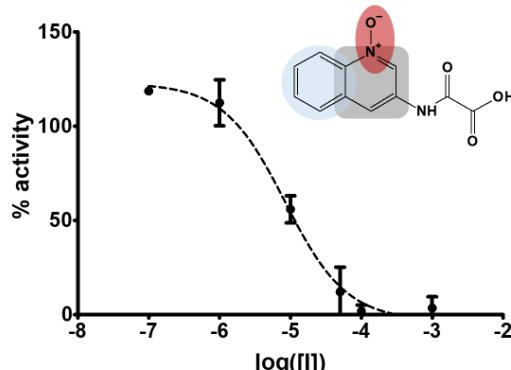
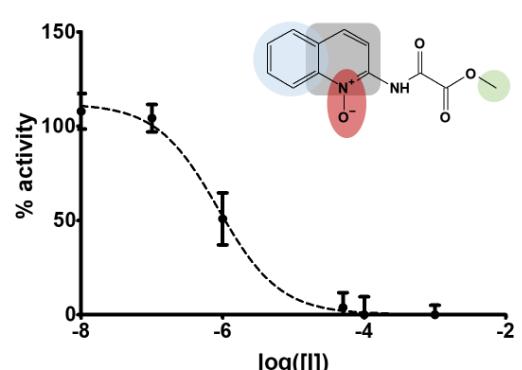
2-(2-(*tert*-butoxy)-2-oxoacetamido)-6-(methoxycarbonyl)pyridine 1-oxide (S22)

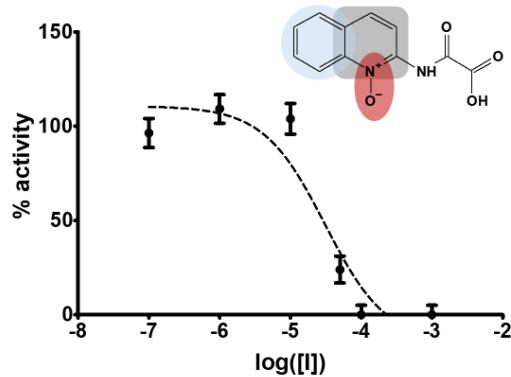
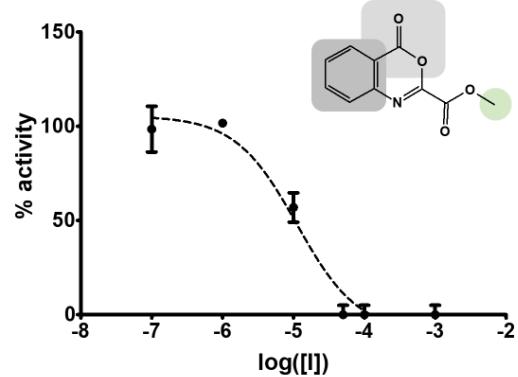
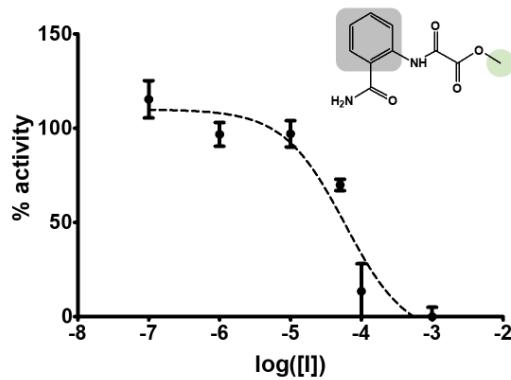
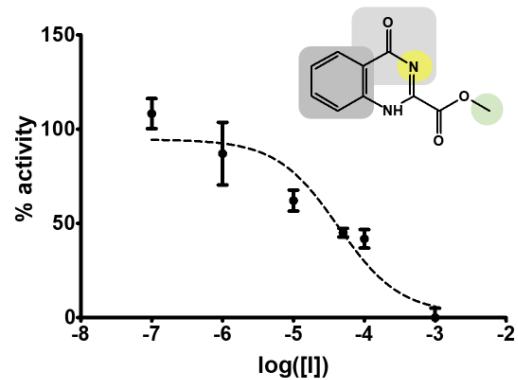
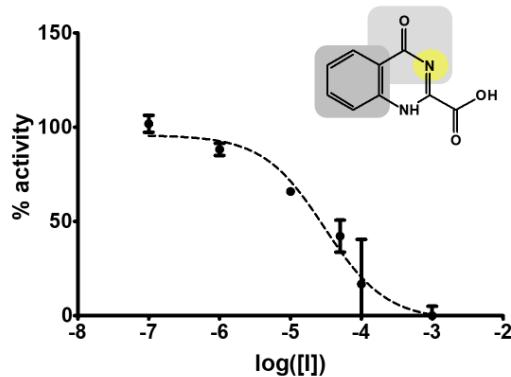
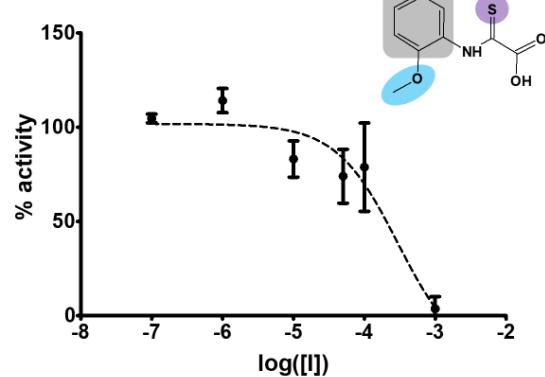
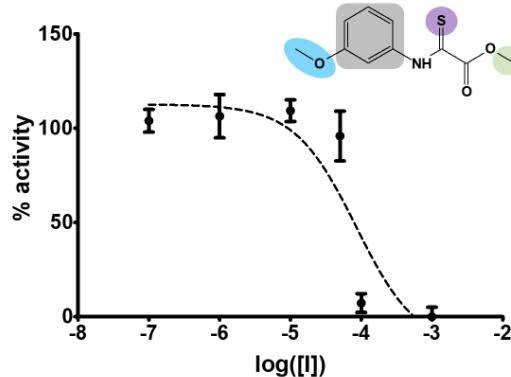
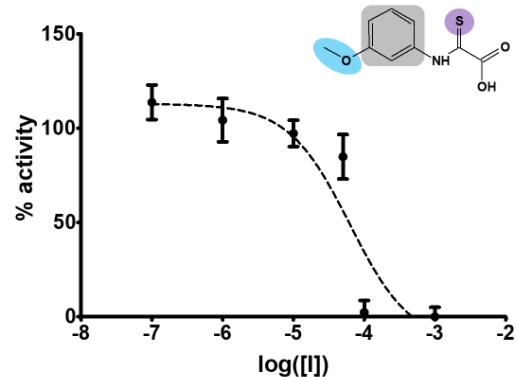


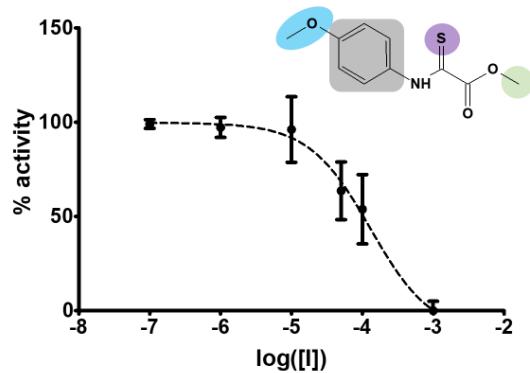
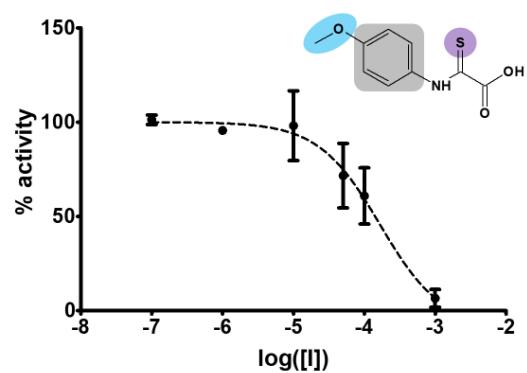
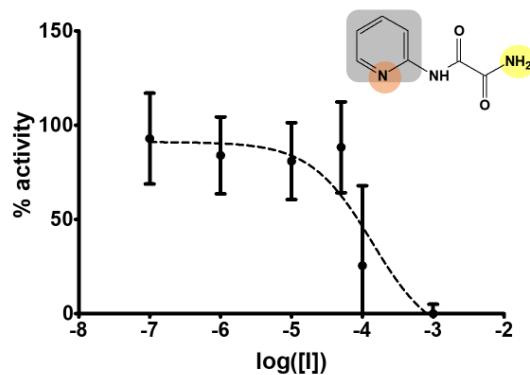
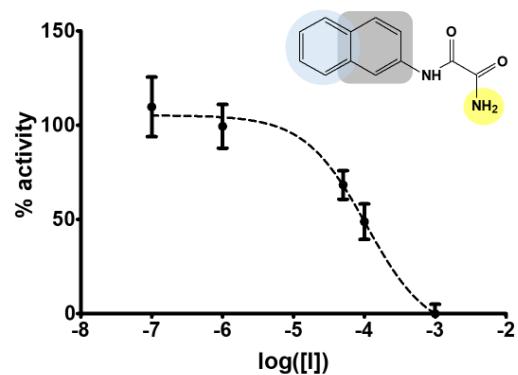
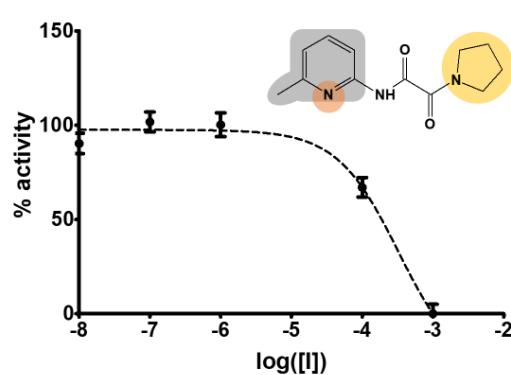
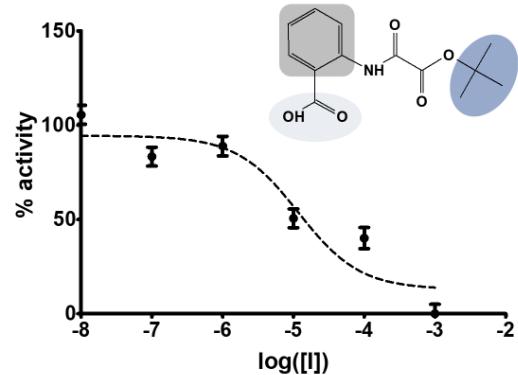
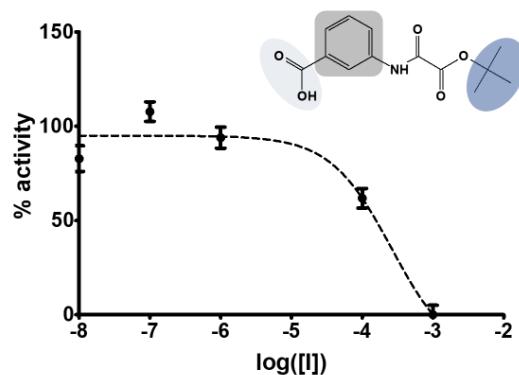
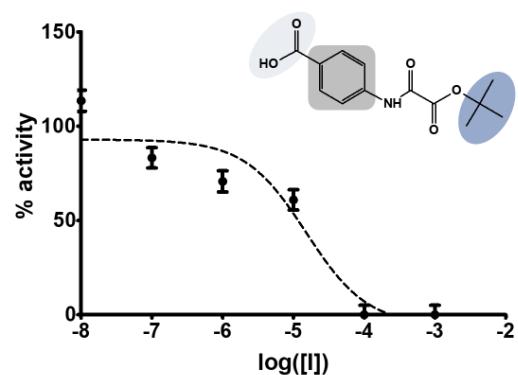
- FAHD1-inhibitor titration curves

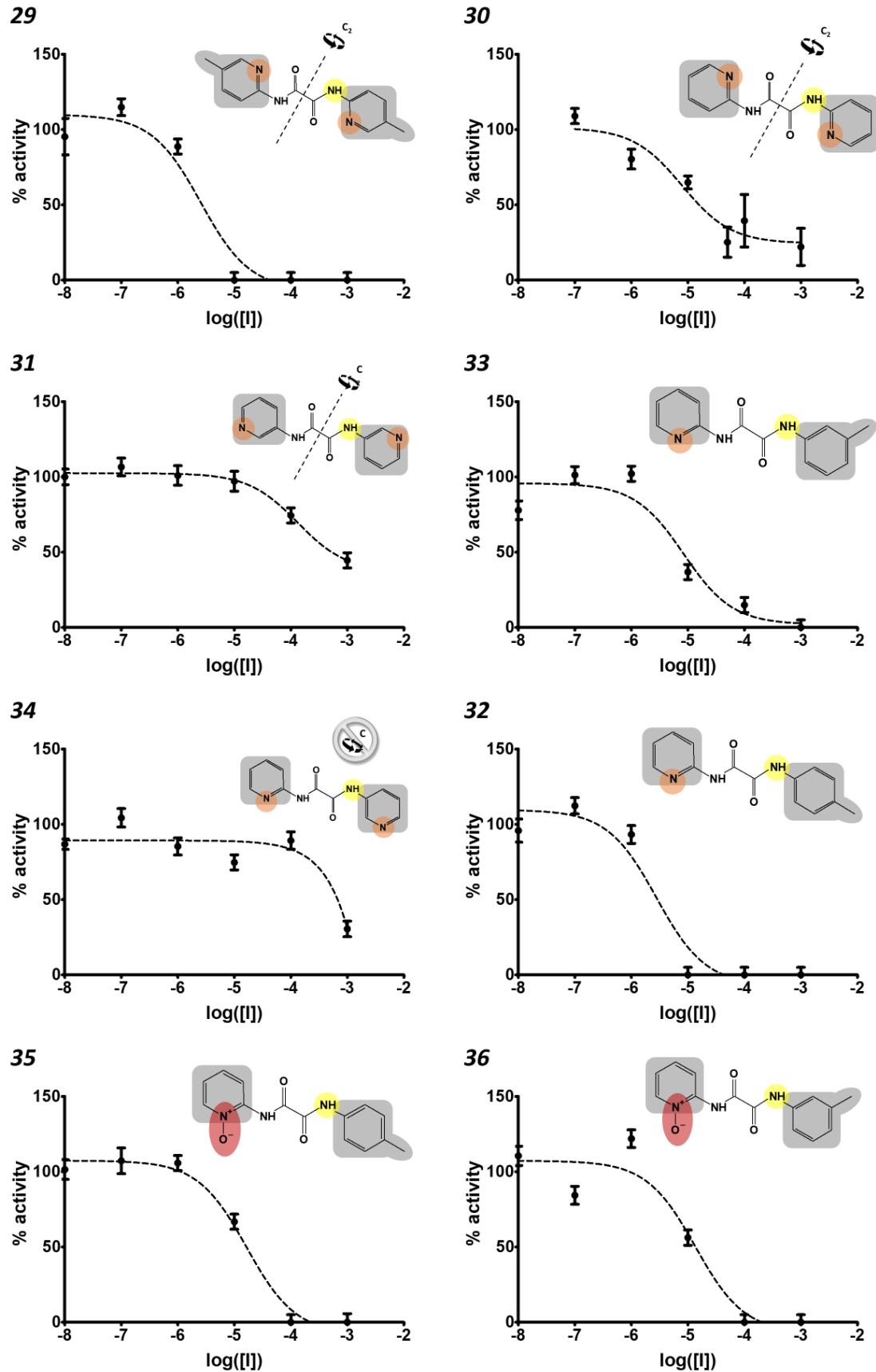


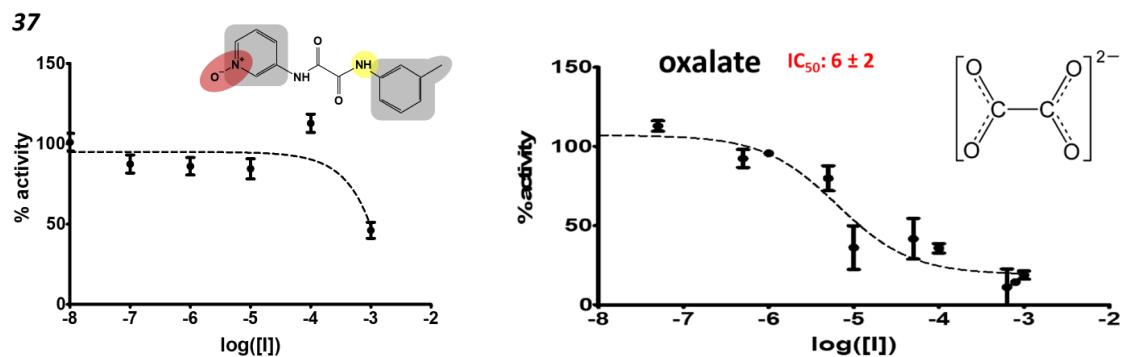
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11b**12a****12b****13a****14a****15a****15b****16a**

16b**17a****18a****19a****19b****20b****21a****21b**

22a**22b****23****24****25****26****27****28**





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