Electronic Supplementary Material (ESI) for Catalysis Science & Technology.

Supplementary Information

Enantioselective Michael addition of aldehydes to maleimides catalysed by surface-adsorbed natural amino acids

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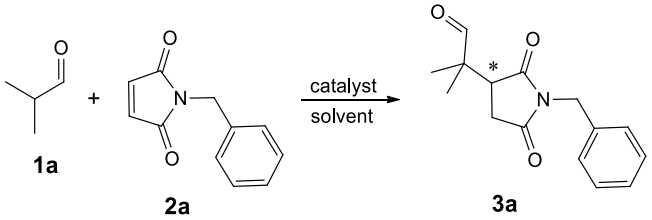
Table of contents page

1. Formulae used for calculating conversions (Conv) and enantiomeric excesses (ee) 2
2. General procedure for preparation of *N*-substituted maleimides 3
3. Analytical data of the prepared *N*-substituted maleimides 4
4. Effect of the reaction conditions in the addition of isobutyraldehyde to *N*-benzylmaleimide 10
5. Structure of the amino acids tested 13
6. Structure of the carbonyl compounds used as nucleophiles 13
7. Results obtained in the addition of isobutyraldehyde to *N*-benzylmaleimide using various amino acids 14
8. Analytical data of the Michael adducts 16
9. Results of UV-Vis spectroscopic investigations 30
10. Thermogravimetric analysis of L-phenylalanine 33
11. XRD diffractograms of selected materials 34
12. FT-IR spectra of hybrid materials 35
13. Adsorption mode on Al_2O_3 37
14. ^{1}H- and ^{13}C-NMR spectra of the prepared materials 38
    - 14.1. ^{1}H- and ^{13}C-NMR spectra of the *N*-substituted maleimides 38
    - 14.2. ^{1}H- and ^{13}C-NMR spectra of the Michael adducts 63
15. Chromatograms and mass spectra of the materials 105
    - 15.1. GC-MSD chromatograms and mass spectra of the *N*-substituted maleimides 105
    - 15.2. GC-MSD chromatograms and mass spectra of the Michael adducts 118
    - 15.3. Separation of the Michael adduct enantiomers by GC-FID 139

1. Formulae used for calculating conversions (Conv) and enantiomeric excesses (ee).

Test reaction:



Enantioselective conjugate addition of isobutyraldehyde to *N*-benzylmaleimide.

Formulae used:

$$\mathrm{Conv}(\mathbf{2a})\ (\%) = \frac{c\mathbf{2a}_0 - c\mathbf{2a}}{c\mathbf{2a}_0} * 100$$

$$\mathrm{ee}(\mathbf{3a})\ (\%) = \frac{|c\mathbf{3a}(S) - c\mathbf{3a}(R)|}{(c\mathbf{3a}(S) + c\mathbf{3a}(R))} * 100$$

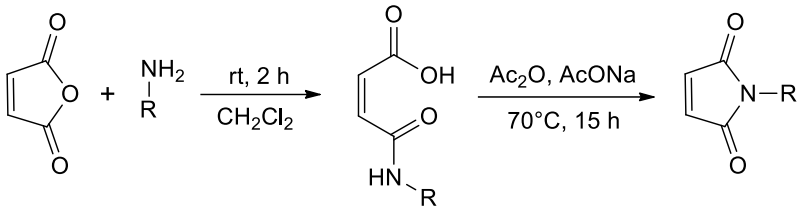
where:

Conv(**2a**) is the conversion of the *N*-benzylmaleimide; $c\mathbf{2a}_0$ is the initial and $c\mathbf{2a}$ is the final concentration of **2a** determined by GC-FID.

ee(**3a**) is the enantiomeric excess of the Michael adduct **3a**; $c\mathbf{3a}(S)$ and $c\mathbf{3a}(R)$ are the concentrations of the *S* and *R* enantiomers of **3a** determined by GC-FID.

In reactions in which Michael adducts having two chiral centres were formed the diastereomeric ratio (dr) was expressed as the ratio of the two enantiomer pairs and the ees of both pairs were calculated according to the above given formula using the corresponding concentrations of the individual enantiomers within one pair, determined by GC-FID.

2. General procedure for preparation of *N*-substituted maleimides



R: substituted phenyl or benzyl group, aliphatic, etc.

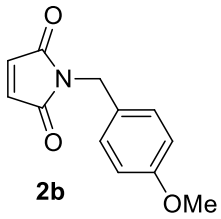
Compounds *N*-benzylmaleimide (**2a**), *N*-phenylmaleimide (**2h**), *N*-methylmaleimide (**4a**), *N*-ethylmaleimide (**4b**) and *N*-*tert*-butylmaleimide (**4k**) were commercial products purchased from Aldrich and were used as received.

The other *N*-substituted maleimides used in the present study were prepared by a modified literature method,[a] using 40 mmol (or 20 mmol) maleic anhydride dissolved in 25 cm^3 (or 12.5 cm^3) CH_2Cl_2 in a round-bottom flask of 100 cm^3 (or 50 cm^3) volume flushed with dry N_2. One equivalent of the corresponding amine dissolved in 15 cm^3 (or 8 cm^3) CH_2Cl_2 was dropwise added to this solution. After 2 h stirring the solvent was evaporated and to the resulted crystalline solid material 12 cm^3 (or 6 cm^3) acetic anhydride and 25 mmol (or 12.5 mmol) anhydrous sodium acetate were added. The solution was stirred overnight (15-20 h) at 70°C. The cooled mixture was poured into 150 cm^3 (or 75 cm^3) ice-water, the organic phase was separated, the aqueous solution was washed twice with 50 cm^3 (or 25 cm^3) CH_2Cl_2 and the unified organic solutions were dried over sicc. Na_2SO_4. Evaporation of the solvent gave the crude product, which was purified either by crystallization in hexane/ethyl acetate mixture or by flash chromatography using hexane/ethyl acetate mixture as eluents (see the analytical data of the compounds). Products were identified and their purity were checked by GC-MSD and ^{1}H- and ^{13}C-NMR (for analytical data, chromatograms and spectra see below).

[a] S. Firoozi, M. Hosseini-Sarvari, M. Koohgard, Green Chem. 20 (2018) 5540-5549, https://doi.org/10.1039/c8gc03297a.

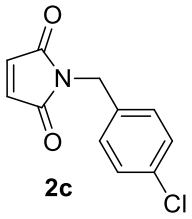
3. Analytical data of the prepared *N*-substituted maleimides

N-(4-methoxybenzyl)maleimide (**2b**),
20 mmol scale, purified by crystallization in hexane/EtOAc 3/2, 2.694 g white solid, yield 62%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.29 (d, 2H, Ar-H), 6.83 (d, 2H, Ar-H), 6.68 (s, 2H, HC=), 4.61 (s, 2H, CH_2), 3.78 (s, 3H, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.4, 159.3, 134.2, 129.9, 128.5, 114.0, 55.2, 40.9.
GC-MSD *m/z*(rel. int.): *217*(M^+, 100), *174*(41) *136*(31), *121*(17), *108*(11).



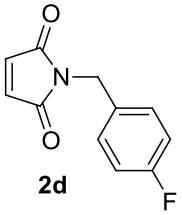
2b

N-(4-chlorobenzyl)maleimide (**2c**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 8/1, 3.059 g transparent liquid, crystallized upon standing at 4°C, yield 69%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.28 (s, 4H, Ar-H), 6.71 (s, 2H, HC=), 4.63 (s, 2H, CH_2).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.2, 134.6, 134.3, 133.9, 129.9, 128.9, 40.8.
GC-MSD *m/z*(rel. int.): *221*(M^+, 100), *186*(35) *158*(19), *140*(63), *130*(22), *89*(20).



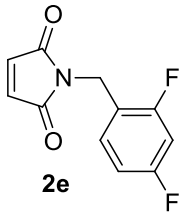
2c

N-(4-fluorobenzyl)maleimide (**2d**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 3/1, 2.955 g transparent oil, yield 72%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.34-7.31 (d, 2H, Ar-H), 7.01-6.97 (d, 2H, Ar-H), 6.70 (s, 2H, HC=), 4.64 (s, 2H, CH_2).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.3, 163.4, 161.4, 134.2, 132.0, 130.4, 130.3, 115.6, 115.5, 40.7.
GC-MSD *m/z*(rel. int.): *205*(M^+, 100), *148*(40) *122*(65), *109*(25), *96*(21).



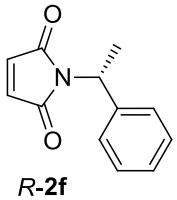
2d

N-(2,4-difluorobenzyl)maleimide (**2e**),
40 mmol scale, flash chromatography: eluted with hexane/EtOAc 8/1, 2.812 g transparent liquid, yield 63%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.31-7.27 (m, 2H, Ar-H), 6.82 (m, 1H, Ar-H), 6.72 (s, 2H, HC=), 4.72 (s, 2H, CH_2).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.0, 163.7, 163.6, 134.3, 131.4, 131.3, 131.2, 119.1, 119.0, 111.5, 111.3, 104.2, 104.0, 103.8, 41.4, 34.7.
GC-MSD *m/z*(rel. int.): *223*(M^+, 100), *166*(35), *140*(67), *127*(38), *114*(30).



2e

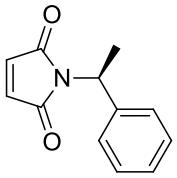
(*R*)-*N*-(1-phenylethyl)maleimide (*R*-**2f**),
20 mmol scale, flash chromatography: eluted with hexane/EtOAc 10/1, 2.978 g pale yellow oil, yield 74%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.41 (m, 2H, Ar-H), 7.31 (m, 2H, Ar-H), 7.25 (m, 1H, Ar-H), 6.61 (s, 2H, HC=), 5.34 (m, 1H), 1.81 (d, 3H, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.5, 140.2, 134.0, 128.5, 127.6, 127.2, 49.6, 17.6.



R-**2f**

GC-MSD *m/z*(rel. int.): *201*(M^+, 100), *186*(65) *158*(29), *120*(35), *104*(46), *77*(47).

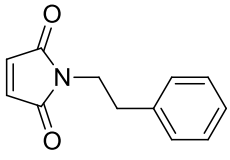
(*S*)-*N*-(1-phenylethyl)maleimide (*S*-**2f**),
20 mmol scale, flash chromatography: eluted with hexane/EtOAc 10/1, 3.018 g pale yellow oil, yield 75%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.40 (m, 2H, Ar-H), 7.31 (m, 2H, Ar-H), 7.25 (m, 1H, Ar-H), 6.61 (s, 2H, HC=), 5.35 (m, 1H), 1.82 (d, 3H, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.5, 140.3, 134.0, 128.5, 127.7, 127.2, 49.7, 17.6.
GC-MSD *m/z*(rel. int.): *201*(M^+, 100), *186*(65) *158*(24), *120*(35), *104*(47), *77*(45).



S-**2f**

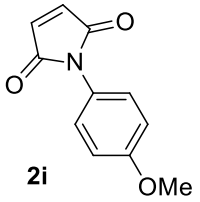
N-(1-phenylethyl)maleimide (*rac*-**2f**),
The racemic compound was prepared by mixing equal amounts (0.3 g) of the two enantiomers, *R*-**2f** and *S*-**2f**.

N-(2-phenylethyl)maleimide (**2g**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 9/1, 2.898 g transparent oil, yield 72%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.30-7.27 (m, 2H, Ar-H), 7.23-7.19 (m, 3H, Ar-H), 6.65 (s, 2H, HC=), 3.76 (tr, 2H, *J* 7.6 Hz, CH_2), 3.76 (tr, 2H, *J* 7.8 Hz, CH_2).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.5, 137.8, 134.0, 128.8, 128.5, 126.7, 39.1, 34.5.
GC-MSD *m/z*(rel. int.): *201*(M^+, 40), *110*(40) *104*(100), *91*(37), *82*(10), *65*(10).



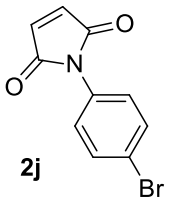
2g

N-(4-methoxyphenyl)maleimide (**2i**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 4/1, 3.251 g light yellow solid, yield 80%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.23 (m, 2H, Ar-H), 6.98 (m, 2H, Ar-H), 6.83 (s, 2H, HC=), 3.83 (s, 3H, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 169.8, 159.2, 134.1, 127.6, 123.7, 114.5, 55.5.
GC-MSD *m/z*(rel. int.): *203*(M^+, 100), *188*(35) *160*(23), *134*(11), *106*(9).



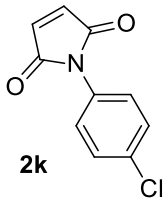
2i

N-(4-bromophenyl)maleimide (**2j**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 6/1, 3.730 g yellow crystals, yield 74%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.59 (d, 2H, *J* 7.6 Hz, Ar-H), 7.26 (d, 2H, *J* 6.3 Hz, Ar-H), 6.85 (s, 2H, HC=).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 169.0, 134.3, 132.3, 130.4, 127.3, 121.6.
GC-MSD *m/z*(rel. int.): *252*(M^+, 100), *207*(10), *183*(16), *116*(22), *90*(16), *54*(20).



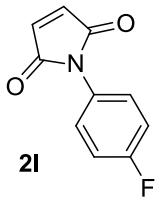
2j

N-(4-chlorophenyl)maleimide (**2k**),
40 mmol scale, purified by crystallization in hexane/EtOAc 10/1, 6.810 g pale yellow solid, yield 82%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.43 (m, 2H, Ar-H), 7.32 (m, 2H, Ar-H), 6.85 (s, 2H, HC=).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 169.1, 134.3, 133.7, 129.9, 129.3, 127.1.
GC-MSD *m/z*(rel. int.): *207*(M^+, 100), *163*(12) *151*(10), *137*(21), *90*(9), *54*(14).



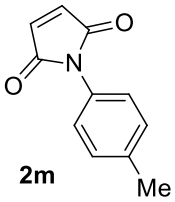
2k

N-(4-fluorophenyl)maleimide (**2l**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 8/1, 2.944 g pale yellow liquid, yield 77%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.34-7.31 (m, 2H, Ar-H), 7.17-7.13 (m, 2H, Ar-H), 6.84 (s, 2H, HC=).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 169.3, 162.8, 160.9, 134.2, 127.9, 127.8, 116.2, 116.0.
GC-MSD *m/z*(rel. int.): *191*(M^+, 100), *147*(17) *135*(15), *121*(29), *109*(15), *54*(19).



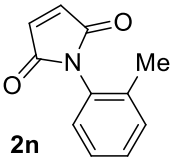
2l

N-(4-methylphenyl)maleimide (**2m**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 6/1, 2.434 g pale yellow oil, yield 65%;
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.27-7.19 (m, 4H, *J* 7.8 Hz, Ar-H), 6.81 (s, 2H, HC=), 2.38 (s, 3H, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 169.6, 138.0, 134.2, 129.8, 128.7, 126.0, 21.1.
GC-MSD *m/z*(rel. int.): *187*(M^+, 100), *158*(10), *143*(8), *130*(23), *117*(18).



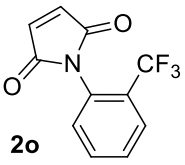
2m

N-(2-methylphenyl)maleimide (**2n**),
40 mmol scale, flash chromatography, eluted with hexane/EtOAc 6/1, 5.841 g transparent liquid, yield 78%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.35-7.27 (m, 3H, Ar-H), 7.11 (d, 1H, *J* 7.1 Hz, Ar-H), 6.85 (s, 2H, HC=), 2.16 (s, 3H, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 169.5, 136.5, 134.4, 131.2, 130.1, 129.4, 128.7, 126.9, 17.8.
GC-MSD *m/z*(rel. int.): *187*(M^+, 100), *169*(80), *141*(18), *130*(55), *104*(20).



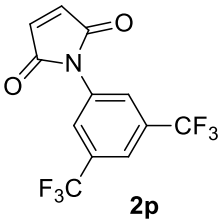
2n

N-((2-trifluoromethyl)phenyl)maleimide (**2o**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 5/1, 3.039 g pale yellow oil, yield 63%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.81 (d, 1H, *J* 7.8 Hz, Ar-H), 7.70-7.59 (m, 2H, Ar-H), 7.28 (d, 1H, *J* 7.8 Hz, Ar-H), 6.89 (s, 2H, HC=).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 169.1, 134.7, 133.1, 131.7, 130.0, 127.7, 127.6, 127.5, 124.0, 121.8.
GC-MSD *m/z*(rel. int.): *241*(M^+, 100), *197*(28) *172*(26), *165*(10), *145*(10), *54*(21).



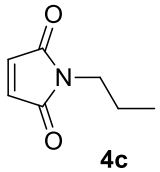
2o

N-((3,5-bistrifluoromethyl)phenyl)maleimide (**2p**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 8/1, 3.834 g pale yellow oil, yield 62%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 7.95 (s, 2H, Ar-H), 7.87 (s, 1H, Ar-H), 6.94 (s, 2H, HC=CH).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 168.3, 134,5, 132.9, 132.7, 132.5, 125.4, 123.9, 121.7, 121.2.
GC-MSD *m/z*(rel. int.): *309*(M^+, 100), *290*(26) *265*(37), *253*(12), *240*(13), *54*(40).



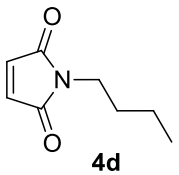
2p

N-propylmaleimide (**4c**),
40 mmol scale, flash chromatography, eluted with hexane/EtOAc 12/1, 4.175 g transparent liquid, yield 75%.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 6.67 (s, 2H, CH=), 3.48 (m, 2H, *J* 7.3 Hz, N-CH_2), 1.61 (m, 2H, *J* 7.4 Hz, CH_2), 0.89 (tr, 3H, *J* 7.4 Hz, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 170.9 (C=O), 134.0 (HC=), 39.5 (N-CH_2), 21.8 (CH_2), 11.1 (CH_3).
GC-MSD *m/z*(rel. int.): *139*(M^+, 40), *110*(100), *98*(9), 82(23), *54*(13).



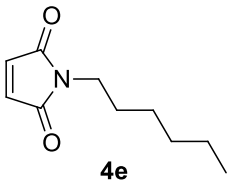
4c

N-butylmaleimide (**4d**),
40 mmol scale, flash chromatography, eluted with hexane/EtOAc 12/1, 4.835 g transparent liquid, yield 79%.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 6.66 (s, 2H, CH=), 3.51 (m, 2H, N-CH_2), 1.57 (m, 2H, CH_2), 1.30 (m, 2H, CH_2), 0.92 (tr, 3H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 170.8 (C=O), 134.0 (HC=), 41.6 (N-CH_2), 37.7 (CH_2), 30.6 (CH_2), 19.9 (CH_2), 13.5 (CH_3).
GC-MSD *m/z* (rel. int.): *153*(M^+, 24), *110*(100) *98*(18), *82*(21), *54*(9).



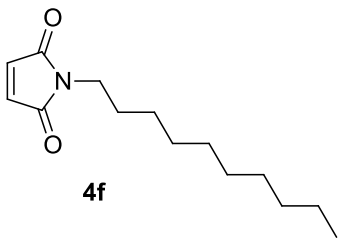
4d

N-hexylmaleimide (**4e**),
40 mmol scale, flash chromatography: eluted with hexane/EtOAc 15/1, 5.655 g transparent liquid, yield 78%.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 6.67 (s, 2H, CH=), 3.50 (m, 2H, N-CH_2), 1.56 (m, 2H, CH_2), 1.28-1.25 (m, 6H, CH_2), 0.86 (tr, 3H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 170.9 (C=O), 134.0 (HC=), 37.9 (N-CH_2), 31.3 (CH_2), 28.5 (CH_2), 26.4 (CH_2), 22.5 (CH_2), 13.9 (CH_3).
GC-MSD *m/z*(rel. int.): *181*(M^+, 22), *124*(21) *110*(100), *99*(31), *82*(22), *55*(17).



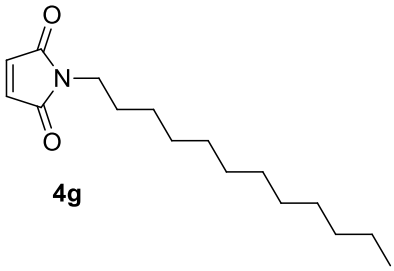
4e

N-decylmaleimide (**4f**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 15/1, 3.085 g transparent liquid which solidifies by time, yield 65%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 6.66 (s, 2H, CH=), 3.50 (tr, 2H, *J* 7.3 Hz, CH_2), 1.60-1.56 (m, 2H, CH_2), 1.30-1.24 (m, 14H, CH_2), 0.87 (tr, 3H, *J* 6.7 Hz, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.8, 134.0, 41.5, 37.9, 31.8, 29.5, 29.2, 29.1, 28.5, 26.7, 22.6.
GC-MSD *m/z*(rel. int.): *237*(M^+, 94), *138*(25) *110*(100), *99*(42), *82*(29), *55*(25).

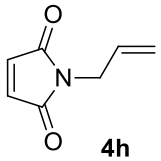


4f

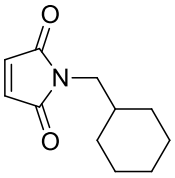
N-dodecylmaleimide (**4g**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 15/1, 3.185 g off-white solid, yield 60%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 6.67 (s, 2H, CH=), 3.50 (tr, 2H, *J* 7.3 Hz, CH_2), 1.60-1.54 (m, 2H, CH_2), 1.31-1.24 (m, 18H, CH_2), 0.88 (tr, 3H, *J* 6.8 Hz, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.9, 134.0, 77.3, 77.0, 76.7, 38.0, 31.9, 29.6, 29.5, 29.5, 29.3, 29.1, 28.5, 26.7, 22.7, 14.0.
GC-MSD *m/z*(rel. int.): *265*(M^+, 100), *138*(20) *110*(86), *99*(35), *82*(24), *55*(21).


4g

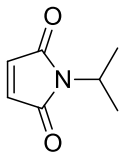
N-allylmaleimide (**4h**),
40 mmol scale, flash chromatography, eluted with hexane/EtOAc 15/1, 4.388 g transparent liquid, yield 80%.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 6.71 (s, 2H, CH=), 5.84-5.76 (m, 1H), 5.19-5.16 (m, 2H), 4.12 (d, *J* 5.6 Hz, 2H).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 170.3, 134.2, 131.5, 117.7, 77.3, 77.0, 76.7, 39.9.
GC-MSD *m/z*(rel. int.): *137*(M^+, 100), *119*(40), *110*(31), *82*(63), *66*(20), *54*(92), *26*(23).


4h

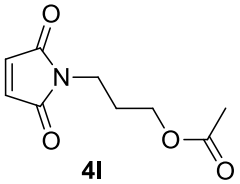
N-(cyclohexylmethyl)maleimide (**4i**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 10/1, 2.937 g pale yellow liquid, yield 76%.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 6.67 (s, 2H, CH=), 3.35 (d, 2H, *J* 7.3 Hz), 1.73-1.60 (m, 6H), 1.23-1.13 (m, 3H), 0.97-0.90 (m, 2H).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 171.1, 133.9, 44.0, 36.9, 30.7, 26.2, 25.6.
GC-MSD *m/z*(rel. int.): *193*(M^+, 80), *110*(100), *99*(71), *83*(45), *55*(59).


4i

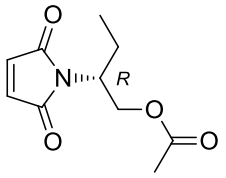
N-isopropylmaleimide (**1j**),
40 mmol scale, flash chromatography, eluted with hexane/EtOAc 15/1, 4.452 g transparent liquid, yield 80%.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 6.61 (s, 2H, CH=), 4.32 (m, 1H, *J* 7.0 Hz, N-CH_2), 1.36 (d, 6H, *J* 7.0 Hz, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 170.8, 133.9, 42.9, 20.0.
GC-MSD *m/z*(rel. int.): *139*(M^+, 24), *124*(100), *98*(8), *80*(18), *69*(12), *54*(11), *41*(10).


4j

N-(3-acetoxypropyl)maleimide (**4l**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 4/1, 2.169 g pale yellow liquid, yield 55%.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 6.67 (s, 2H, HC=), 4.01 (tr, 2H, *J* 6.1 Hz, CH_2), 3.58 (tr, 2H, *J* 6.9 Hz, CH_2), 2.00 (s, 3H, CH_3), 1.90 (m, 2H, CH_2).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.8, 170.5, 134.1, 61.5, 34.8, 27.4, 20.7.
GC-MSD *m/z*(rel. int.): *197*(M^+, 0), *151*(23) *138*(100), *110*(29), *98*(13), *43*(25).


4l

(*R*)-*N*-[(1-acetyloxymethyl)propyl]maleimide (**4m**),
20 mmol scale, flash chromatography, eluted with hexane/EtOAc 10/1, 2.197 g transparent liquid, yield 52%.



4m

^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 6.67 (s, 2H, HC=), 4.34 (m, 2H), 4.18 (m, 1H), 1.97 (s, 3H, CH_3), 0.87 (tr, 3H, *J* 7.4 Hz, CH_3).

^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 170.8, 170.5, 133.9, 63.5, 52.3, 21.8, 20.6, 10.6.

GC-MSD *m/z*(rel. int.): *211*(M^+, 0), *151*(23) *138*(100), *110*(29), *98*(14), *43*(26).

4. Effect of the reaction conditions in the addition of isobutyraldehyde to *N*-benzylmaleimide

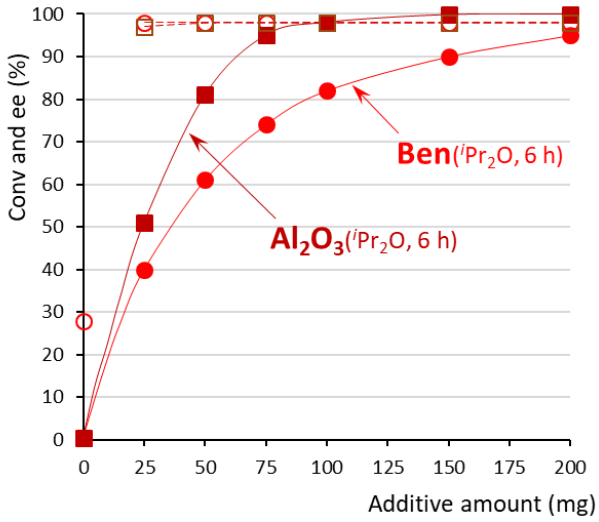


Fig. S1 Effect of the additive amount on the Conv (closed symbols) and ee (open symbols) obtained in the addition of isobutyraldehyde (**1a**) to *N*-benzylmaleimide (**2a**) using Ben (●, ○) or Al_2O_3 (■, □) additive in diisopropyl ether. Reaction conditions: 0.03 mmol (10 mol%) L-Phe, 1.2 mmol **1a**, 0.3 mmol **2a**, 1 cm^3 $^iPr_2O$, rt, 6 h.

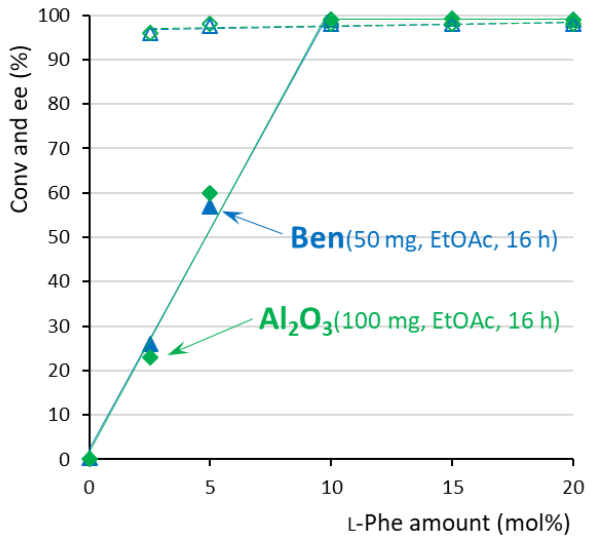


Fig. S2 Effect of L-Phe amount on the Conv (closed symbols) and ee (open symbols) in the addition of isobutyraldehyde (**1a**) to *N*-benzylmaleimide (**2a**) using 50 mg Ben (▲, △) or 100 mg Al_2O_3 (◆, ◇) additive. Reaction conditions: 1.2 mmol **1a**, 0.3 mmol **2a**, 1 cm^3 solvent, rt, 16 h.

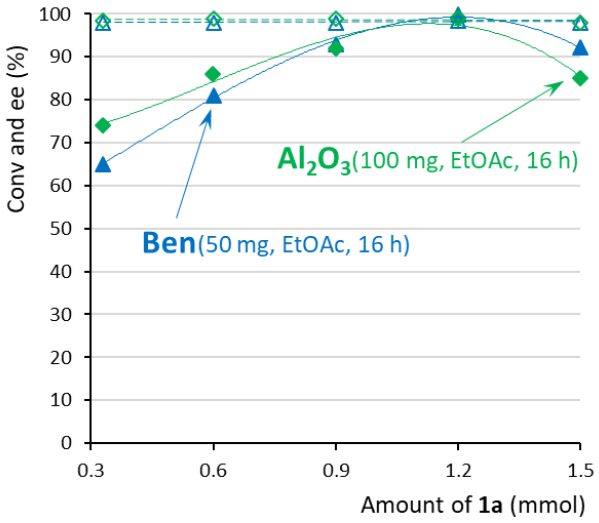


Fig. S3 Effect of the isobutyraldehyde (**1a**) amount on the Conv (closed symbols) and ee (open symbols) in the addition to *N*-benzylmaleimide (**2a**) using 50 mg Ben (▲, △) or 100 mg Al_2O_3 (◆, ◇) additive. Reaction conditions: L-Phe 0.03 mmol (10 mol%), 0.3 mmol **2a**, 1 cm^3 EtOAc, rt, 16 h.

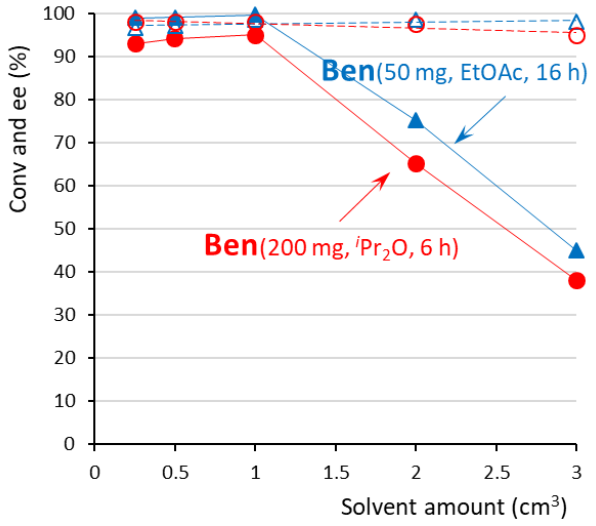


Fig. S4 Effect of the solvent amount on the Conv (closed symbols) and ee (open symbols) in the addition isobutyraldehyde (**1a**) to *N*-benzylmaleimide (**2a**) using 50 mg Ben in EtOAc for 16 h (▲, △) and 200 mg Ben in $^iPr_2O$ for 6 h (●, ○). Reaction conditions: L-Phe 0.03 mmol (10 mol%), 1.2 mmol **1a**, 0.3 mmol **2a**, rt.

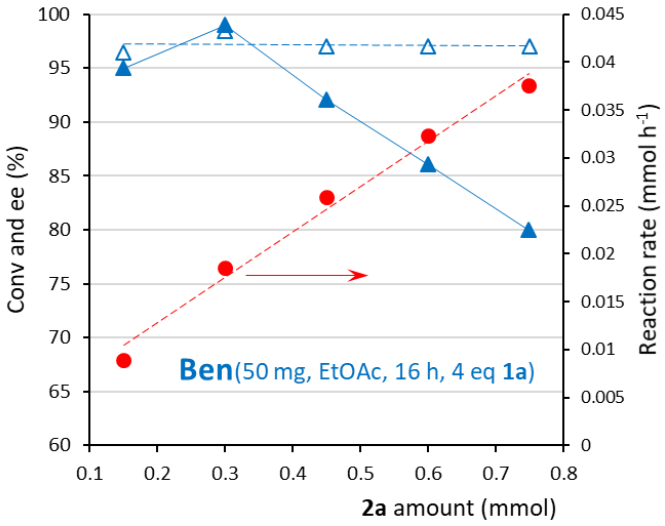


Fig. S5 Effect of the *N*-benzylmaleimide (**2a**) amount on the Conv (▲) and ee (△) using L-Phe 0.03 mmol, 50 mg Ben, 4 eq **1a**, 1 cm^3 EtOAc, 16 h, rt; red data points (●) are the calculated reaction rates.

5. Structure of the amino acids tested

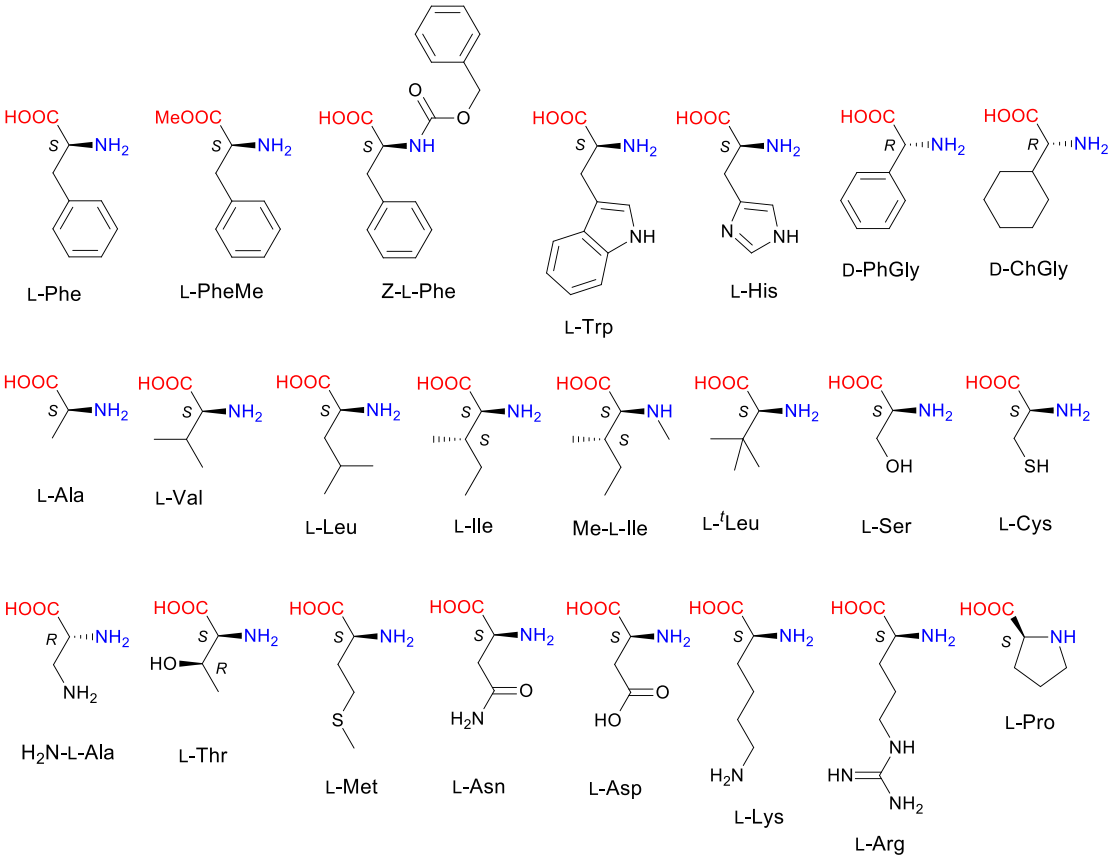


Fig. S6 Structure of the amino acids and amino acid derivatives tested in the addition of isobutyraldehyde (**1a**) to *N*-benzylmaleimide (**2a**).

6. Structure of the carbonyl compounds used as nucleophiles

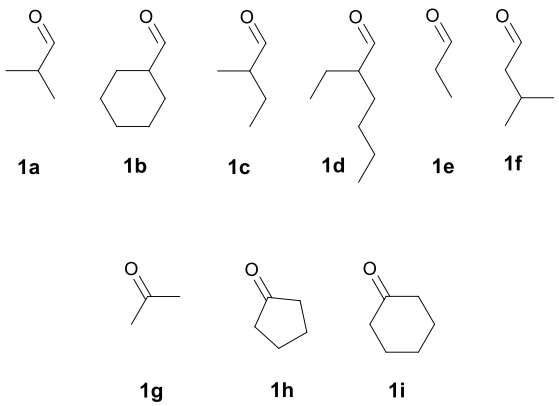


Fig. S7 Structure of the aldehydes and ketones used as nucleophiles.

7. Results obtained in the addition of isobutyraldehyde to *N*-benzylmaleimide using various amino acids

Table S1 Michael addition of **1a** to **2a** catalysed by amino acids in the presence of Ben[a]

Entry	Amino acid	pK_1; pK_2 ($pK_{side\text{-}group}$)	Conv (%)[b]	ee (%) (*config.*)[b]
1[c]	L-Phe	2.20; 9.31	>99	98 (*S*)
2	L-Phe	2.20; 9.31	94	97 (*S*)
3	L-PheMe[d]		10	13 (*S*)
4	Z-L-Phe		<1	nd
5	L-Trp	2.46; 9.41	91	95 (*S*)
6	L-His	1.80; 9.33 (6.04)	25	78 (*S*)
7[c]	D-PhGly		20	92 (*R*)
8	D-PhGly		25	90 (*R*)
9	D-ChGly		36	92 (*R*)
10	L-Ala	2.35; 9.87	25	71 (*S*)
11	L-Val	2.39; 9.74	53	96 (*S*)
12	L-Leu	2.33; 9.74	63	92 (*S*)
13	L-Ile	2.32; 9.76	66	96 (*S*)
14	Me-L-Ile		6	12 (*R*)
15[c]	L-^{t}Leu		72	97 (*S*)
16	L-^{t}Leu		73	97 (*S*)
17	L-Ser	2.19; 9.21	10	60 (*S*)
18	L-Cys	1.92; 10.70 (8.37)	30	95 (*S*)
19	H_2N-D-Ala[d]		19	24 (*R*)
20	L-Thr	2.09; 9.10	4	61 (*S*)
21	L-Met	2.13; 9.28	70	90 (*S*)
22	L-Asn	2.14; 8.72	6	56 (*S*)
23	L-Asp	1.99; 9.90 (3.90)	1	5 (*S*)
24	L-Lys[d]	2.16; 9.06 (10.54)	72	24 (*S*)
25	L-Arg[d]	1.82; 8.99 (12.48)	23	62 (*S*)
26	L-Pro	1.95; 10.64	3	2 (*S*)

[a] Reaction conditions: 0.03 mmol amino acid, 0.3 mmol **2a**, 1.2 mmol **1a**, 50 mg Ben, 1 cm^3 EtOAc, 50°C, 6 h; nd: not determined. [b] Conversion, ee and configuration of the excess enantiomer determined by GC. [c] 16 h reaction at rt. [d] Used as HCl salt.

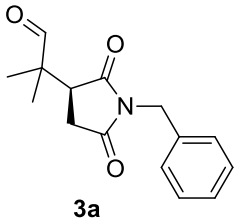
Table S2 Michael addition of **1a** to **2a** catalysed by amino acids in the presence of Al_2O_3[a]

Entry	Amino acid	Conv (%)[b]	ee (%) (*config.*)[b]
1	L-Phe	99	99 (*S*)
2	L-Trp	99	97 (*S*)
3	L-His	79	73 (*S*)
4	D-PhGly	59	96 (*R*)
5	D-ChGly	67	96 (*R*)
6	L-Ala	79	86 (*S*)
7	L-Val	84	98 (*S*)
8	L-Leu	77	94 (*S*)
9	L-Ile	86	98 (*S*)
10	L-^{t}Leu	88	98 (*S*)
11	L-Cys	44	89 (*S*)
12	H_2N-D-Ala[c]	41	16 (*R*)
13	L-Met	96	90 (*S*)
14	L-Lys[c]	99	28 (*S*)
15	L-Arg[c]	26	57 (*S*)

[a] Reaction conditions: 0.03 mmol amino acid, 0.3 mmol **2a**, 1.2 mmol **1a**, 100 mg Al_2O_3, 1 cm^3 EtOAc, rt, 16 h. [b] Conversion, ee and configuration of the excess enantiomer determined by GC. [c] Used as HCl salt.

8. Analytical data of the Michael adducts

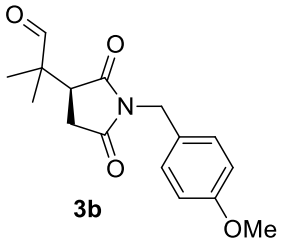
(*S*)-2-(1-benzyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3a**),



3a

Table 4, entry 1: flash chromatography, eluted with hexane/ethyl acetate 4/1, 72 mg white solid, yield 92 %, ee 98.5 %.
Fig. 13, 6 mmol scale: flash chromatography, eluted with hexane/ethyl acetate 4/1, 1.35 g white solid, yield 87 %, ee 97 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.48 (s, 1H, CH=O), 7.36-7.28 (m, 5H, Ar-H), 4.64 (q, 2H, *J* 14.1 Hz), 3.02 (dd, 1H, *J* 5.4, 9.4 Hz), 2.81 (dd, 1H, *J* 9.4, 18.3 Hz), 2.44 (dd, 1H, *J* 5.4, 18.3 Hz), 1.16 (s, 6H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 175.3, 135.6, 128.7, 128.6, 127.9, 48.0, 44.9, 42.4, 31.4, 19.9, 19.1.
GC-MSD *m/z*(rel. int.): *259*(M^+, 1), *231*(100), *216*(48), *189*(13), *138*(30), *106*(21), *91*(80), *83*(16), *69*(30), *55*(8), *41*(13).
GC-FID analysis: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 165°C - 95 min, 5°C/min to 180°C - 9 min, total time 202 min; retention times: **2a** 55.6 min, *R*-**3a** 174.3 min, *S*-**3a** 179.6 min.

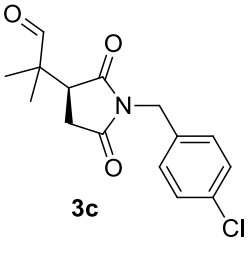
(*S*)-2-(1-(4-methoxybenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3b**),



3b

Table 4, entry 2: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 1/1, 78 mg white solid, yield 90 %, ee 97.5 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.49 (s, 1H, CH=O), 7.30 (d, 2H, *J* 8.6 Hz, Ar-H), 6.82 (d, 2H, *J* 8.7 Hz, Ar-H), 4.58 (q, 2H, *J* 14.0 Hz), 3.78 (s, 3H, CH_3), 3.01 (dd, 1H, *J* 5.4, 9.4 Hz), 2.79 (dd, 1H, *J* 9.4, 18.3 Hz), 2.42 (dd, 1H, *J* 5.4, 18.3 Hz), 1.15 (d, 6H, *J* 5.8 Hz, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.3, 175.3, 159.4, 130.2, 128.0, 114.0, 55.2, 47.9, 45.0, 41.9, 31.5, 19.8, 19.1.
GC-MSD *m/z*(rel. int.): *289*(M^+, 5), *261*(8), *162*(11), *136*(8), *121*(100), *77*(4), *69*(4), *55*(3), *41*(3).
GC-FID analysis: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 110°C - 10 min, 1°C/min to 165°C - 5 min, 5°C/min to 180°C - 67 min, 5°C/min to 190°C - 108 min, total time 250 min; retention times: **2b** 74.1 min, *R*-**3b** 196.7 min, *S*-**3b** 201.4 min.

(*S*)-2-(1-(4-chlorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3c**),

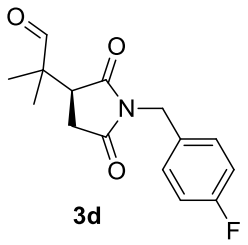


3c

Table 4, entry 3: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 2/1, 78 mg white solid, yield 88 %, ee 97.5 %.
Fig. 13, 3 mmol scale: crystallized in hexane/ethyl acetate, 0.69 g pale yellow solid, yield 78 %, ee 97 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.46 (s, 1H, CH=O), 7.32-7.26 (m, 4H, Ar-H), 4.60 (q, 2H, *J* 14.2 Hz), 3.00 (dd, 1H, *J* 5.5, 9.4 Hz), 2.81 (dd, 1H, *J* 9.4, 18.3 Hz), 2.44 (dd, 1H, *J* 5.5, 18.3 Hz), 1.17 (d, 6H, *J* 12.3 Hz, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.4, 175.2, 134.1, 133.9, 130.2, 128.9, 128.8, 44.9, 41.7, 31.6, 20.0, 19.3.
GC-MSD *m/z*(rel. int.): *293*(M^+, 1), *265*(74), *250* (33), *223* (12), *138*(35), *125*(100), *83*(20), *69*(29), *55*(9), *41*(13).

GC-FID analysis: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 110°C - 10 min, 1°C/min to 165°C - 70 min, 5°C/min to 180°C - 67 min, 5°C/min to 190°C - 53 min, total time 195 min; retention times: **2c** 72.6 min, *R*-**3c** 178.1 min, *S*-**3c** 181.4 min.

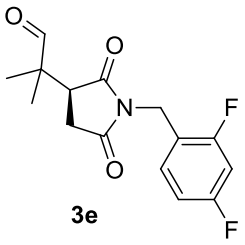
(*S*)-2-(1-(4-fluorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3d**),



3d

Table 4, entry 4: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 1/1, 75 mg white solid, yield 90 %, ee 97.5 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.47 (s, 1H, CH=O), 7.36 (m, 2H, Ar-H), 6.98 (m, 2H, Ar-H), 4.61 (q, 2H), 3.01 (dd, 1H), 2.81 (dd, 1H), 2.44 (dd, 1H), 1.17 (d, 6H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.4, 175.3, 163.4, 131.5, 130.8, 130.7, 130.6, 115.7, 115.6, 115.5, 115.4, 48.1, 44.9, 41.7, 31.5, 20.0, 19.3.
GC-MSD *m/z*(rel. int.): *277*(M^+, 1), *249*(72), *234*(24), *207*(10), *138*(23), *124*(16), *109*(100), *83*(20), *69*(32), *55*(7), *41*(10).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 165°C - 95 min, 5°C/min to 180°C - 9 min, total time 202 min; retention times: **2d** 59.8 min, *R*-**3d** 187.8 min, *S*-**3d** 192.4 min.

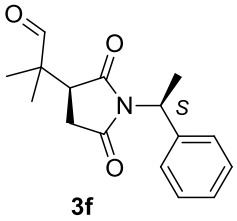
(*S*)-2-(1-(2,4-difluorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3e**),



3e

Table 4, entry 5: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 2/1, 81 mg white solid, yield 91 %, ee 97 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.46 (s, 1H, CH=O), 7.33 (m, 1H, Ar-H), 6.81 (d, 2H, Ar-H), 4.69 (s, 2H), 3.00 (dd, 1H), 2.82 (dd, 1H), 2.47 (dd, 1H), 1.21 (d, 6H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.2, 175.0, 131.6, 131.5, 111.5, 11.5, 111.3, 104.2, 104.0, 103.8, 48.1, 44.9, 35.7, 31.6, 20.1, 19.5.
GC-MSD *m/z*(rel. int.): *295*(M^+, 1), *267*(100), *252*(48), *225*(13), *138*(30), *127*(21), *83*(16), *69*(30), *55*(8), *41*(13).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 165°C - 95 min, 5°C/min to 180°C - 9 min, total time 202 min; retention times: **2e** 49.2 min, *R*-**3e** 128.5 min, *S*-**3e** 130.6 min.

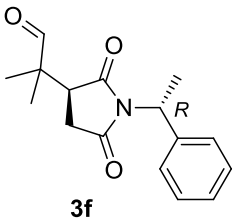
(*S*)-2-((*S*)-1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal ((*,*S*)-**3f**),



3f

Table 4, entry 7: flash chromatography, eluted with hexane/ethyl acetate 6/1, 65.6 mg white solid, yield 80 %, ee 98 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.47 (s, 1H, CH=O), 7.41 (d, 2H, Ar-H), 7.30 (m, 2H, Ar-H), 7.25 (m, 1H, Ar-H), 5.40 (q, 1H, *J* 7,2 Hz), 2.97 (dd, 1H, *J* 5.4, 9.5 Hz), 2.73 (dd, 1H, *J* 9.5, 18.3 Hz), 2.40 (dd, 1H, *J* 5.4, 18.3 Hz), 1.79 (d, 3H, *J* 7.3 Hz, CH_3), 1.11 (d, 6H, *J* 5.3 Hz, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.4, 175.4, 139.5, 128.4, 127.8, 127.4, 50.4, 48.0, 44.7, 31.3, 19.9, 18.8, 16.5.
GC-MSD *m/z*(rel. int.): *273*(M^+, 1), *245*(28), *160* (14), *141* (28), *126*(59), *105*(100), *77*(21), *69*(15), *55*(8), *41*(12).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 158°C - 112 min, 4°C/min to 180°C - 29.5 min, total time 235 min; retention times: *S*-**2f** 55.2 min, (*S,S*)-**3f** 211.4 min, (*R,S*)-**3f** 212.1 min.

(*S*)-2-((*R*)-1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal ((*,*R*)-**3f**), Table 4, entry 8: flash chromatography, eluted with hexane/ethyl acetate 6/1, 64.0 mg white solid, yield 78 %, ee 98 %.



3f

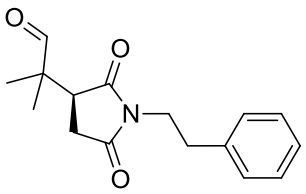
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.49 (s, 1H, CH=O), 7.42 (m, 2H, Ar-H), 7.32 (m, 2H, Ar-H), 7.27 (m, 1H, Ar-H), 5.42 (q, 1H, *J* 7.3 Hz), 2.94 (dd, 1H, *J* 5.4, 9.5 Hz), 2.76 (dd, 1H, *J* 9.5, 18.3 Hz), 2.40 (dd, 1H, *J* 5.4, 18.3 Hz), 1.79 (d, 3H, *J* 7.3Hz, CH_3), 1.16 (d, 6H, *J* 5.1 Hz, CH_3).

^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.7, 177.5, 175.4, 139.3, 128.4, 127.8, 127.5, 50.2, 48.1, 44.6, 31.3, 20.0, 18.9, 16.3.

GC-MSD *m/z*(rel. int.): *273*(M^+, 3), *245*(57), *160* (19), *141* (36), *126*(68), *105*(100), *77*(20), *69*(13), *55*(6), *41*(9).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 158°C - 112 min, 4°C/min to 180°C - 29.5 min, total time 235 min; retention times: *R*-**2f** 56.7 min, (*R,R*)-**3f** 208.9 min, (*S,R*)-**3f** 214.1 min.

(*S*)-2-(1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3g**), Table 4, entry 9: crystallization in hexane/ethyl acetate 10/1, 72.2 mg white solid, yield 88 %; ee 95.5 %.



3g

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.47 (s, 1H, CH=O), 7.28-7.2 (m, 5H, Ar-H), 3.75 (m, 2H), 2.96 (dd, 1H, *J* 5.5, 9.3 Hz), 2.90 (tr, 2H, *J* 7.1 Hz), 2.73 (dd, 1H, *J* 9.4, 18.2 Hz), 2.36 (dd, 1H, *J* 5.5, 18.2 Hz), 1.13 (s, 6H, CH_3).

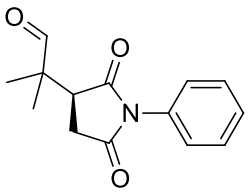
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.4, 175.4, 137.7, 128.8, 128.5, 126.7, 47.8, 44.9, 39.9, 33.4, 31.3, 19.8, 19.0.

GC-MSD *m/z*(rel. int.): *273*(M^+, 1), *245*(41), *230*(20), *104*(100), *91*(14), *69*(10), *55*(4), *41*(6).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 165°C - 72 min, 5°C/min to 180°C - 45 min, total time 215 min; retention times: **2g** 64.1 min, (*R*)-**3g** 200.4 min, (*S*)-**3g** 202.8 min.

(*S*)-2-(1-phenyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3h**), Table 4, entry 10: crystallization in hexane/ethyl acetate 10/1, 66.2 mg white solid, yield 90 %; ee 97 %.

Fig. 13, 6 mmol scale: crystallization in hexane/ethyl acetate, 1.18 g off-white solid, yield 80 %, ee 97 %.



3h

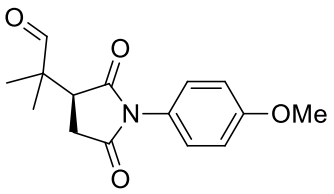
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.49 (s, 1H, CH=O), 7.44 (tr, 2H, *J* 7.2 Hz, Ar-H), 7.36 (tr, 1H, *J* 7.4, Ar-H), 7.25 (d, 2H, *J* 8.7 Hz, Ar-H), 3.12 (m, 1H), 2.94 (m, 1H), 2.58 (m, 1H), 1.28 (d, 6H, *J* 15.9 Hz, CH_3).

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 202.7, 176.8, 174.7, 131.8, 129.1, 128.7, 126.5, 48.5, 45.0, 31.8, 20.2, 19.6.

GC-MSD *m/z*(rel. int.): *245*(M^+, 1), *217*(75), *202* (100), *175*(13), *147*(18), *119*(18), *93*(40), *83*(52), *69*(33), *55*(12), *41*(23).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 165°C - 102 min, 5°C/min to 180°C - 10 min, total time 210 min; retention times: **2h** 54.0 min, (*R*)-**3h** 198.5 min, (*S*)-**3h** 200.5 min.

(*S*)-2-(1-(4-methoxyphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3i**),



3i

Table 4, entry 11: flash chromatography, eluted with hexane/ethyl acetate 3/1, 70.2 mg off-white solid, yield 85 %, ee 89 %.

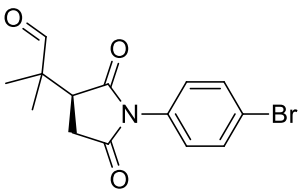
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.53 (s, 1H, CH=O), 7.19 (m, 2H), 6.97 (m, 2H), 3.82 (s, 3H, CH_3), 3.13 (dd, 1H), 2.95 (dd, 1H), 2.60 (dd, 1H), 1.31 (s, 6H, CH_3).

^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.0, 174.9, 159.7, 127.7, 114.6, 55.5, 48.4, 45.1, 31.8, 20.2, 19.6.

GC-MSD *m/z*(rel. int.): *275*(M^+, 21), *247*(19), *232* (55), *177*(32), *149*(19), *134*(26), *123*(100), *108*(32), *69*(33), *55*(19), *41*(26).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 110°C - 10 min, 1°C/min to 165°C - 5 min, 5°C/min to 180°C - 67 min, 5°C/min to 190°C - 108 min, total time 250 min; retention times: **2i** 73.0 min, (*R*)-**3i** 217.1 min, (*S*)-**3i** 221.1 min.

(*S*)-2-(1-(4-bromophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal, (**3j**),



3j

Table 4, entry 12: flash chromatography, eluted with hexane/ethyl acetate 3/1, 87.5 mg pale yellow solid, yield 90 %, ee 95 %.

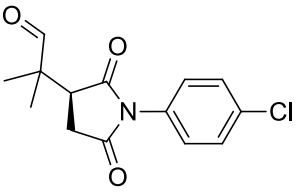
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.47 (s, 1H, CH=O), 7.58 (m, 2H), 7.18 (m, 2H), 3.10 (dd, 1H), 2.95 (dd, 1H), 2.59 (dd, 1H), 1.30 (d, 6H, CH_3).

^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 176.5, 174.3, 132.3, 130.8, 128.0, 122.5, 48.7, 45.0, 31.9, 20.5, 19.9.

GC-MSD *m/z*(rel. int.): *324*(M^+, 1), *223*(5), *297*(51), *295*(55), *282*(94), *280*(100), *253*(13), *225*(22), *197*(17), *171*(42), *83*(93), *69*(45), *55*(19), *41*(29).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 110°C - 10 min, 1°C/min to 165°C - 5 min, 5°C/min to 180°C - 67 min, 5°C/min to 190°C - 108 min, total time 250 min; retention times: **2j** 78.2 min, (*R*)-**3j** 233.9 min, (*S*)-**3j** 239.1 min.

(*S*)-2-(1-(4-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3k**),



3k

Table 4, entry 13: flash chromatography, eluted with hexane/ethyl acetate 4/1, 73.8 mg pale yellow solid, yield 88 %, ee 95 %.

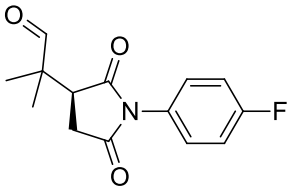
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.49 (s, 1H, CH=O), 7.43 (m, 2H, Ar-H), 7.25 (m, 2H, Ar-H), 3.11 (dd, 1H), 2.96 (dd, 1H), 2.62 (dd, 1H), 1.31 (d, 6H, CH_3).

^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 176.5, 174.3, 134.5, 130.4, 129.3, 127.8, 48.6, 45.1, 32.0, 20.4, 19.9.

GC-MSD *m/z*(rel. int.): *279*(M^+, 5), *251*(55), *236* (100), *209*(15), *181*(24), *153*(17), *127*(38), *83*(68), *69*(32), *55*(13), *41*(20).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 110°C - 10 min, 1°C/min to 165°C - 5 min, 5°C/min to 180°C - 97 min, 5°C/min to 190°C - 38 min, total time 210 min; retention times: **2k** 67.4 min, (*R*)-**3k** 193.5 min, (*S*)-**3k** 196.8 min.

(*S*)-2-(1-(4-fluorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3l**),



3l

Table 4, entry 14: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 1/1.5, 69.5 mg off-white solid, yield 88 %, ee 96 %. Fig. 13, 3 mmol scale: crystallization in toluene, 0.65 g light beige solid, yield 82 %, ee 96 %.

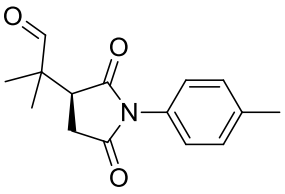
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.50 (s, 1H, CH=O), 7.25 (m, 2H, Ar-H), 7.16 (m, 2H, Ar-H), 3.11 (dd, 1H), 2.97 (dd, 1H), 2.61 (dd, 1H), 1.31 (d, 6H, CH_3).

^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.7, 176.8, 174.6, 163.3, 128.4, 128.3, 116.3, 116.1, 48.6, 44.9, 31.9, 20.4, 19.9.

GC-MSD *m/z*(rel. int.): *263*(M^+, 3), *235*(65), *220* (100), *193*(14), *165*(23), *137*(18), *111*(33), *83*(60), *69*(29), *55*(11), *41*(17).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 165°C - 95 min, 5°C/min to 180°C - 9 min, total time 202 min; retention times: **2l** 55.0 min, (*R*)-**3l** 194.0 min, (*S*)-**3l** 196.5 min.

(*S*)-2-(1-(4-methylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3m**),



3m

Table 4, entry 15: flash chromatography, eluted with hexane/ethyl acetate 3/1, 68.5 mg off-white solid, yield 88 %, ee 96 %.

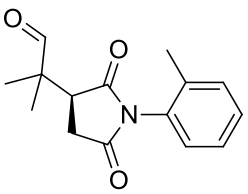
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.52 (s, 1H, CH=O), 7.25 (m, 2H), 7.13 (m, 2H), 3.13 (dd, 1H), 2.95 (dd, 1H), 2.60 (dd, 1H), 2.37 (s, 3H, CH_3), 1.29 (d, 6H, CH_3).

^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 176.8, 174.8, 138.8, 129.8, 129.2, 126.3, 48.4, 45.1, 31.8, 21.2, 20.2, 19.6.

GC-MSD *m/z*(rel. int.): *359*(M^+, 9), *231*(64), *216*(100), *188*(12), *161*(25), *107*(57), *83*(25), *69*(18), *55*(7), *41*(13).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 110°C - 10 min, 1°C/min to 165°C - 5 min, 5°C/min to 180°C - 97 min, 5°C/min to 190°C - 38 min, total time 210 min; retention times: **2m** 56.2 min, (*R*)-**3m** 176.0 min, (*S*)-**3m** 178.9 min.

(*S*)-2-(1-(2-methylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3n**),



3n

Table 4, entry 16: flash chromatography, eluted with hexane/ethyl acetate 3/1, 62.2 mg off-white solid, yield 80 %, ee 96 %.

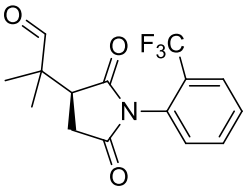
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.46 (d, 1H, CH=O), 7.27-7.20 (m, 3H, Ar-H), 6.97 (m, 1H, Ar-H), 3.11 (m, 1H), 2.92 (m, 1H), 2.61 (m, 1H), 2.12 (d, 3H, CH_3), 1.29 (d, 6H, CH_3).

^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 202.5, 176.7, 174.6, 174.5, 135.9, 135.5, 131.2, 131.0, 129.5, 128.0, 126.9, 126.8, 48.5, 48.1, 45.5, 45.2, 32.1, 31.9, 20.4, 20.3, 19.8, 17.8, 17.6.

GC-MSD *m/z*(rel. int.): *359*(M^+, 3), *231*(59), *216*(100), *188*(9), *161*(18), *107*(45), *83*(37), *69*(26), *55*(9), *41*(18).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 110°C - 10 min, 1°C/min to 165°C - 25 min, 5°C/min to 175°C - 68 min, 5°C/min to 190°C - 27 min, total time 190 min; retention times: **2n** 46.7 min, (*R*)-**3n** 153.1 min, (*S*)-**3n** 155.5 min.

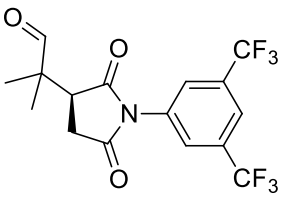
(*S*)-2-(1-(2-trifluoromethylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3o**),



3o

Table 4, entry 17: flash chromatography, eluted with hexane/ethyl acetate 4/1, 75.2 mg off-white solid, yield 80 %, ee 95 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.59-9.46 (m, 1H, CH=O), 7.81-7.57 (m, 3H), 7.34-7.19 (m, 1H), 3.38 (m, 0.45H), 3.08 (m, 0.55H), 3.01 (m, 1H), 2.65 (m, 1H), 1.34-1.22 (m, 6H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.7, 176.7, 176.1, 174.3, 133.3, 130.9, 130.8, 130.1, 130.0, 127.6, 127.5, 127.4, 45.9, 45.2, 32.5, 31.7, 29.7, 20.8, 20.5, 19.4, 18.9.
GC-MSD *m/z*(rel. int.): *313*(M^+, 1), *285*(84), *270*(28), *250*(100), *222*(29), *168*(21), *83*(97), *69*(98), *55*(21), *41*(37).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 110°C - 10 min, 1°C/min to 155°C - 15 min, 5°C/min to 180°C - 50 min, 5°C/min to 190°C - 8 min, total time 135 min; retention times: **2o** 39.0 min, (*R*)-**3o** 104.5 min, (*S*)-**3o** 106.0 min.

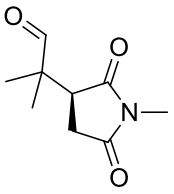
(*S*)-2-(1-(3,5-ditrifluoromethylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3p**),



3p

Table 4, entry 18: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 2/1, 100.6 mg off-white solid, yield 88 %, ee 95 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.46 (s, 1H, CH=O), 7.88 (m, 3H), 3.10 (dd, 1H), 3.01 (dd, 1H), 2.68 (dd, 1H), 1.40 (d, 6H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 176.1, 173.7, 133.4, 132.7, 132.5, 126.7, 123.8, 122.3, 122.2, 49.1, 45.0, 32.2, 20.8, 20.5.
GC-MSD *m/z*(rel. int.): *381*(M^+, 0), *362*(15), *353*(26), *338*(43), *83*(100), *69*(70), *55*(25), *41*(31).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 95 min, 1.5°C/min to 180°C - 5 min, total time 180 min; retention times: **2p** 42.5 min, (*R*)-**3p** 171.6 min, (*S*)-**3p** 172.4 min.

(*S*)-2-methyl-2-(1-methyl-2,5-dioxopyrrolidin-3-yl)propanal (**5a**),

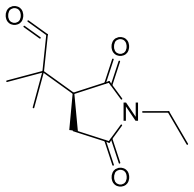


5a

Table 5, entry 1: flash chromatography, eluted with hexane/EtOAc 5/1; 38.5 mg pale yellow oil, yield 70 %, ee 95 %.
^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 9.47 (s, 1H, CH=O), 3.01 (dd, 1H, *J* 5.4, 9.3 Hz), 2.94 (s, 1H, CH_3), 2.78 (dd, 1H, *J* 9.3, 18.3 Hz), 2.42 (dd, 1H, *J* 5.4, 18.3 Hz), 1.17 (d, 6H, *J* 5.2 Hz, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 202.7, 177.8, 175.8, 47.9, 45.0, 31.4, 24.8, 20.0, 19.2.
GC-MSD *m/z*(rel. int.): *183*(M^+, 2), *155*(41), *140*(66), *113*(61), *83*(30), *69*(100), *55*(13), *41*(33).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 60°C - 5 min, 2°C/min to 120°C - 15 min, 2°C/min to 165°C - 12.5 min, 3°C/min to 186°C - 13 min, total time 105 min; retention times: **4a** 12.9 min, (*R*)-**5a** 92.1 min, (*S*)-**5a** 92.9 min.

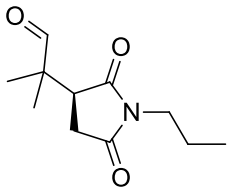
(*S*)-2-(1-ethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5b**),
Table 5, entry 2: flash chromatography, eluted with hexane/EtOAc 4/1; 48.5 mg pale yellow oil, yield 82 %, ee 95 %.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.49 (s, 1H, CH=O), 3.53 (q, 2H, *J* 7.2 Hz), 3.00 (dd, 1H, *J* 5.3, 9.4 Hz), 2.77 (dd, 1H, *J* 9.4, 18.3 Hz), 2.40 (dd, 1H, *J* 5.3, 18.3 Hz) 1.19 (d, 6H, *J* 4.0 Hz, CH_3), 1.14 (tr, 3H, *J* 7.2 Hz, CH_3).
^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 202.7, 177.5, 175.5, 47.9, 44.9, 33.7, 31.4, 19.9, 19.0, 12.8.
GC-MSD *m/z*(rel. int.): *197*(M^+, 3), *169*(30), *154*(59), *127*(55), *83*(24), *69*(100), *55*(13), *41*(29).


5b

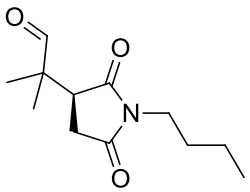
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 60°C - 5 min, 2°C/min to 120°C - 15 min, 2°C/min to 170°C - 10 min, 4°C/min to 190°C - 10 min, total time 100 min; retention times: **4b** 25.4 min, (*R*)-**5b** 79.7 min, (*S*)-**5b** 81.3 min.

(*S*)-2-(1-propyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5c**),
Table 5, entry 3: flash chromatography, eluted with hexane/EtOAc 3/1; 47.5 mg transparent oil, yield 75 %, ee 97 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.51 (s, 1H, CH=O), 3.46 (tr, 2H, *J* 7.3 Hz), 3.01 (dd, 1H, *J* 5.4, 9.4 Hz), 2.80 (dd, 1H, *J* 9.4, 18.3 Hz), 2.43 (dd, 1H, *J* 5.4, 18.3 Hz) 1.59 (m, 2H, CH_2), 1.20 (d, 6H, *J* 5.1 Hz, CH_3), 1.20 (tr, 3H, *J* 7.4 Hz, CH_3).


5c

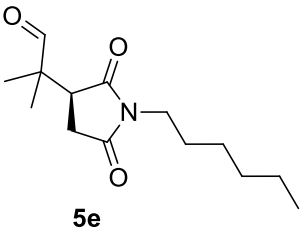
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.7, 177.7, 175.8, 44.9, 40.4, 31.4, 20.9, 20.0, 19.1, 11.2.
GC-MSD *m/z*(rel. int.): *211*(M^+, 5), *183*(45), *168*(100), *141*(71), *126*(18), *83*(28), *69*(99), *55*(16), *41*(30).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 60°C - 5 min, 2°C/min to 120°C - 15 min, 2°C/min to 170°C - 11 min, 5°C/min to 185°C - 15 min, total time 108 min; retention times: **4c** 29.6 min, (*S*)-**5c** 82.4 min, (*R*)-**5c** 84.3 min.

(*S*)-2-(1-butyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5d**),
Table 5, entry 4: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 2/1; 47.3 mg transparent oil, yield 70 %, ee 97 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.52 (s, 1H, CH=O), 3.50 (tr, 1H, *J* 7.4 Hz, CH), 3.00 (dd, 1H, *J* 5.4, 9.4 Hz), 2.78 (dd, 1H, *J* 9.4, 18.2 Hz), 2.42 (dd, 1H, *J* 5.4, 18.2 Hz), 1.56 (m, 2H, CH_2), 1.31 (m, 2H, CH_2), 1.21 (s, 6H, CH_3), 0.93 (tr, 3H, *J* 7.4 Hz, CH_3).


5d

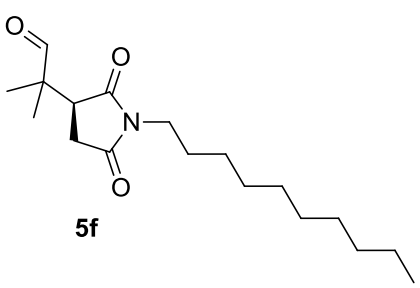
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.7, 175.7, 47.9, 45.0, 38.7, 31.4, 29.7, 20.0, 20.0, 19.1, 13.5.
GC-MSD *m/z*(rel. int.): *225*(M^+, 5), *197*(40), *182*(100), *155*(49), *126*(21), *83*(23), *69*(89), *55*(17), *41*(28).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 160°C, 10°C/min to 180°C - 33 min, total time 125 min; retention times: **4d** 16.6 min, (*R*)-**5d** 82.5 min, (*S*)-**5d** 83.7 min.

(*S*)-2-(1-hexyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5e**),
Table 5, entry 5: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 2/1; 60.8 mg transparent oil, yield 80 %, ee 96 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.52 (s, 1H, CH=O), 3.49 (tr, 2H, *J* 7.5 Hz), 3.01 (dd, 1H, *J* 5.4, 9.3 Hz), 2.78 (dd, 1H, *J* 9.4, 18.2 Hz), 2.42 (dd, 1H, *J* 5.4, 18.2 Hz), 1.56 (m, 2H, CH_2), 1.29 (s, 6H, CH_2), 1.20 (s, 6H, CH_3), 0.88 (m, 3H, CH_3).


5e

^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.6, 175.7, 47.9, 45.0, 39.0, 31.4, 31.3, 27.6, 26.5, 22.4, 20.0, 19.1, 13.9.
GC-MSD *m/z*(rel. int.): *253*(M^+, 23), *254*(22), *210*(66), *183*(32), *126*(22), *83*(21), *69*(100), *55*(25), *41*(39).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 160°C - 40 min, 8°C/min to 184°C - 7 min, total time 140 min; retention times: **4e** 35.9 min, (*R*)-**5e** 124.0 min, (*S*)-**5e** 126.6 min.

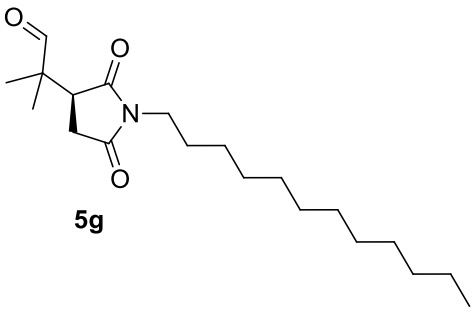
(*S*)-2-(1-decyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5f**),
Table 5, entry 6: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 4/1; 74.3 mg transparent oil, yield 80 %, ee 95 %.
Fig. 13, 3 mmol scale: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 4/1, 0.84 g pale yellow oil solid, yield 90 %, ee 93 %.



5f

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.51 (s, 1H, CH=O), 3.47 (tr, 2H), 3.01 (dd, 1H), 2.78 (dd, 1H), 2.42 (dd, 1H), 1.54 (m, 2H, CH_2), 1.25 (m, 14H, CH_2), 1.19 (d, 6H, CH_3), 0.87 (tr, 3H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.7, 177.7, 175.8, 47.9, 44.9, 38.9, 31.8, 31.3, 29.5, 29.4, 29.2, 29.1, 27.6, 26.8, 22.6, 20.0, 19.0, 14.1.
GC-MSD *m/z*(rel. int.): *309*(M^+, 5), *280*(66), *266*(100), *238*(15), 1*83*(26), *69*(57), *55*(14), *41*(19).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 150°C - 10 min, 5°C/min to 185°C - 83 min, 5°C/min to 190°C - 9 min, total time 110 min; retention times: **4f** 16.8 min, (*S*)-**5f** 85.5 min, (*R*)-**5f** 89.3 min.

(*S*)-2-(1-dodecyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5g**),
Table 5, entry 7: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 2/1; 86.1 mg transparent oil, yield 85 %, ee 96 %.

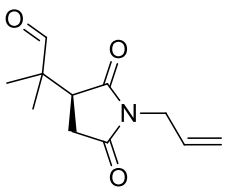


5g

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.52 (s, 1H, CH=O), 3.48 (tr, 2H, *J* 7.3 Hz), 3.01 (dd, 1H, *J* 5.4, 9.43 Hz), 2.78 (dd, 1H, *J* 9.4, 18.2 Hz), 2.42 (dd, 1H, *J* 5.4, 18.2 Hz), 1.56 (m, 2H, CH_2), 1.29-1.26 (m, 18H, CH_2), 1.20 (s, 6H, CH_3), 0.88 (m, 3H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.6, 175.7, 47.9, 45.0, 39.0, 31.9, 31.4, 29.6, 29.5, 29.3, 29.1, 27.6, 26.8, 22.7, 20.0, 19.1, 14.1.
GC-MSD *m/z*(rel. int.): *337*(M^+, 6), *308*(73), *294*(100), *266*(16), *183*(28), *126*(16), *69*(58), *55*(15), *41*(18).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 150°C - 10 min, 5°C/min to 185°C - 83 min, 5°C/min to 195°C - 78 min, total time 180 min; retention times: **4g** 24.1 min, (*S*)-**5g** 139.9 min, (*R*)-**5g** 143.5 min.

(*S*)-2-(1-allyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5h**),

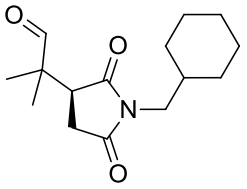
Table 5, entry 8: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 3/1, 54.0 mg transparent oil, yield 86 %, ee 96 %.



5h

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.50 (s, 1H, CH=O), 5.77 (m, 1H, CH), 5.21 (dd, 2H, *J* 17.1, 30.1 Hz), 4.10 (d, 2H, *J* 5.9 Hz), 3.03 (dd, 1H, *J* 5.5, 9.4 Hz), 2.81 (dd, 1H, *J* 9.4, 18.3 Hz), 2.46 (dd, 1H, *J* 5.5, 18.3 Hz), 1.22 (d, 6H, *J* 4.0 Hz, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.2, 175.1, 130.6, 118.5, 48.0, 45.0, 40.9, 31.5, 20.0, 19.3.
GC-MSD *m/z*(rel. int.): *209*(M^+, 1), *181*(70), *166*(80), *139*(24), *109*(63), *83*(63), *69*(100), *55*(23), *41*(40).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 60°C - 5 min, 2°C/min to 120°C - 15 min, 2°C/min to 170°C - 11 min, 5°C/min to 185°C - 15 min, total time 108 min; retention times: **4h** 31.5 min, (*S*)-**5h** 83.4 min, (*R*)-**5h** 85.8 min.

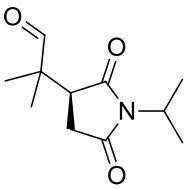
(*S*)-2-(1-cyclohexylmethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5i**),
Table 5, entry 9: crystallization in hexane/ethyl acetate 10/1, 63.7 mg white solid, yield 80 %, ee 96 %.



5i

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.52 (s, 1H, CH=O), 3.33 (d, 2H, *J* 7.2 Hz), 3.02 (dd, 1H, *J* 5.7, 9.3 Hz), 2.79 (dd, 1H, *J* 9.4, 18.2 Hz), 2.44 (dd, 1H, *J* 5.6, 18.2 Hz), 1.71-1.60 (m, 6H, CH_2), 1.21-1.17 (m, 8H), 0.94 (m, 2H, CH_2).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.7, 177.9, 175.9, 47.8, 45.0, 36.1, 31.3, 30.7, 26.2, 25.6, 20.1, 19.1.
GC-MSD *m/z*(rel. int.): *265*(M^+, 3), *237*(31), *222*(50), *183*(32), *170*(100), *126*(24), *83*(19), *69*(46), *55*(28), *41*(24).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 165°C - 102 min, 5°C/min to 180°C - 10 min, total time 210 min; retention times: **4i** 50.0 min, (*R*)-**5i** 164.6 min, (*S*)-**5i** 168.1 min.

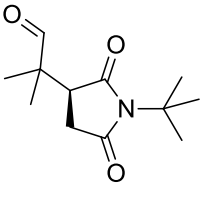
(*S*)-2-(1-isopropyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5j**),
Table 5, entry 10: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 2/1, 53.9 mg transparent oil, yield 85 %, ee 97 %.



5j

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.52 (s, 1H, CH=O), 3.37 (m, 1H, CH), 2.96 (dd, 1H, *J* 5.3, 9.4 Hz), 2.74 (dd, 1H, *J* 9.5, 18.2 Hz), 2.38 (dd, 1H, *J* 5.3, 18.2 Hz), 1.37 (d, 6H, *J* 7.0 Hz CH_3), 1.18 (m, 6H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.7, 177.7, 175.6, 48.0, 44.7, 43.9, 31.3, 20.0, 19.1, 18.8.
GC-MSD *m/z*(rel. int.): *211*(M^+, 4), *183*(39), *168*(41), *141*(57), *126*(53), *99*(23), *83*(23), *69*(100), *55*(16), *41*(31).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 80°C - 10 min, 1.5°C/min to 113°C - 30 min, 0.5°C/min to 137°C - 0 min, 8°C/min to 185°C - 9 min, total time 125 min; retention times: **4j** 17.0 min, (*S*)-**5j** 105.2 min, (*R*)-**5j** 107.8 min.

(*S*)-2-(1-*tert*-butyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5k**),
Table 5, entry 11: flash chromatography, eluted with hexane/EtOAc 6/1, 43.9 mg pale yellow oil, yield 65 %, ee 94 %.

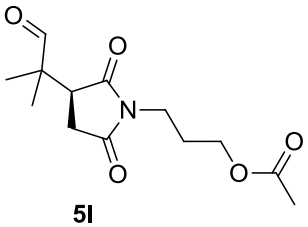


5k

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.54 (s, 1H, CH=O), 2.95 (dd, 1H, *J* 5.4, 9.8 Hz), 2.66 (dd, 1H, *J* 9.8, 18.0 Hz), 2.34 (dd, 1H, *J* 5.4, 18.0 Hz), 1.54 (s, 9H, CH_3), 1.12 (s, 6H, CH_3).

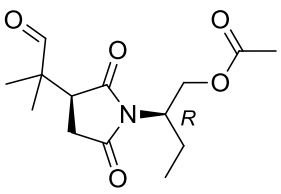
^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 202.9, 178.7, 176.6, 129.3, 127.5, 58.7, 47.8, 45.0, 31.5, 28.3, 20.2, 17.9.
GC-MSD *m/z*(rel. int.): *225*(M^+, 4), *210*(15), *197*(14), *170*(64), *153*(21), *141*(35), *126*(100), *111*(20), *99*(22), *83*(31), *69*(42), *57*(32), *41*(43).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 60°C - 5 min, 2°C/min to 120°C - 15 min, 2°C/min to 170°C - 15 min, 5°C/min to 185°C - 15 min, total time 108 min; retention times: **4k** 31.5 min, (*S*)-**5k** 74.6 min, (*R*)-**5k** 75.3 min.

(*S*)-2-((3-acetoxypropyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5l**),
Table 5, entry 12: flash chromatography, eluted with hexane/EtOAc 1/1, 71.1 mg pale yellow oil, yield 88 %, ee 95 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.47 (s, 1H, CH=O), 4.04 (tr, 2H, *J* 6.3 Hz), 3.58 (tr, 2H, *J* 7.0 Hz), 2.97 (dd, 1H, *J* 5.5, 9.4 Hz), 2.77 (dd, 1H, *J* 9.4, 18.2 Hz), 3.43
(dd, 1H, *J* 5.5, 18.2 Hz), 2.03 (s, 3H, CH_3), 1.91 (m, 2H, CH_2), 1.21 (d, 6H, *J* 15.8 Hz, CH_3).


5l

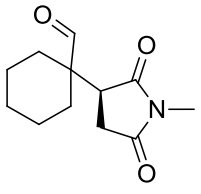
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.6, 177.5, 175.4, 170.8, 61.5, 48.0, 44.9, 35.7, 31.5, 26.7, 20.8, 20.1, 19.4.
GC-MSD *m/z*(rel. int.): *269*(M^+, 0), *209*(15), *180*(71), *166*(100), *139*(25), *83*(17), *69*(39), *55*(10), *41*(25).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 100°C - 5 min, 2°C/min to 150°C - 15 min, 2°C/min to 184°C - 15 min, 1°C/min to 195°C - 42 min, total time 130 min; retention times: **4l** 41.2 min, (*R*)-**5l** 95.7 min, (*S*)-**5l** 97.2 min.

(*S*)-2-(*R*)-N-[1-[(acetyloxy)methyl]propyl]-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5m**),
Table 5, entry 13: flash chromatography, eluted with hexane/EtOAc 1/1, 68.0 mg pale yellow oil, yield 80 %, ee 94 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.53 (s, 1H, CH=O), 4.46 (m, 1H), 4.31 (m, 1H), 4.24 (m, 1H), 3.05 (m, 1H), 2.78 (m, 1H), 2.44 (m, 1H), 1.99 (s, 3H, CH_3), 1.74 (m, 1H), 1.22 (d, 6H, CH_3), 0.90 (m, 3H, CH_3).


5m

^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.5, 62.7, 53.3, 44.7, 31.2, 31.1, 21.1, 20.7, 20.1, 19.0, 10.5.
GC-MSD *m/z*(rel. int.): *283*(M^+, 0), *255*(5), *223*(14), *210*(38), *194*(30), *180*(100), *170*(54), *153*(22), *125*(19), *83*(21), *69*(42), *55*(16), *41*(37).
GC-FID: column HP-Chiral-20B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 2°C/min to 180°C - 50 min, total time 100 min; retention times: *R*-**4m** 36.2 min, (*S*,*R*)-**5m** 76.5 min, (*R*,*R*)-**5m** 78.5 min.

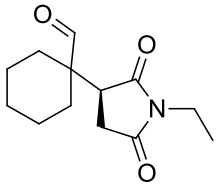
(*S*)-1-(1-methyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6a**),
Table 6, entry 1: crystallization in hexane/EtOAc 10/1, 60.3 mg white solid, yield 90 %, ee 99 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.55 (s, 1H, CH=O), 3.03 (m, 1H), 2.98 (s, 3H), 2.69 (dd, 1H), 2.50 (dd, 1H), 1.95 (m, 1H), 1.81-1.78 (m, 2H), 1.61-1.55 (m, 7H).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 204.7, 204.6, 177.8, 175.7, 51.4, 43.6, 31.1, 28.8, 27.9, 25.1, 24.8, 21.6, 21.4.


6a

GC-MSD *m/z*(rel. int.): *223*(M^+, 0), *195*(23), *152*(21), *140*(18), *113*(100), *81*(24), *67*(38), *55*(15), *41*(11).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 80°C - 10 min, 1°C/min to 130°C - 20 min, 3°C/min to 151°C - 20 min, 6°C/min to 181°C - 20 min, total time 132 min; retention times: **4a** 11.7 min, (*S*)-**6a** 127.7 min, (*R*)-**6a** 127.8 min.

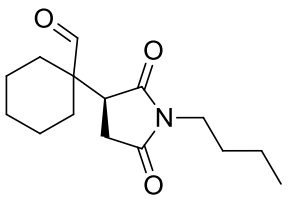
(*S*)-1-(1-ethyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6b**),
Table 6, entry 2: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 3/1, 62.6 mg off-white solid, yield 88 %, ee 97 %.



6b

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.54 (s, 1H, CH=O), 3.54 (q, 2H, *J* 7.2 Hz), 3.00 (dd, 1H, *J* 5.6, 9.3 Hz), 2.66 (dd, 1H, *J* 9.3, 18.2 Hz), 2.50 (dd, 1H, *J* 5.6, 18.1 Hz), 1.93 (m, 1H) 1.79 (m, 2H, CH_2), 1.56-1.39 (m, 7H), 1.15 (tr, 6H, *J* 7.2 Hz, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 204.7, 177.5, 175.5, 51.4, 43.4, 33.7, 31.1, 28.8, 27.8, 25.1, 21.6, 21.4, 12.8.
GC-MSD *m/z*(rel. int.): *237*(M^+, 1), *208*(39), *166*(19), *154*(19), *127*(100), *109*(23), *81*(15), *67*(23), *55*(12), *41*(9).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 60°C - 5 min, 2°C/min to 120°C - 15 min, 2°C/min to 170°C - 20 min, 3°C/min to 188°C - 19 min, total time 120 min; retention times: **4b** 24.9 min, (*S*)-**6b** 113.7 min, (*R*)-**6b** 115.0 min.

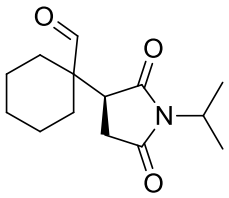
(*S*)-1-(1-butyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6c**),
Table 6, entry 3: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 3/1, 67.7 mg off-white solid, yield 85 %, ee 97 %.



6c

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.55 (s, 1H, CH=O), 3.48 (tr, 2H), 3.01 (dd, 1H, *J* 5.6, 9.3 Hz), 2.67 (dd, 1H, *J* 9.3, 18.2 Hz), 2.49 (dd, 1H, *J* 5.6, 18.1 Hz), 1.95 (m, 1H), 1.79 (m, 2H), 1.57-1.54 (m, 7H), 1.31 (m, 4H), 0.92 (tr, 3H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 204.7, 177.8, 175.7, 51.4, 43.5, 38.6, 31.1, 29.6, 28.9, 27.8, 25.1, 21.6, 21.5, 20.0, 13.6.
GC-MSD *m/z*(rel. int.): *265*(M^+, 1), *236*(32), *194*(16), *182*(24), *155*(100), *109*(28), *81*(14), *67*(23), *55*(16), *41*(11).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 70°C - 5 min, 2°C/min to 120°C - 25 min, 2°C/min to 170°C - 30 min, 3°C/min to 188°C - 29 min, total time 145 min; retention times: **4d** 28.0 min, (*S*)-**6c** 136.0 min, (*R*)-**6c** 137.5 min.

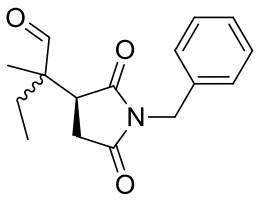
(*S*)-1-(1-isopropyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6d**),
Table 6, entry 4: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 2/1, 60.3 mg transparent oil, yield 80 %, ee 98 %.



6d

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.55 (s, 1H, CH=O), 4.35 (m, 1H), 2.95 (dd, 1H, *J* 5.6, 9.3 Hz), 2.62 (dd, 1H, *J* 9.3, 18.2 Hz), 2.44 (dd, 1H, *J* 5.6, 18.1 Hz), 1.94-1.90 (m, 1H), 1.81-1.74 (m, 2H), 1.63-1.43 (m, 7H), 1.37 (d, 6H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 204.7, 177.8, 175.7, 51.4, 43.8, 43.2, 31.1, 28.8, 27.6, 25.1, 21.6, 21.5, 19.1.
GC-MSD *m/z*(rel. int.): *251*(M^+, 1), *222*(31), *180*(11), *168*(20), *141*(100), *109*(28), *99*(35), *81*(12), *67*(18), *55*(11), *41*(9).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 160°C - 40 min, 8°C/min to 184°C - 7 min, total time 140 min; retention times: **4j** 8.1 min, (*S*)-**6d** 132.5 min, (*R*)-**6d** 133.5 min.

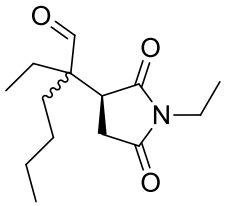
2-((*S*)-1-benzyl-2,5-dioxopyrrolidin-3-yl)-2-methylbutanal (**6e**),
Table 6, entry 5: flash chromatography, eluted with hexane/ethyl acetate 5/1, 70.5 mg transparent oil, yield 86 %, dr 60/40, ee 99, 97 %.



6e

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.59 (s, 1H, CH=O), 7.35 (m, 2H, Ar-H), 7.29 (m, 3H, Ar-H), 4.63 (m, 2H), 3.21 (dd, 1H), 2.77 (dd, 1H), 2.49 (dd, 1H), 1.62 (m, 2H, CH_2), 1.02 (s, 3H, CH_3), 0.86 (tr, 3H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 203.1, 177.4, 175.4, 135.6, 128.7, 128.6, 128.0, 50.9, 44.0, 42.5, 30.6, 27.4, 14.6, 7.9.
GC-MSD *m/z*(rel. int.): *273*(M^+, 1), *245*(57), *230*(15), *216*(45), *189*(28), *138*(26), *91*(100), *83*(24), *69*(13), *55*(19), *41*(8).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 165°C - 95 min, 5°C/min to 180°C - 47 min, total time 240 min; retention times: **2a** 57.3 min, **6e** enantiomer pairs: 205.7 min, 208.3 min and 224.1 min, 226.3 min.

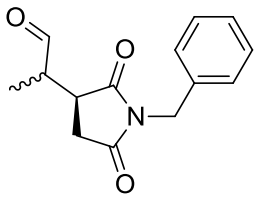
2-ethyl-2-((*S*)-1-ethyl-2,5-dioxopyrrolidin-3-yl)hexanal (**6f**),
Table 6, entry 6: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 1/3, 49.4 mg transparent oil, yield 65 %, dr 62/38, ee 98, 98 %.



6f

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.59 (d, 1H, CH=O), 3.54 (m, 2H), 3.01 (dd, 1H), 2.77 (dd, 1H), 2.52 (dd, 1H), 1.90-1.60 (m, 4H), 1.36-1.22 (m, 4H), 1.16 (tr, 3H, CH_3), 0.92 (m, 6H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 203.9, 177.9, 175.7, 54.0, 53.9, 42.2, 42.0, 33.7, 31.6, 31.5, 30.4, 25.8, 24.9, 23.8, 13.8, 12.7, 8.2.
GC-MSD *m/z*(rel. int.): *253*(M^+, 2), *224*(32), *196*(25), *182*(100), *168*(99), *127*(26), *83*(70), *69*(28), *55*(8), *41*(14).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 60°C - 5 min, 2°C/min to 120°C - 15 min, 2°C/min to 164°C - 48 min, 4°C/min to 190°C - 8.5 min, total time 135 min; retention times: **4b** 25.2 min, **6f** enantiomer pairs: 99.9 min, 101.3 min and 108.7 min, 113.3 min.

2-((*S*)-1-benzyl-2,5-dioxopyrrolidin-3-yl)propanal (**6g**),
Table 6, entry 7: flash chromatography, eluted with hexane/ethyl acetate 2/1, 44.1 mg pale yellow oil, yield 60 %, dr 52/48, ee 90, 88 %.

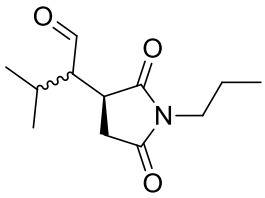


6g

Syn isomers: ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 9.57 (s, 1H, CH=O), 7.38 (d, 2H, *J* 7.2 Hz, Ar-H), 7.33-7.25 (m, 3H, Ar-H), 4.69 (m, 2H, *J* 14.2 Hz), 3.10 (m, 1H), 2.97 (m, 1H), 2.78 (dd, 1H, *J* 9.5, 18.0 Hz), 2.45 (dd, 1H, *J* 5.6, 18.0 Hz), 1.29 (d, 3H, *J* 7.7 Hz, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 201.4, 178.0, 175.6, 135.6, 128.6, 127.9, 46.1, 42.5, 40.7, 31.7, 11.2.
Anti isomers: ^{1}H NMR (500 MHz, $CDCl_3$) δ (ppm): 9.66 (s, 1H, CH=O), 7.37 (d, 2H, *J* 7.2 Hz, Ar-H), 7.33-7.25 (m, 3H, Ar-H), 4.67 (m, 2H, *J* 13.9, 16.8 Hz), 3.27 (m, 1H), 3.08 (m, 1H), 2.84 (dd, 1H, *J* 9.5, 18.5 Hz), 2.36 (dd, 1H, *J* 5.2, 18.5 Hz), 1.11 (d, 3H, *J* 7.5 Hz, CH_3).
^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 201.3, 178.2, 175.6, 135.6, 128.7, 128.6, 128.0, 46.5, 42.5, 39.3, 31.0, 9.2.
GC-MSD *m/z*(rel. int.): *259*(M^+, 1), *231*(100), *216*(49), *189*(12), *138*(27), *106*(29), *91*(97), *83*(16), *69*(28), *55*(8), *41*(14).

GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 100°C - 5 min, 2°C/min to 150°C - 15 min, 2°C/min to 184°C - 15 min, 1°C/min to 195°C - 42 min, total time 130 min; retention times: **2a** 38.4 min, **6g** enantiomer pairs: 99.0 min, 99.8 min and 108.5 min, 114.4 min.

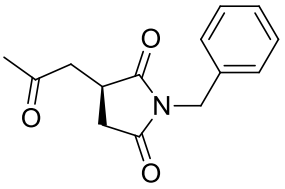
2-((*S*)-2,5-dioxo-1-propylpyrrolidin-3-yl)-3-methylbutanal (**6h**),



6h

Table 6, entry 8: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 1/3, 41.9 mg pale yellow oil, yield 62 %, dr 66/34, ee 87, 88 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 9.85 (s, 0.2H, CH=O), 9.72 (s, 0.7H, CH=O), 3.49 (m, 2H), 3.23 (m, 0.2H), 3.07 (m, 0.8H), 2.87 (m, 1H), 2.71-2.41 (m, 2H), 1.28 (m, 1H), 1.62 (m, 3H, CH_3), 1.20 (d, 2H), 1.07 (m, 3H), 0.93 (tr, 3H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 202.8, 202.5, 179.5, 176.3, 58.6, 57.1, 40.7, 40.6, 38.1, 37.9, 32.9, 32.2, 28.0, 27.7, 21.5, 21.1, 21.0, 20.9, 20.8, 20.1, 11.2.
GC-MSD *m/z*(rel. int.): *225*(M^+, 1), *182*(9), *154*(100), *141*(52), *112*(17), *97*(12), *85*(14), *69*(18), *55*(14), *41*(15).
GC-FID: column Hydrodex g-TBDAc, 25 m, d 0.25 mm, hp: 20 psi, t_{col}: 60°C - 5 min, 2°C/min to 120°C - 15 min, 2°C/min to 170°C - 15 min, 5°C/min to 185°C - 15 min, total time 108 min; retention times: **4c** 29.4 min, **6h** enantiomer pairs: 90.9 min, 94.7 min and 96.8 min, 100.2 min.

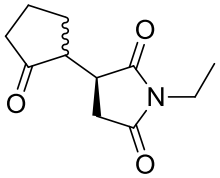
(*R*)-1-benzyl-3-(2-oxopropyl)pyrrolidine-2,5-dione (**6i**),



6i

Table 6, entry 9: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 1/1, 58.9 mg pale yellow oil, yield 80 %, ee 30 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 7.38-7.28 (m, 5H, Ar-H), 4.67 (q, 2H), 3.04 (m, 2H), 2.95-2.83 (m, 2H), 2.38 (dd, 1H), 2.17 (s, 3H, CH_3).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 205.0, 178.9, 175.8, 135.8, 128.6, 127.9, 43.5, 42.6, 35.6, 34.7, 29.7.
GC-MSD *m/z*(rel. int.): *245*(M^+, 48), *159*(100), *132*(46), *106*(90), *91*(97), *65*(22), *43*(84).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 165°C - 100 min, 1°C/min to 181°C - 59 min, total time 270 min; retention times: **2a** 57.5 min, (*R*)-**6i** 199.2 min, (*S*)-**6i** 201.5 min.

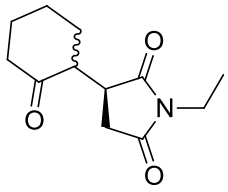
(3*S*)-1-ethyl-3-(2-oxocyclopentyl)pyrrolidine-2,5-dione (**6j**),



6j

Table 6, entry 10: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 1/1, 34.5 mg pale yellow oil, yield 55 %, dr 60/40, ee 54, 18 %.
^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 3.54-3.56 (m, 2H), 3.26-3.30 (m, 1H), 2.75-2.81 (m 2H), 2.33-2.40 (m, 2H), 2.07-2.12 (m, 3H),1.81-1.86 (m, 1H), 1.70-1.74 (m, 1H), 1.14-1.17 (m, 3H).
^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 217.7, 178.7, 175.8, 49.7, 38.6, 37.7, 33.8, 31.8, 26.9, 24.9, 20.3, 12.9
GC-MSD *m/z*(rel. int.): *209*(M^+, 1), *127*(100), *83*(44), *67*(12), *54*(24), *44*(4), *39*(10), *27*(7).
GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 160°C - 0 min, 10°C/min to 180°C - 33 min, total time 125 min; retention times: **4b** 7.4 min, **6j** enantiomer pairs: 90.9 min, 93.0 min and 97.7 min, 98.6 min.

(3*S*)-1-ethyl-3-(2-oxocyclohexyl)pyrrolidine-2,5-dione (**6k**),



6k

Table 6, entry 11: flash chromatography, eluted with hexane/*tert*-butyl methyl ether 1/1, 33.5 mg pale yellow oil, yield 50 %, dr 58/42, ee 26, 14 %.

^{1}H-NMR (500 MHz, $CDCl_3$) δ (ppm): 3.55-3.58 (m, 2H), 3.12-3.16 (m, 0.24H), 3.13-3.14 (m, 0.73H), 2.96-2.99 (m, 0.75H), 2.85-2.90 (m, 0.76H), 2.63-2.68 (m, 0.52H), 2.42-2.46 (m, 1.0H), 2.29-2.37 (m, 2.0H), 2.09-2.17 (m, 1.3H), 1.93-1.95(m, 1.8H, CH_2), 1.59-1.83(m, 3.1H), 1.16-1.19 (m, 3.1H).

^{13}C-NMR (125 MHz, $CDCl_3$) δ (ppm): 51.4, 50.4, 51.9, 41.7, 41.2, 39.8, 33.8, 33.7, 32.7, 32.0, 31.9, 29.3, 27.1, 27.0, 25.0, 34.9, 12.8, 12.5

GC-MSD *m/z*(rel. int.): *223*(M^+, 1), *127*(100), *97*(74), *80*(11), *68*(16), *55*(14).

GC-FID: column Cyclosil-B, 30 m, d 0.25 mm, hp: 25 psi, t_{col}: 100°C - 10 min, 1°C/min to 150°C - 20 min, 1°C/min to 160°C, 10°C/min to 180°C - 33 min, total time 125 min; retention times: **4b** 7.4 min, **6k** enantiomer pairs: 102.8 min, 104.6 min and 109.4 min, 112.0 min.

9. Results of UV-Vis spectroscopic investigations

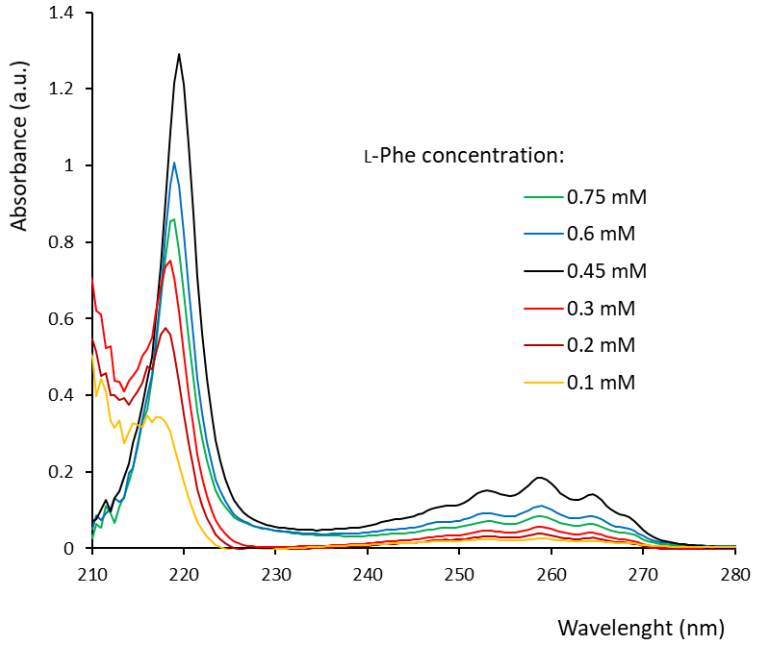


Fig. S8 UV-Vis spectra of L-Phe solutions of increasing concentrations in MeOH used for calibration.

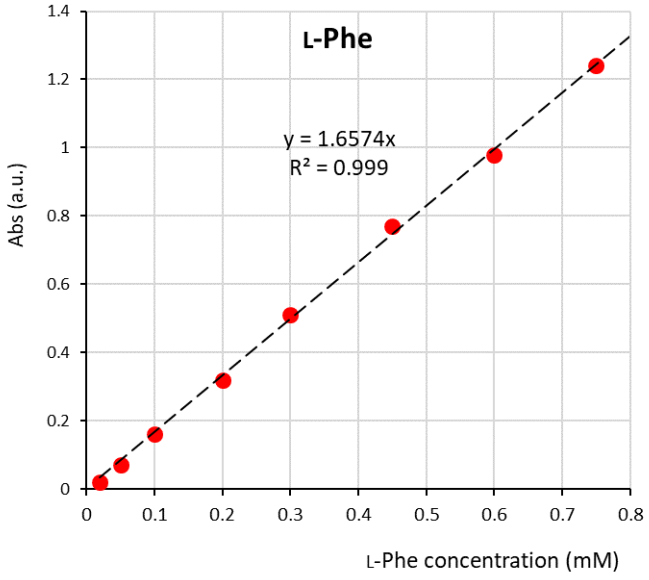


Fig. S9 Calibration plot used to determine the concentration of L-Phe remained in or desorbed to the liquid phase.

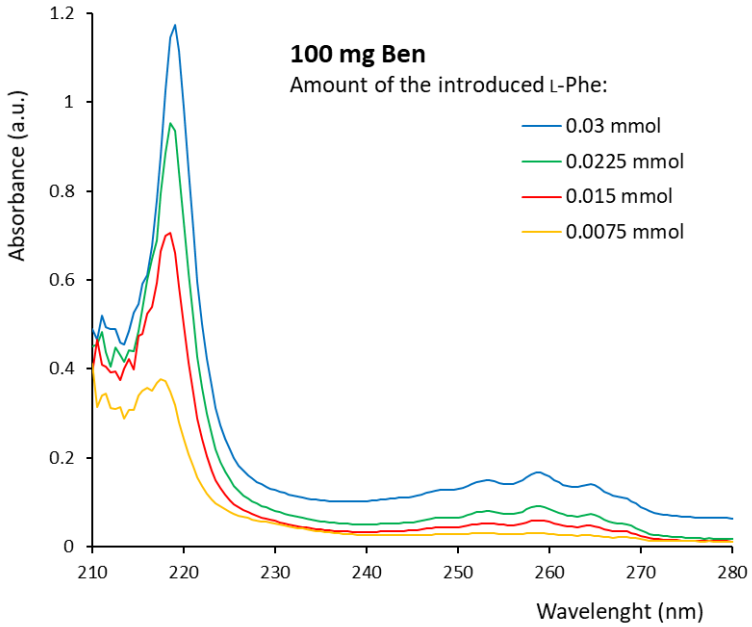


Fig. S10 UV-Vis spectra of L-Phe solutions obtained from the supernatant resulted following stirring the given amount of amino acid and 100 mg Ben in 1 cm^3 MeOH for 2 h.

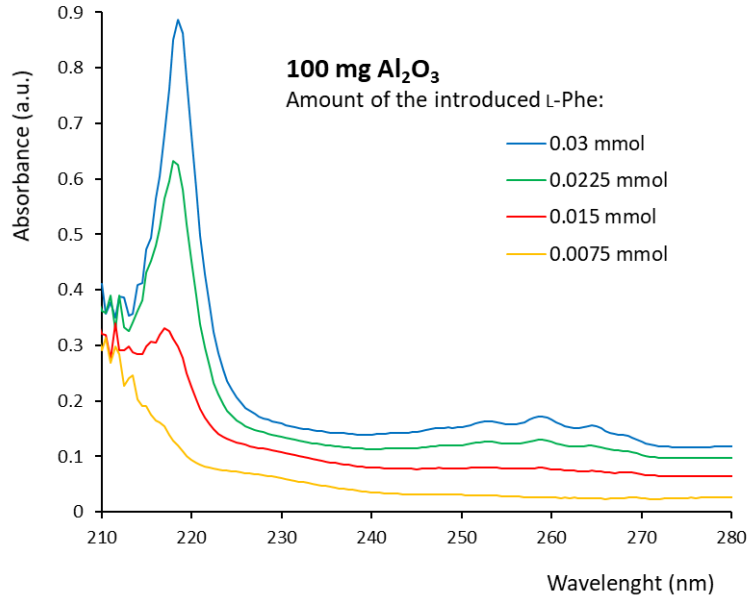


Fig. S11 UV-Vis spectra of L-Phe solutions obtained from the supernatant resulted following stirring the given amount of amino acid and 100 mg Al_2O_3 in 1 cm^3 MeOH for 2 h.

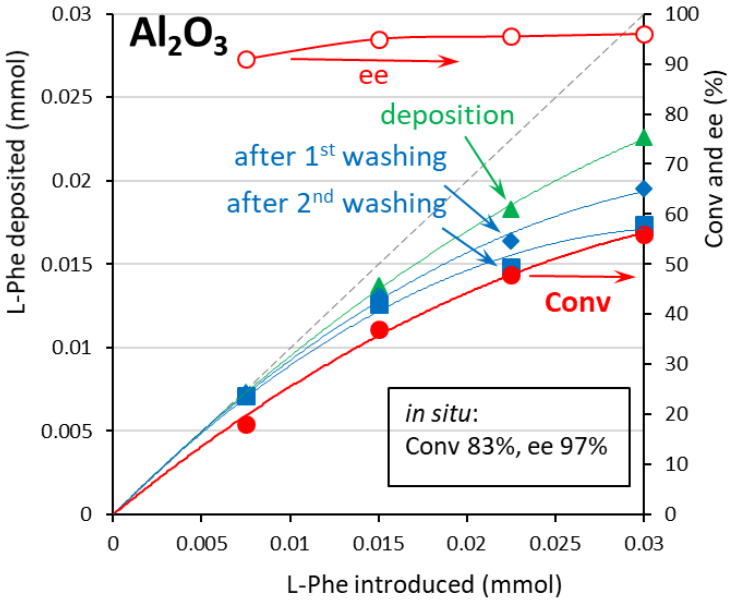


Fig. S12 Amount of L-Phe deposited on 100 mg Al_2O_3 from 1 cm^3 MeOH determined by UV-Vis spectroscopy; ▲ L-Phe amount included after 2 h stirring at rt, ◆ L-Phe amount remained following another 2 h washing using 1 cm^3 MeOH, ■ L-Phe amount remained following a 2nd 2 h washing using 1 cm^3 MeOH. Conv ● and ee ○ obtained applying the prepared materials as catalysts in the addition of **1a** to **4a**. Reaction conditions: 1.2 mmol **1a**, 0.3 mmol **4a**, 1 cm^3 EtOAc, rt, 16 h; the result obtained under identical conditions with catalyst formed *in situ* using 0.03 mmol L-Phe is given in the frame.

10. Thermogravimetric analysis of L-phenylalanine

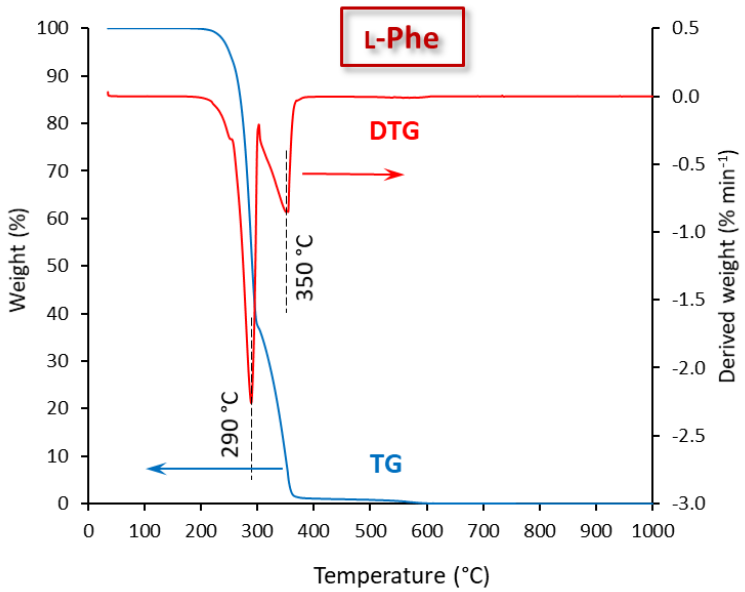


Fig. S13 Thermogravimetric analysis of L-Phe.

11. XRD diffractograms of selected materials

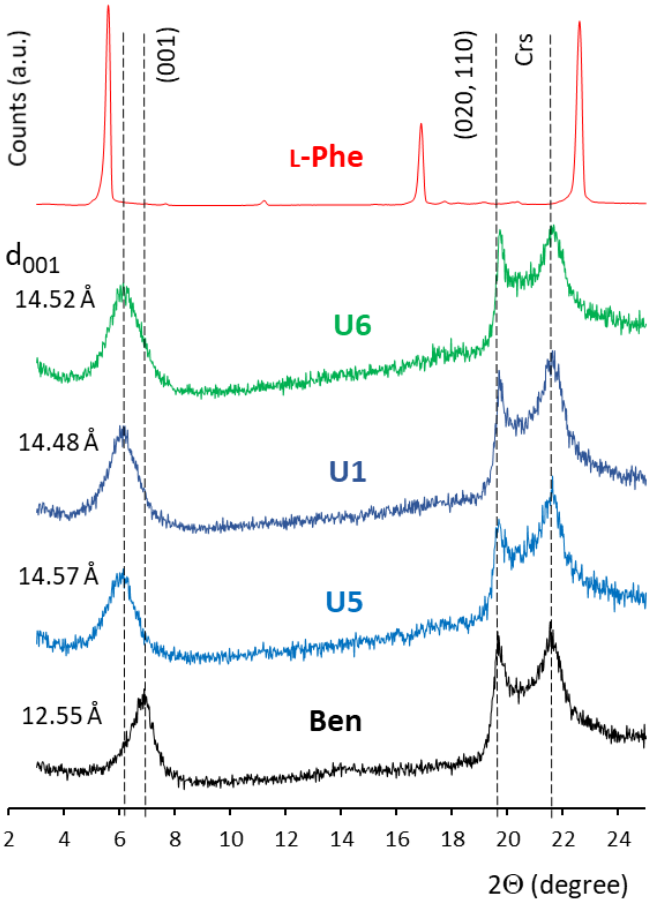


Fig. S14 X-ray diffractograms of Ben, L-Phe, and that of *in situ* obtained materials after reactions of **1a** and **2a**; material formed from 200 mg Ben and 0.03 mmol L-Phe in $^{i}Pr_2O$ after a 6 h reaction at rt, Conv 95 %, ee 98 %, (**U5**); material formed from 200 mg Ben and 0.03 mmol L-Phe in $^{i}Pr_2O$ after a 6 h reaction at 50°C, Conv >99 %, ee 96.5 %, (**U1**); material formed from 50 mg Ben and 0.03 mmol L-Phe in EtOAc after a 16 h reaction at 50°C, Conv >99 %, ee 97.5 %, (**U6**).

12. FT-IR spectra of hybrid materials

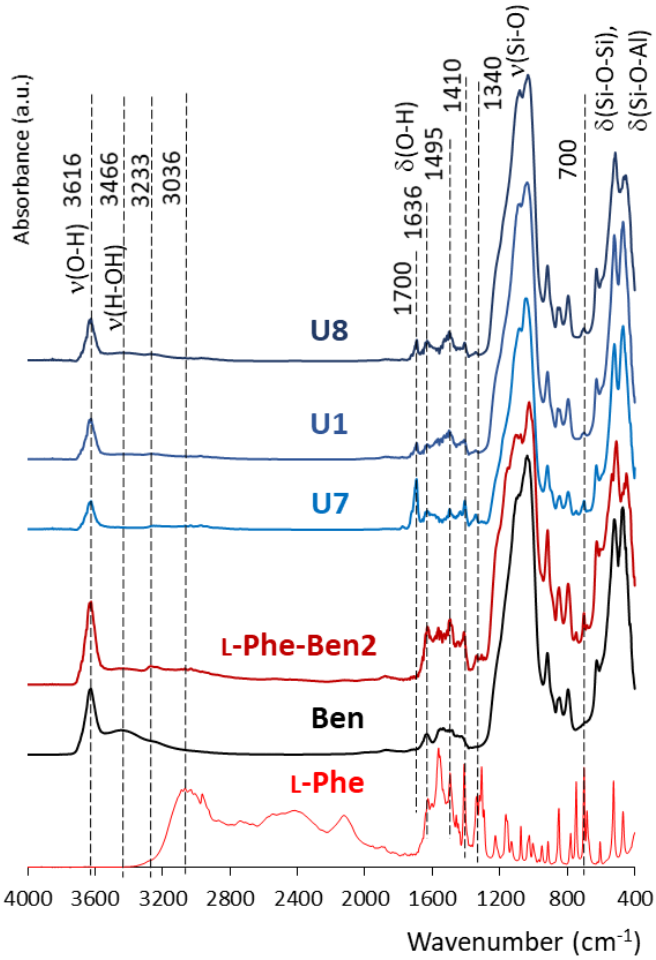


Fig. S15 Full FT-IR spectra of L-Phe, Ben, L-Phe-Ben2 and three materials used in reactions of **1a** and **2a** in 1 cm^3 $^{i}Pr_2O$; **U1** (see Fig. 7); 55 mg of L-Phe-Ben2 after first use in a 6 h reaction at 50°C, Conv 99 %, ee 96.5 %, (**U7**); *in situ* formed material from 200 mg Ben and 0.03 mmol L-Phe, in 6 h reactions at rt: **U5** after ten uses, last run Conv 62 %, ee 98 % (**U8**).

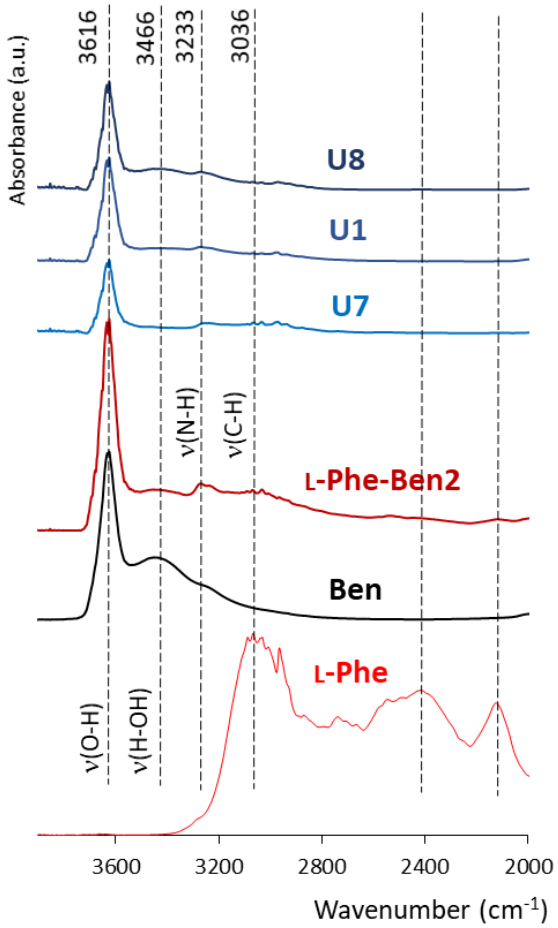


Fig. S16 Selected ranges of the FT-IR spectra of L-Phe, Ben, L-Phe-Ben2 and three materials used in reactions of **1a** and **2a** in 1 cm^3 $^iPr_2O$; **U1** (see Fig. 7); 55 mg of L-Phe-Ben2 after first use in a 6 h reaction at 50°C, Conv 99 %, ee 96.5 %, (**U7**); *in situ* formed material from 200 mg Ben and 0.03 mmol L-Phe, in 6 h reactions at rt: **U5** after ten uses, last run Conv 62 %, ee 98 % (**U8**).

13. Adsorption mode on Al_2O_3

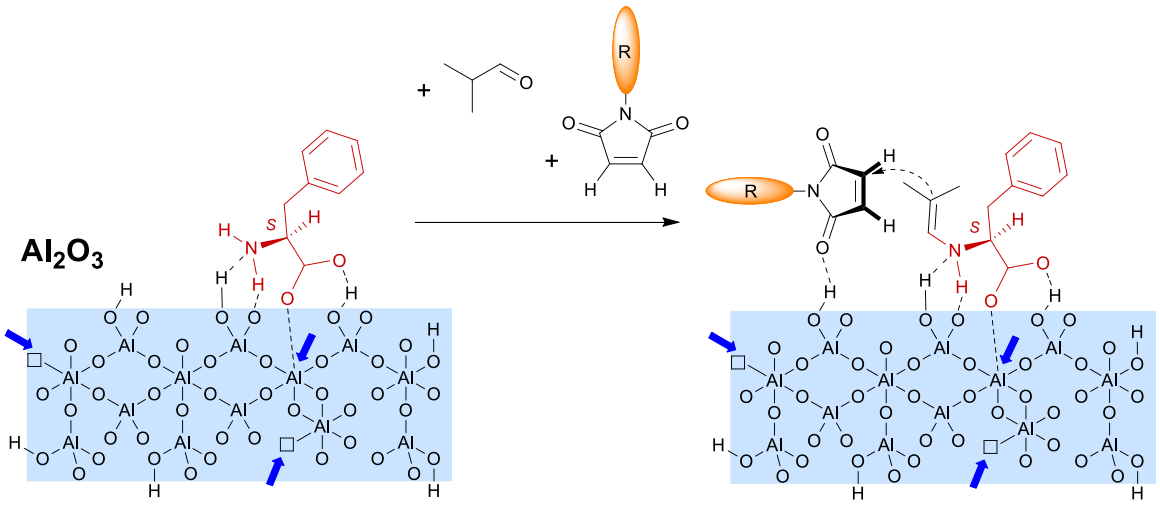
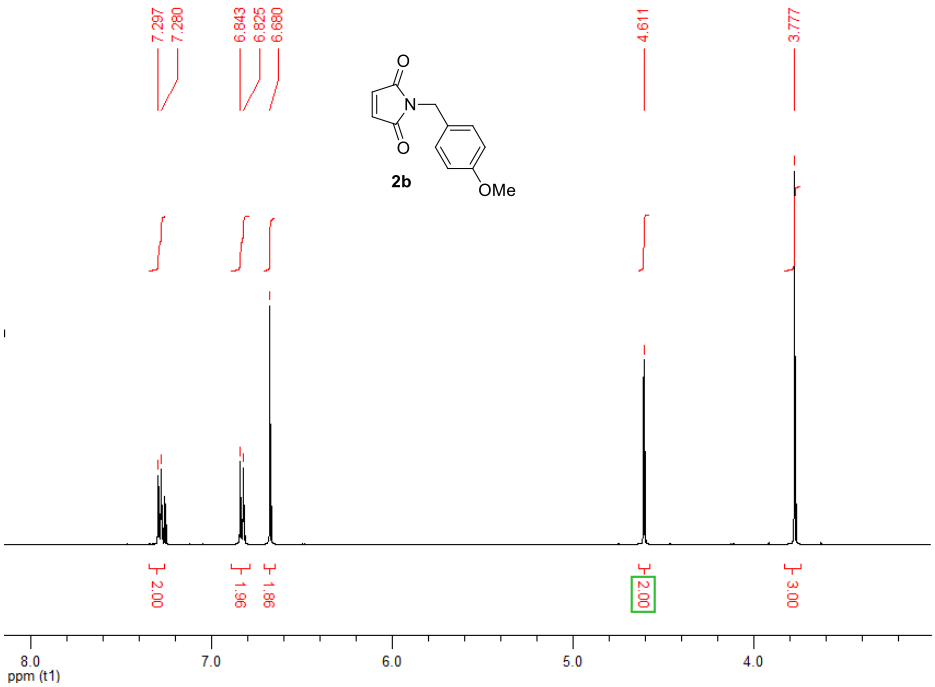


Fig. S17 Possible adsorption mode of L-Phe on Al_2O_3 surface and the structure of the surface transition state.

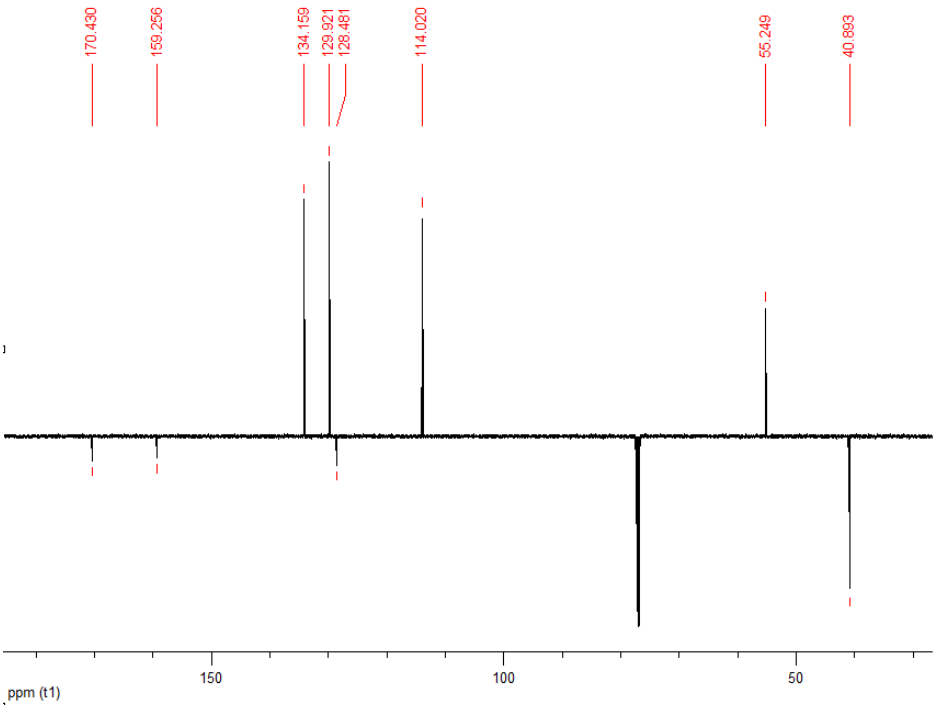
14. ^{1}H- and ^{13}C-NMR spectra of the materials

14.1. ^{1}H- and ^{13}C-NMR spectra of the *N*-substituted maleimides

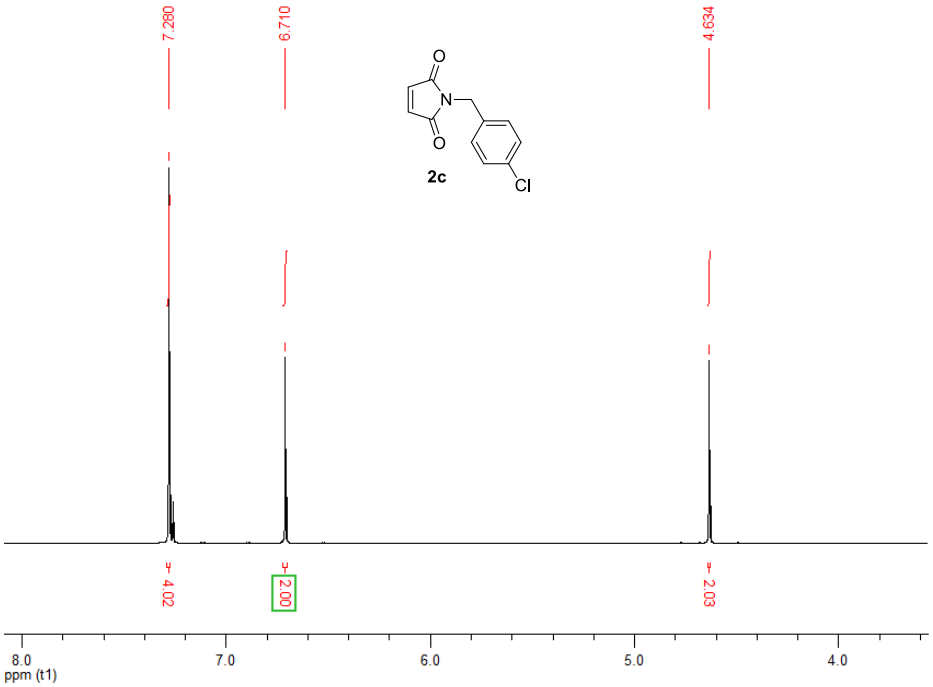
N-(4-methoxybenzyl)maleimide (**2b**),
^{1}H-NMR, 500 MHz, $CDCl_3$



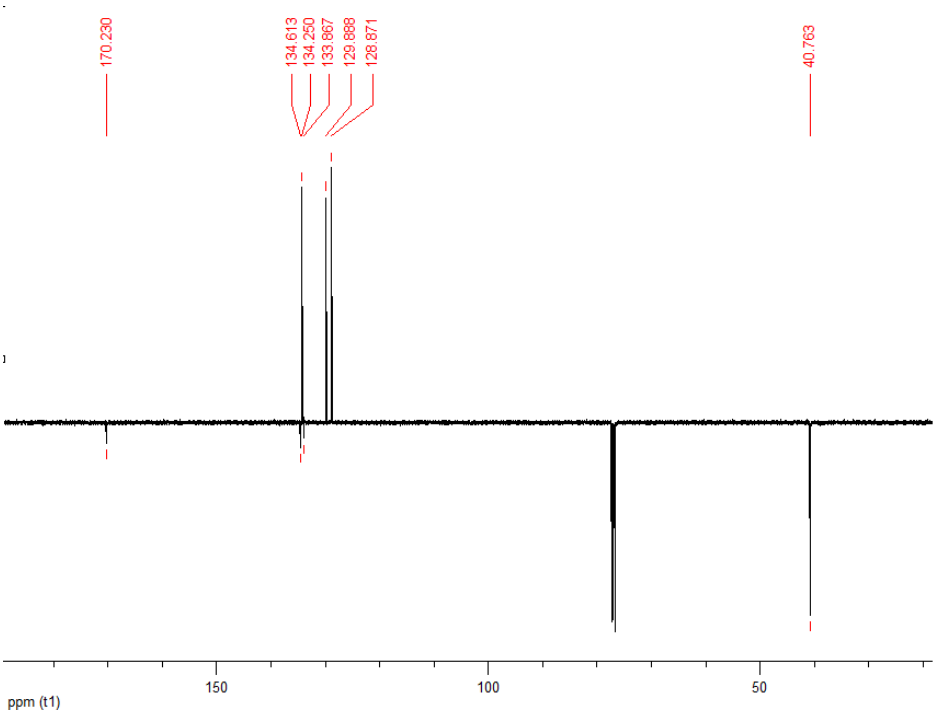
^{13}C-NMR, 125 MHz, $CDCl_3$



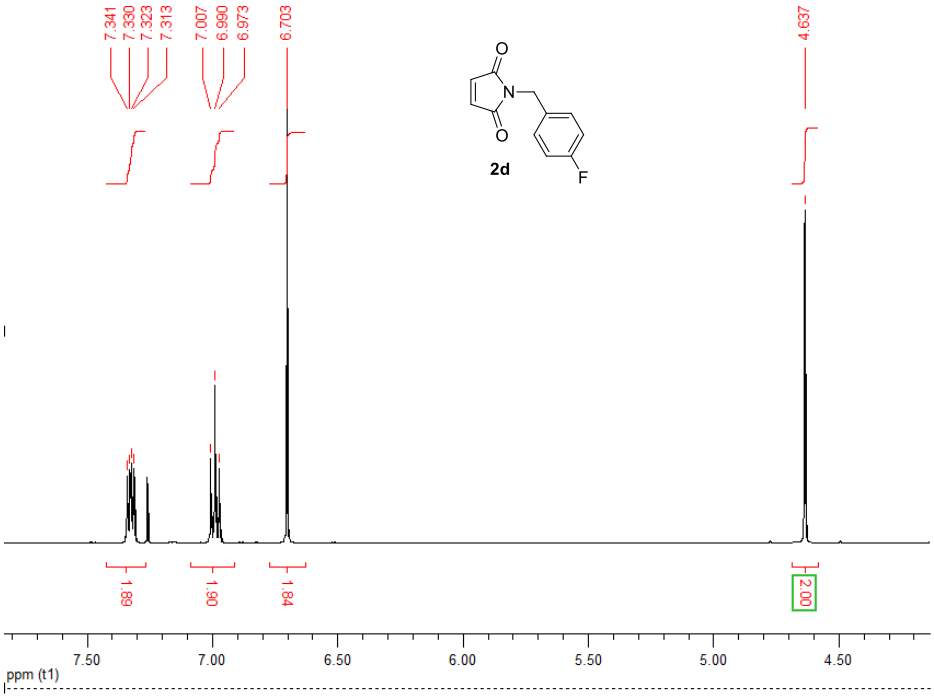
N-(4-chlorobenzyl)maleimide (**2c**),
^{1}H-NMR, 500 MHz, $CDCl_3$



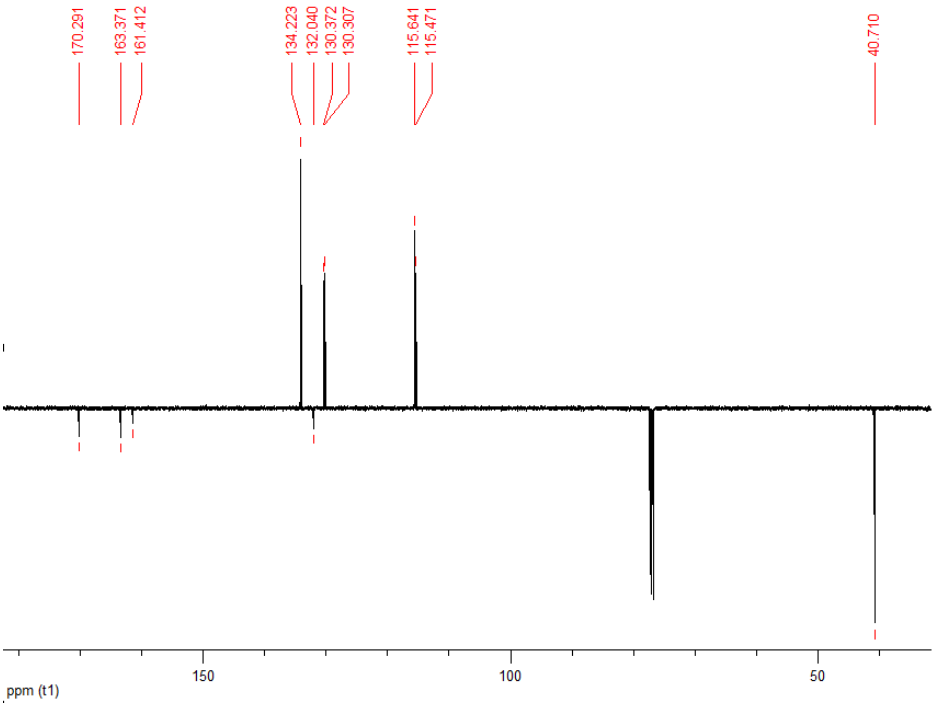
^{13}C-NMR, 125 MHz, $CDCl_3$



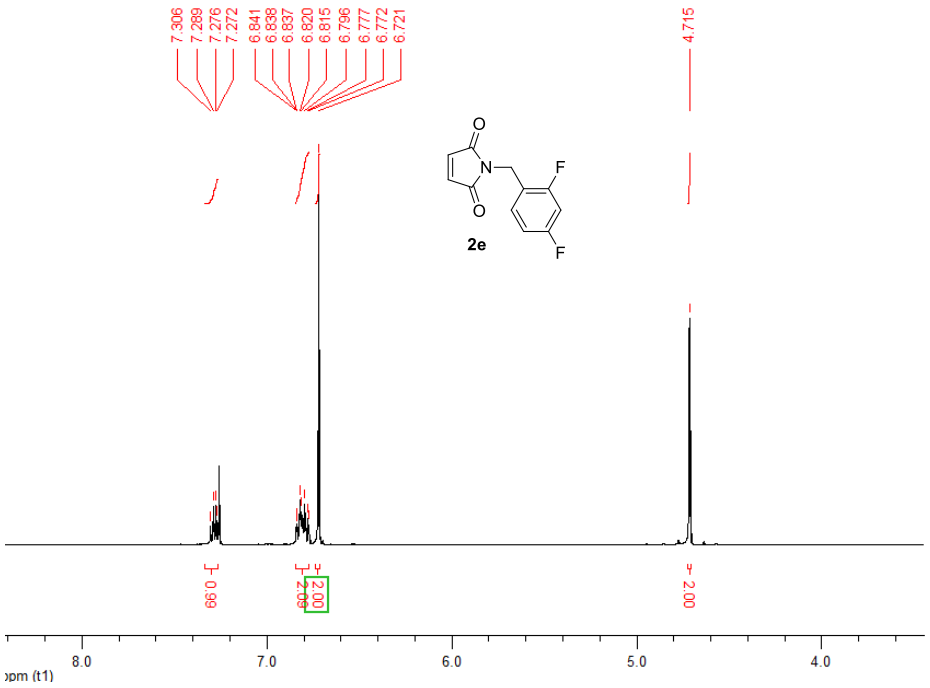
N-(4-fluorobenzyl)maleimide (**2d**),
^{1}H-NMR, 500 MHz, $CDCl_3$



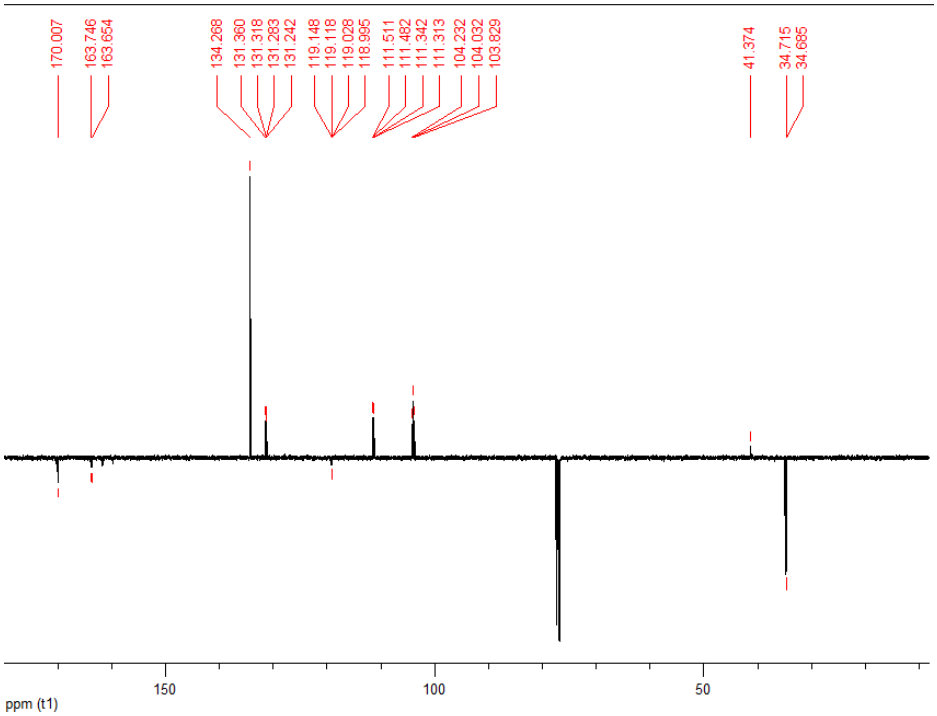
^{13}C-NMR, 125 MHz, $CDCl_3$



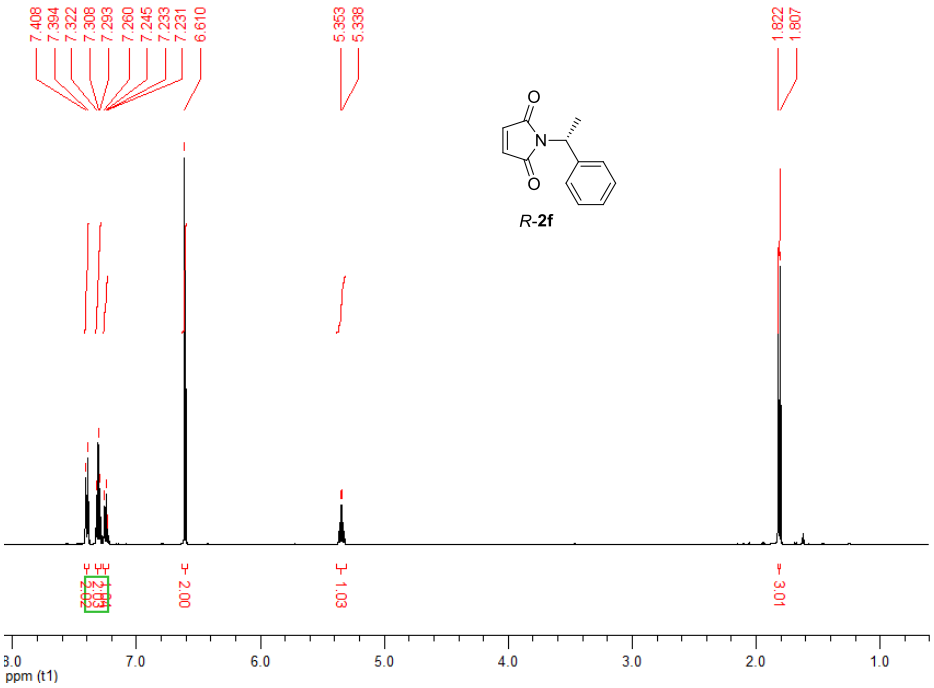
N-(2,4-difluorobenzyl)maleimide (**2e**),
^{1}H-NMR, 500 MHz, $CDCl_3$



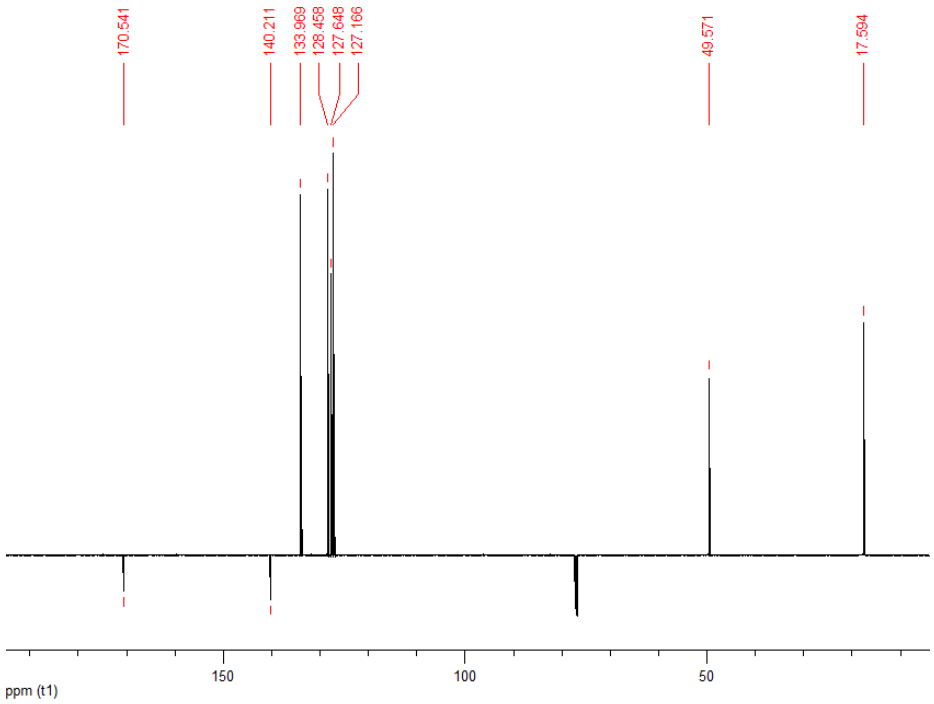
^{13}C-NMR, 125 MHz, $CDCl_3$



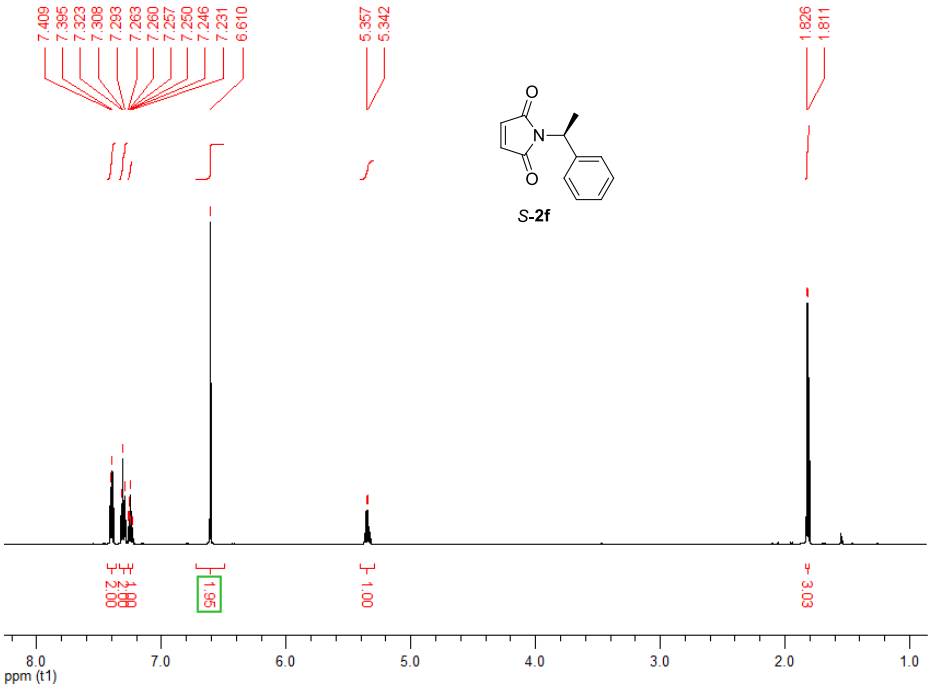
(*R*)-*N*-(1-phenylethyl)maleimide (*R*-**2f**),
^{1}H-NMR, 500 MHz, $CDCl_3$



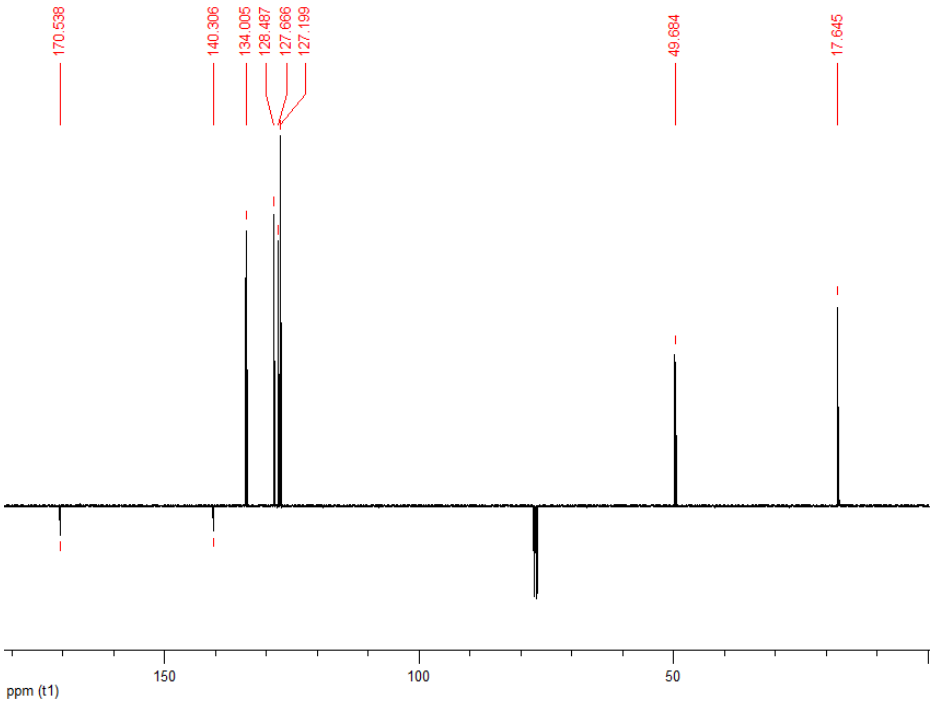
^{13}C-NMR, 125 MHz, $CDCl_3$



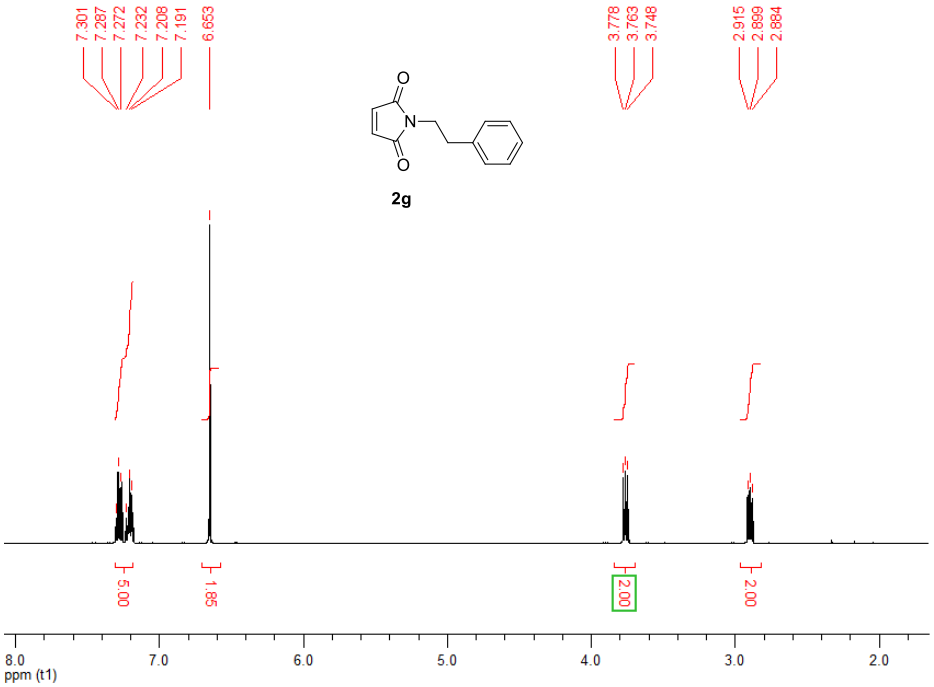
(*S*)-*N*-(1-phenylethyl)maleimide (*S*-**2f**),
^{1}H-NMR, 500 MHz, $CDCl_3$



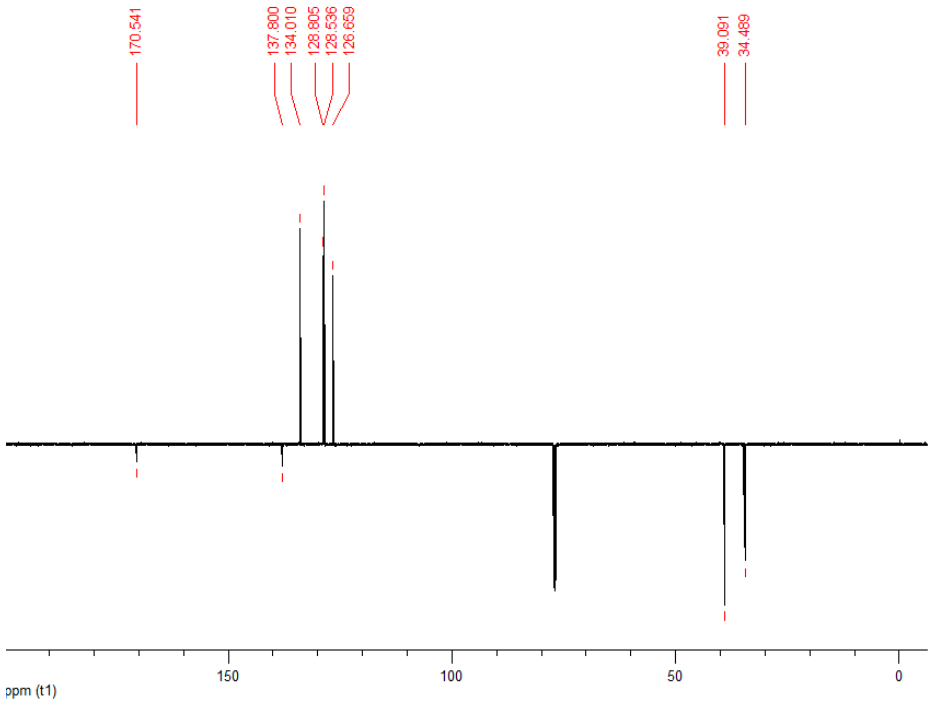
^{13}C-NMR, 125 MHz, $CDCl_3$



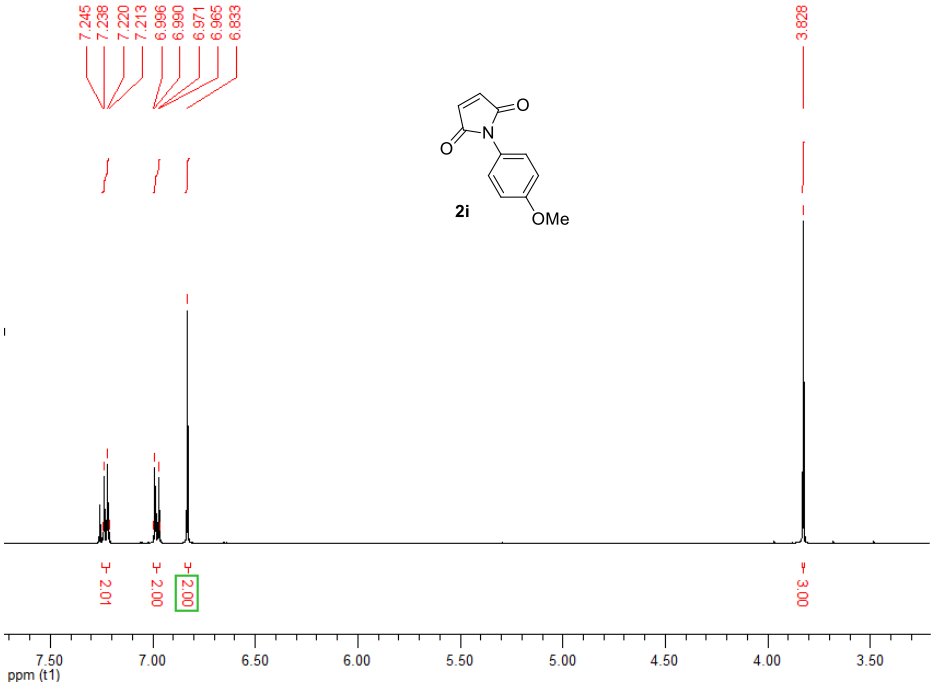
N-(2-phenylethyl)maleimide (**2g**),
^{1}H-NMR, 500 MHz, $CDCl_3$



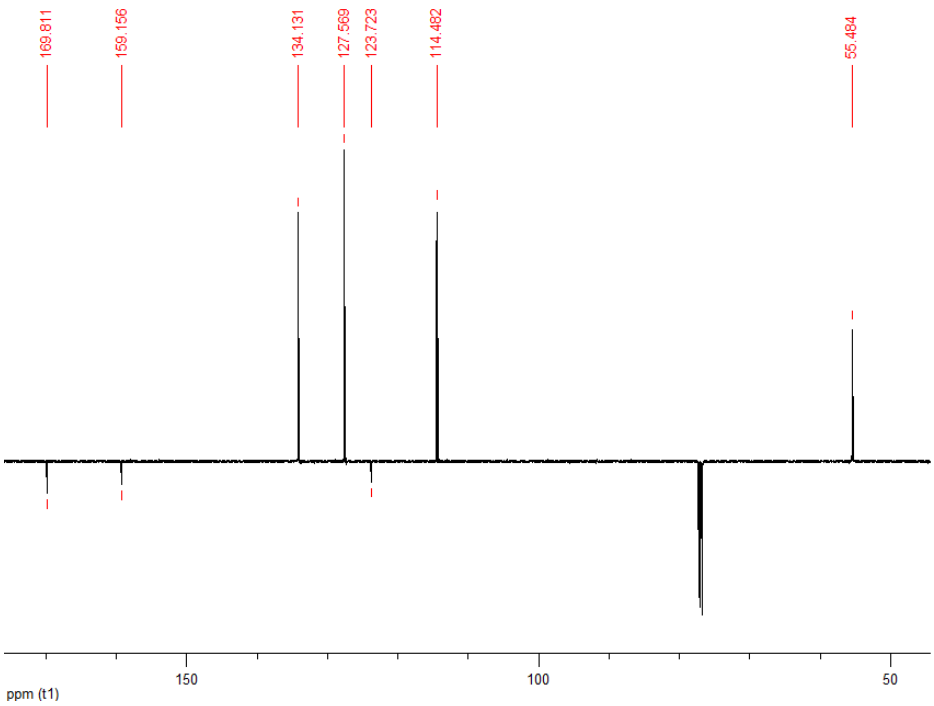
^{13}C-NMR, 125 MHz, $CDCl_3$



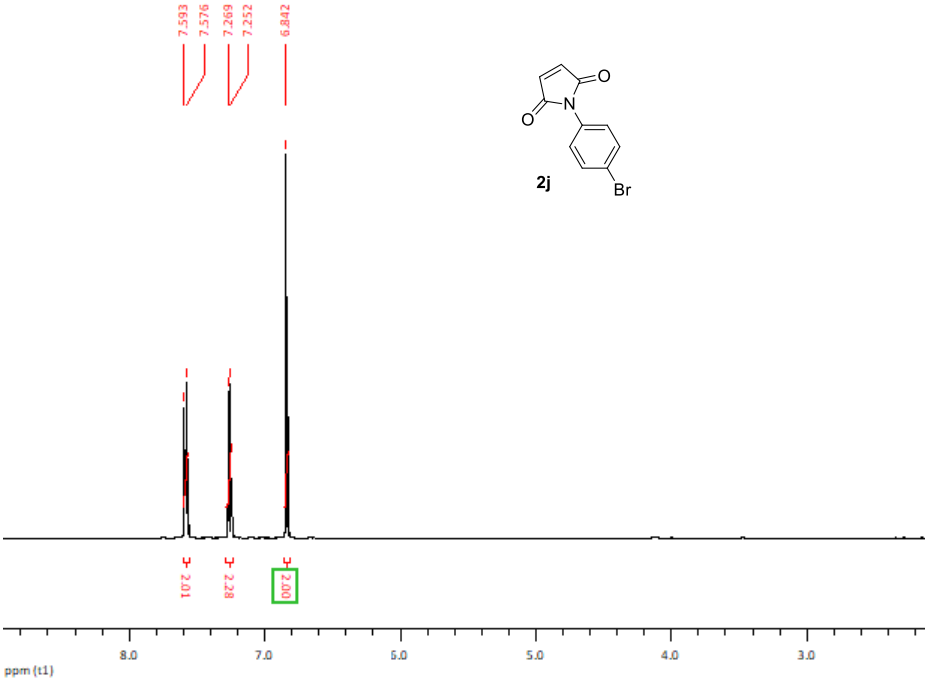
N-(4-methoxyphenyl)maleimide (**2i**),
^{1}H-NMR, 500 MHz, $CDCl_3$



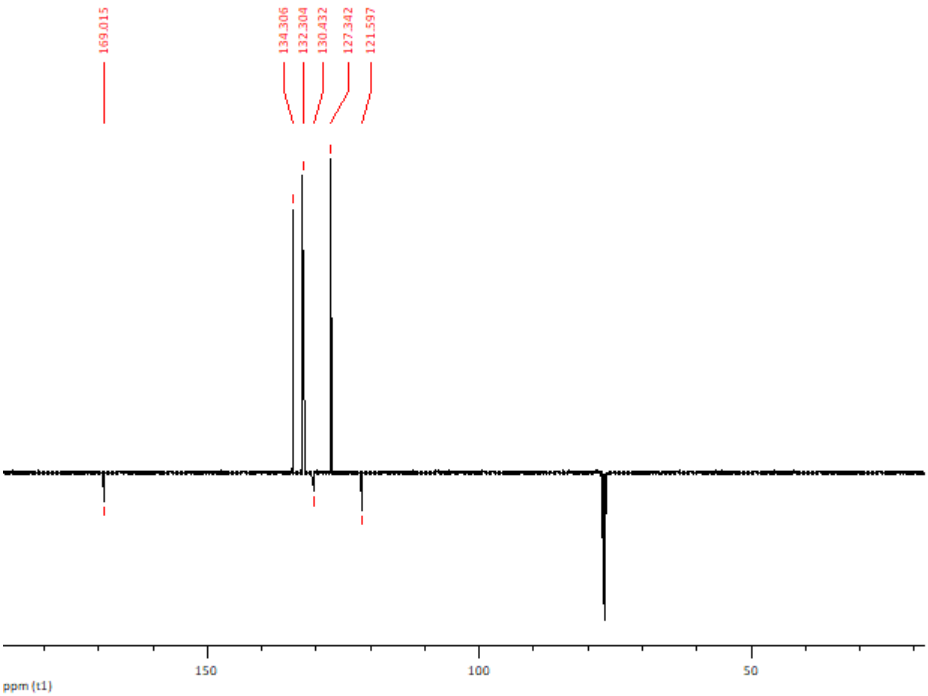
^{13}C-NMR, 125 MHz, $CDCl_3$



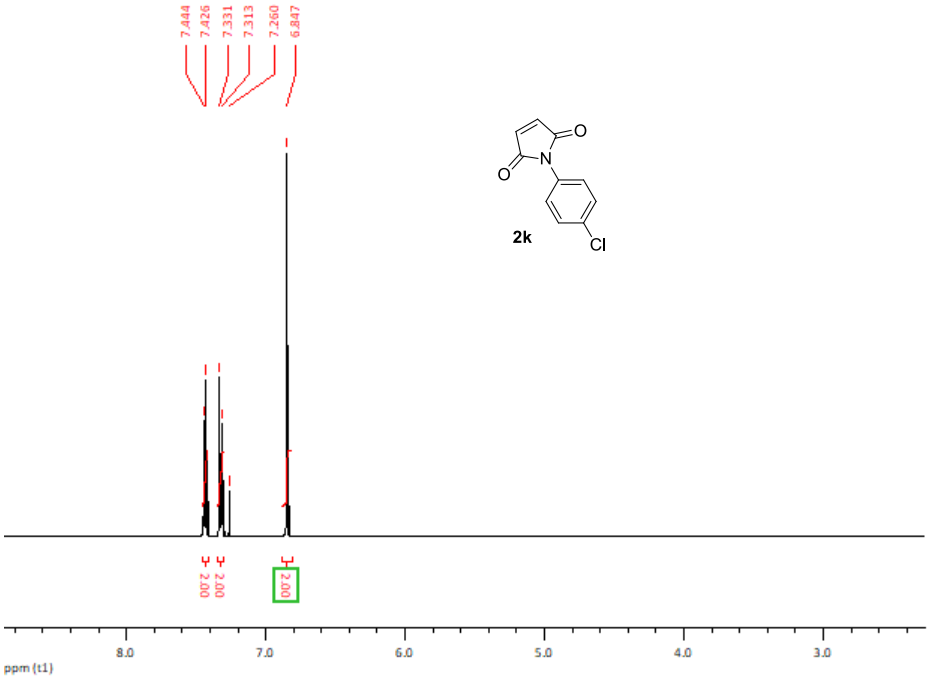
N-(4-bromophenyl)maleimide (**2j**),
^{1}H-NMR, 500 MHz, $CDCl_3$



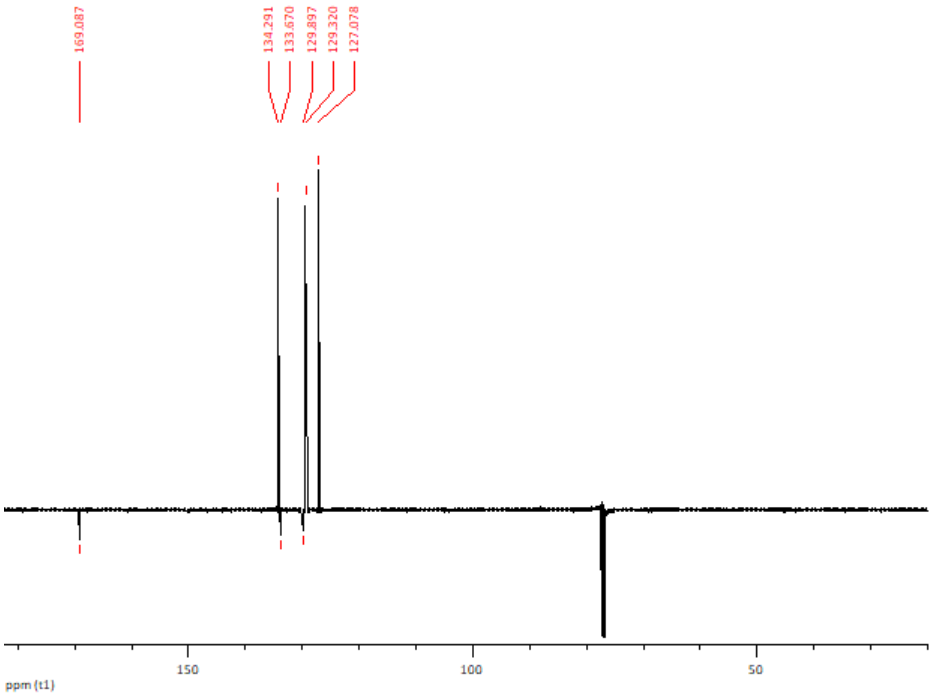
^{13}C-NMR, 125 MHz, $CDCl_3$



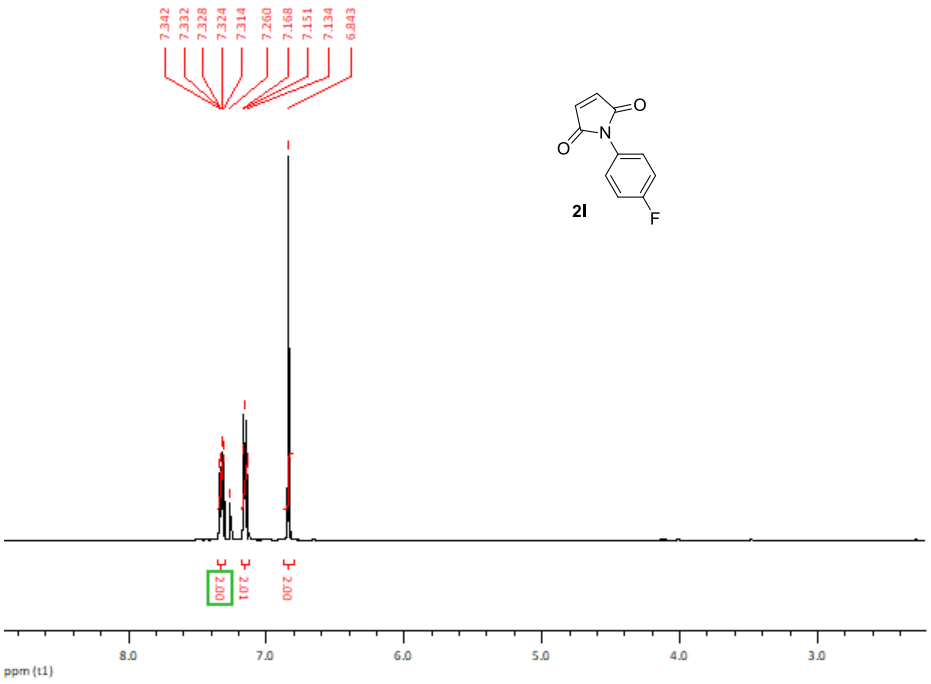
N-(4-chlorophenyl)maleimide (**2k**),
^{1}H-NMR, 500 MHz, $CDCl_3$



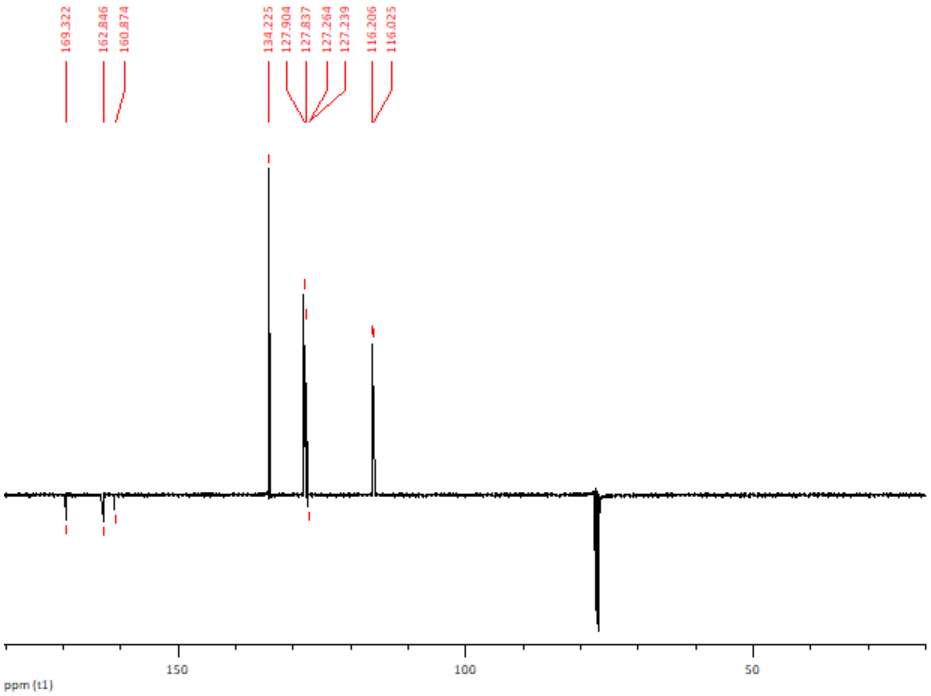
^{13}C-NMR, 125 MHz, $CDCl_3$



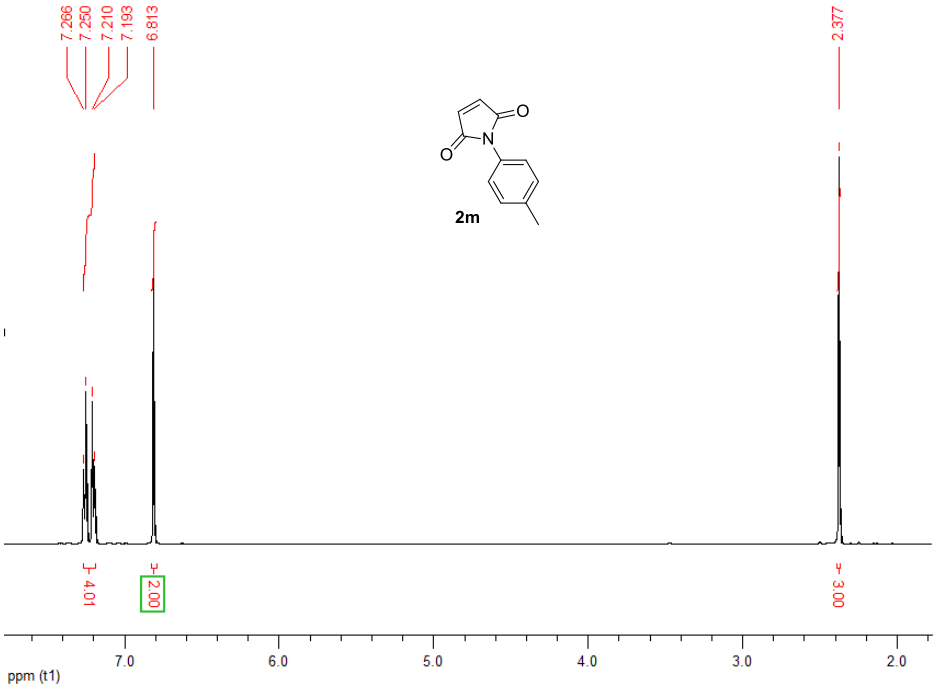
N-(4-fluorophenyl)maleimide (**2l**),
^{1}H-NMR, 500 MHz, $CDCl_3$



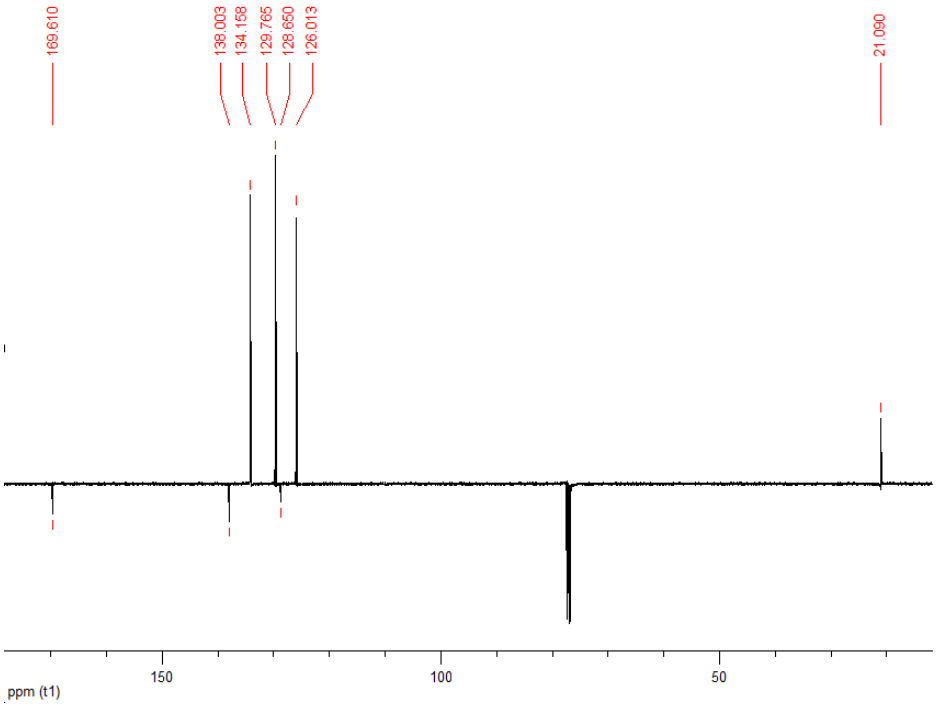
^{13}C-NMR, 125 MHz, $CDCl_3$



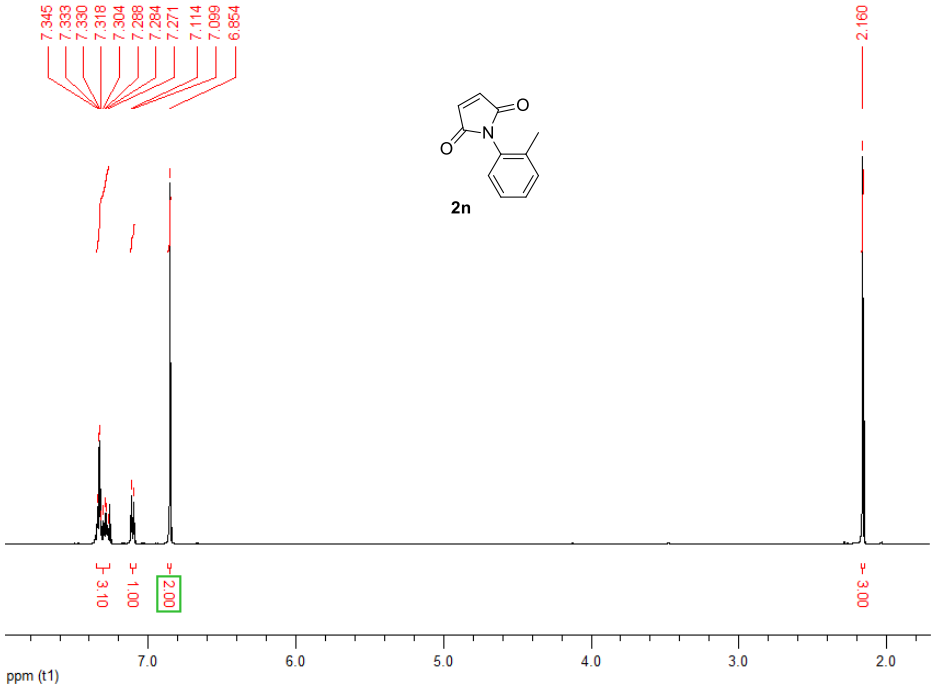
N-(4-methylphenyl)maleimide (**2m**),
^{1}H-NMR, 500 MHz, $CDCl_3$



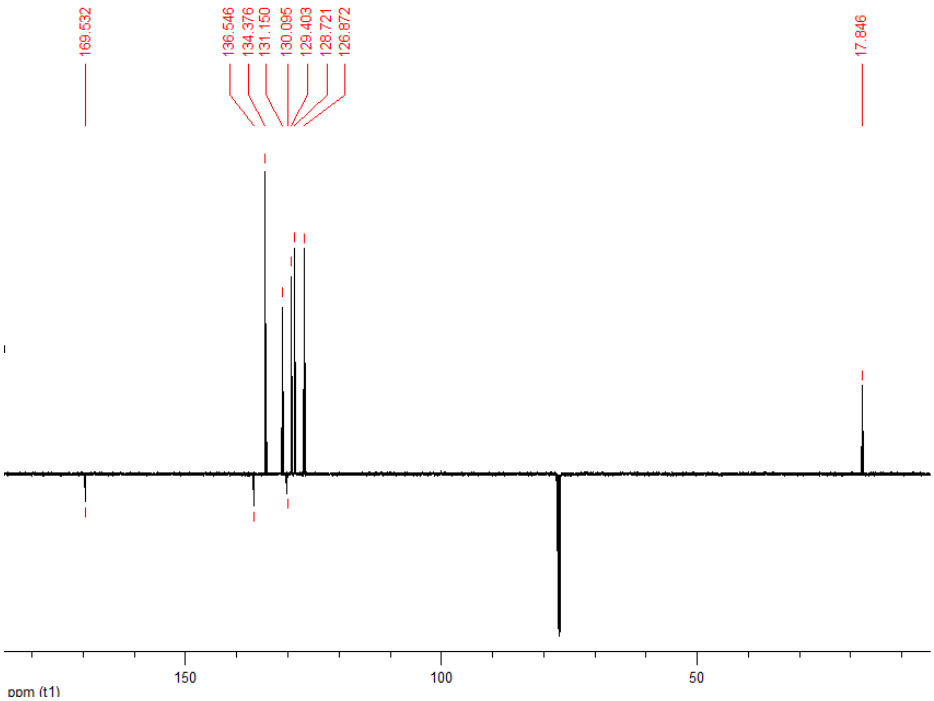
^{13}C-NMR, 125 MHz, $CDCl_3$



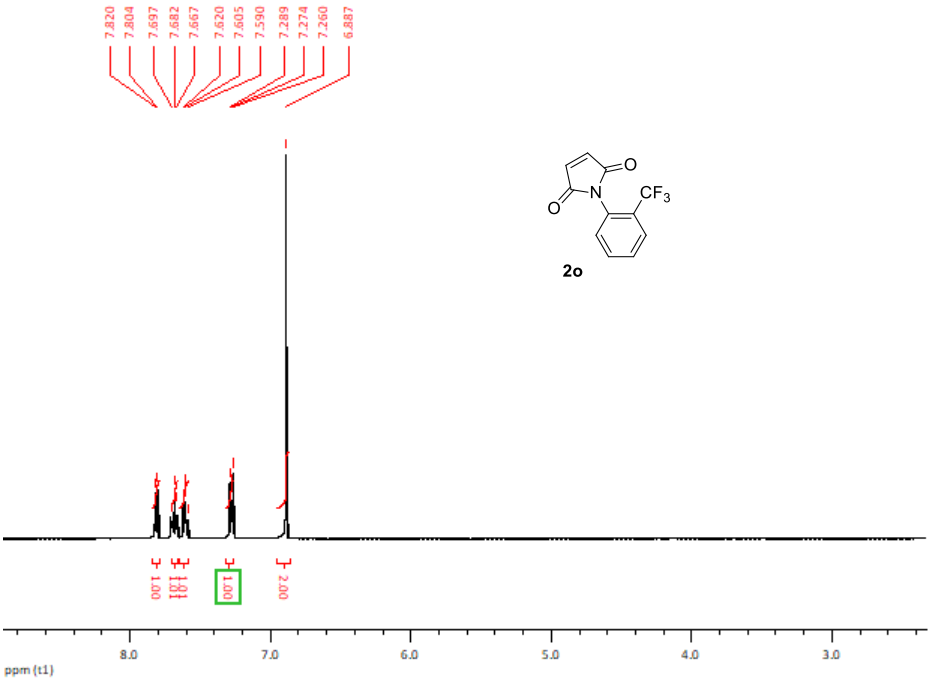
N-(2-methylphenyl)maleimide (**2n**),
^{1}H-NMR, 500 MHz, $CDCl_3$



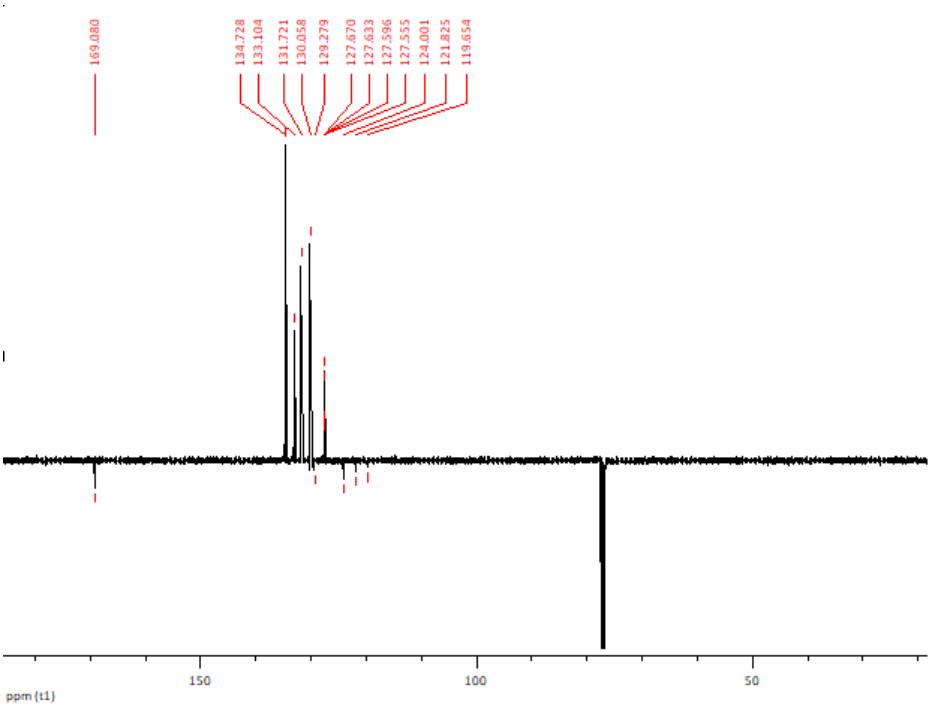
^{13}C-NMR, 125 MHz, $CDCl_3$



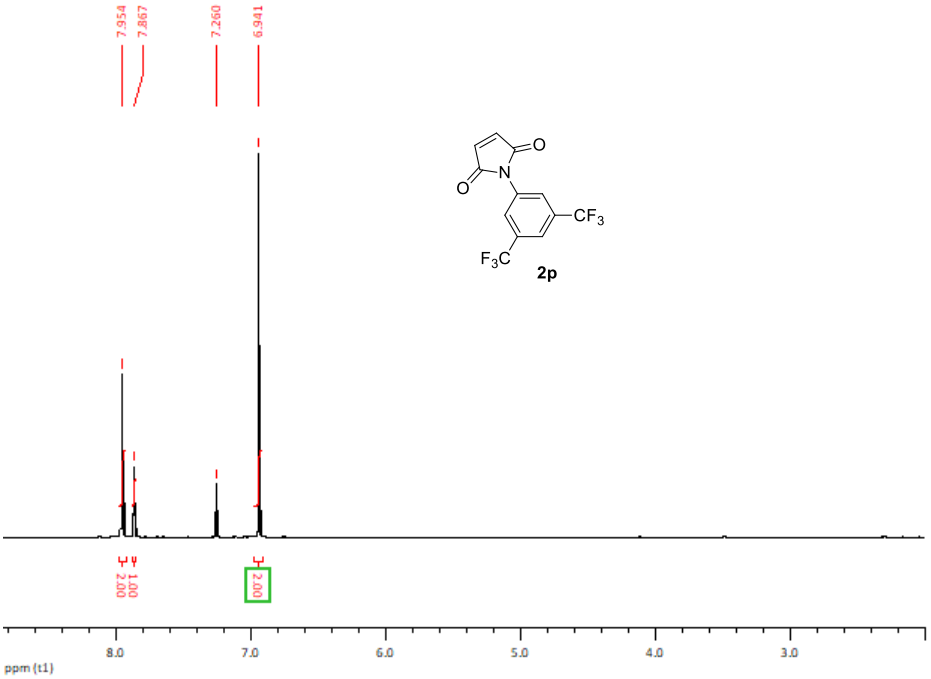
N-((2-trifluoromethyl)phenyl)maleimide (**2o**),
^{1}H-NMR, 500 MHz, $CDCl_3$



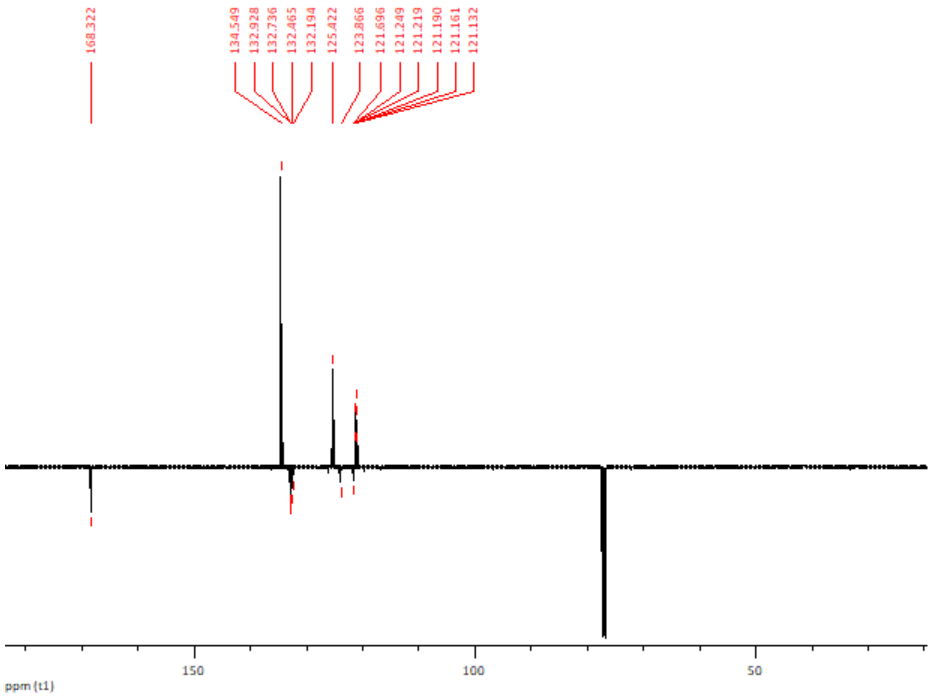
^{13}C-NMR, 125 MHz, $CDCl_3$



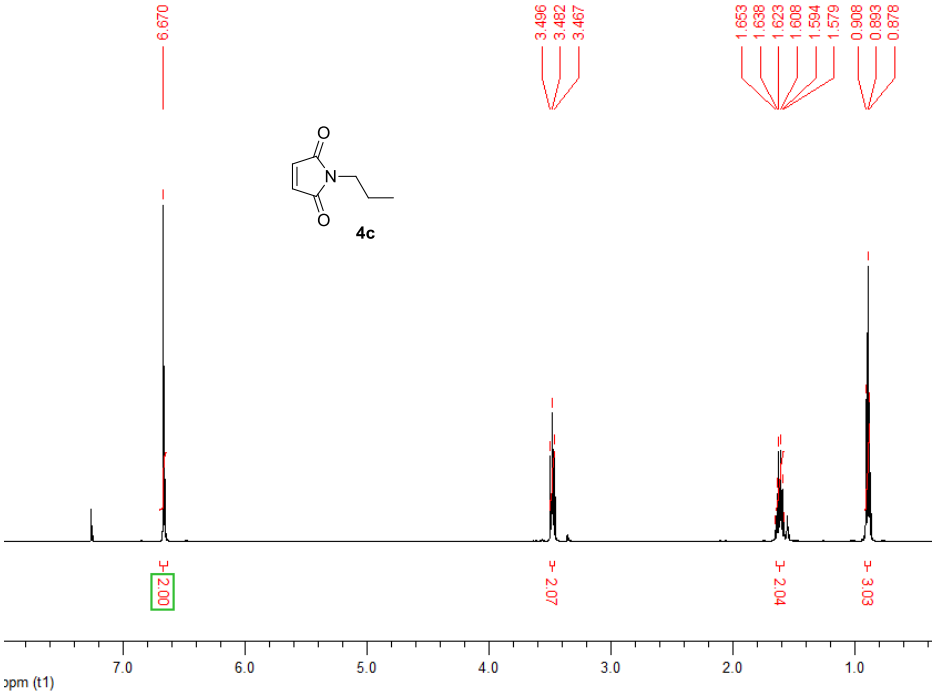
N-((3,5-bistrifluoromethyl)phenyl)maleimide (**2p**),
^{1}H-NMR, 500 MHz, $CDCl_3$



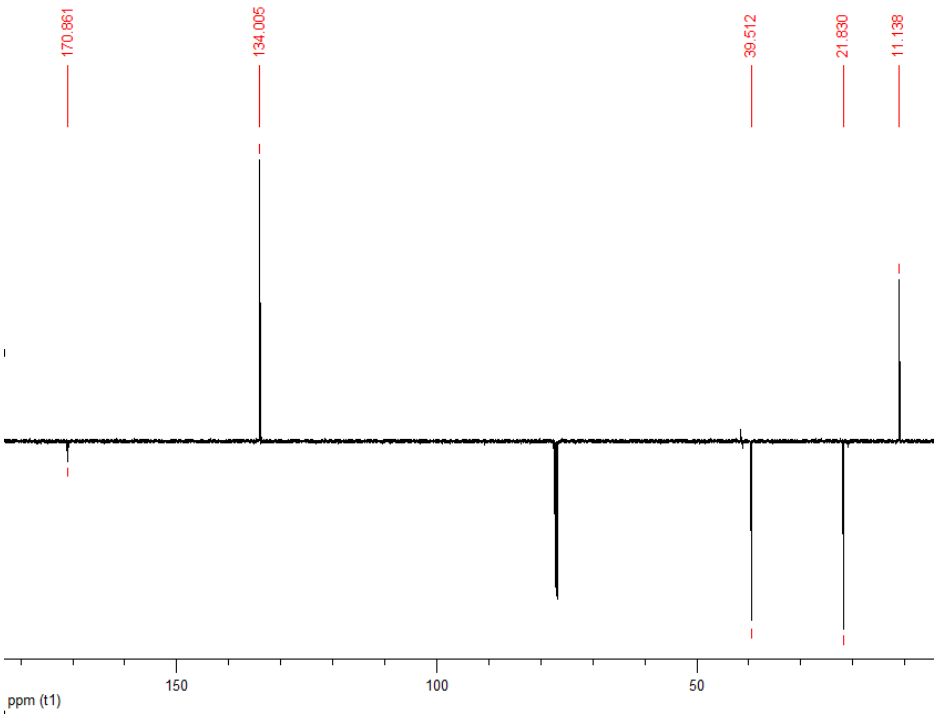
^{13}C-NMR, 125 MHz, $CDCl_3$



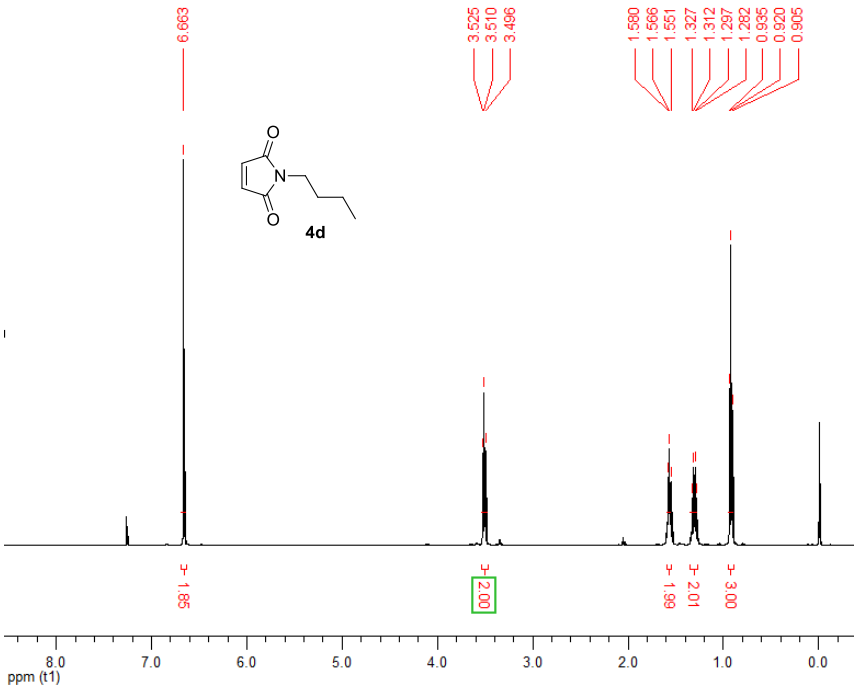
N-propylmaleimide (**4c**),
^{1}H-NMR, 500 MHz, $CDCl_3$



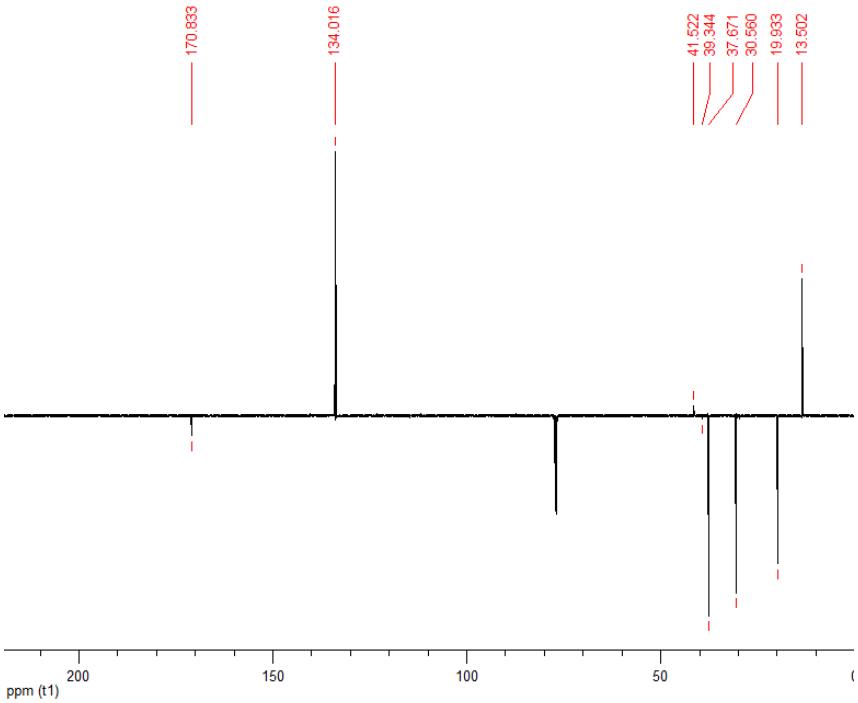
^{13}C-NMR, 125 MHz, $CDCl_3$



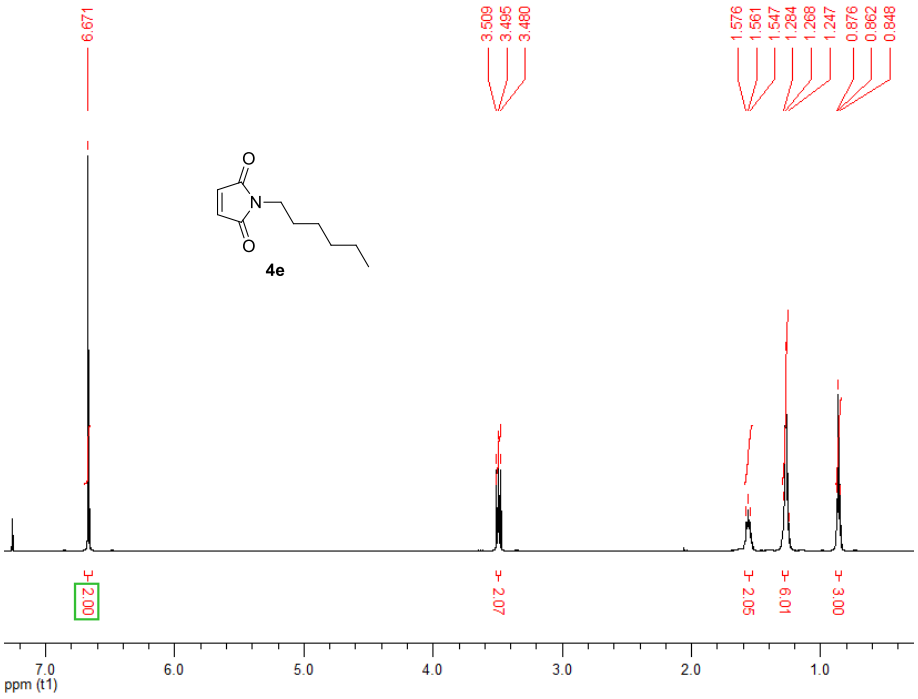
N-butylmaleimide (**4d**),
^{1}H-NMR, 500 MHz, $CDCl_3$



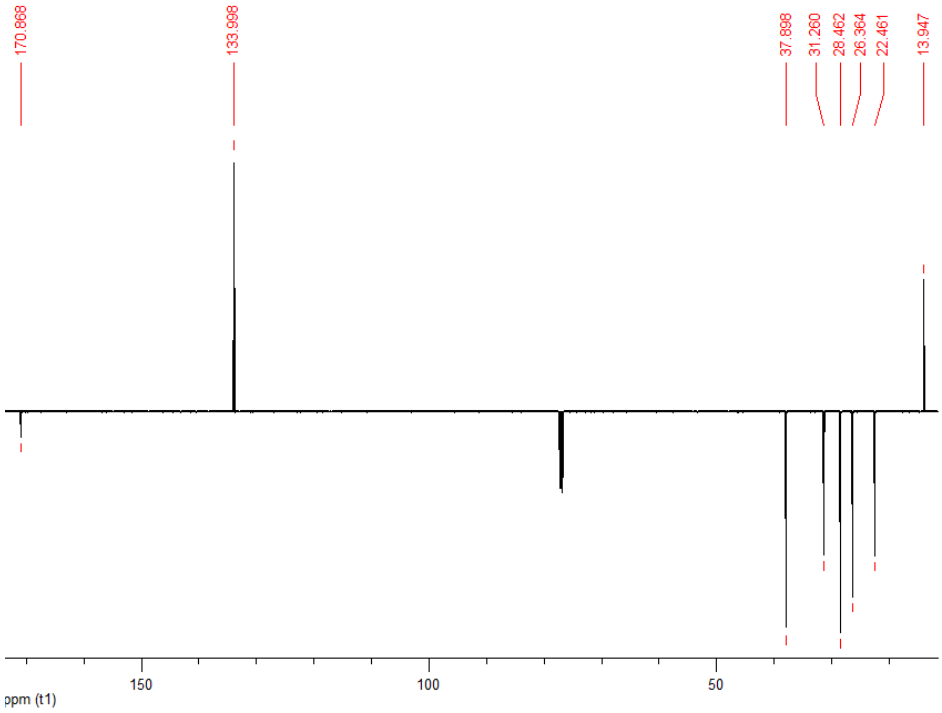
^{13}C-NMR, 125 MHz, $CDCl_3$



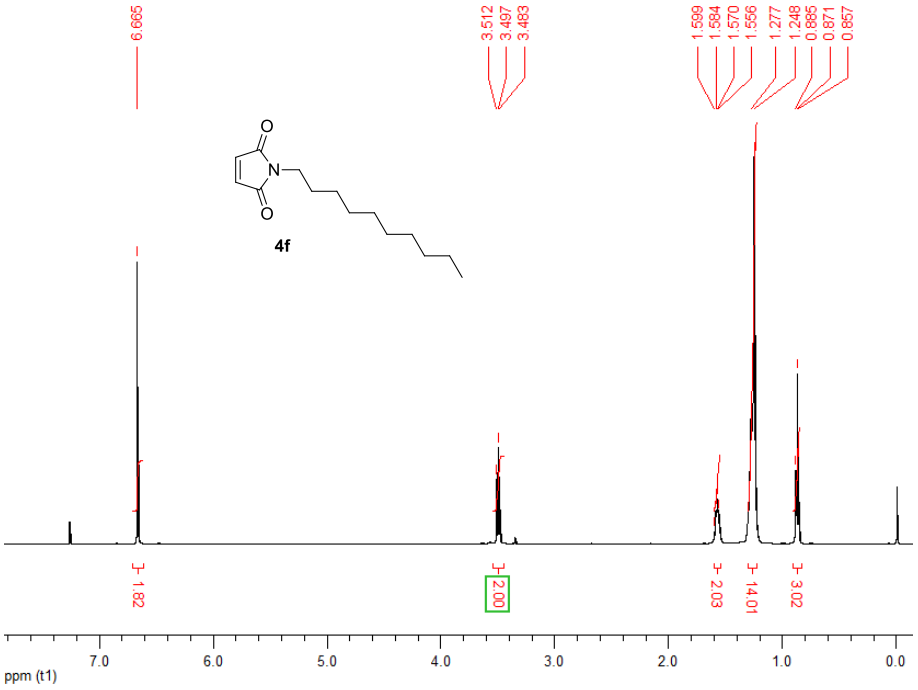
N-hexylmaleimide (**4e**),
^{1}H-NMR, 500 MHz, $CDCl_3$



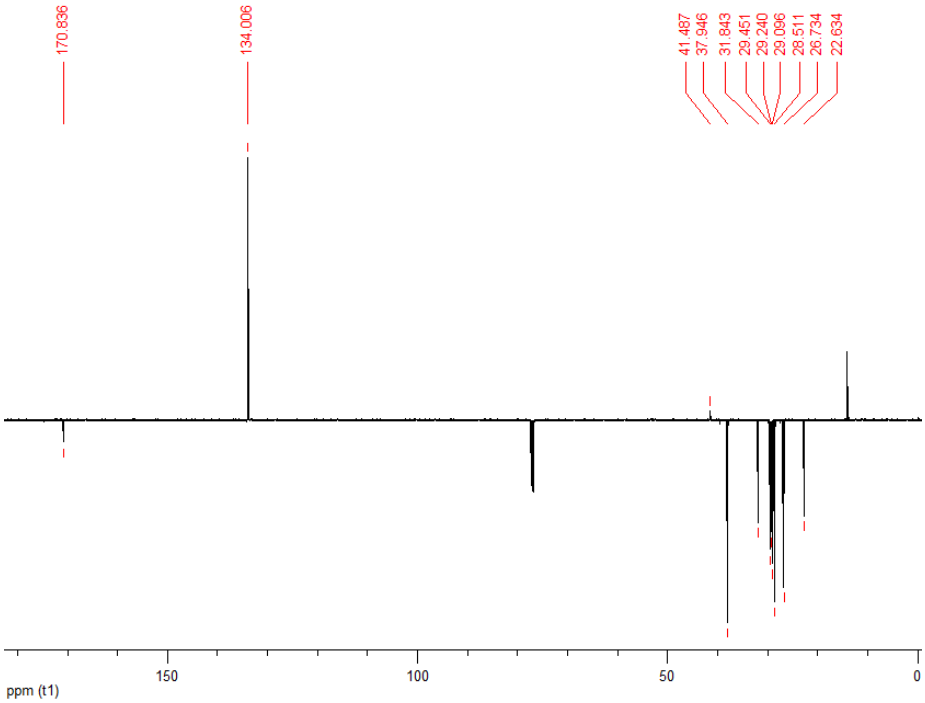
^{13}C-NMR, 125 MHz, $CDCl_3$



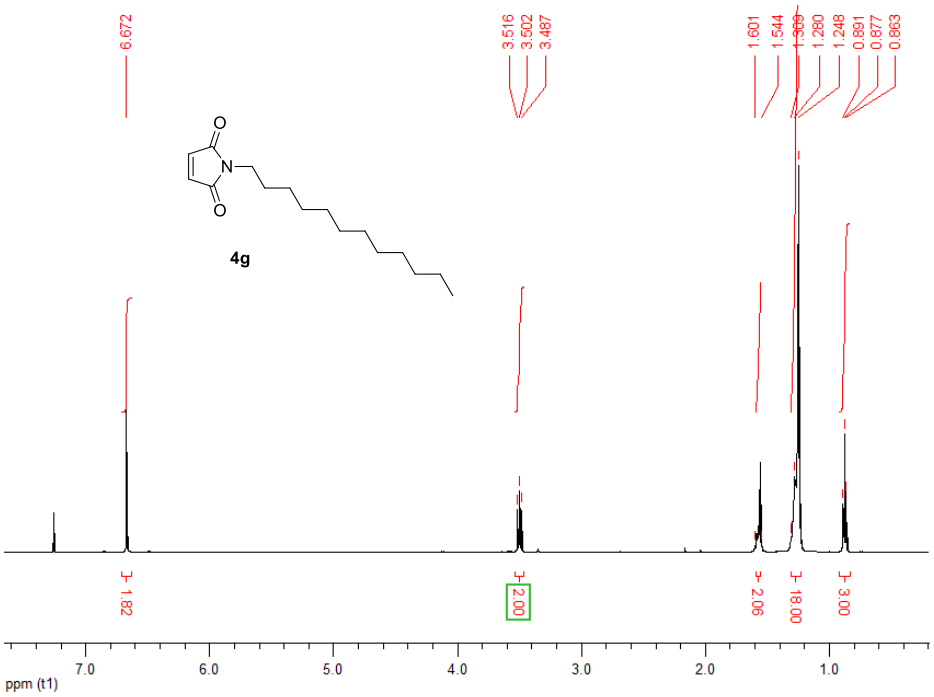
N-decylmaleimide (**4f**),
^{1}H-NMR, 500 MHz, $CDCl_3$



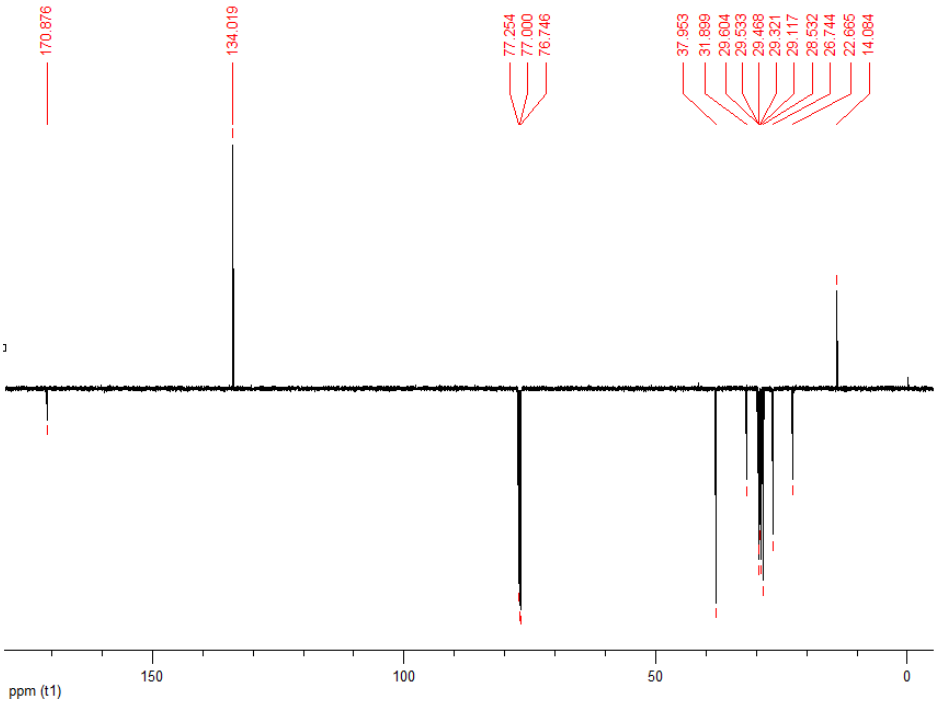
^{13}C-NMR, 125 MHz, $CDCl_3$



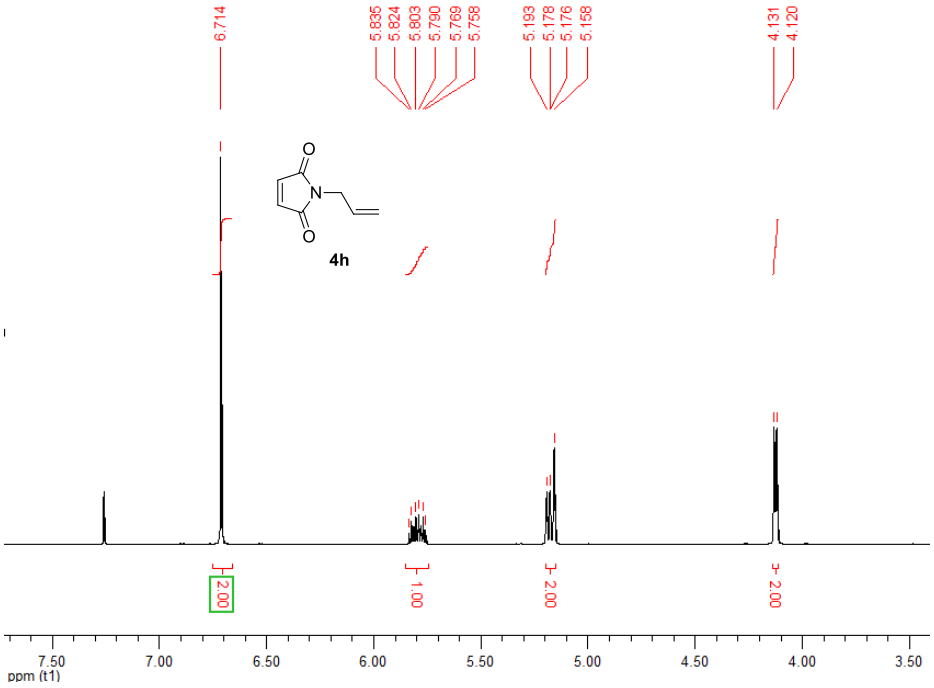
N-dodecylmaleimide (**4g**),
^{1}H-NMR, 500 MHz, $CDCl_3$



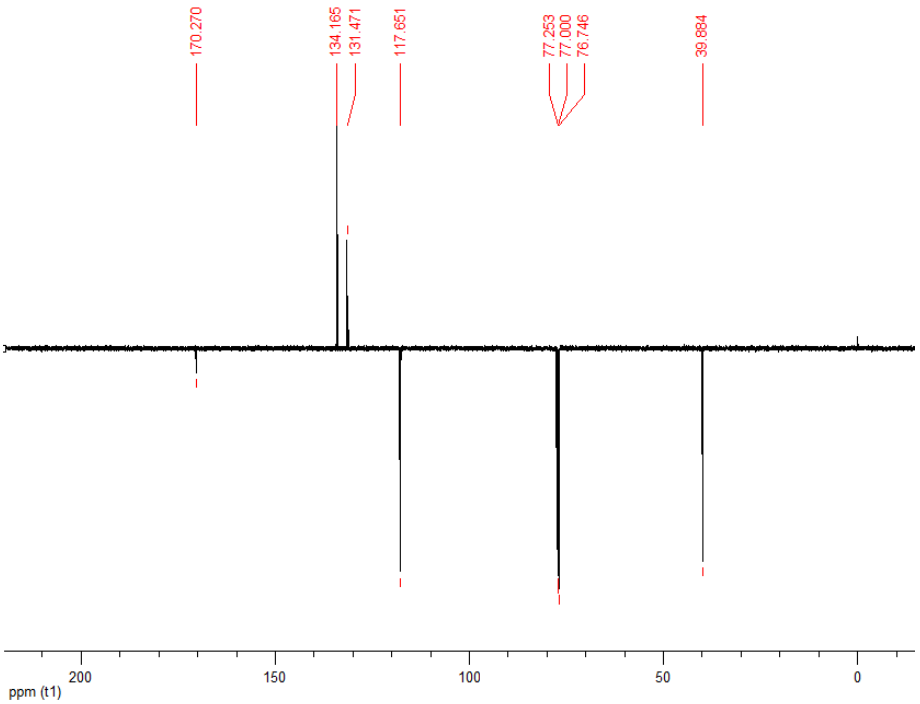
^{13}C-NMR, 125 MHz, $CDCl_3$



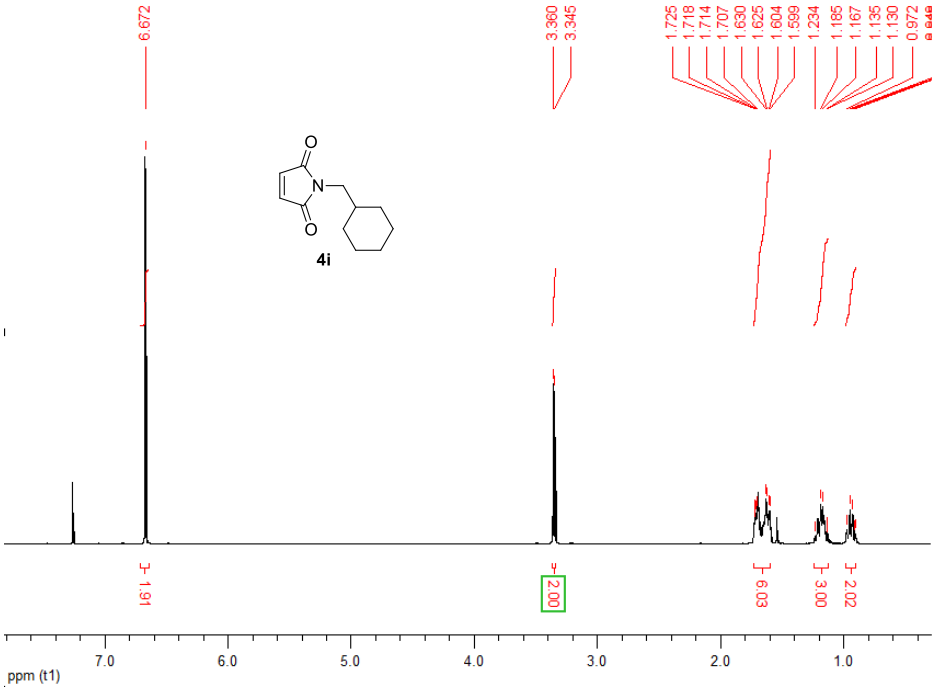
N-allylmaleimide (**4h**),
^{1}H-NMR, 500 MHz, $CDCl_3$



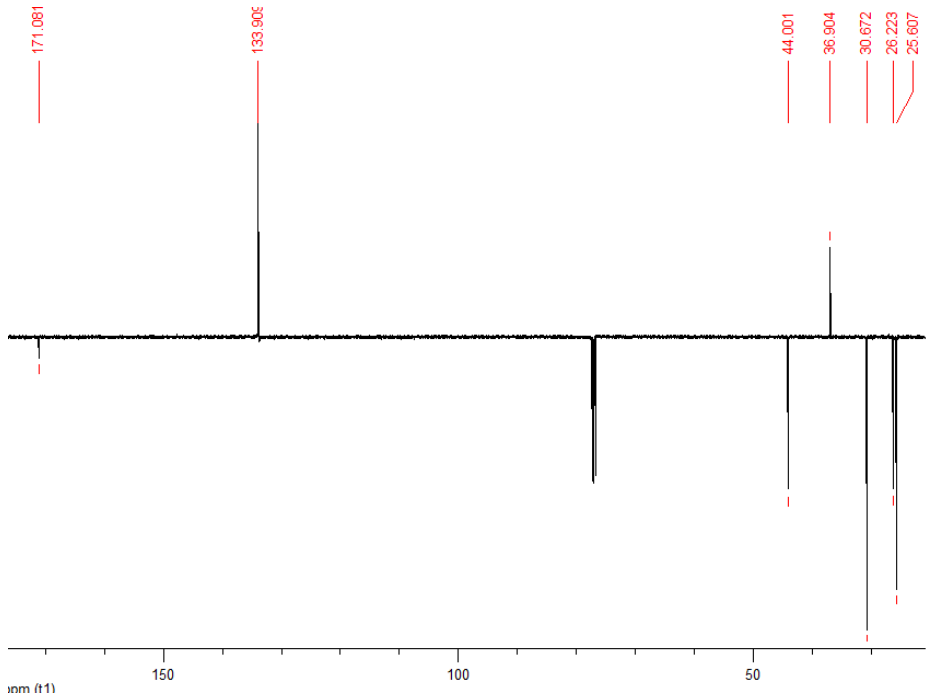
^{13}C-NMR, 125 MHz, $CDCl_3$



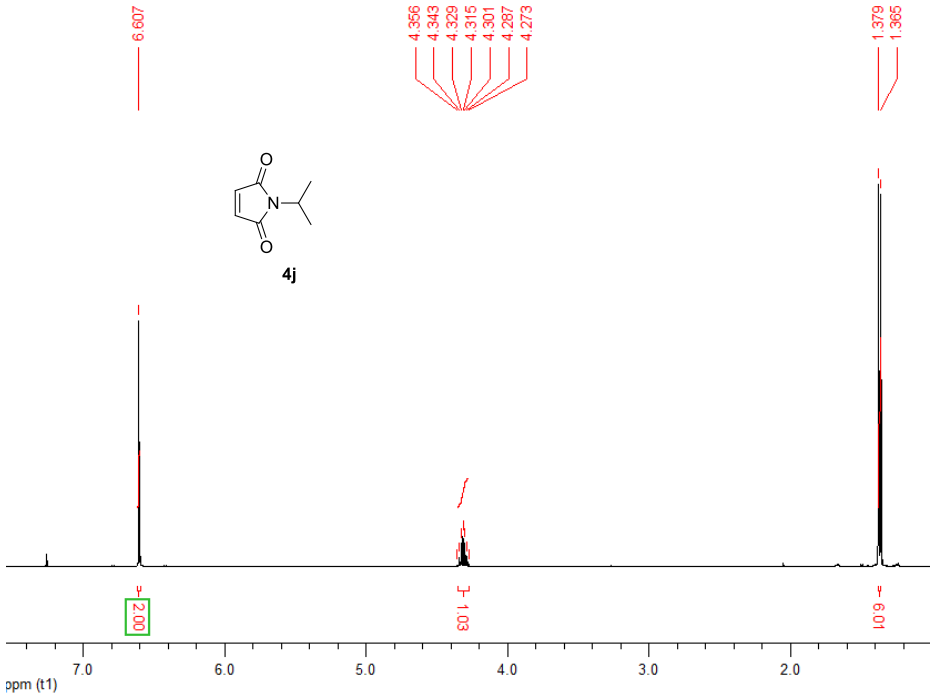
N-(cyclohexylmethyl)maleimide (**4i**),
^{1}H-NMR, 500 MHz, $CDCl_3$



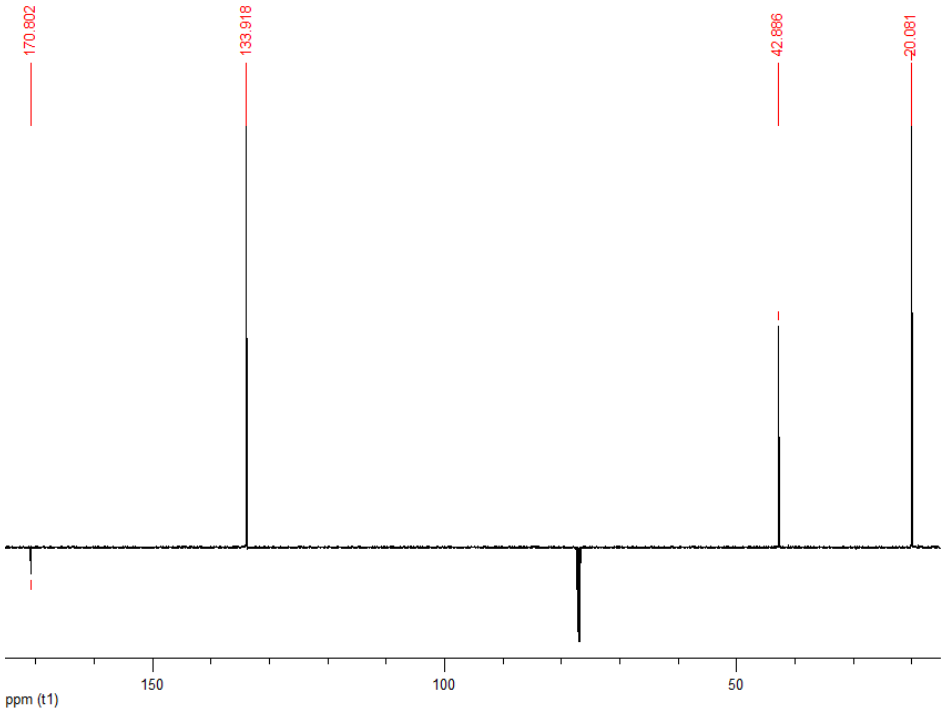
^{13}C-NMR, 125 MHz, $CDCl_3$



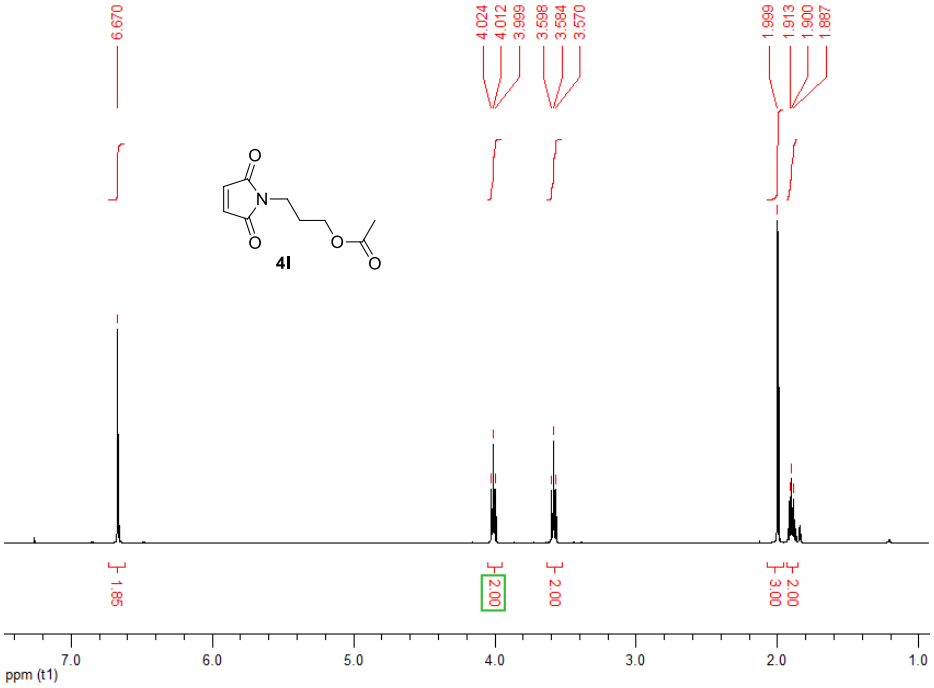
N-isopropylmaleimide (**4j**),
^{1}H-NMR, 500 MHz, $CDCl_3$



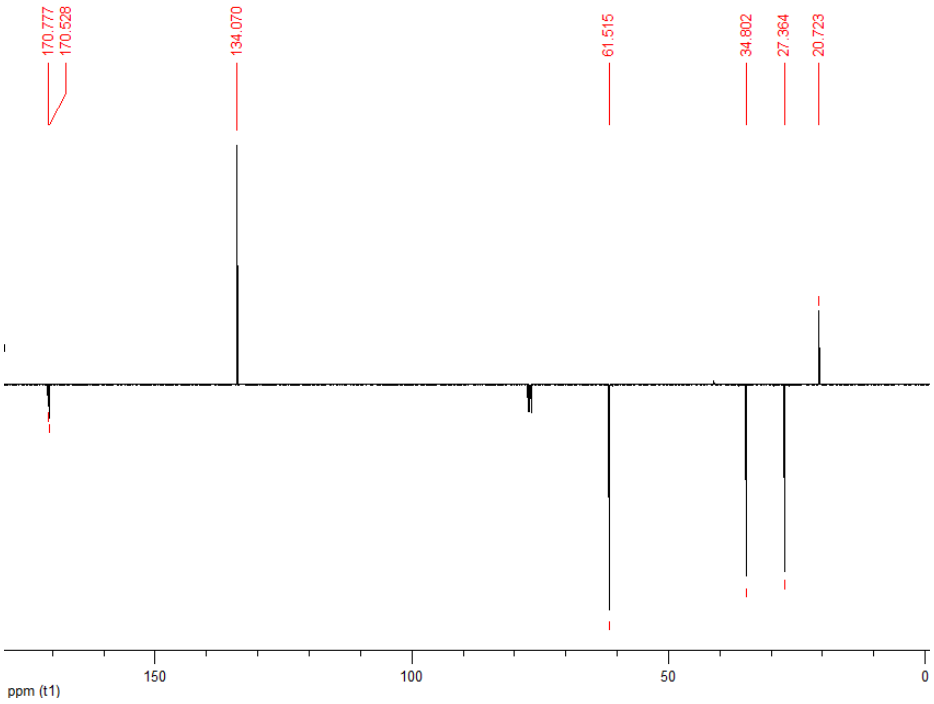
^{13}C-NMR, 125 MHz, $CDCl_3$



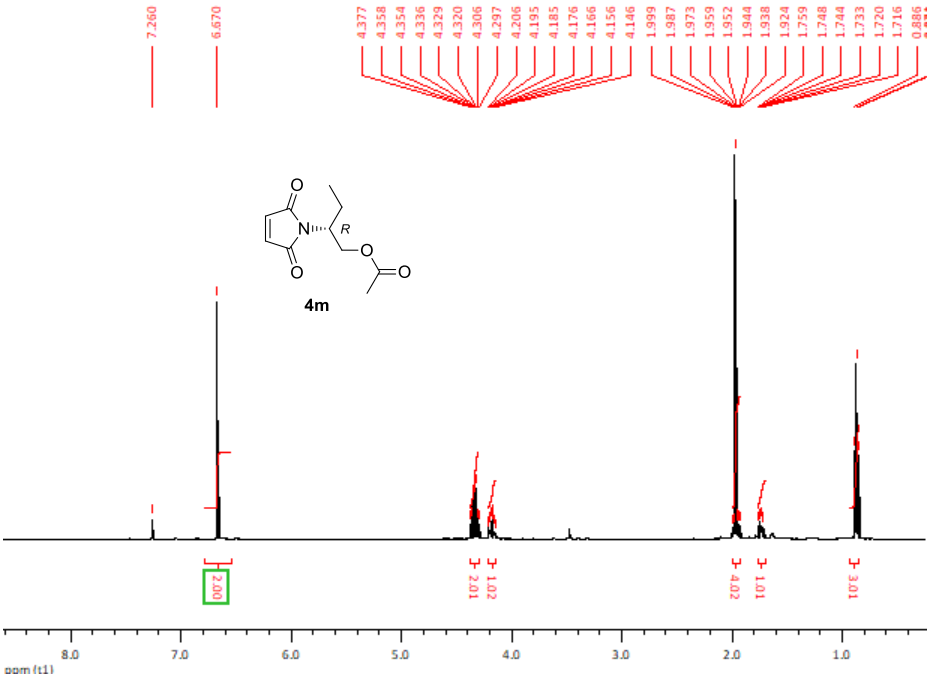
N-(3-acetoxypropyl)maleimide (**4l**),
^{1}H-NMR, 500 MHz, $CDCl_3$



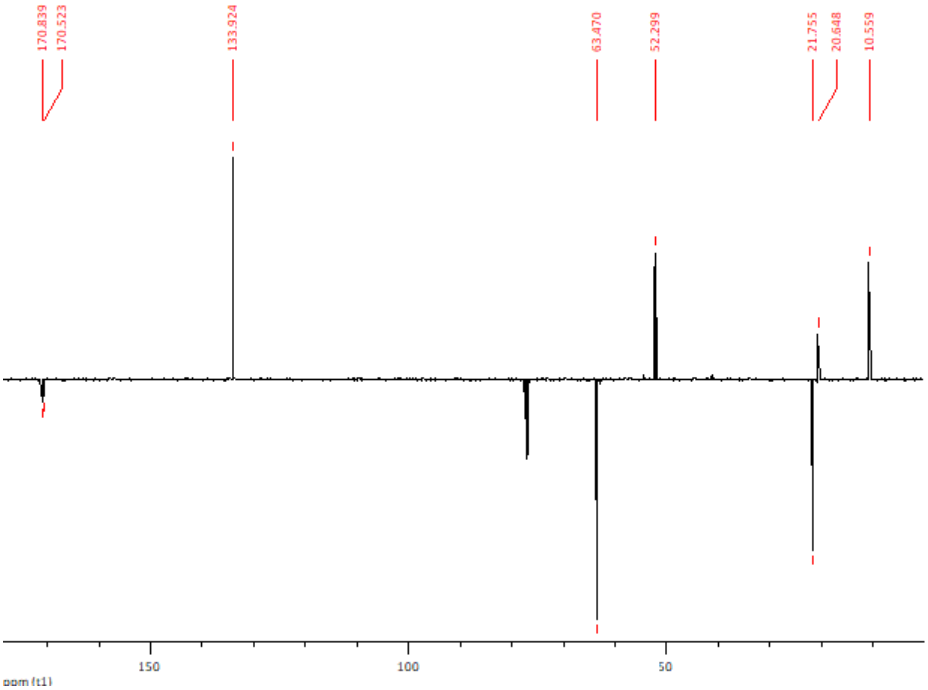
^{13}C-NMR, 125 MHz, $CDCl_3$



(*R*)-*N*-[(1-acetyloxymethyl)propyl]maleimide (**4m**),
^{1}H-NMR, 500 MHz, $CDCl_3$

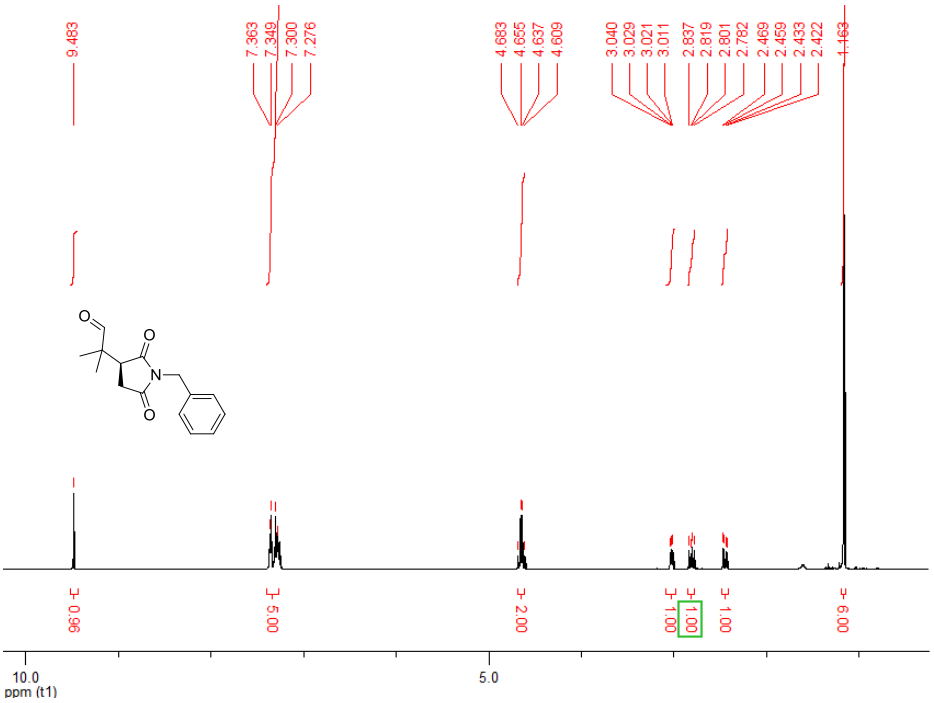


^{13}C-NMR, 125 MHz, $CDCl_3$

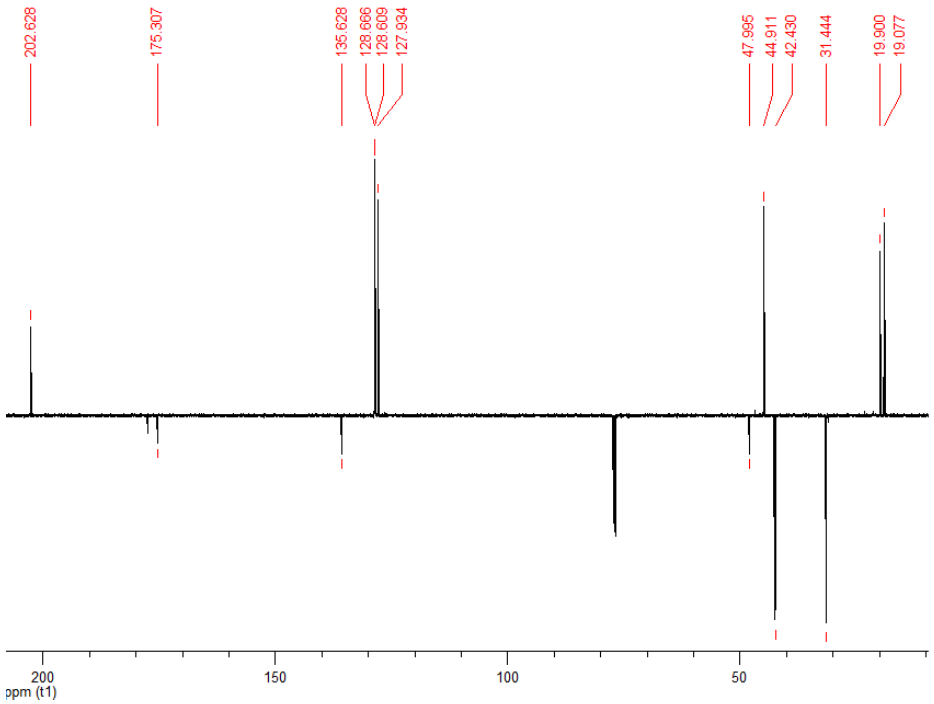


14.2. ^{1}H- and ^{13}C-NMR spectra of the Michael adducts

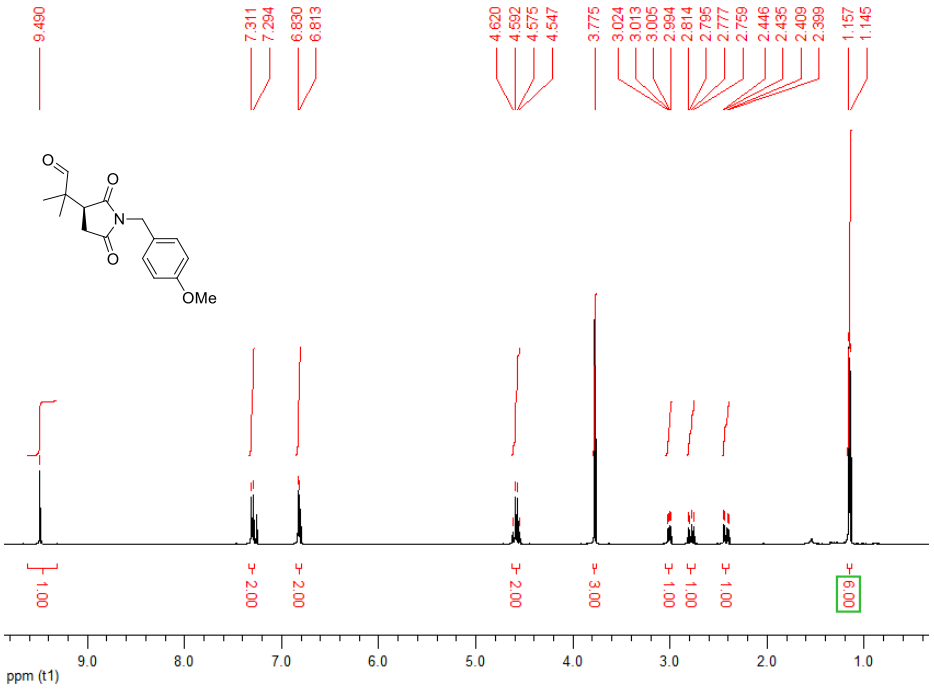
(*S*)-2-(1-benzyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3a**),
^{1}H-NMR, 500 MHz, $CDCl_3$



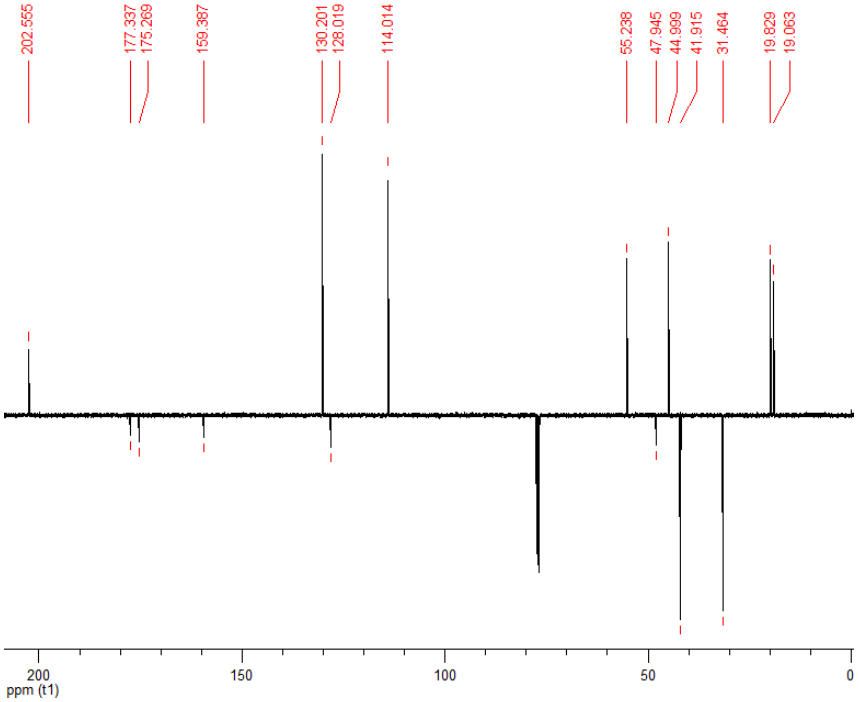
^{13}C-NMR, 125 MHz, $CDCl_3$



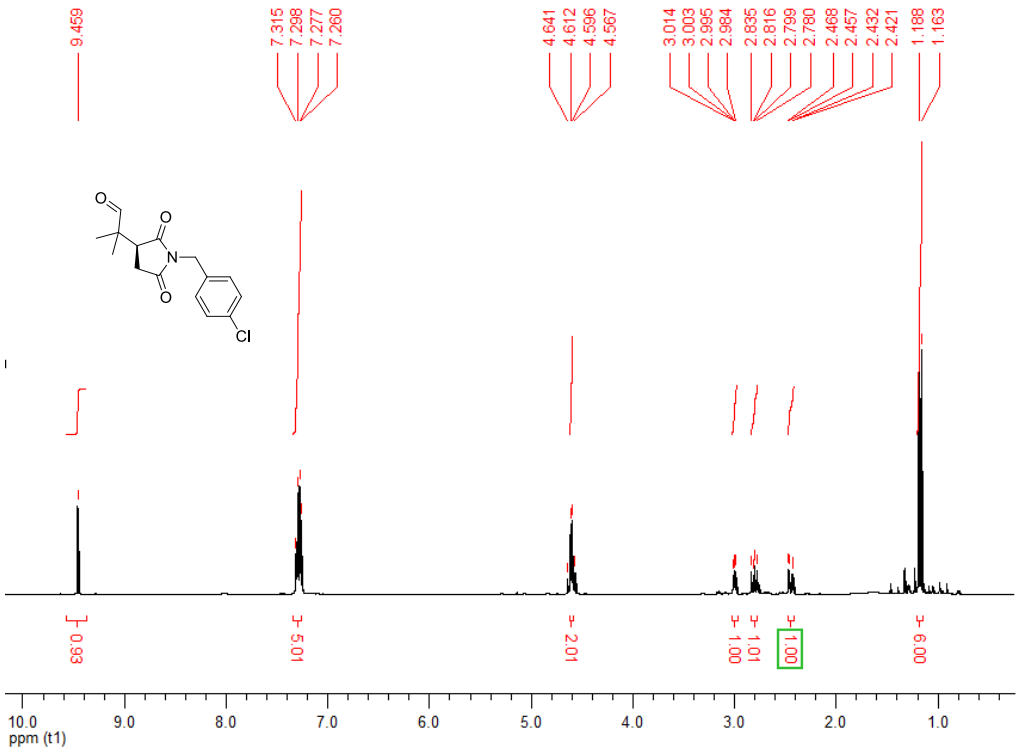
(*S*)-2-(1-(4-methoxybenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3b**),
^{1}H-NMR, 500 MHz, $CDCl_3$



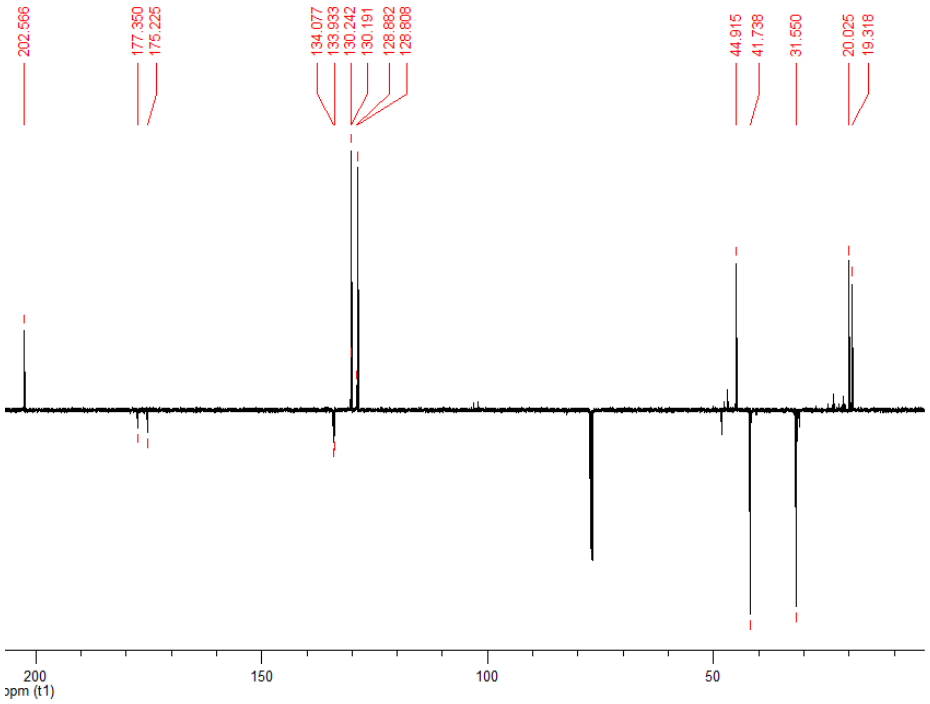
^{13}C-NMR, 125 MHz, $CDCl_3$



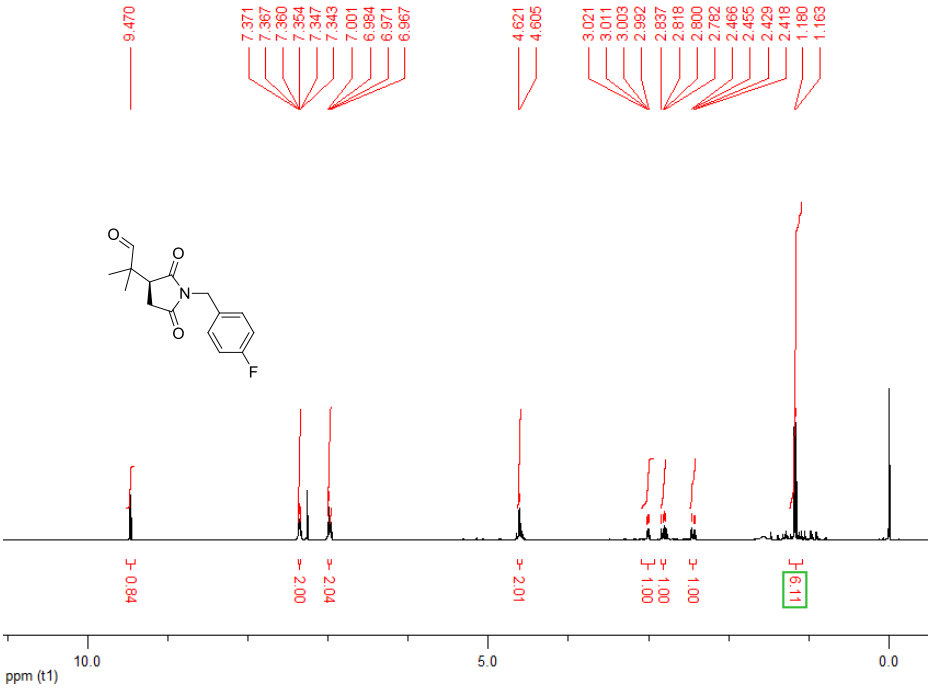
(*S*)-2-(1-(4-chlorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3c**),
^{1}H-NMR, 500 MHz, $CDCl_3$



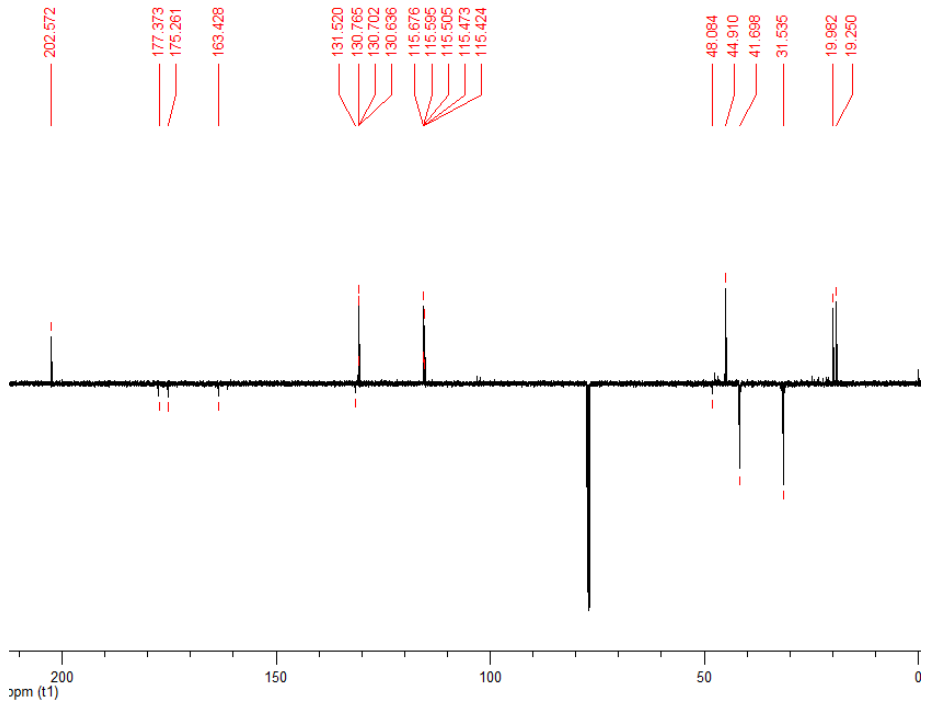
^{13}C-NMR, 125 MHz, $CDCl_3$



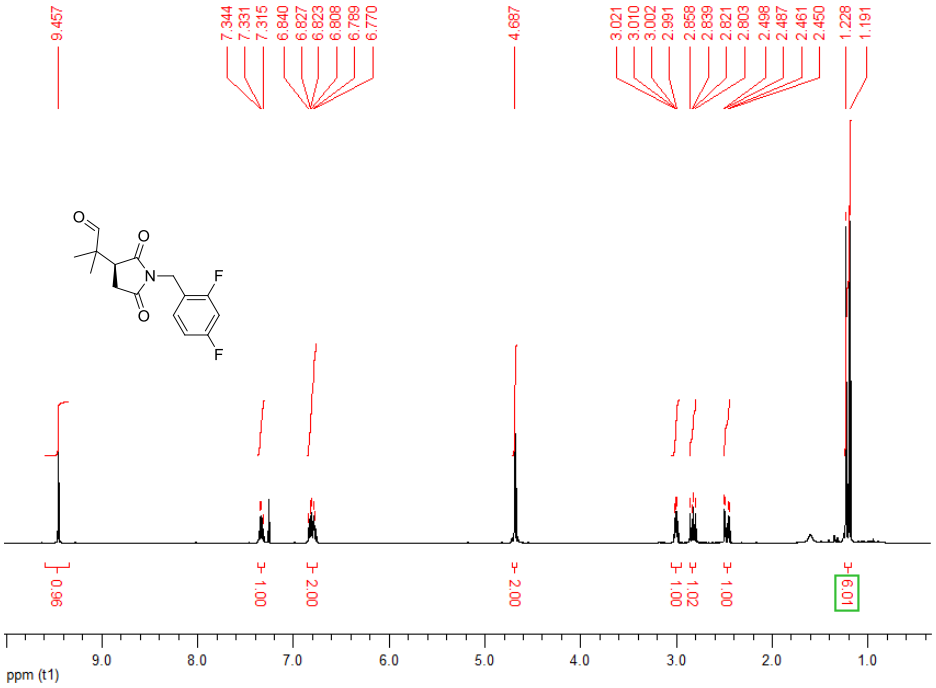
(*S*)-2-(1-(4-fluorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3d**),
^{1}H-NMR, 500 MHz, $CDCl_3$



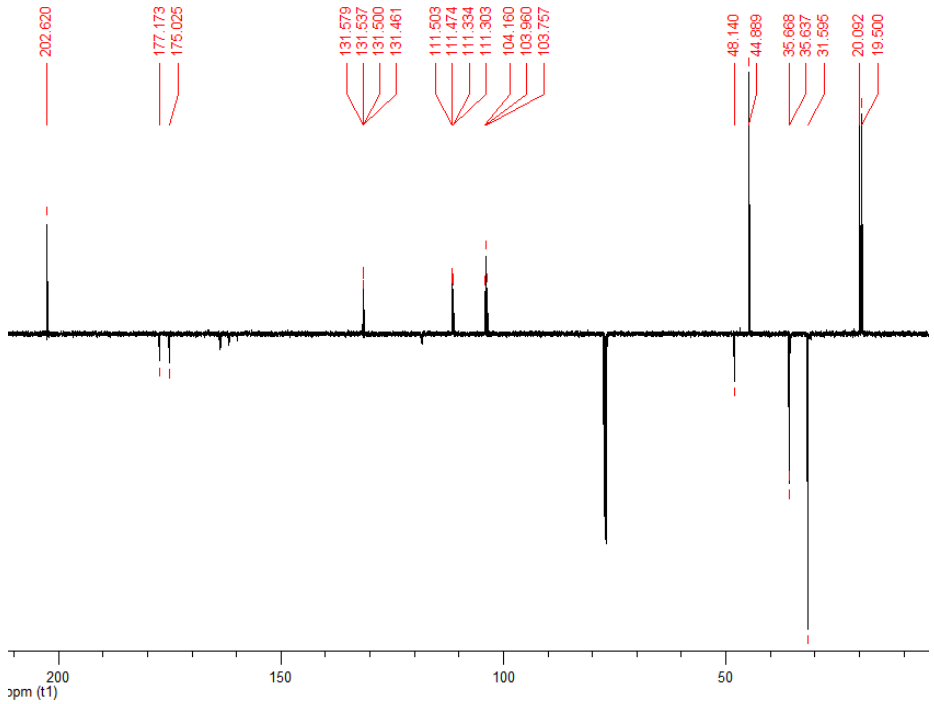
^{13}C-NMR, 125 MHz, $CDCl_3$



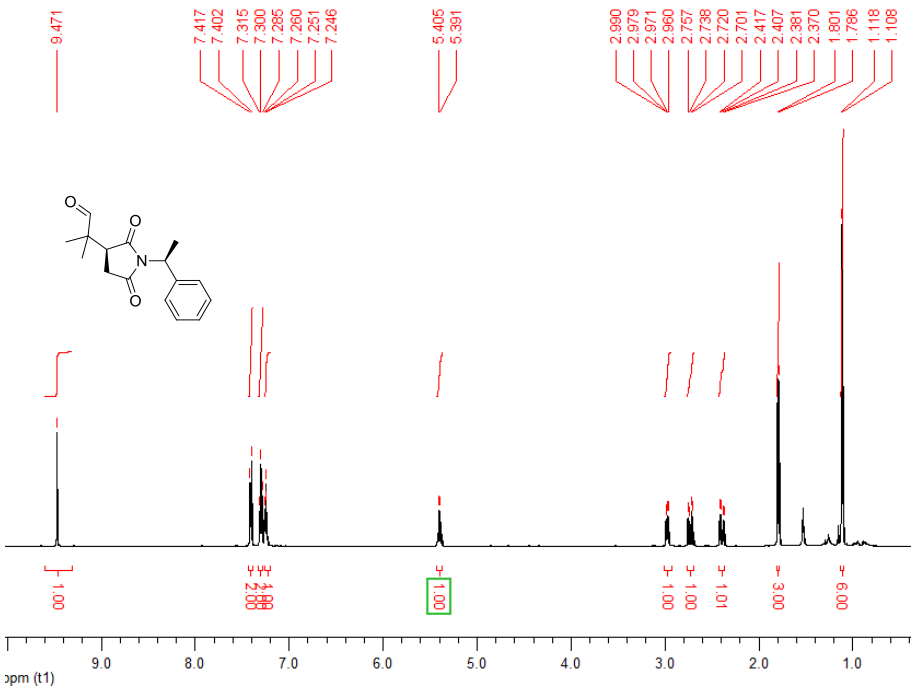
(*S*)-2-(1-(2,4-difluorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3e**),
^{1}H-NMR, 500 MHz, $CDCl_3$



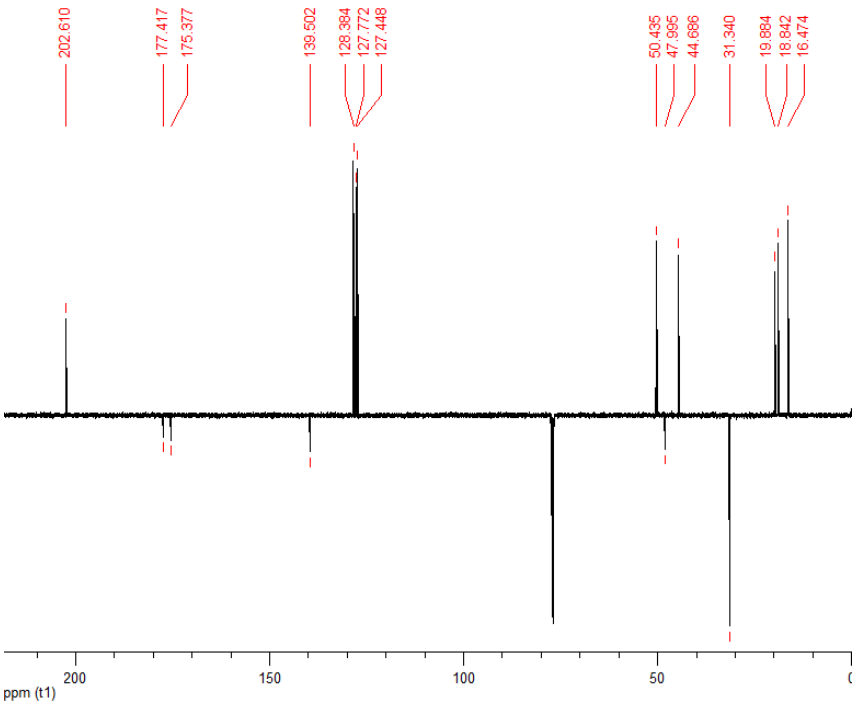
^{13}C-NMR, 125 MHz, $CDCl_3$



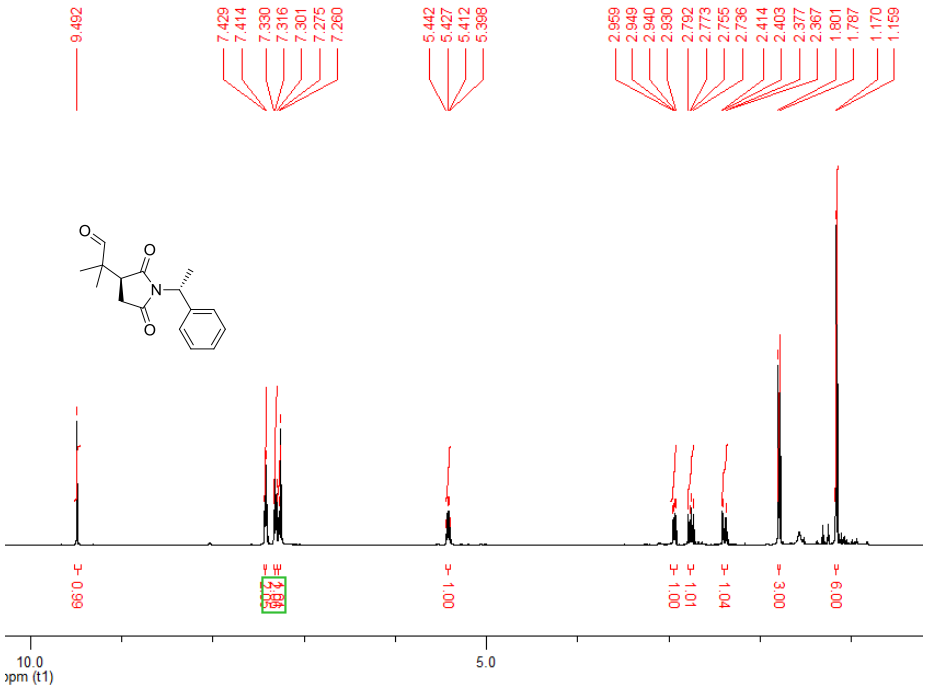
(*S*)-2-((*S*)-1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal ((*S*,*S*)-**3f**),
^{1}H-NMR, 500 MHz, $CDCl_3$



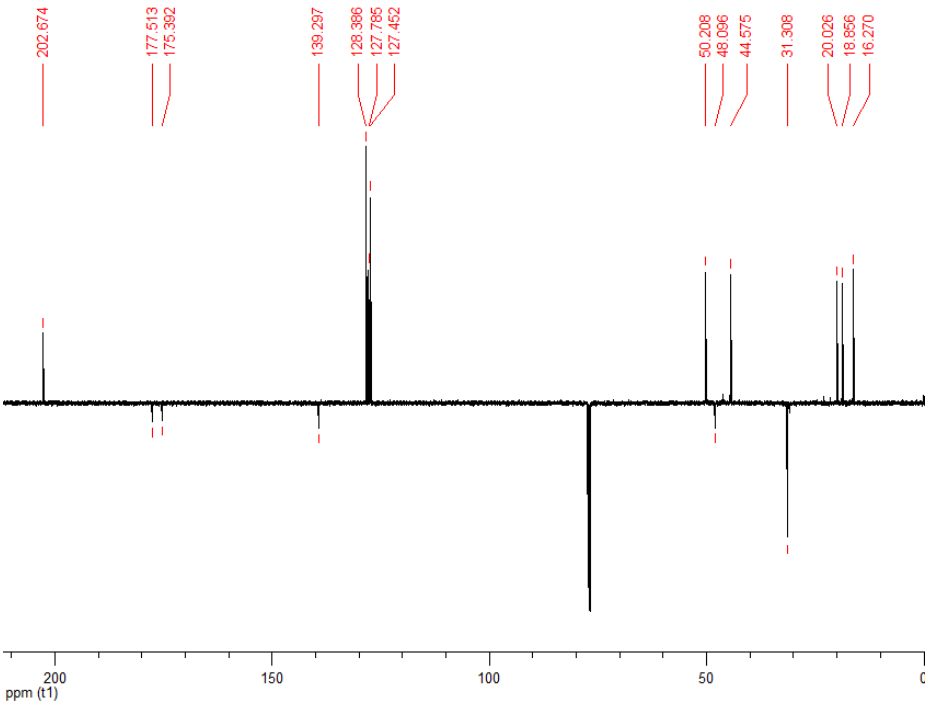
^{13}C-NMR, 125 MHz, $CDCl_3$



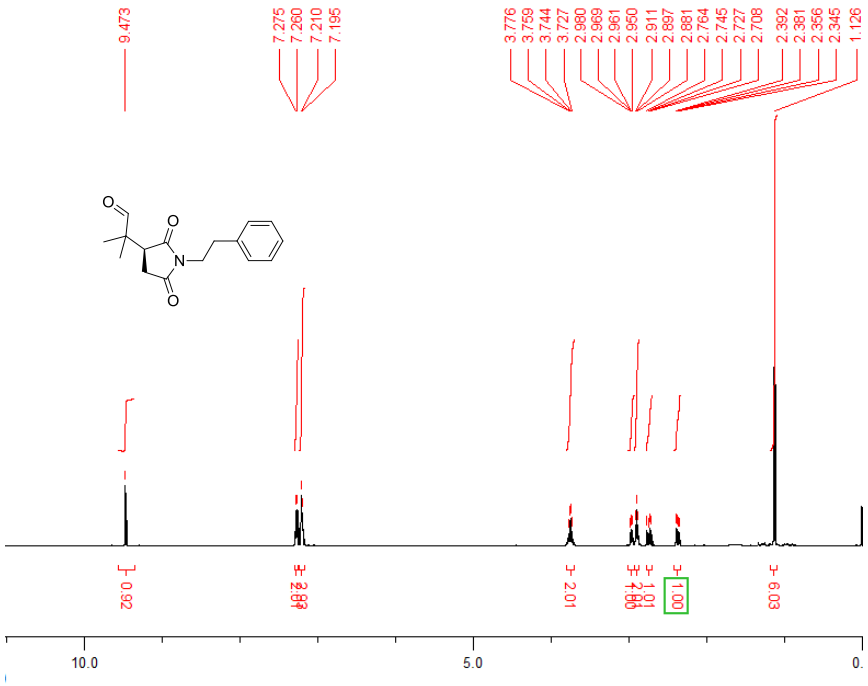
(*S*)-2-((*R*)-1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal ((*S*,*R*)-**3f**),
^{1}H-NMR, 500 MHz, $CDCl_3$



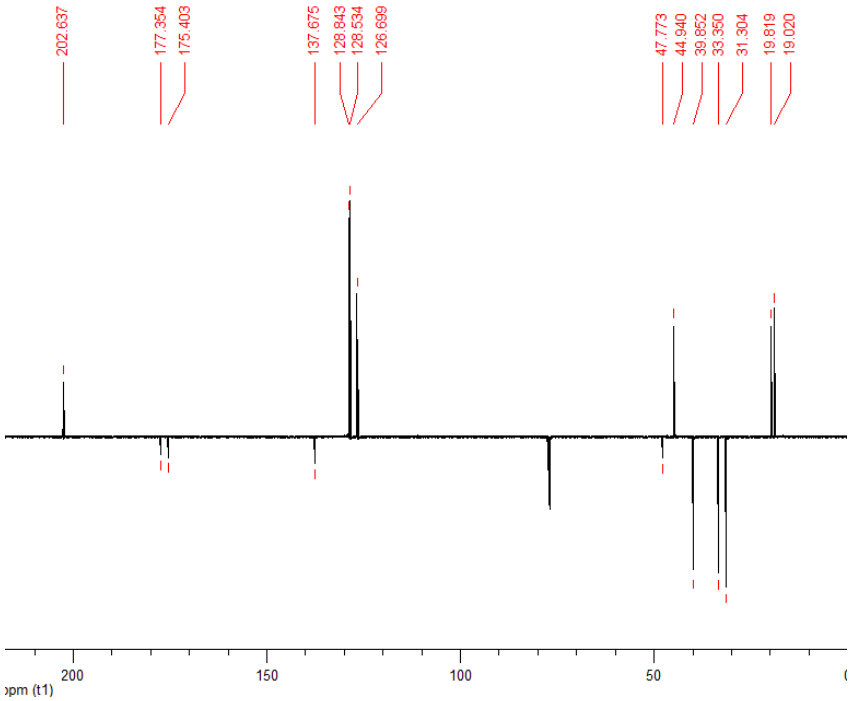
^{13}C-NMR, 125 MHz, $CDCl_3$



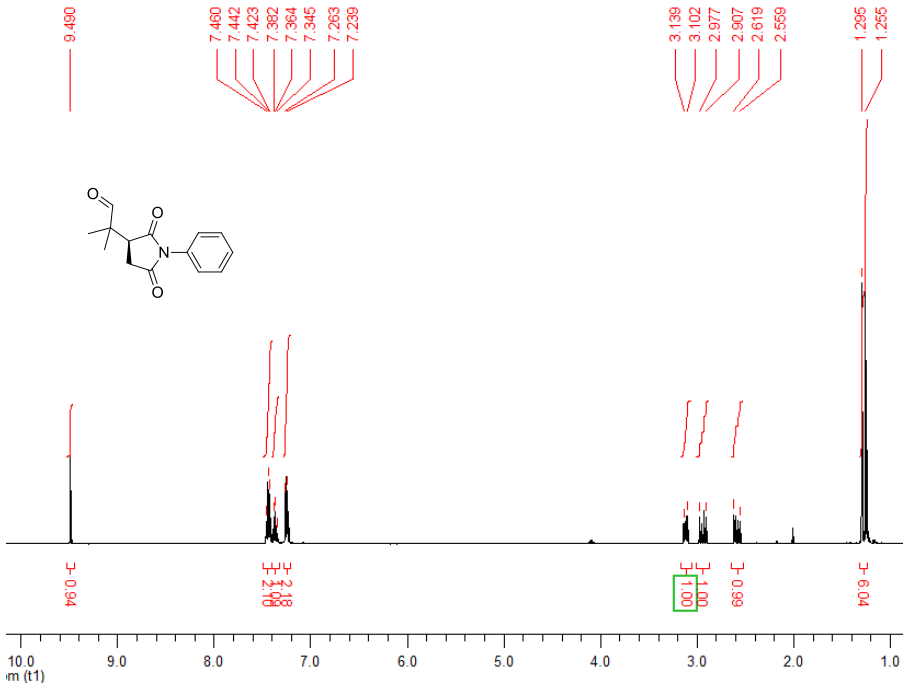
(*S*)-2-(1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3g**),
^{1}H-NMR, 500 MHz, $CDCl_3$



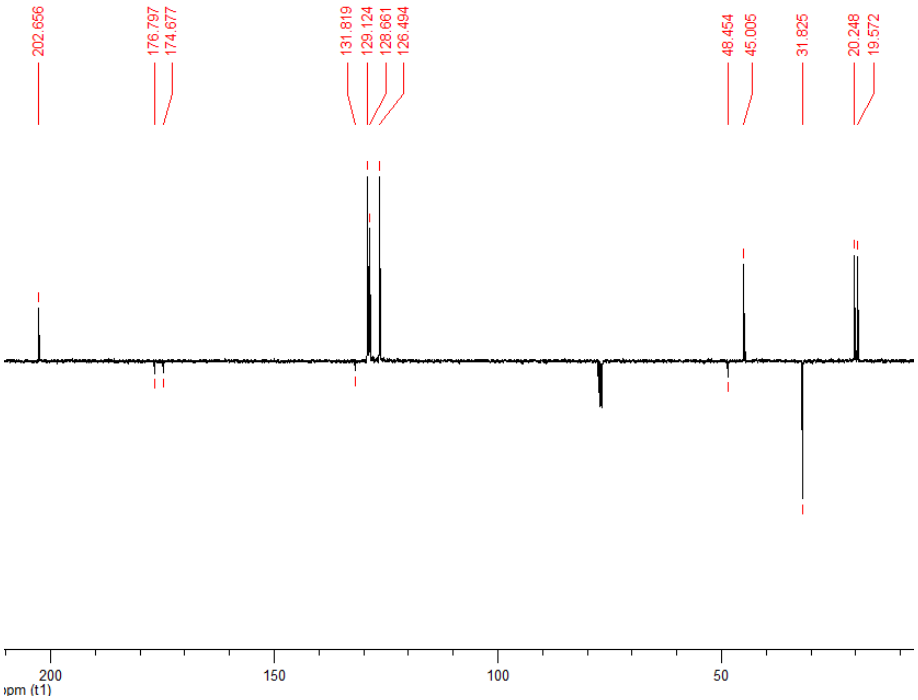
^{13}C-NMR, 125 MHz, $CDCl_3$



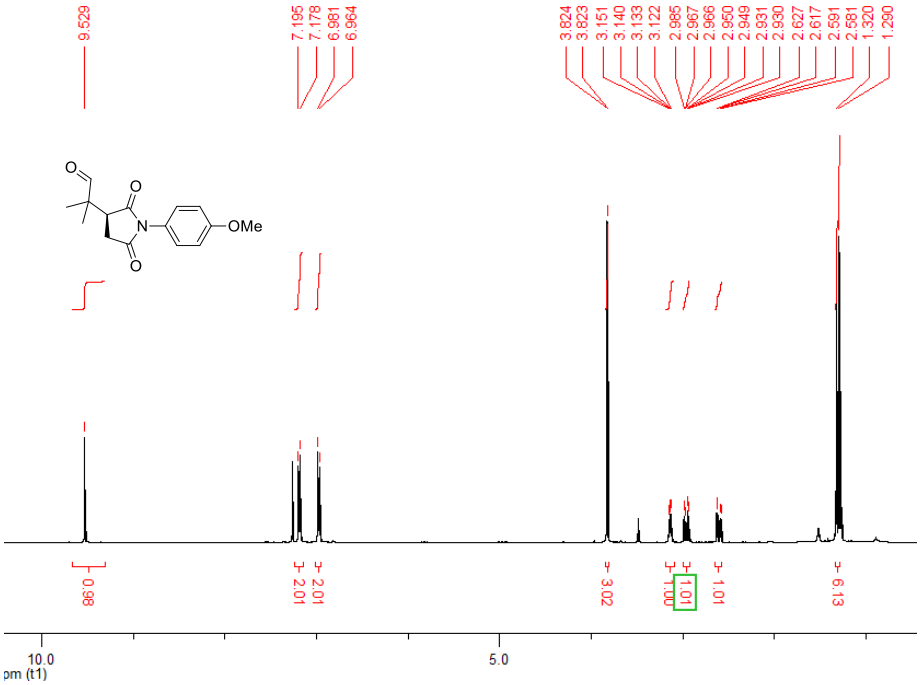
(*S*)-2-(1-phenyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3h**),
^{1}H-NMR, 500 MHz, $CDCl_3$



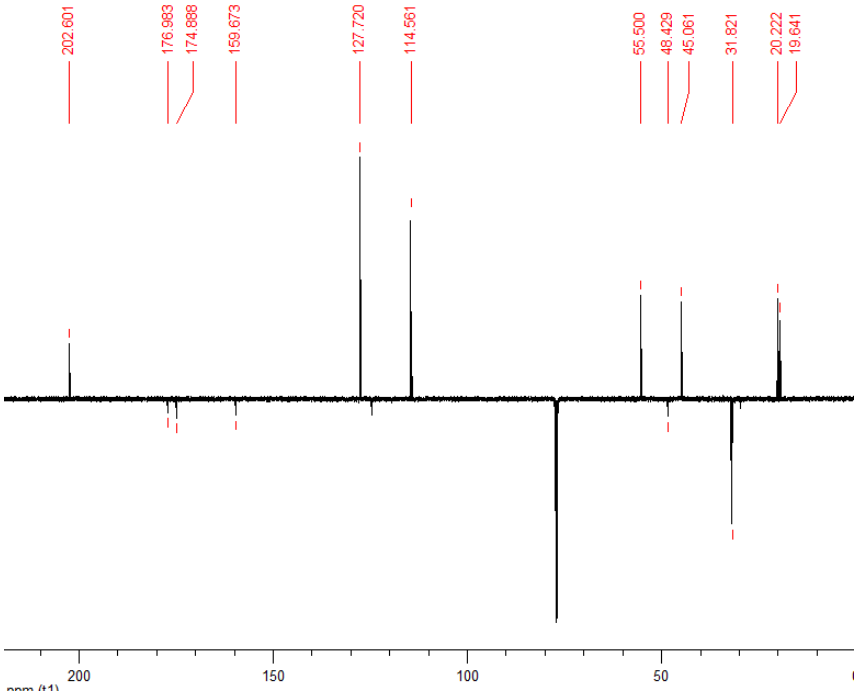
^{13}C-NMR, 125 MHz, $CDCl_3$



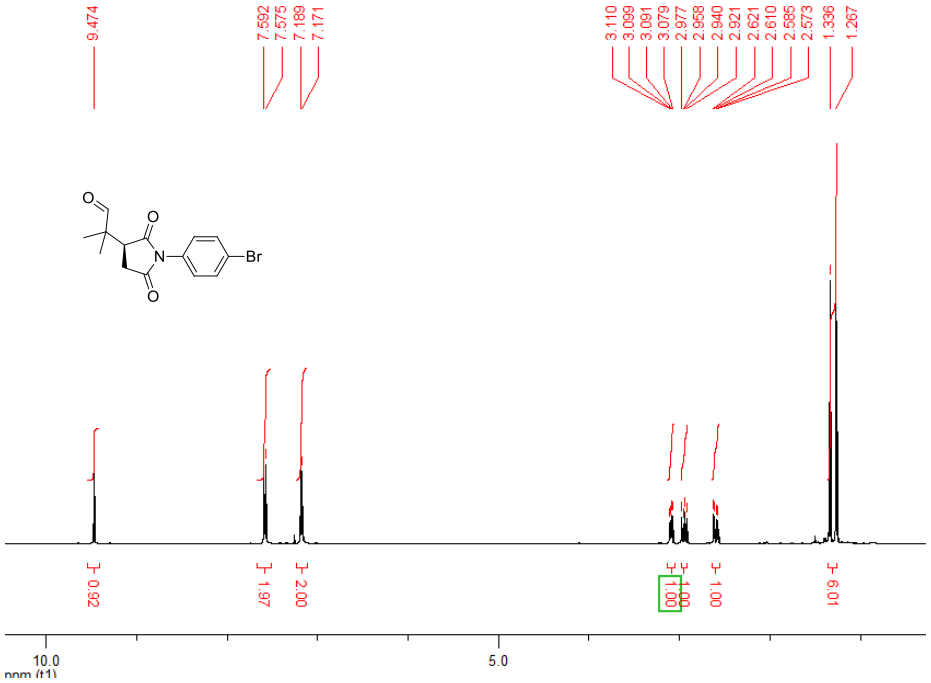
(*S*)-2-((1-(4-methoxyphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3i**),
^{1}H-NMR, 500 MHz, $CDCl_3$



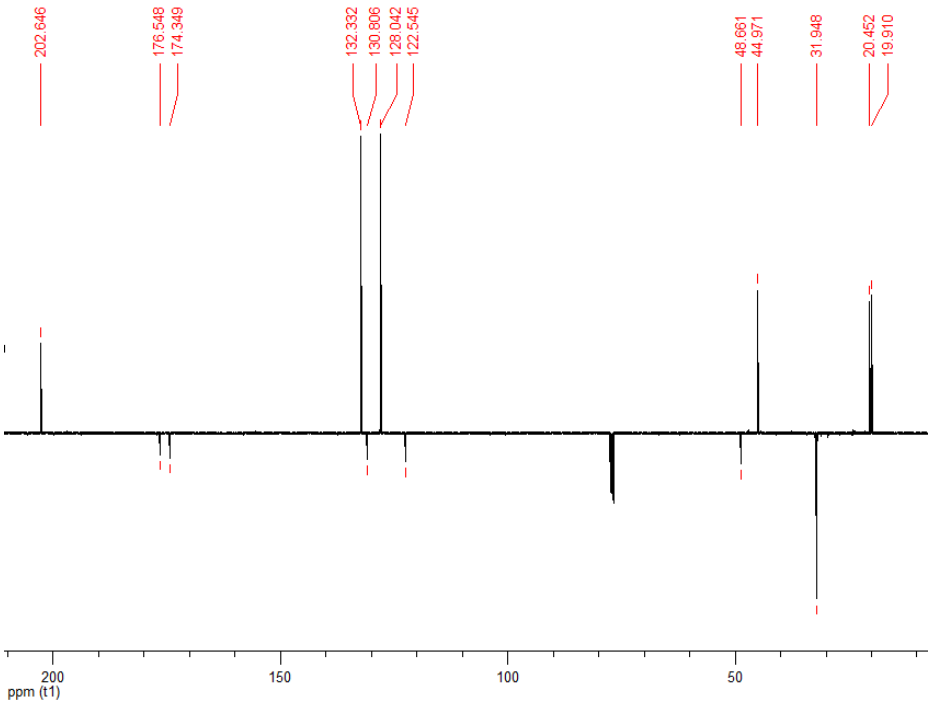
^{13}C-NMR, 125 MHz, $CDCl_3$



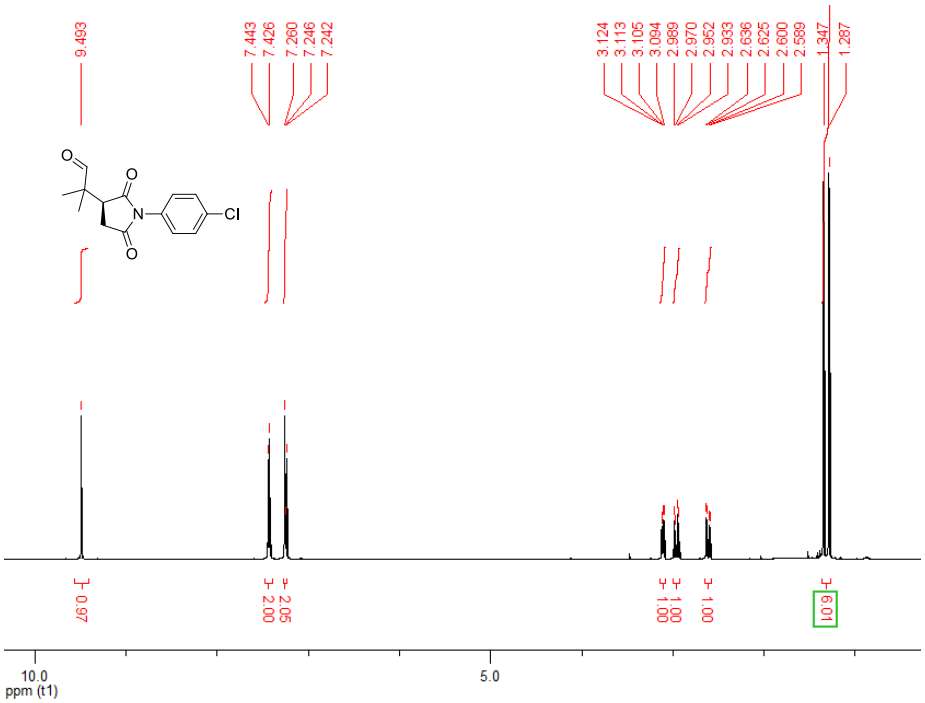
(*S*)-2-(1-(4-bromophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3j**),
^{1}H-NMR, 500 MHz, $CDCl_3$



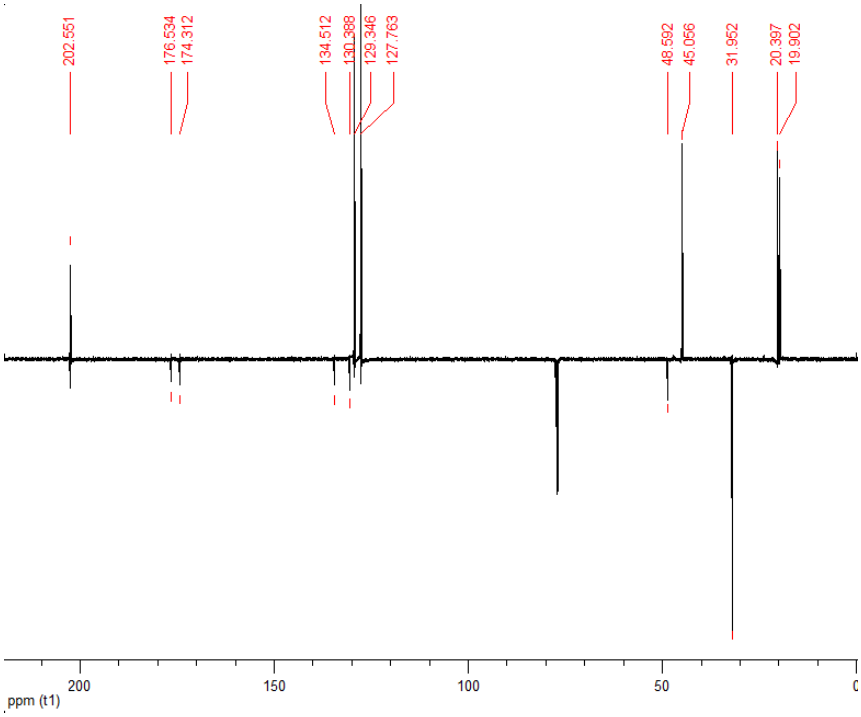
^{13}C-NMR, 125 MHz, $CDCl_3$



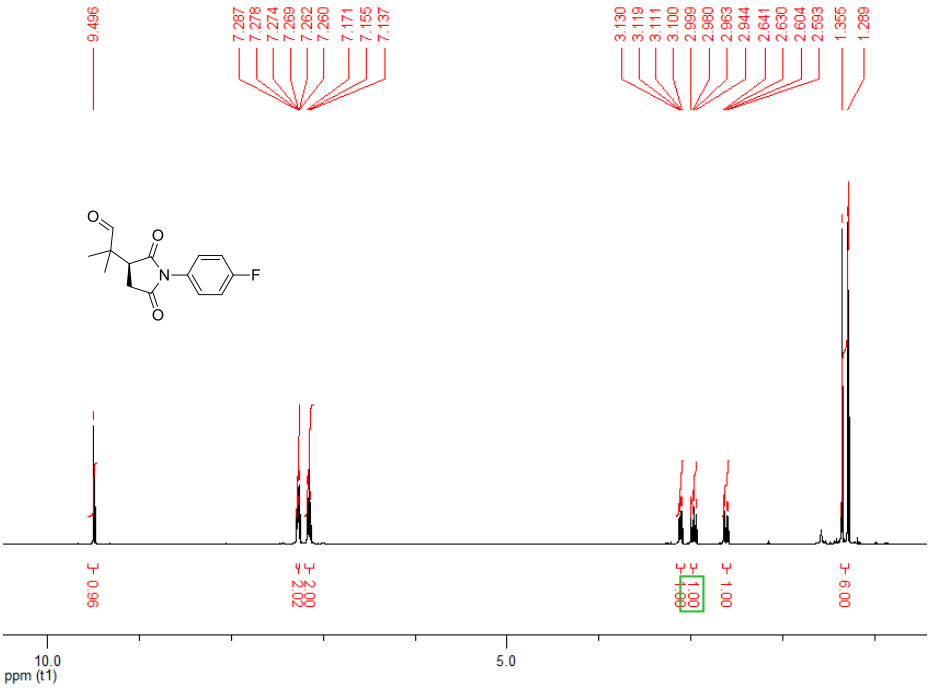
(*S*)-2-(1-(4-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3k**),
^{1}H-NMR, 500 MHz, $CDCl_3$



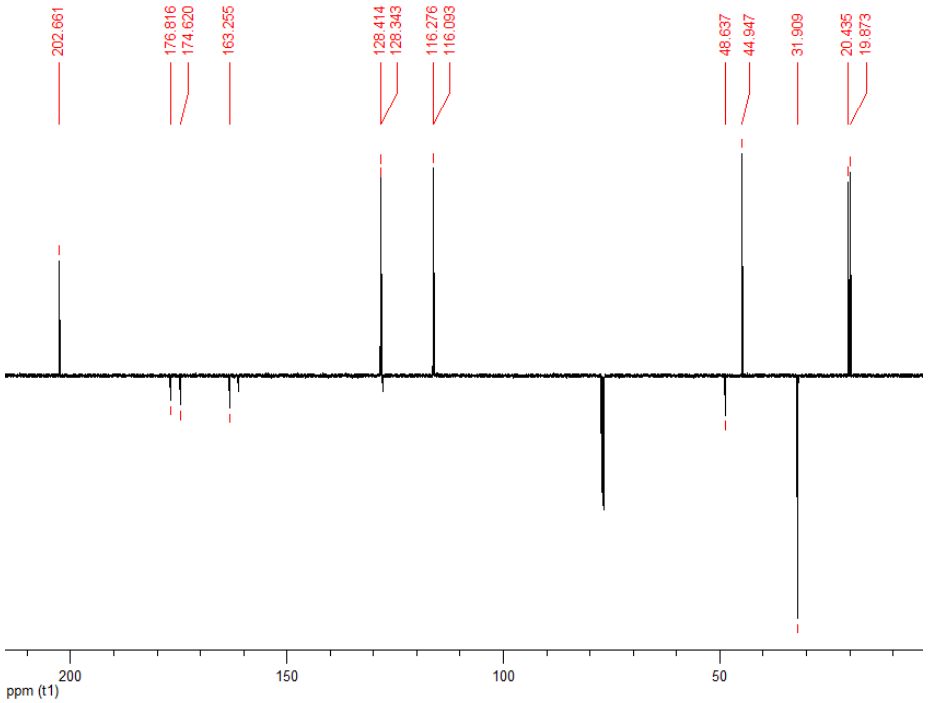
^{13}C-NMR, 125 MHz, $CDCl_3$



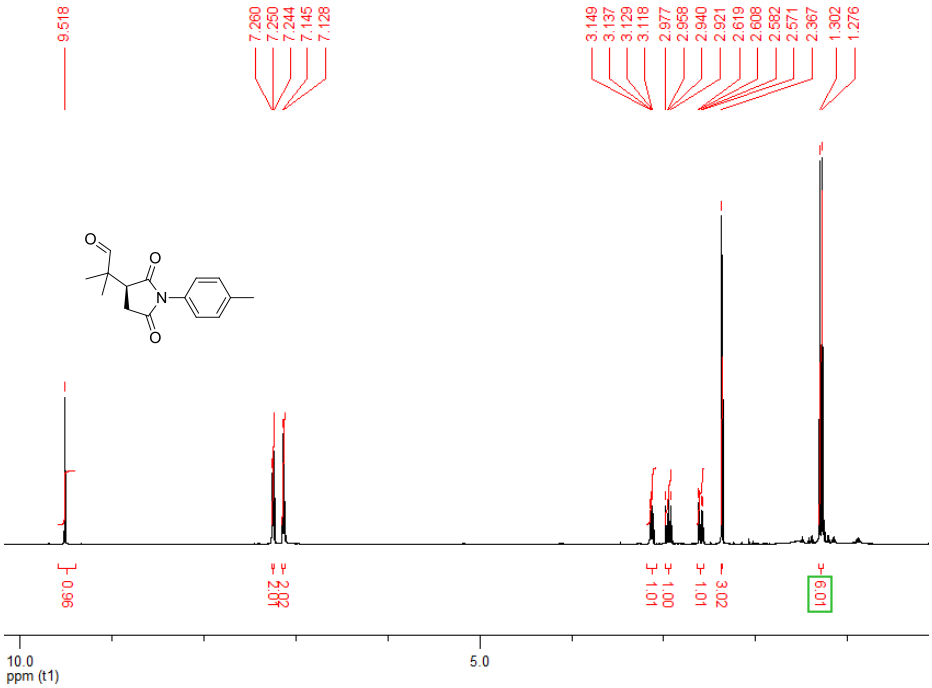
(*S*)-2-(1-(4-fluorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3l**),
^{1}H-NMR, 500 MHz, $CDCl_3$



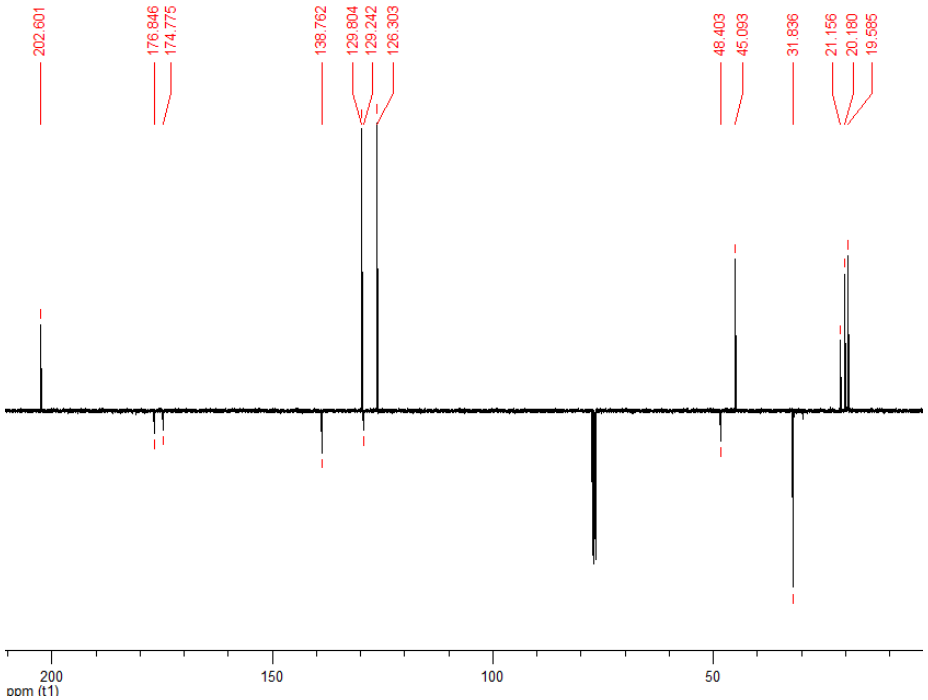
^{13}C-NMR, 125 MHz, $CDCl_3$



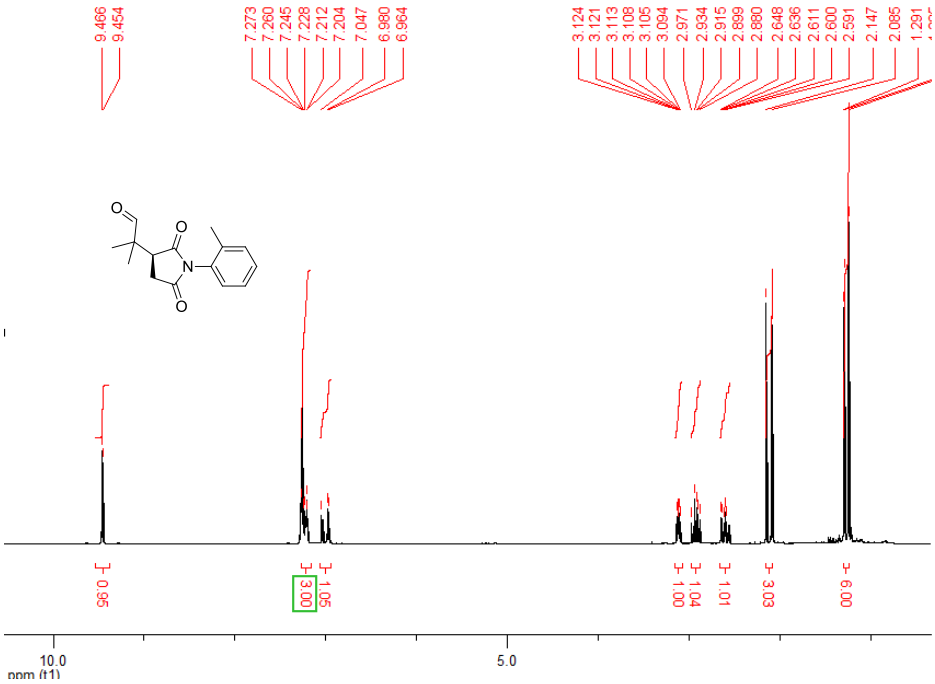
(*S*)-2-(1-(4-methylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3m**),
^{1}H-NMR, 500 MHz, $CDCl_3$



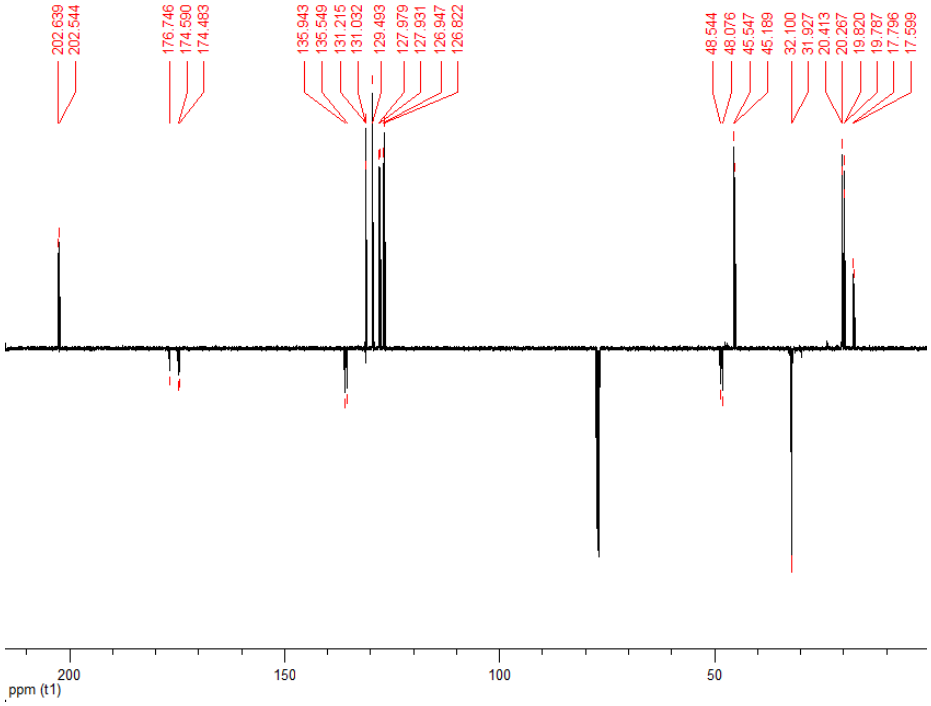
^{13}C-NMR, 125 MHz, $CDCl_3$



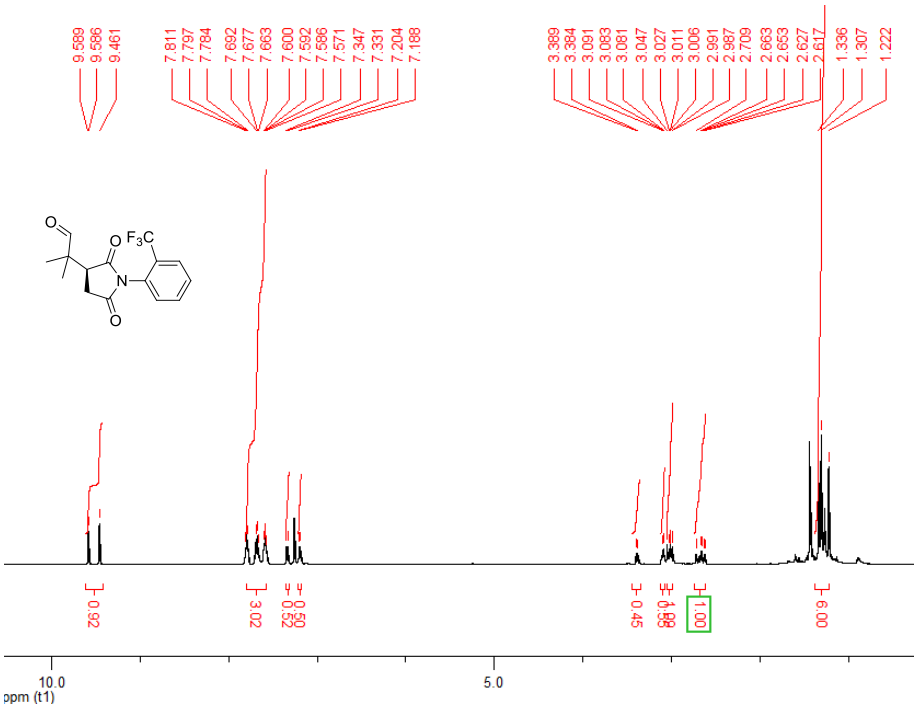
(*S*)-2-(1-(2-methylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3n**),
^{1}H-NMR, 500 MHz, $CDCl_3$



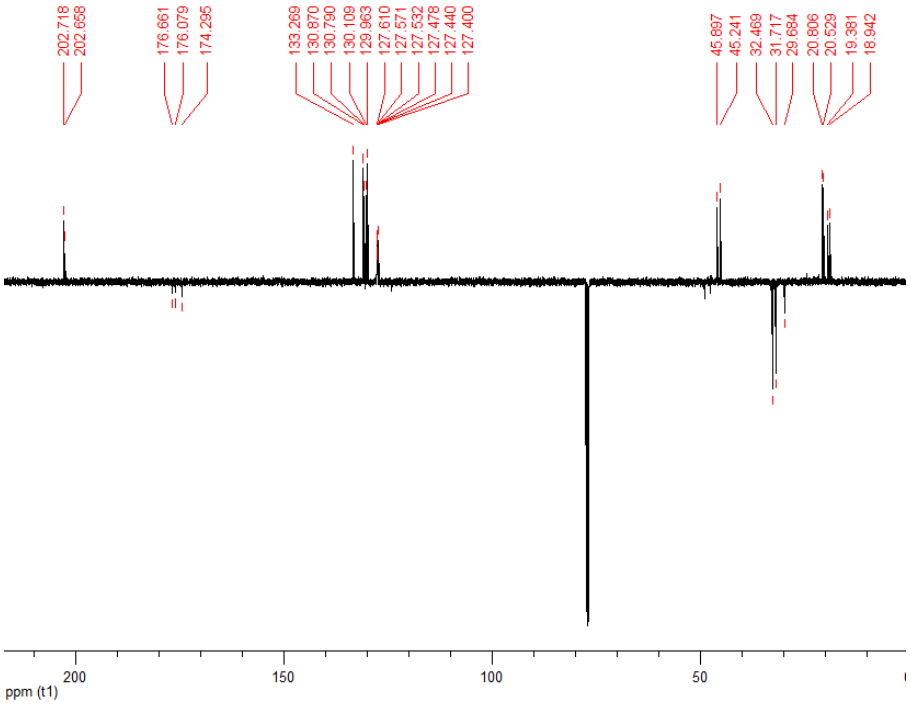
^{13}C-NMR, 125 MHz, $CDCl_3$



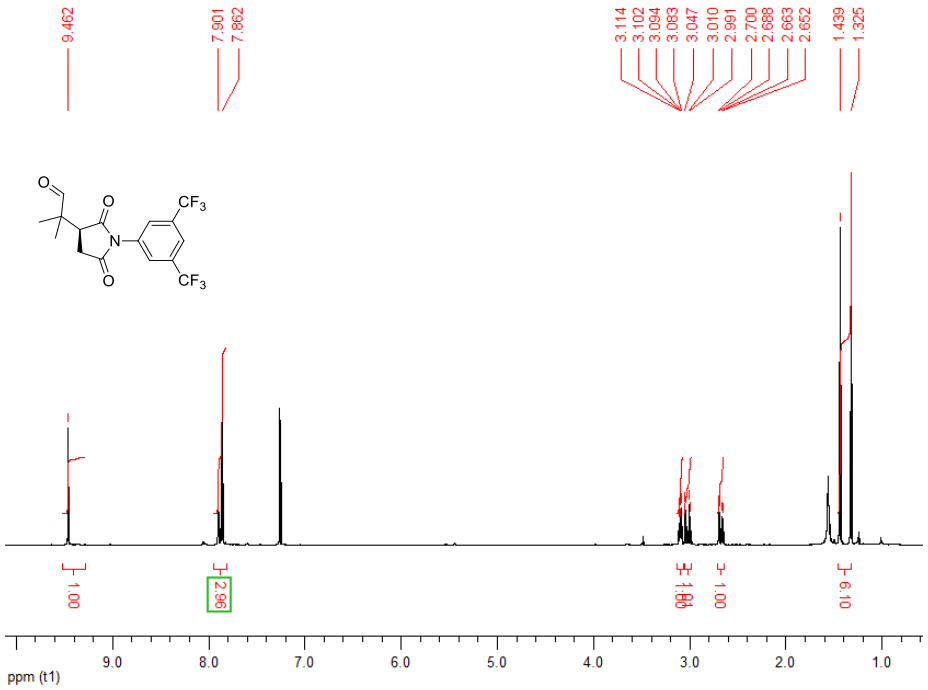
(*S*)-2-(1-(2-trifluoromethylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3o**),
^{1}H-NMR, 500 MHz, $CDCl_3$



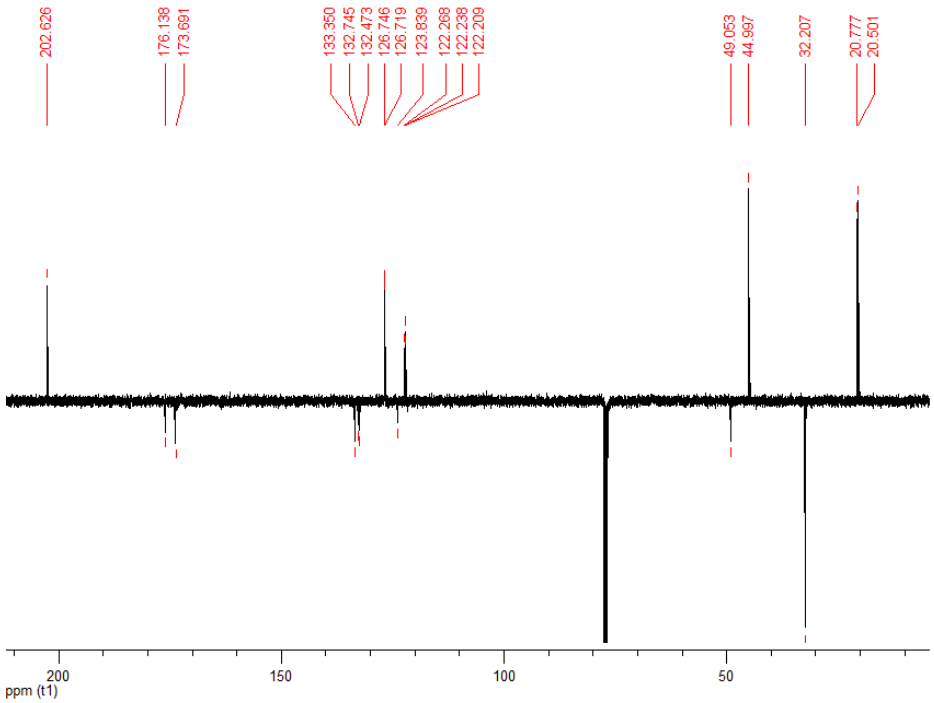
^{13}C-NMR, 125 MHz, $CDCl_3$



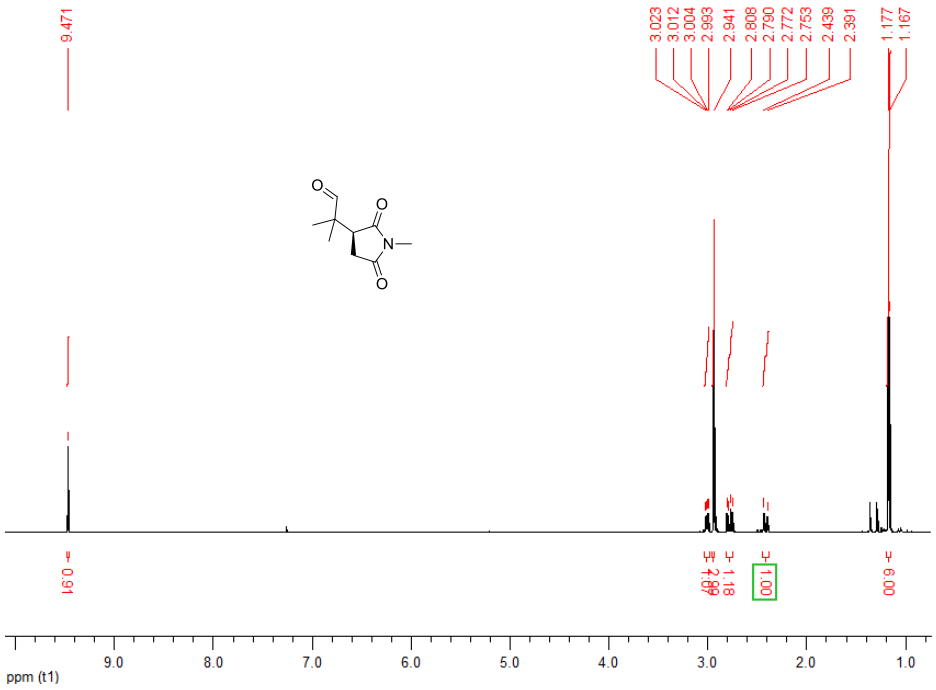
(*S*)-2-(1-(3,5-ditrifluoromethylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3p**), ^{1}H-NMR, 500 MHz, $CDCl_3$



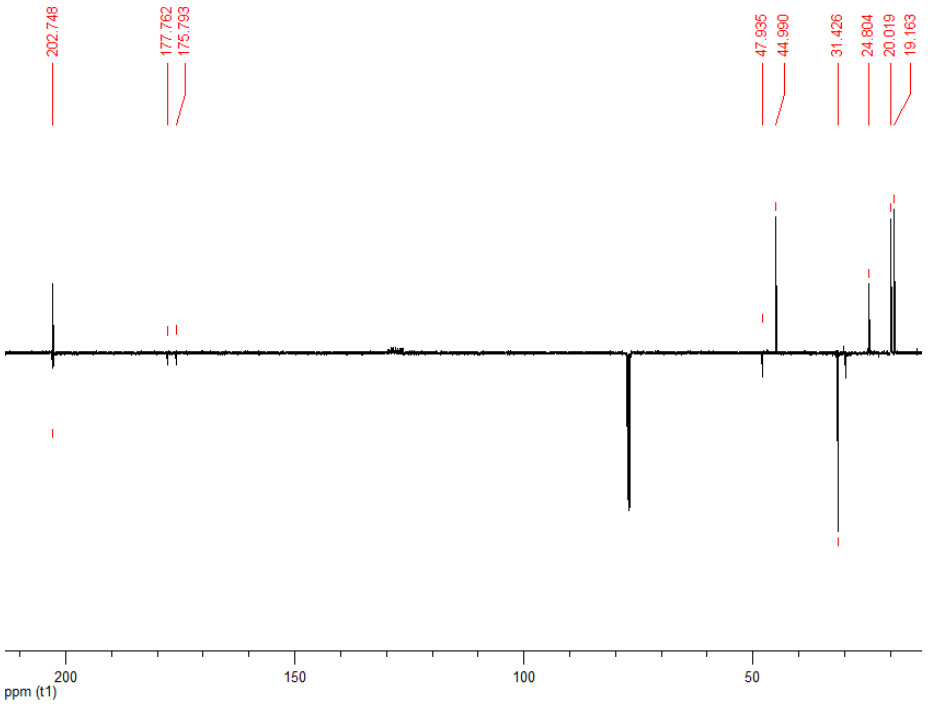
^{13}C-NMR, 125 MHz, $CDCl_3$



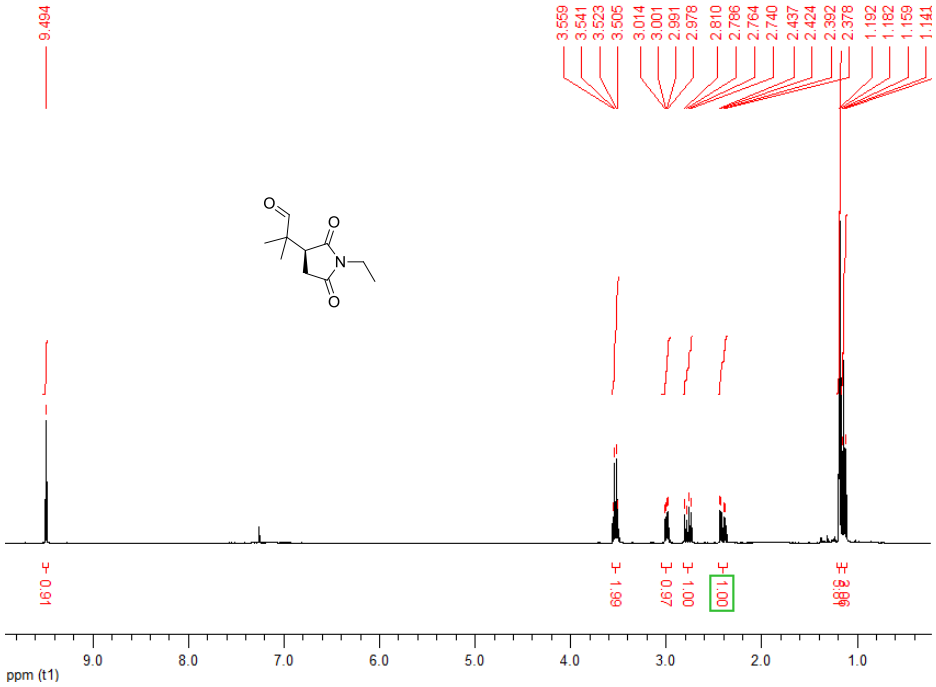
(*S*)-2-methyl-2-(1-methyl-2,5-dioxopyrrolidin-3-yl)propanal (**5a**),
^{1}H-NMR, 500 MHz, $CDCl_3$



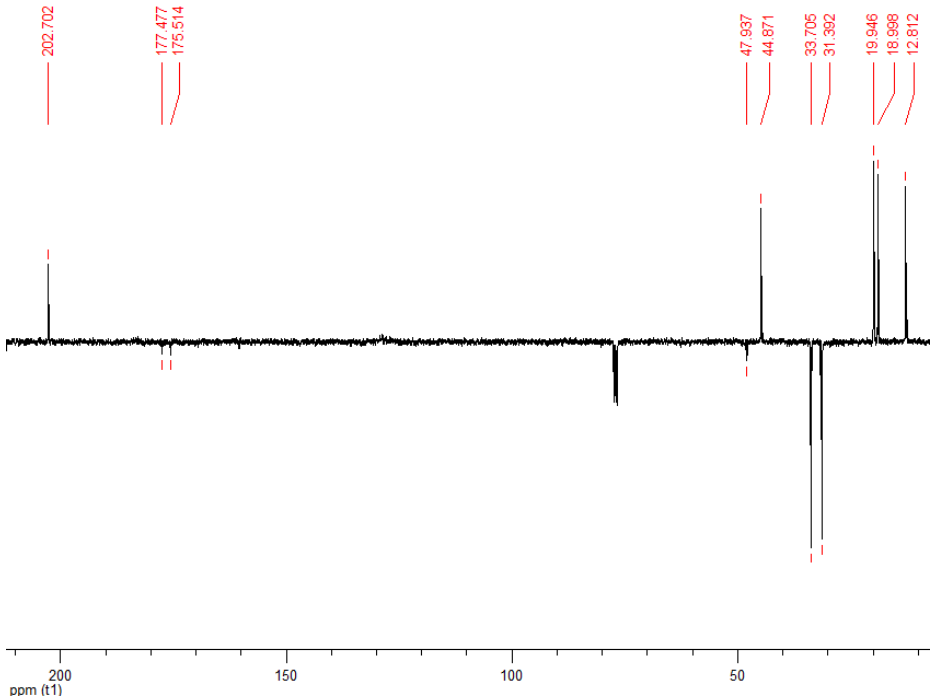
^{13}C-NMR, 125 MHz, $CDCl_3$



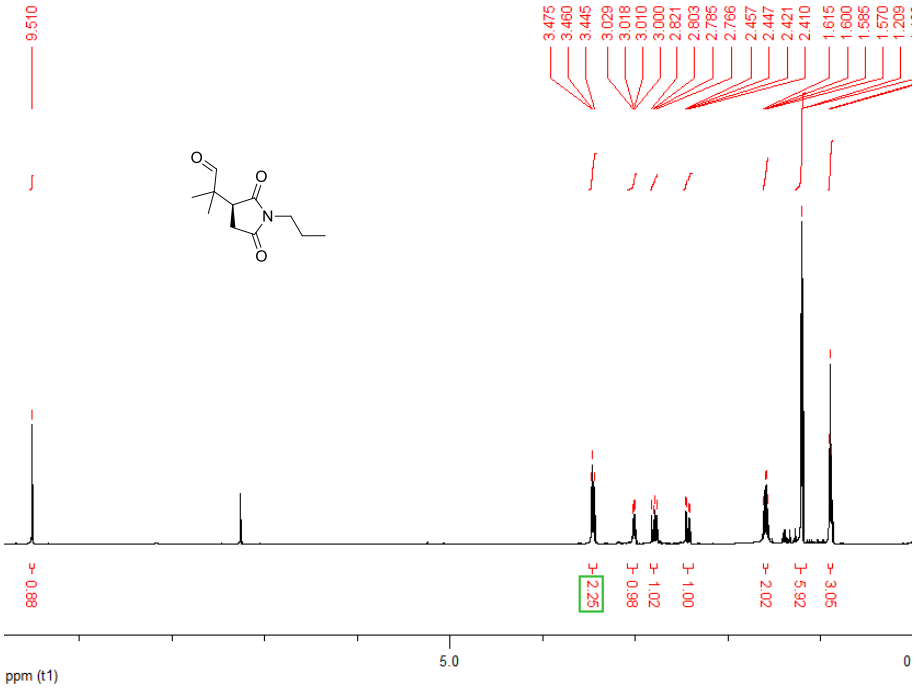
(*S*)-2-(1-ethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5b**),
^{1}H-NMR, 500 MHz, $CDCl_3$



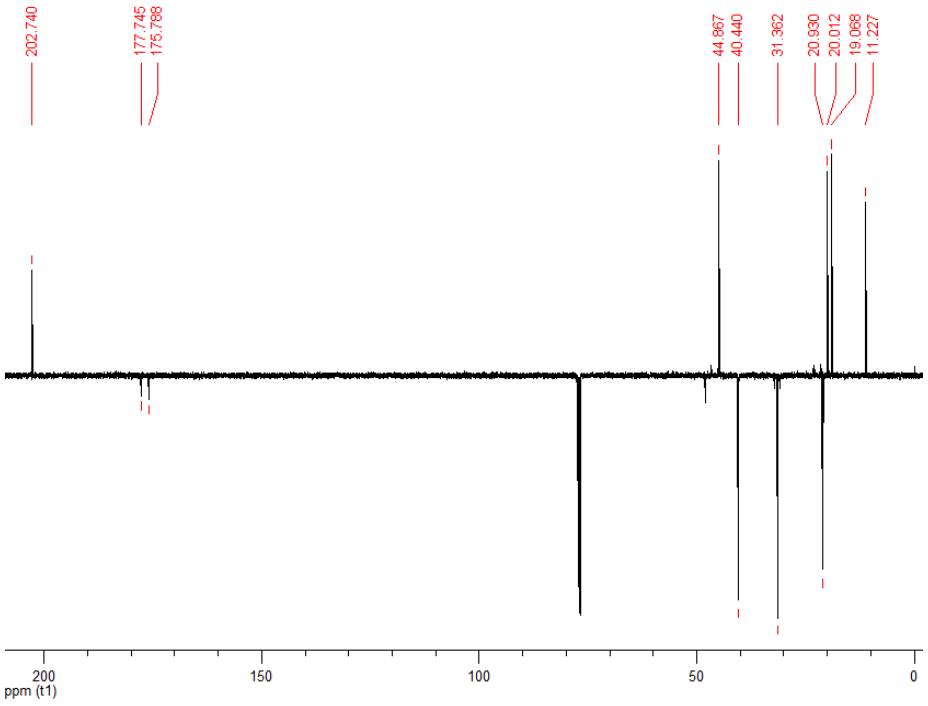
^{13}C-NMR, 125 MHz, $CDCl_3$



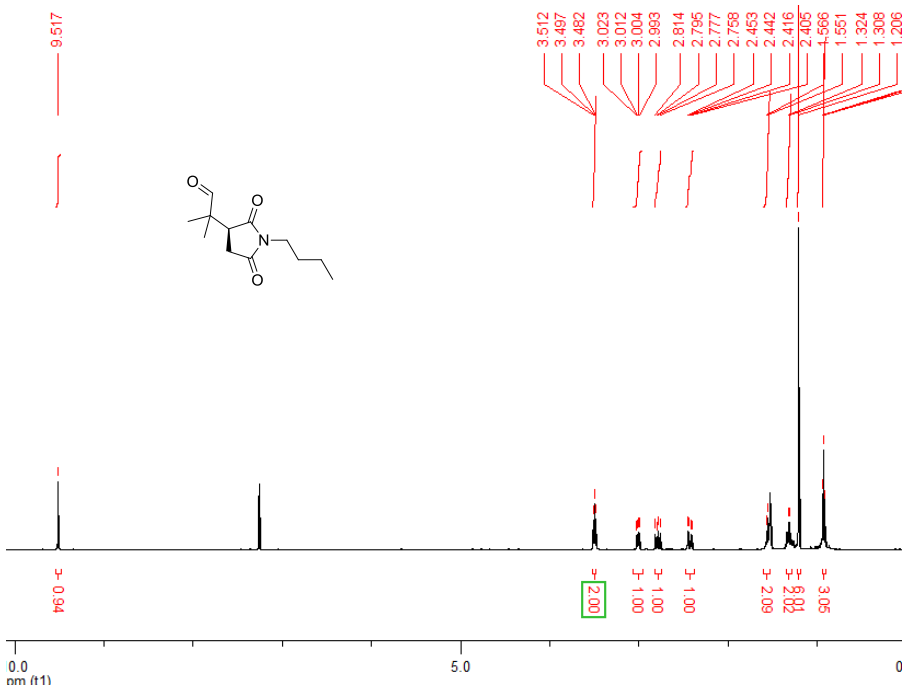
(*S*)-2-(1-propyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5c**),
^{1}H-NMR, 500 MHz, $CDCl_3$



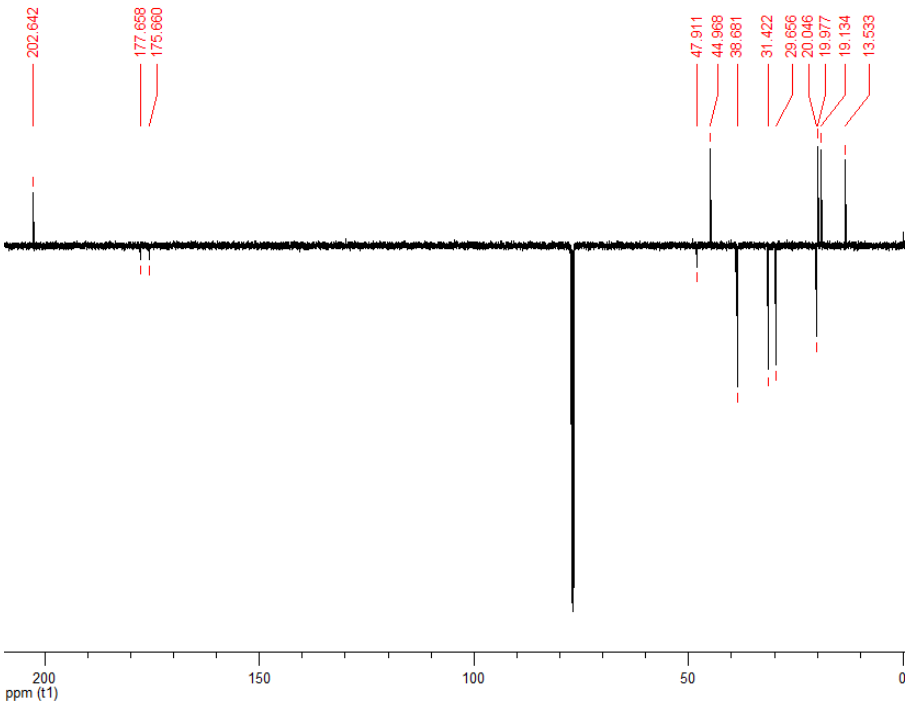
^{13}C-NMR, 125 MHz, $CDCl_3$



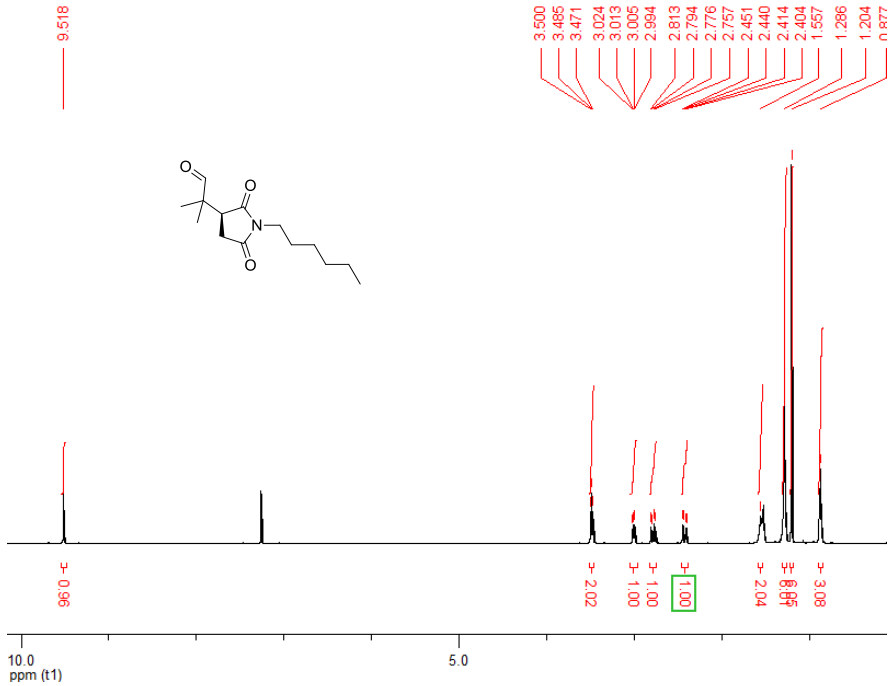
(*S*)-2-(1-butyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5d**),
^{1}H-NMR, 500 MHz, $CDCl_3$



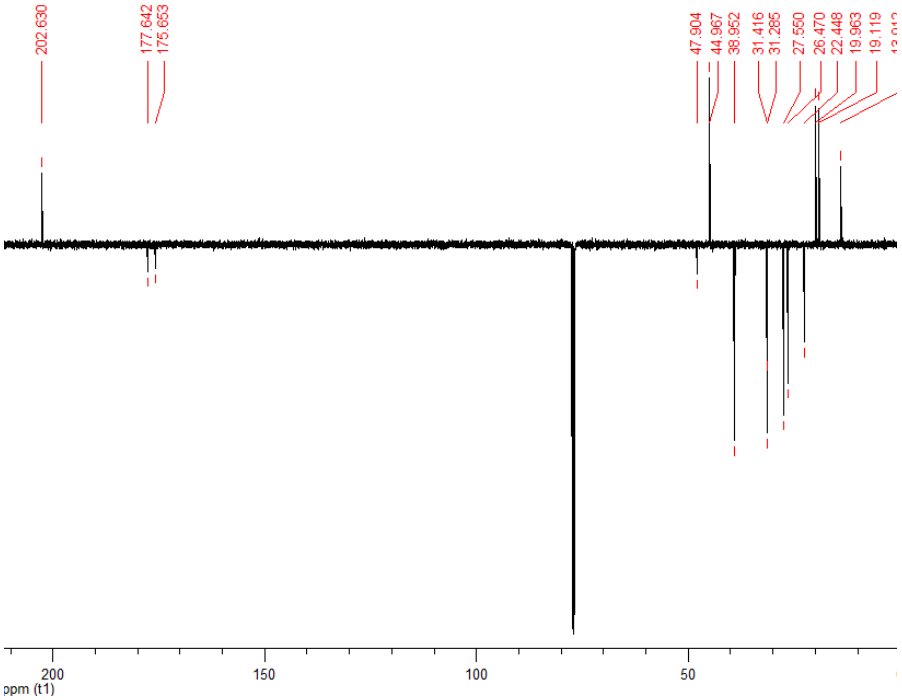
^{13}C-NMR, 125 MHz, $CDCl_3$



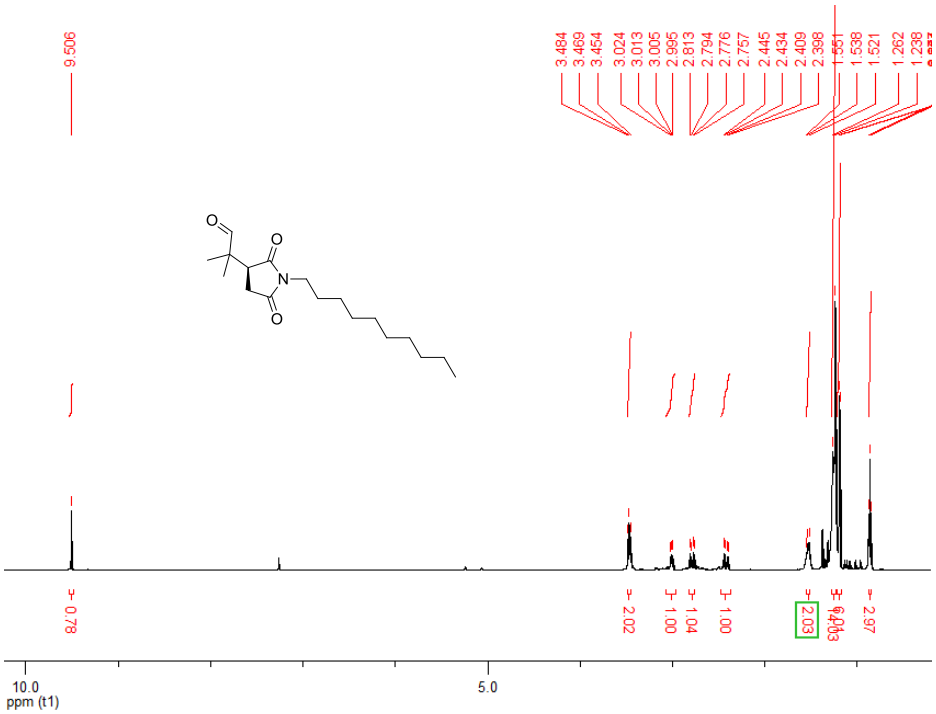
(*S*)-2-(1-hexyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5e**),
^{1}H-NMR, 500 MHz, $CDCl_3$



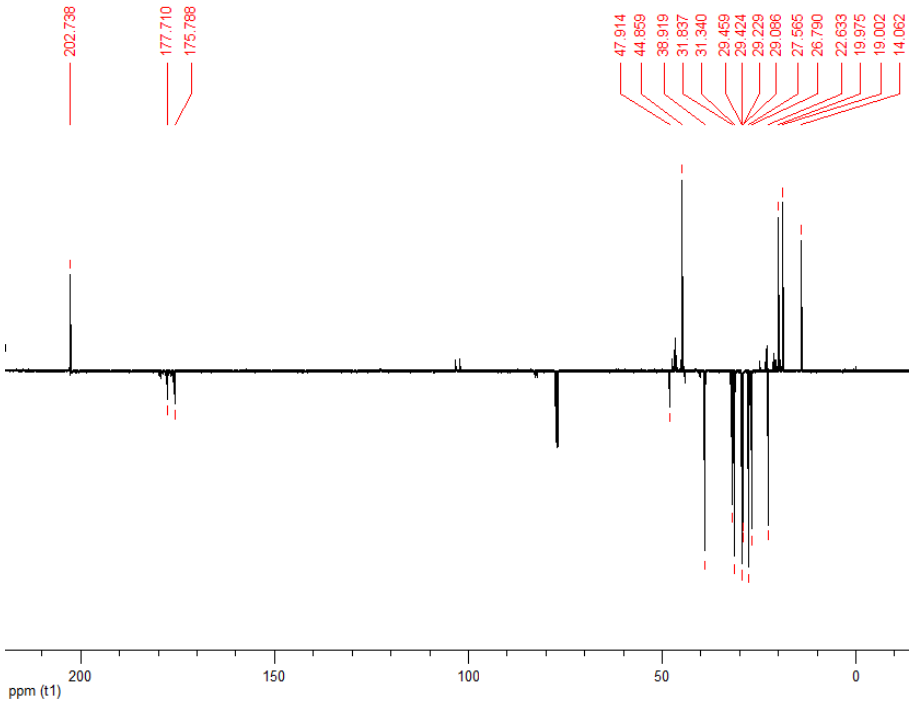
^{13}C-NMR, 125 MHz, $CDCl_3$



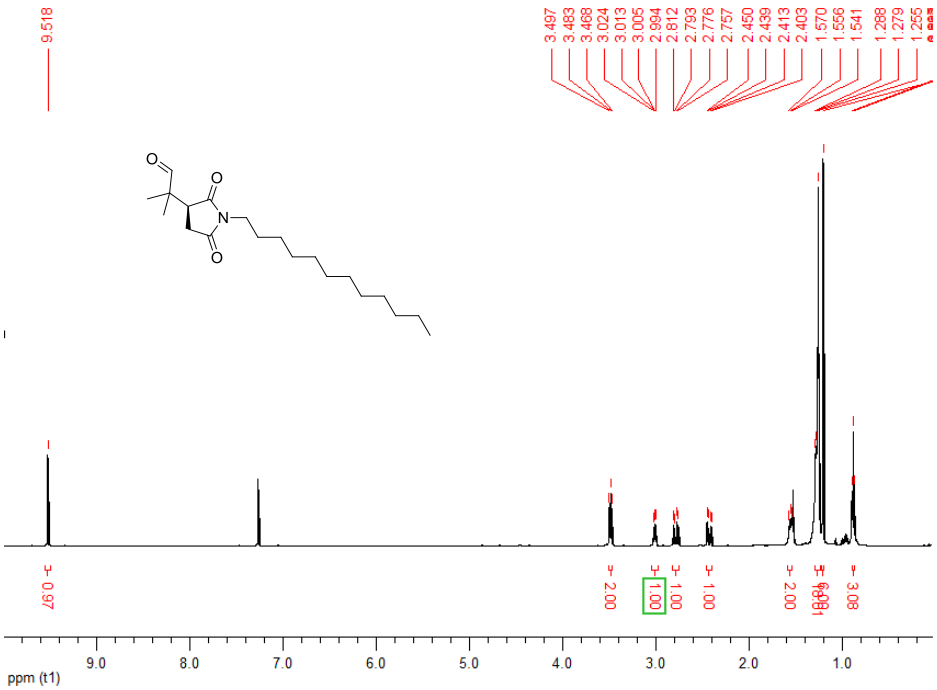
(*S*)-2-(1-decyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5f**),
^{1}H-NMR, 500 MHz, $CDCl_3$



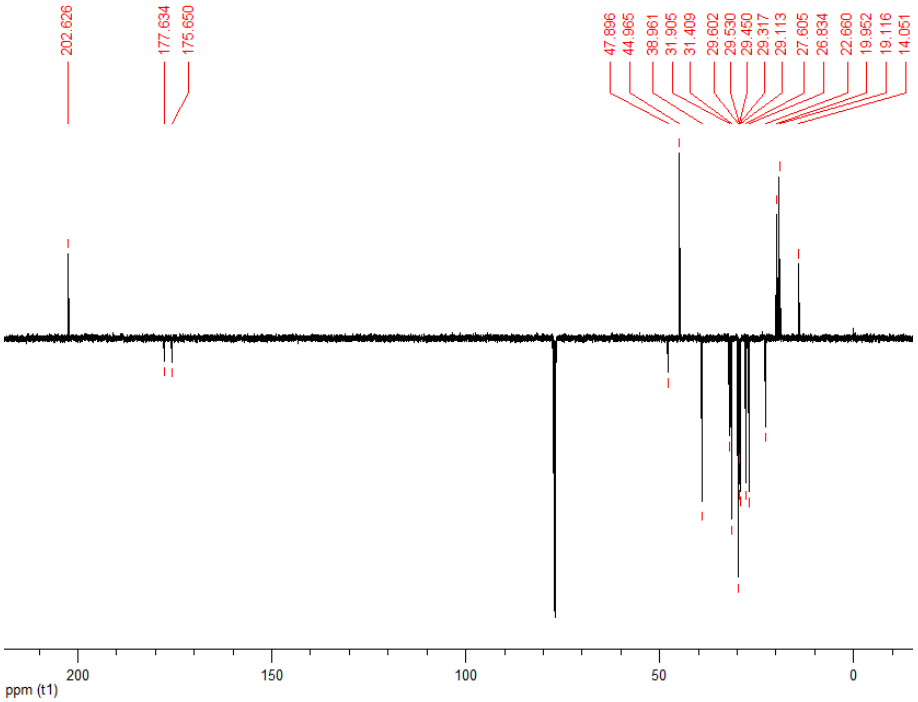
^{13}C-NMR, 125 MHz, $CDCl_3$



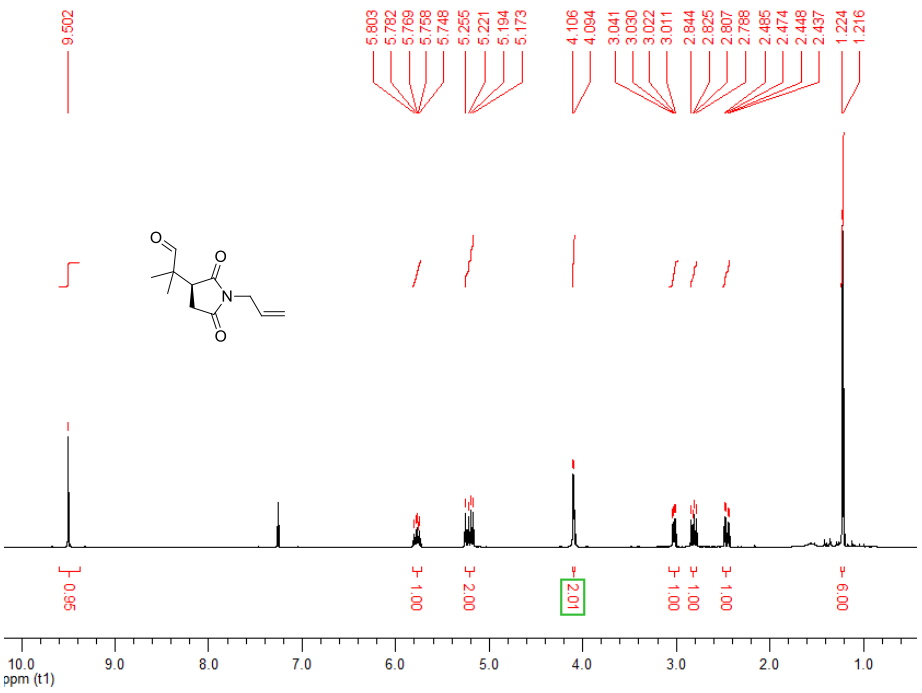
(*S*)-2-(1-dodecyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5g**),
^{1}H-NMR, 500 MHz, $CDCl_3$



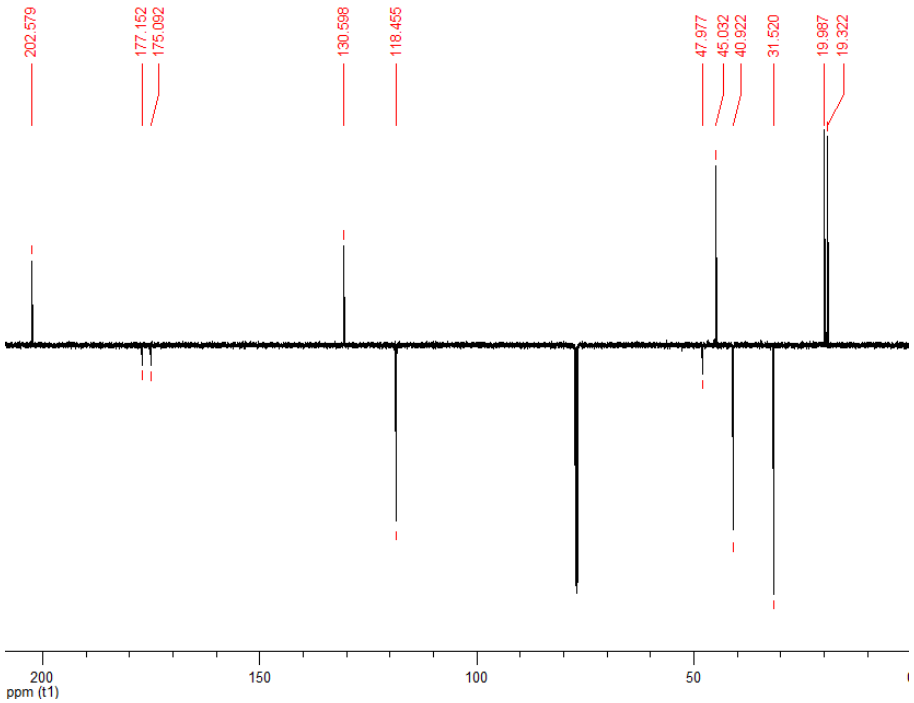
^{13}C-NMR, 125 MHz, $CDCl_3$



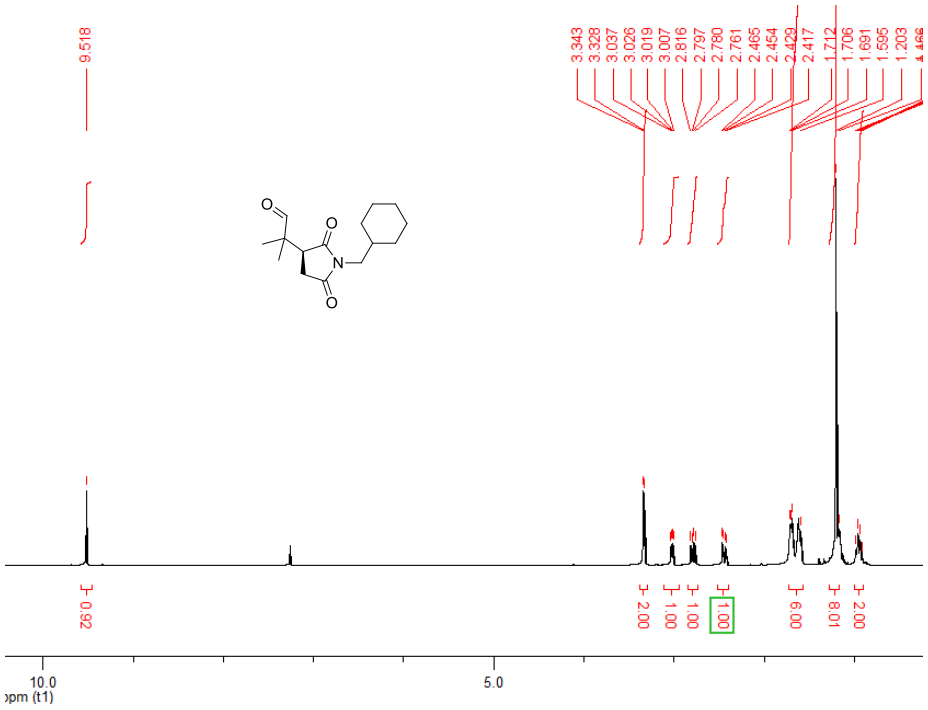
(*S*)-2-(1-allyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5h**),
^{1}H-NMR, 500 MHz, $CDCl_3$



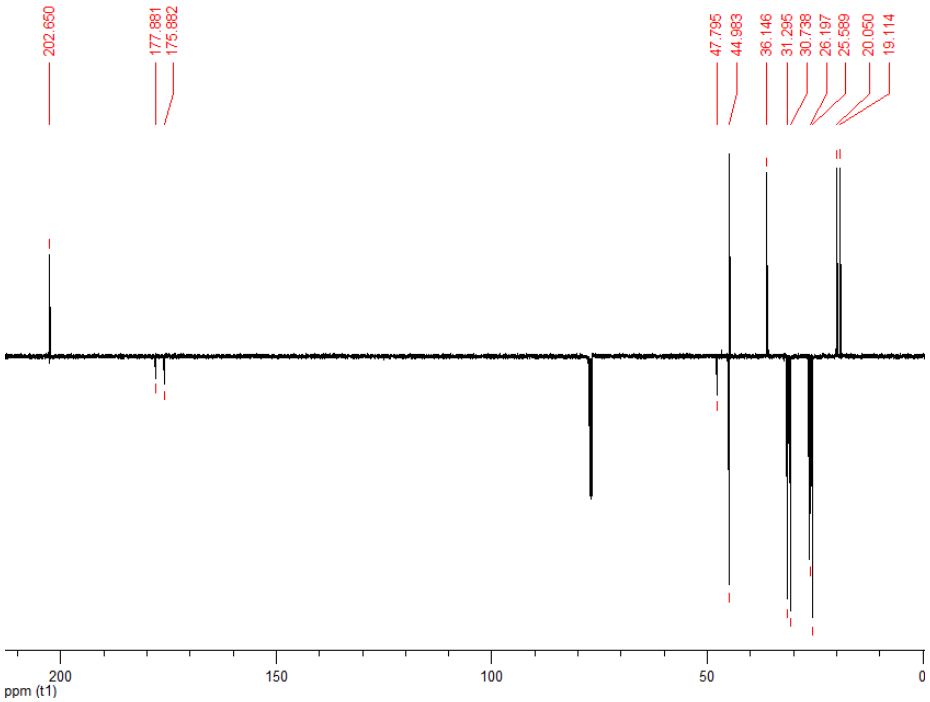
^{13}C-NMR, 125 MHz, $CDCl_3$



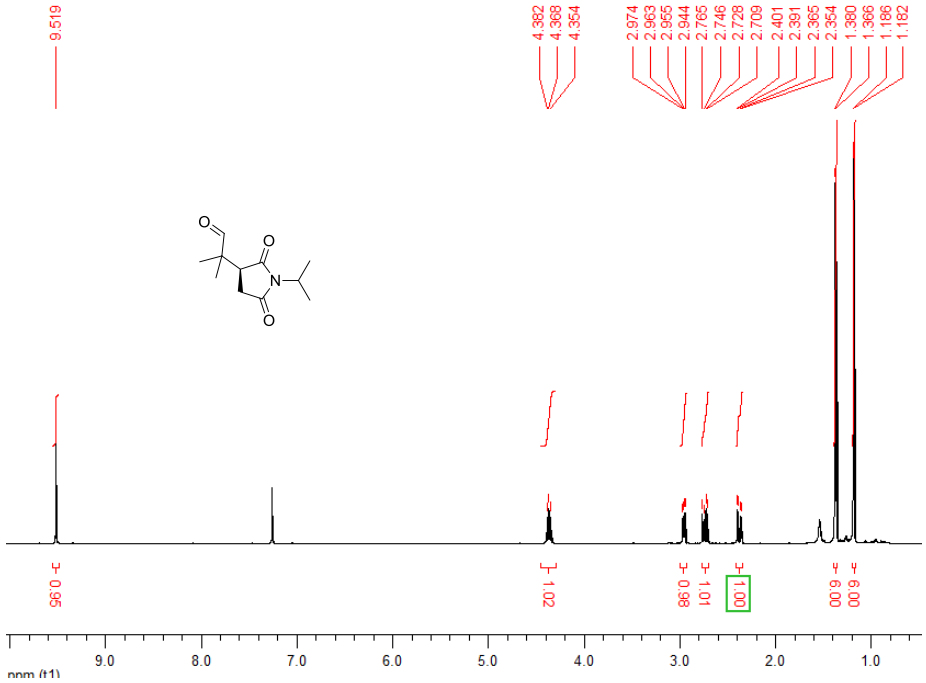
(*S*)-2-(1-cyclohexylmethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5i**),
^{1}H-NMR, 500 MHz, $CDCl_3$



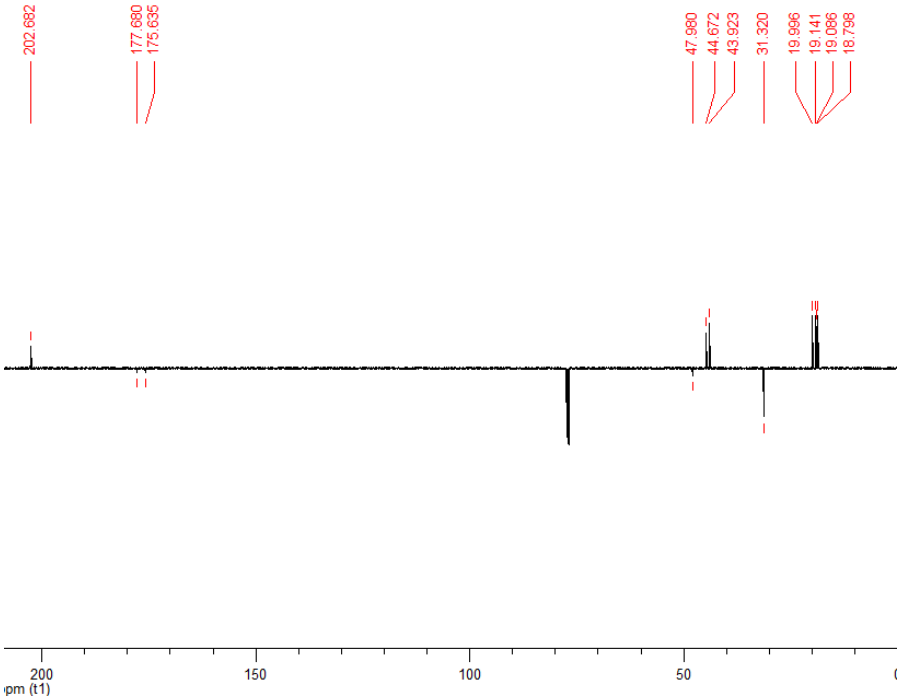
^{13}C-NMR, 125 MHz, $CDCl_3$



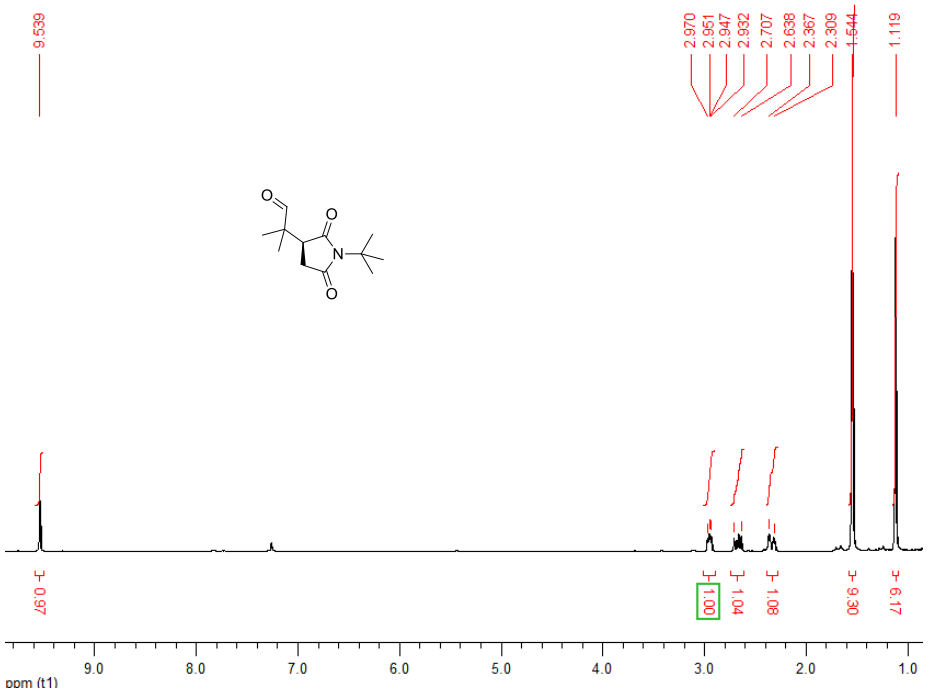
(*S*)-2-(1-isopropyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5j**),
^{1}H-NMR, 500 MHz, $CDCl_3$



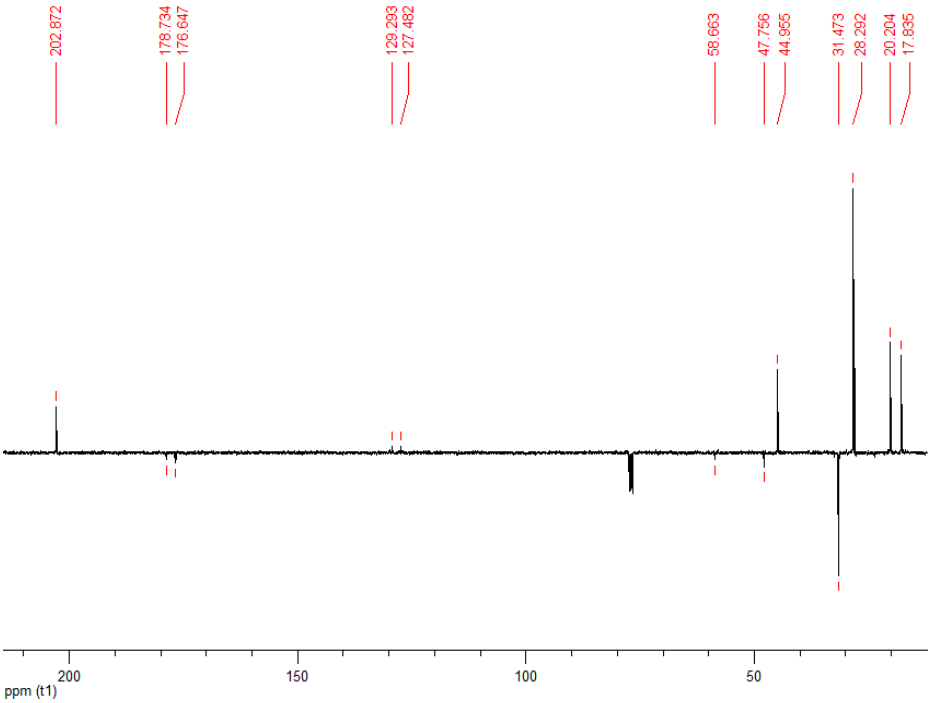
^{13}C-NMR, 125 MHz, $CDCl_3$



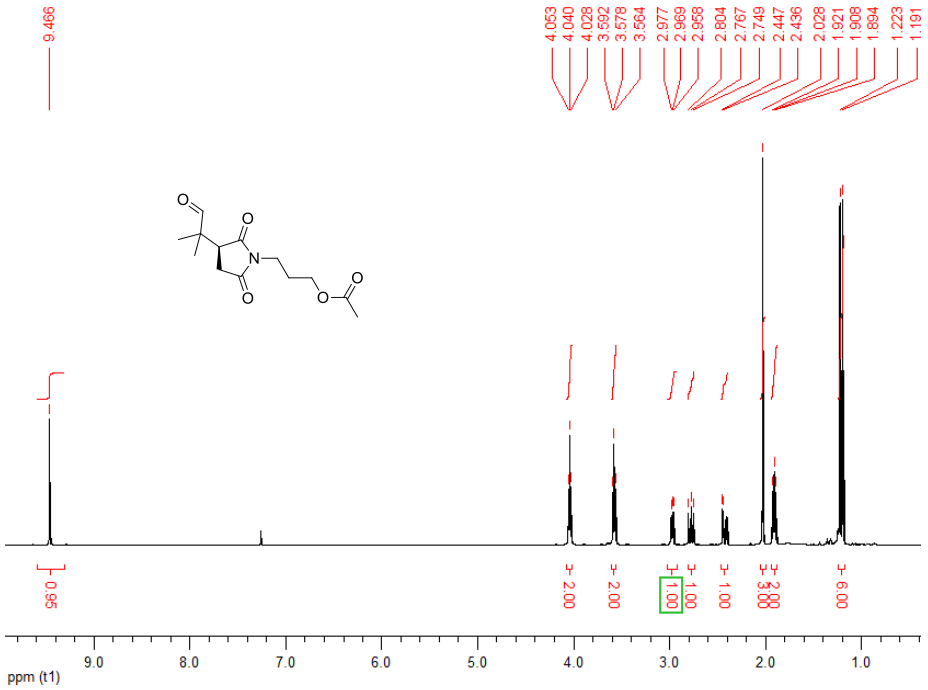
(*S*)-2-(1-*tert*-butyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5k**),
^{1}H-NMR, 500 MHz, $CDCl_3$



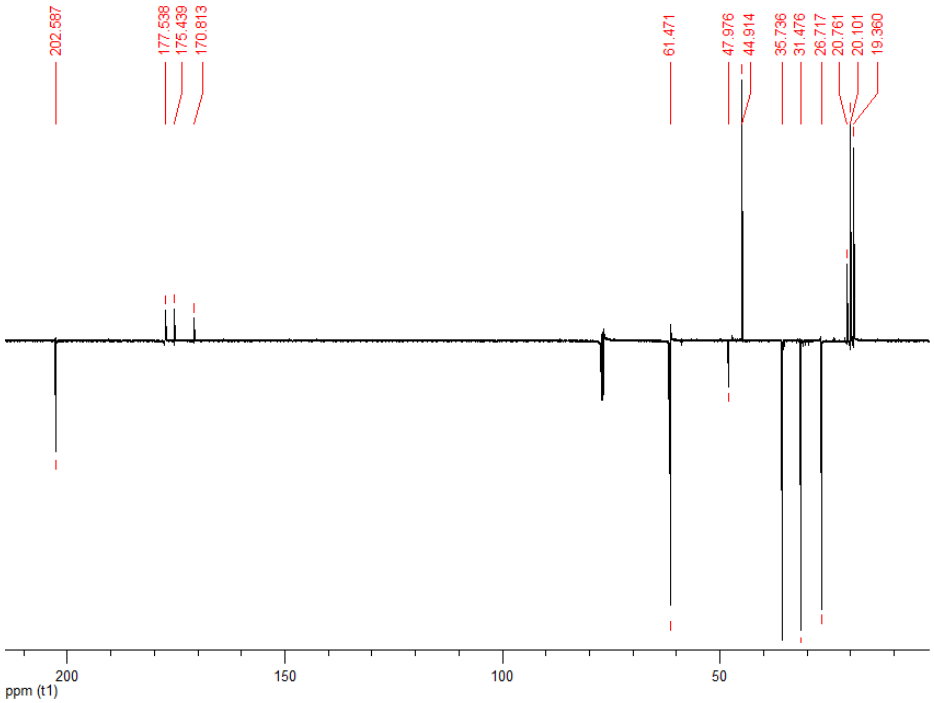
^{13}C-NMR, 125 MHz, $CDCl_3$



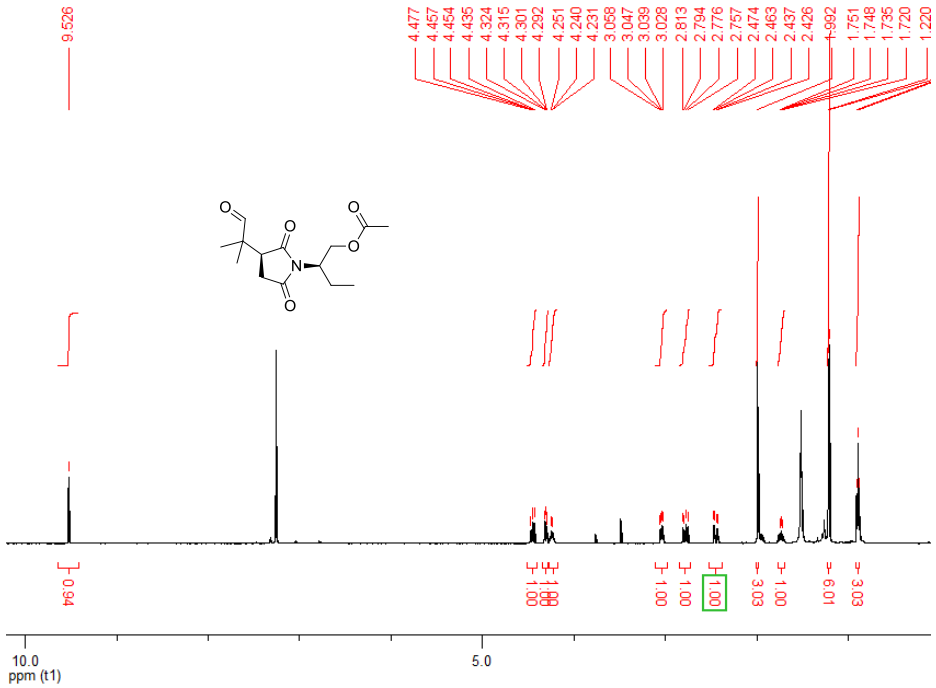
(*S*)-2-((3-acetoxypropyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5l**),
^{1}H-NMR, 500 MHz, $CDCl_3$



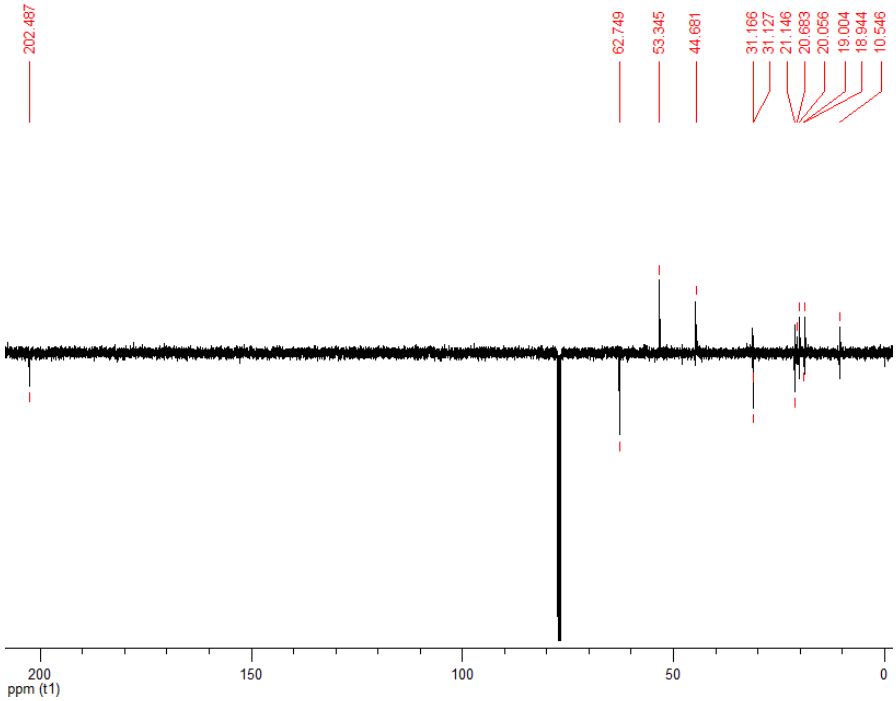
^{13}C-NMR, 125 MHz, $CDCl_3$



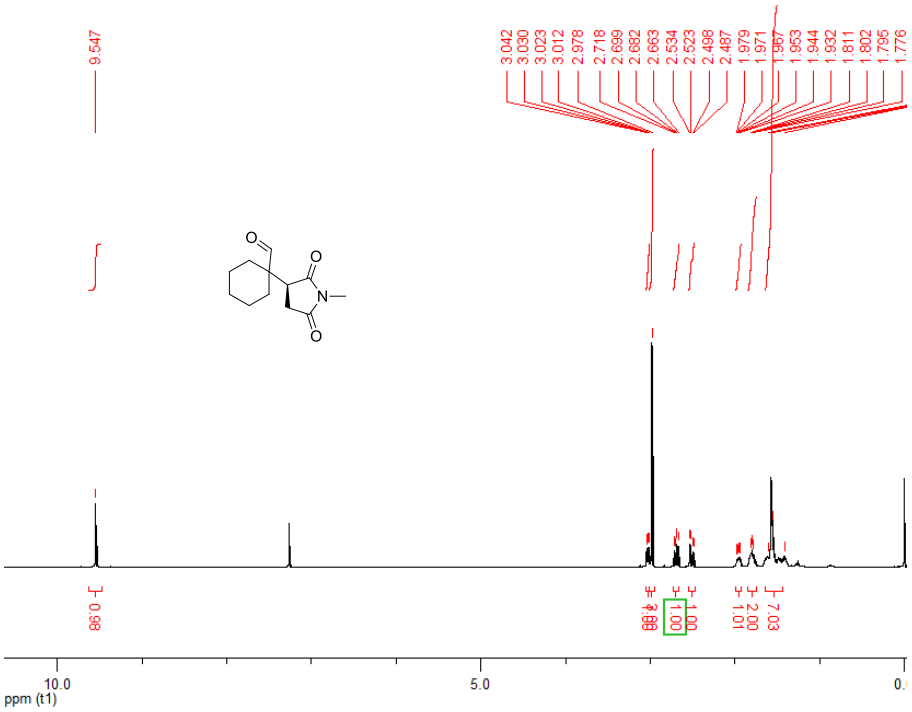
(*S*)-2-(*R*)-*N*-[1-[(acetyloxy)methyl]propyl]-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5m**),
^{1}H-NMR, 500 MHz, $CDCl_3$



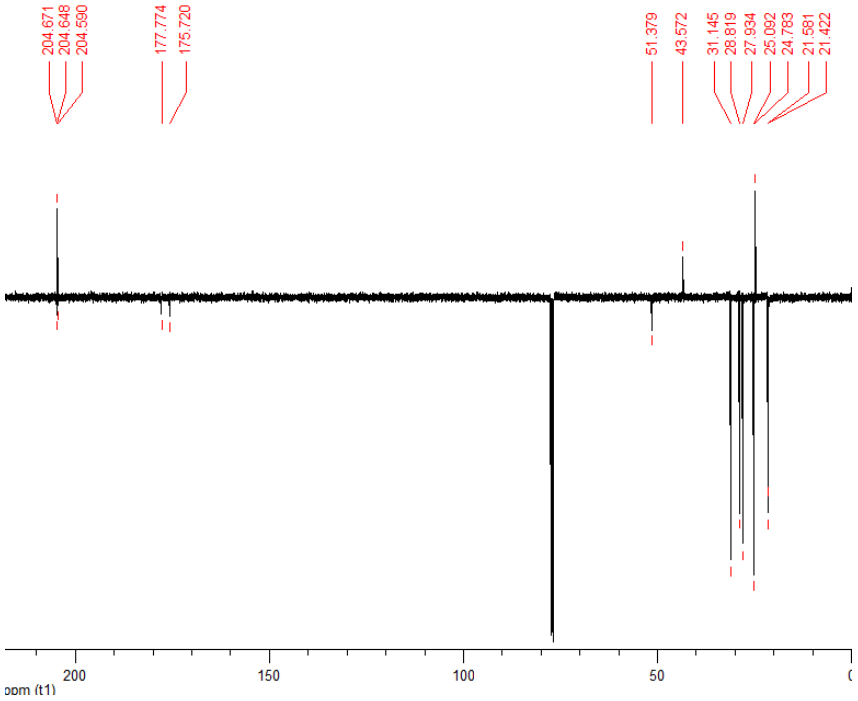
^{13}C-NMR, 125 MHz, $CDCl_3$



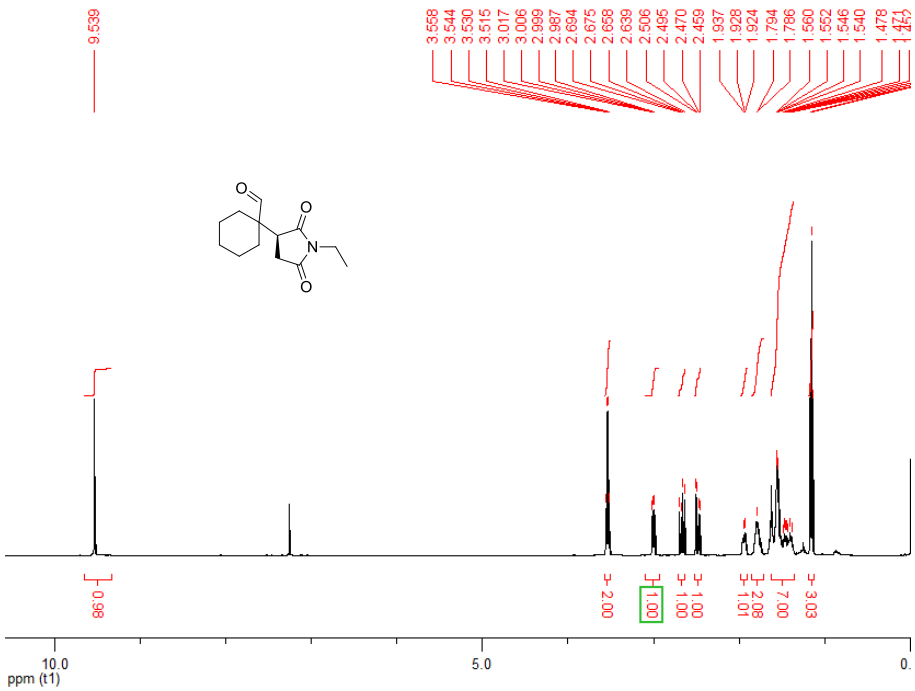
(*S*)-1-(1-methyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6a**),
^{1}H-NMR, 500 MHz, $CDCl_3$



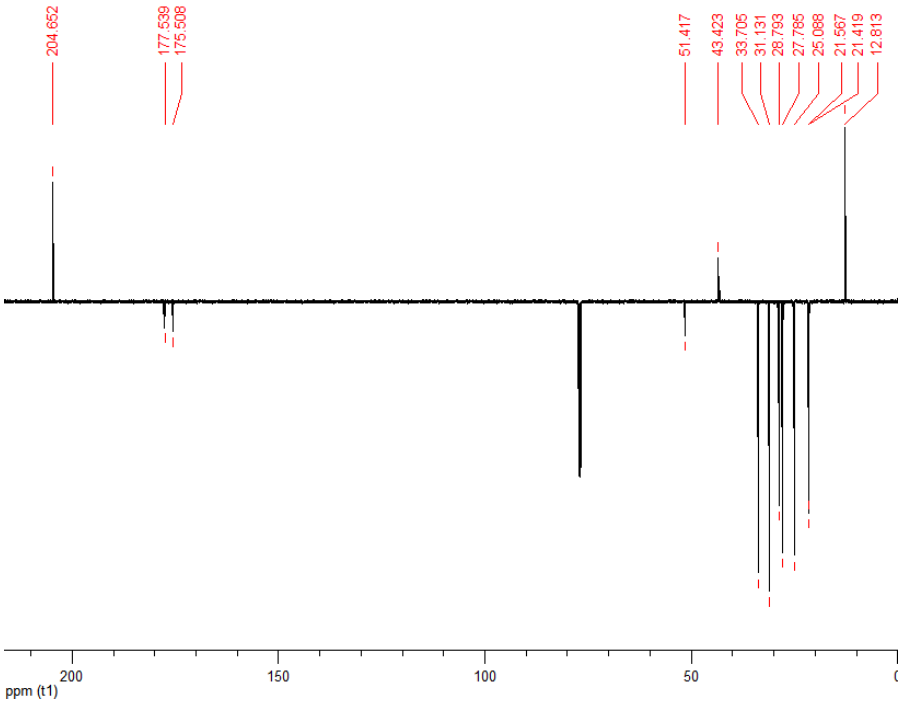
^{13}C-NMR, 125 MHz, $CDCl_3$



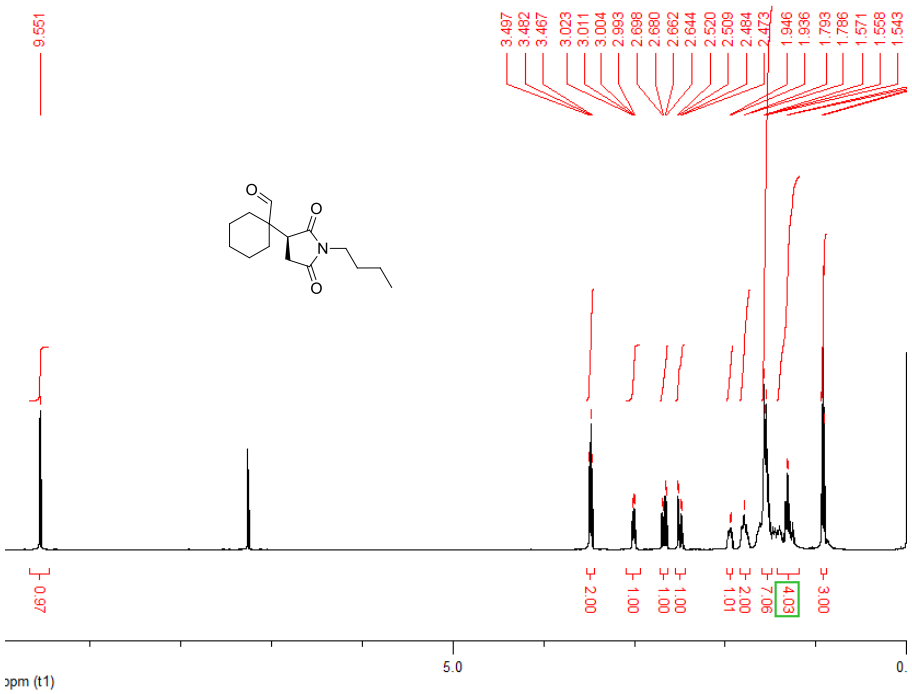
(*S*)-1-(1-ethyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6b**),
^{1}H-NMR, 500 MHz, $CDCl_3$



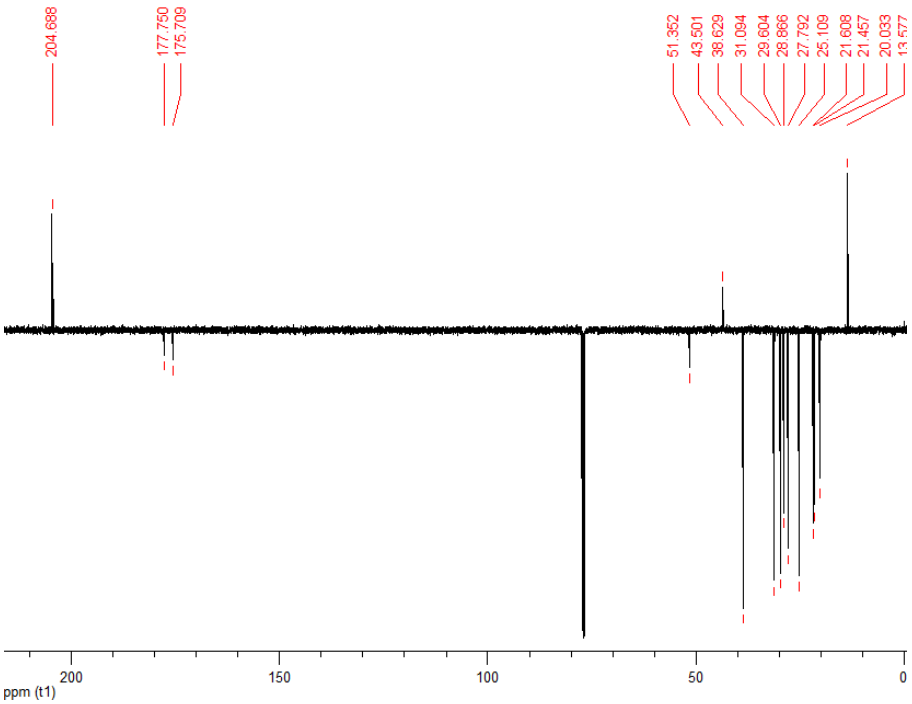
^{13}C-NMR, 125 MHz, $CDCl_3$



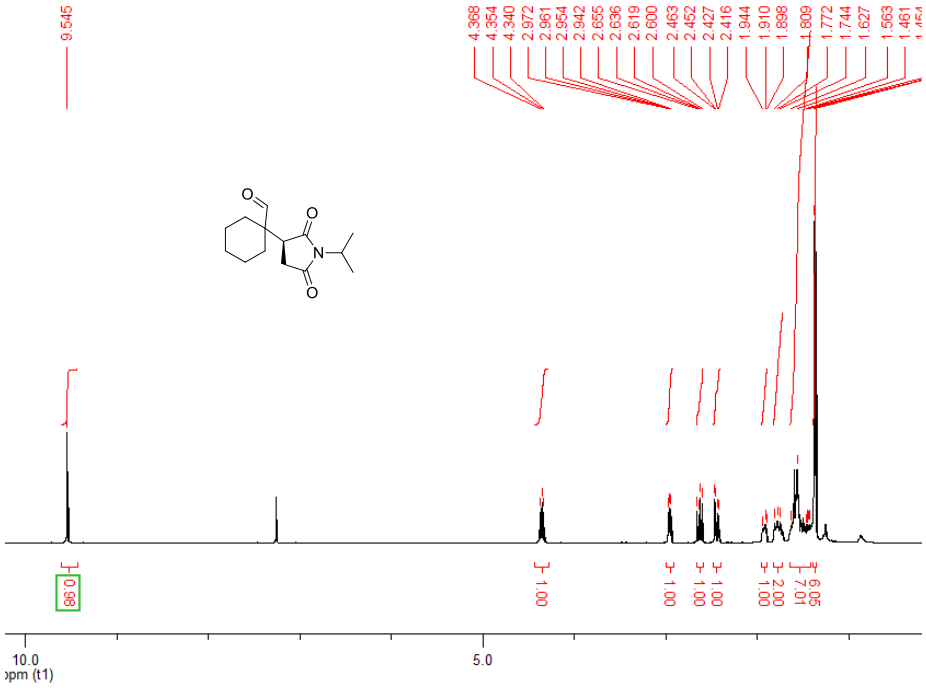
(*S*)-1-(1-butyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6c**),
^{1}H-NMR, 500 MHz, $CDCl_3$



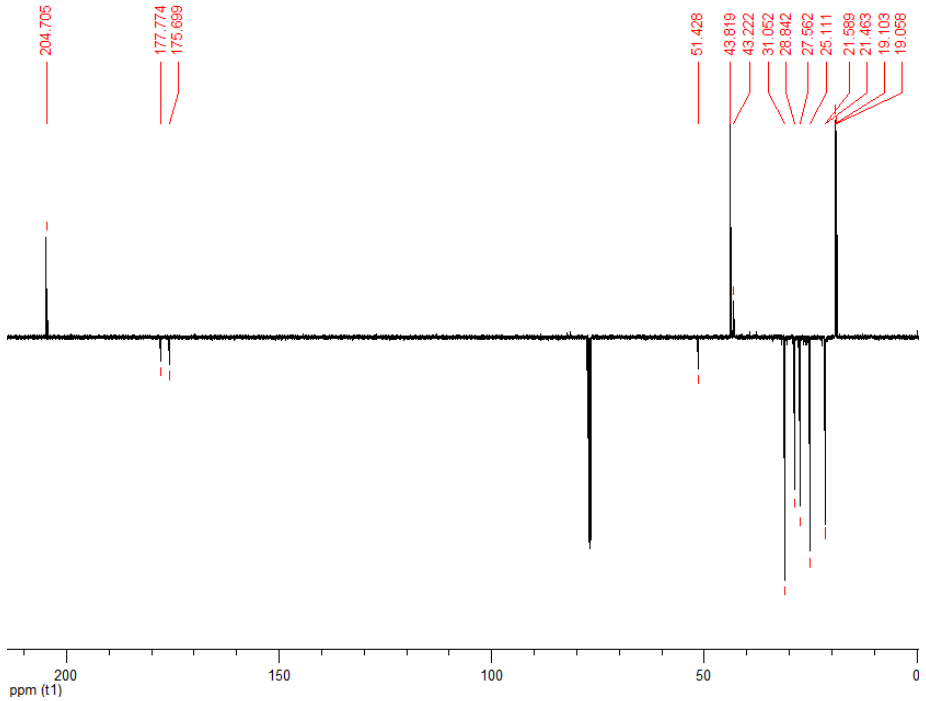
^{13}C-NMR, 125 MHz, $CDCl_3$



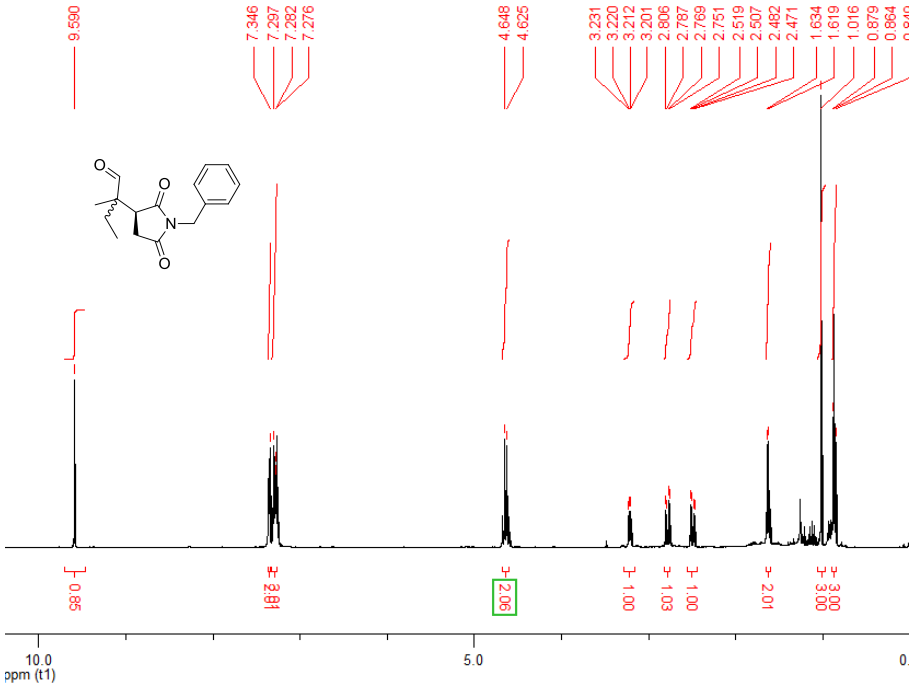
(*S*)-1-(1-isopropyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6d**),
^{1}H-NMR, 500 MHz, $CDCl_3$



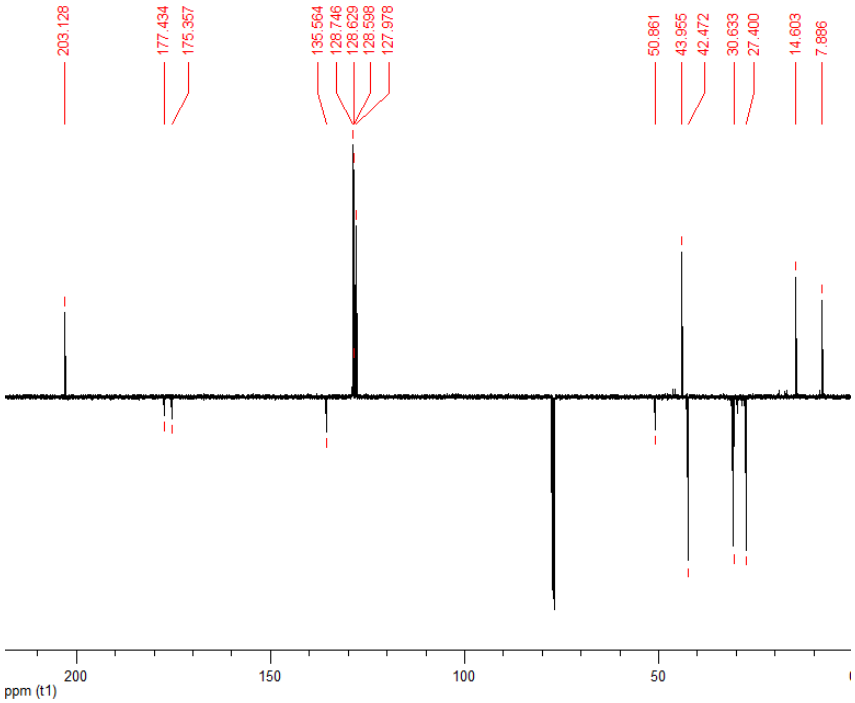
^{13}C-NMR, 125 MHz, $CDCl_3$



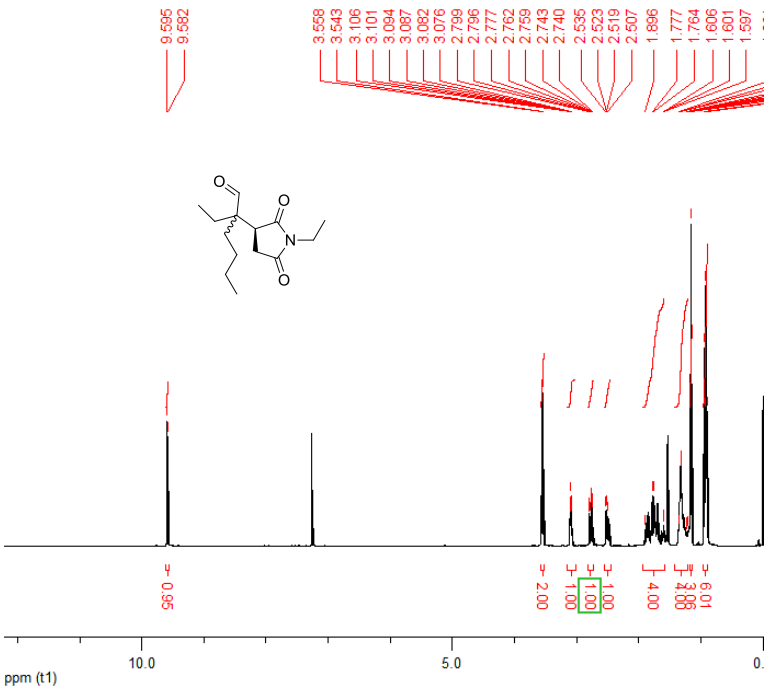
2-((*S*)-1-benzyl-2,5-dioxopyrrolidin-3-yl)-2-methylbutanal (**6e**),
^{1}H-NMR, 500 MHz, $CDCl_3$



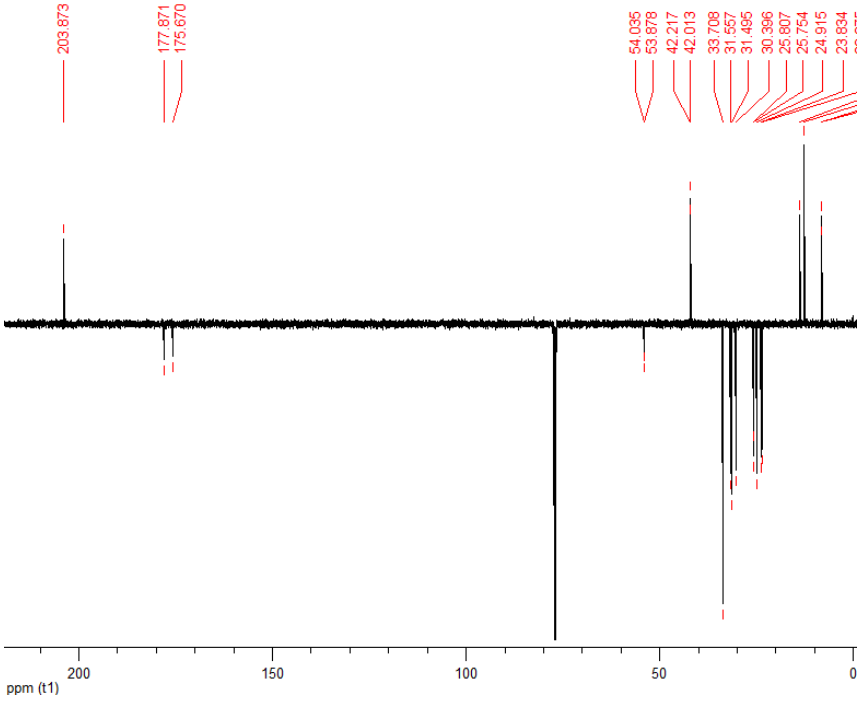
^{13}C-NMR, 125 MHz, $CDCl_3$



2-ethyl-2-((*S*)-1-ethyl-2,5-dioxopyrrolidin-3-yl)hexanal (**6f**),
^{1}H-NMR, 500 MHz, $CDCl_3$

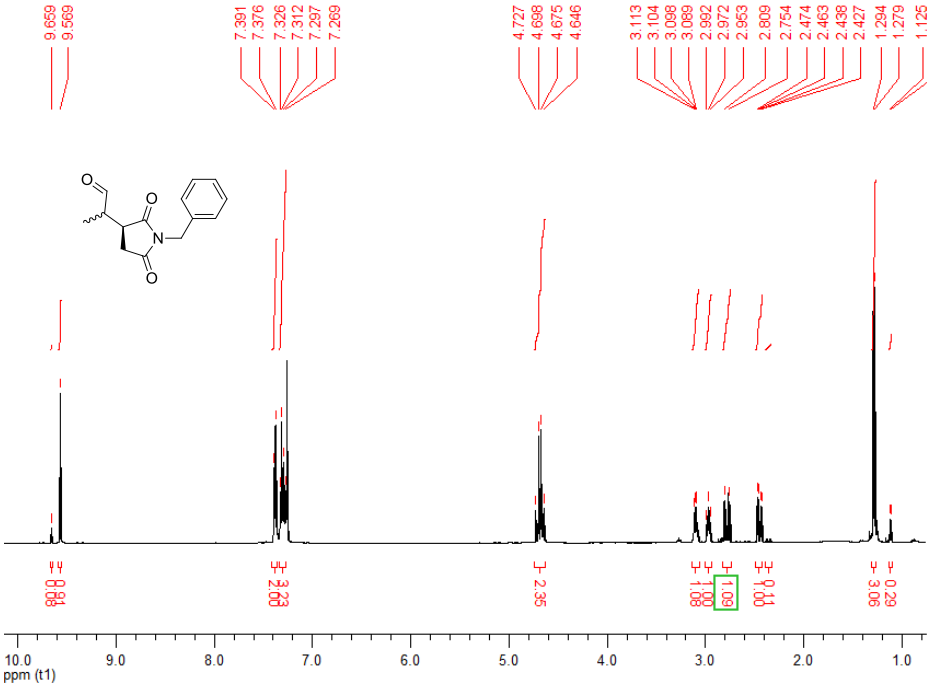


^{13}C-NMR, 125 MHz, $CDCl_3$

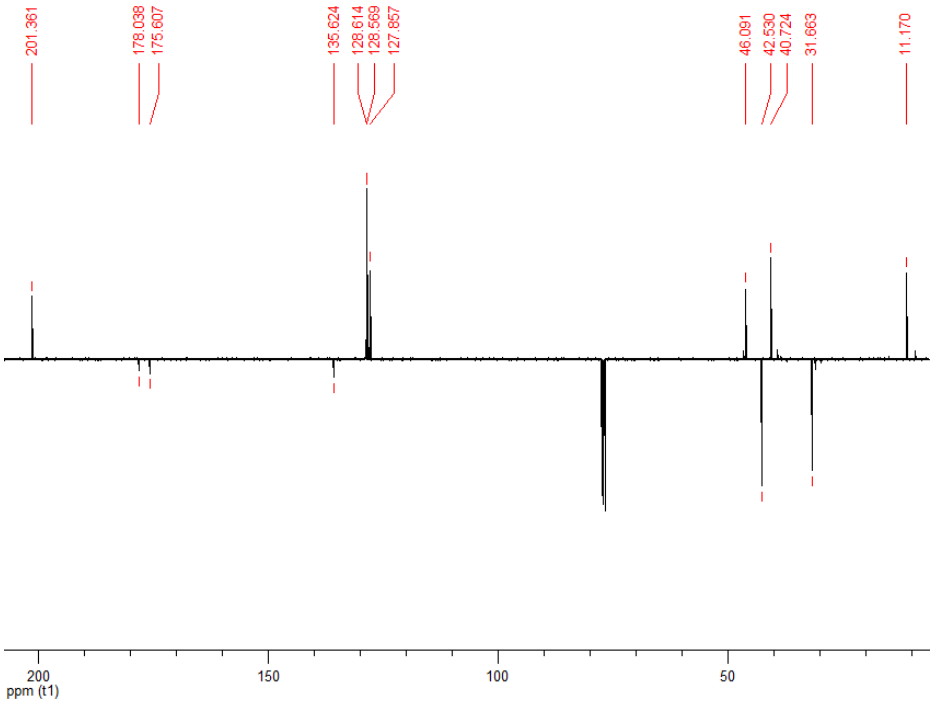


2-((*S*)-1-benzyl-2,5-dioxopyrrolidin-3-yl)propanal (**6g**), *syn*

^{1}H-NMR, 500 MHz, $CDCl_3$

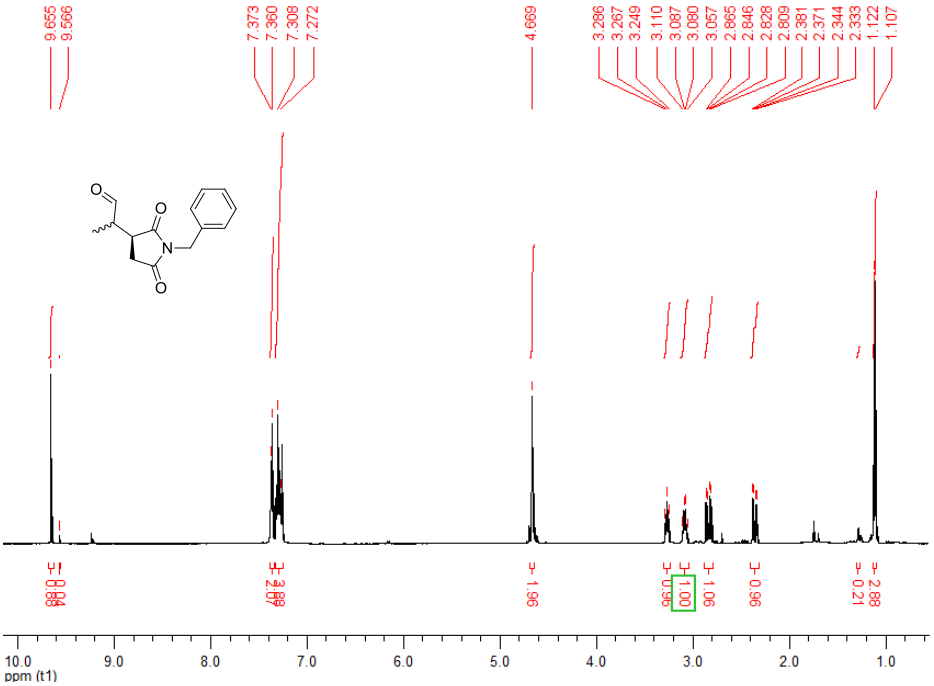


^{13}C-NMR, 125 MHz, $CDCl_3$

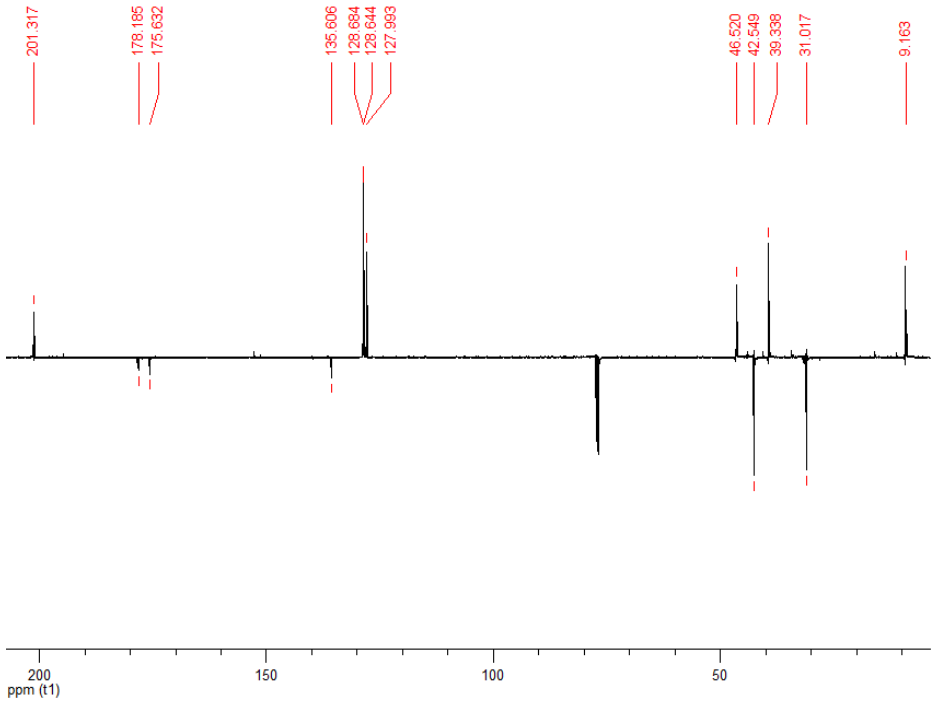


2-((*S*)-1-benzyl-2,5-dioxopyrrolidin-3-yl)propanal (**6g**), *anti*

^{1}H-NMR, 500 MHz, $CDCl_3$

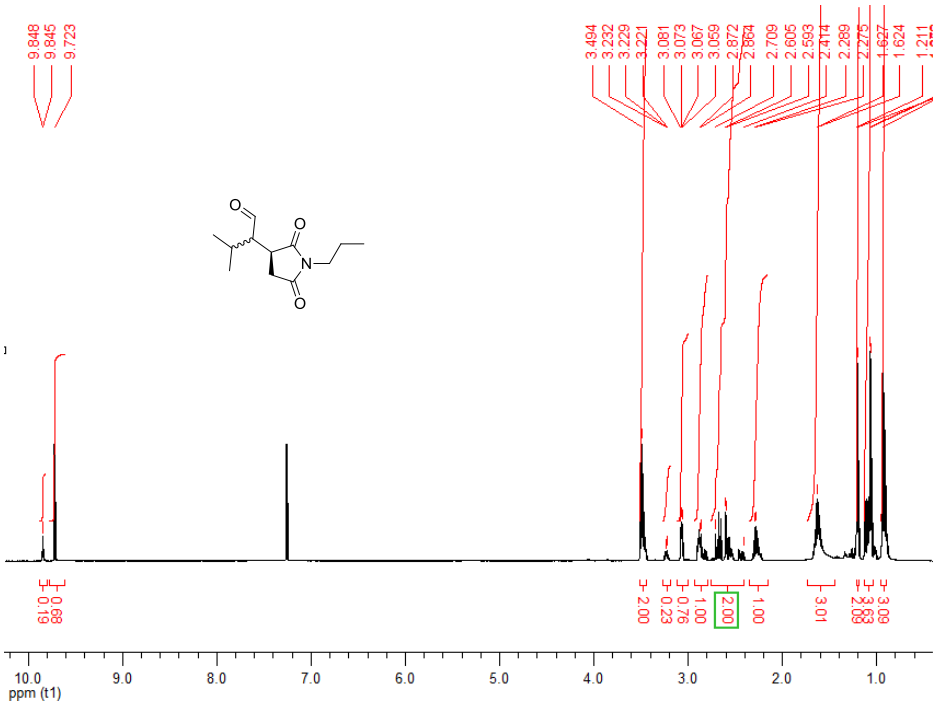


^{13}C-NMR, 125 MHz, $CDCl_3$

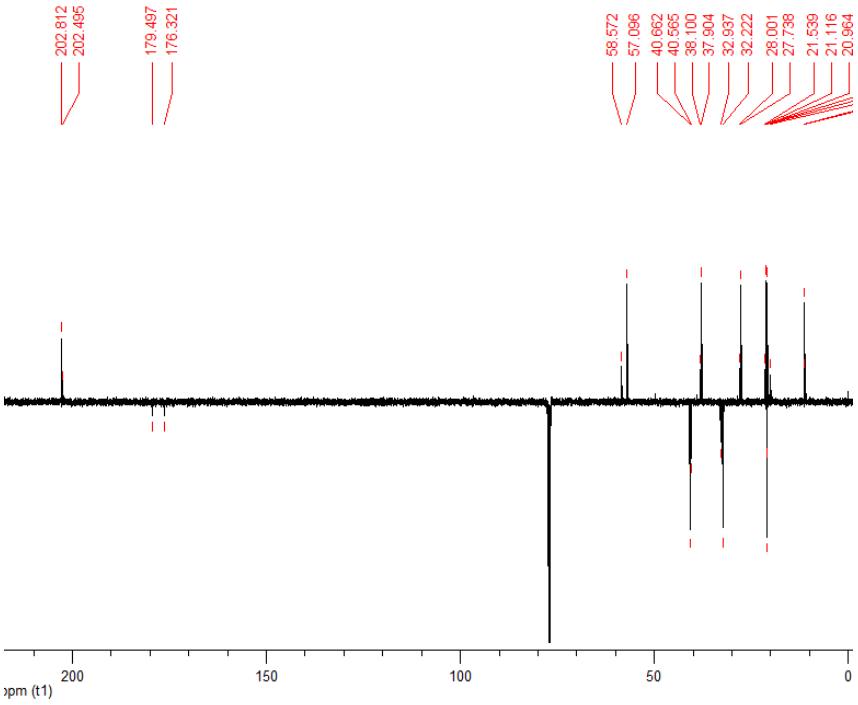


2-((*S*)-2,5-dioxo-1-propylpyrrolidin-3-yl)-3-methylbutanal (**6h**), *syn* + *anti*

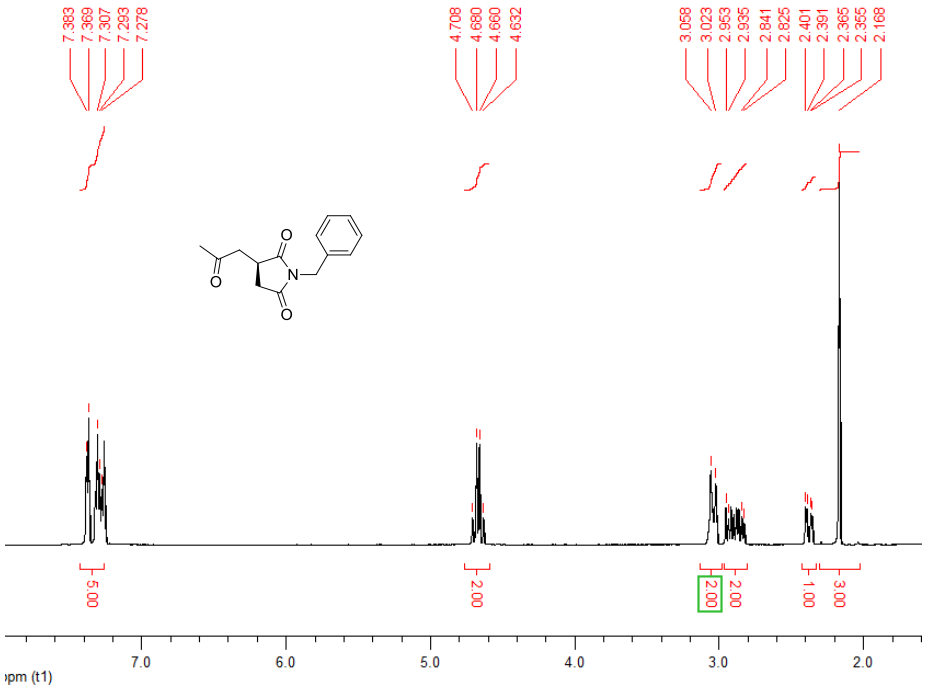
^{1}H-NMR, 500 MHz, $CDCl_3$



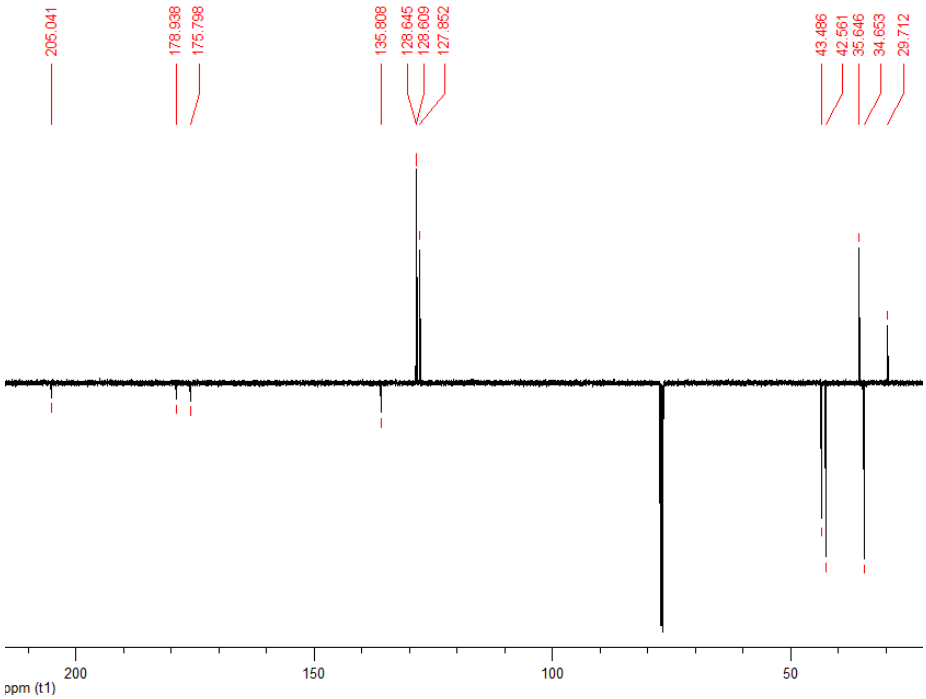
^{13}C-NMR, 125 MHz, $CDCl_3$



(*R*)-1-benzyl-3-(2-oxopropyl)pyrrolidine-2,5-dione (**6i**),
^{1}H-NMR, 500 MHz, $CDCl_3$

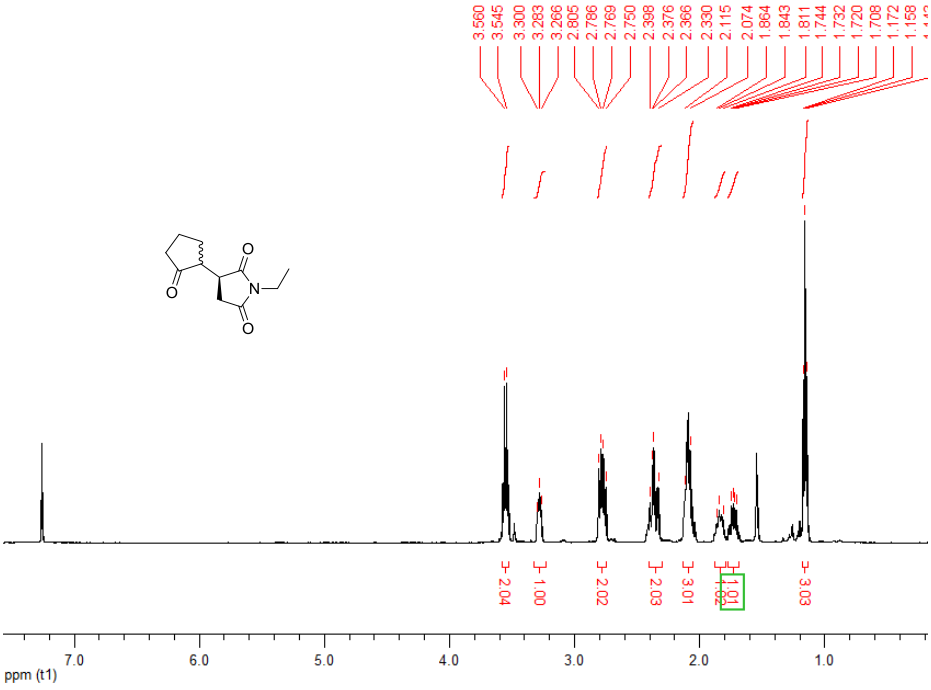


^{13}C-NMR, 125 MHz, $CDCl_3$

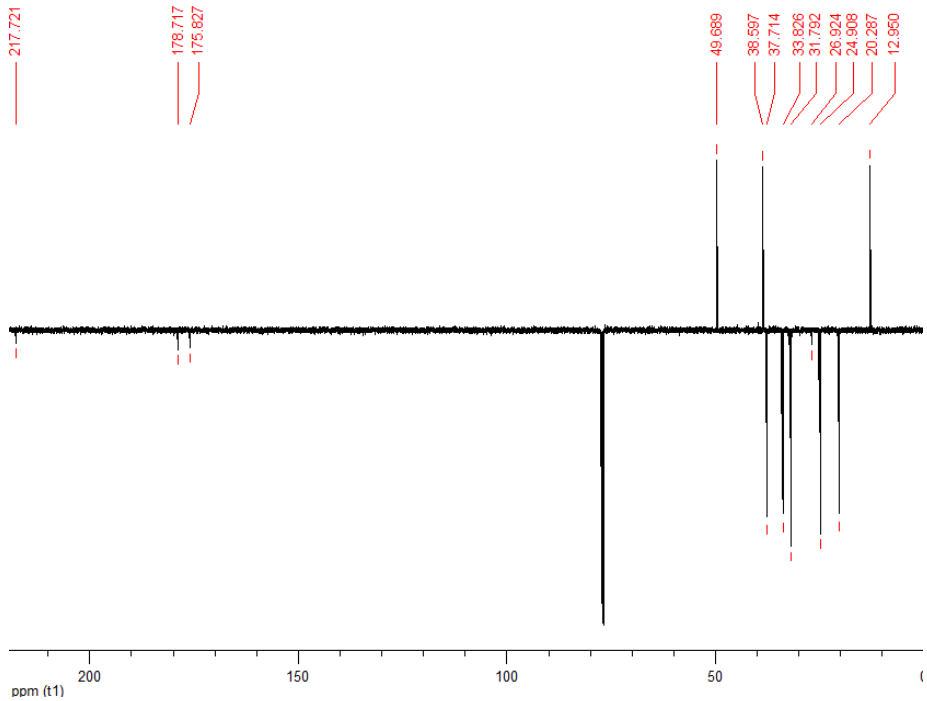


(3*S*)-1-ethyl-3-(2-oxocyclopentyl)pyrrolidine-2,5-dione (**6j**), *syn* + *anti*

^{1}H-NMR, 500 MHz, $CDCl_3$

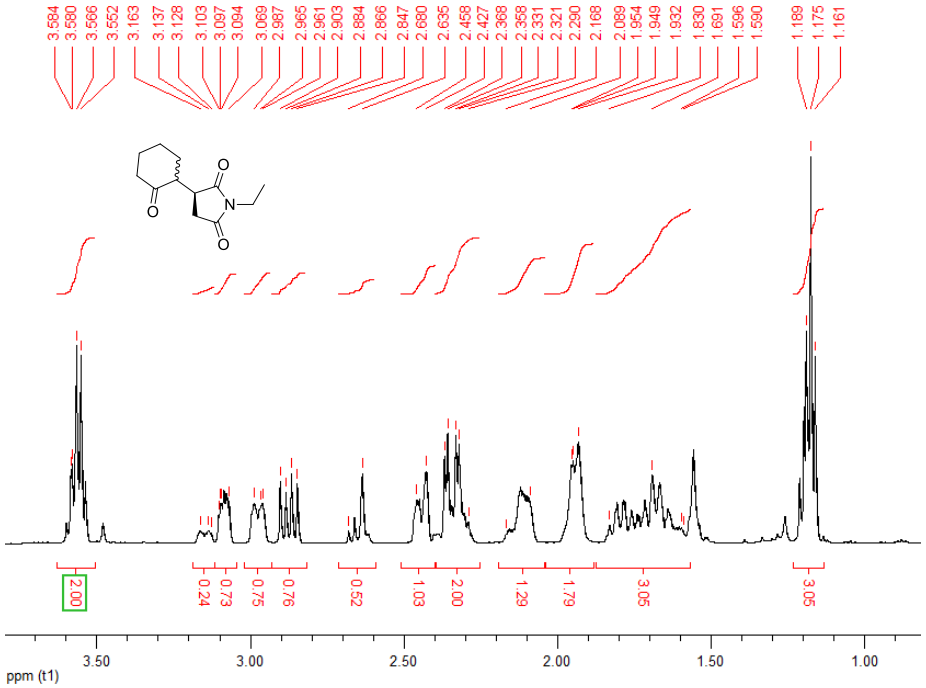


^{13}C-NMR, 125 MHz, $CDCl_3$

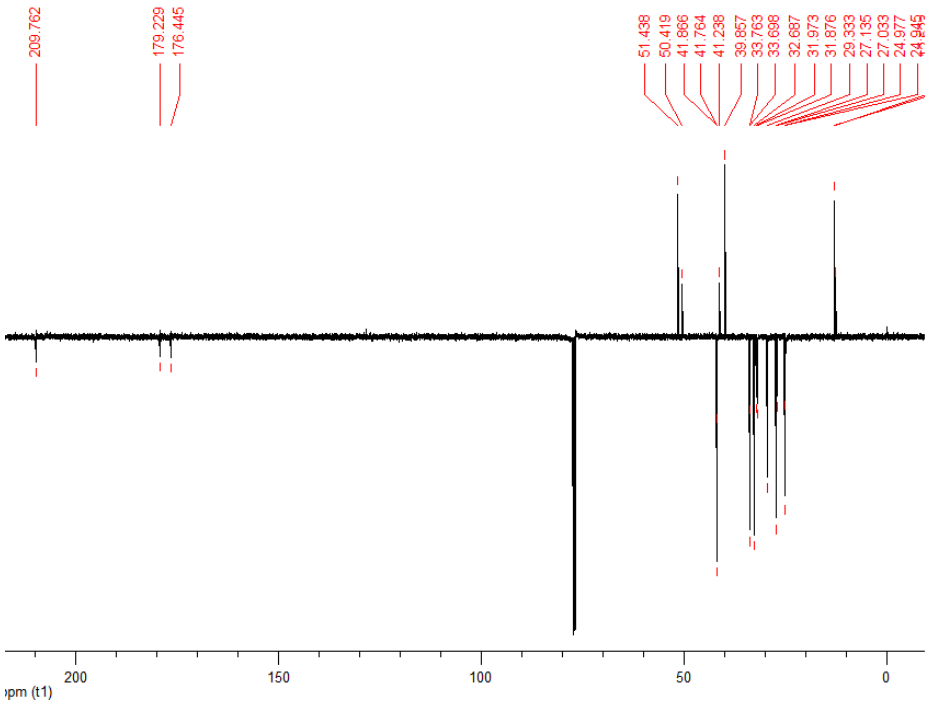


(3*S*)-1-ethyl-3-(2-oxocyclohexyl)pyrrolidine-2,5-dione (**6k**), *syn* + *anti*

^{1}H-NMR, 500 MHz, $CDCl_3$



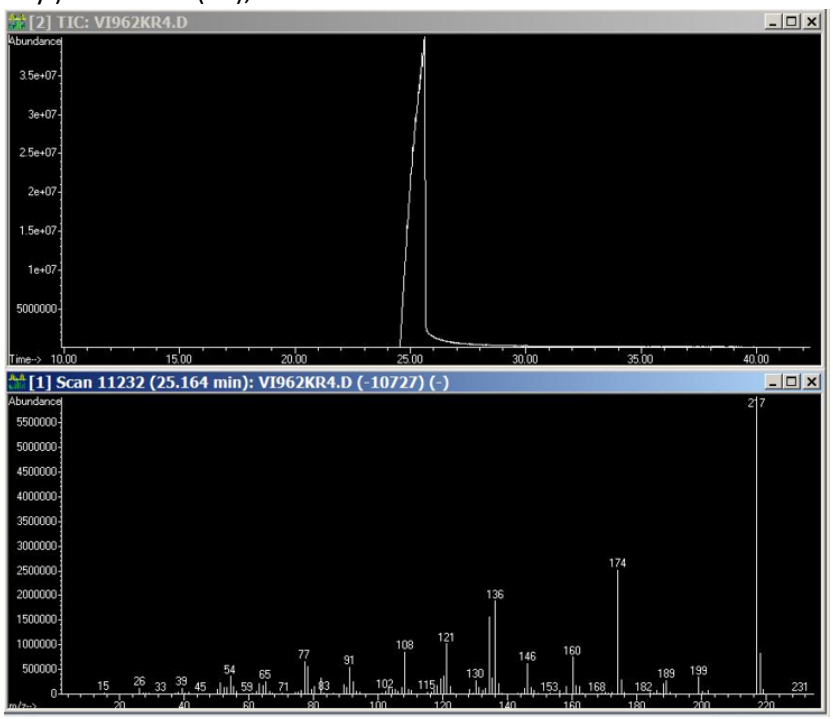
^{13}C-NMR, 125 MHz, $CDCl_3$



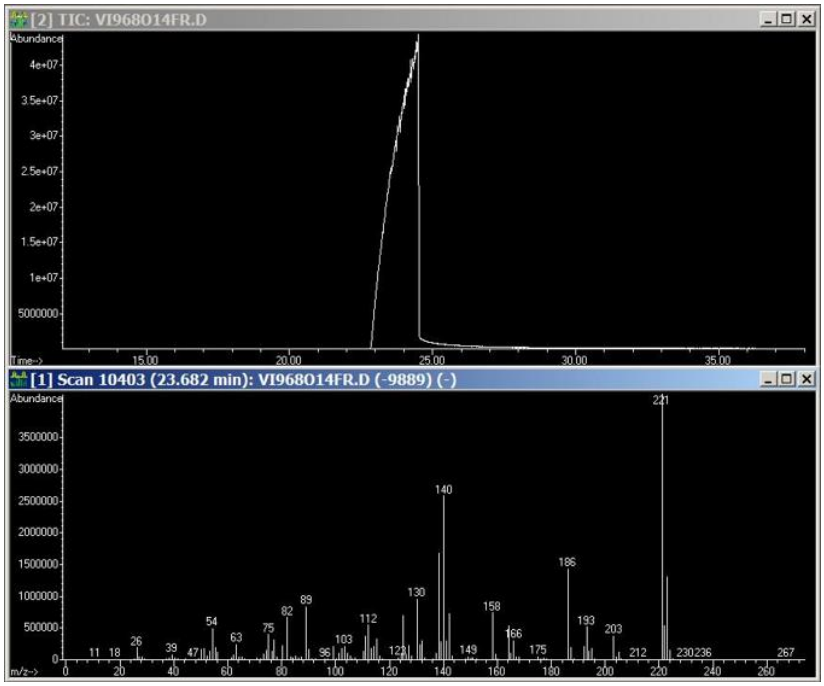
15. Chromatograms and mass spectra of the materials

15.1. GC-MSD chromatograms and mass spectra of the *N*-substituted maleimides

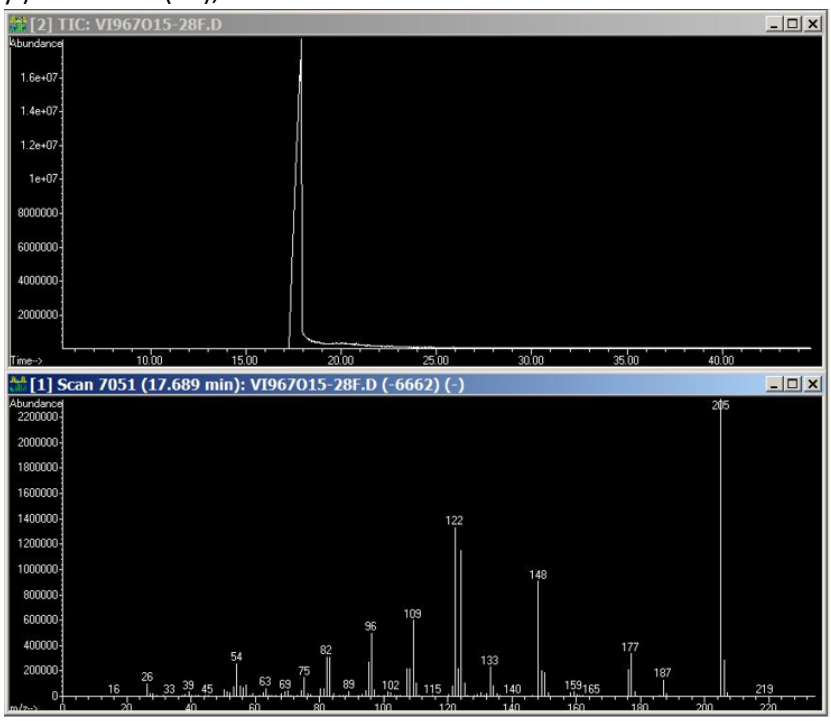
N-(4-methoxybenzyl)maleimide (**2b**),



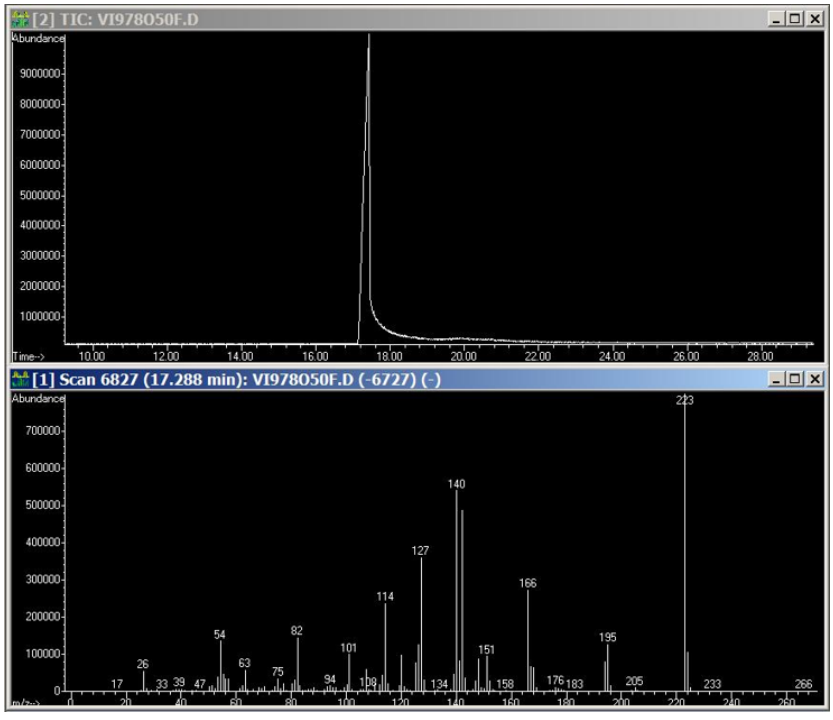
N-(4-chlorobenzyl)maleimide (**2c**),



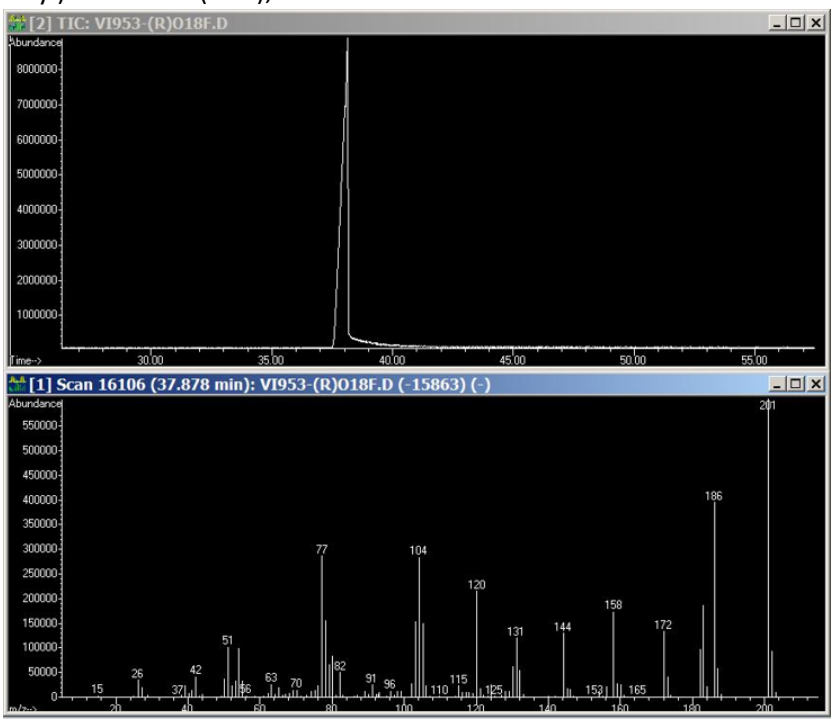
N-(4-fluorobenzyl)maleimide (**2d**),



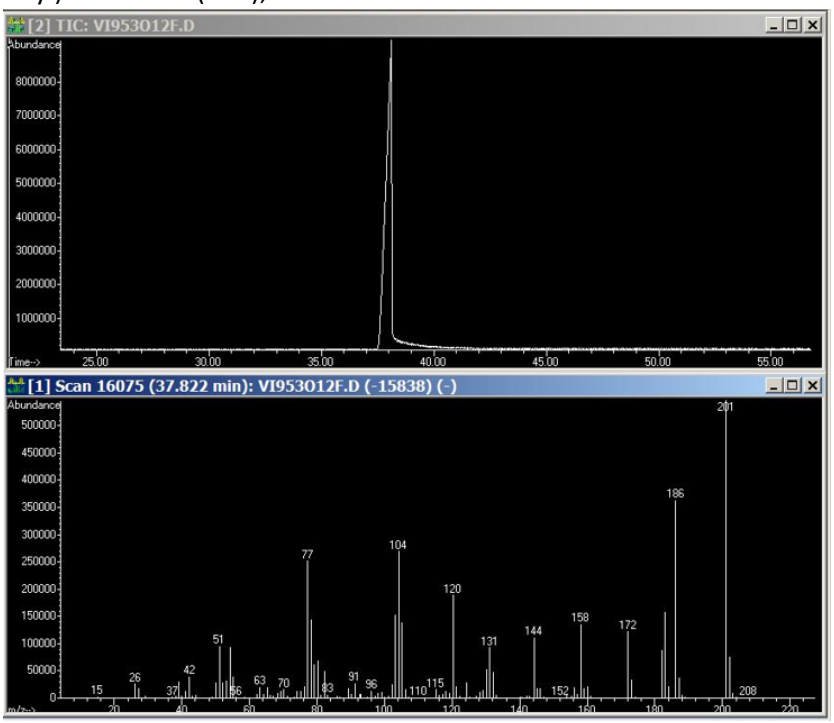
N-(2,4-difluorobenzyl)maleimide (**2e**),



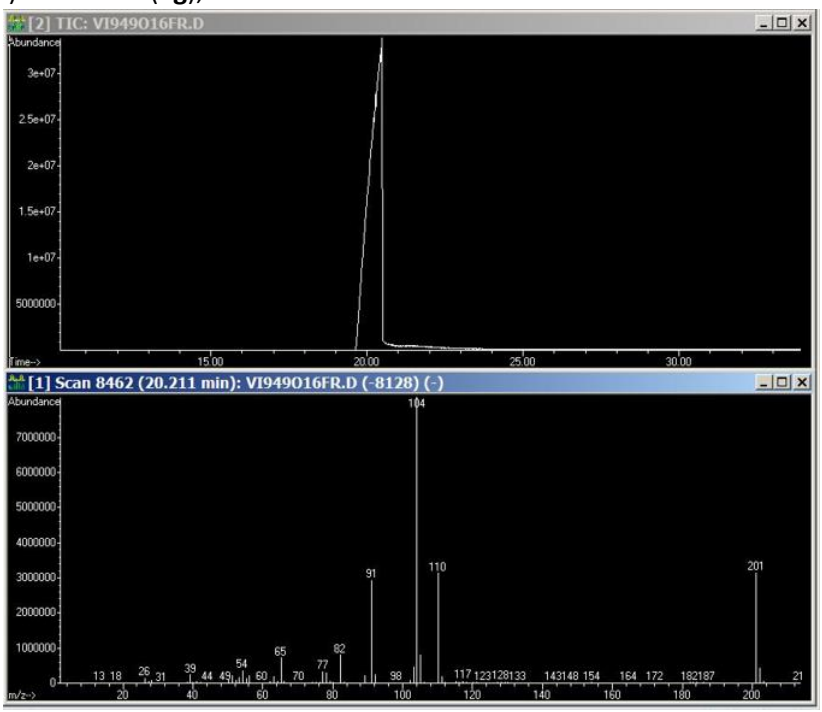
(*R*)-*N*-(1-phenylethyl)maleimide (*R*-**2f**),



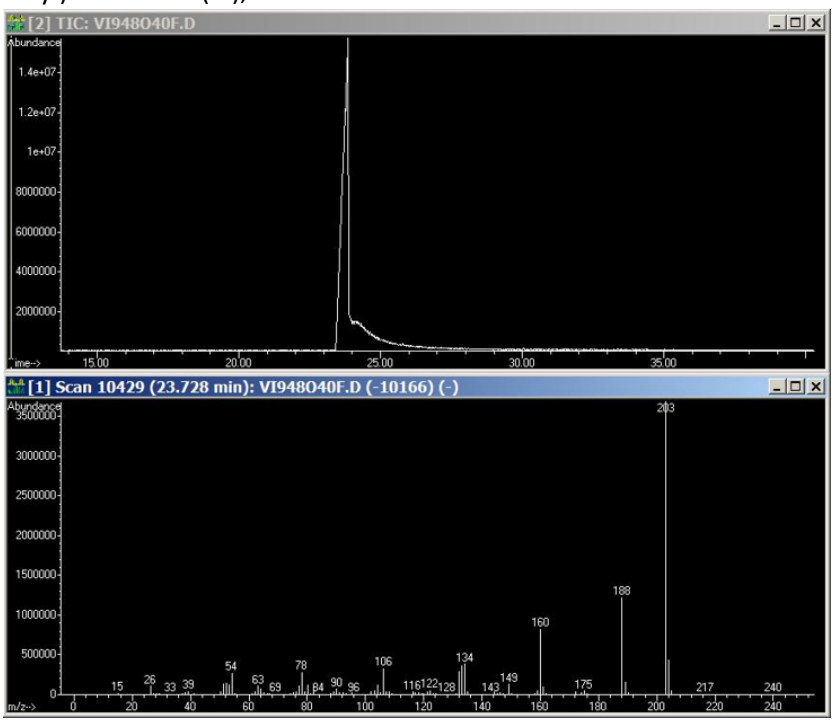
(*S*)-*N*-(1-phenylethyl)maleimide (*S*-**2f**),



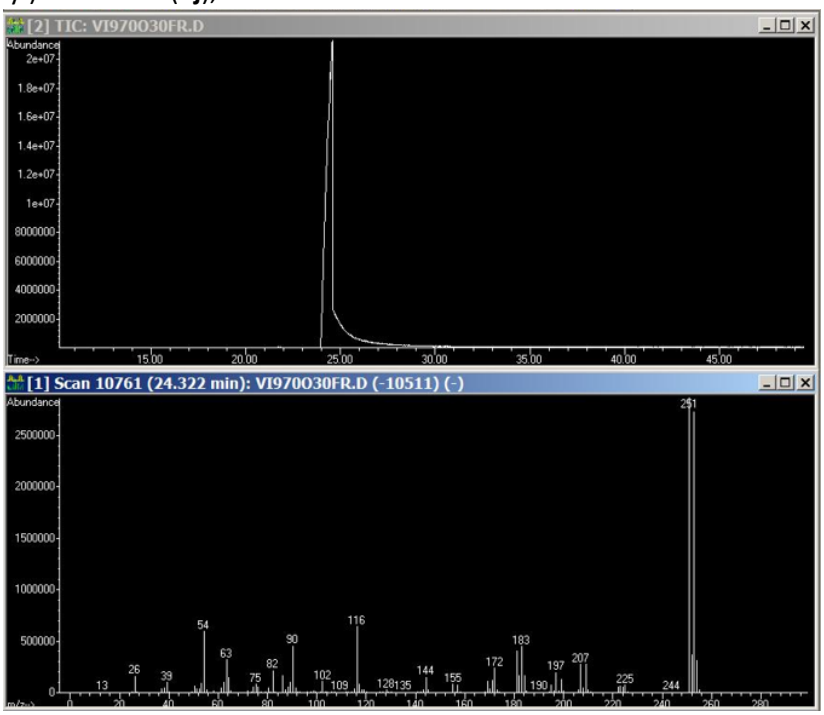
N-(2-phenylethyl)maleimide (**2g**),



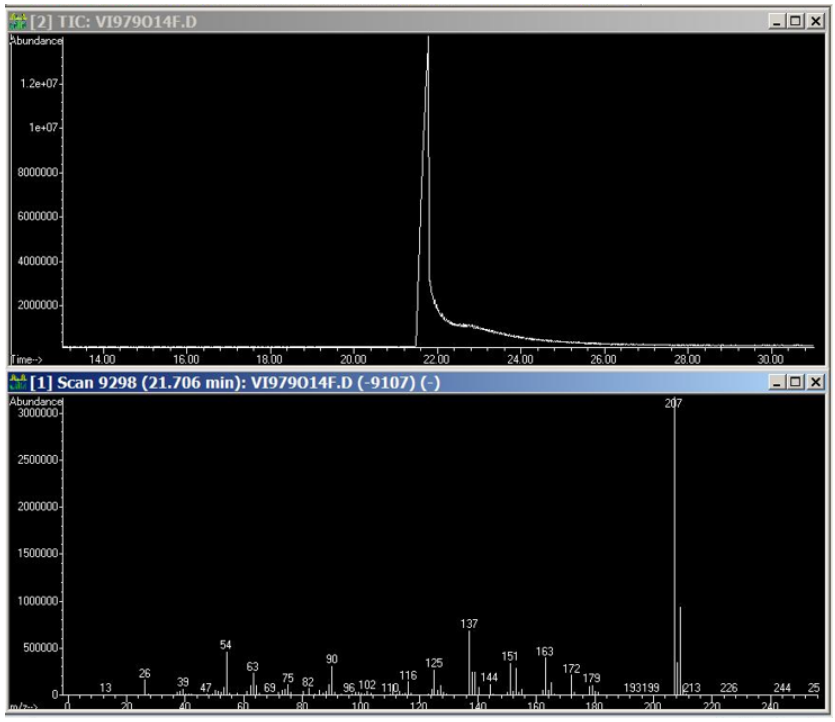
N-(4-methoxyphenyl)maleimide (**2i**),



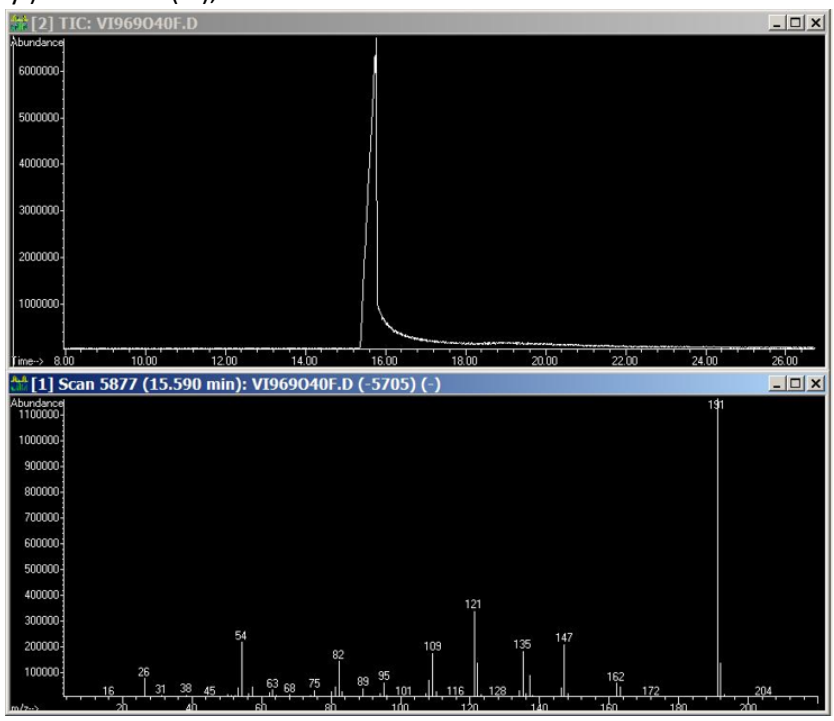
N-(4-bromophenyl)maleimide (**2j**),



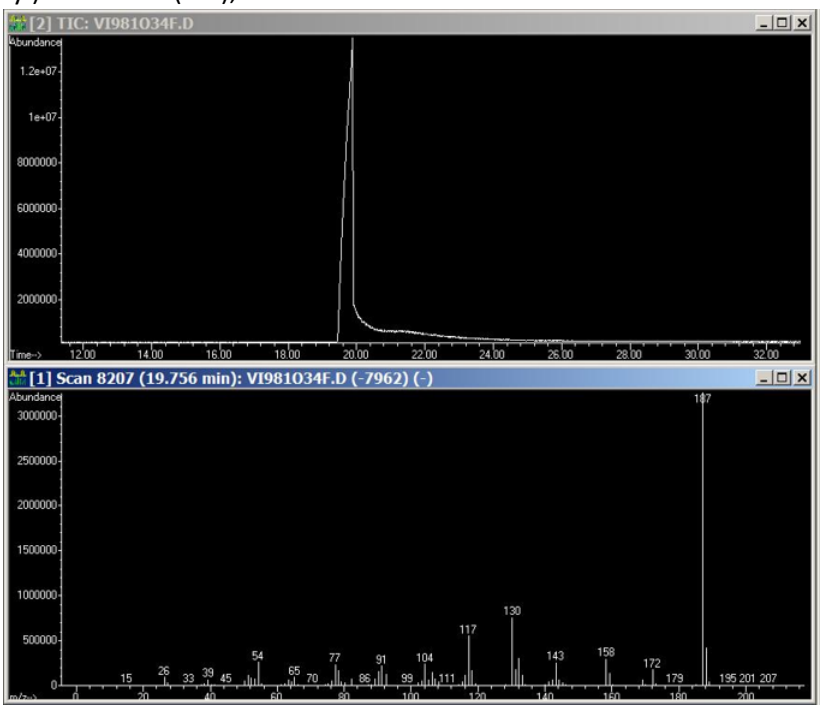
N-(4-chlorophenyl)maleimide (**2k**),



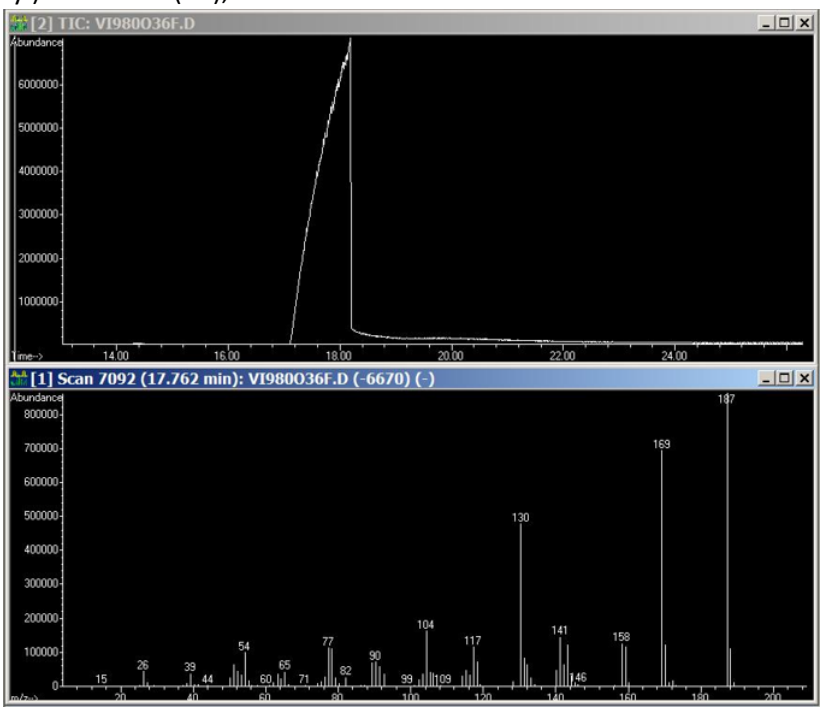
N-(4-fluorophenyl)maleimide (**2l**),



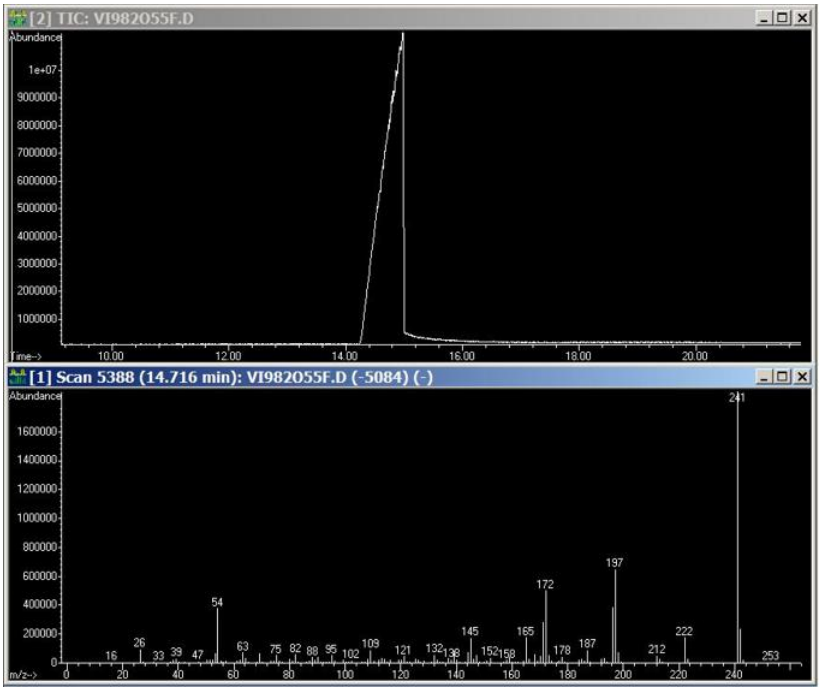
N-(4-methylphenyl)maleimide (**2m**),



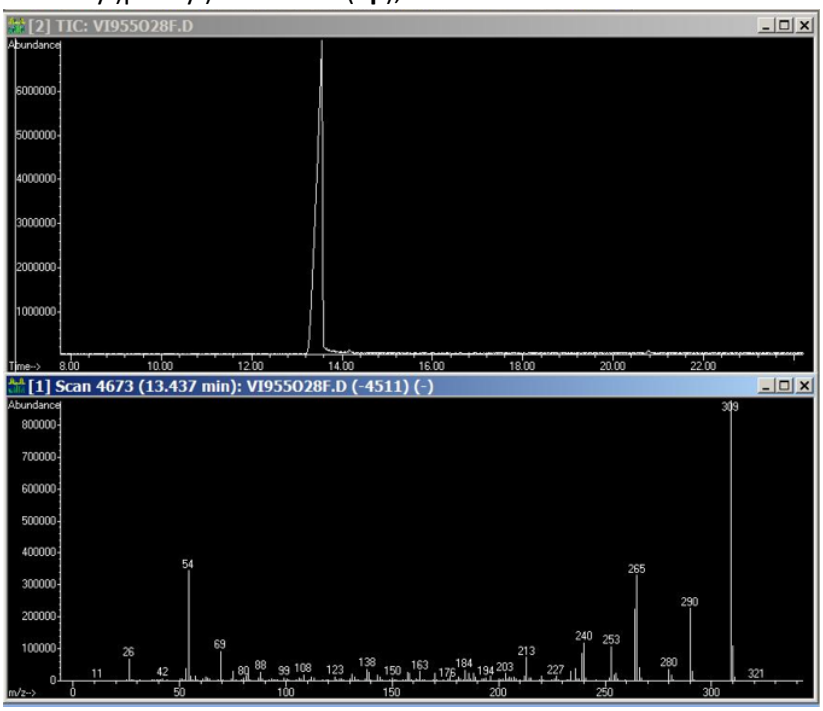
N-(2-methylphenyl)maleimide (**2n**),



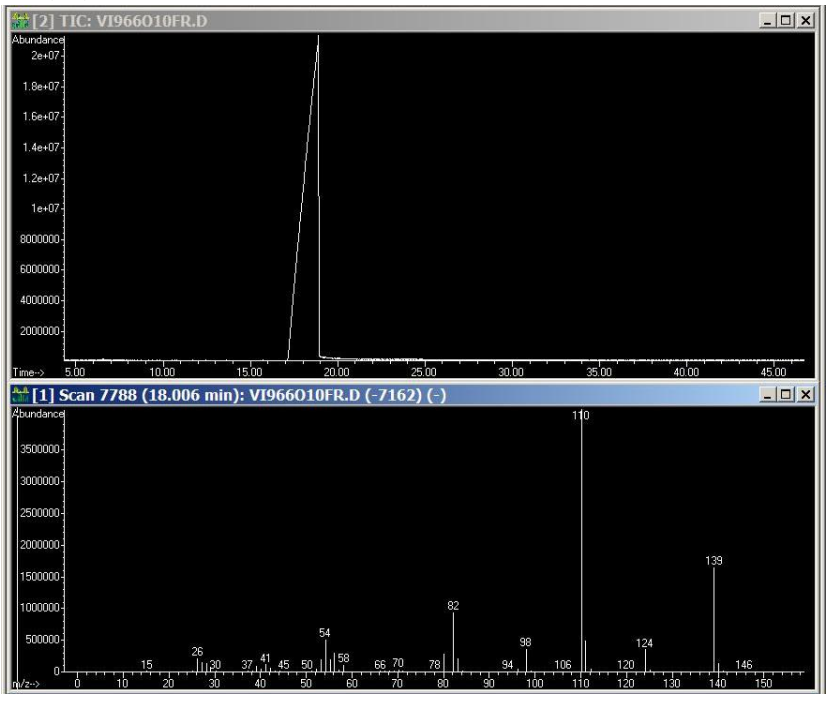
N-((2-trifluoromethyl)phenyl)maleimide (**2o**),



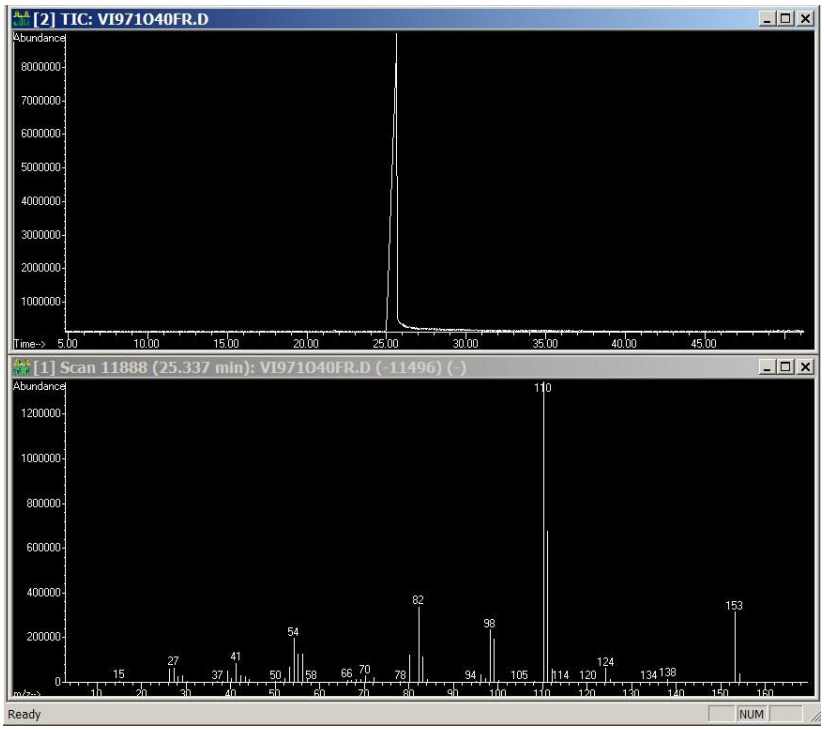
N-((3,5-bistrifluoromethyl)phenyl)maleimide (**2p**),



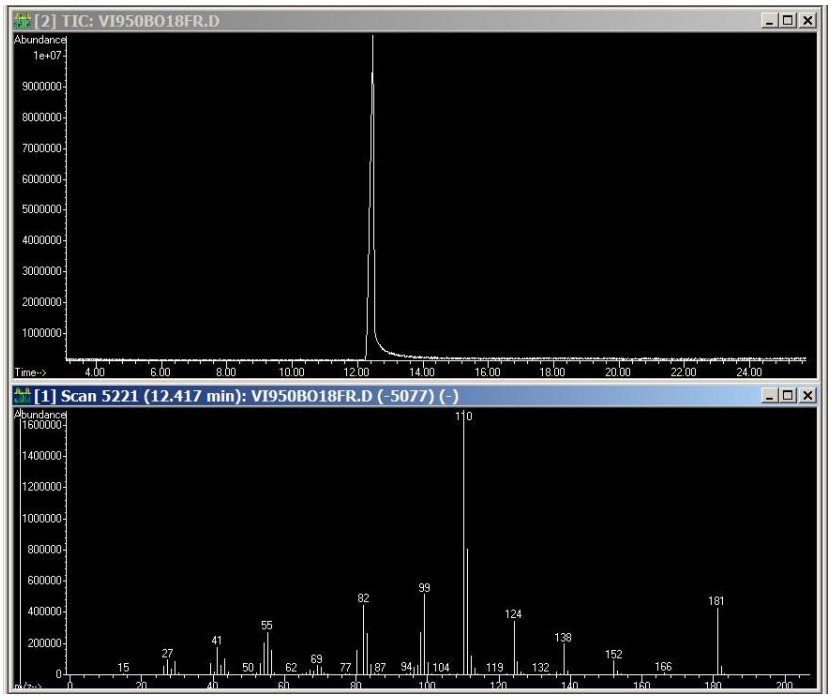
N-propylmaleimide (**4c**),



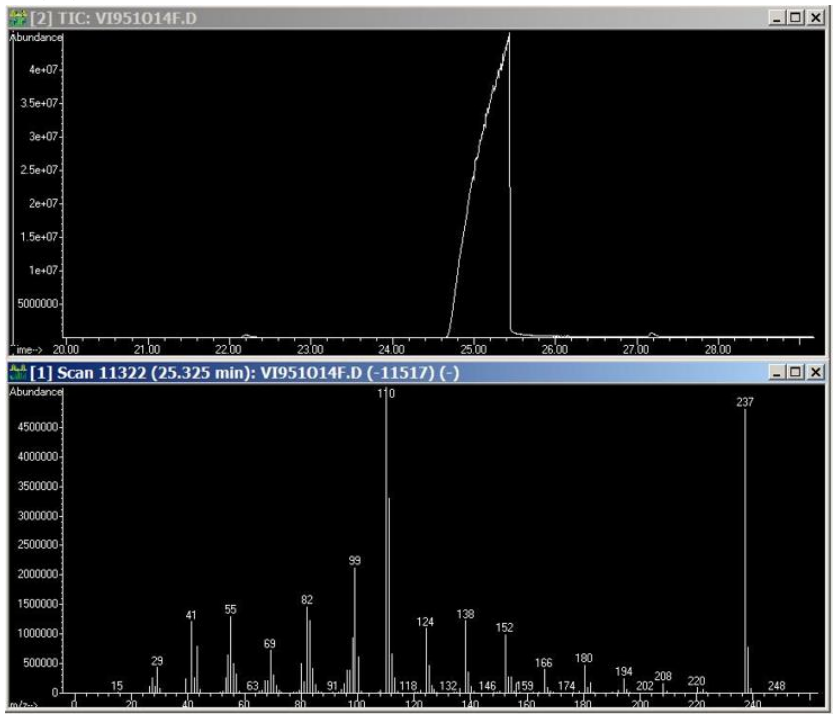
N-butylmaleimide (**4d**),



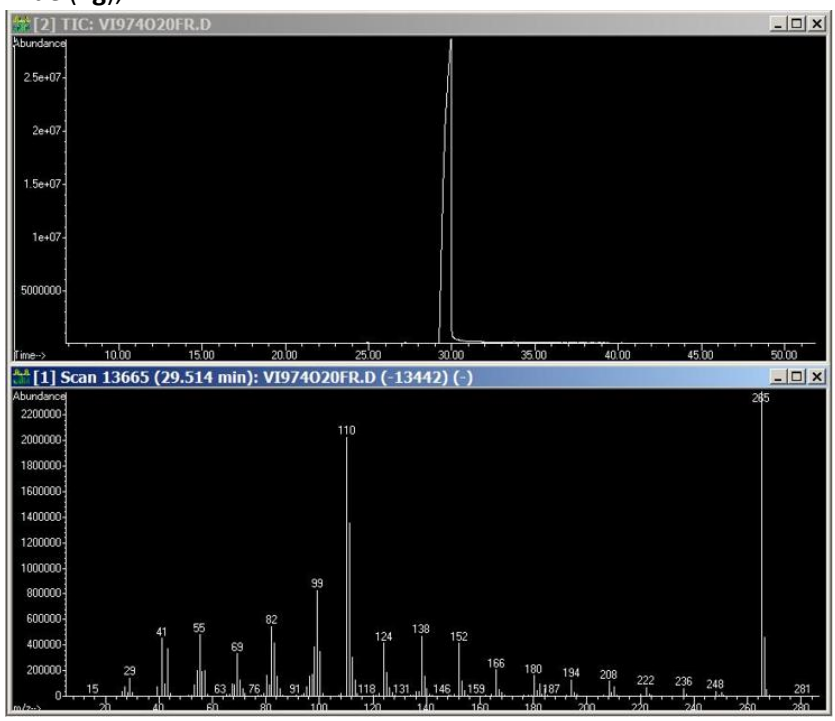
N-hexylmaleimide (**4e**),



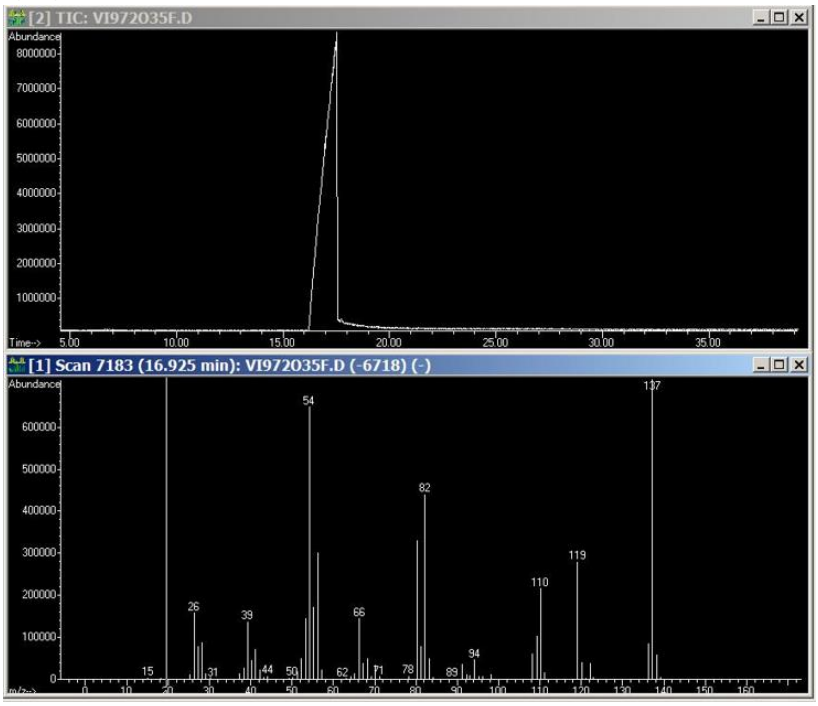
N-decylmaleimide (**4f**),



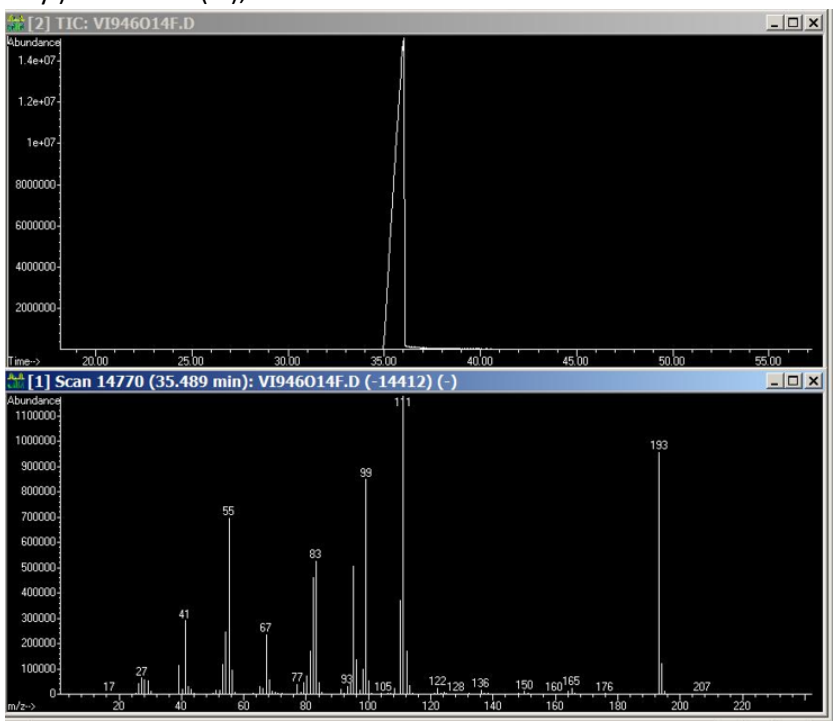
N-dodecylmaleimide (**4g**),



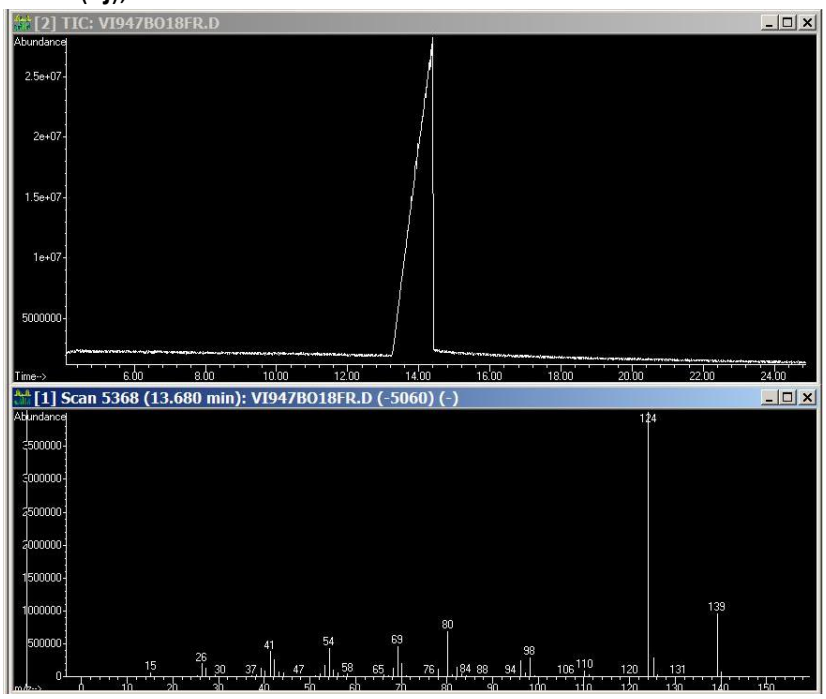
N-allylmaleimide (**4h**),



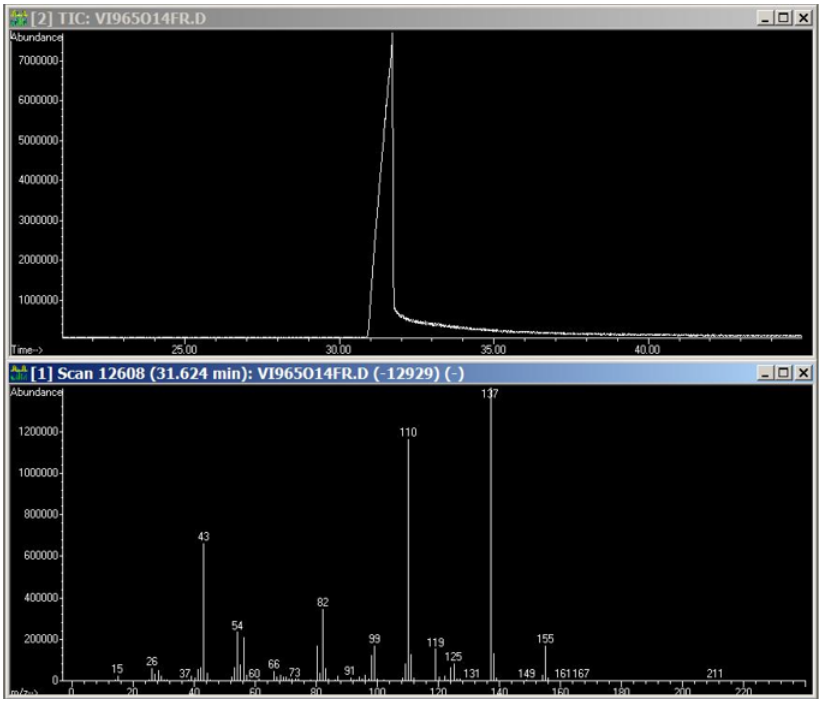
N-(cyclohexylmethyl)maleimide (**4i**),



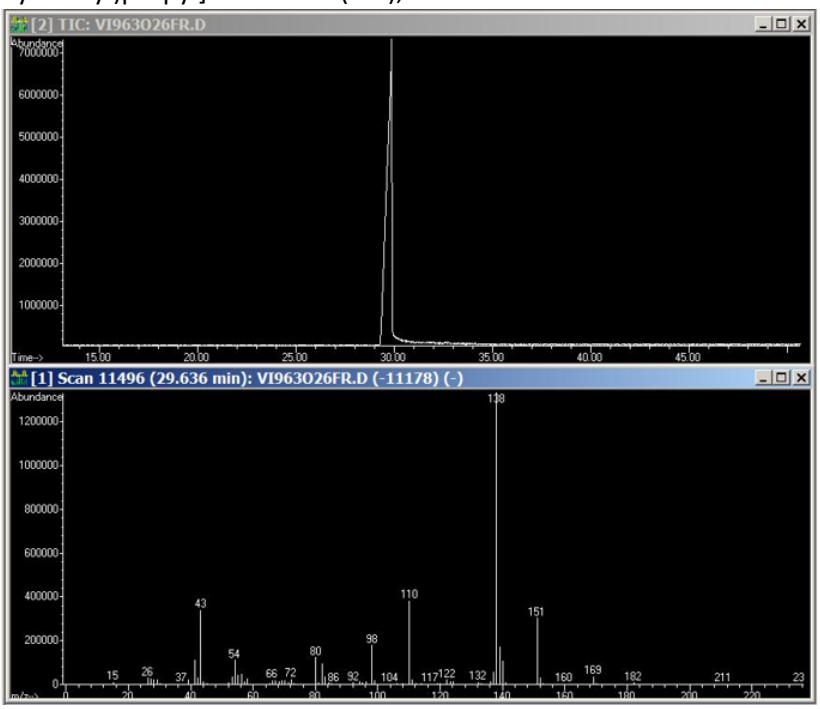
N-isopropylmaleimide (**4j**),



N-(3-acetoxypropyl)maleimide (**4l**),

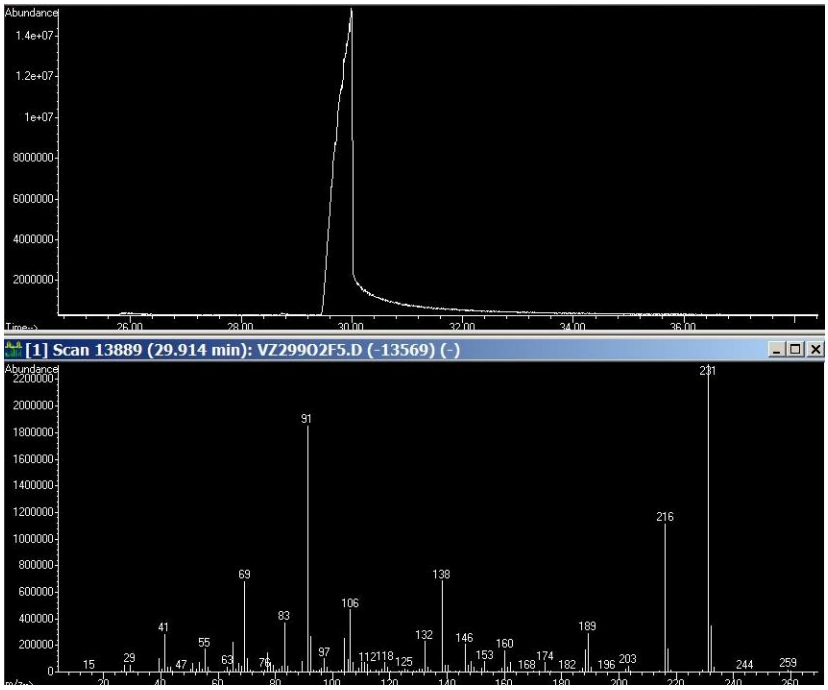


(*R*)-*N*-[(1-acetyloxymethyl)propyl]maleimide (**4m**),

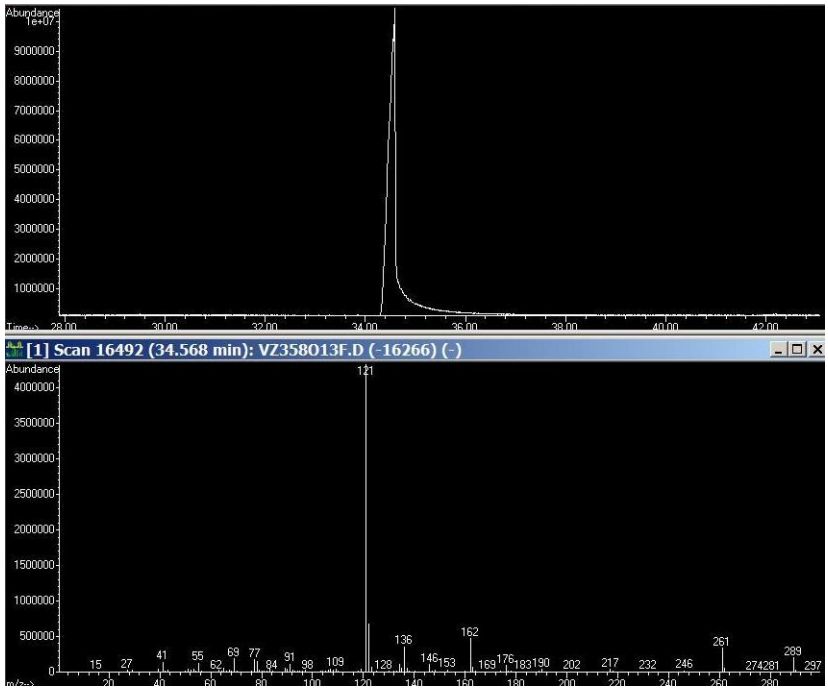


15.2. GC-MSD chromatograms and mass spectra of the Michael adducts

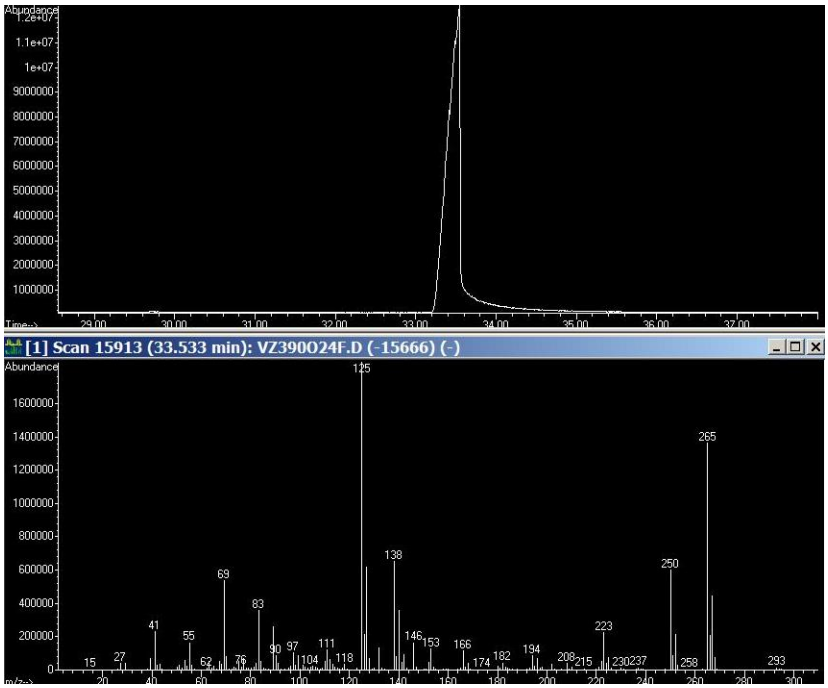
2-(1-benzyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3a**),



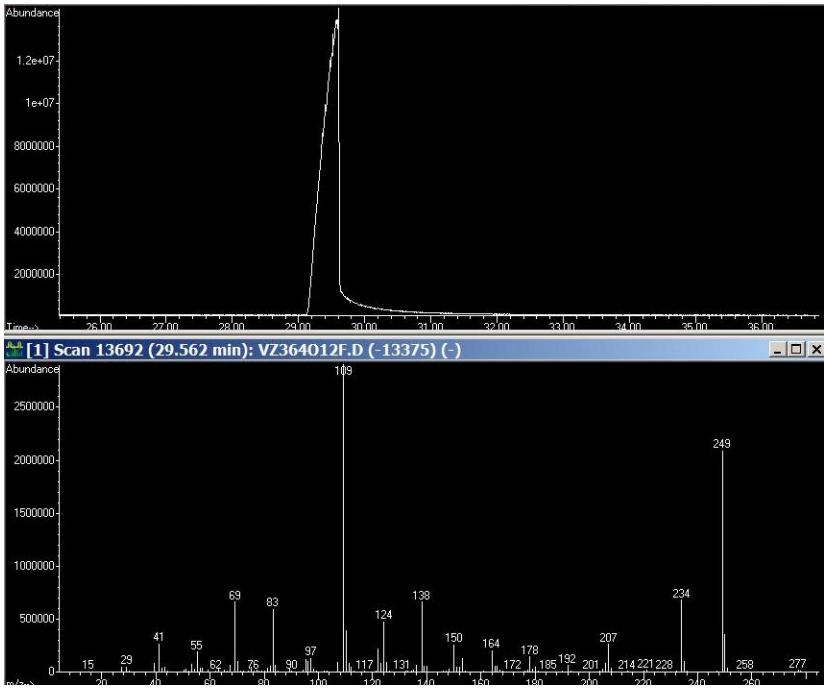
2-(1-(4-methoxybenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3b**),



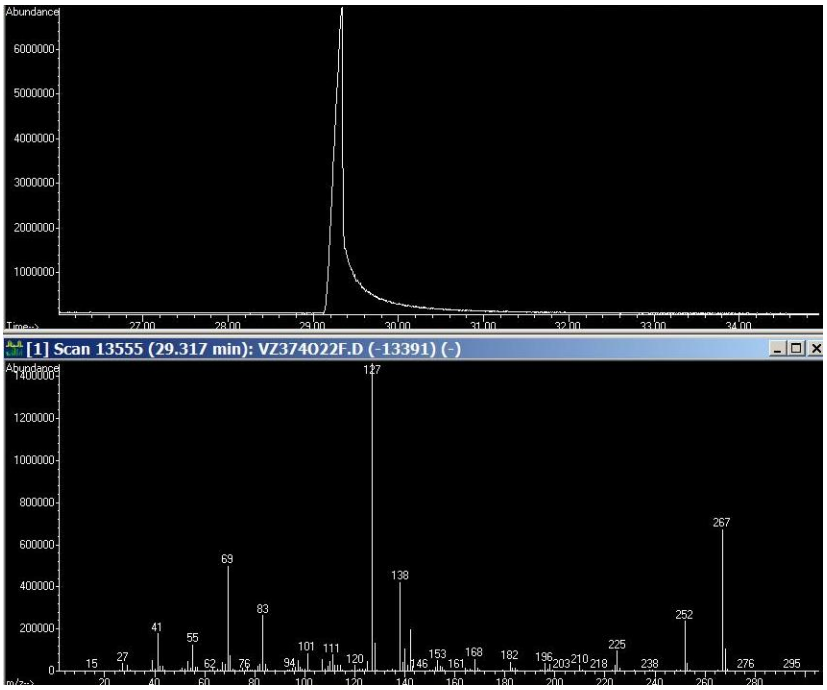
2-(1-(4-chlorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3c**),



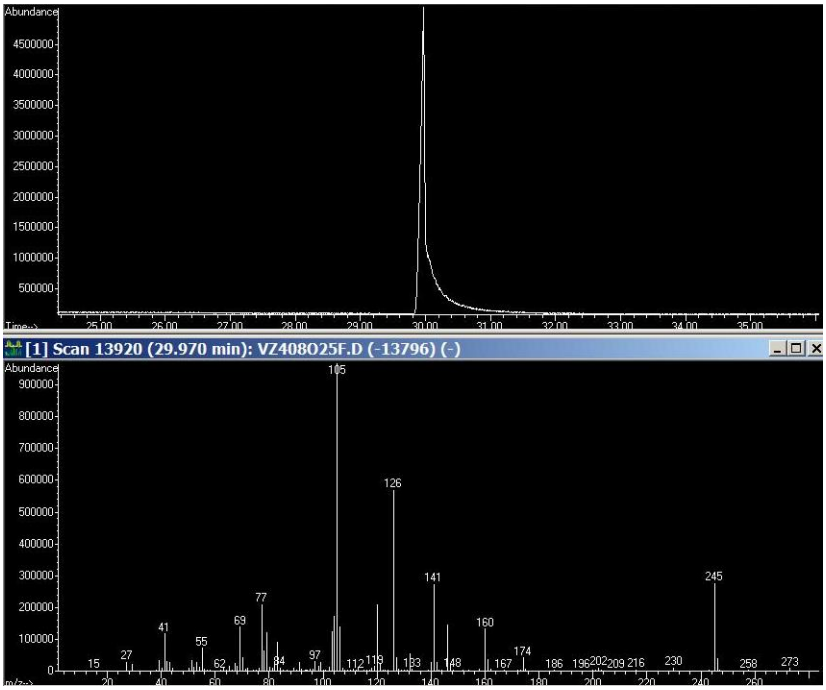
2-(1-(4-fluorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3d**),



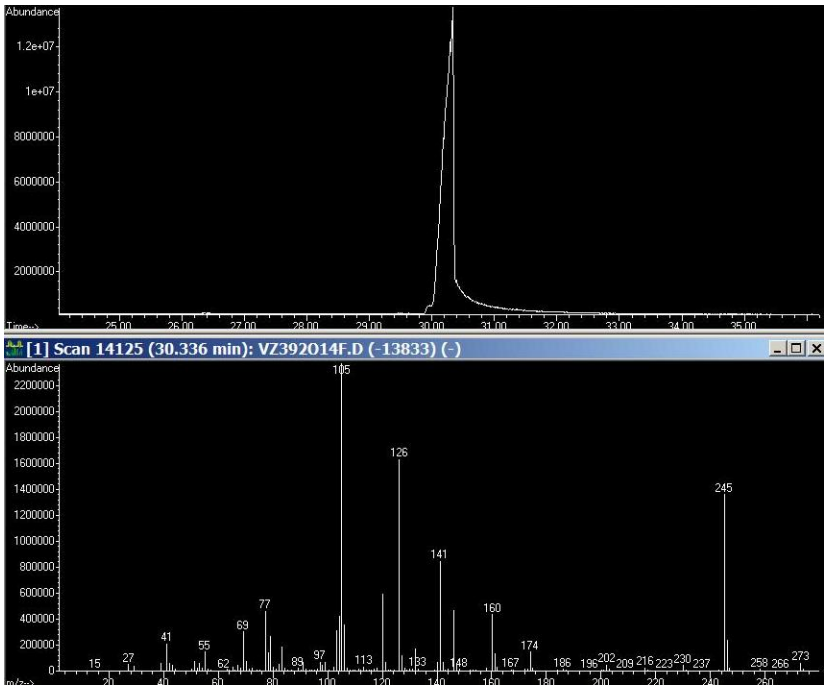
2-(1-(2,4-difluorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3e**),



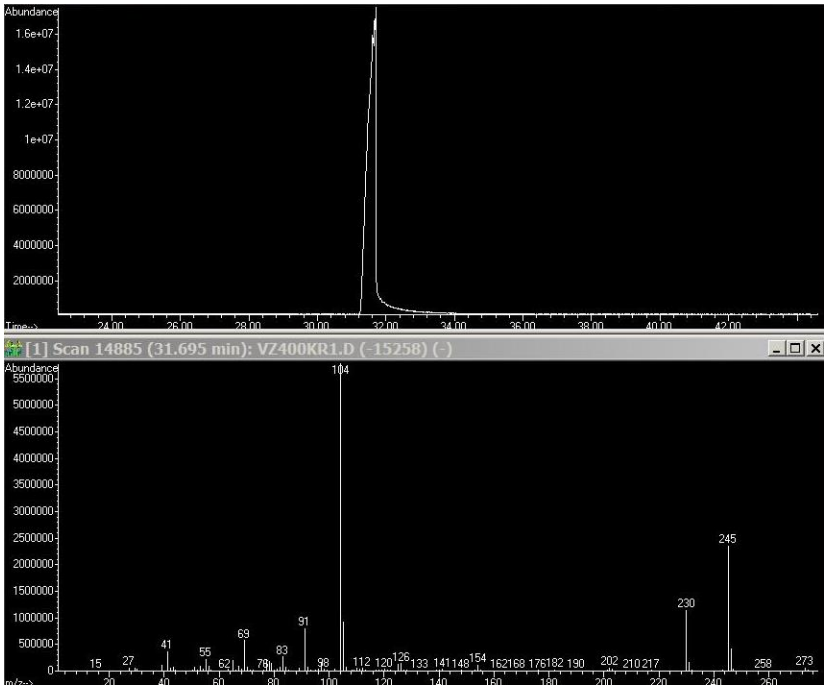
2-((*S*)-1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal ((*S*,*S*)-**3f**),



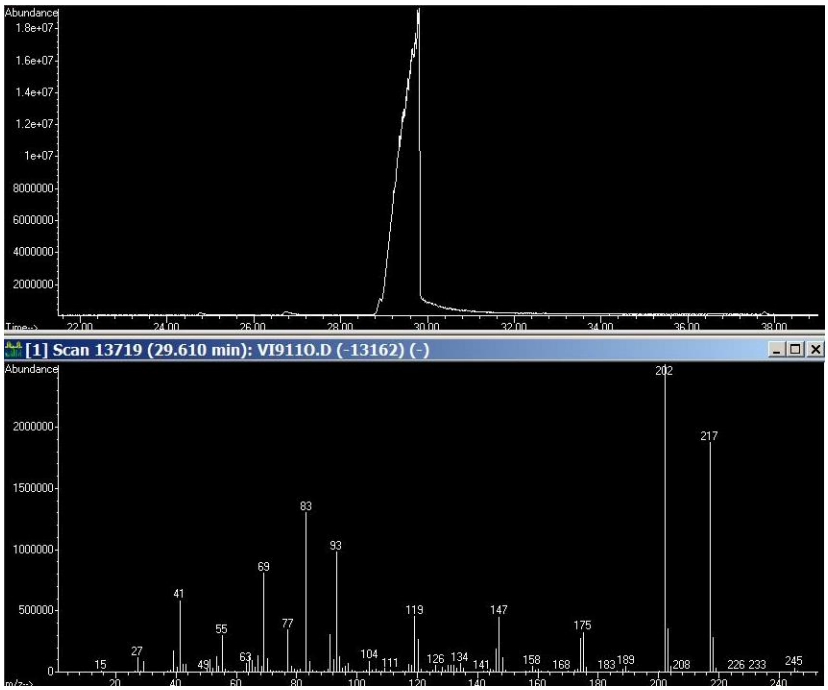
2-((*R*)-1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal ((*S*,*R*)-**3f**),



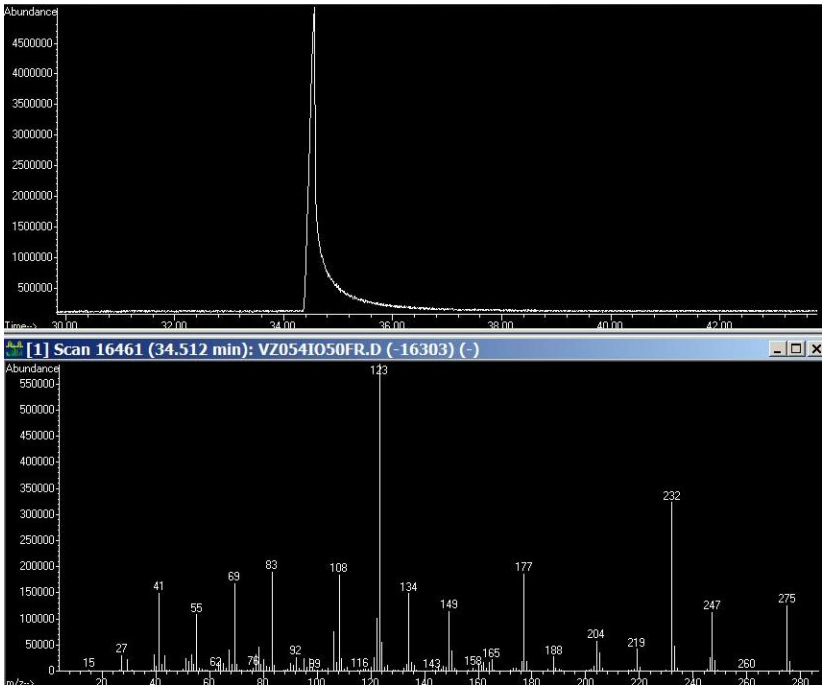
2-(1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3g**),



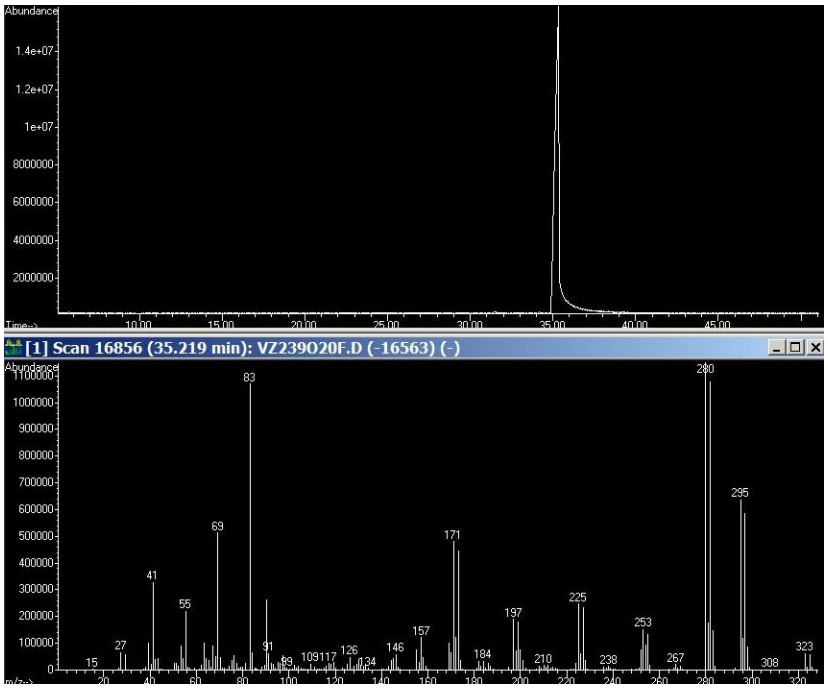
2-(1-phenyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3h**),



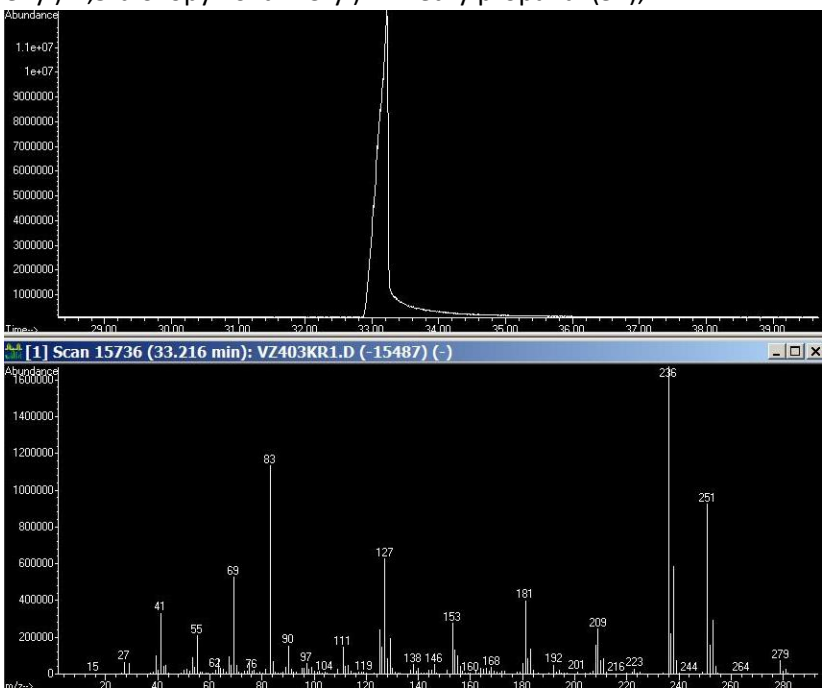
2-(1-(4-methoxyphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3i**),



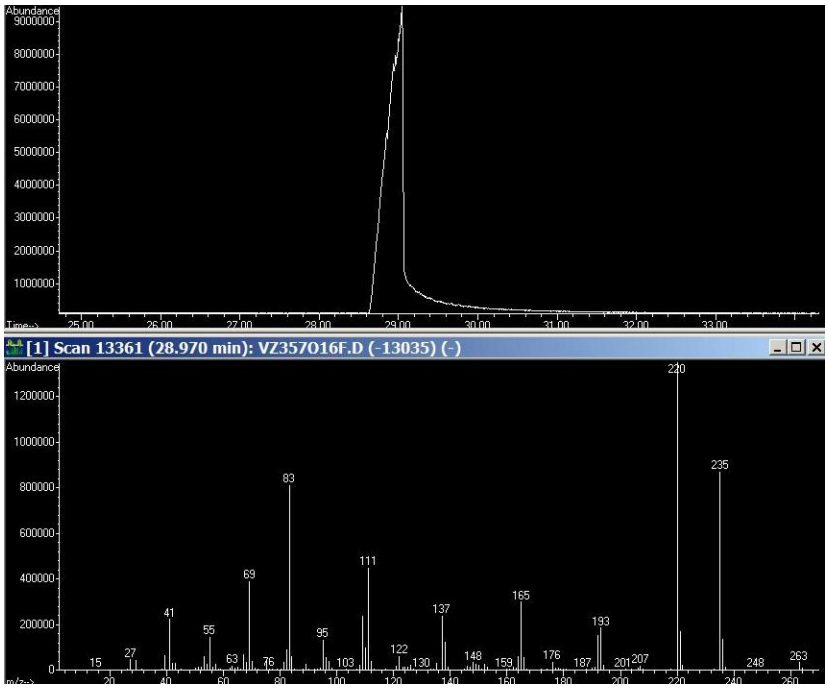
2-(1-(4-bromophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3j**),



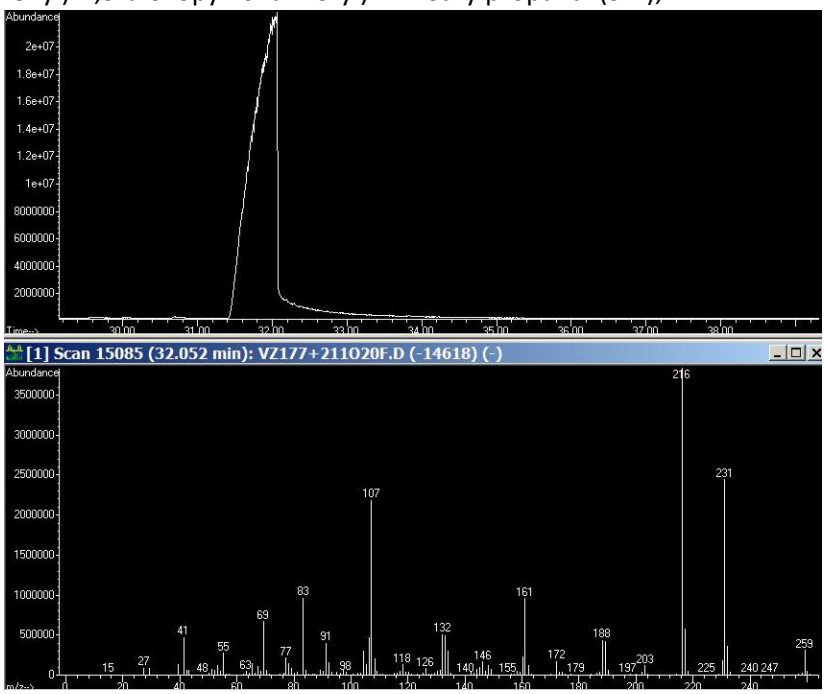
2-(1-(4-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3k**),



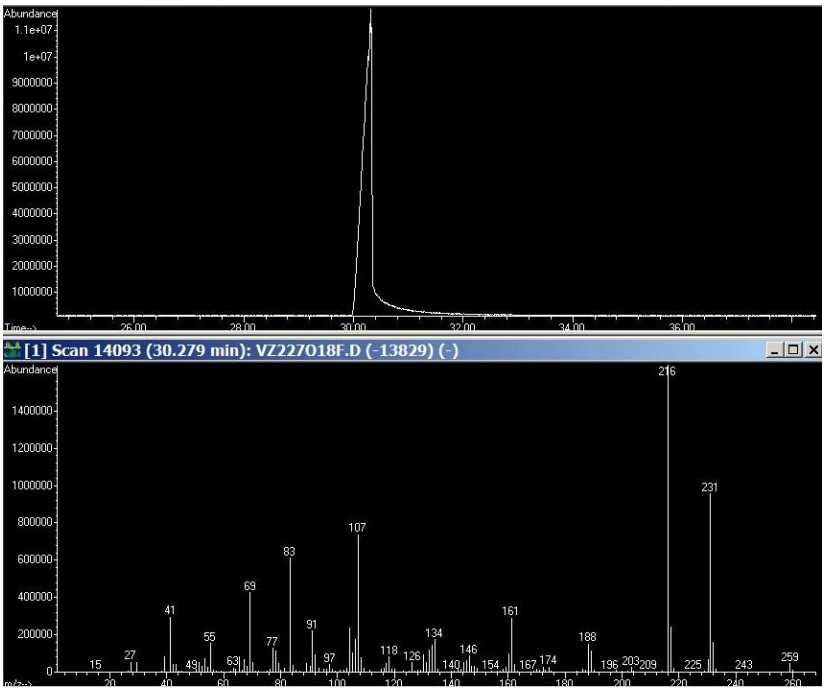
2-(1-(4-fluorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3l**),



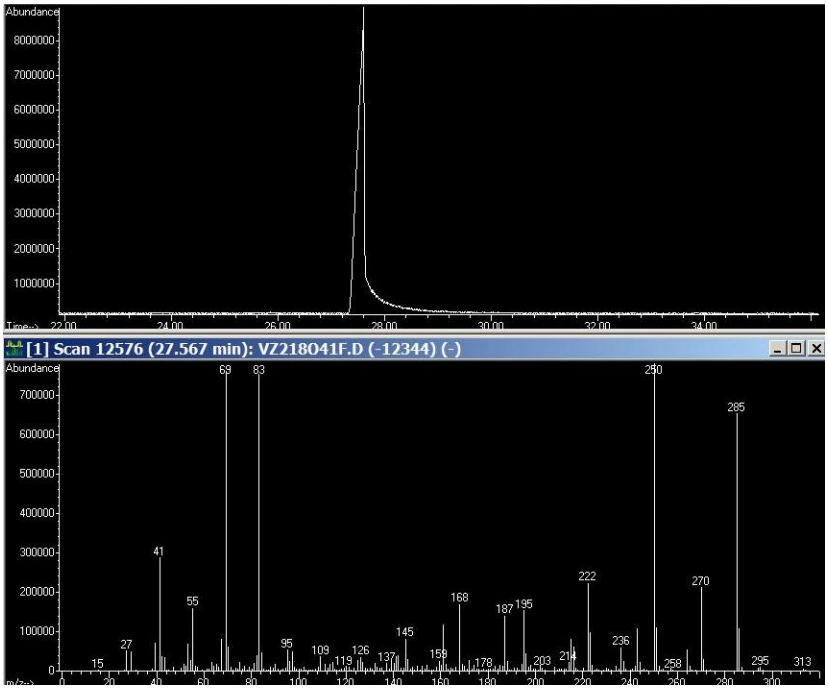
2-(1-(4-methylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3m**),



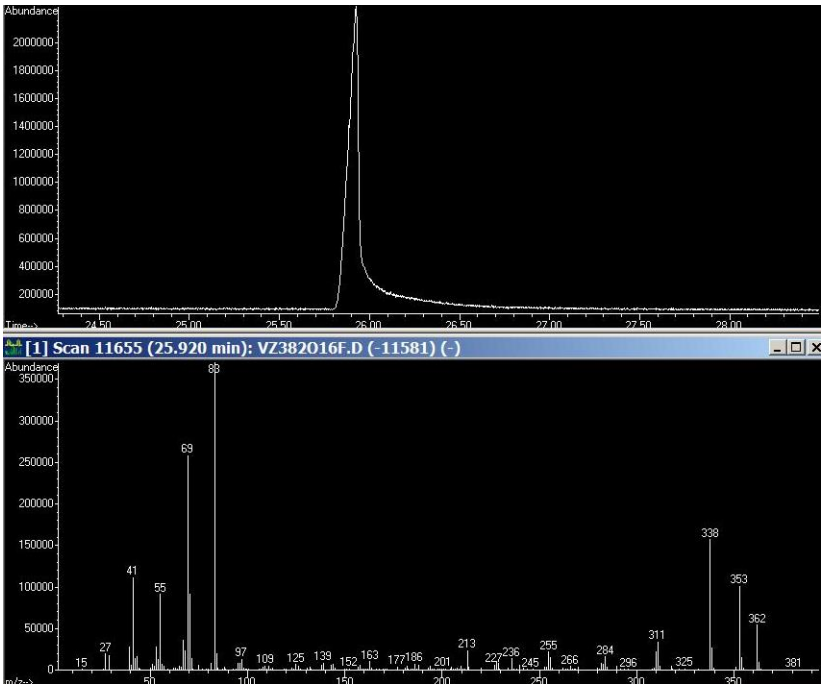
2-(1-(2-methylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3n**),



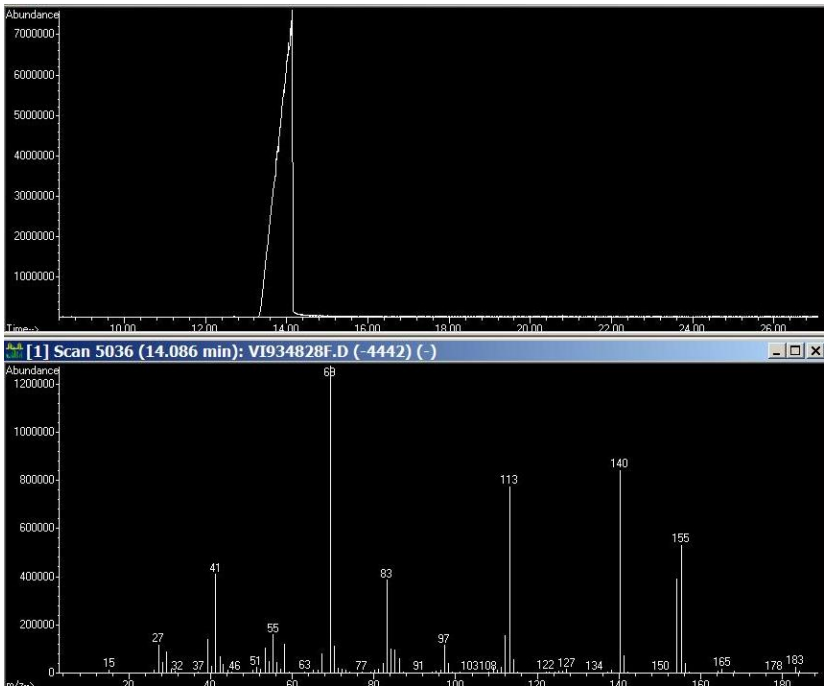
2-(1-(2-trifluoromethylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3o**),



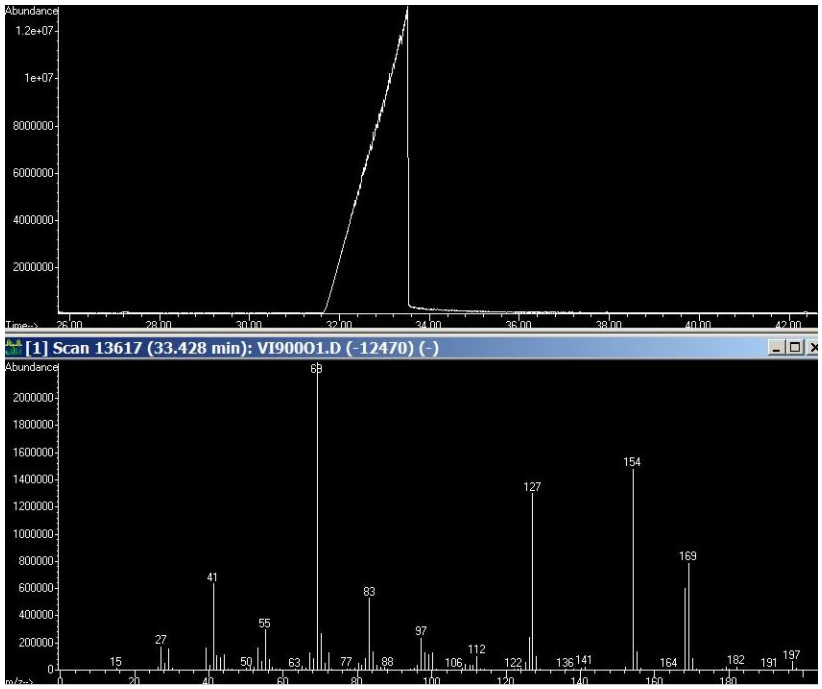
2-(1-(3,5-ditrifluoromethylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3p**),



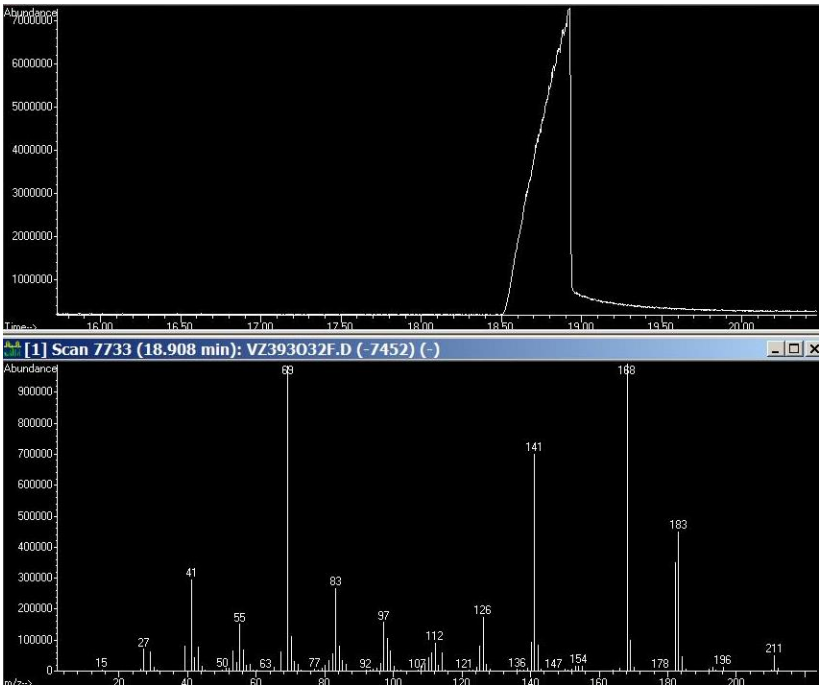
2-methyl-2-(1-methyl-2,5-dioxopyrrolidin-3-yl)propanal (**5a**),



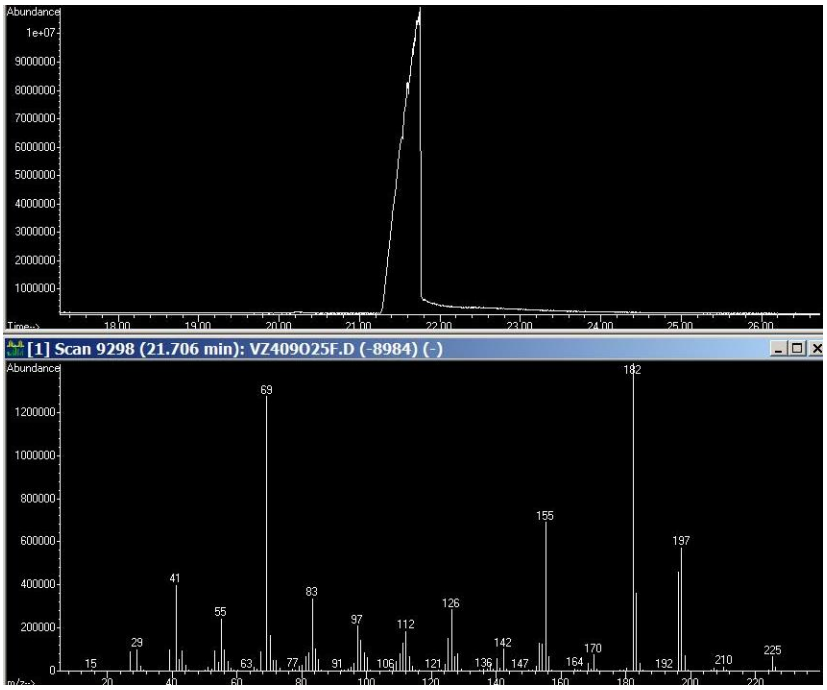
2-(1-ethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5b**),



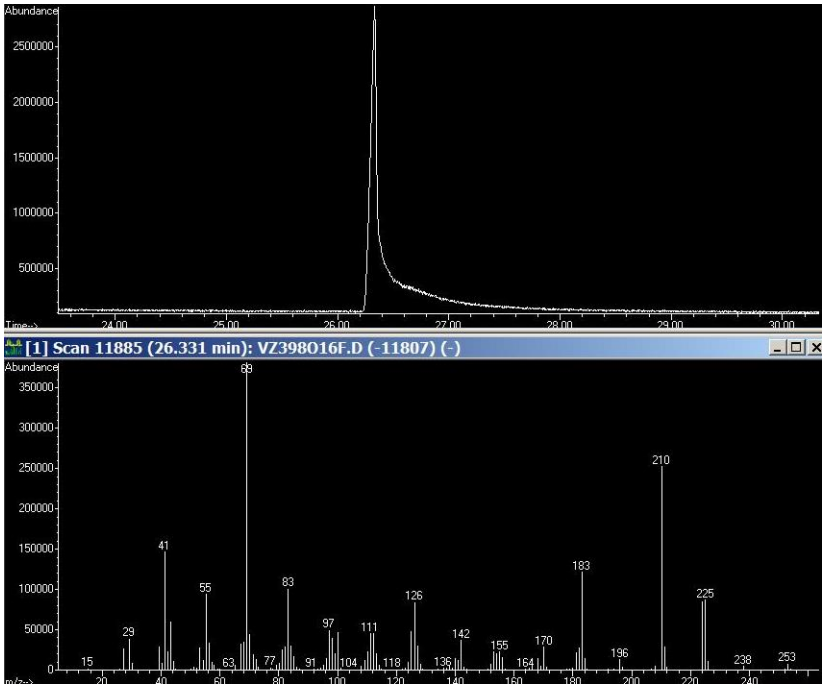
2-(1-propyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5c**),



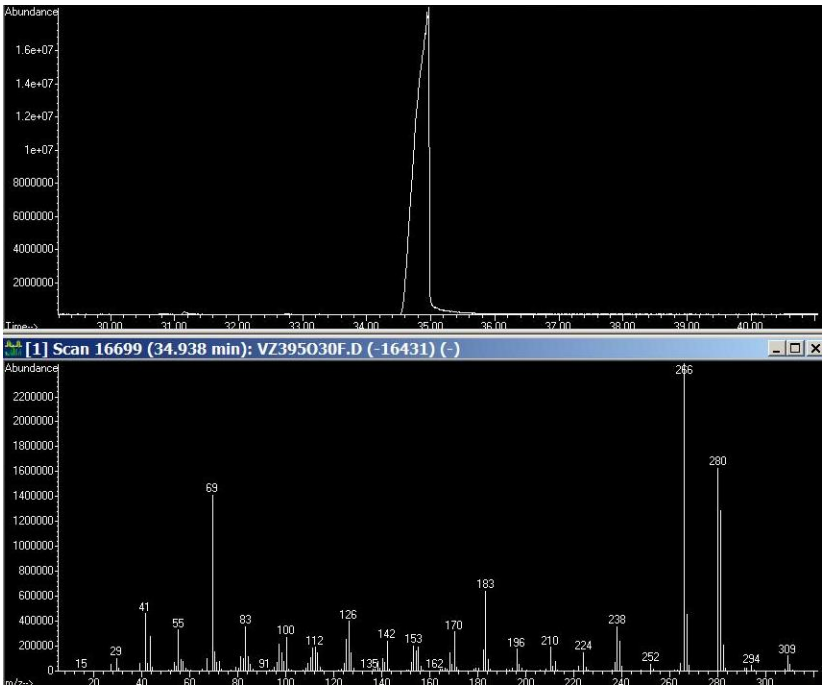
2-(1-butyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5d**),



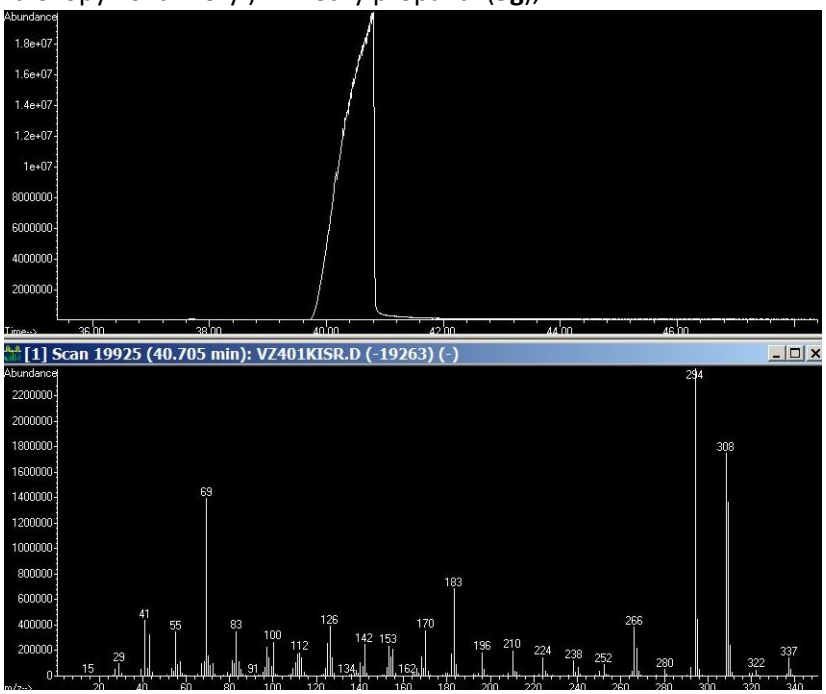
2-(1-hexyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5e**),



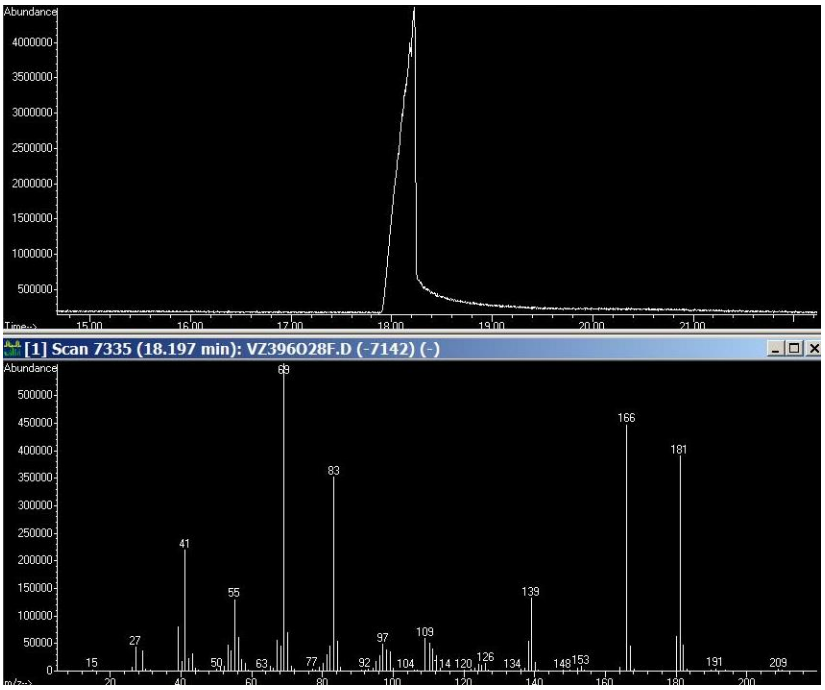
2-(1-decyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5f**),



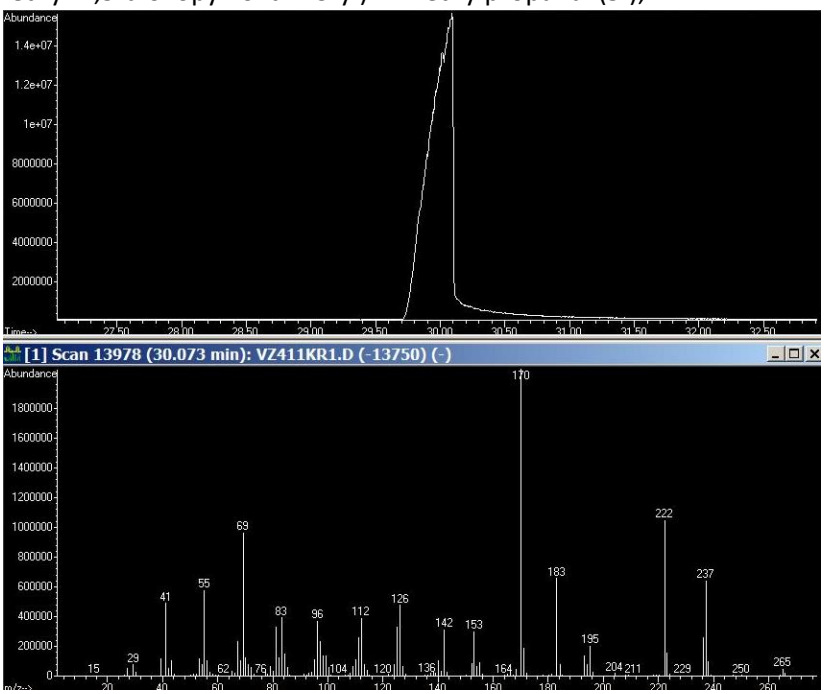
2-(1-dodecyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5g**),



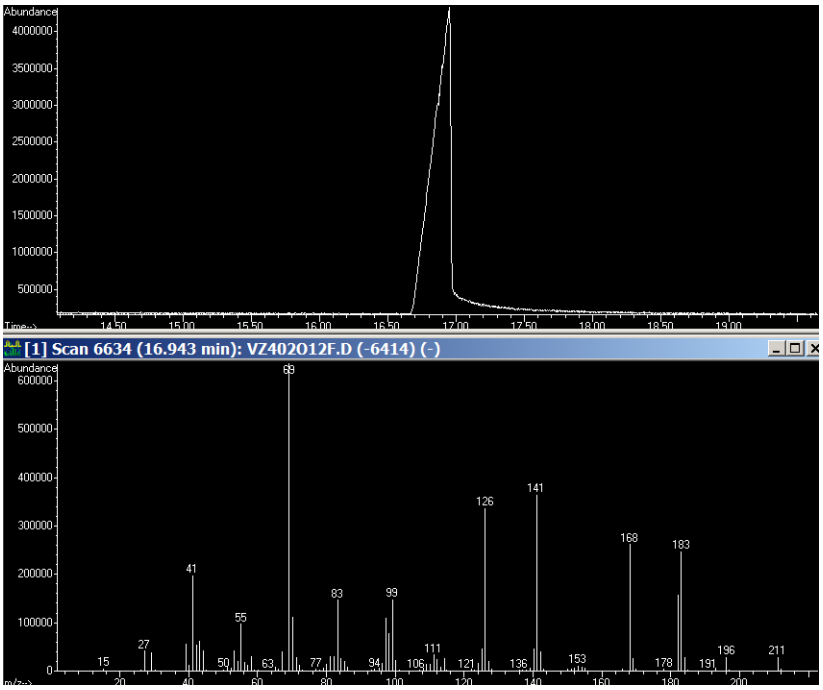
2-(1-allyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5h**),



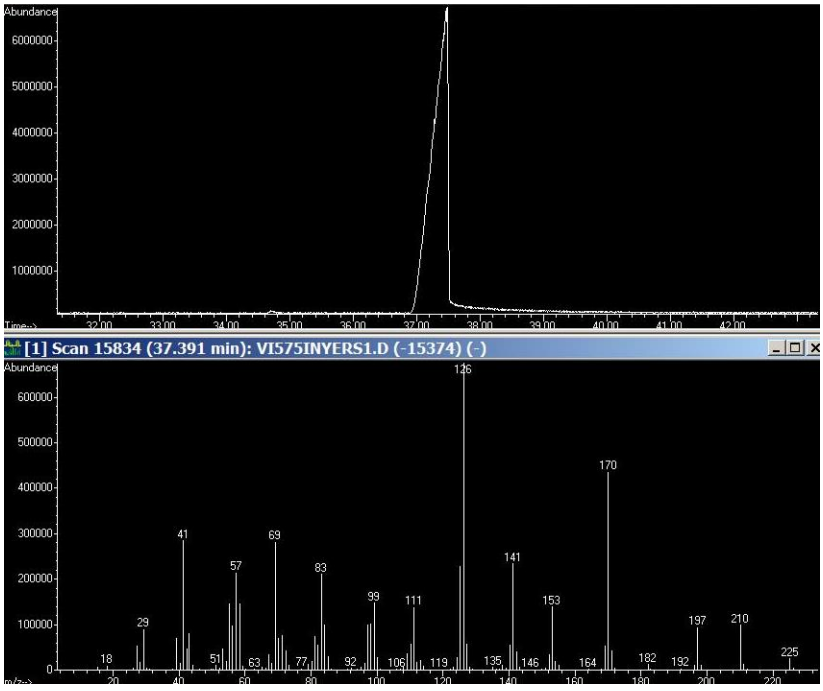
2-(1-cyclohexylmethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5i**),



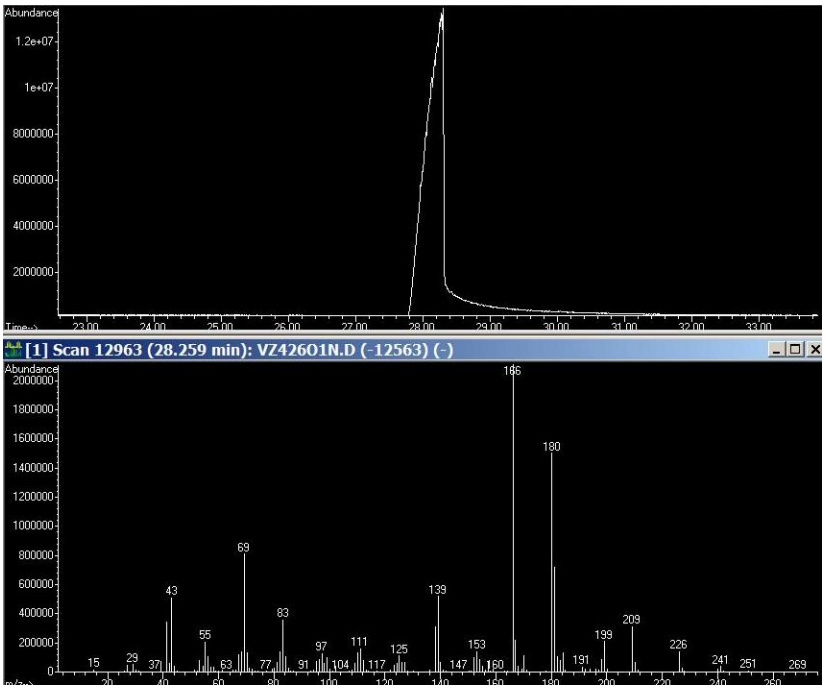
2-(1-isopropyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5j**),



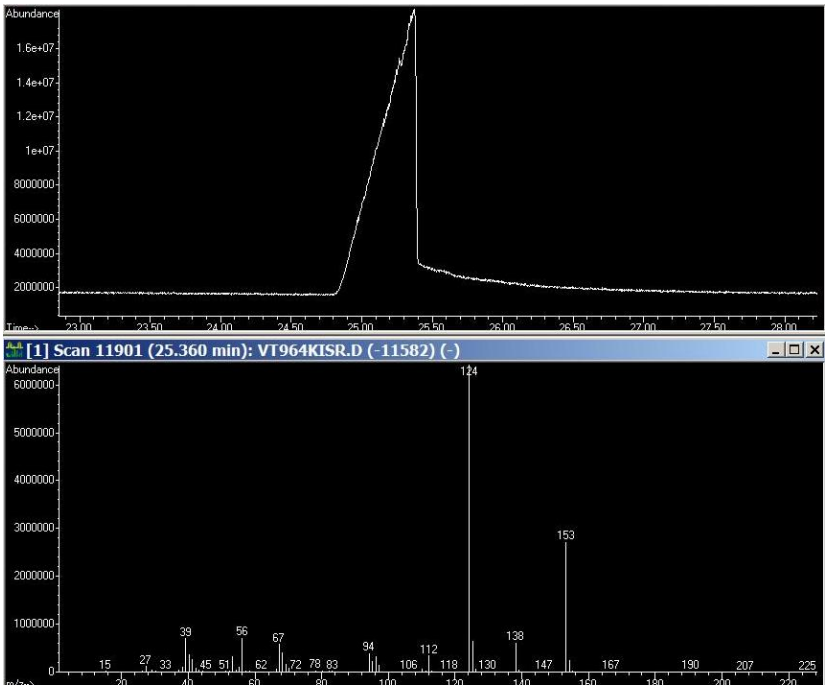
2-(1-*tert*-butyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5k**),



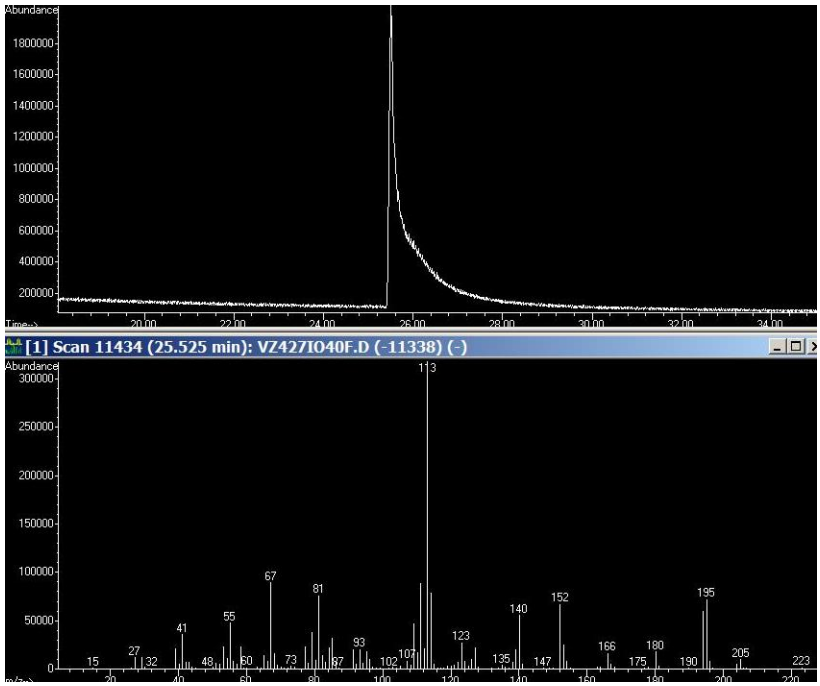
2-(1-(3-acetoxypropyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5l**),



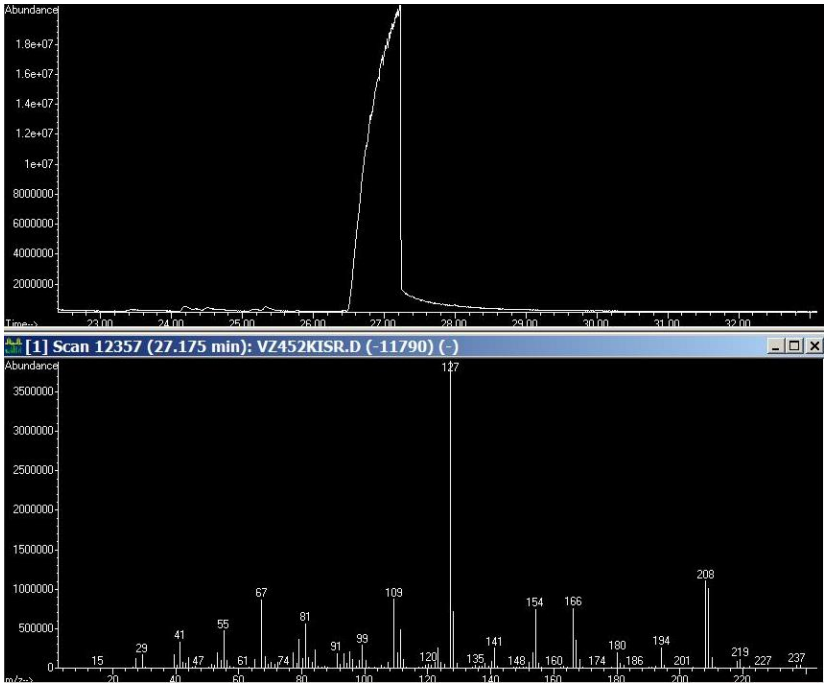
2-methyl-2-((*3R*)-4-methyl-2,5-dioxo-1-propylpyrrolidin-3-yl)propanal (**5m**),



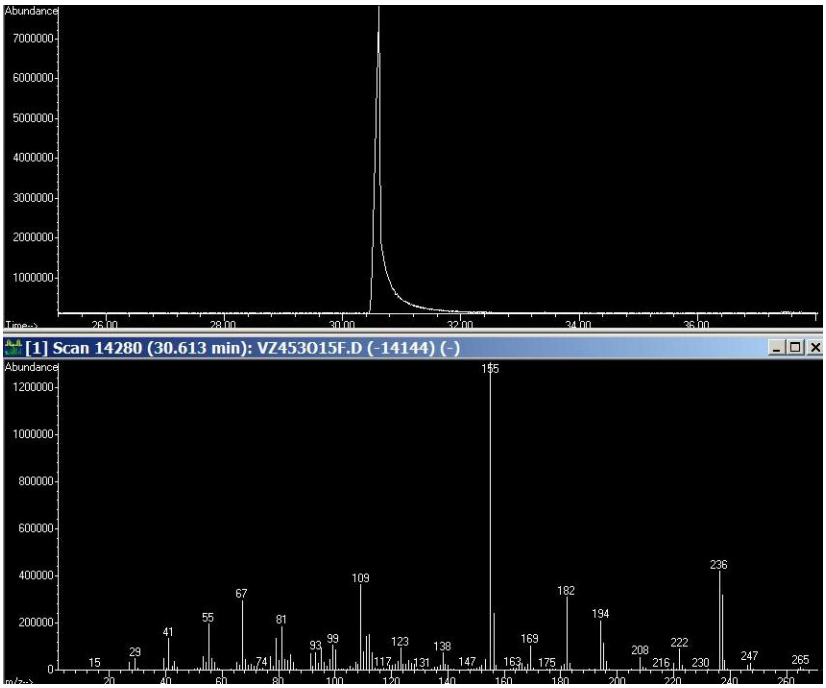
1-(1-methyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6a**),



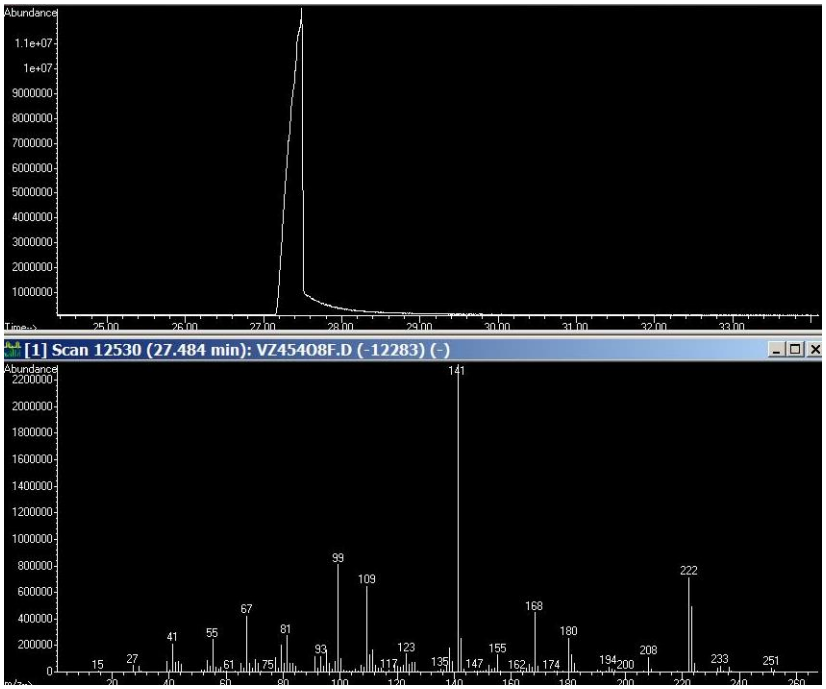
1-(1-ethyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6b**),



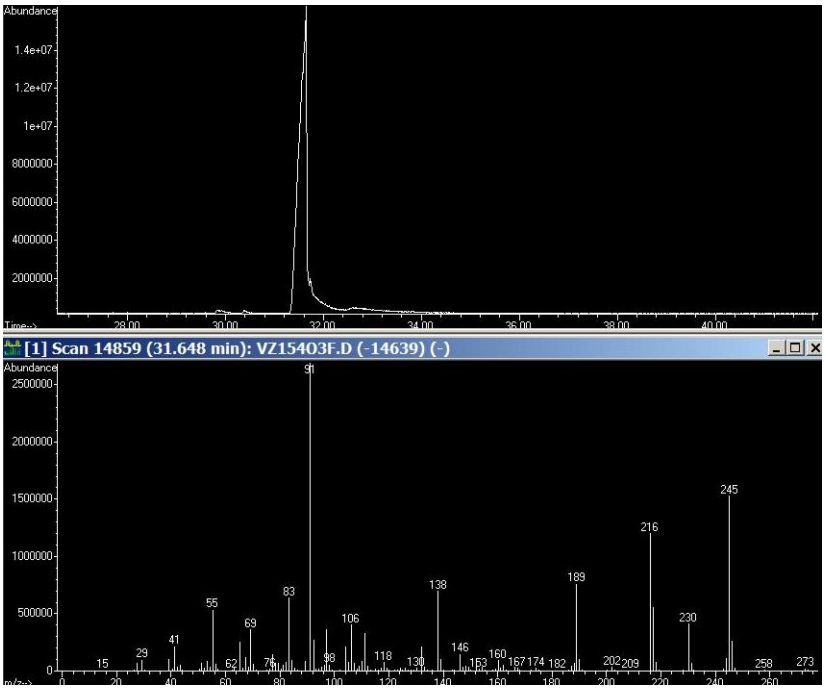
1-(1-butyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6c**),



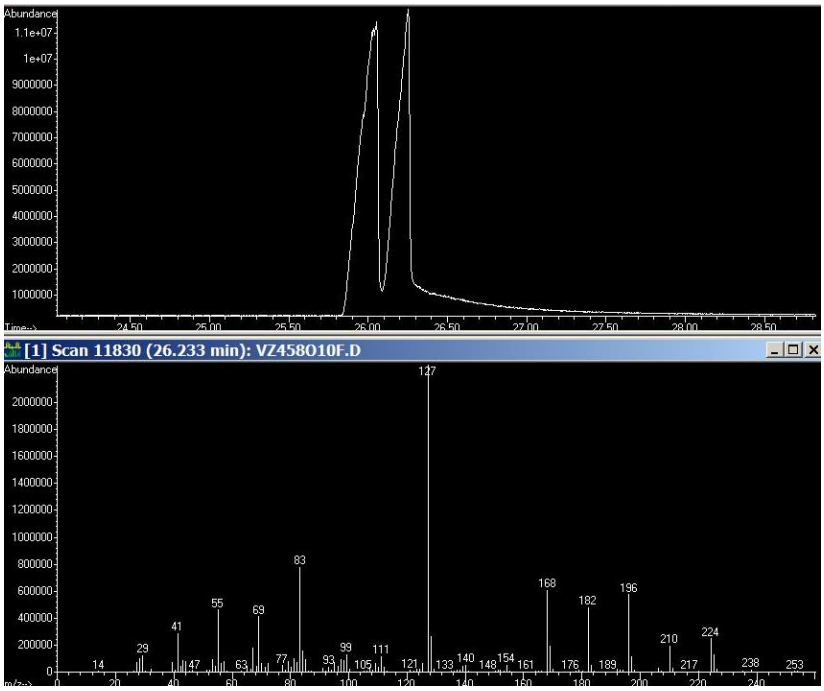
1-(1-isopropyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6d**),



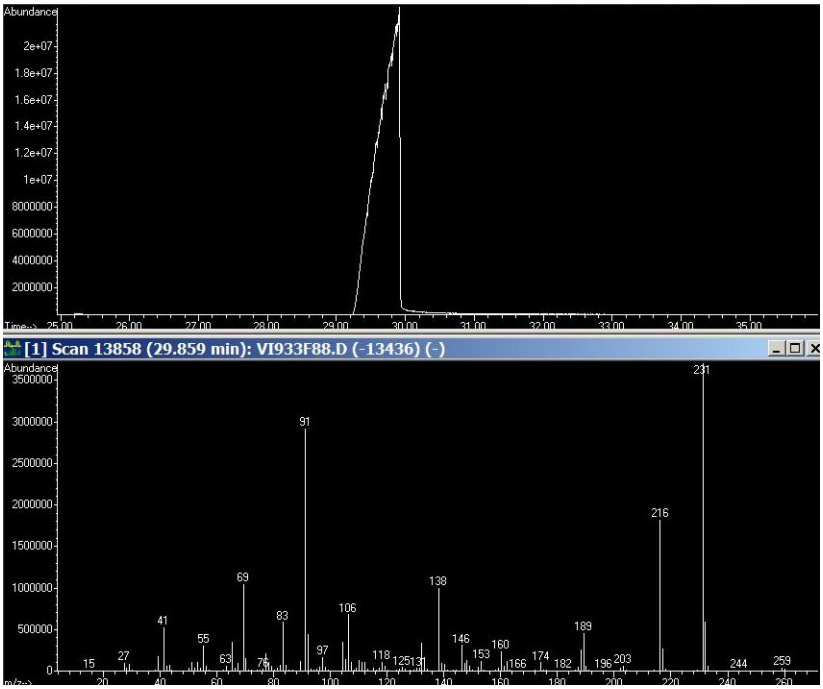
2-(1-benzyl-2,5-dioxopyrrolidin-3-yl)-2-methylbutanal (**6e**),



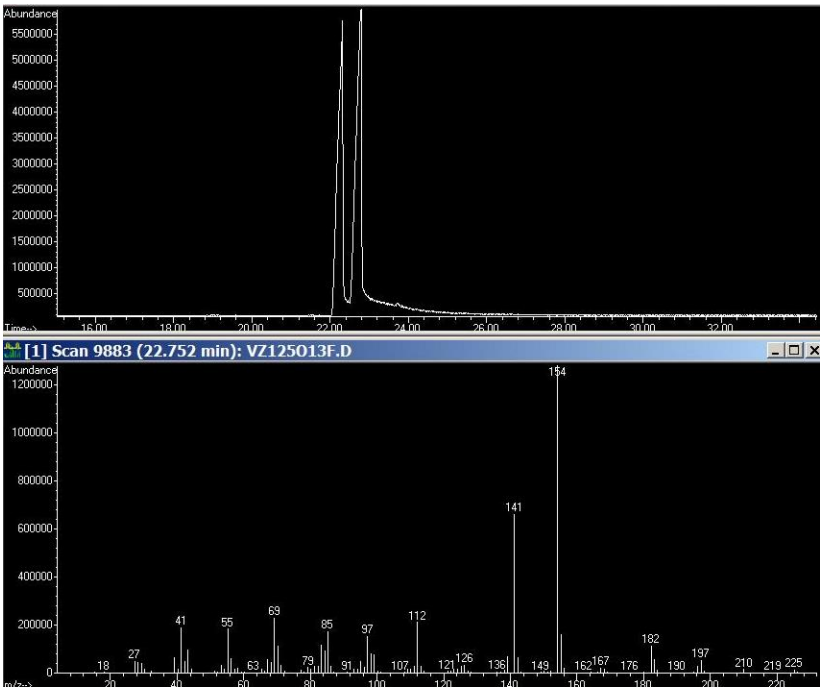
2-ethyl-2-(1-ethyl-2,5-dioxopyrrolidin-3-yl)hexanal (**6f**),



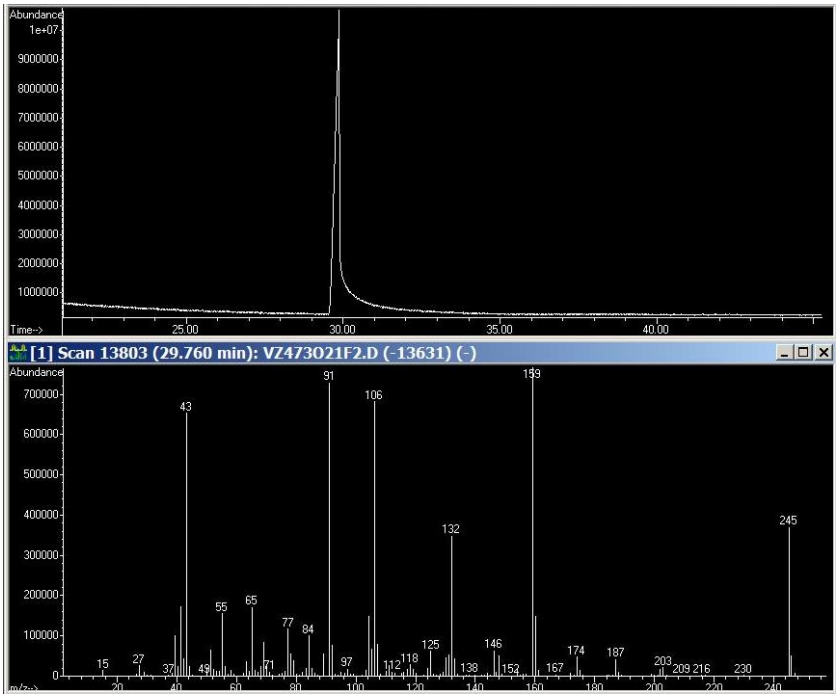
2-(1-benzyl-2,5-dioxopyrrolidin-3-yl)propanal (**6g**),



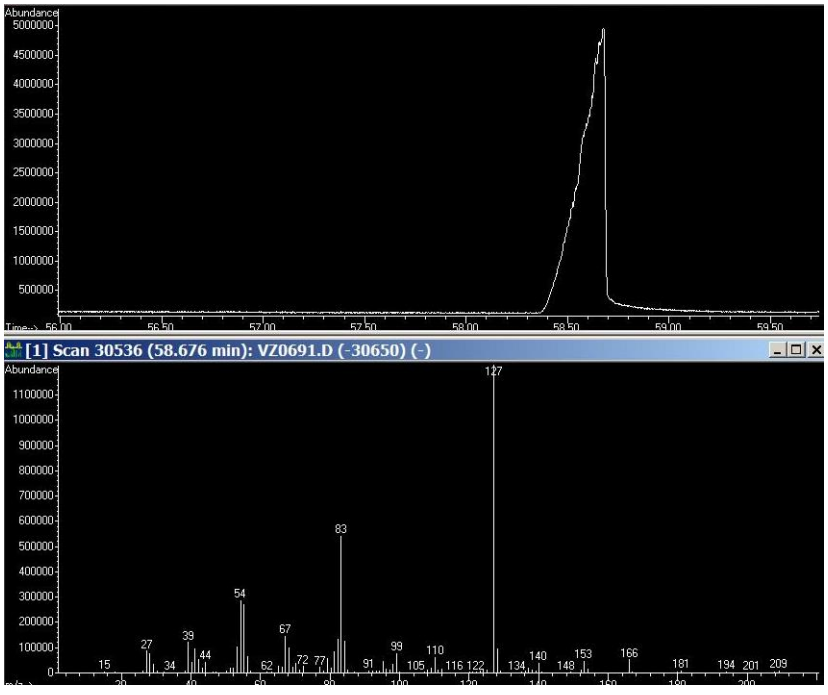
2-(1-propyl-2,5-dioxopyrrolidin-3-yl)-3-methylbutanal (**6h**),



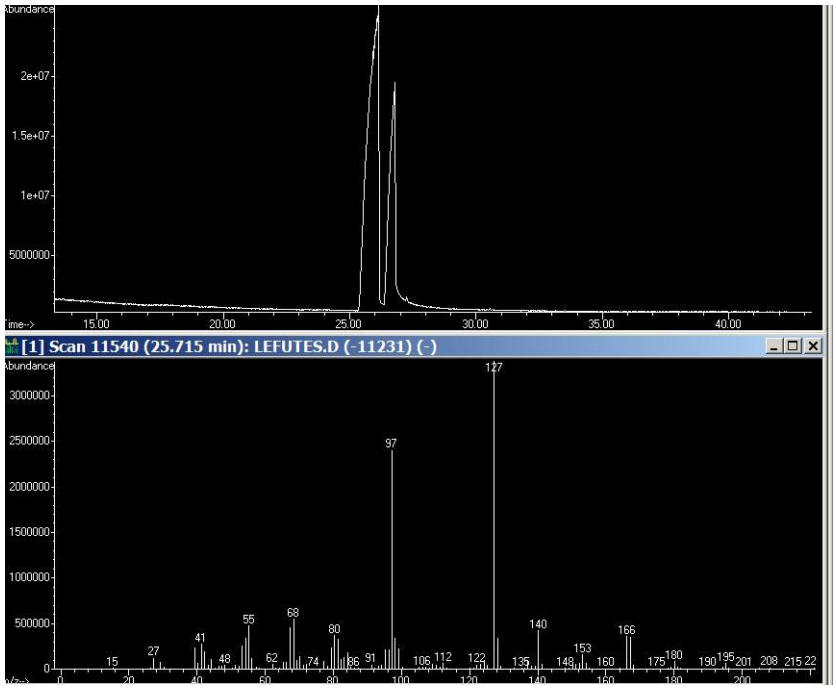
1-benzyl-3-(2-oxopropyl)pyrrolidine-2,5-dione (**6i**),



1-ethyl-3-(2-oxocyclopentyl)pyrrolidine-2,5-dione (**6j**),

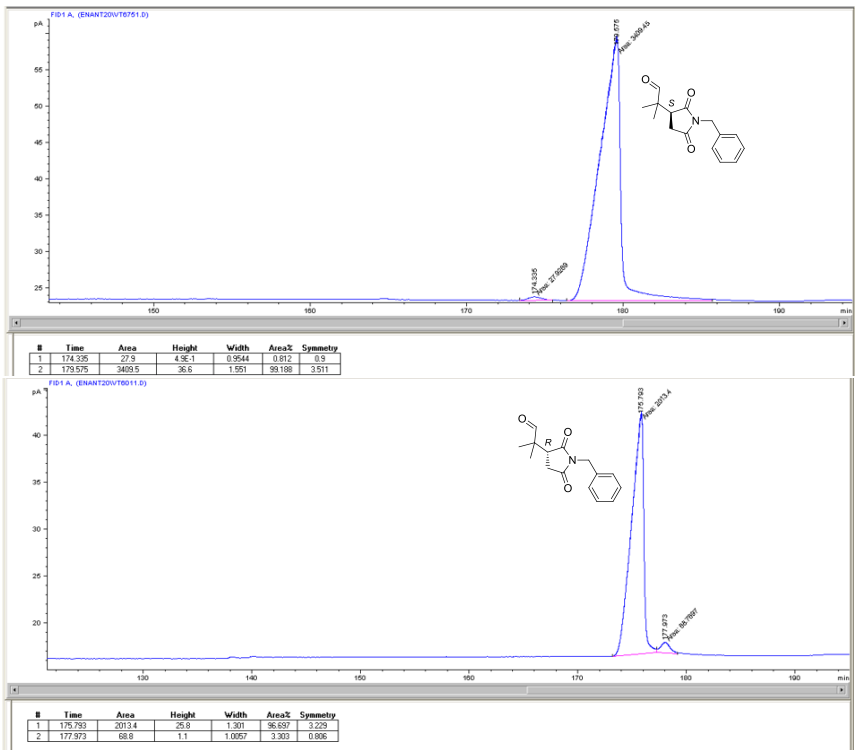


1-ethyl-3-(2-oxocyclohexyl)pyrrolidine-2,5-dione (**6k**),



15.3. Separation of the Michael adduct enantiomers by GC-FID

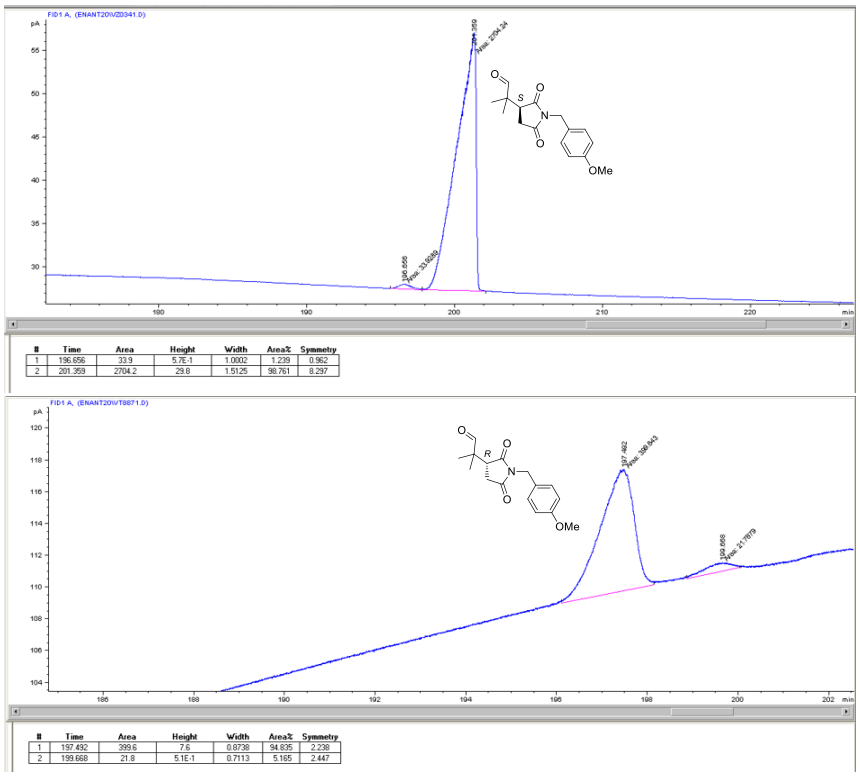
2-(1-benzyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3a**),



#	Time	Area	Height	Width	Area%	Symmetry
1	174.335	27.9	4.9E-1	0.9544	0.812	0.9
2	179.575	3409.5	36.6	1.551	99.188	3.511

#	Time	Area	Height	Width	Area%	Symmetry
1	175.793	2013.4	25.8	1.301	96.697	3.229
2	177.973	68.8	1.1	1.0057	3.303	0.806

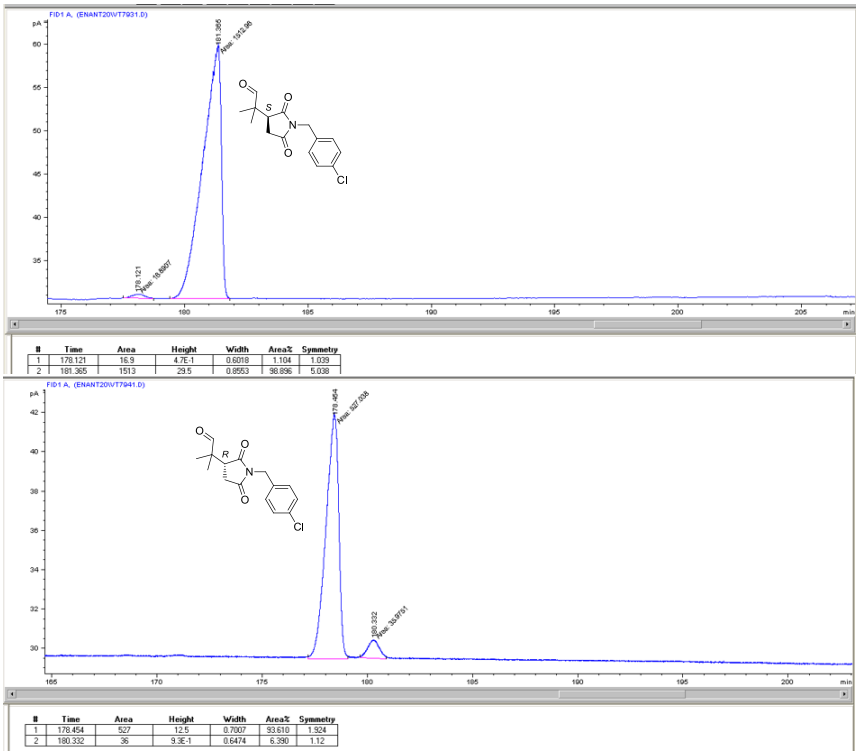
2-(1-(4-methoxybenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3b**),



#	Time	Area	Height	Width	Area%	Symmetry
1	196.656	33.9	5.7E-1	1.0002	1.239	0.962
2	201.359	2704.2	29.8	1.5125	98.761	8.297

#	Time	Area	Height	Width	Area%	Symmetry
1	197.492	399.6	7.6	0.8738	94.835	2.238
2	199.668	21.8	5.1E-1	0.7113	5.165	2.447

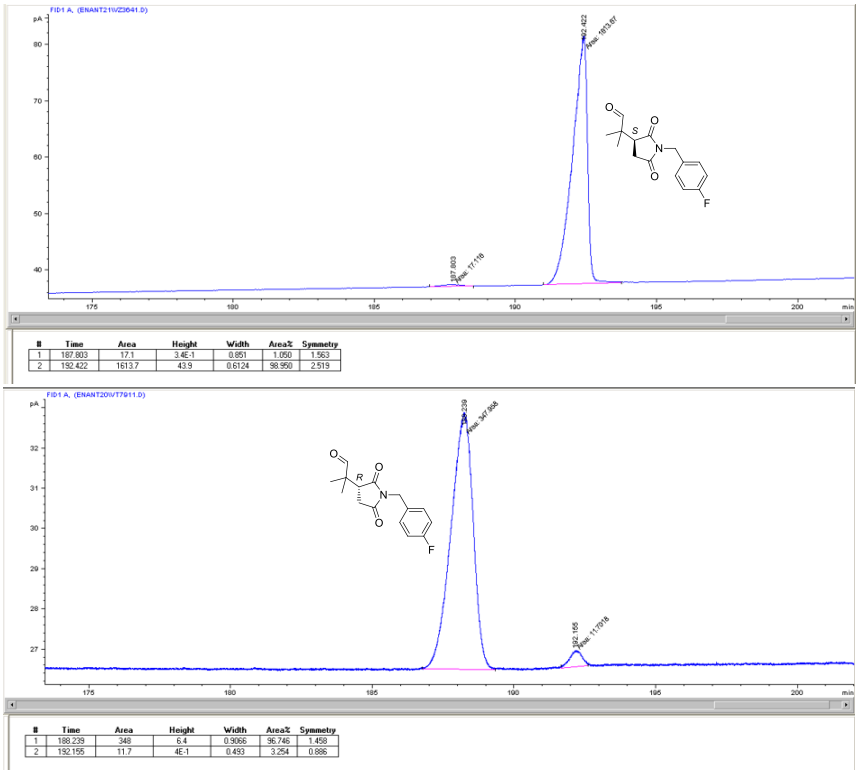
2-(1-(4-chlorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3c**),



#	Time	Area	Height	Width	Area%	Symmetry
1	178.121	16.9	4.7E-1	0.6018	1.104	1.039
2	181.365	1513	29.5	0.8553	98.896	5.038

#	Time	Area	Height	Width	Area%	Symmetry
1	178.454	527	12.5	0.7007	93.610	1.924
2	180.332	36	9.3E-1	0.6474	6.390	1.12

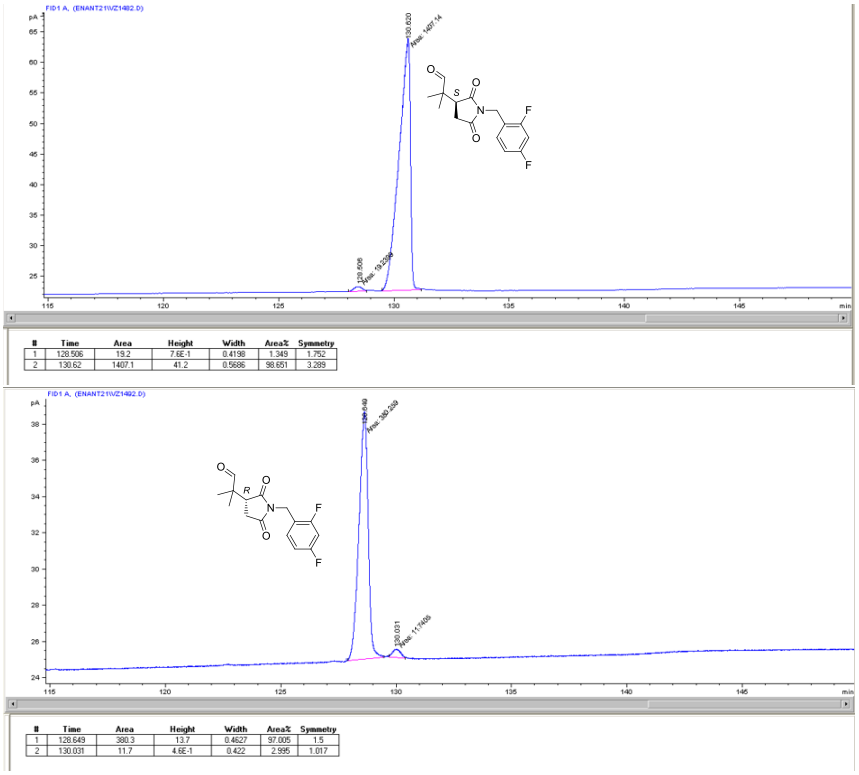
2-(1-(4-fluorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3d**),



#	Time	Area	Height	Width	Area%	Symmetry
1	187.803	17.1	3.4E-1	0.851	1.050	1.563
2	192.422	1613.7	43.9	0.6124	98.950	2.519

#	Time	Area	Height	Width	Area%	Symmetry
1	188.239	348	6.4	0.9068	96.746	1.459
2	192.155	11.7	4E-1	0.493	3.254	0.886

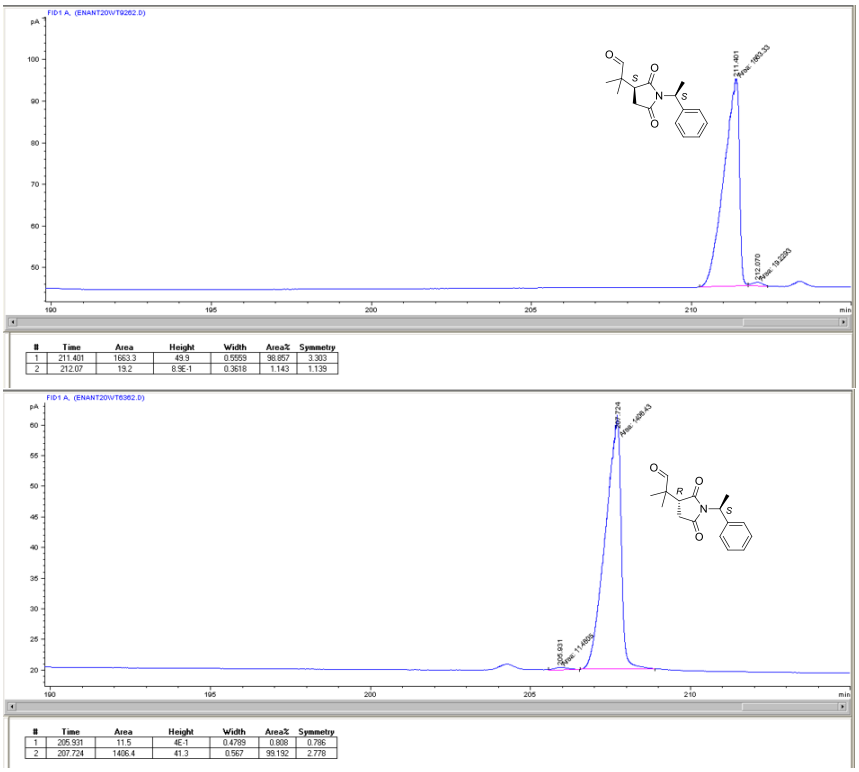
2-(1-(2,4-difluorobenzyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3e**),



#	Time	Area	Height	Width	Area%	Symmetry
1	128.506	19.2	7.6E-1	0.4198	1.349	1.752
2	130.62	1407.1	41.2	0.5686	98.651	3.289

#	Time	Area	Height	Width	Area%	Symmetry
1	128.649	380.3	13.7	0.4627	97.005	1.5
2	130.031	11.7	4.6E-1	0.422	2.995	1.017

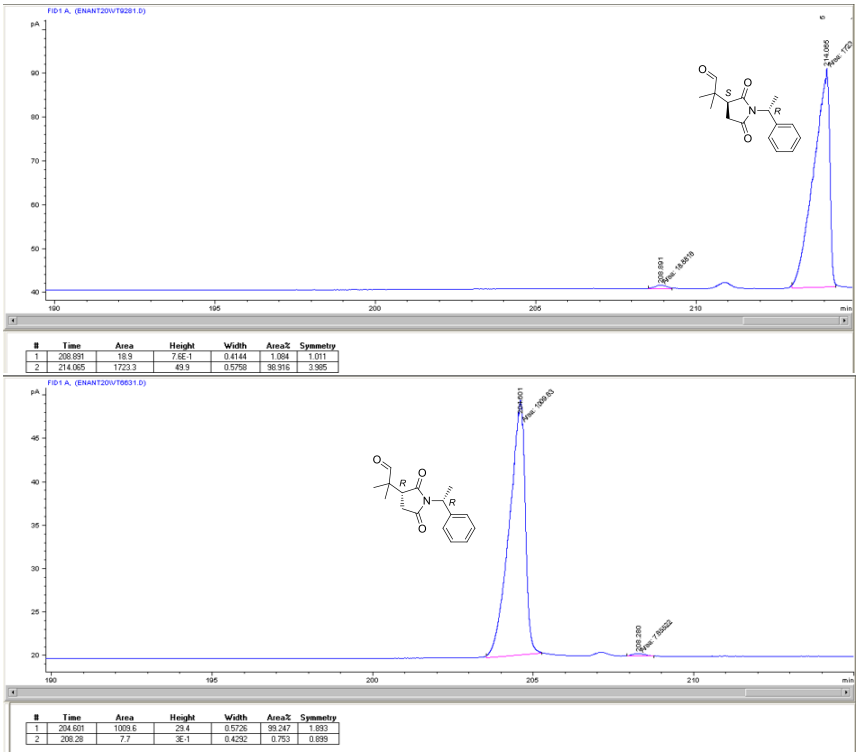
2-((*S*)-1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3f**),



#	Time	Area	Height	Width	Area%	Symmetry
1	211.401	163.3	49.9	0.5559	98.857	3.303
2	212.07	19.2	8.9E-1	0.3618	1.143	1.139

#	Time	Area	Height	Width	Area%	Symmetry
1	205.931	11.5	4E-1	0.4789	0.808	0.786
2	207.724	1406.4	41.3	0.567	99.192	2.778

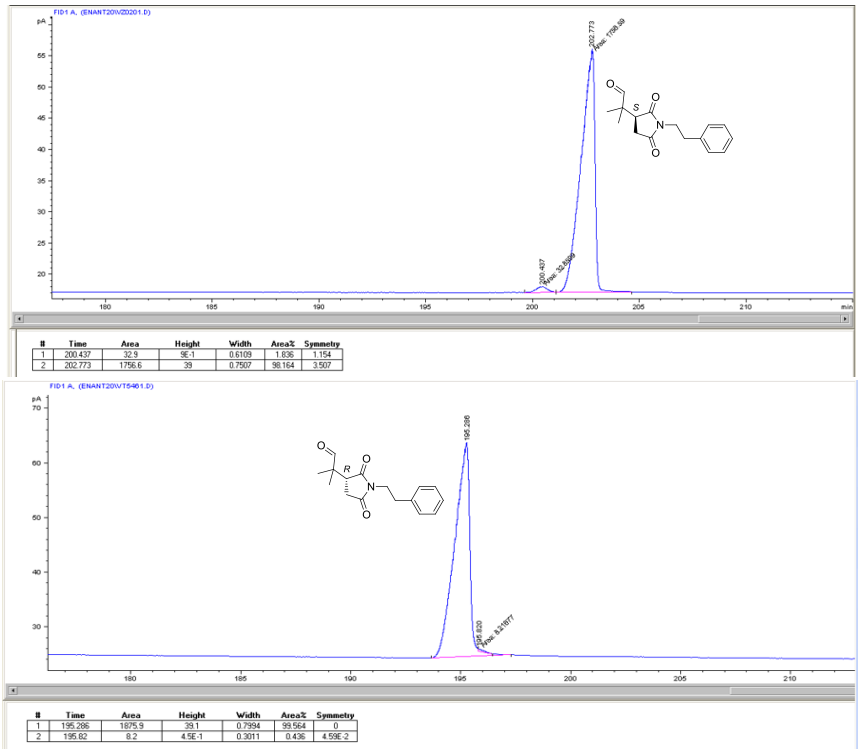
2-((*R*)-1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3f**),



#	Time	Area	Height	Width	Area%	Symmetry
1	208.891	18.9	7.6E-1	0.4144	1.084	1.011
2	214.065	1723.3	49.9	0.5758	98.916	3.985

#	Time	Area	Height	Width	Area%	Symmetry
1	204.601	1009.6	29.4	0.5726	99.247	1.893
2	208.28	7.7	3E-1	0.4292	0.753	0.899

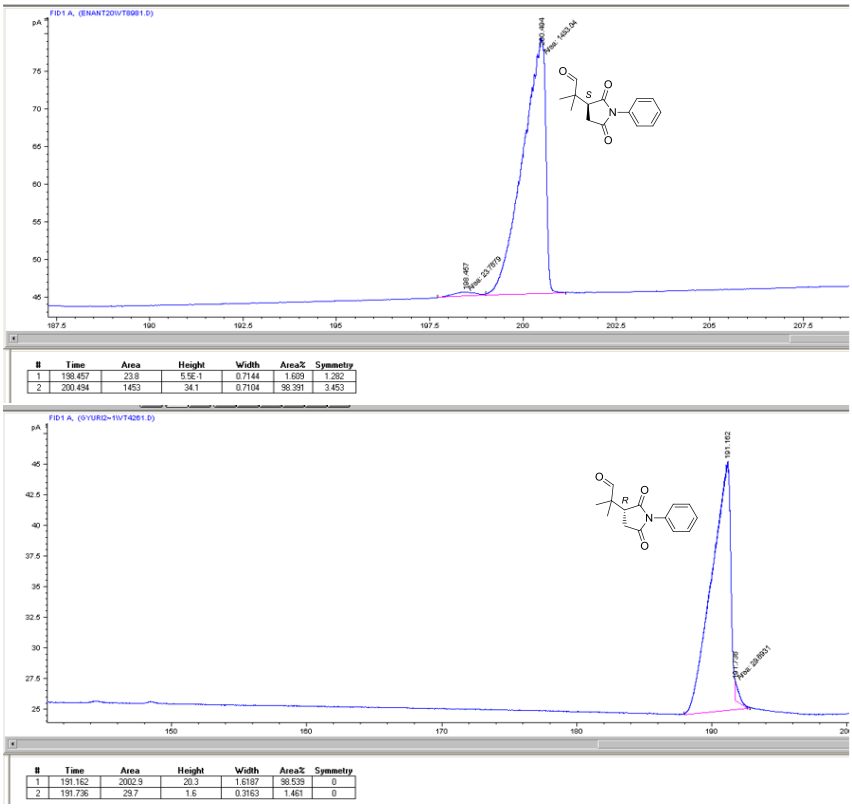
2-(1-phenylethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3g**),



#	Time	Area	Height	Width	Area%	Symmetry
1	200.437	32.9	9E-1	0.6109	1.836	1.154
2	202.773	1756.6	39	0.7507	98.164	3.507

#	Time	Area	Height	Width	Area%	Symmetry
1	195.286	1875.9	39.1	0.7994	99.564	0
2	195.82	8.2	4.5E-1	0.3011	0.436	4.59E-2

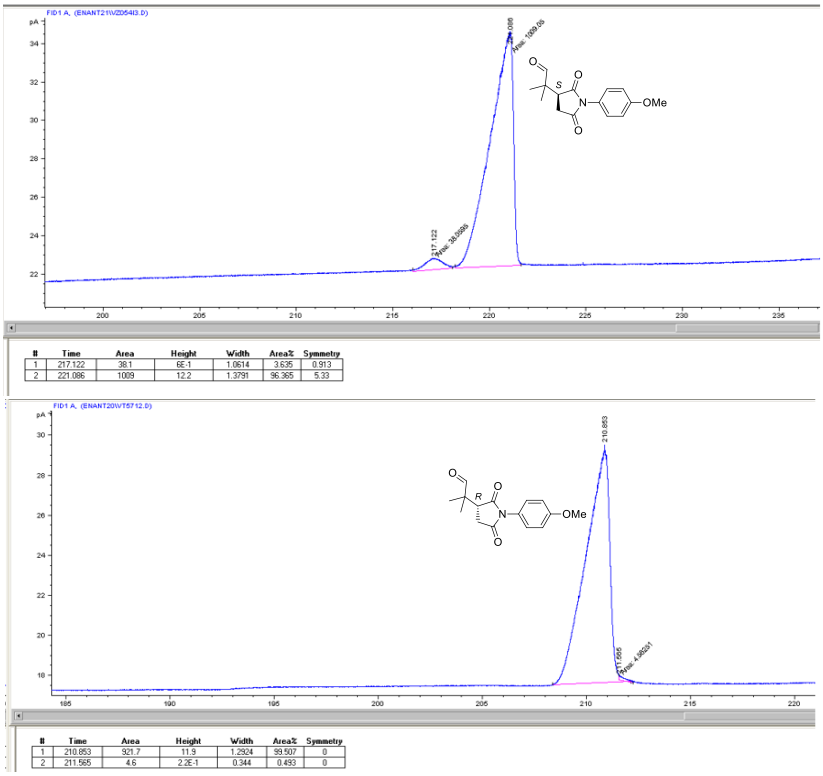
2-(1-phenyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3h**),



#	Time	Area	Height	Width	Area%	Symmetry
1	198.457	23.8	5.5E-1	0.7144	1.689	1.282
2	200.494	1453	34.1	0.7104	98.391	3.453

#	Time	Area	Height	Width	Area%	Symmetry
1	191.162	2002.9	20.3	1.6187	98.539	0
2	191.736	29.7	1.6	0.3163	1.461	0

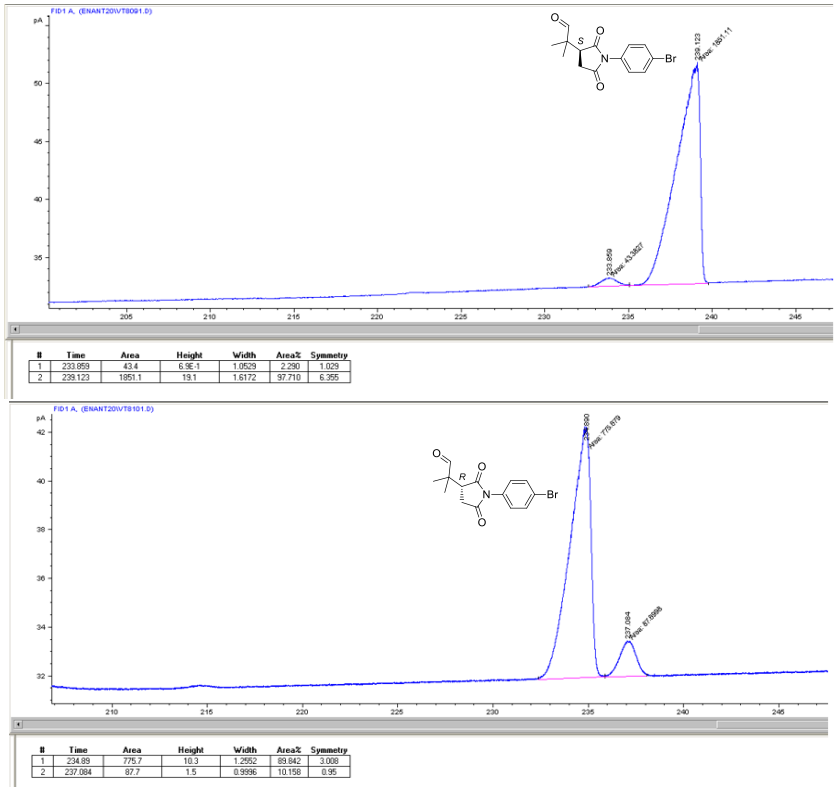
2-(1-(4-methoxyphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3i**),



#	Time	Area	Height	Width	Area%	Symmetry
1	217.122	38.1	6E-1	1.0614	3.635	0.913
2	221.086	1009	12.2	1.3791	96.365	5.33

#	Time	Area	Height	Width	Area%	Symmetry
1	210.853	921.7	11.9	1.2924	99.507	0
2	211.565	4.6	2.2E-1	0.344	0.493	0

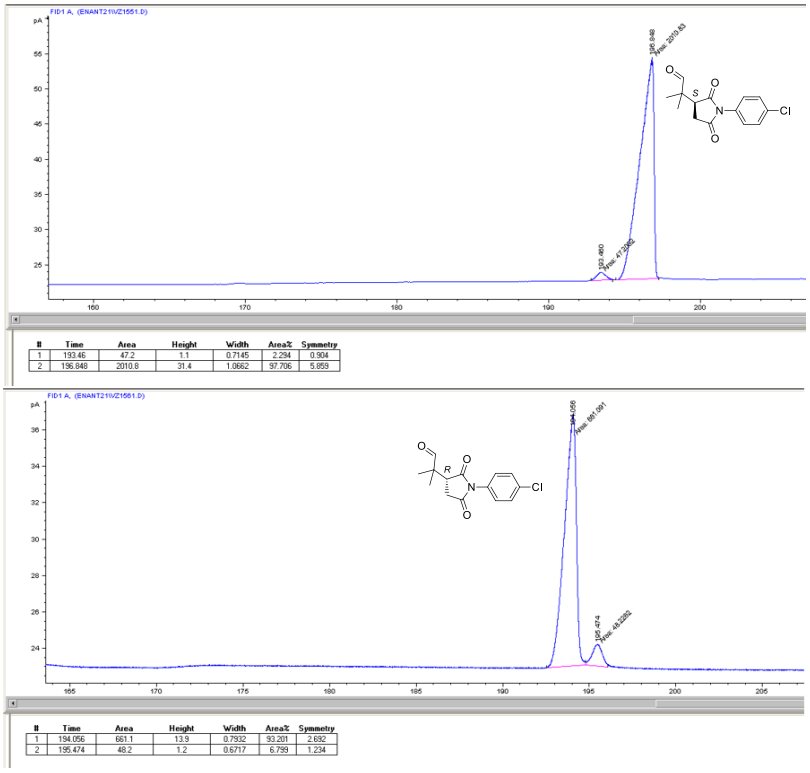
2-(1-(4-bromophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3j**),



#	Time	Area	Height	Width	Area%	Symmetry
1	233.859	43.4	6.9E-1	1.0529	2.290	1.029
2	239.123	1851.1	19.1	1.6172	97.710	6.355

#	Time	Area	Height	Width	Area%	Symmetry
1	234.89	775.7	10.3	1.2552	89.842	3.008
2	237.084	87.7	1.5	0.9996	10.158	0.95

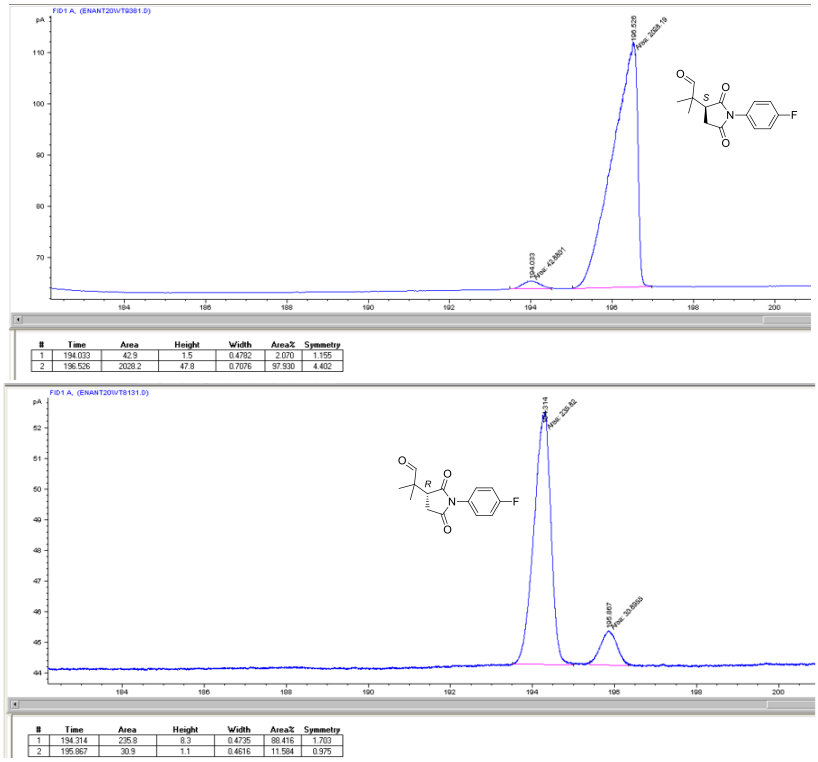
2-(1-(4-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3k**),



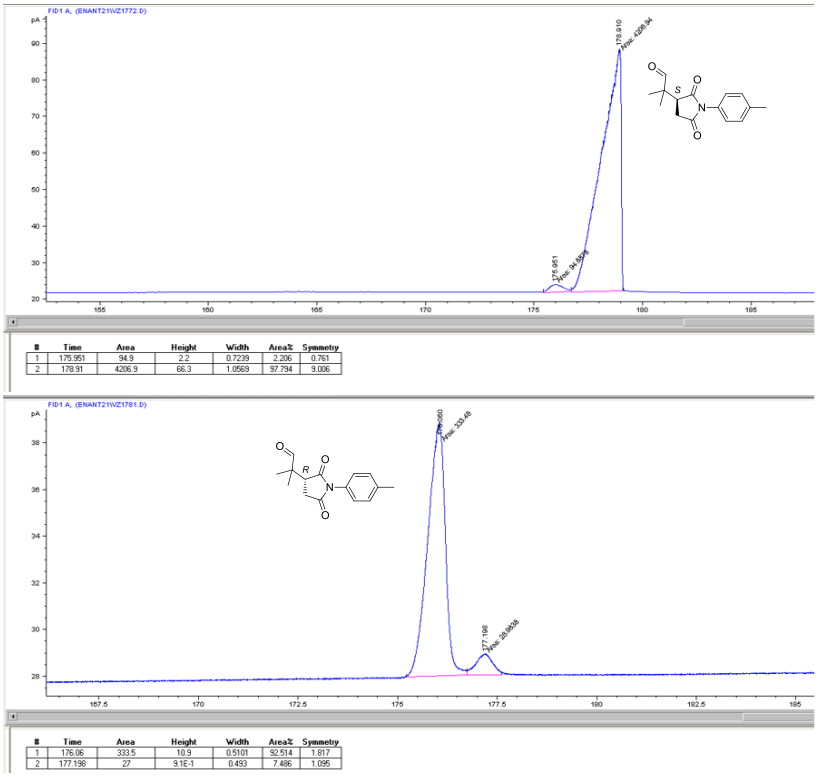
#	Time	Area	Height	Width	Area%	Symmetry
1	193.46	47.2	1.1	0.7145	2.294	0.904
2	196.848	2010.8	31.4	1.0662	97.706	5.859

#	Time	Area	Height	Width	Area%	Symmetry
1	194.056	661.1	13.9	0.7932	93.201	2.682
2	195.474	48.2	1.2	0.6717	6.799	1.234

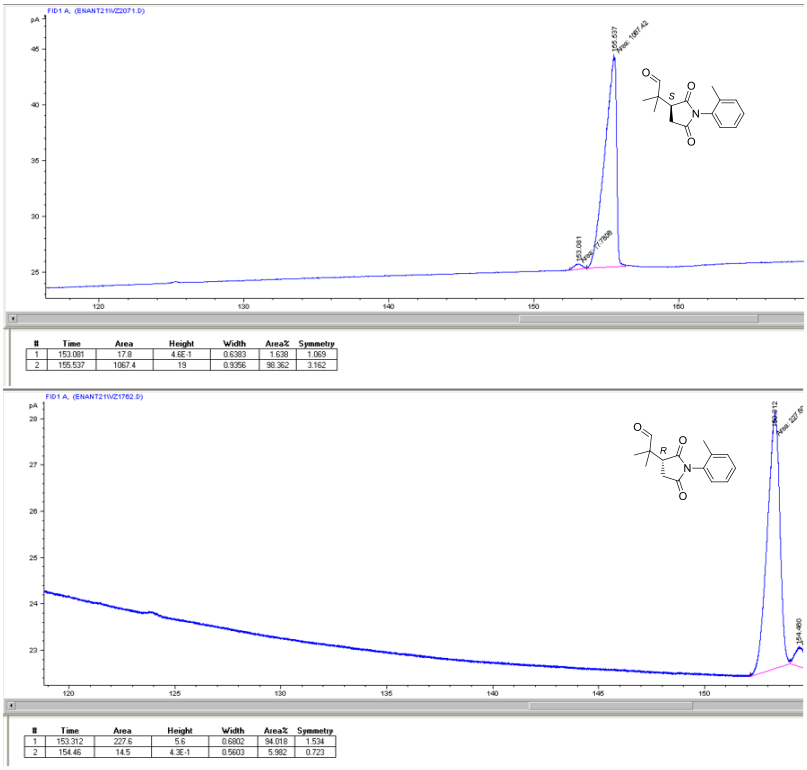
2-(1-(4-fluorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3l**),



2-(1-(4-methylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3m**),



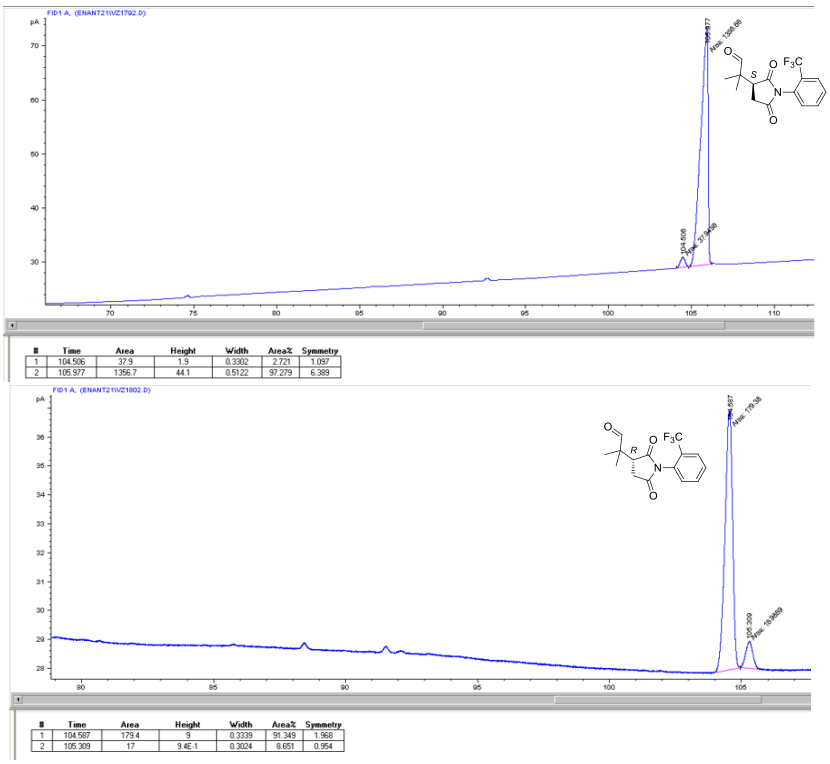
2-(1-(2-methylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3n**),



#	Time	Area	Height	Width	Area%	Symmetry
1	153.081	17.8	4.6E-1	0.6383	1.638	1.069
2	155.537	1067.4	19	0.9356	98.362	3.162

#	Time	Area	Height	Width	Area%	Symmetry
1	153.312	227.6	5.6	0.6802	94.018	1.534
2	154.46	14.5	4.3E-1	0.5603	5.982	0.723

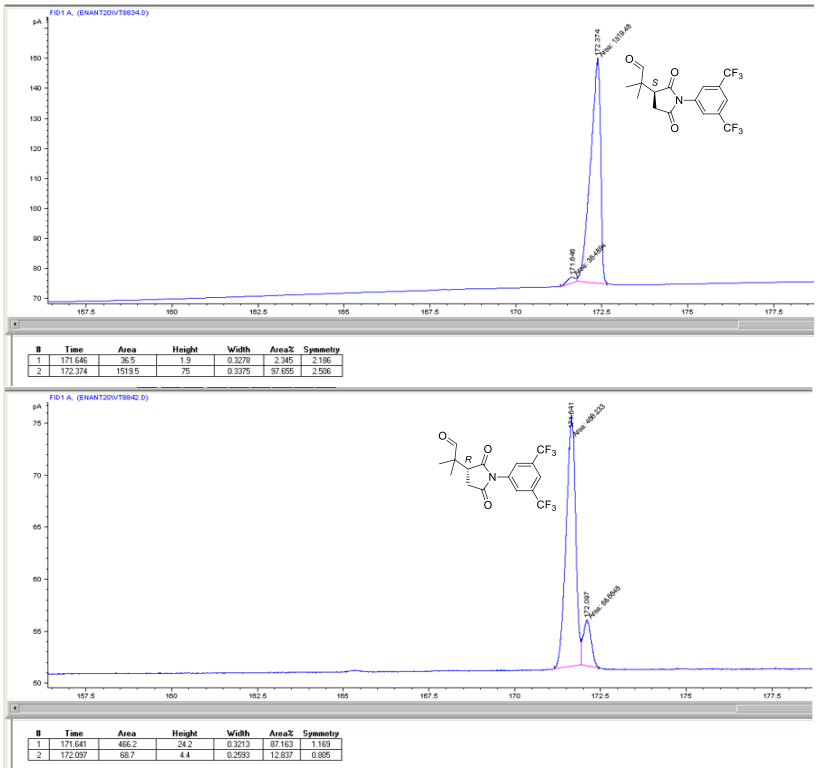
2-(1-(2-trifluoromethylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3o**),



#	Time	Area	Height	Width	Area%	Symmetry
1	104.506	37.9	1.9	0.3302	2.721	1.097
2	105.977	1356.7	44.1	0.5122	97.279	6.389

#	Time	Area	Height	Width	Area%	Symmetry
1	104.587	179.4	9	0.3339	91.349	1.968
2	105.309	17	9.4E-1	0.3024	8.651	0.954

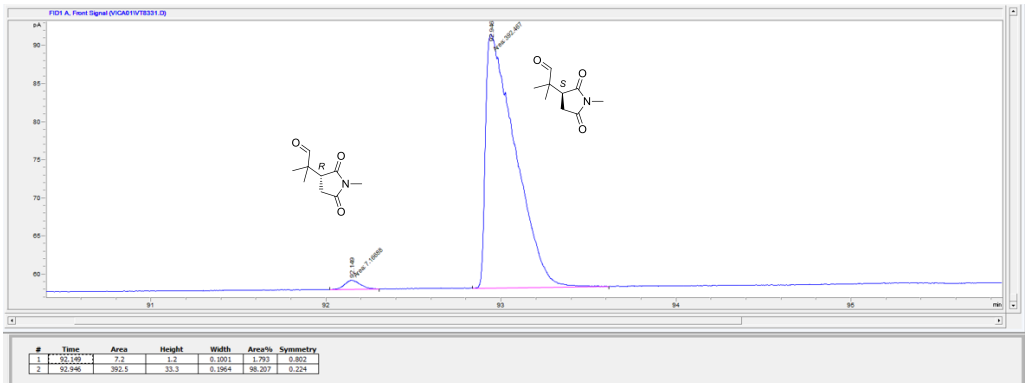
2-(1-(3,5-ditrifluoromethylphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**3p**),



#	Time	Area	Height	Width	Area%	Symmetry
1	171.646	36.5	1.9	0.3278	2.345	2.186
2	172.374	1519.5	75	0.3375	97.655	2.506

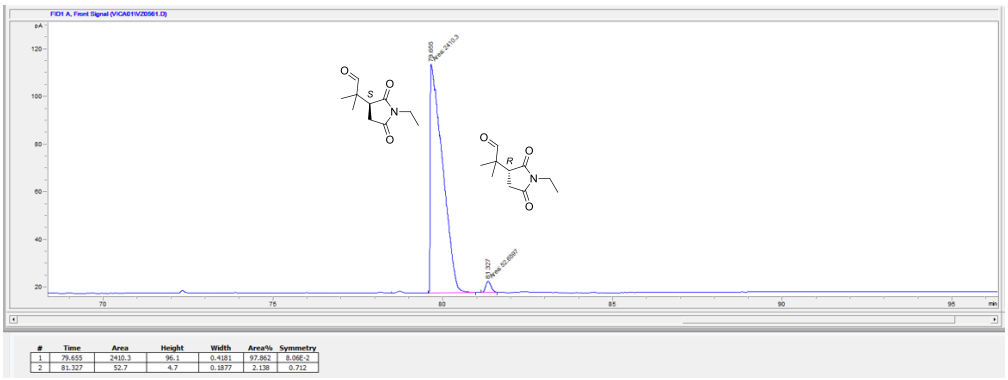
#	Time	Area	Height	Width	Area%	Symmetry
1	171.641	466.2	24.2	0.3213	87.163	1.169
2	172.097	68.7	4.4	0.2593	12.837	0.685

2-methyl-2-(1-methyl-2,5-dioxopyrrolidin-3-yl)propanal (**5a**),



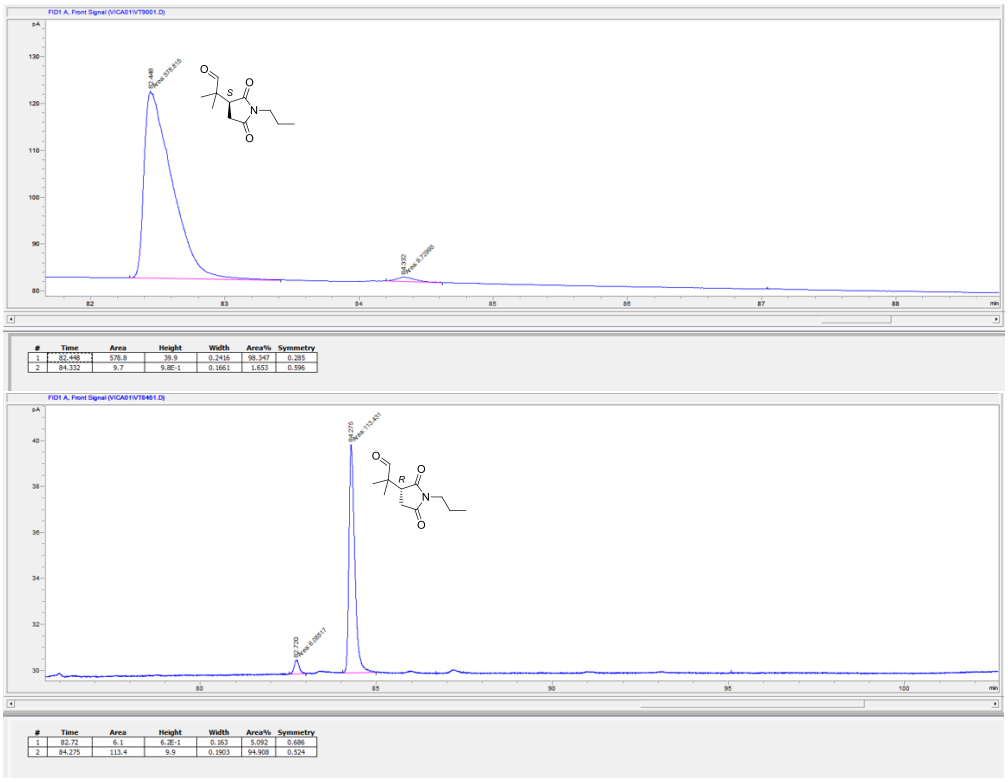
#	Time	Area	Height	Width	Area%	Symmetry
1	92.149	7.2	1.2	0.1001	1.793	0.802
2	92.946	392.5	33.3	0.1964	98.207	0.224

2-methyl-2-(1-ethyl-2,5-dioxopyrrolidin-3-yl)propanal (**5b**),



#	Time	Area	Height	Width	Area%	Symmetry
1	79.655	2410.3	96.1	0.4181	97.862	8.09E-2
2	81.327	52.7	4.7	0.1877	2.138	0.712

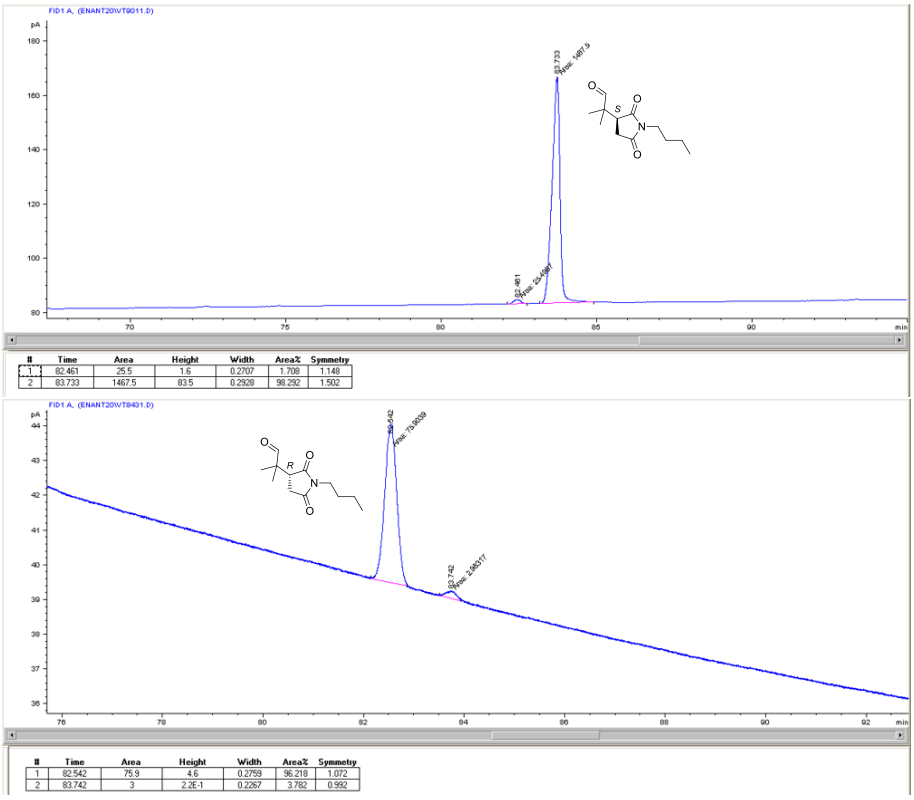
2-methyl-2-(1-propyl-2,5-dioxopyrrolidin-3-yl)propanal (**5c**),



#	Time	Area	Height	Width	Area%	Symmetry
1	82.448	578.8	39.9	0.2416	98.347	0.285
2	84.332	9.7	9.8E-1	0.1661	1.653	0.596

#	Time	Area	Height	Width	Area%	Symmetry
1	82.72	6.1	6.2E-1	0.163	5.092	0.686
2	84.275	113.4	9.9	0.1903	94.908	0.524

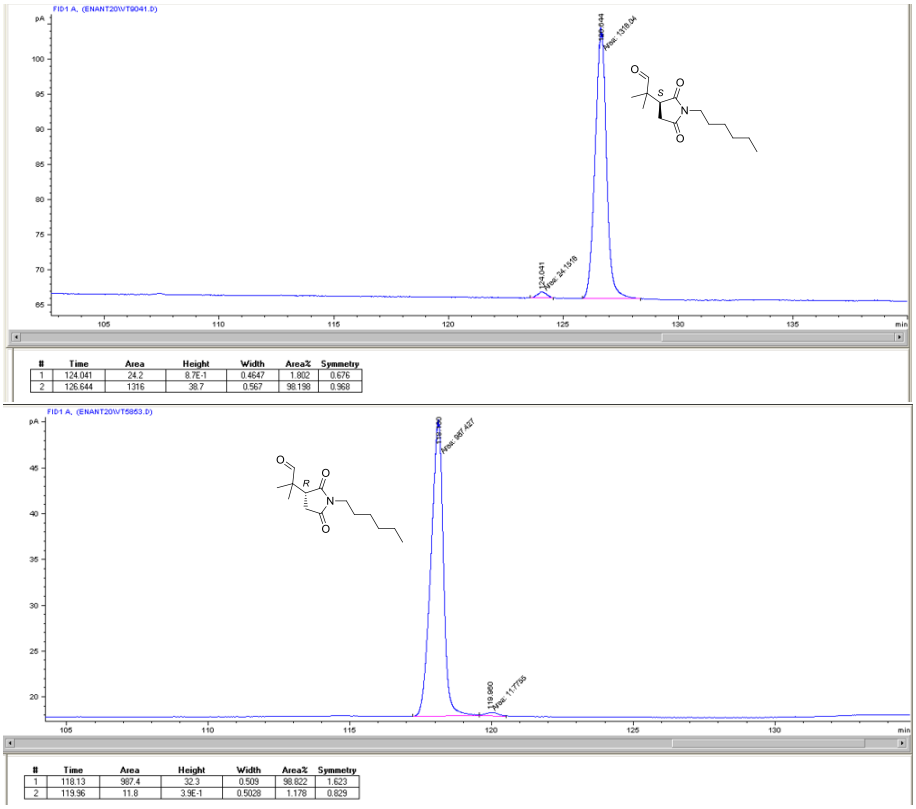
2-methyl-2-(1-butyl-2,5-dioxopyrrolidin-3-yl)propanal (**5d**),



#	Time	Area	Height	Width	Area%	Symmetry
1	82.461	25.5	1.6	0.2707	1.708	1.148
2	83.733	1467.5	83.5	0.2928	98.292	1.502

#	Time	Area	Height	Width	Area%	Symmetry
1	82.542	75.9	4.6	0.2759	96.218	1.072
2	83.742	3	2.2E-1	0.2267	3.782	0.992

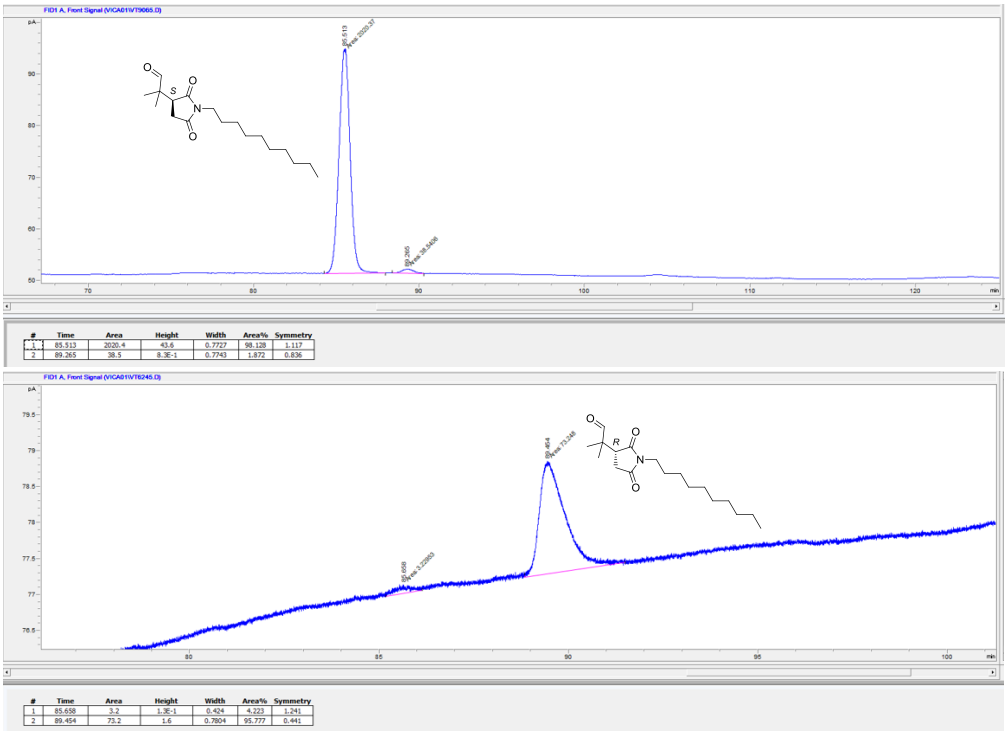
2-methyl-2-(1-hexyl-2,5-dioxopyrrolidin-3-yl)propanal (**5e**),



#	Time	Area	Height	Width	Area%	Symmetry
1	124.041	24.2	8.7E-1	0.4647	1.802	0.676
2	126.644	1316	38.7	0.567	98.198	0.968

#	Time	Area	Height	Width	Area%	Symmetry
1	118.13	987.4	32.3	0.509	98.822	1.623
2	119.96	11.8	3.9E-1	0.5028	1.178	0.829

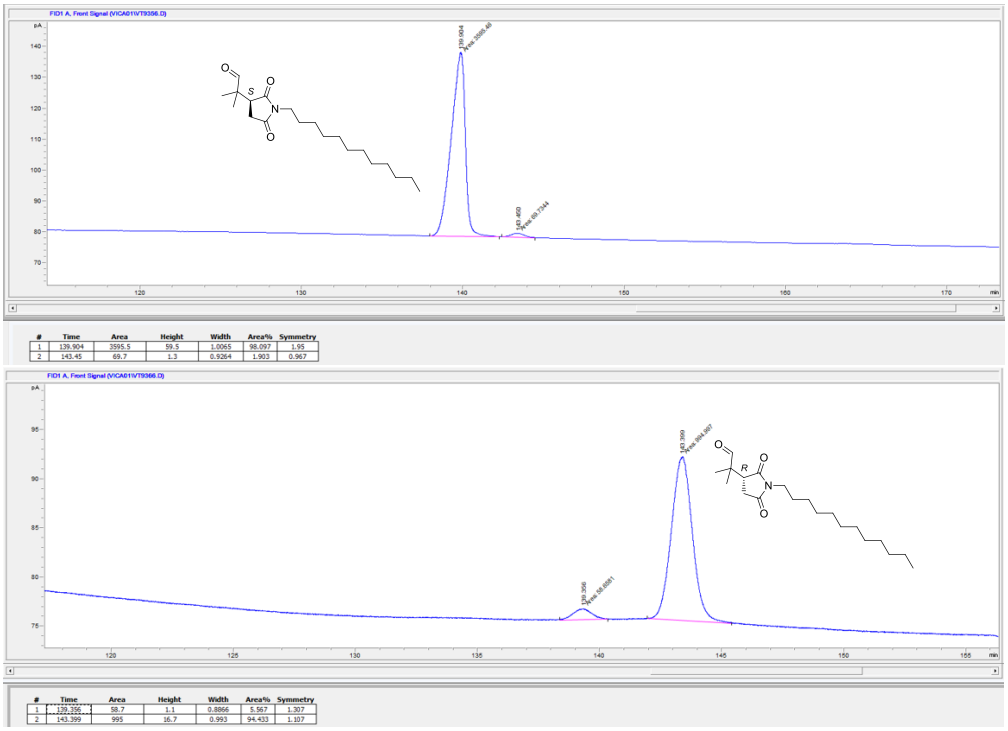
2-methyl-2-(1-decyl-2,5-dioxopyrrolidin-3-yl)propanal (**5f**),



#	Time	Area	Height	Width	Area%	Symmetry
1	85.513	2020.4	43.6	0.7727	98.128	1.117
2	89.265	38.5	8.3E-1	0.7743	1.872	0.836

#	Time	Area	Height	Width	Area%	Symmetry
1	85.658	3.2	1.3E-1	0.424	4.223	1.241
2	89.454	73.2	1.6	0.7804	95.777	0.441

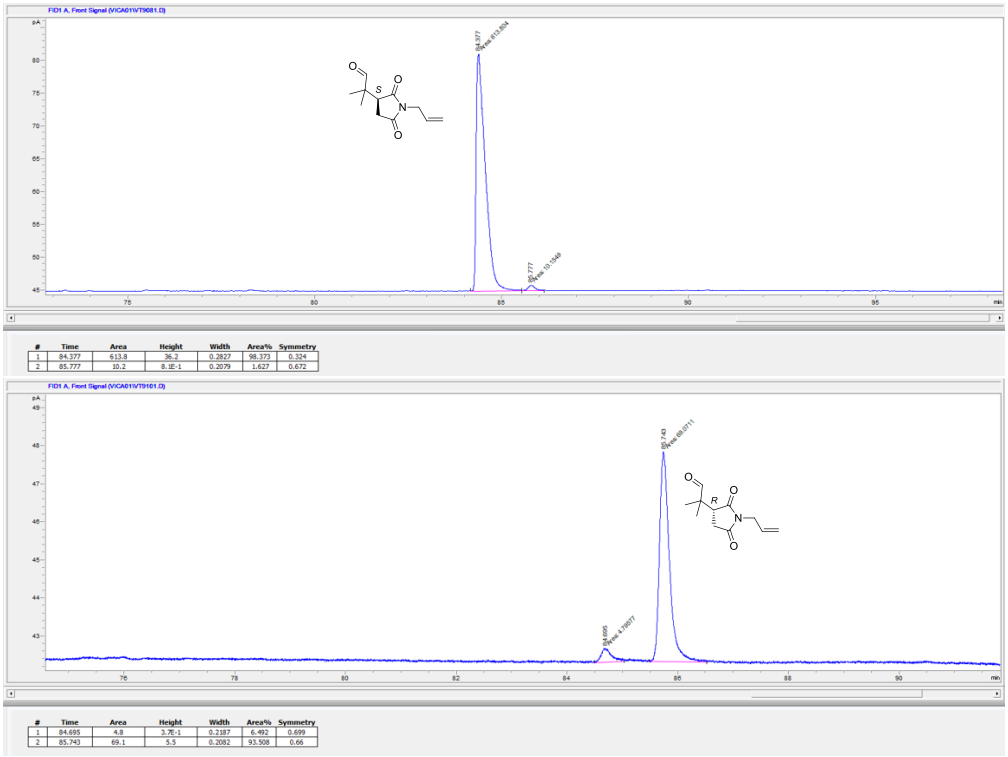
2-methyl-2-(1-dodecyl-2,5-dioxopyrrolidin-3-yl)propanal (**5g**),



#	Time	Area	Height	Width	Area%	Symmetry
1	139.904	3995.5	59.5	1.0065	98.097	1.95
2	143.45	69.7	1.3	0.9264	1.903	0.967

#	Time	Area	Height	Width	Area%	Symmetry
1	139.356	58.7	1.1	0.8866	5.567	1.307
2	143.399	995	16.7	0.993	94.433	1.107

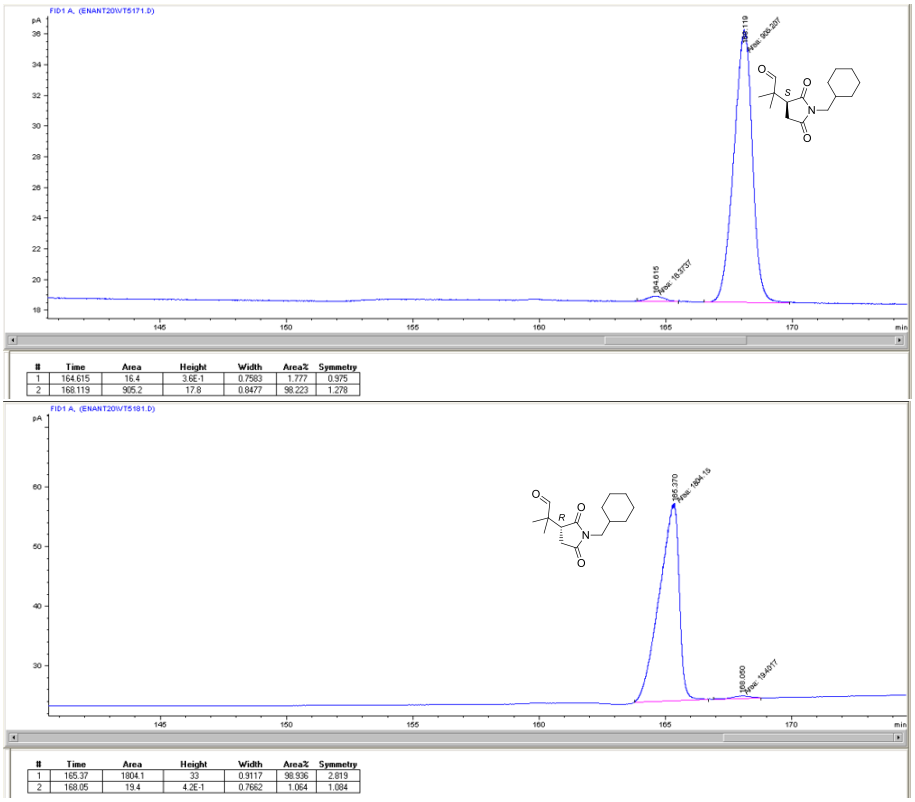
2-methyl-2-(1-allyl-2,5-dioxopyrrolidin-3-yl)propanal (**5h**),



#	Time	Area	Height	Width	Area%	Symmetry
1	84.377	613.8	36.2	0.2827	98.373	0.324
2	85.777	10.2	8.1E-1	0.2079	1.627	0.672

#	Time	Area	Height	Width	Area%	Symmetry
1	84.695	4.8	3.7E-1	0.2187	6.492	0.699
2	85.743	69.1	5.5	0.2082	93.508	0.66

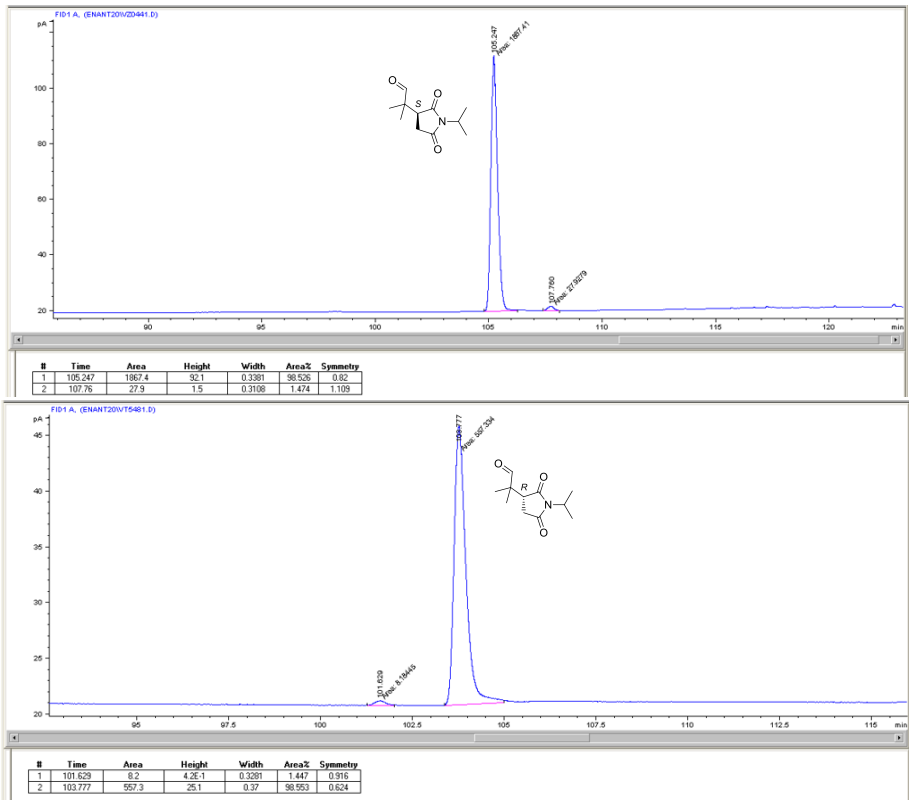
2-(1-cyclohexylmethyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5i**),



#	Time	Area	Height	Width	Area%	Symmetry
1	164.615	16.4	3.6E-1	0.7583	1.777	0.975
2	168.119	905.2	17.8	0.8477	98.223	1.278

#	Time	Area	Height	Width	Area%	Symmetry
1	165.37	1804.1	33	0.9117	98.936	2.819
2	168.05	19.4	4.2E-1	0.7662	1.064	1.084

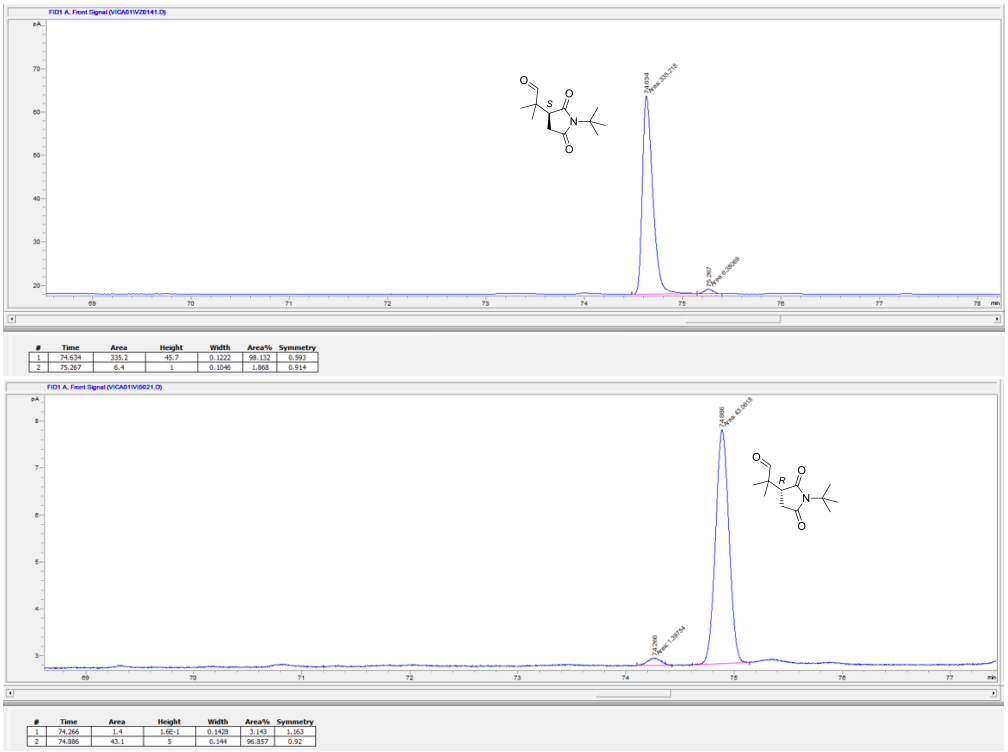
2-(1-isopropyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5j**),



#	Time	Area	Height	Width	Area%	Symmetry
1	105.247	1867.4	92.1	0.3381	98.526	0.82
2	107.76	27.9	1.5	0.3108	1.474	1.109

#	Time	Area	Height	Width	Area%	Symmetry
1	101.629	8.2	4.2E-1	0.3281	1.447	0.916
2	103.777	557.3	25.1	0.37	98.553	0.624

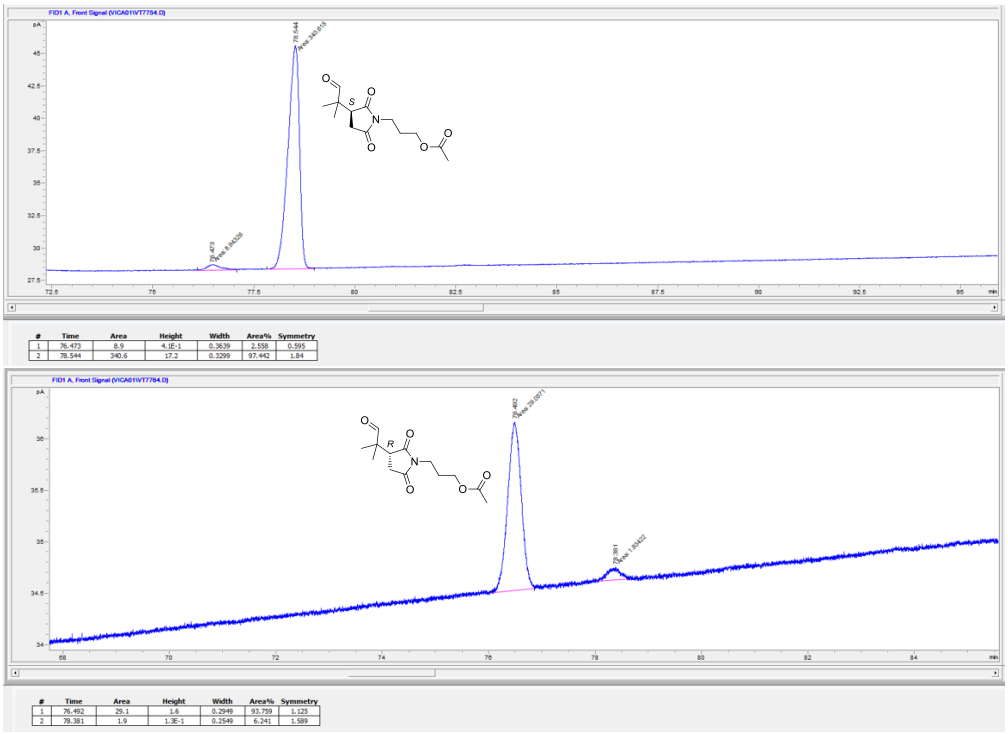
2-(1-*tert*-butyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5k**),



#	Time	Area	Height	Width	Area%	Symmetry
1	74.634	335.2	45.7	0.1222	98.132	0.593
2	75.267	6.4	1	0.1046	1.868	0.914

#	Time	Area	Height	Width	Area%	Symmetry
1	74.266	1.4	1.6E-1	0.1428	3.143	1.163
2	74.886	43.1	5	0.144	96.857	0.92

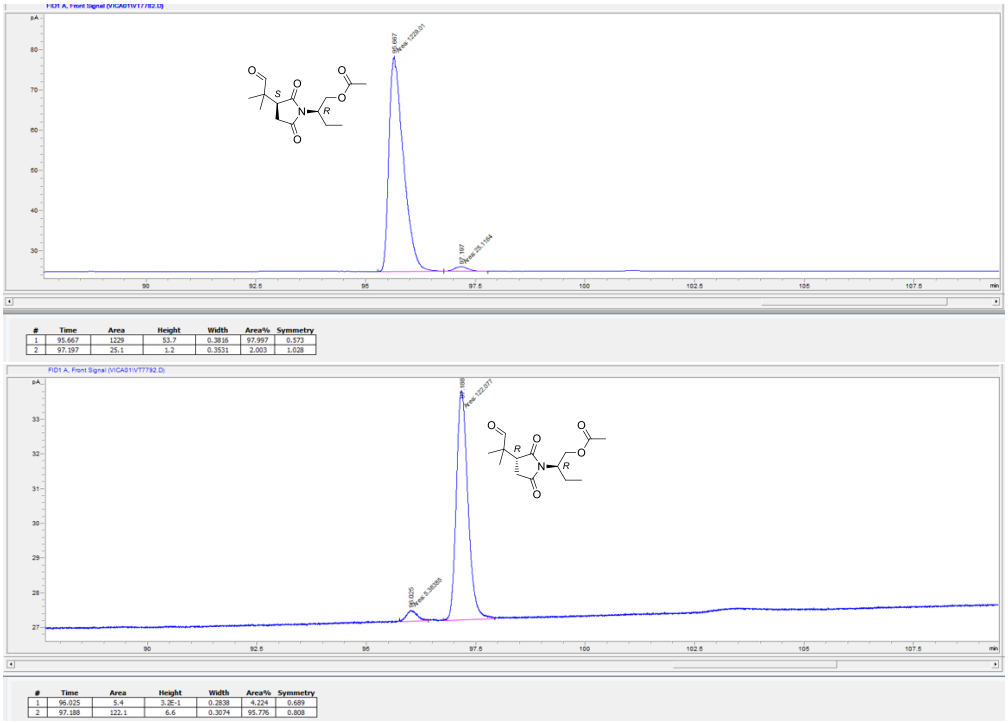
2-(1-(3-acetoxypropyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5l**),



#	Time	Area	Height	Width	Area%	Symmetry
1	76.473	8.9	4.1E-1	0.3639	2.558	0.595
2	78.544	340.6	17.2	0.3299	97.442	1.84

#	Time	Area	Height	Width	Area%	Symmetry
1	76.492	29.1	1.6	0.2949	93.759	1.125
2	78.381	1.9	1.3E-1	0.2549	6.241	1.589

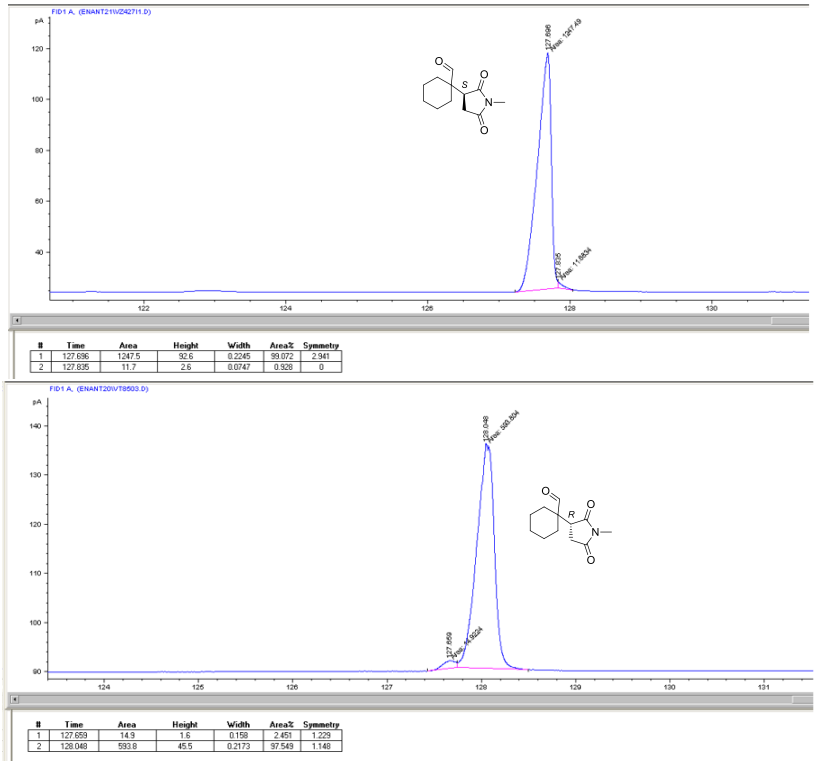
2-(*R*)-*N*-[1-[(acetyloxy)methyl]propyl]-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (**5m**),



#	Time	Area	Height	Width	Area%	Symmetry
1	95.667	1229	53.7	0.3816	97.997	0.573
2	97.197	25.1	1.2	0.3531	2.003	1.028

#	Time	Area	Height	Width	Area%	Symmetry
1	96.025	5.4	3.2E-1	0.2838	4.224	0.689
2	97.188	122.1	6.6	0.3074	95.776	0.808

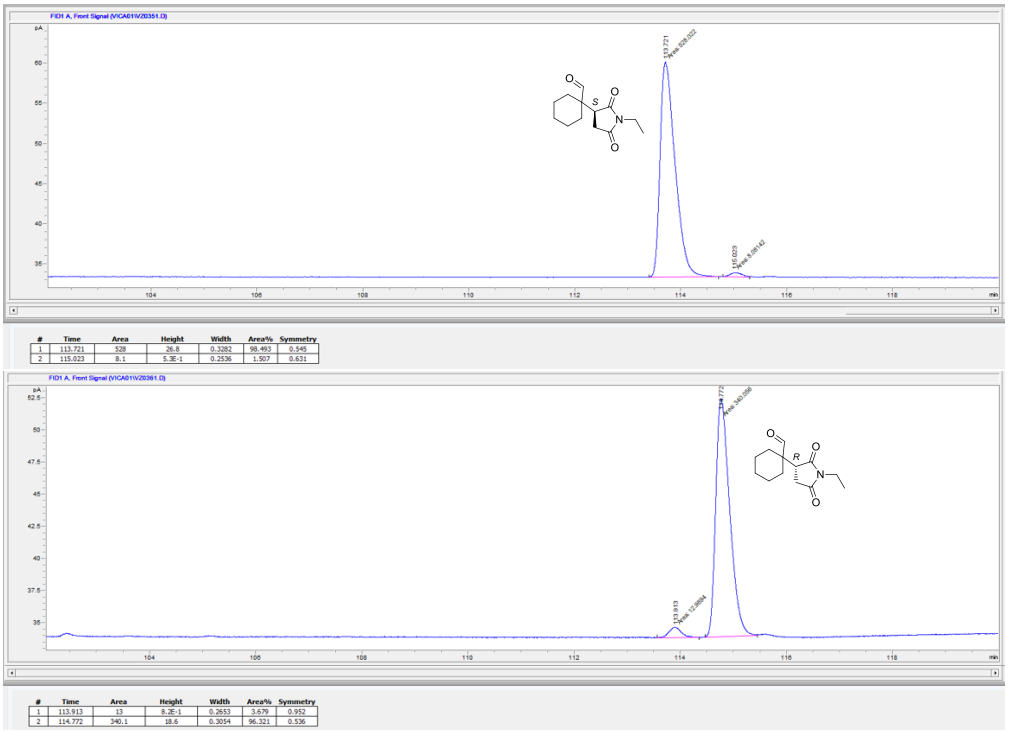
1-(1-methyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6a**),



#	Time	Area	Height	Width	Area%	Symmetry
1	127.696	1247.5	92.6	0.2245	99.072	2.941
2	127.835	11.7	2.6	0.0747	0.928	0

#	Time	Area	Height	Width	Area%	Symmetry
1	127.659	14.9	1.6	0.158	2.451	1.229
2	128.048	593.8	45.5	0.2173	97.549	1.148

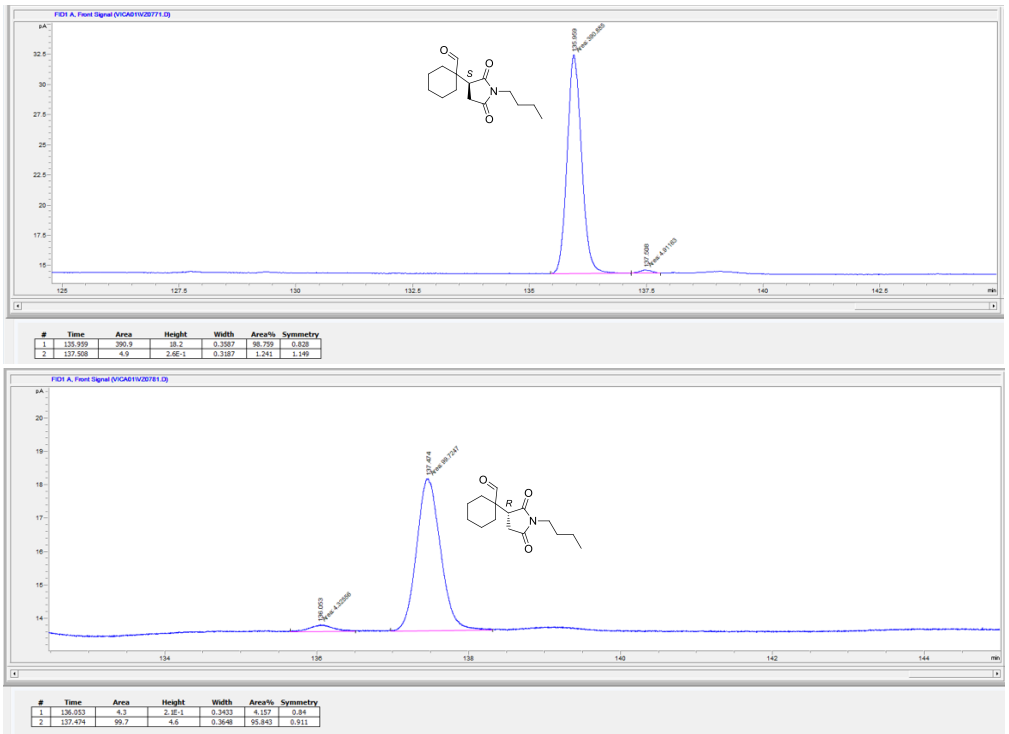
1-(1-ethyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6b**),



#	Time	Area	Height	Width	Area%	Symmetry
1	113.721	528	26.8	0.3282	98.493	0.545
2	115.023	8.1	5.3E-1	0.2536	1.507	0.631

#	Time	Area	Height	Width	Area%	Symmetry
1	113.913	13	8.2E-1	0.2653	3.679	0.952
2	114.772	340.1	18.6	0.3054	96.321	0.536

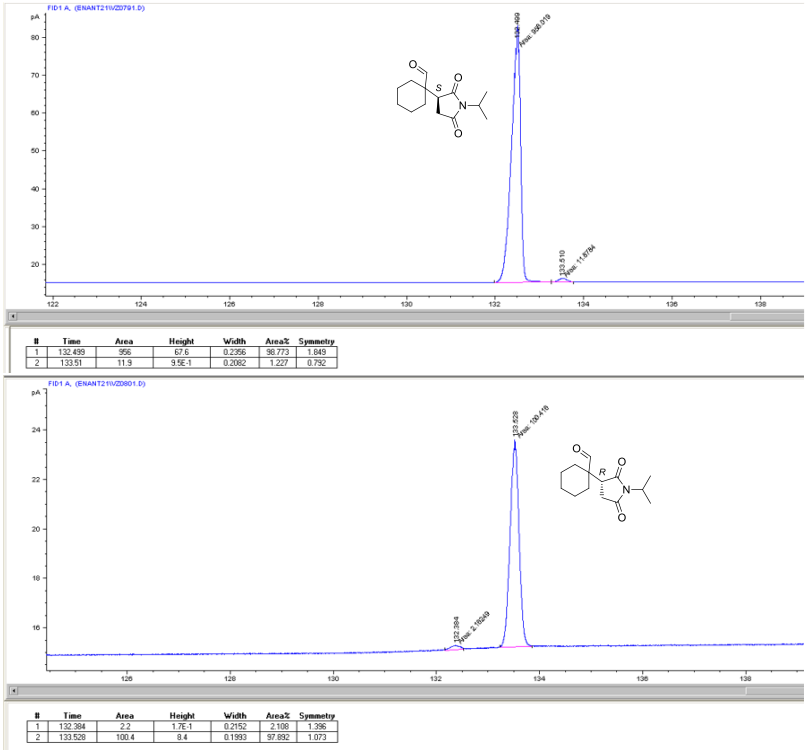
1-(1-butyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6c**),



#	Time	Area	Height	Width	Area%	Symmetry
1	135.959	390.9	18.2	0.3587	98.759	0.828
2	137.508	4.9	2.6E-1	0.3187	1.241	1.149

#	Time	Area	Height	Width	Area%	Symmetry
1	136.053	4.3	2.1E-1	0.3433	4.157	0.84
2	137.474	99.7	4.6	0.3648	95.843	0.911

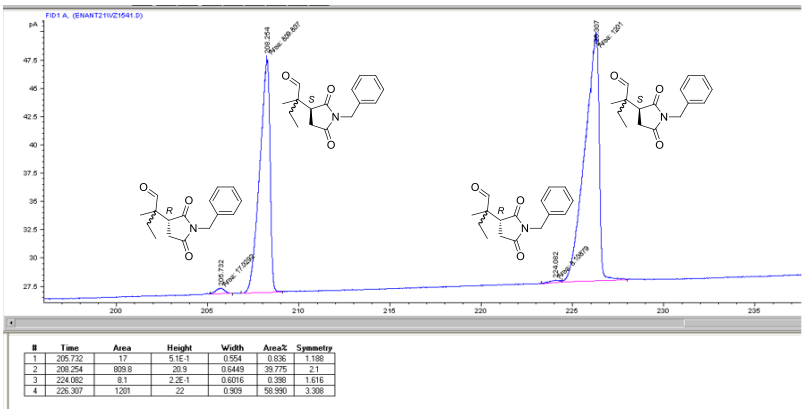
1-(1-isopropyl-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (**6d**),



#	Time	Area	Height	Width	Area%	Symmetry
1	132.499	956	67.6	0.2356	98.773	1.849
2	133.51	11.9	9.5E-1	0.2082	1.227	0.792

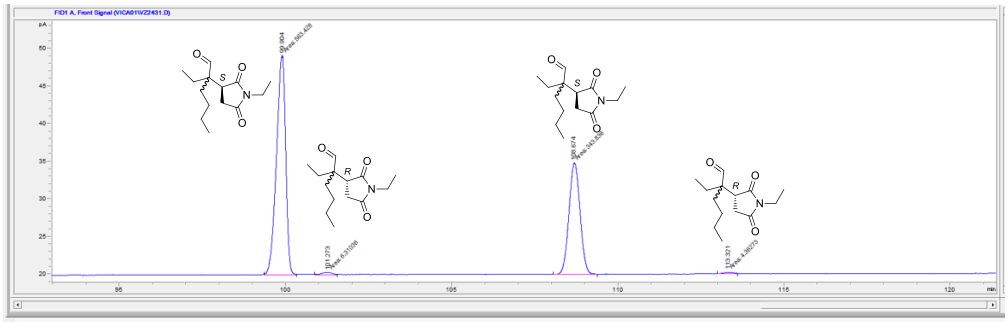
#	Time	Area	Height	Width	Area%	Symmetry
1	132.384	2.2	1.7E-1	0.2152	2.108	1.396
2	133.529	100.4	8.4	0.1993	97.892	1.073

2-(1-benzyl-2,5-dioxopyrrolidin-3-yl)-2-methylbutanal (**6e**),



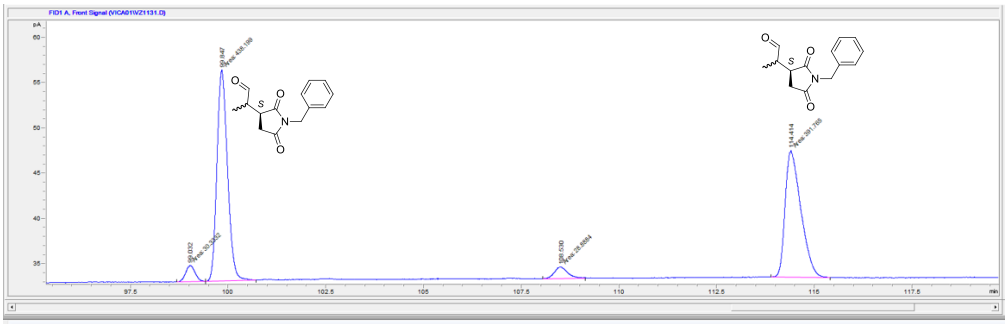
#	Time	Area	Height	Width	Area%	Symmetry
1	205.732	17	5.1E-1	0.554	0.836	1.188
2	208.254	809.8	20.9	0.6449	39.775	2.1
3	224.082	8.1	2.2E-1	0.6016	0.398	1.616
4	226.307	1201	22	0.909	58.990	3.308

2-ethyl-2-(1-ethyl-2,5-dioxopyrrolidin-3-yl)hexanal (**6f**),

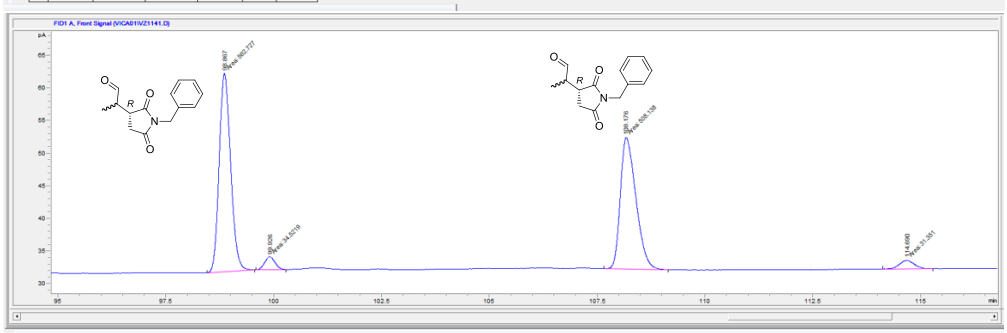


#	Time	Area	Height	Width	Area%	Symmetry
1	99.904	563.4	29.2	0.3214	61.380	1.7
2	101.273	6.3	3.2E-1	0.3252	0.687	1.406
3	108.674	343.8	14.8	0.3867	37.457	0.792
4	113.321	4.4	2.3E-1	0.3184	0.475	0.883

2-(1-benzyl-2,5-dioxopyrrolidin-3-yl)propanal (**6g**),

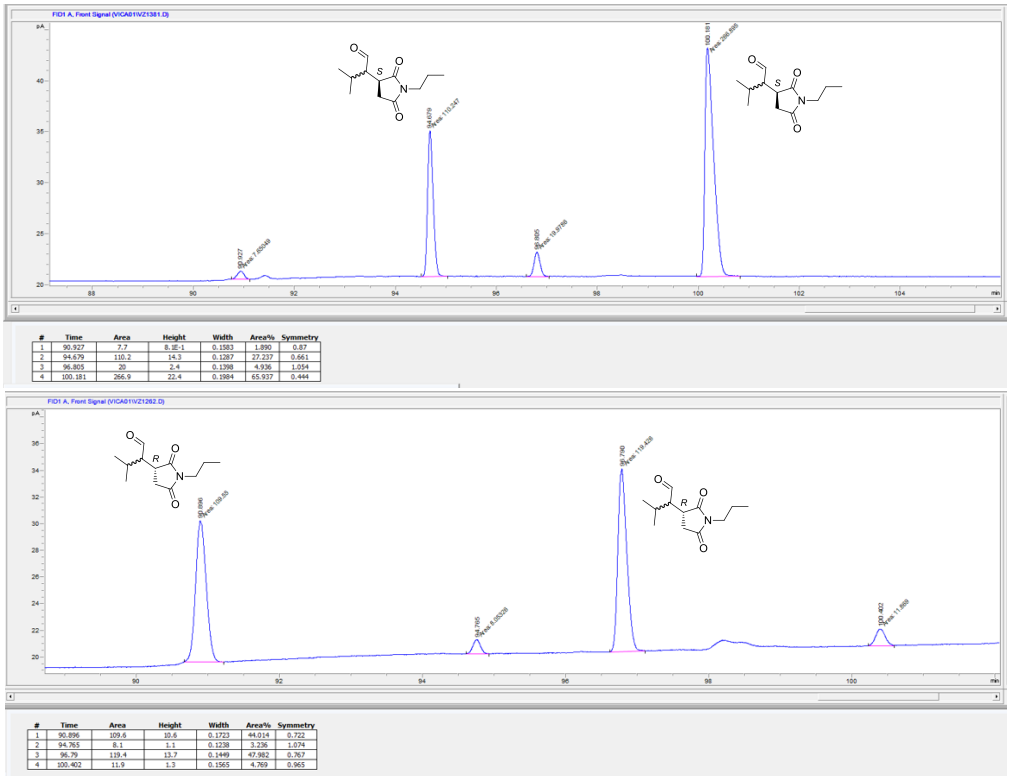


#	Time	Area	Height	Width	Area%	Symmetry
1	99.032	30.3	1.8	0.2803	3.411	0.786
2	99.847	438.2	23.3	0.3131	49.281	0.78
3	108.53	28.9	1.3	0.3783	3.249	0.96
4	114.414	391.8	14	0.4668	44.059	0.573



#	Time	Area	Height	Width	Area%	Symmetry
1	98.867	562.7	30.5	0.3077	49.504	0.783
2	99.926	34.5	2	0.284	3.037	1.003
3	108.176	508.1	20.2	0.4196	44.701	0.541
4	114.69	31.4	1.3	0.3922	2.758	0.977

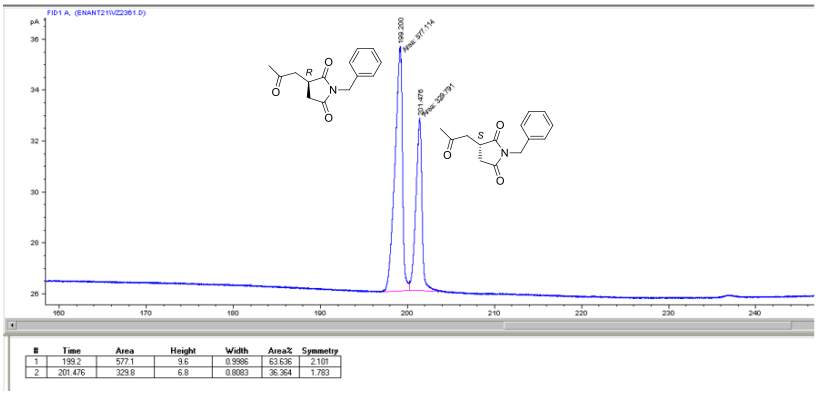
2-(2,5-dioxo-1-propylpyrrolidin-3-yl)-3-methylbutanal (**6h**),



#	Time	Area	Height	Width	Area%	Symmetry
1	90.927	7.7	8.3E-1	0.1583	1.890	0.87
2	94.679	110.2	14.3	0.1287	27.237	0.661
3	96.805	20	2.4	0.1398	4.936	1.054
4	100.181	266.9	22.4	0.1984	65.937	0.444

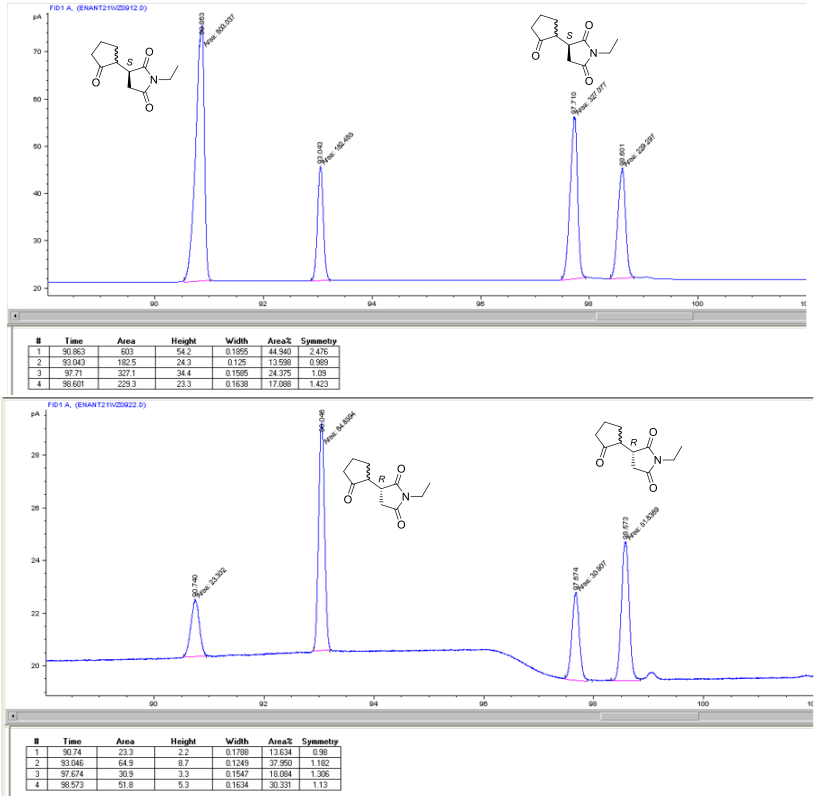
#	Time	Area	Height	Width	Area%	Symmetry
1	90.896	109.6	10.6	0.1723	44.014	0.722
2	94.765	8.1	1.1	0.1238	3.236	1.074
3	96.79	119.4	13.7	0.1449	47.982	0.767
4	100.402	11.9	1.3	0.1565	4.769	0.965

1-benzyl-3-(2-oxopropyl)pyrrolidine-2,5-dione (**6i**),



#	Time	Area	Height	Width	Area%	Symmetry
1	199.2	577.1	9.6	0.9986	63.636	2.101
2	201.476	329.8	6.8	0.8083	36.364	1.783

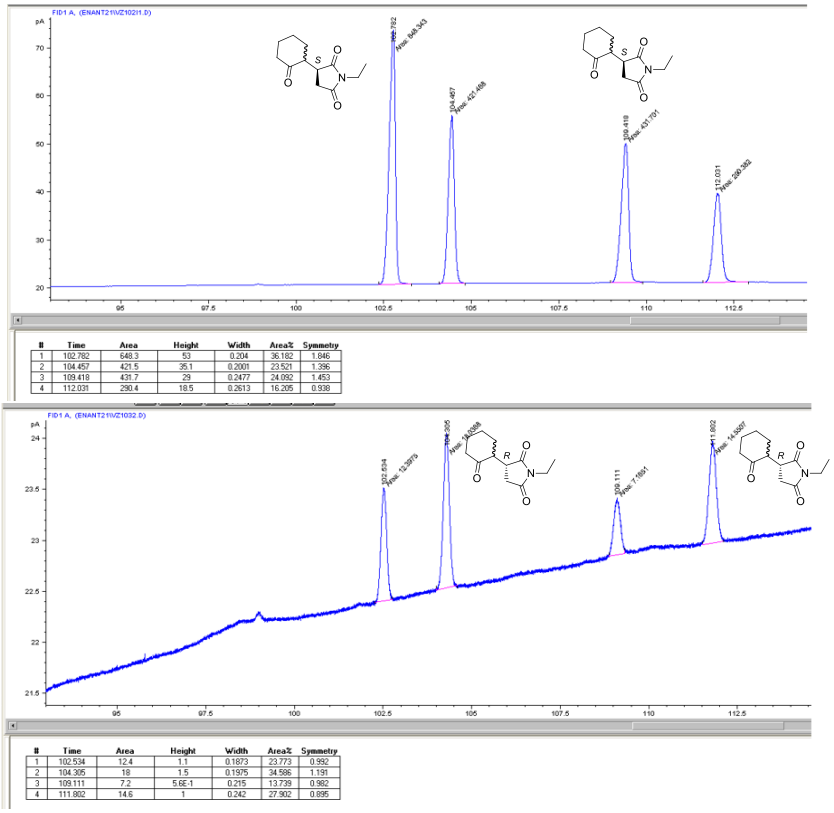
1-ethyl-3-(2-oxocyclopentyl)pyrrolidine-2,5-dione (**6j**),



#	Time	Area	Height	Width	Area%	Symmetry
1	90.863	603	54.2	0.1855	44.940	2.476
2	93.043	182.5	24.3	0.125	13.598	0.989
3	97.71	327.1	34.4	0.1585	24.375	1.09
4	98.601	229.3	23.3	0.1638	17.088	1.423

#	Time	Area	Height	Width	Area%	Symmetry
1	90.74	23.3	2.2	0.1788	13.634	0.98
2	93.046	64.9	8.7	0.1249	37.950	1.182
3	97.674	30.9	3.3	0.1547	18.084	1.306
4	98.573	51.8	5.3	0.1634	30.331	1.13

1-ethyl-3-(2-oxocyclohexyl)pyrrolidine-2,5-dione (**6k**),



#	Time	Area	Height	Width	Area%	Symmetry
1	102.782	648.3	53	0.204	36.182	1.846
2	104.457	421.5	35.1	0.2001	23.521	1.396
3	109.418	431.7	29	0.2477	24.092	1.453
4	112.031	290.4	18.5	0.2613	16.205	0.938

#	Time	Area	Height	Width	Area%	Symmetry
1	102.534	12.4	1.1	0.1873	23.773	0.992
2	104.305	18	1.5	0.1975	34.586	1.191
3	109.111	7.2	5.6E-1	0.215	13.739	0.982
4	111.802	14.6	1	0.242	27.902	0.895