

SUPPLEMENTARY MATERIAL

One-pot synthesis of substituted piperidinones and 3,4-dihydropyrimidinones using highly active, recyclable supported ionic liquid phase organocatalyst

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Spectral data of 1-ethyl-3-methylimidazolium ethylsulphate (Ionic liquid)

¹H-NMR (400 MHz, CDCl₃): Cationic part, δ 1.32 (t, 3H, CH₃), 3.16 (s, 1H, CH), 3.79 (s, 3H, N-CH₃), 4.07-4.13 (q, 2H, CH₂), 7.38-7.44 (m, 2H, CH); Anionic part, δ 1.01 (t, 3H, CH₃), 3.80-3.87 (q, 2H, CH₂).

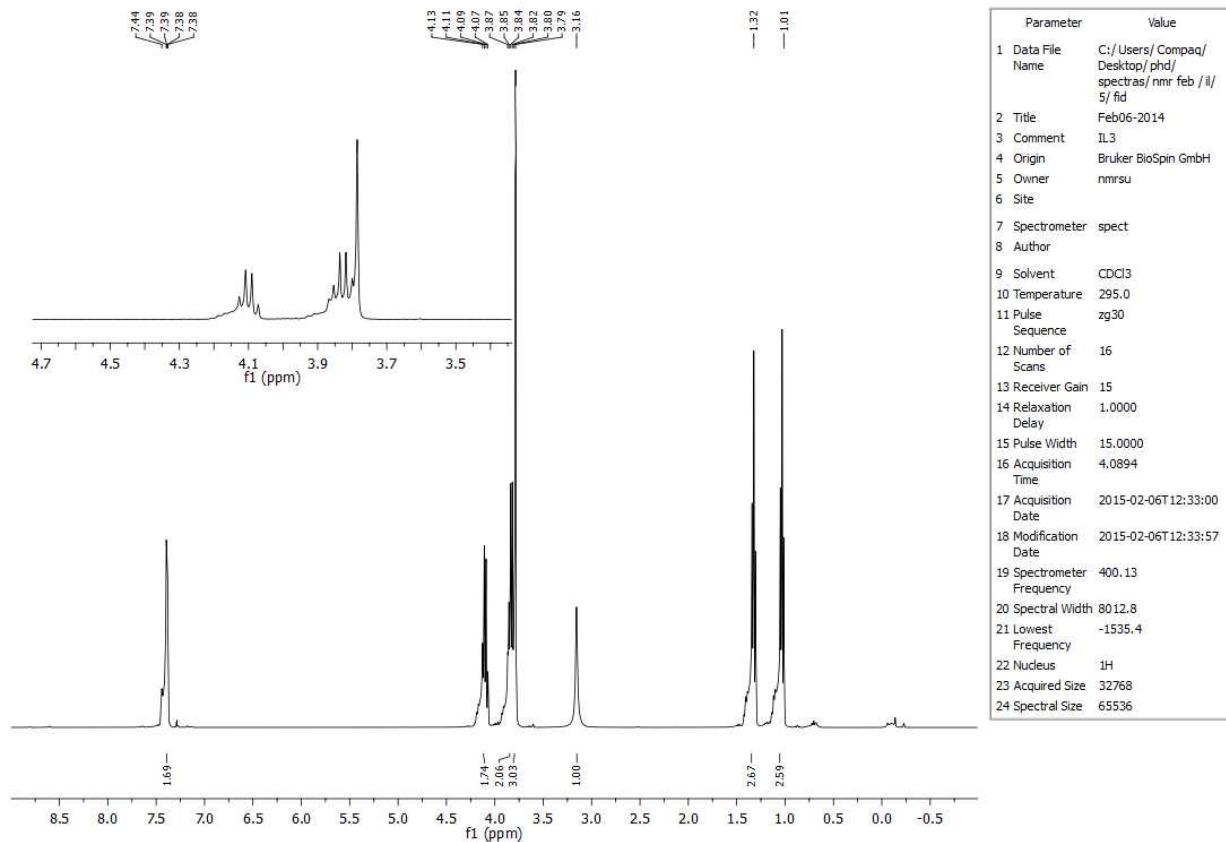


Fig. S1 ¹H NMR of 1-ethyl-3-methylimidazolium ethylsulphate

¹³C-NMR (100 MHz, CDCl₃): Cationic part, δ 15.0, 36.0, 44.8, 122.0, 123.8, 136.4;
Anionic part, 15.3, 63.0.

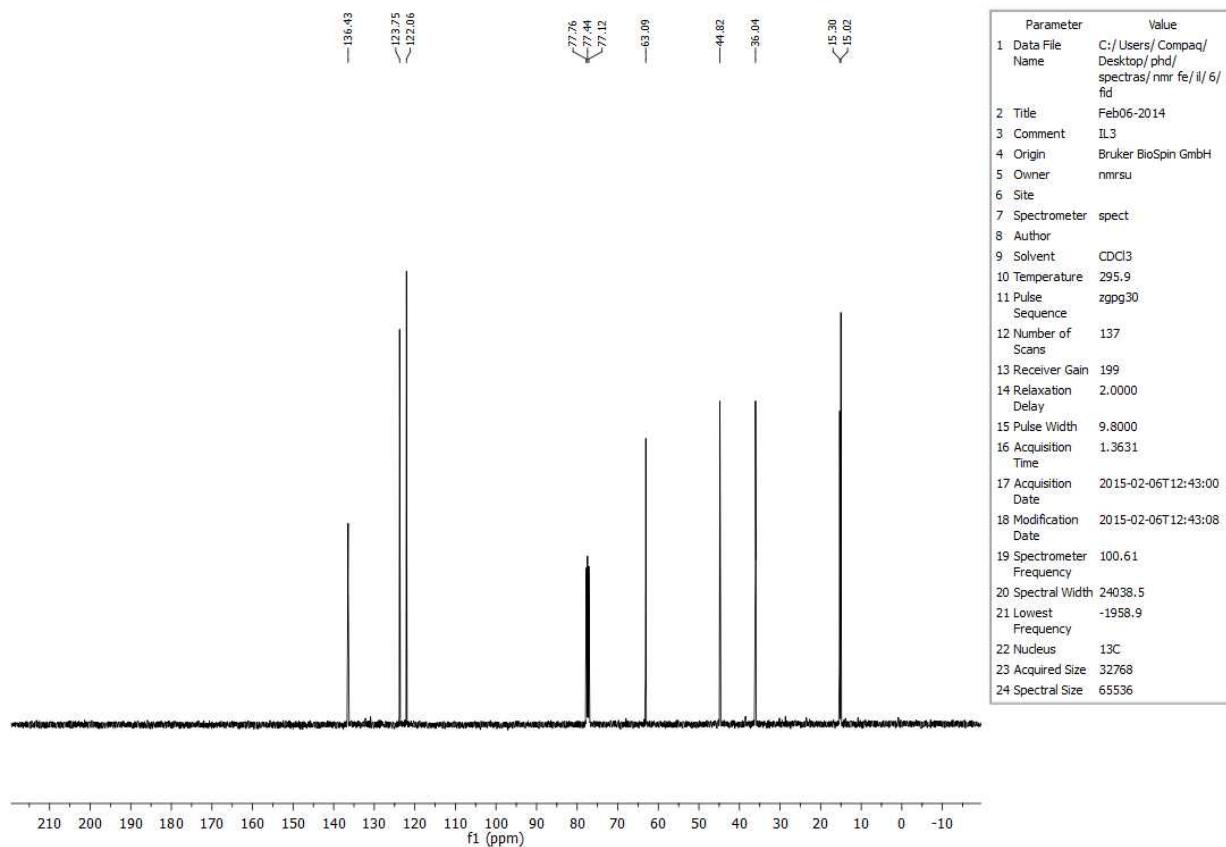


Fig. S2 ¹³C NMR of 1-ethyl-3-methylimidazolium ethylsulphate

MS (ESI): 111 (M+1).

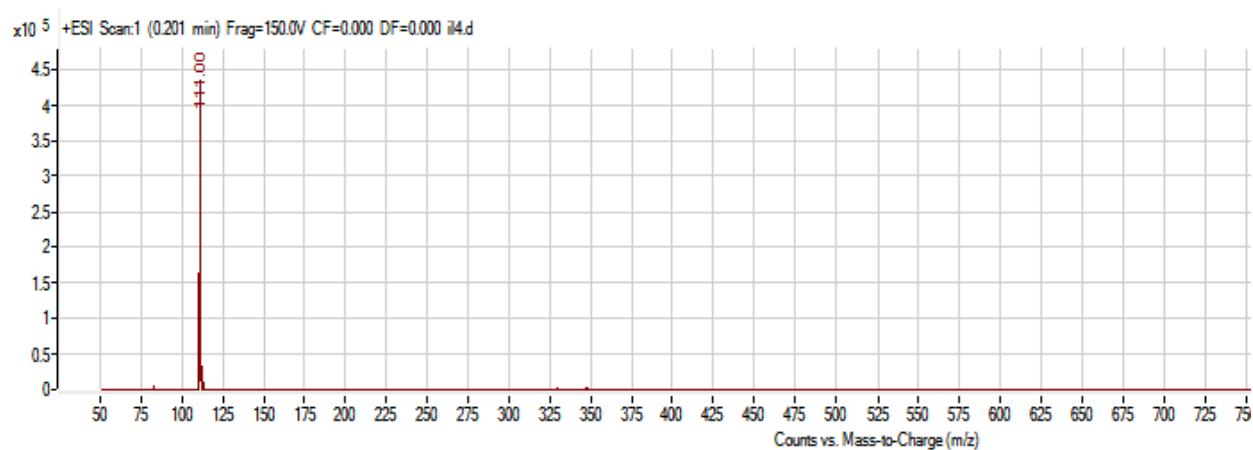


Fig. S3 Mass spectra of 1-ethyl-3-methylimidazolium ethylsulphate

Spectral Data of some 2,6-diarylpiriperidinones

3-methyl-2,6-diphenylpiperidin-4-one (1a)

¹H-NMR (400 MHz, CDCl₃): δ 0.87 (d, 3H, -CH₃), 2.66-2.83 (m, 3H, CH, CH₂), 3.67 (d, 1H, CH), 4.13 (d, 1H, CH), 4.83 (bs, 1H, -NH), 7.29-7.65 (m, 8H, Ar-H), 8.14 (d, 2H, Ar-H).

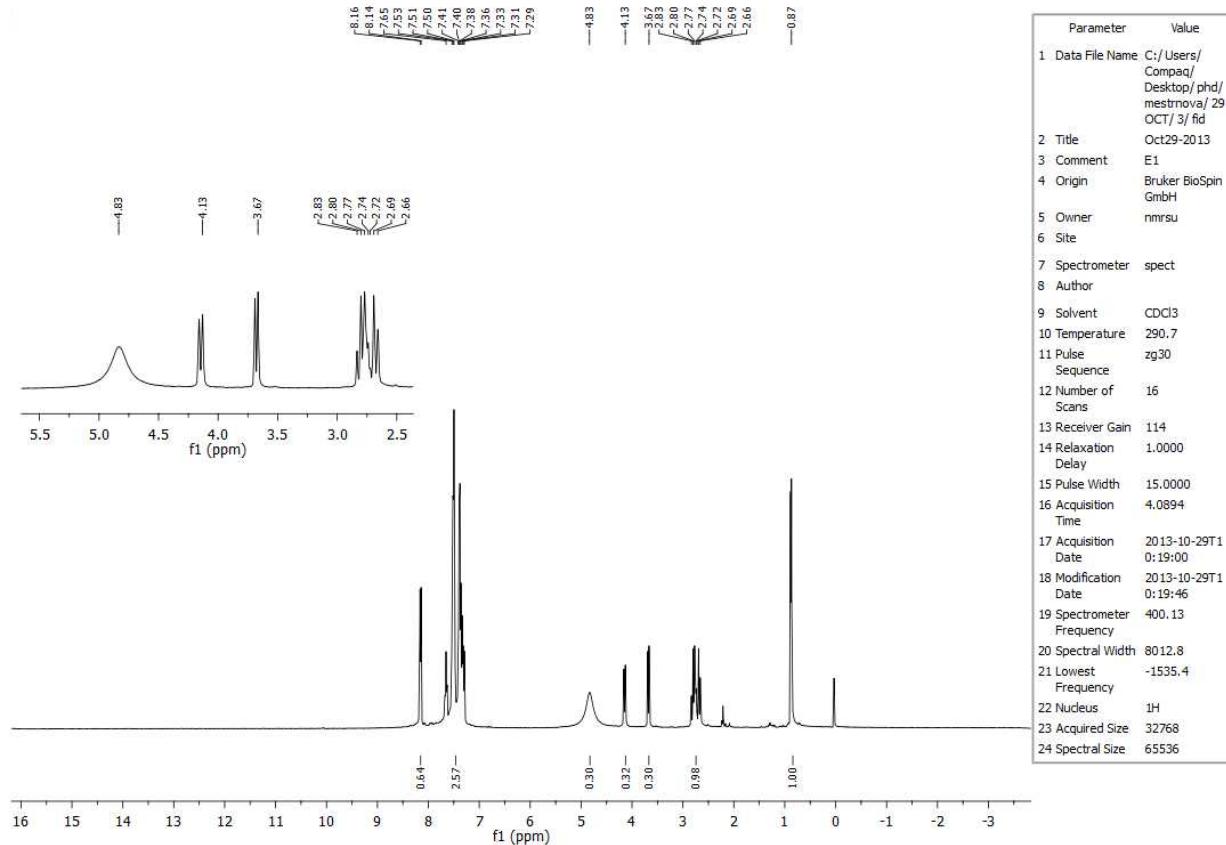


Fig. S4 ¹H-NMR of 3-methyl-2,6-diphenylpiperidin-4-one (1a)

¹³C-NMR (100 MHz, CDCl₃): δ 10.2, 50.8, 51.7, 61.6, 68.5, 126.6, 127.9, 128.5, 128.7, 130.2, 133.7, 141.7, 142.6, 209.8.

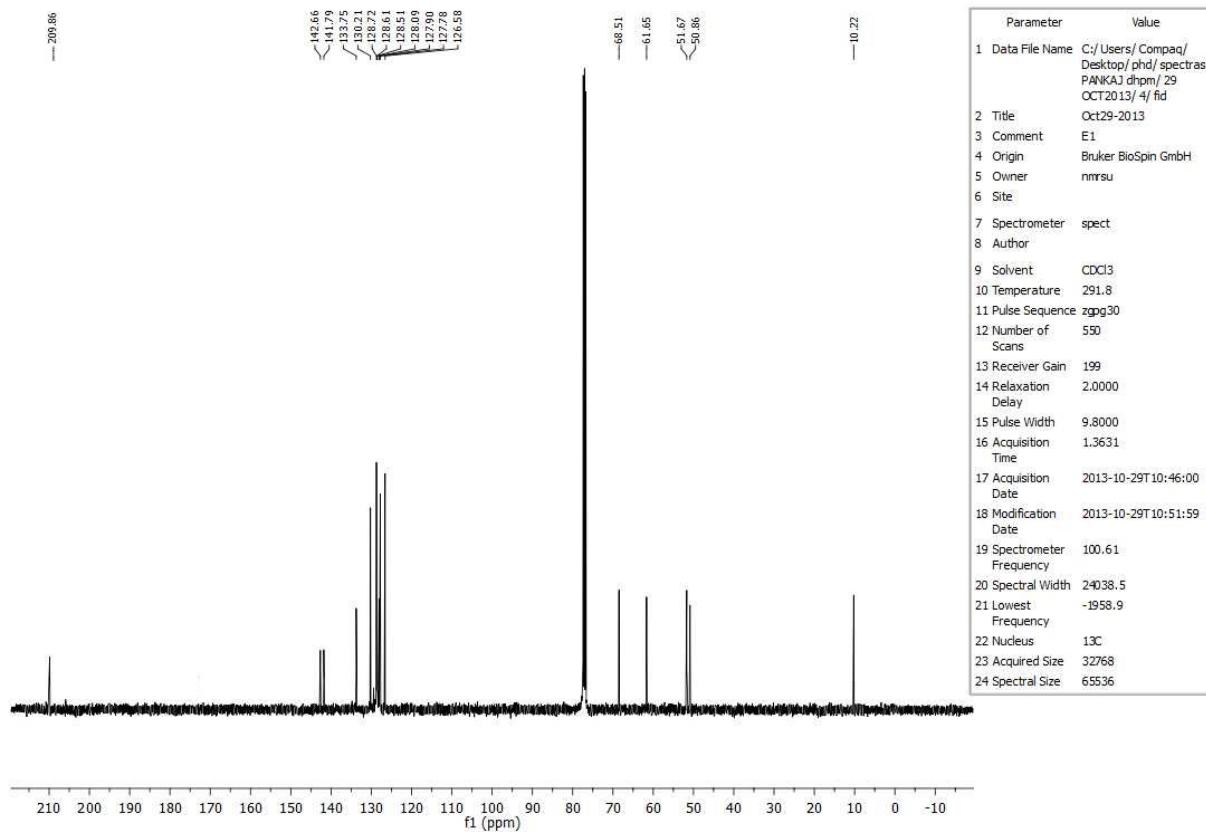


Fig. S5 ¹³C NMR spectra of 3-methyl-2,6-diphenylpiperidin-4-one (1a)

MS (ESI): 266 (M+1).

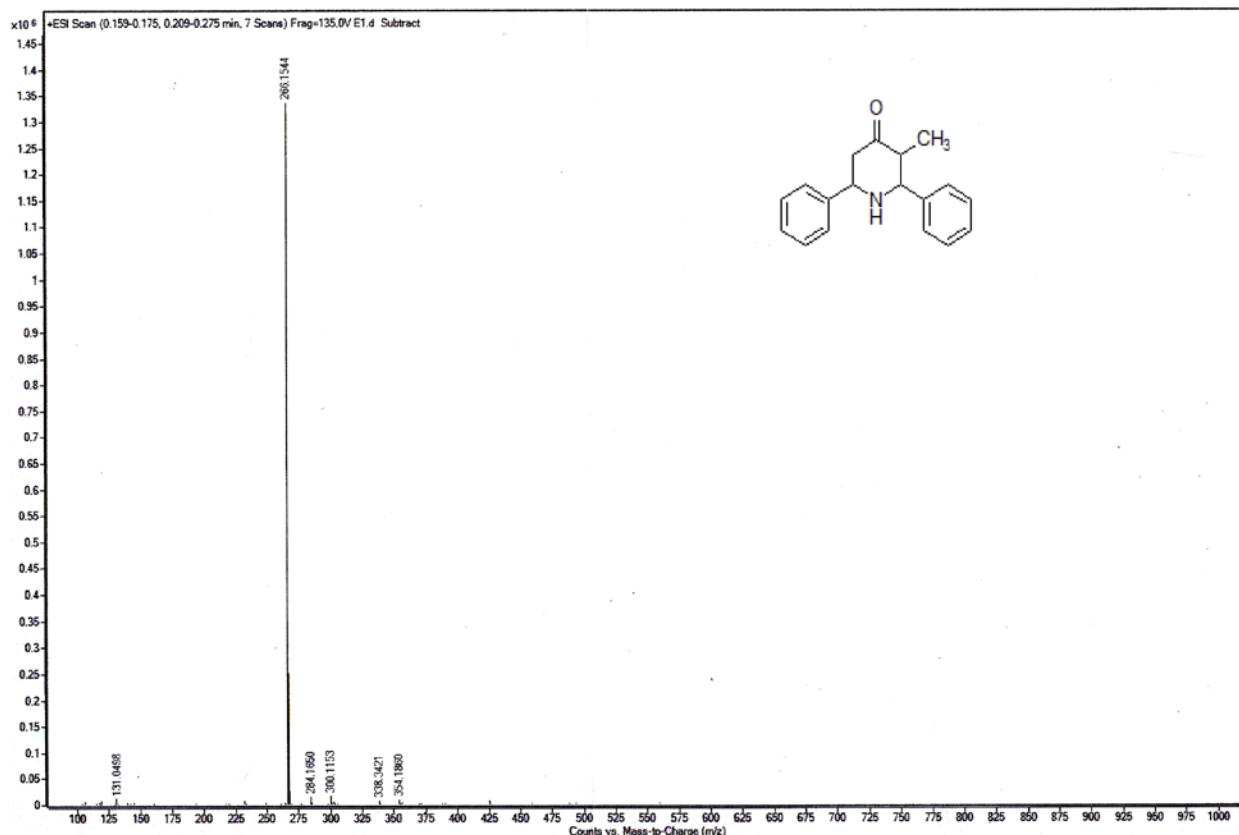


Fig. S6 Mass spectra of 3-methyl-2,6-diphenylpiperidin-4-one (1a)

2,6-Bis-(4-methoxyphenyl)-3-methylpiperidin-4-one (1b)

¹H-NMR (400 MHz, CDCl₃): δ 0.85 (d, 3H, -CH₃), 2.20 (bs, 1H, -NH), 2.59-2.73 (m, 3H, -CH₂, CH), 3.58 (d, 1H, -CH), 3.83 (s, 6H, 2x-OCH₃), 4.07 (d, 1H, CH), 6.89-6.93 (m, 4H, Ar-H), 7.38 (d, 2H, Ar-H), 7.41 (d, 2H, Ar-H).

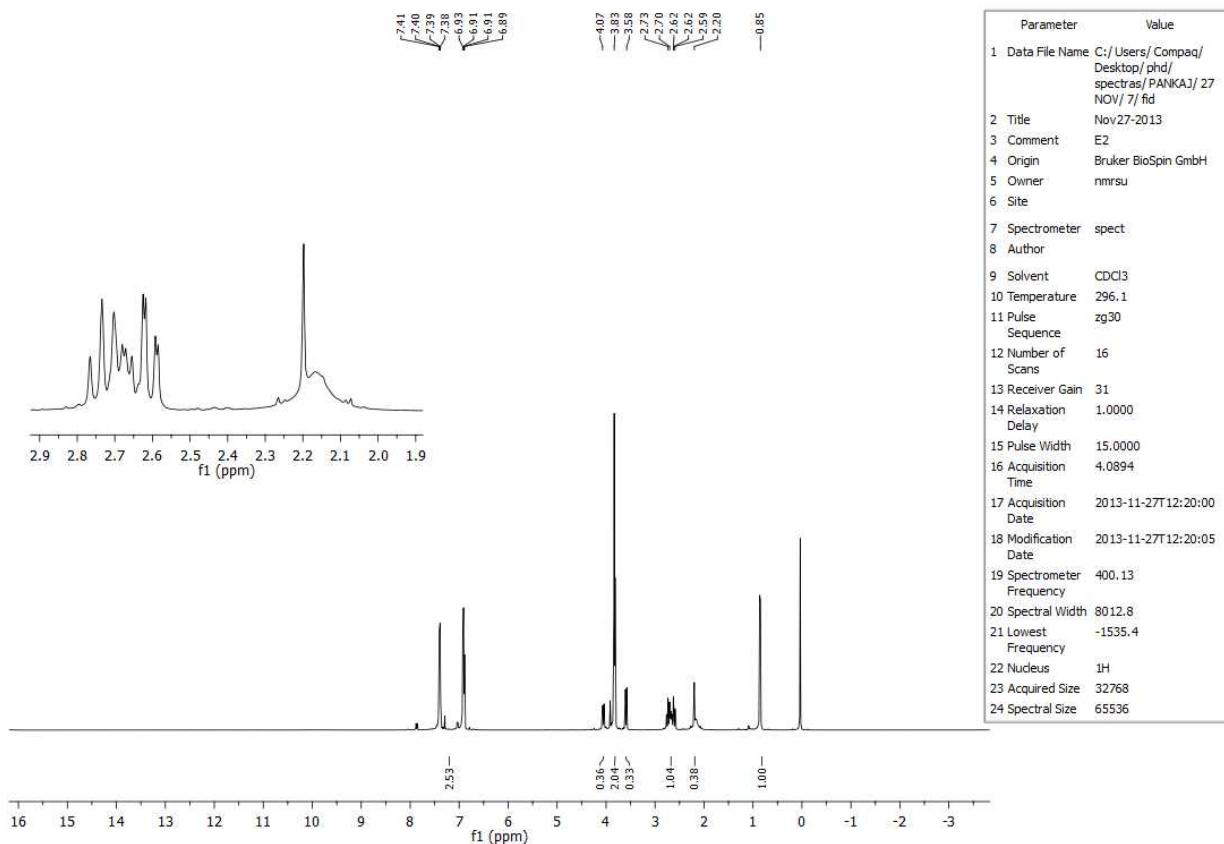


Fig. S7 ¹H NMR spectra of 2,6-Bis-(4-methoxyphenyl)-3-methylpiperidin-4-one (1b)

¹³C-NMR (100 MHz CDCl₃): δ 10.2, 51.9, 55.3, 61.0, 67.9, 113.8, 114.00, 114.33, 127.6, 128.8, 134.2, 135.1, 159.1, 159.3, 209.3.

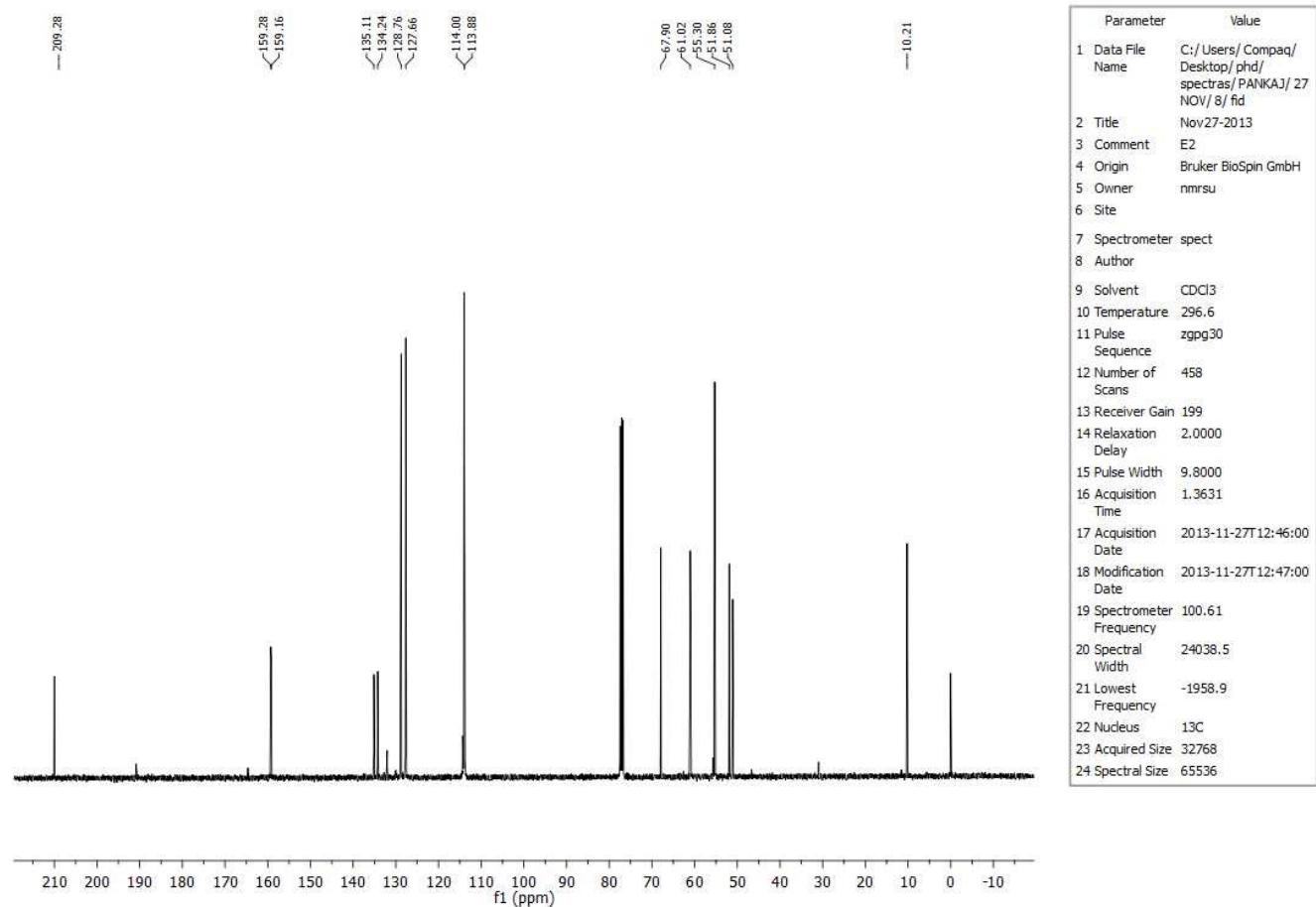


Fig. S8 ¹³C NMR spectra of 2,6-Bis-(4-methoxyphenyl)-3-methylpiperidin-4-one (1b)

MS (ESI): 326 (M+1).

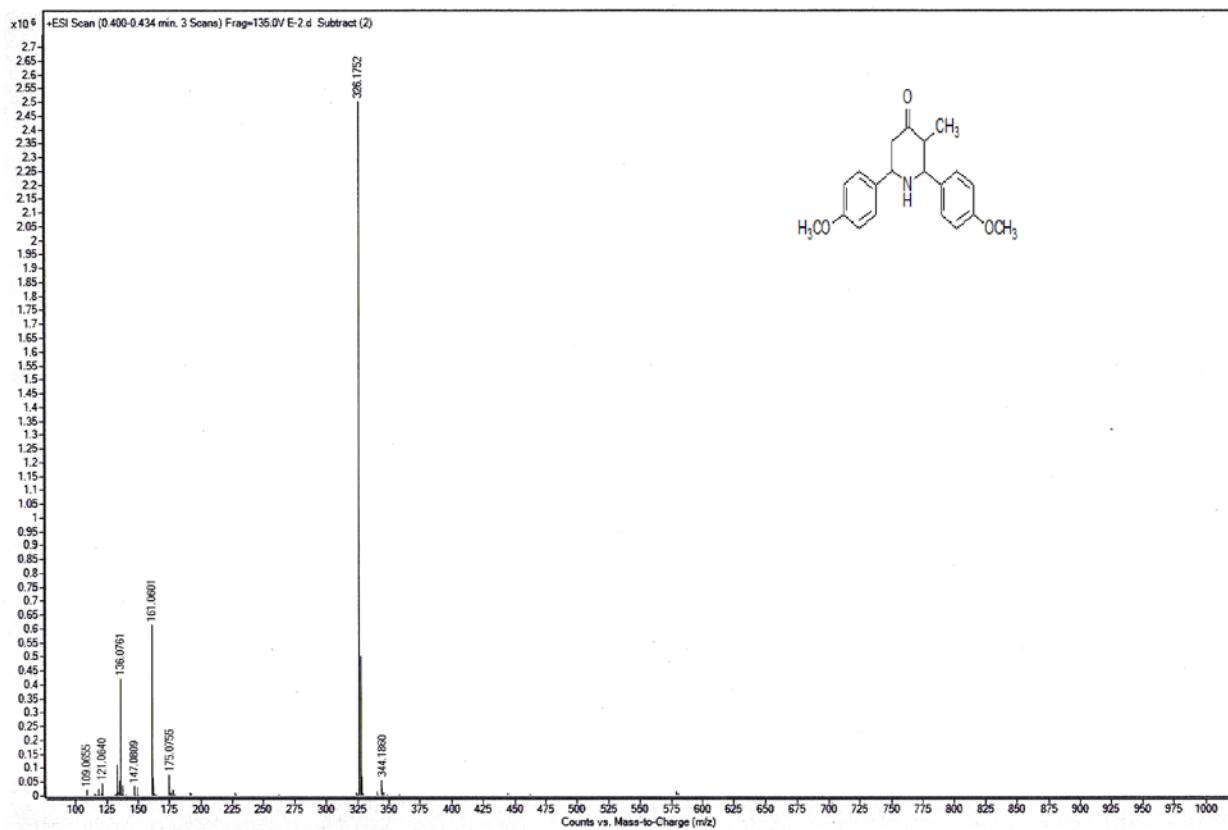


Fig. S9 Mass spectra 2,6-Bis-(4-methoxyphenyl)-3-methylpiperidin-4-one (1b)

3-Methyl-2,6-di-p-tolylpiperidin-4-one (1d)

¹H-NMR (400 MHz, CDCl₃): δ 0.87 (d, 3H, -CH₃), 2.37 (s, 3H, CH₃), 2.47 (s, 3H, CH₃), 2.75 (m, 3H, -CH, CH₂), 3.62 (d, 1H, CH), 4.12 (q, 1H, CH), 5.22 (bs, 1H, -NH), 7.17-7.37 (m, 6H, Ar-H), 8.05 (d, 2H, Ar-H).

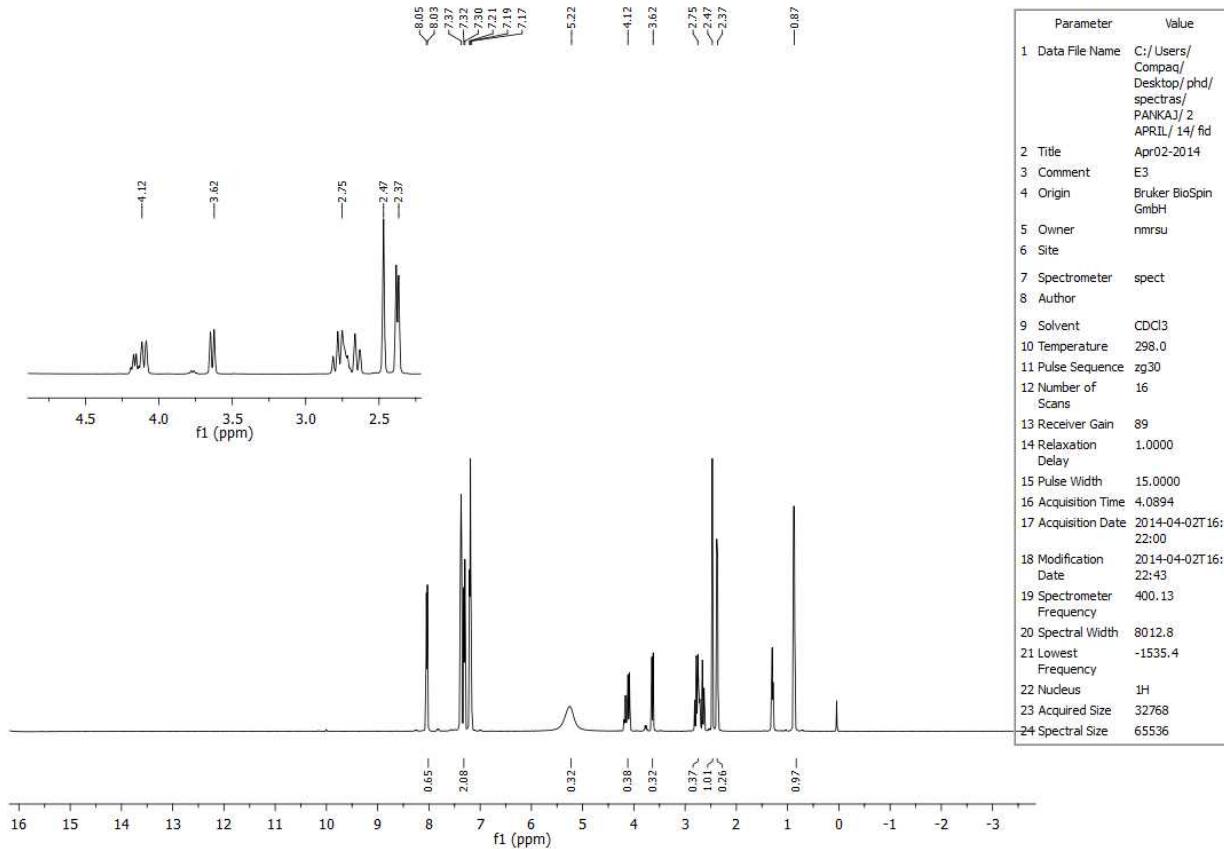


Fig. S10 ¹H-NMR of 3-Methyl-2,6-di-p-tolylpiperidin-4-one (1d)

¹³C-NMR (100 MHz, CDCl₃): δ 10.2, 21.1, 50.9, 51.6, 61.4, 68.3, 126.4, 127.6, 129.2, 130.2, 137.5, 138.9, 139.8, 144.5, 210.0.

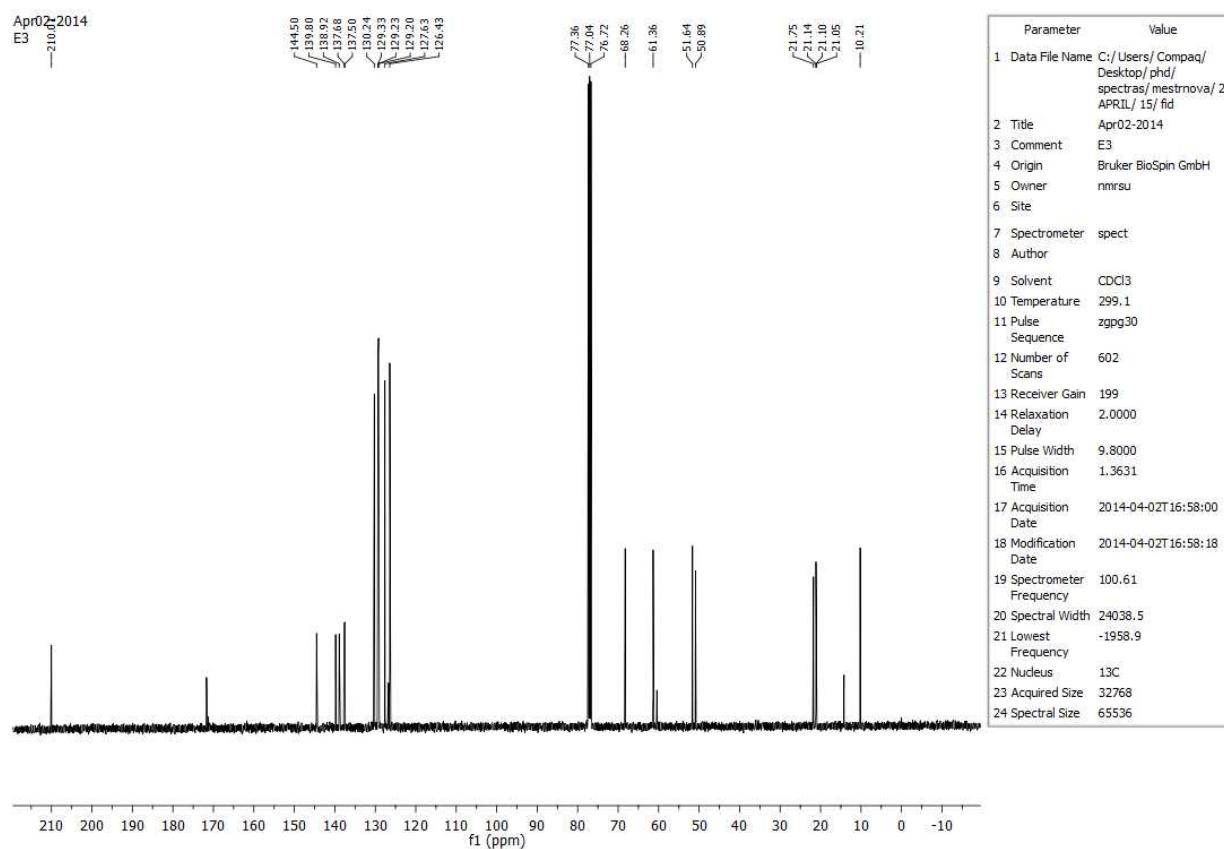


Fig. S11 ¹³C-NMR of 3-Methyl-2,6-di-p-tolylpiperidin-4-one (1d)

Spectral Data of some selected 3,4-dihydropyrimidinones

5-(Ethoxycarbonyl)-6-methyl-4-phenyl-3, 4-dihydropyrimidin-2(1H)-one (4a)

¹H-NMR (400 MHz, DMSO-d₆): δ 1.08-1.11 (t, 3H, -OCH₂CH₃), 2.25 (s, 3H, -CH₃), 3.96-4.01 (q, 2H -OCH₂CH₃), 5.16 (d, 1H, -CH), 7.23-7.34 (m, 5H, Ar-H), 7.74 (bs, 1H, -NH), 9.19 (bs, 1H, -NH).

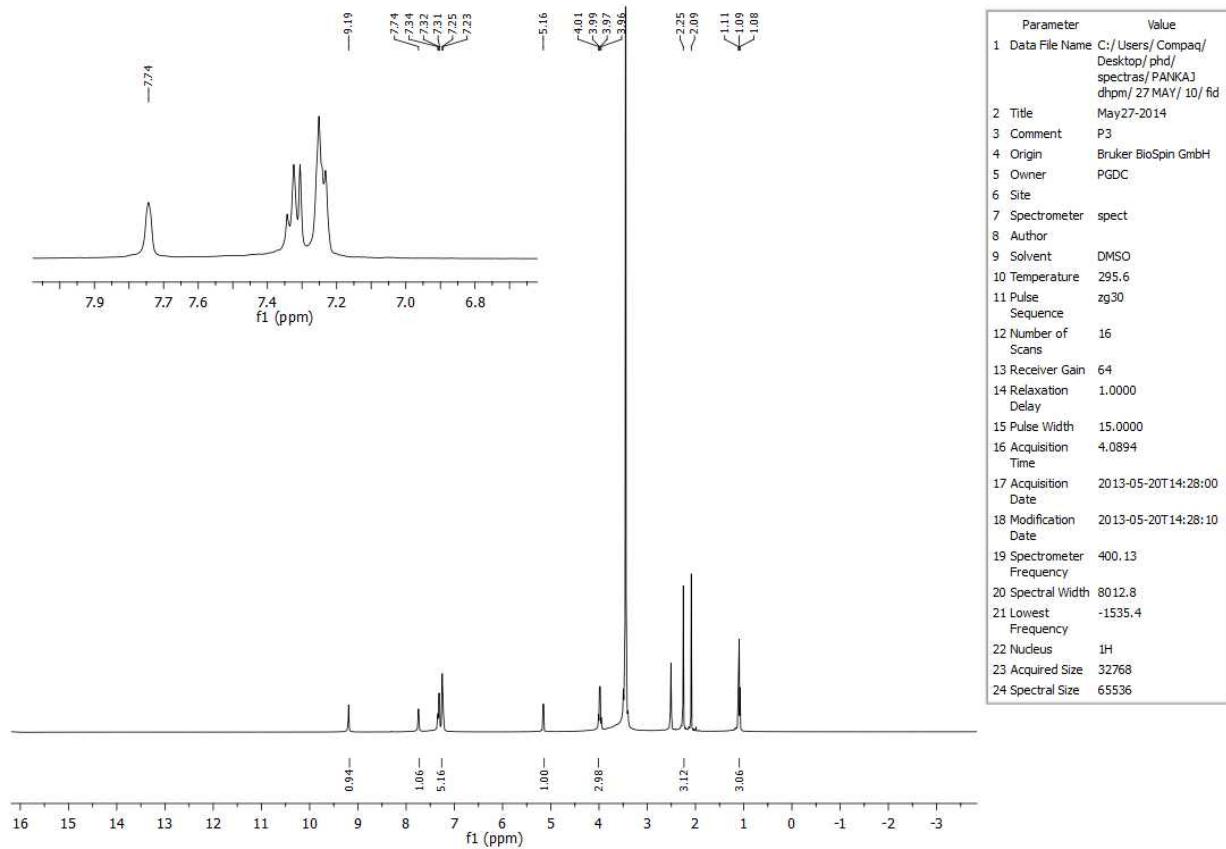


Fig. S12 ¹H NMR spectra of 5-(Ethoxycarbonyl)-6-methyl-4-phenyl-3, 4-dihydropyrimidin-2(1H)-one (4a)

¹³C-NMR (100 MHz, DMSO-d₆): δ 14.5, 18.2, 54.4, 59.7, 99.8, 126.7, 127.8, 128.9, 145.3, 148.8, 152.6, 165.8.

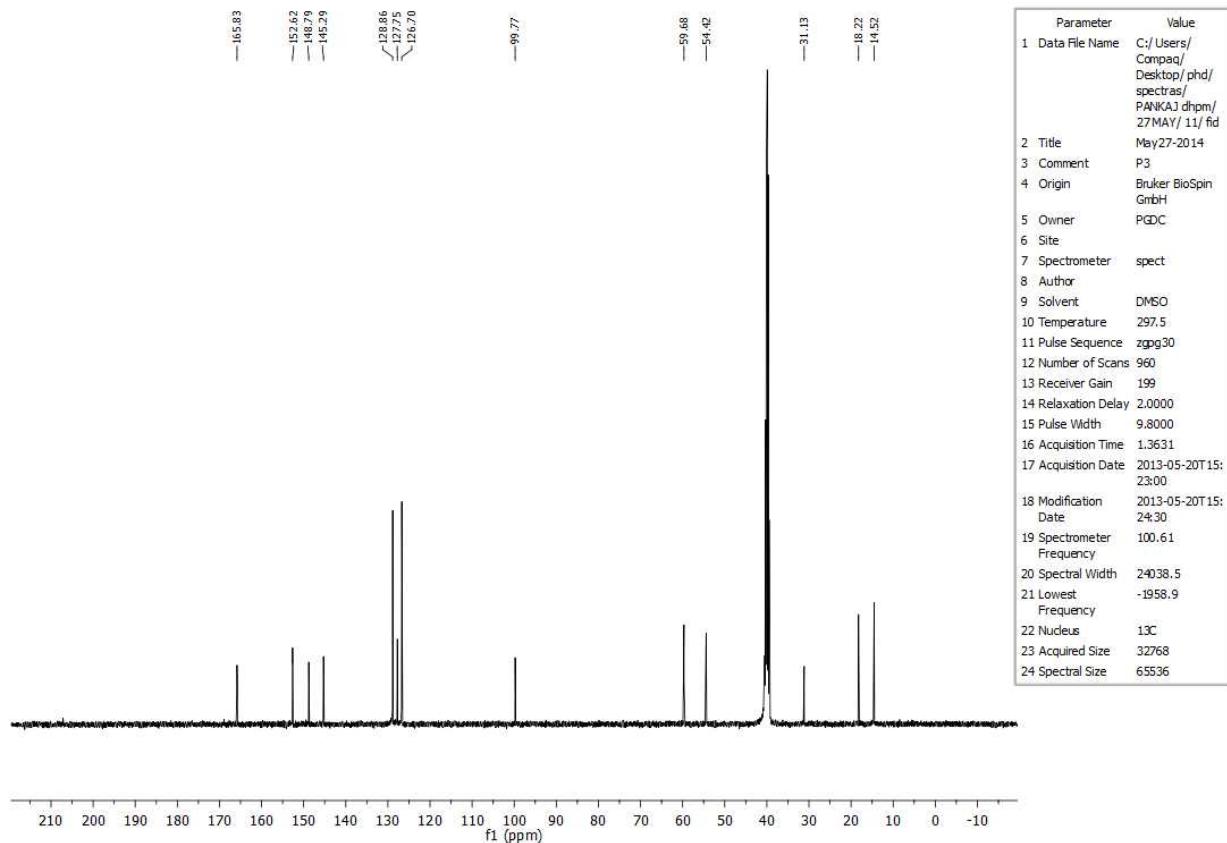


Fig. S13 ¹³C NMR spectra of 5-(Ethoxycarbonyl)-6-methyl-4-phenyl-3, 4-dihydropyrimidin-2(1H)-one (4a)

MS (ESI): 261 (M+1).

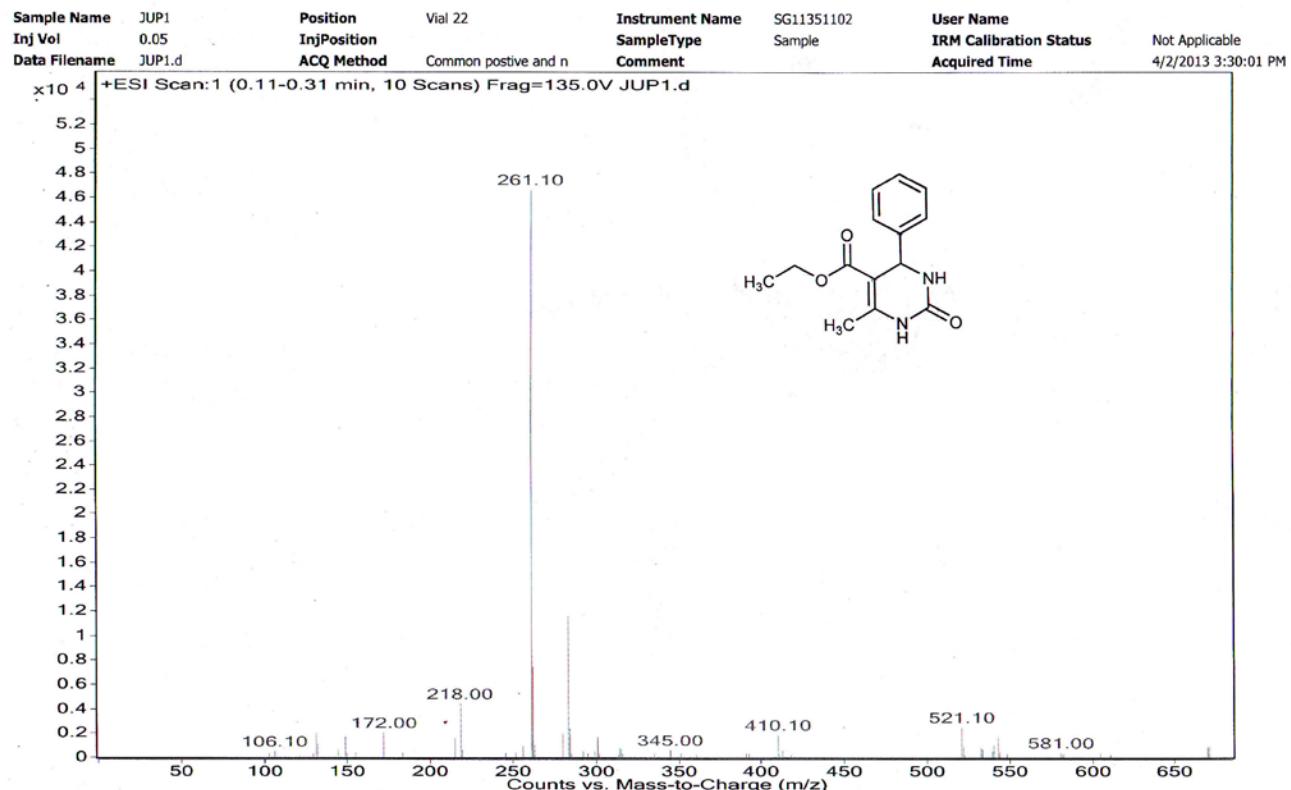


Fig. S14 Mass spectra of 5-(Ethoxycarbonyl)-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1H)-one (4a)

5-(Ethoxycarbonyl)-4-(4-methoxyphenyl)-6-methyl-3, 4-dihydropyrimidin-2-(1H)-one (4b)

$^1\text{H-NMR}$ (DMSO- d_6): δ 1.09-1.12 (t, 3H, -OCH₂CH₃), 2.24 (s, 3H, -CH₃), 3.37 (s, 3H, -OCH₃), 3.97-3.98 (q, 2H, -OCH₂CH₃), 5.09 (d, 1H, -CH), 6.87 (d, 2H, Ar-H), 7.13 (d, 2H, Ar-H), 7.68 (bs, 1H, -NH), 9.16 (bs, 1H, -NH).

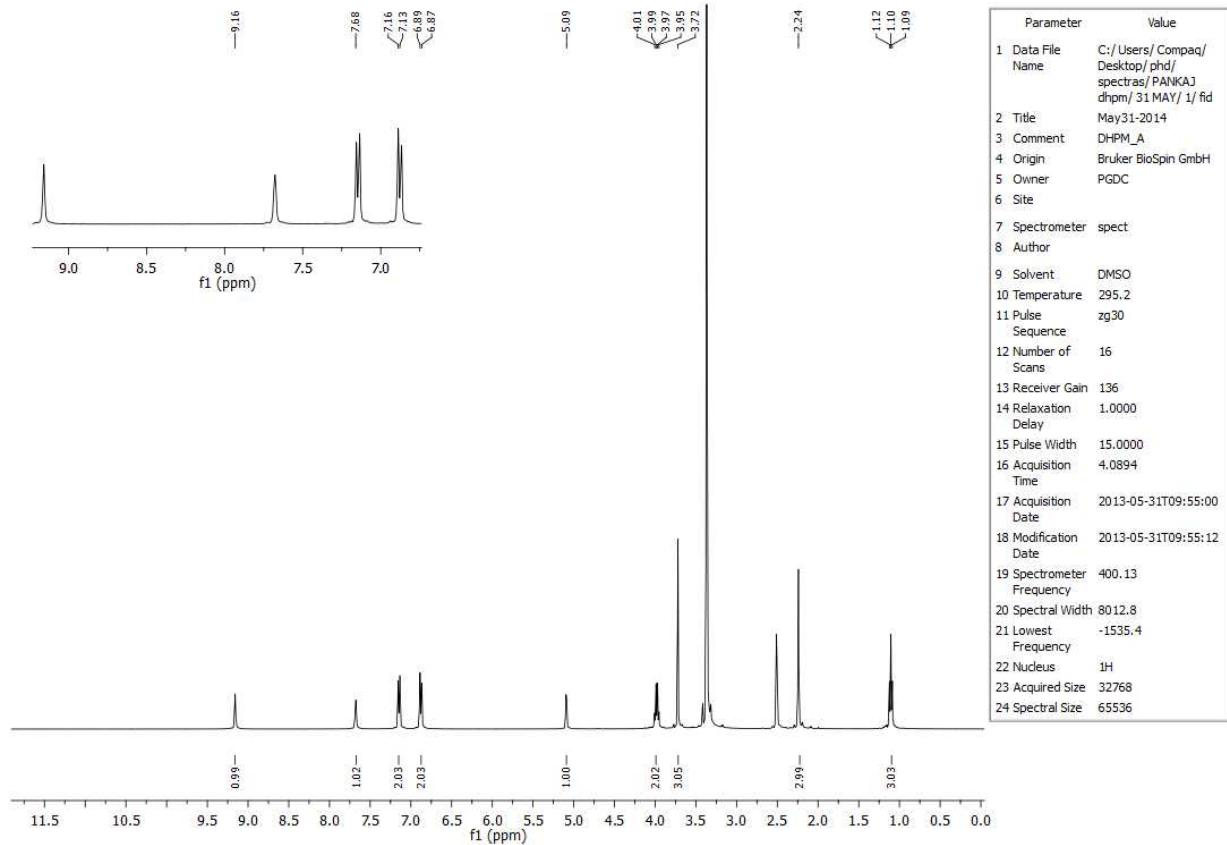


Fig. S15 ^1H NMR spectra 5-(Ethoxycarbonyl)-4-(4-methoxyphenyl)-6-methyl-3, 4-dihydropyrimidin-2-(1H)-one (4b)

¹³C-NMR (DMSO-d₆): δ 14.6, 18.2, 53.8, 55.5, 59.6, 100.0, 114.2, 127.9, 137.5, 148.5, 152.6, 158.9, 165.8.

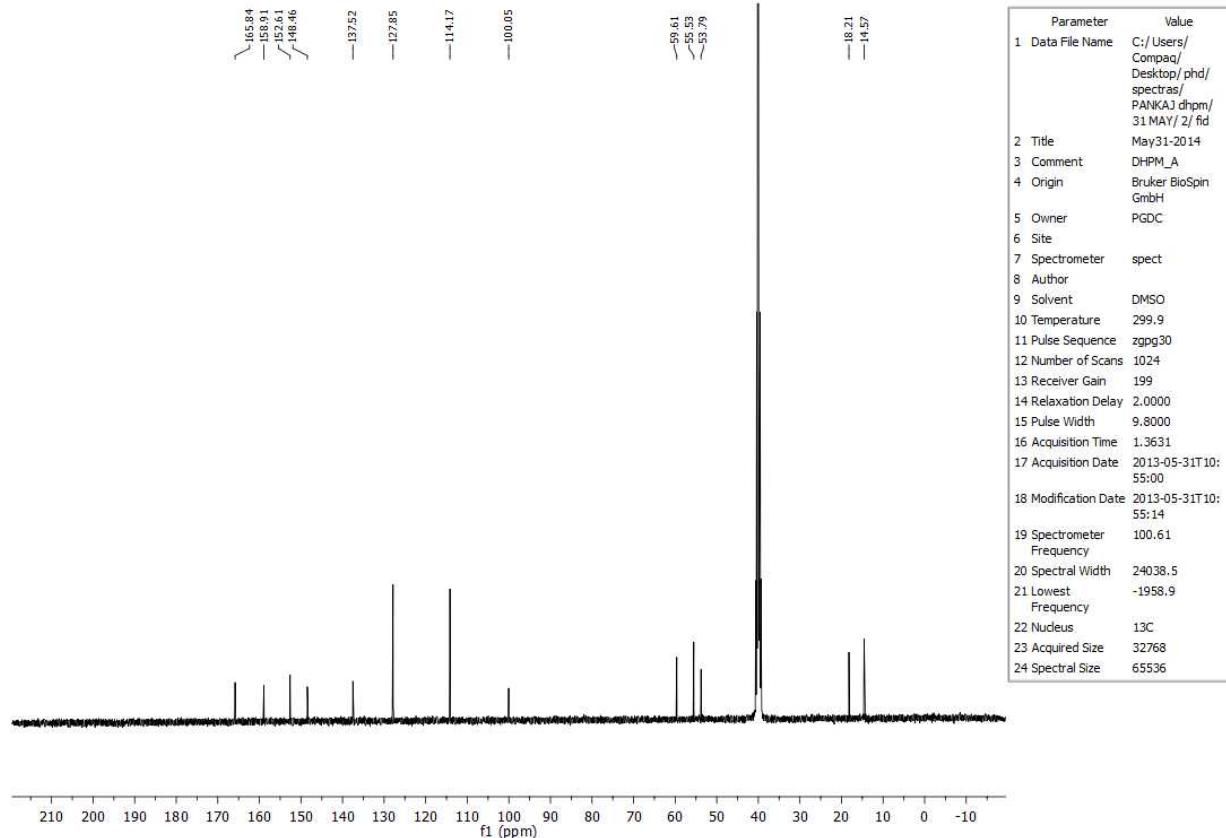


Fig. S16 ¹³C NMR spectra 5-(Ethoxycarbonyl)-4-(4-methoxyphenyl)-6-methyl-3,4-dihydropyrimidin-2-(1H)-one (4b)

5-(Ethoxycarbonyl)-4-(3-nitrophenyl)-6-methyl-3, 4-dihydropyrimidin-2(1H)-one (4e)

¹H-NMR (400 MHz, DMSO-d₆): δ 1.08-1.12 (t, 3H, -OCH₂CH₃), 2.29 (s, 3H, -CH₃), 3.96-4.02 (q, 2H, -OCH₂CH₃), 5.26 (d, 1H, -CH), 7.50-7.52 (m, 2H, Ar-H), 8.21-8.24 (m, 2H, Ar-H), 7.91 (bs, 1H, -NH), 9.36 (bs, 1H, -NH).

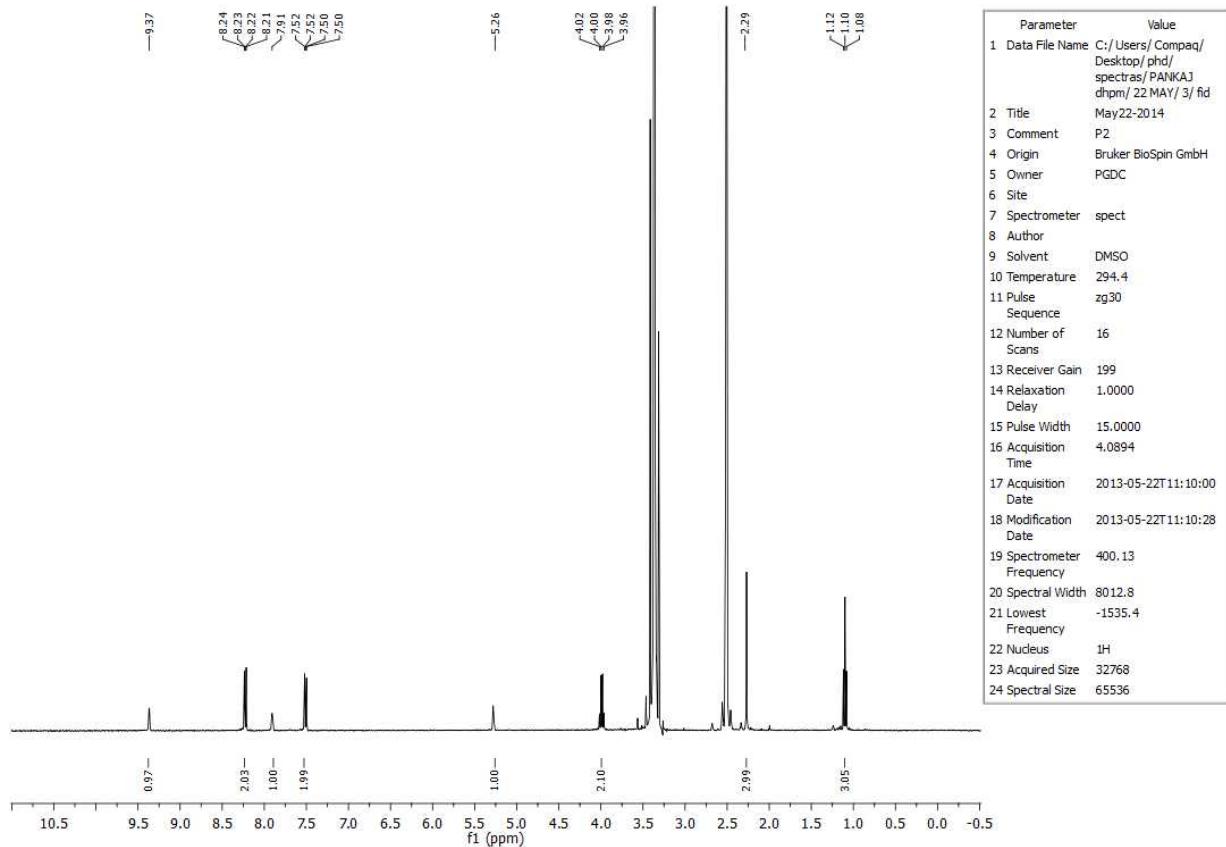


Fig. S17 ¹H NMR spectra 5-(Ethoxycarbonyl)-4-(3-nitrophenyl)-6-methyl-3, 4-dihydropyrimidin-2(1H)-one (4e)

5-(Ethoxycarbonyl)-6-methyl-4-styryl-3, 4-dihydropyrimidin-2(1H)-one (4j)

¹H-NMR (400 MHz, DMSO-d₆): δ 1.18-1.22 (t, 3H, -OCH₂CH₃), 2.20 (s, 3H, -CH₃), 4.06-4.11 (q, 2H, -OCH₂CH₃), 4.74 (d, 1H, -CH), 6.16-6.22 (dd, 1H, -CH=C-H), 6.34-6.38 (d, 1H, H-C=CH) 7.23-7.41 (m, 5H, Ar-H), 7.55 (bs, 1H, -NH), 9.14 (bs, 1H, -NH).

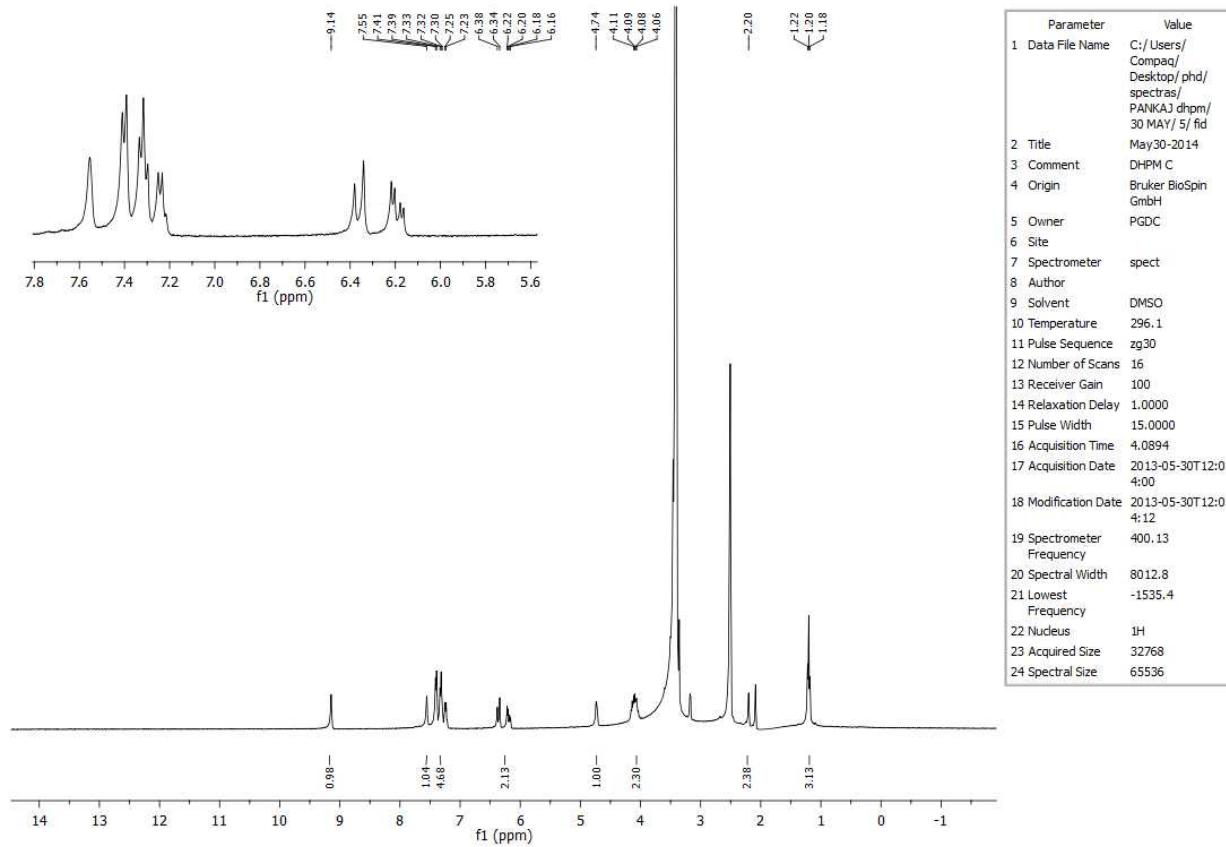


Fig. S18 ¹H NMR spectra 5-(Ethoxycarbonyl)-6-methyl-4-styryl-3, 4-dihydropyrimidin-2(1H)-one (4j)

¹³C-NMR (100 MHz, DMSO-d₆): δ 14.7, 18.2, 52.3, 59.7, 98.3, 126.8, 128.0, 128.6, 129.1, 130.4, 136.3, 149.0, 153.0, 165.7.

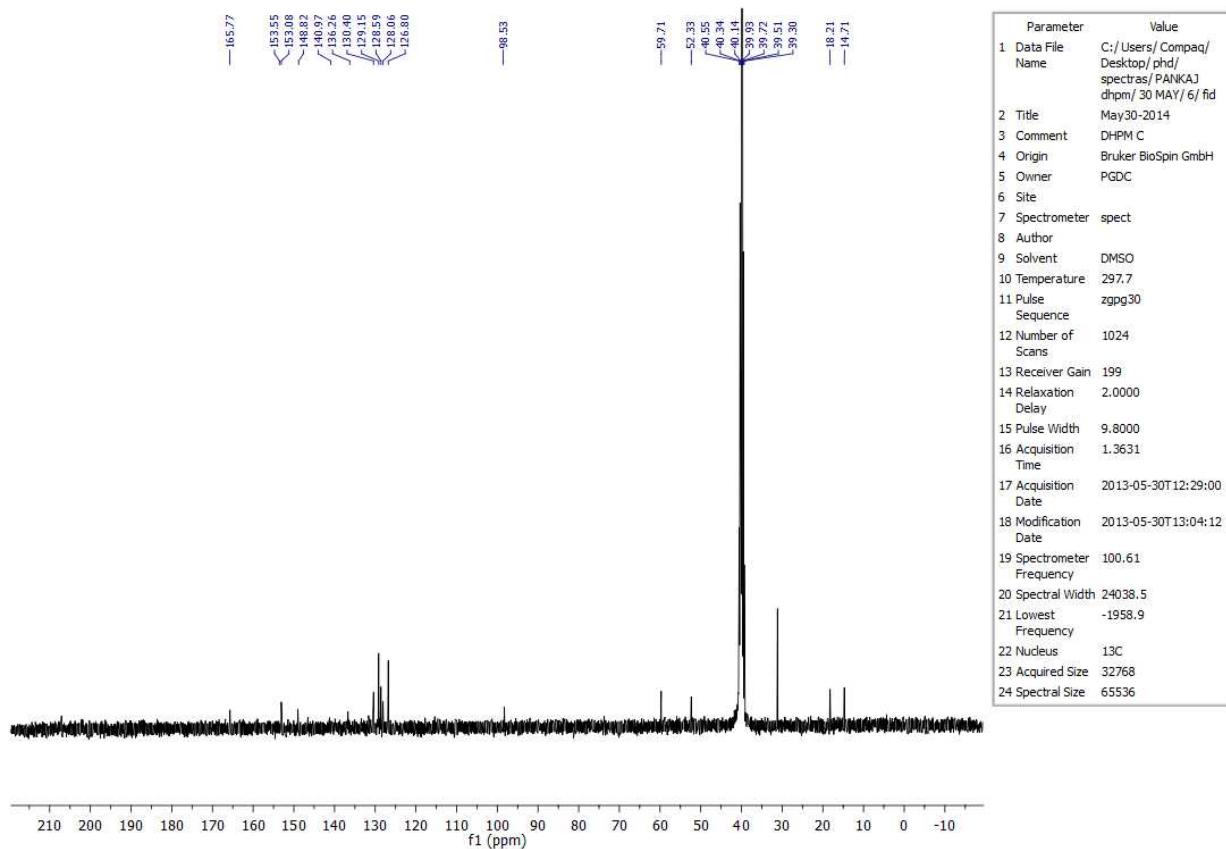


Fig. S19 ¹³C NMR spectra 5-(Ethoxycarbonyl)-6-methyl-4-styryl-3, 4-dihdropyrimidin-2(1H)-one (4j)

5-(Ethoxycarbonyl)-6-methyl-3, 4-dihydropyrimidin-2-(1H)-one (4l)

¹H-NMR (400 MHz, DMSO-d₆): δ 1.17-1.19 (t, 3H, -OCH₂CH₃), 2.15 (s, 3H, -CH₃), 3.88(s, 1H, -CH), 4.32-4.08 (q, 2H, -OCH₂CH₃), 7.03 (bs, 1H, -NH), 8.86 (bs, 1H, -NH).

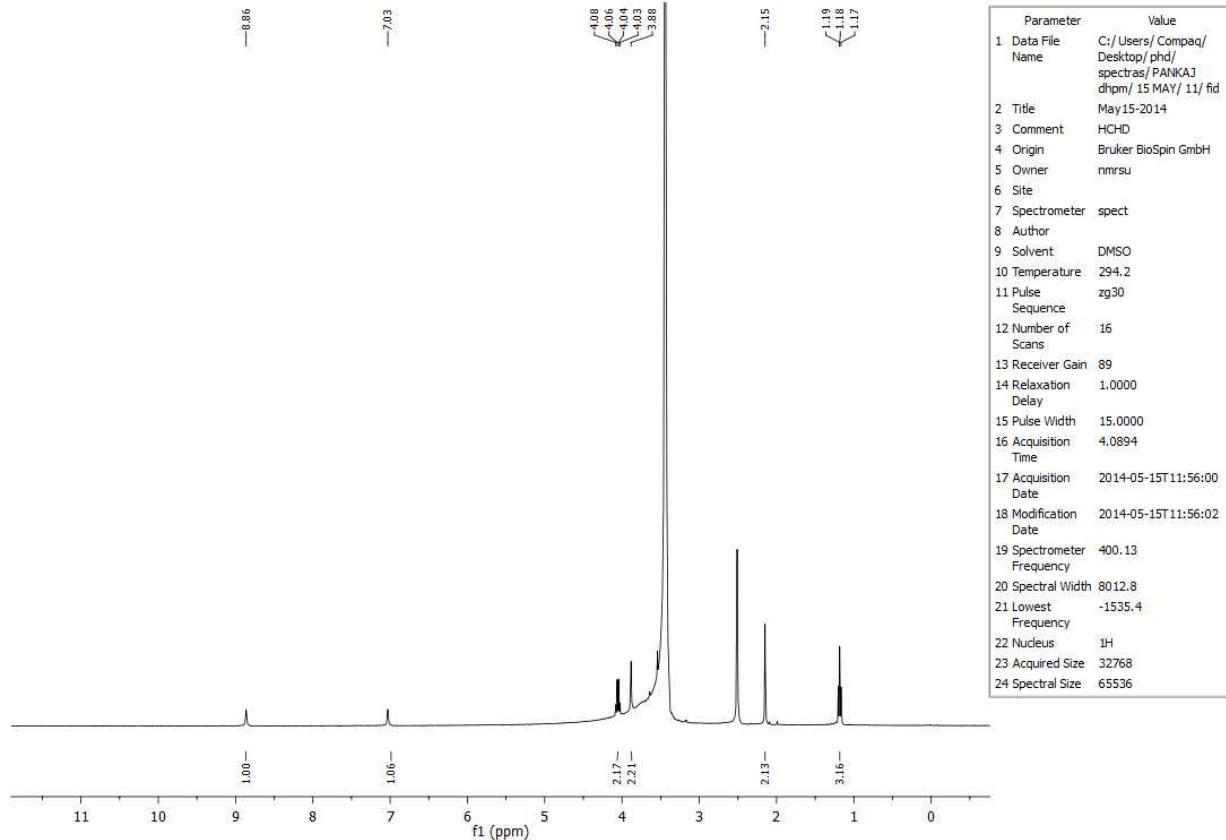


Fig. S20 ¹H NMR spectra of 5-(Ethoxycarbonyl)-6-methyl-3, 4-dihydropyrimidin-2-(1H)-one (4l)

¹³C-NMR (100 MHz, DMSO-d₆): δ 14.6, 17.8, 36.7, 59.7, 95.4, 148.9, 153.4, 165.9.

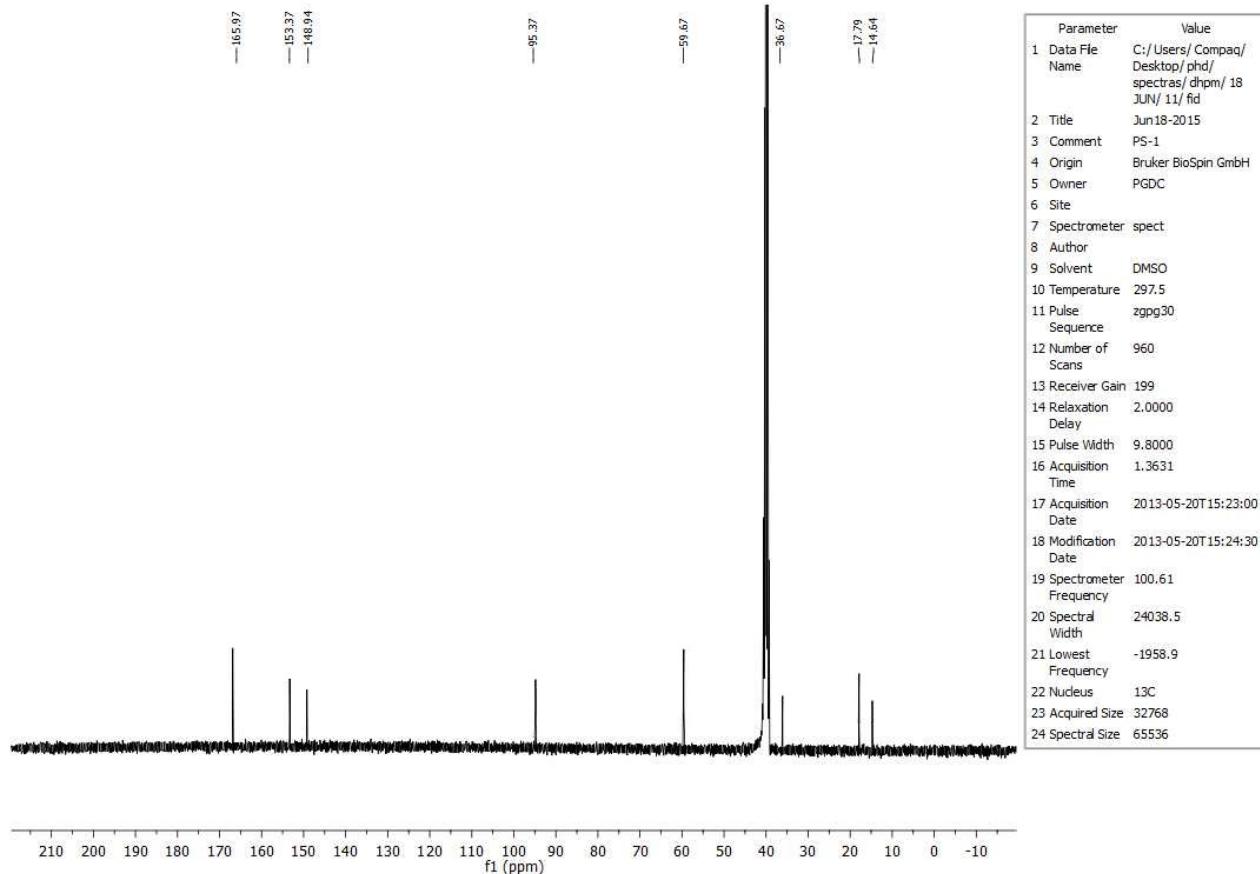


Fig. S21 ¹³C NMR spectra of 5-(Ethoxycarbonyl)-6-methyl-3, 4-dihydropyrimidin-2-(1H)-one (4l)