

Supporting information

A novel, rapid and green method of phosphorylation under ultrasound irradiation and catalyst free conditions

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1. General	2
2. Typical experimental procedure for the phosphorylation	2
<i>Scheme 1</i> .phosphorylation of various structurally N-acylamines.....	3
<i>Scheme 2</i> .phosphorylation of various structurally N-acylaminoesters.....	3
<i>Scheme 3</i> .phosphorylation of various structurally N-acylaminoalcohols.....	3
<i>Scheme 4</i> : phosphorylation of various structurally N-acylsulfonamides.....	3
 3. Spectral Data	 4
 ¹ H NMR spectrum:Dimethyl (2-oxo-2-(phenylamino)ethyl)phosphonate.....	 9
¹³ CNMRspectrum: Dimethyl (2-oxo-2-(phenylamino)ethyl)phosphonate.....	9
¹ H NMR spectrum:Dimethyl (2-(benzylamino)-2-oxoethyl)phosphonate.....	10
¹³ CNMRspectrum:Dimethyl (2-(benzylamino)-2-oxoethyl)phosphonate.....	10
IR spectrum: Dimethyl (2-(benzylamino)-2-oxoethyl)phosphonate.....	11
¹ H NMR spectrum: (S)-Methyl 2-(2-(diethoxyphosphoryl)acetamido)-4-methylpentanoate...	11
¹³ C NMR spectrum:(S)-Methyl 2-(2-(diethoxyphosphoryl)acetamido)-4-methylpentanoate....	12
³¹ P NMR spectrum: (S)-Methyl 2-(2-(diethoxyphosphoryl)acetamido)-4-methylpentanoate..	12
¹ H NMR spectrum: (S)-Diethyl (2-((1-hydroxy-3-methylbutan-2-yl)amino)-2-oxoethyl)phosphonate.....	13
¹³ C NMR spectrum:(S)-Diethyl (2-((1-hydroxy-3-methylbutan-2-yl)amino)-2-oxoethyl)phosphonate.....	13
³¹ P NMR spectrum:(S)-Diethyl (2-((1-hydroxy-3-methylbutan-2-yl)amino)-2-oxoethyl)phosphonate.....	14
¹ H NMR spectrum: N-phenyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide.....	14
¹ H NMR spectrum: N-3-fluorophenyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide...	15
¹ H NMR spectrum:N-4-methoxyphenyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide	15

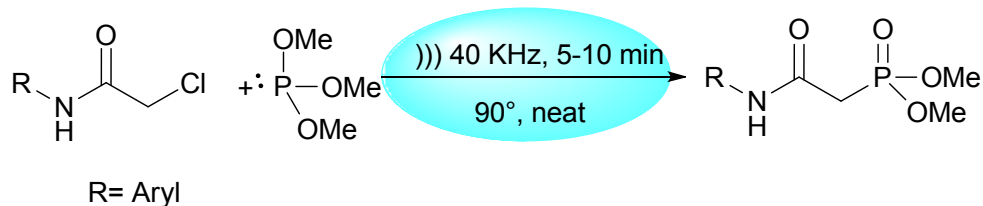
1. General:

All chemicals and solvents were purchased from common commercial sources and were used as received without any further purification. All reactions were monitored by TLC on silica Merck 60 F₂₅₄ percolated aluminum plates and were developed by spraying with ninhydrin solution. Column chromatography was performed with Merck silica gel (230-400 mesh). Proton nuclear magnetic resonance (¹H NMR) spectra were recorded on a Brücker spectrometer at 250, 300 or 400 MHz. Chemical shifts are reported in δ units (ppm) with TMS as reference (δ 0.00). All coupling constants (*J*) are reported in Hertz. Multiplicity is indicated by one or more of the following: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). Carbon nuclear magnetic resonance (¹³C NMR) spectra were recorded on a Brücker at 60, 75 or 100 MHz. Chemical shifts are reported in δ units (ppm) relative to CDCl₃ (δ 77.0). Phosphorus nuclear magnetic resonance (³¹P NMR) spectra were recorded on a Brücker at 160 MHz. Chemical shifts are reported in δ units (ppm) relative to CDCl₃ (δ 00.0). Infrared spectra were recorded on a SHIMADZU FT-IR 8000 spectrometer. Elemental analyses were recorded on a EURO E.A 3700. Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes. Ultrasound assisted reactions were carried out using a FUNGILAB ultrasonic bath with a frequency of 40 kHz and a nominal power of 250 W. The reactions were carried out in an open glass tube (diameter: 25 mm; thickness: 1 mm; volume: 20 mL) at 90°.

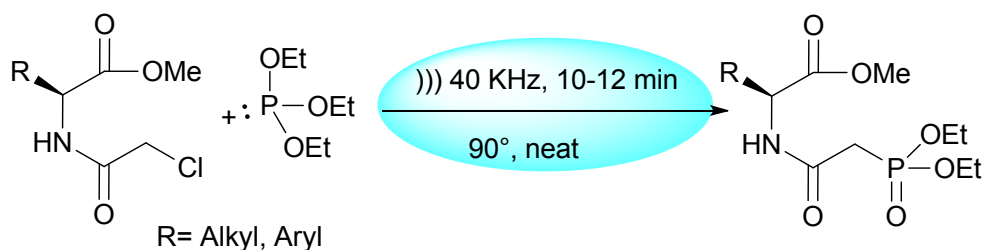
2. Typical experimental procedure for the phosphorylation:

In a 10 ml round bottom flask taken *N*-acylsulfonamide (1 mmol) and triethylphosphite or trimethylphosphite (1 mmol) was added. Then reaction mixture was subjected to the ultrasonication for appropriate time. After completion of the reaction, as indicated by TLC, silica gel; dichloromethane:methanol (9,5:0.5).

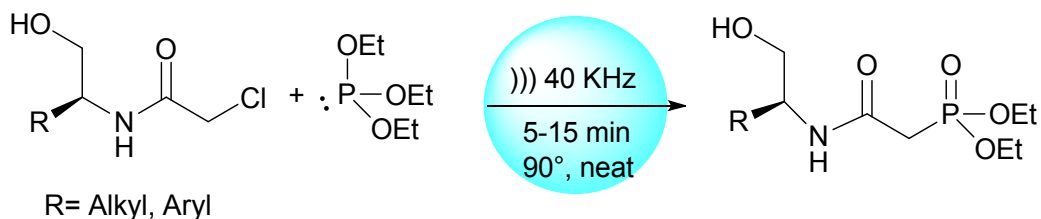
Surplus reactants were removed by column chromatography eluted with dichloromethane



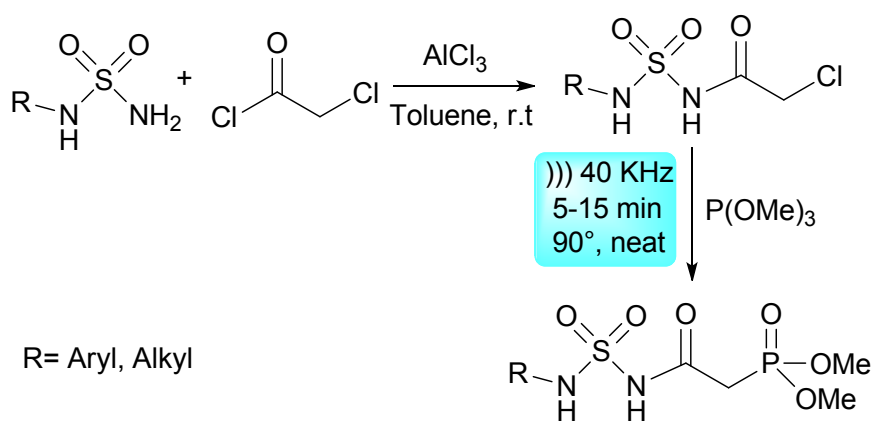
Scheme 1: Ultrasound assisted phosphorylation of various structurally *N*-acylamines.



Scheme 2: Ultrasound assisted phosphorylation of various structurally *N*-acylaminoesters.

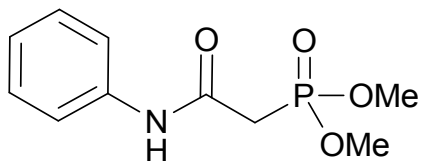


Scheme 3: Ultrasound assisted phosphorylation of various structurally *N*-acylaminoalcohols.



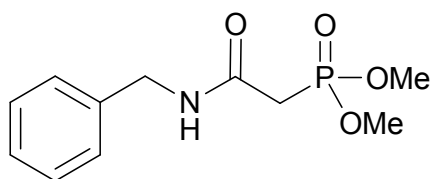
Scheme 4: Ultrasound assisted phosphorylation of various structurally *N*-acylsulfonamides.

3. Spectral data:



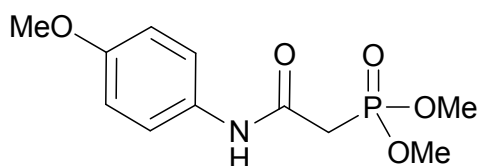
Dimethyl (2-oxo-2-(phenylamino)ethyl)phosphonate (Table 1, Entry 1a)

Yellow oil. Yield 90%. $R_f = 0.42$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$:95/05). ν_{max} (KBr)/ cm^{-1} 3280.62, 1674.25, 1510.15, 1319.00, 1231.25, 1039.67. δ_p (100 CDCl_3) 27.4. δ_H (250 MHz, CDCl_3) 2.85 (s, 1H, $\text{CH}_2\text{-CO}$), 2.95 (s, 1H, $\text{CH}_2\text{-CO}$), 3.72 (s, 3H, $\text{CH}_3\text{-O}$), 3.78 (s, 3H, $\text{CH}_3\text{-O}$), 4.44 (s, 1H, NH), 7.30 (m, 5H, H-Ar). δ_C (62 MHz, CDCl_3) 33,34, 42.7, 52.1, 126.9, 127.1, 136.2, 162.4. Anal. Calc. for $\text{C}_{10}\text{H}_{15}\text{NO}_4\text{P}$: C 49.39, H 5.80, N 5.76. Found: C 49.35, H 5.85, N 5.75%. $M=243$.



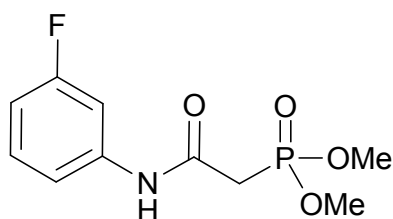
Dimethyl (2-(benzylamino)-2-oxoethyl)phosphonate (Table 1, Entry 2a)

Yellow oil. Yield 89%. $R_f = 0.40$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}$:95/05). ν_{max} (KBr)/ cm^{-1} 3284.73, 1660, 1555.42, 1314.19, 1247.25, 1034.23. δ_p (100 CDCl_3) 27.6. δ_H (250 MHz, CDCl_3) 1.45 (s, 1H, $\text{CH}_2\text{-NH}$), 1.54 (s, 1H, $\text{CH}_2\text{-NH}$), 3.08 (s, 1H, $\text{CH}_2\text{-CO}$), 3.13 (s, 1H, $\text{CH}_2\text{-CO}$), 3.85 (2d, 6H, J_1 11.02, J_2 11.20, 2 $\text{CH}_3\text{-O}$), 7.10 (m, 1H, H-Ar), 7.31 (t, 2H, J 7.02 H-Ar), 7.54 (d, 2H, J 8.32, H-Ar), 9.32 (s, 1H, NH-CO). δ_C (62 MHz, CDCl_3) 34.35, 36.42, 53.55, 53.65, 120.49, 124.63, 129.05, 133.3, 162.2. Anal. Calc. for $\text{C}_{11}\text{H}_{16}\text{NO}_4\text{P}$: C 51.36, H 6.27, N 5.45. Found: C 51.35, H 6.30, N 5.48%. $M=257$.



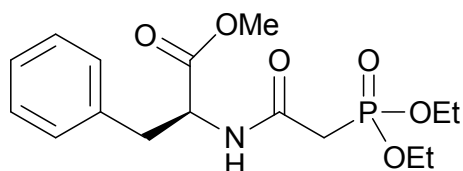
Dimethyl (2-((4-methoxyphenyl)amino)-2-oxoethyl)phosphonate (Table 1, Entry 3a)

Yellow oil. Yield 84%. $R_f = 0.48$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). ν_{max} (KBr)/ cm^{-1} 3274.05, 1684.16, 1498, 1234.15, 1046. δ_{P} (100 CDCl_3) 27.4. δ_{H} (250 MHz, CDCl_3) 2.85 (s, 1H, $\text{CH}_2\text{-CO}$), 2.97 (s, 1H, $\text{CH}_2\text{-CO}$), 3.64 (s, 3H, $\text{CH}_3\text{-O}$), 3.68 (s, 3H, $\text{CH}_3\text{-O}$), 3.74 (s, 3H, $\text{CH}_3\text{-O}$), 5.20 (s, 1H, NH), 7.21(m, 5H, H-Ar). δ_{C} (62 MHz, CDCl_3) 33, 35, 39, 44.16, 56.06, 125.4, 126.8, 137.3, 139.1, 164.2. δ_{C} (62 MHz, CDCl_3) 41, 51.1, 51.7, 52.5, 120.9, 125.3, 140.4, 156.7, 164.2. Anal. Calc. for $\text{C}_{11}\text{H}_{16}\text{NO}_5\text{P}$: C 48.36, H 5.90, N 5.13. Found: C 48.29, H 5.85, N 5.15%. $M=273$.



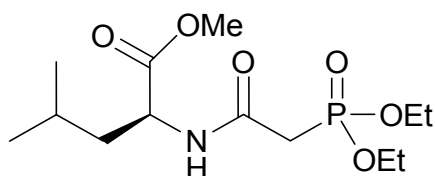
Dimethyl (2-((3-fluorophenyl)amino)-2-oxoethyl)phosphonate (Table 1, Entry 4a)

Yellow oil. Yield 86%. $R_f = 0.43$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). ν_{max} (KBr)/ cm^{-1} 3290, 1684.14, 1512.98, 1239.34, 1067.25. δ_{P} (100 CDCl_3) 27.4. δ_{H} (250 MHz, CDCl_3) 2.78 (s, 1H, $\text{CH}_2\text{-CO}$), 2.85 (s, 1H, $\text{CH}_2\text{-CO}$), 3.71 (s, 3H, $\text{CH}_3\text{-O}$), 3.79 (s, 3H, $\text{CH}_3\text{-O}$), 6.90 (m, 1H, H-Ar), 7.12-7.18 (m, 2H, H-Ar), 7.21 (m, 1H, H-Ar), 9.12 (s, 1H, NH-CO). δ_{C} (62 MHz, CDCl_3) 41.15, 41.92, 50.93, 120.16, 121.8, 123.69, 125.10, 134.41, 139.4, 161.28. Anal. Calc. for $\text{C}_{10}\text{H}_{13}\text{NO}_4\text{FP}$: C 45.99, H 5.02, N 5.36. Found: C 46.05, H 5.05, N 5.39%. $M=261$.



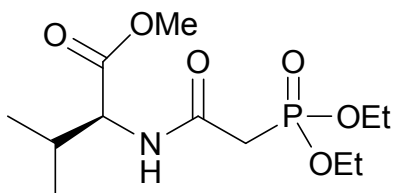
(S)-Methyl 2-(2-(diethoxyphosphoryl)acetamido)-3-phenylpropanoate (Table 2, Entry 1b)

Oil. Yield 80%. $R_f = 0.57$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). ν_{max} (KBr)/ cm^{-1} 3264, 1745, 1663, 1245, 1161. δ_{P} (160 CDCl_3) 16.4. δ_{H} (400 MHz, CDCl_3) 1.25 (2t, 6H, $J_1 7.01$, $J_2 5.30$ Hz, $2\text{CH}_3\text{-P}$), 2.8 (s, 1H, $\text{CH}_2\text{-CO}$), 2.95 (s, 1H, $\text{CH}_2\text{-CO}$), 3.05-3.25 (2dd, 2H, $J_1 7.09$, $J_2 5.45$ Hz, $\text{CH}_2\text{-Ph}$), 3.75 (s, 3H, $\text{CH}_3\text{-O}$), 4.15 (m, 4H, $\text{CH}_2\text{-O-P}$), 4.8 (m, 1H, *CH), 7.26 (d, 1H, $J 2.2$, NH). δ_{C} (100 MHz, CDCl_3) 16.29, 16.31, 34.41, 37.77, 52.35, 53.83, 62.76, 62.85, 127.19, 128.58, 129.26, 135.95, 161, 173. Anal. Calc. for $\text{C}_{16}\text{H}_{24}\text{NO}_6\text{P}$: C 53.78, H 6.77, N 3.92. Found: C 53.75, H 6.82, N 3.90%. $M=357$.



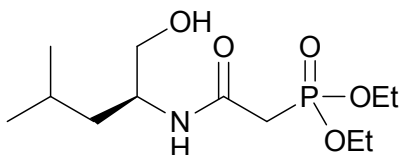
(S)-Methyl 2-(2-(diethoxyphosphoryl)acetamido)-4-methylpentanoate (Table 2, Entry 2b)

Oil. Yield 82%. $R_f = 0.6$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). $\nu_{\text{max}}(\text{KBr})/\text{cm}^{-1}$ 3274, 1743, 1674, 1245, 1161. $\delta_{\text{p}}(160 \text{ CDCl}_3)$ 16.33. δ_{H} (400 MHz, CDCl_3) 0.95 (2d, 6H, J 8.01 Hz, $2\text{CH}_3\text{-iBu}$), 1.3 (t, 6H, J 7.05 Hz, $\text{CH}_3\text{-CH}_2\text{O}$), 1.5-1.7 (m, 3H, CH-iBu , $\text{CH}_2\text{-iBu}$), 2.82 (d, 1H, $\text{CH}_2\text{-CO}$), 2.95 (s, 1H, $\text{CH}_2\text{-CO}$), 3.7 (s, 3H, $\text{CH}_3\text{-O}$), 4.1 (m, 4H, $\text{CH}_2\text{-O-P}$), 4.54 (m, 1H, $^*\text{CH}$), 7.1 (d, 1H, J 7.81, NH). δ_{C} (100 MHz, CDCl_3) 16.27, 16.31, 21.6, 22.79, 24.65, 34.23, 40.96, 51.05, 52.19, 62.5, 62.50, 163.95, 173. Anal. Calc. for $\text{C}_{13}\text{H}_{26}\text{NO}_6\text{P}$: C 41.29, H 8.11, N 4.33. Found: C 41.25, H 8.19, N 4.25%. $M=323$.



(S)-methyl 2-(2-(diethoxyphosphoryl)acetamido)-3-methylbutanoate (Table 2, Entry 3b)

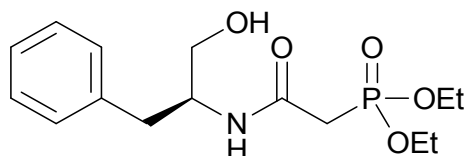
Oil. Yield 88%. $R_f = 0.67$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). $\nu_{\text{max}}(\text{KBr})/\text{cm}^{-1}$ 3280.69, 1731, 1651, 1250, 1155. $\delta_{\text{p}}(160 \text{ CDCl}_3)$ 16.31. δ_{H} (400 MHz, CDCl_3) 0.95 (2d, 6H, J 6.90 Hz, $2\text{CH}_3\text{-iPr}$), 1.4 (t, 6H, J 7.07 Hz, $\text{CH}_3\text{-CH}_2$), 2.2 (m, 1H, CH-iPr), 2.83 (s, 1H, $\text{CH}_2\text{-CO}$), 2.98 (d, 1H, $\text{CH}_2\text{-CO}$), 3.75 (s, 3H, $\text{CH}_3\text{-O}$), 4.15 (m, 4H, $2\text{CH}_2\text{-O}$), 4.5 (m, 1H, $^*\text{CH}$), 7.15 (d, 1H, J 8.1 Hz, NH). δ_{C} (100 MHz, CDCl_3) 16.21, 16.23, 18.50, 19.25, 29.71, 34.23, 50.96, 52, 62.61, 62.62, 163.8, 171.9. Anal. Calc. for $\text{C}_{12}\text{H}_{24}\text{NO}_6\text{P}$: C 46.60, H 7.82, N 4.53. Found: C 46.55, H 7.72, N 4.61%. $M=309$.



(S)-Diethyl 2-((1-hydroxy-4-methylpentan-2-yl)amino)-2-oxoethylphosphonate (Table 3, Entry 1c)

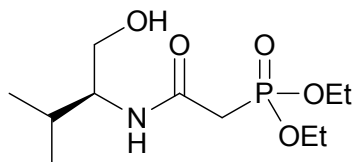
Oil. Yield 79%. $R_f = 0.62$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). $\nu_{\text{max}}(\text{KBr})/\text{cm}^{-1}$ 3490, 3180, 1650, 1230, 1112. $\delta_{\text{p}}(160 \text{ CDCl}_3)$ 16.33. δ_{H} (400 MHz, CDCl_3) 0.98 (2d, 6H, J 2.75 Hz, $2\text{CH}_3\text{-iBu}$), 1.4 (m,

8H, 2CH₃-CH₂-CH₂-iBu), 1.65 (m, 1H, CH-iBu), 2.45 (s, 1H, OH), 2.85 (s, 1H, CH₂-CO), 2.99 (s, 1H, CH₂-CO), 3.50 (dd, 1H, *J* 5.37 Hz, CH₂-OH), 3.75 (dd, 1H, *J* 3.37 Hz, CH₂-OH), 4.2 (m, 5H, 2CH₂-O, *CH), 6.8 (d, 1H, *J* 7.89 Hz, NH). δ_C (100 MHz, CDCl₃) 16.3, 16.35, 21.98, 23.12, 24.74, 35, 39.86, 50.75, 62.69, 62.74, 65.76, 164.79. Anal. Calc. for C₁₂H₂₆NO₅P: C 41.29, H 8.11, N 4.33. Found: C 41.25, H 8.19, N 4.25%. M=295.



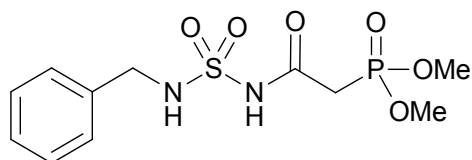
(S)-Diethyl (2-((1-hydroxy-3-phenylpropan-2-yl)amino)-2-oxoethyl)phosphonate (Table 3, Entry 2c)

Oil. Yield 75%. R_f = 0.55 (CH₂Cl₂/MeOH:95/05). $\nu_{\max}$ (KBr)/cm⁻¹ 3880, 3580, 3190, 1650, 1296, 1112. δ_P (160 CDCl₃) 16.36. δ_H (400 MHz, CDCl₃) 1.4 (t, 6H, *J* 7.18 Hz, 2CH₃-CH₂), 2.82 (m, 4H, CH₂-CO, CH₂-Ph), 3.05 (s, 1H, OH), 3.55 (dd, 1H, *J* 4.68 Hz, CH₂-OH), 3.75 (dd, 1H, *J* 3.47 Hz, CH₂-OH), 4.2 (m, 5H, CH₃-CH₂, *CH), 7.05 (d, 1H, *J* 8.07, NH), 7.3 (m, 5H, H-Ar). δ_C (100MHz, CDCl₃) 16.3, 16.34, 35.48, 38.54, 52.74, 53.80, 62.80, 127.22, 128.68, 129.55, 135.98, 164.79. Anal. Calc. for C₁₂H₂₆NO₅P: C 54.71, H 7.35, N 4.25. Found: C 54.65, H 7.30, N 4.29%. M=329.



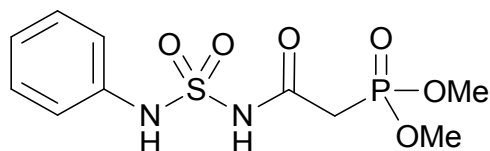
(S)-Diethyl (2-((1-hydroxy-3-methylbutan-2-yl)amino)-2-oxoethyl)phosphonate (Table 3, Entry 3c)

Oil. Yield 83%. R_f = 0.45 (CH₂Cl₂/MeOH:95/05). $\nu_{\max}$ (KBr)/cm⁻¹ 3484, 3298, 1650, 1240, 1151. δ_P (160 CDCl₃) 16.34. δ_H (400 MHz, CDCl₃) 0.94 (2d, 6H, *J* 6.56 Hz, 2CH₃-iPr), 1.05 (t, 3H, *J* 6.6 Hz, CH₃-CH₂), 1.25 (t, 3H, *J* 5.24 Hz, CH₃-CH₂), 1.95 (m, 1H, CH-iPr), 2.88 (d, 1H, CH₂-CO), 2.97 (d, 1H, CH₂-CO), 3.55-3.75 (m, 2H, CH₂-OH), 4.15 (m, 4H, 2CH₂-O), 4.16 (m, 1H, *CH), 6.80 (d, 1H, *J* 9.64 Hz, NH). δ_C (100 MHz, CDCl₃) 16.21, 16.23, 18.14, 19.12, 28.98, 34.99, 50.20, 62.22, 62.30, 64, 163.79. Anal. Calc. for C₁₁H₂₄NO₅P: C 46.97, H 8.60, N 4.98. Found: C 46.92, H 8.67, N 4.94%. M=281.



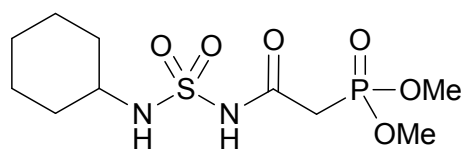
N-benzyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide (Table 4, Entry 1d)

Yellow oil. Yield 85%. $R_f = 0.42$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). ν_{max} (KBr)/ cm^{-1} 3350, 1725, 1658, 1364, 1249, 1159. δ_p (160 CDCl_3) 27.9. δ_H (400 MHz, CDCl_3) 2.95 (s, 1H, $\text{CH}_2\text{-CO}$), 3.09 (s, 1H, $\text{CH}_2\text{-CO}$), 3.75 (s, 1H, $\text{CH}_2\text{-Ar}$), 3.66 (s, 1H, $\text{CH}_2\text{-Ar}$), 3.82 (2s, 6H, 2 $\text{CH}_3\text{-O}$), 7.25 (m, 5H, H-Ar), 8.82 (s, 1H, NH-CO). δ_C (100 MHz, CDCl_3) 35, 41.2, 52, 126.6, 126.9, 128.6, 141.2, 165.1. Anal. Calc. for $\text{C}_{11}\text{H}_{17}\text{N}_2\text{O}_6\text{SP}$: C 41.25, H 5.35, N 8.75. Found: C 41.35, H 5.25, N 8.85%. $M=336$.



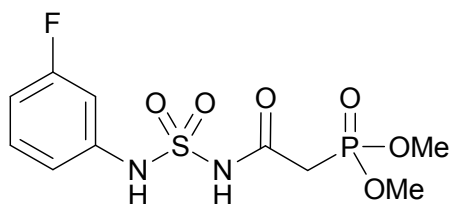
N-phenyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide (Table 4, Entry 2d)

Yellow oil. Yield 90%. $R_f = 0.44$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). ν_{max} (KBr)/ cm^{-1} 3320, 1715, 1610, 1361, 1234, 1149. δ_p (160 CDCl_3) 27.3. δ_H (400 MHz, CDCl_3) 2.97 (s, 1H, $\text{CH}_2\text{-CO}$), 3.02 (s, 1H, $\text{CH}_2\text{-CO}$), 3.82 (2s, 6H, 2 $\text{CH}_3\text{-O}$), 6.90 (m, 1H, H-Ar), 7.12-7.50 (2m, 4H, H-Ar), 9.32 (s, 1H, NH-CO). δ_C (100 MHz, CDCl_3) 36, 52, 120.9, 125.3, 129.6, 139.2, 160.4. Anal. Calc. for $\text{C}_{10}\text{H}_{15}\text{N}_2\text{O}_6\text{SP}$: C 37.26, H 4.96, N 8.69. Found: C 37.35, H 4.85, N 8.75%. $M=322$.



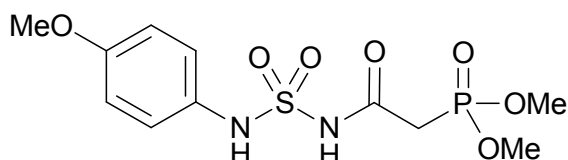
N-cyclohexyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide (Table 4, Entry 3d)

Yellow oil. Yield 80%. $R_f = 0.50$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). ν_{max} (KBr)/ cm^{-1} 3269, 1700, 1614, 1368, 1249, 1150. δ_p (100 CDCl_3) 29.8. δ_H (250 MHz, CDCl_3) 1.28 (m, 4H, 2 $\text{CH}_2\text{-cyc}$), 1.35 (m, 2H, $\text{CH}_2\text{-cyc}$), 1.55 (m, 2H, $\text{CH}_2\text{-cyc}$), 8.5 (m, 2H, $\text{CH}_2\text{-cyc}$), 2.80 (s, 1H, $\text{CH}_2\text{-CO}$), 2.91 (s, 1H, $\text{CH}_2\text{-CO}$), 3.66 (m, 1H, CH-NH), 3.91 (2s, 6H, 2 $\text{CH}_3\text{-O}$), 8.65 (s, 1H, NH-CO). δ_C (62 MHz, CDCl_3) 24, 25, 32, 42.9, 43, 52, 170. Anal. Calc. for $\text{C}_{10}\text{H}_{21}\text{N}_2\text{O}_6\text{SP}$: C 36.58, H 3.45, N 8.53. Found: C 36.65, H 3.25, N 8.85%. $M=328$.



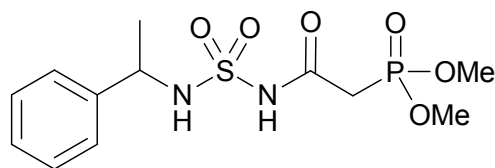
N-3-fluorophenyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide (Table 4, Entry 4d)

Yellow oil. Yield 90%. $R_f = 0.45$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). ν_{max} (KBr)/ cm^{-1} 3368, 1720, 1650, 1364, 1245, 1159. $\delta_p(100 \text{ CDCl}_3)$ 28.4. $\delta_H(250 \text{ MHz, CDCl}_3)$ 3.03 (s, 1H, $\text{CH}_2\text{-CO}$), 3.11 (s, 1H, $\text{CH}_2\text{-CO}$), 3.92 (2s, 6H, $2\text{CH}_3\text{-O}$), 6.9 (m, 1H, H-Ar), 7.35 (m, 2H, H-Ar), 7.65 (m, 1H, H-Ar), 9.02 (s, 1H, NH-CO). $\delta_C(62 \text{ MHz, CDCl}_3)$ 41.2, 52, 105.9, 110.4, 115, 129.6, 139.4, 163, 170. Anal. Calc. for $\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_6\text{FSP}$: C 35.30, H 4.15, N 8.26. Found: C 35.45, H 4.05, N 8.35%. $M=340$.



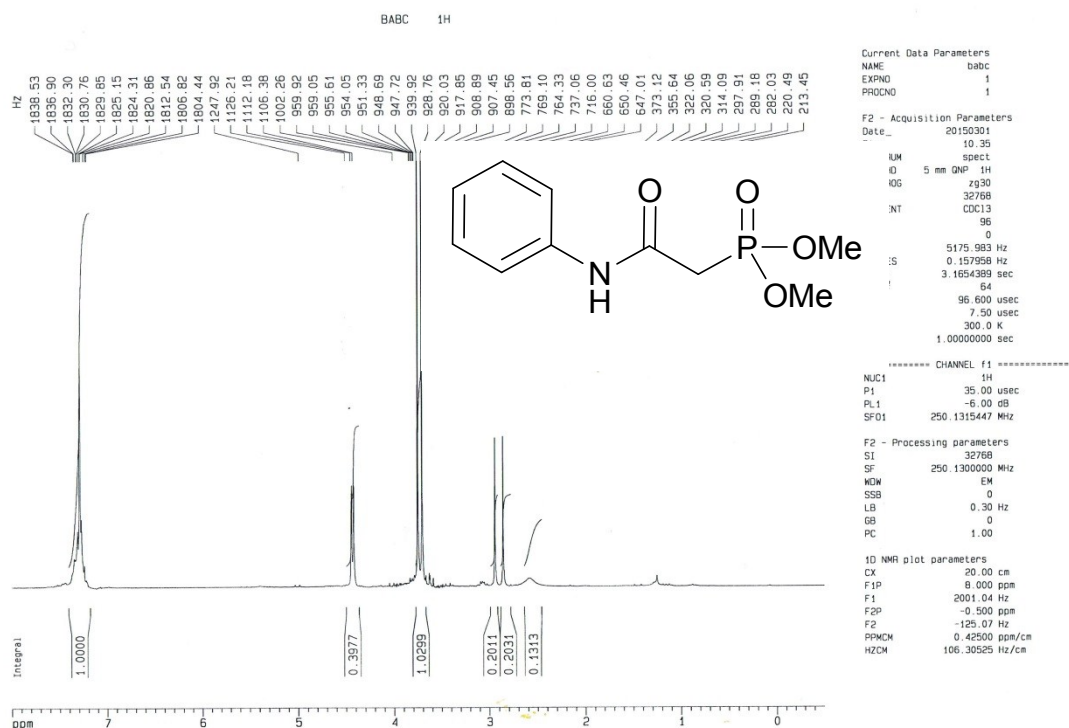
N-4-methoxyphenyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide (Table 4, Entry 5d)

Yellow oil. Yield 88%. $R_f = 0.48$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). ν_{max} (KBr)/ cm^{-1} 3310, 1714, 1590, 1364, 1260, 1151. $\delta_p(100 \text{ CDCl}_3)$ 28.1. $\delta_H(250 \text{ MHz, CDCl}_3)$ 2.94 (s, 1H, $\text{CH}_2\text{-CO}$), 2.99 (s, 1H, $\text{CH}_2\text{-CO}$), 3.90 (3s, 9H, $3\text{CH}_3\text{-O}$), 6.9-7.35 (2d, 4H, $J_1=J_2$ 7.36, H-Ar), 8.82 (s, 1H, NH-CO). $\delta_C(62 \text{ MHz, CDCl}_3)$ 41.2, 52, 105.9, 110.4, 115, 129.6, 139.4, 163, 170. Anal. Calc. for $\text{C}_{11}\text{H}_{17}\text{N}_2\text{O}_7\text{SP}$: C 37.50, H 4.85, N 7.96. Found: C 37.55, H 4.98, N 7.85%. $M=352$.

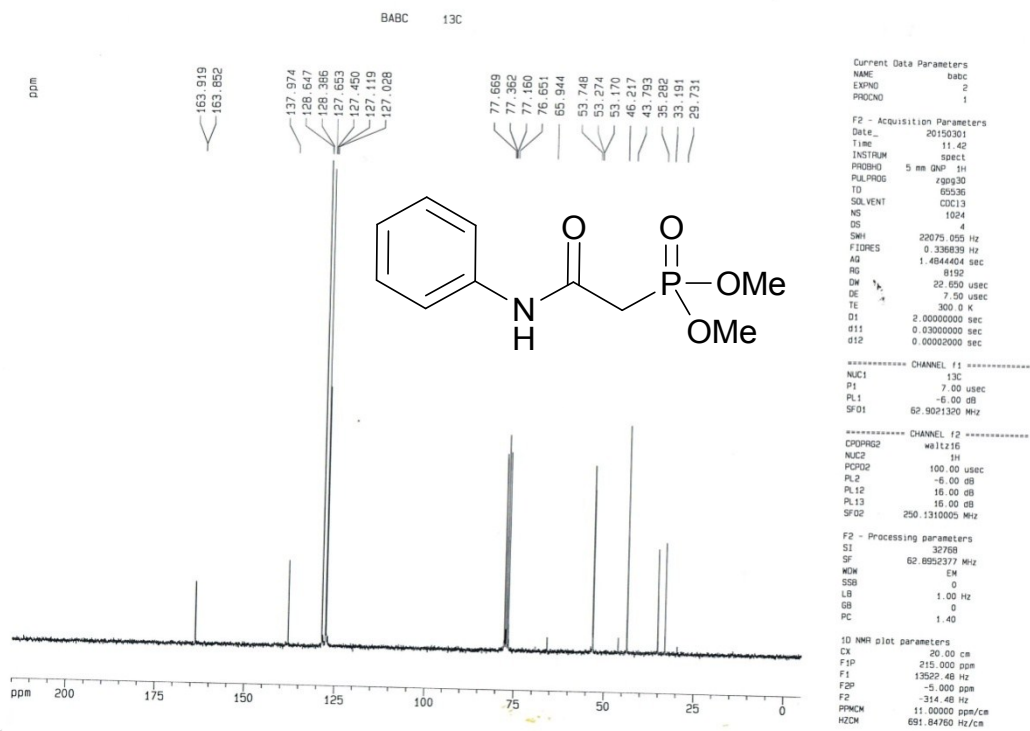


N-1-phenylethyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide (Table 4, Entry 6d)

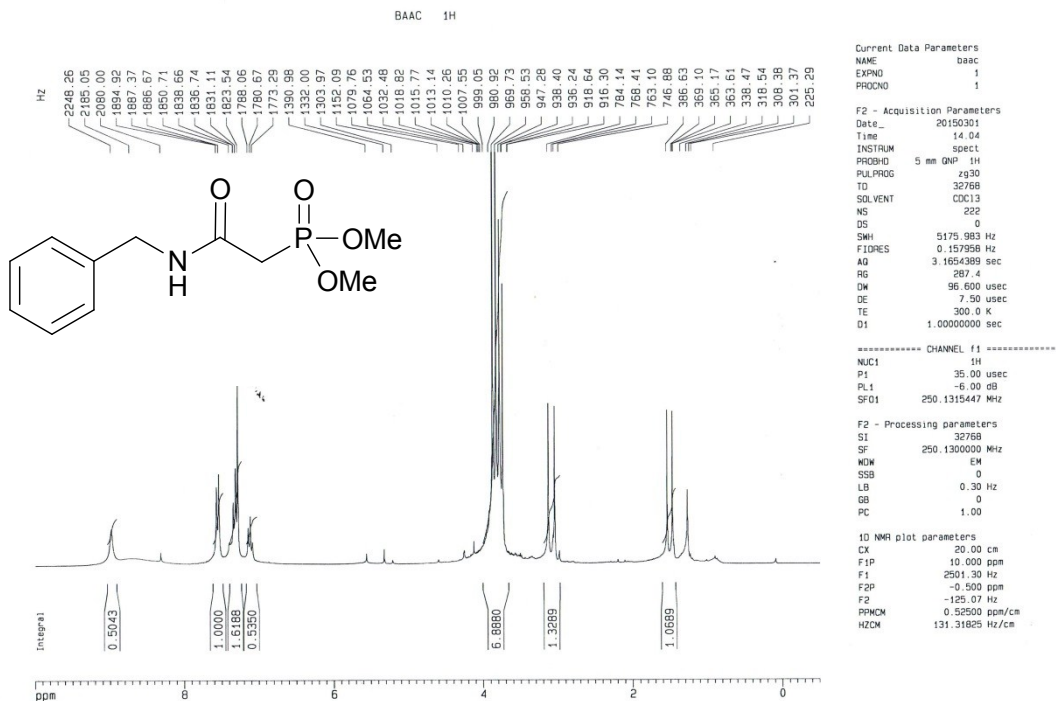
Yellow oil. Yield 75%. $R_f = 0.42$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}:95/05$). ν_{max} (KBr)/ cm^{-1} 3260, 1710, 1612, 1361, 1250, 1158. $\delta_p(160 \text{ CDCl}_3)$ 27.9. $\delta_H(400 \text{ MHz, CDCl}_3)$ 1.28 (d, 3H, J 6.93 Hz, CH_3), 2.92 (s, 1H, $\text{CH}_2\text{-CO}$), 3.01 (s, 1H, $\text{CH}_2\text{-CO}$), 3.80 (2s, 6H, $2\text{CH}_3\text{-O}$), 4.35 (m, 1H, CH^*), 5.55 (d, 1H, J 6.20 Hz, NH-CH), 7.50 (m, 5H, H-Ar), 8.62 (s, 1H, NH-CO). $\delta_C(100 \text{ MHz, CDCl}_3)$ 19, 41, 42, 46, 52, 126.5, 128.1, 143.6, 170. Anal. Calc. for $\text{C}_{12}\text{H}_{19}\text{N}_2\text{O}_6\text{SP}$: C 41.14, H 4.15, N 8.00. Found: C 41.25, H 4.19, N 8.05%. $M=350$.



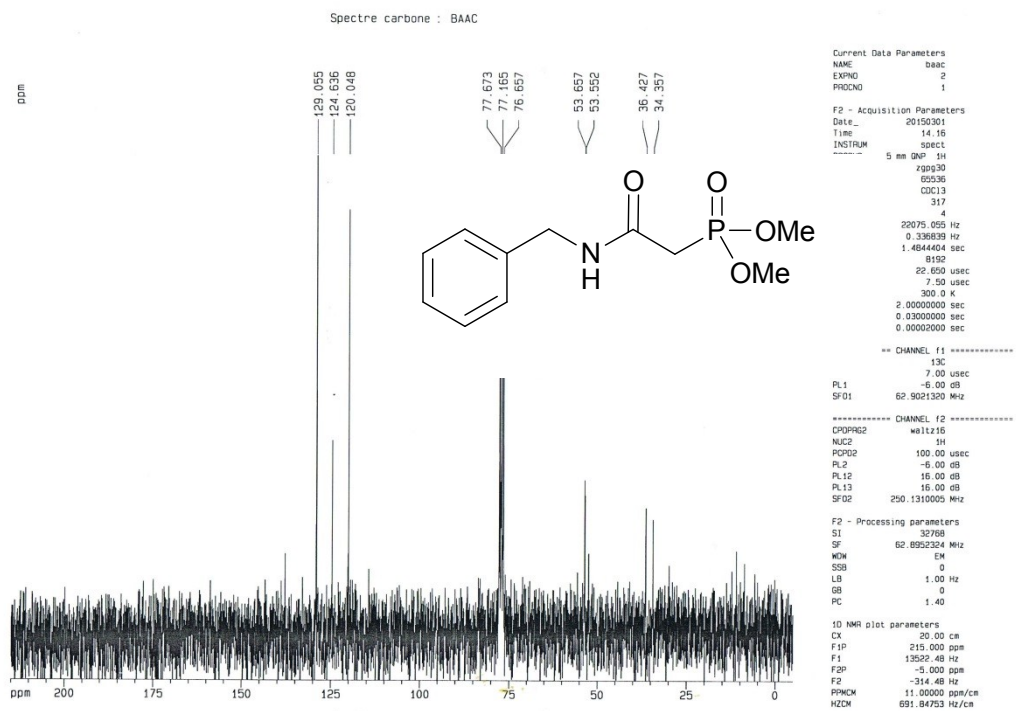
¹H NMR spectrum: Dimethyl (2-oxo-2-(phenylamino)ethyl)phosphonate



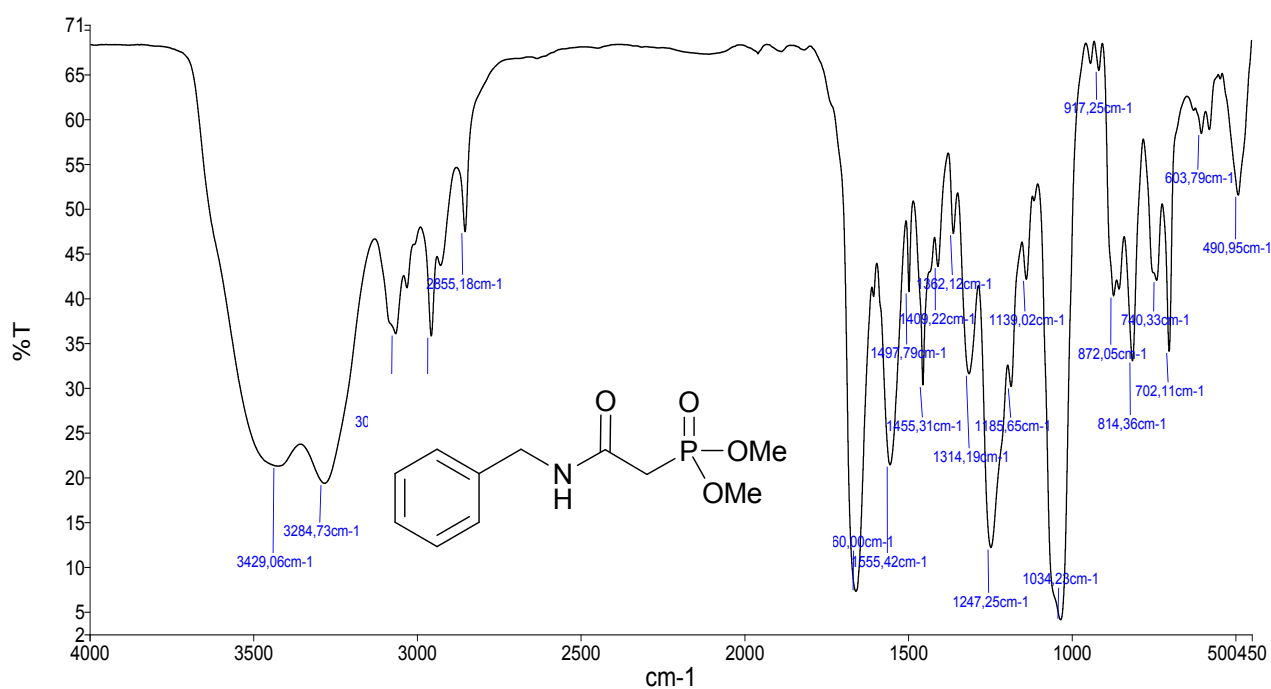
¹³C NMR spectrum: Dimethyl (2-oxo-2-(phenylamino)ethyl)phosphonate



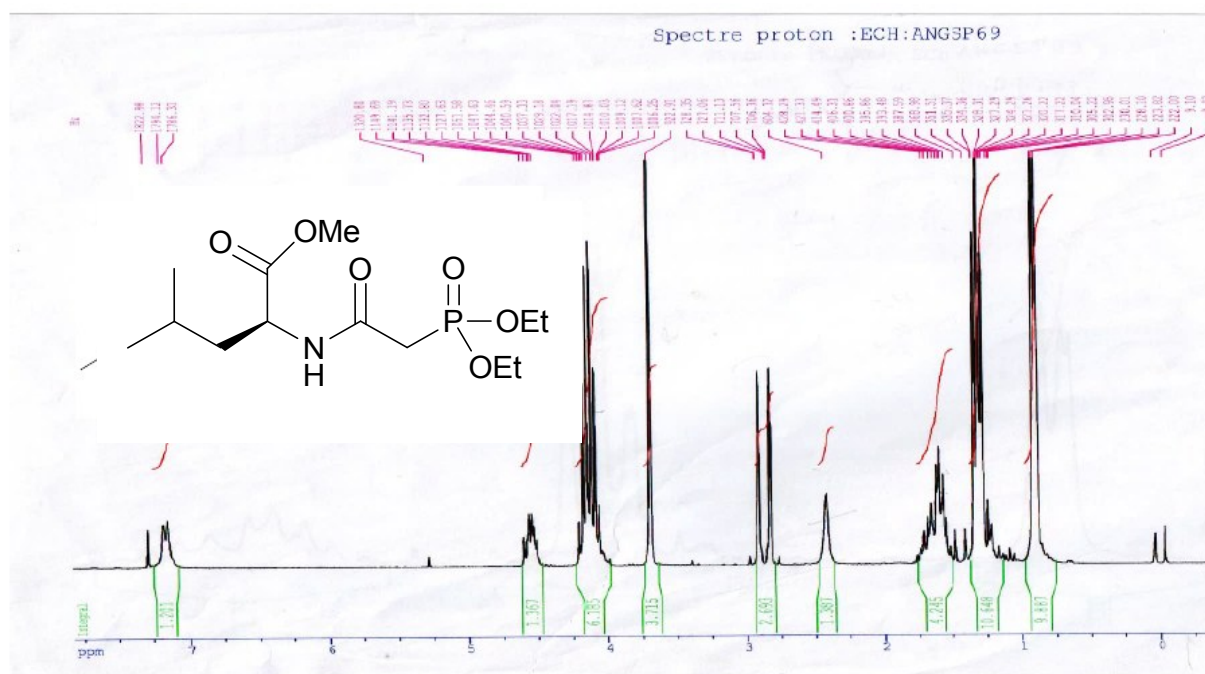
¹H NMR spectrum: Dimethyl (2-(benzylamino)-2-oxoethyl)phosphonate



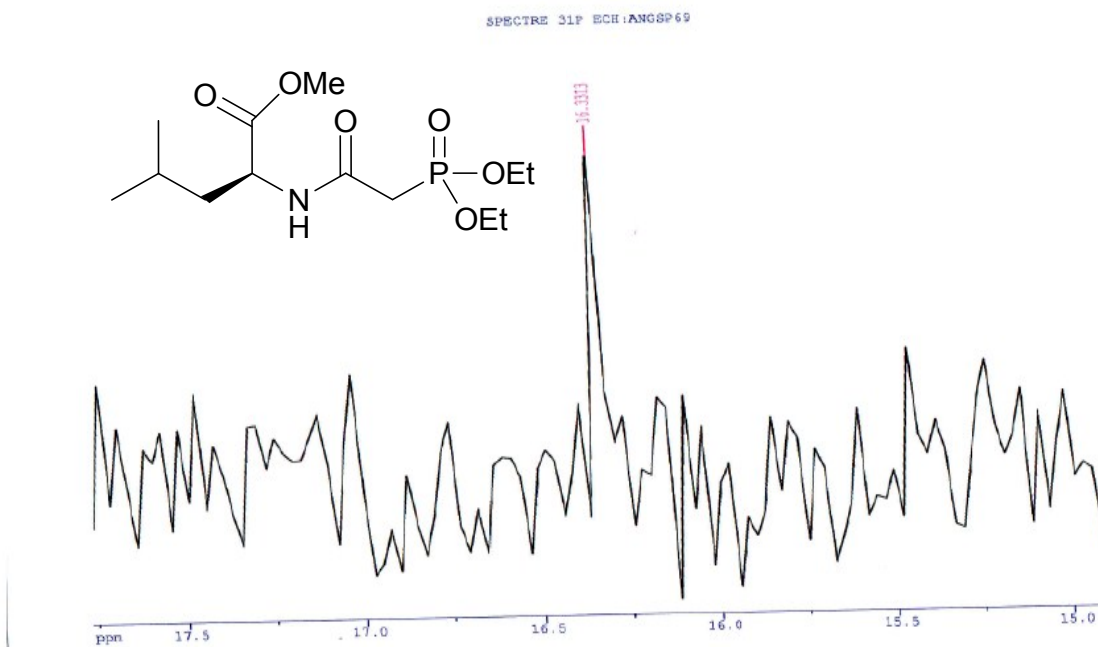
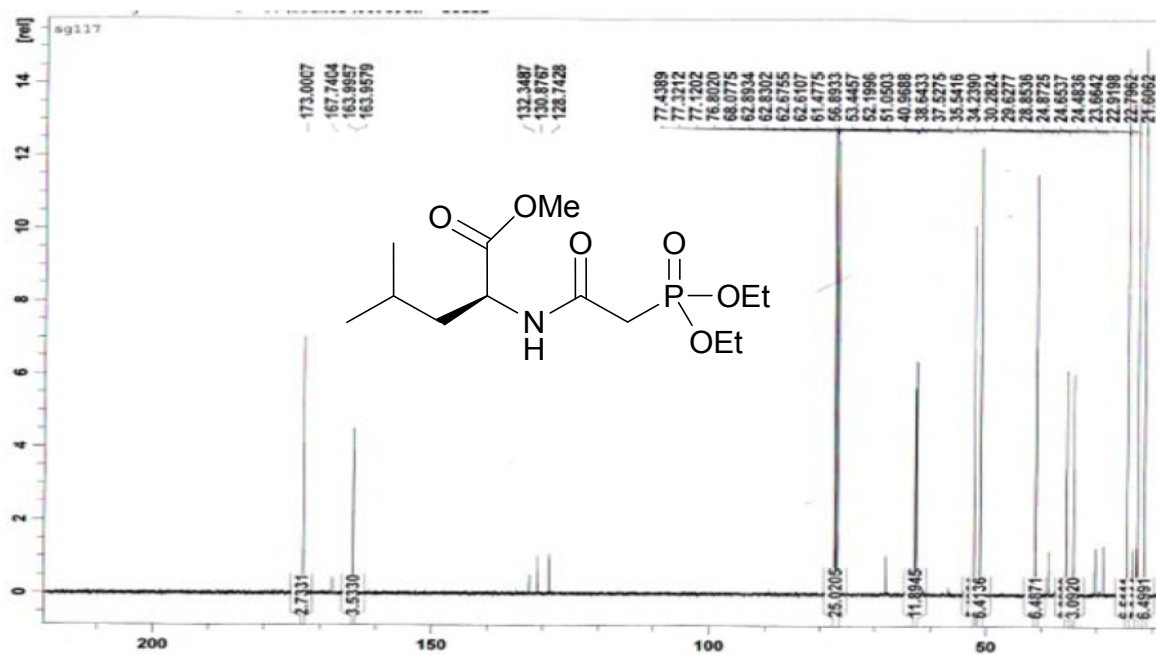
¹³C NMR spectrum: Dimethyl (2-(benzylamino)-2-oxoethyl)phosphonate

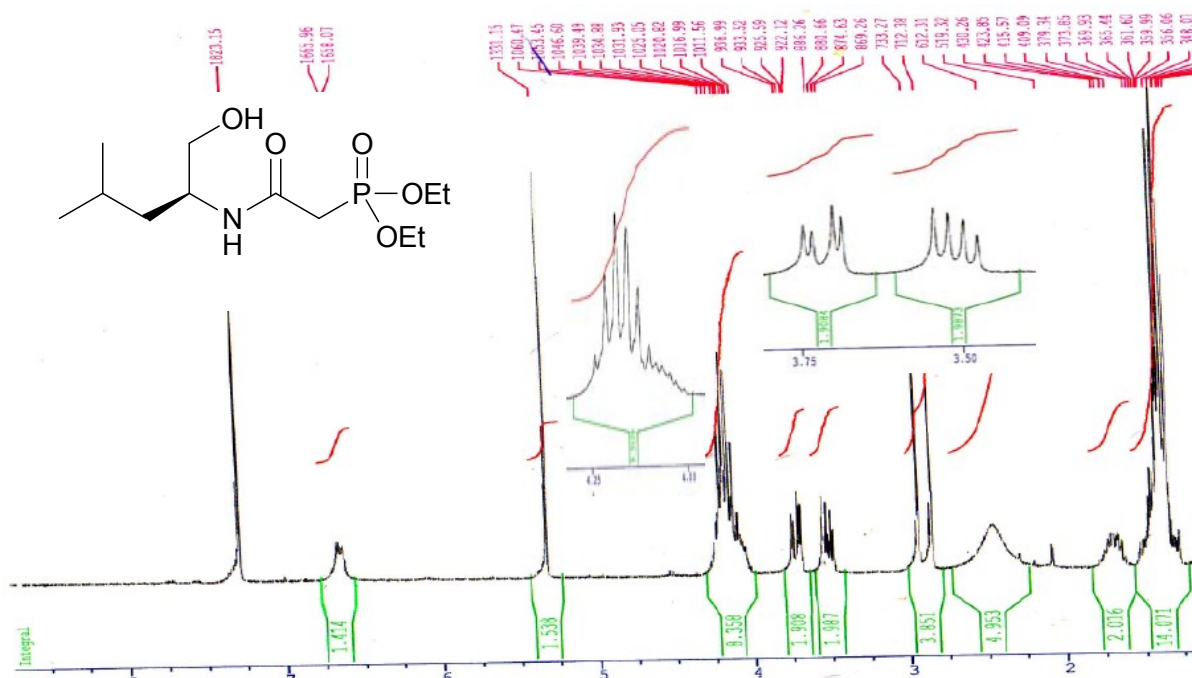


IR spectrum: Dimethyl (2-oxo-2-(benzylamino)ethyl)phosphonate

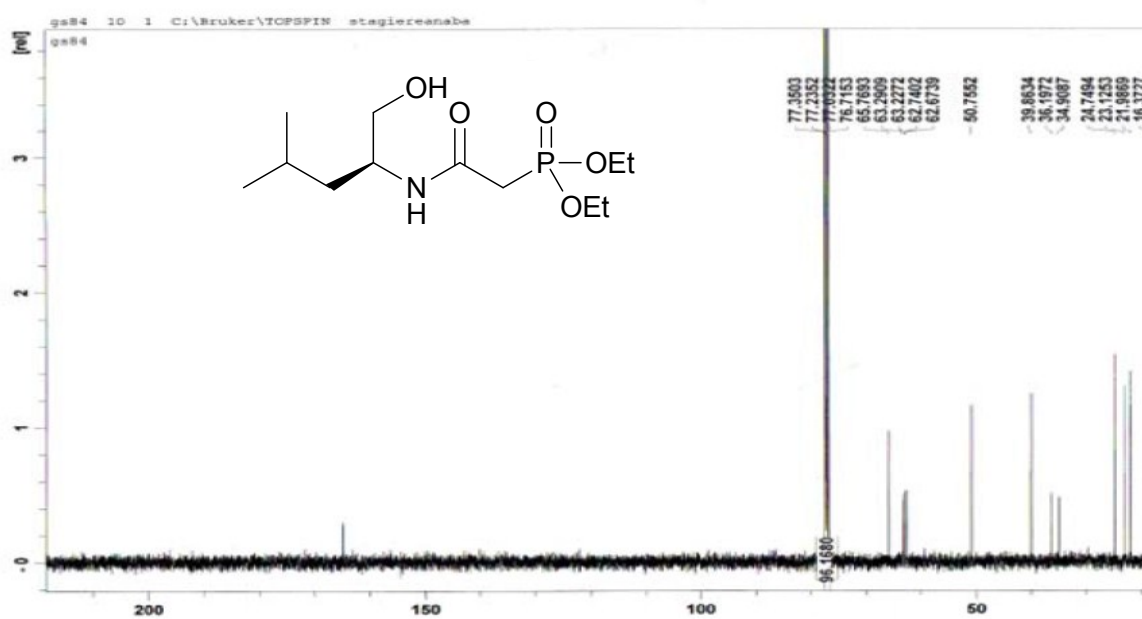


¹H NMR spectrum: (S)-Methyl 2-(2-(diethoxyphosphoryl)acetamido)-4-methylpentanoate

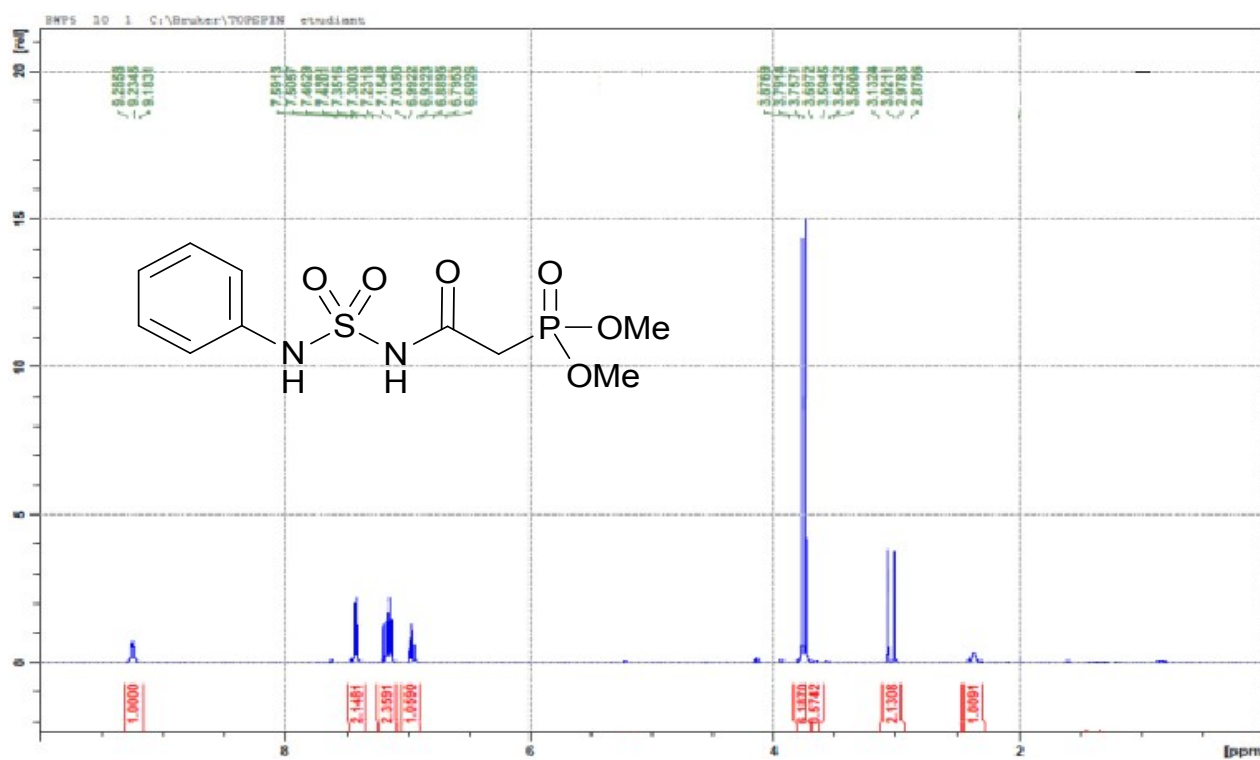
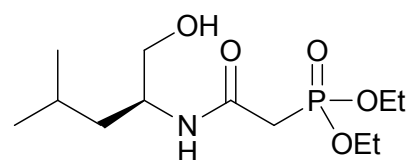




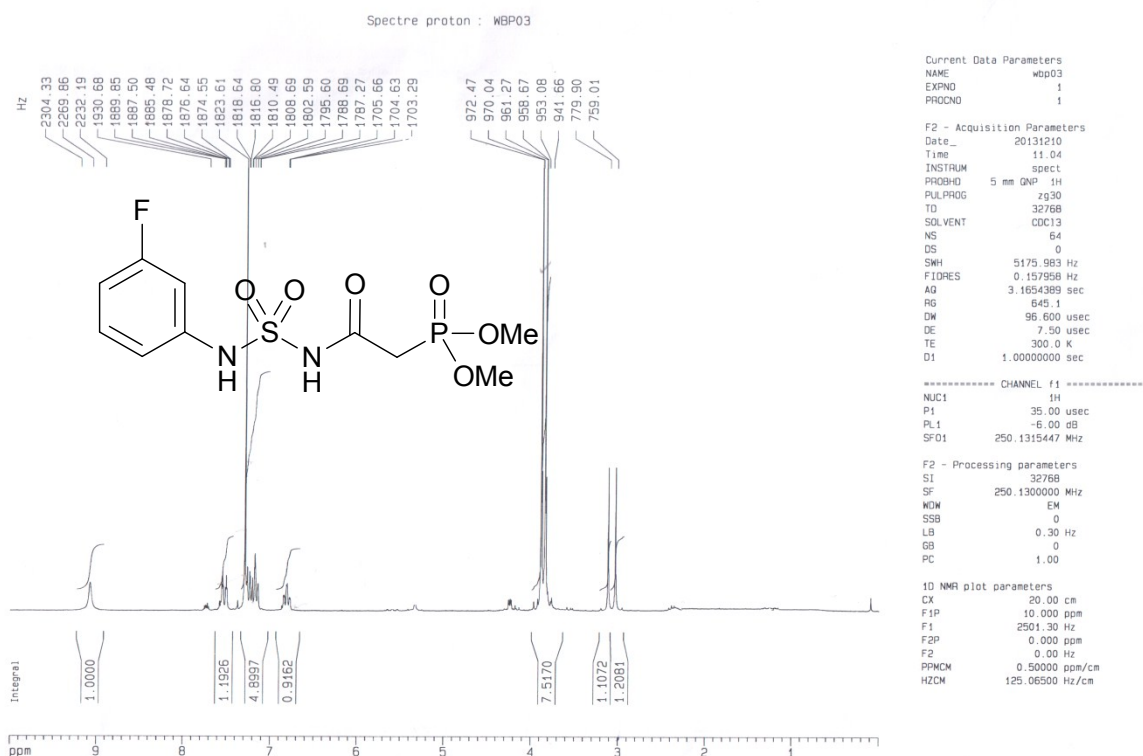
¹H NMR spectrum: (S)-Diethyl 2-((1-hydroxy-4-methylpentan-2-yl)amino)-2-oxoethylphosphonate



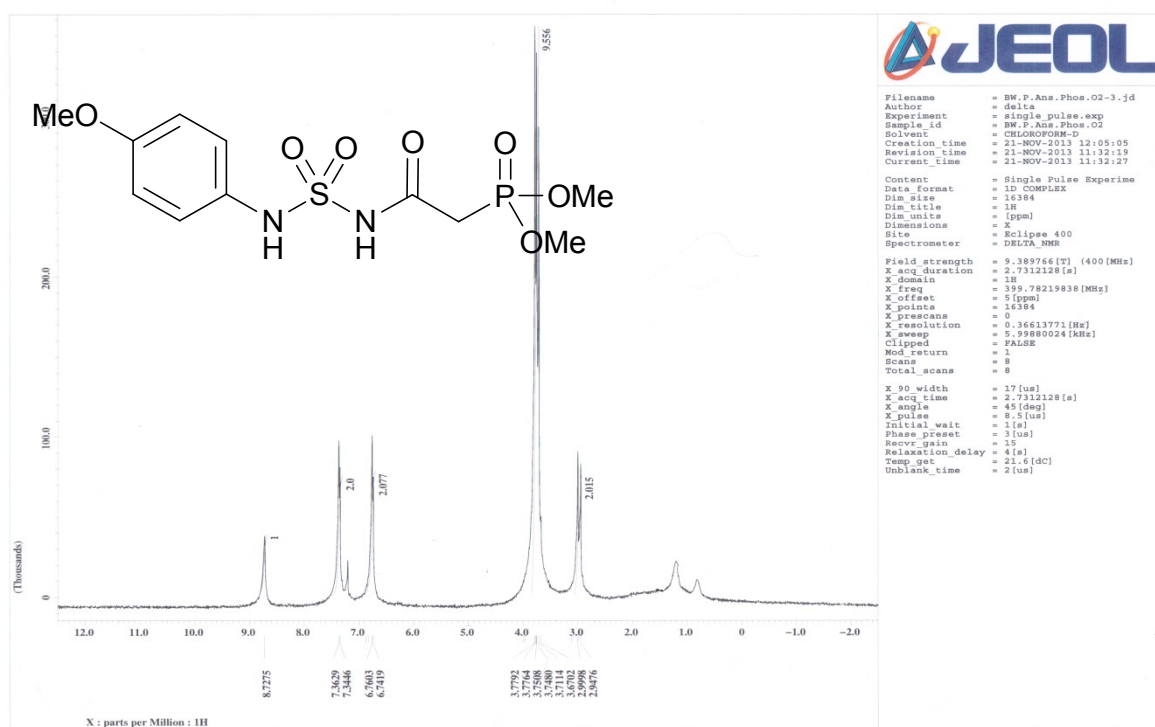
¹³C NMR spectrum: (S)-Diethyl 2-((1-hydroxy-4-methylpentan-2-yl)amino)-2-oxoethylphosphonate



¹H NMR spectrum: N-phenyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide



¹H NMR spectrum: N-3-fluorophenyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide



¹H NMR spectrum: N-4-methoxyphenyl-(1-(2-dimethoxyphosphoryl) acetamide) sulfamide