10.1071/CH14349_AC

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SUPPLEMENTARY MATERIAL

Reactions of Methyl Ketones and (Hetero)arylcarboxamides with *N,N*-Dimethylacetamide Dimethyl Acetal.

A Simple Metal-free Synthesis of 2,4,6-Trisubstituted Pyridines

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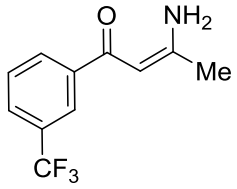
c) FARMA GRS, Seidlova cesta 70, 8000 Novo mesto, Slovenia

Table of contents:

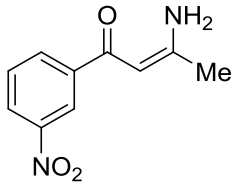
(*Z*)-3-amino-1-(3-(trifluoromethyl)phenyl)but-2-en-1-one (5c)[46]



The product is already known and characterized, but we found no crystal structure of the product, thus we prepared the crystal [46].

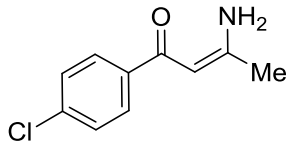
Prepared from 3-(dimethylamino)-1-(3-(trifluoromethyl)phenyl)but-2-en-1-one (655 mg, 2.5 mmol) and ammonium acetate (2.0 g, 25.9 mmol) in 11 mL of methanol. The reaction mixture was stirred at ambient temperature for 6 h. Volatile components were evaporated under *vacuo*, and the product was isolated from the residue via extraction. Yield: 89 % (512 mg).

(Z)-3-amino-1-(3-nitrophenyl)but-2-en-1-one (5e)



Prepared from 3-(dimethylamino)-1-(3-nitrophenyl)but-2-en-1-one (542 mg, 2.3 mmol) and ammonium acetate (2.0 g, 25.9 mmol) in 12 mL of methanol. The reaction mixture was left to stir at ambient temperature for 4 h. The precipitated product was collected with vacuum filtration, and washed with methanol. Yield: 71 % (337 mg).

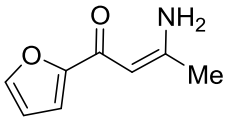
(*Z*)-3-amino-1-(4-chlorophenyl)but-2-en-1-one (5g)[47]



Although the product is already known the crystal structure of the product was not determined, thus we prepared the crystal [47].

The compound was prepared from 1-(4-chlorophenyl)-3-(dimethylamino)but-2-en-1-one (627 mg, 2.80 mmol) and ammonium acetate (2.160 g, 28.0 mmol) in 10 mL of methanol. The reaction mixture was stirred at ambient temperature for 6 h. Volatile components were evaporated under *vacuo*, and the product was isolated from the residue via extraction. Yield: 96 % (525 mg).

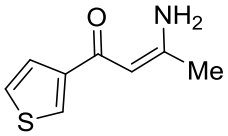
(*Z*)-3-amino-1-(furan-2-yl)but-2-en-1-one (5i)[47]



Although the product is already known the crystal structure of the product was not determined, thus we prepared the crystal [47].

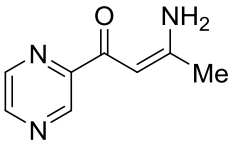
The compound was prepared from 3-(dimethylamino)-1-(furan-2-yl)but-2-en-1-one (503 mg, 2.80 mmol) and ammonium acetate (2.160 g, 28.0 mmol) in 12 mL of methanol. The reaction mixture was stirred at ambient temperature for 6 h. Volatile components were evaporated under *vacuo,* and the product was isolated from the residue via extraction. Yield: 95 % (401 mg).

(Z)-3-amino-1-(thiophen-3-yl)but-2-en-1-one (5j)



Prepared from 3-(dimethylamino)-1-(thiophen-3-yl)but-2-en-1-one (441 mg, 2.3 mmol) and ammonium acetate (1.40 g, 18 mmol) in 7 mL of methanol. The reaction mixture was left to stir at ambient temperature for 4 h. Volatile components were evaporated under *vacuo*, and the product was isolated from the residue via extraction. Yield: 95 % (367 mg).

(Z)-3-amino-1-(pyrazin-2-yl)but-2-en-1-one (5m)



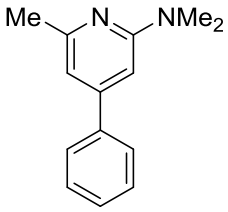
Prepared from 3-(dimethylamino)-1-(pyrazin-2-yl)but-2-en-1-one (668 mg, 3.5 mmol) and ammonium acetate (2.15 g, 28 mmol) in 10 mL of methanol. The reaction mixture was stirred at ambient temperature for 6 h. Volatile components were evaporated under *vacuo*, and the product was isolated from the residue via extraction. Yield: 95 % (547 mg).

Crystalographic data for Enaminones **5**.

Crystal data	**5c**	**5g**	**5i**	**5j**	**5m**
Formula	$C_{11}H_{10}F_3NO$	$C_{10}H_{10}ClNO$	$C_8H_9NO_2$	C_8H_9NOS	$C_8H_9N_3O$
M_r	229.20	195.64	151.16	167.22	163.18
Cell setting, space group	Monoclinic, $P12_1/c1$	Orthorhombic, *Pccn*	Triclinic, $P\bar{1}$	Tetragonal, $P4_1$	Monoclinic, $P2_1$
a (Å)	12.8117(5)	8.0757(4)	9.2279(6)	8.2883(3)	3.8781(3)
b (Å)	10.5100(4)	26.8322(14)	9.3829(9)	8.2883(3)	9.3987(6)
c (Å)	7.7540(3)	9.4110(5)	9.5326(5)	12.6091(6)	10.9937(6)
α (°)	90	90	94.307(6)	90	90
β (°)	100.323(3)	90	90.066(5)	90	93.937(6)
γ (°)	90	90	91.582(7)	90	90
V (Å^3)	1027.18(7)	2039.27(18)	822.73(10)	866.19(8)	399.76(5)
Z	4	8	4	4	4
D_x (Mg m^{-3})	1.482	1.274	1.220	1.282	1.356
μ (mm^{-1})	0.132	0.334	0.089	0.315	0.095
F(000)	472	816	320	352	172
Crystal form, colour	Prism, colourless	Platelet, colourless	Platelet, colourless	Platelet, colourless	Platelet, colourless
Crystal size (mm)	0.40×0.30×0.20	0.40×0.15×0.10	0.45×0.30×0.02	0.25×0.20×0.02	0.50×0.10×0.05
Data collection					
T (K)	150	293	293	293	293
Radiation type, wavelength	Mo Kα, λ=0.7107 Å	Mo Kα, λ=0.7107 Å	Mo Kα, λ=0.7107 Å	Mo Kα, λ=0.7107 Å	Mo Kα, λ=0.7107 Å
Diffractometer	SuperNova-Atlas	SuperNova-Atlas	SuperNova-Atlas	SuperNova-Atlas	SuperNova-Atlas
Data collection method	ω scans	ω scans	ω scans	ω scans	ω scans
Absorption	Multi-scan	Multi-scan	Multi-scan	Multi-scan	Multi-scan

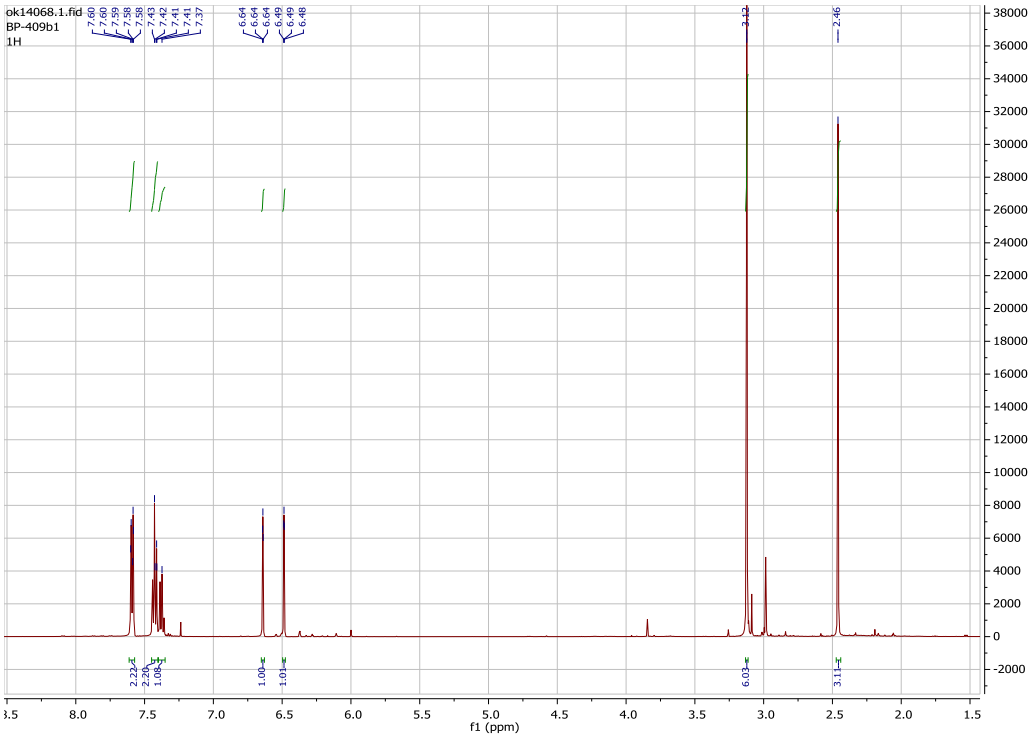
correction					
No. of measured, independent and observed reflections	5349, 2347, 1853	8090, 2342, 1376	8730, 3763, 2691	3966, 1767, 1414	3200, 1986, 1368
Criterion for observed reflections	>2sigma(I)	>2sigma(I)	>2sigma(I)	>2sigma(I)	>2sigma(I)
R_{int}	0.0231	0.0287	0.0207	0.0206	0.0446
θ range (°)	3.23−27.48	3.04−27.48	2.94−27.48	2.94−27.45	3.72−30.23
h range	−13 → 16	−9 → 10	−11 → 11	−10 → 10	−5 → 4
k range	−11 → 13	−34 → 34	−12 → 8	−8 → 10	−12 → 13
l range	−9 → 10	−11 → 12	−12 → 11	−16 → 16	−15 → 14
Refinement					
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
R (on F_{obs}), *wR* (on F_{obs}), *S*	0.0751, 0.2011, 1.070	0.0483, 0.1266, 1.033	0.0584, 0.1595, 1.031	0.0515, 0.1207, 1.041	0.0523, 0.1149, 1.025
No. of contributing reflections	2347	2342	3763	1767	1986
No. of parameters	134	127	210	102	118
No. of restraints	0	0	2	3	1
$(\Delta/\sigma)_{max}$, $(\Delta/\sigma)_{ave}$	0.000, 0.000	0.024, 0.001	0.000, 0.000	0.004, 0.000	0.000, 0.000
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (eÅ^{-3})	0.938, −0.791	0.135, −0.268	0.342, −0.396	0.288, −0.273	0.155, −0.143

N,N,6-trimethyl-4-phenylpyridin-2-amine (10a)

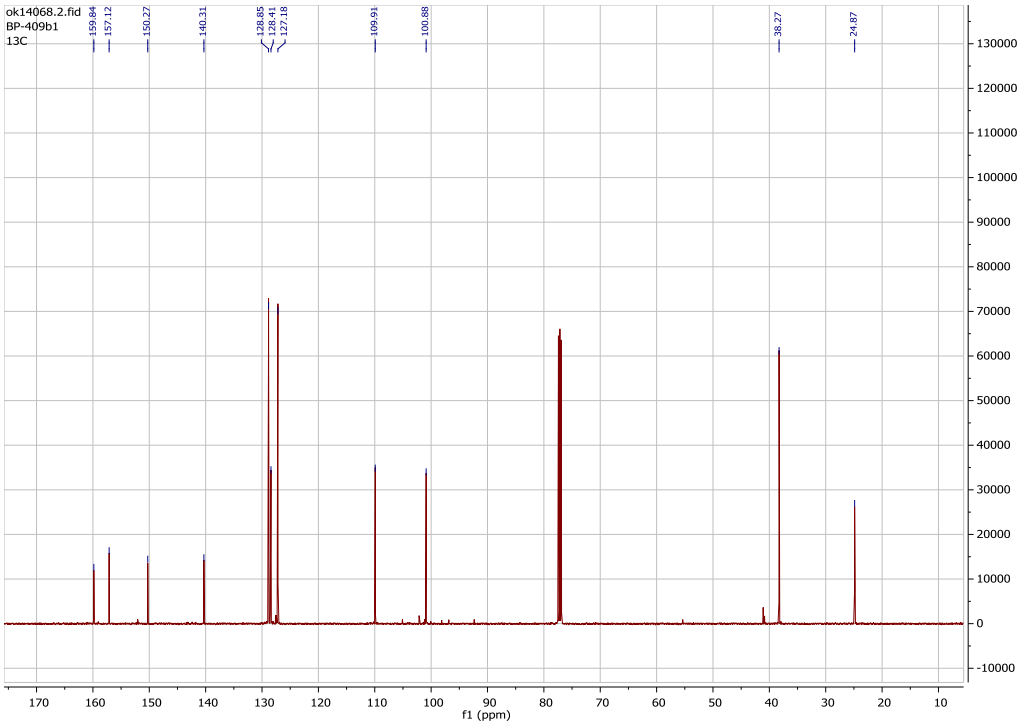


Prepared from (*Z*)-3-amino-1-phenylbut-2-en-1-one (250 mg, 1.55 mmol) and DMADMA (0.453 mL, 3.10 mmol), 135 °C, 10 min. Column chromatography, Ethyl acetate : Petroleum ether = 1 : 5. Yield: 35 % (115 mg).

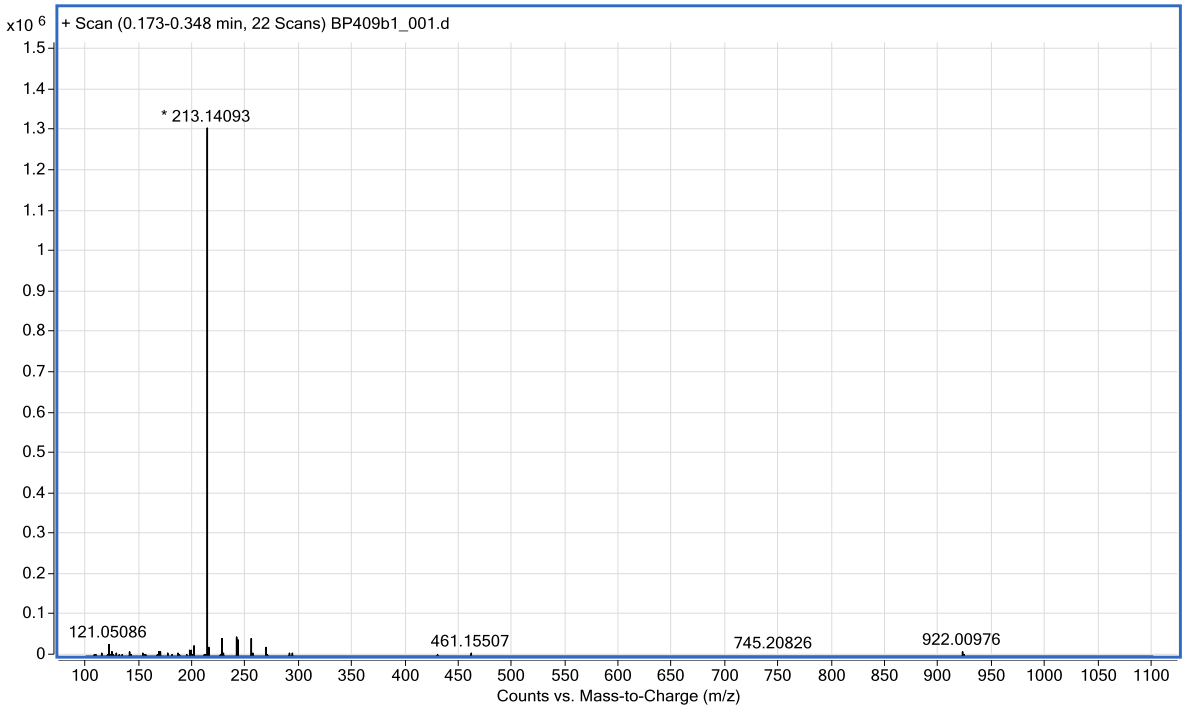
^{1}H NMR:



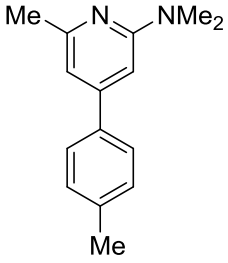
^{13}C NMR:


ok14068.2.fid
BP-409b1
13C
159.84
157.12
150.27
140.31
128.85
128.41
127.18
109.91
100.88
38.27
24.87
f1 (ppm)

MS:

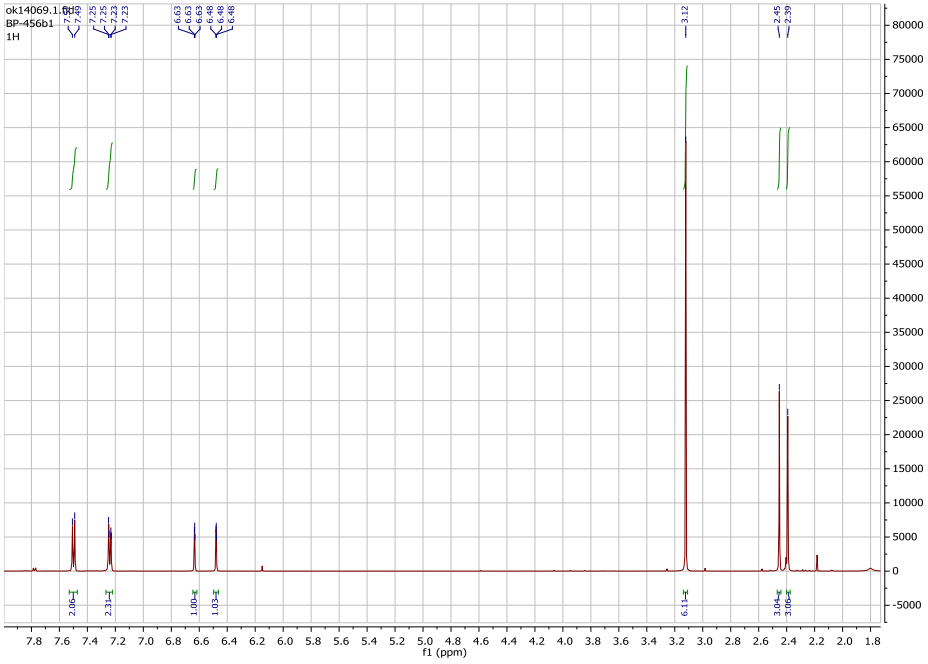

x10 6
+ Scan (0.173-0.348 min, 22 Scans) BP409b1_001.d
* 213.14093
121.05086
461.15507
745.20826
922.00976
Counts vs. Mass-to-Charge (m/z)

N,N,6-trimethyl-4-(p-tolyl)pyridin-2-amine (10b)

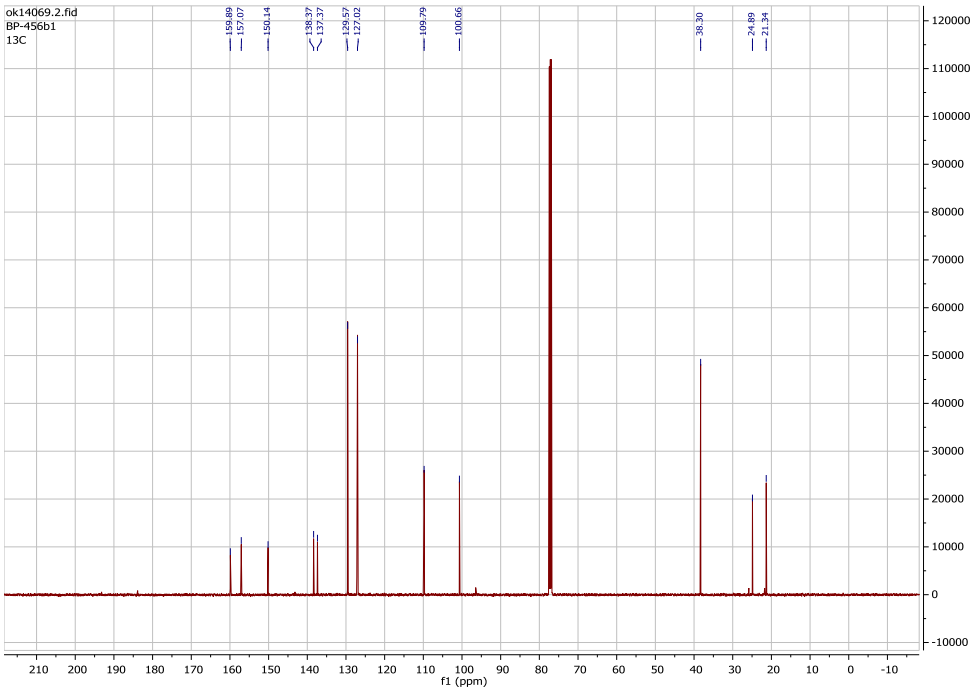


The product was prepared from (*Z*)-3-amino-1-(*p*-tolyl)but-2-en-1-one (272 mg, 1.55 mmol) and DMADMA (0.454 mL, 3.11 mmol), 130 °C, 8 min. Column chromatography, Ethyl acetate : Hexane = 1 : 5. Yield: 43 % (151 mg).

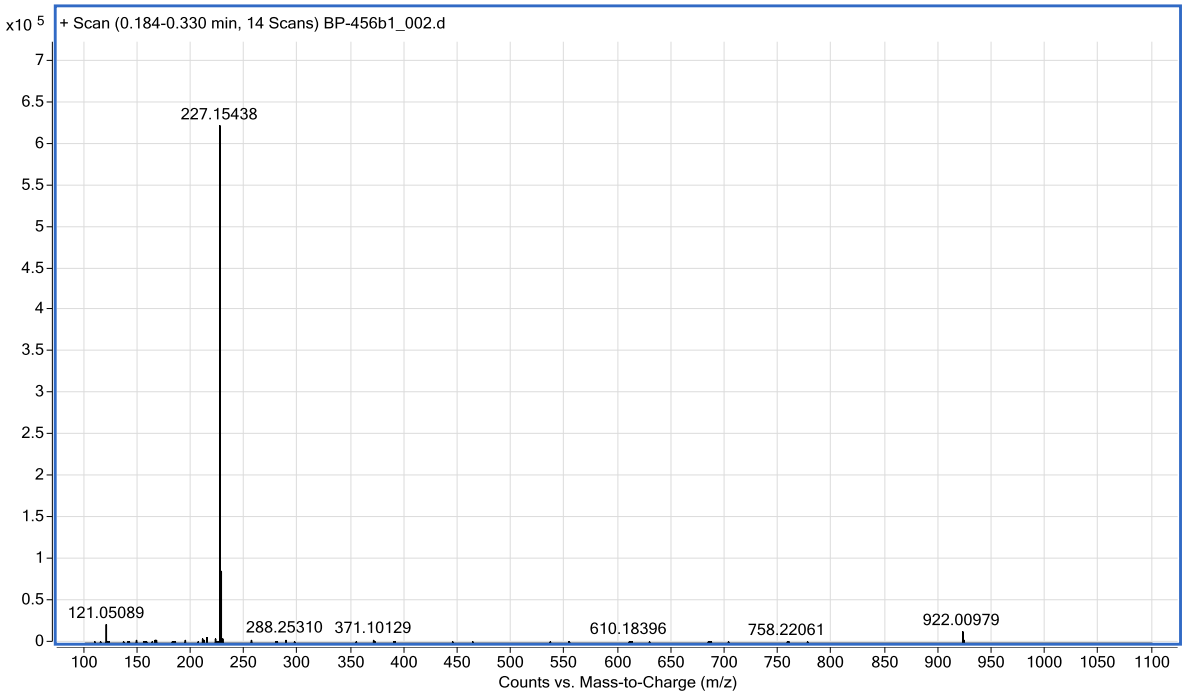
^{1}H NMR:



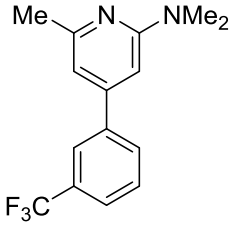
^{13}C NMR:


ok14069.2.fid
BP-456b1
13C
159.89
157.07
150.14
138.37
137.37
129.57
127.02
109.79
100.66
38.30
24.89
21.34
f1 (ppm)

MS:

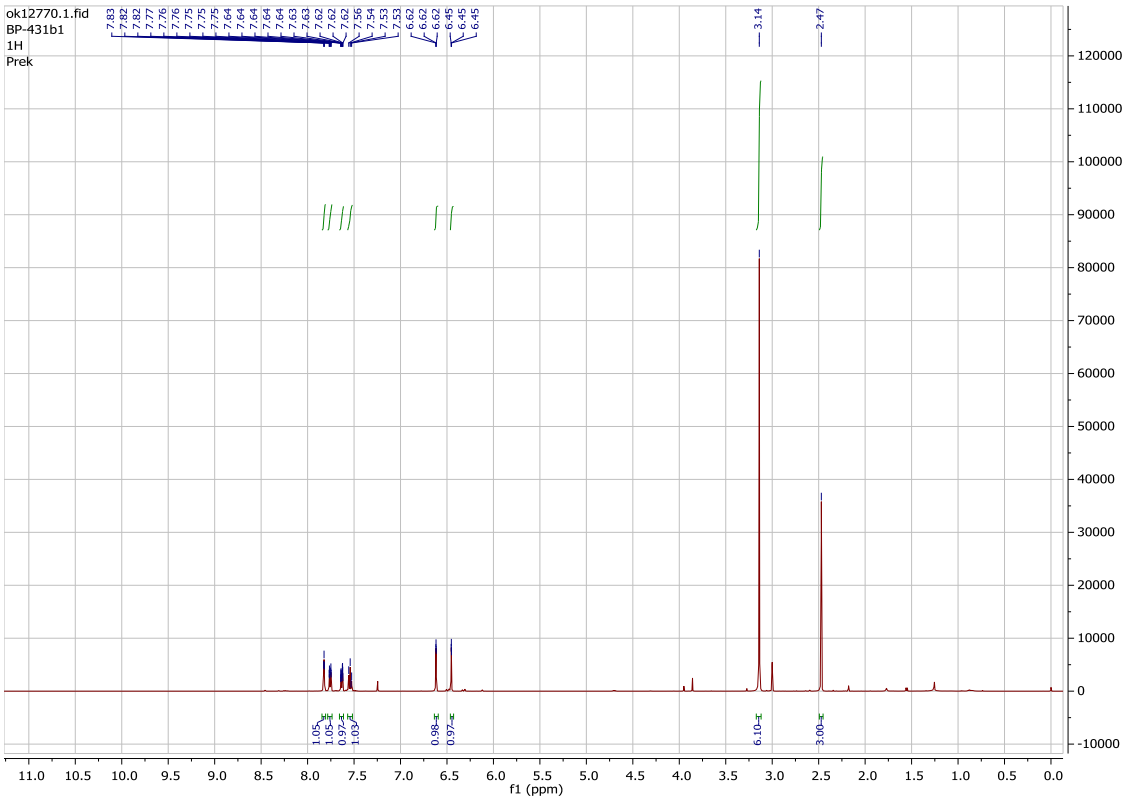

x10 5
+ Scan (0.184-0.330 min, 14 Scans) BP-456b1_002.d
227.15438
121.05089
288.25310
371.10129
610.18396
758.22061
922.00979
Counts vs. Mass-to-Charge (m/z)

N,N,6-trimethyl-4-(3-(trifluoromethyl)phenyl)pyridin-2-amine (10c)

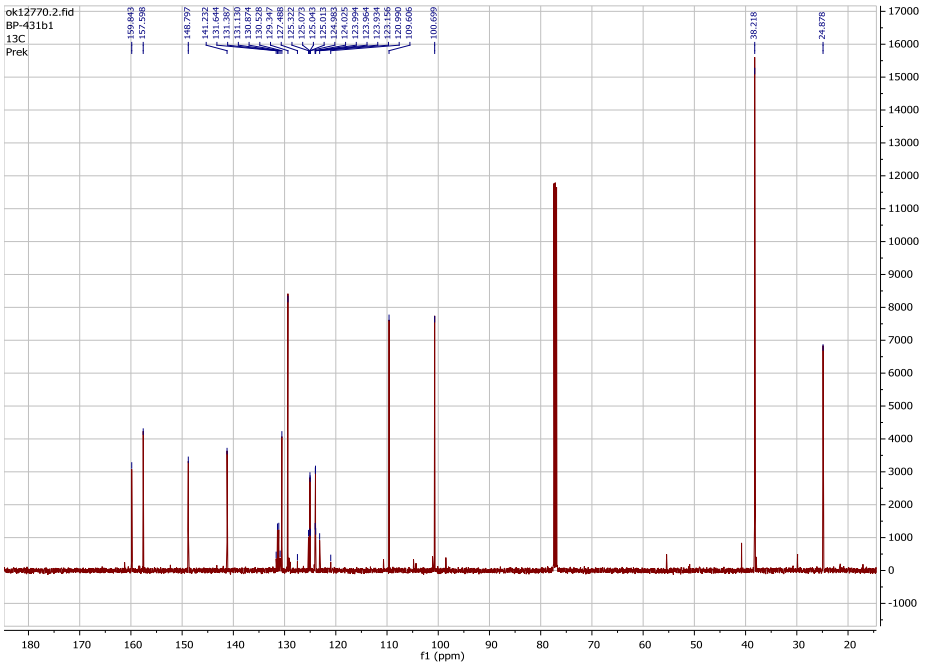


Prepared from (Z)-3-amino-1-(3-(trifluoromethyl)phenyl)but-2-en-1-one (284 mg, 1.24 mmol) and DMADMA (0.657 mL, 4.50 mmol), 130 °C, 2 min. Column chromatography, Ethyl acetate : Petroleum ether = 1 : 10. Yield: 13 % (46 mg).

^{1}H NMR:



^{13}C NMR: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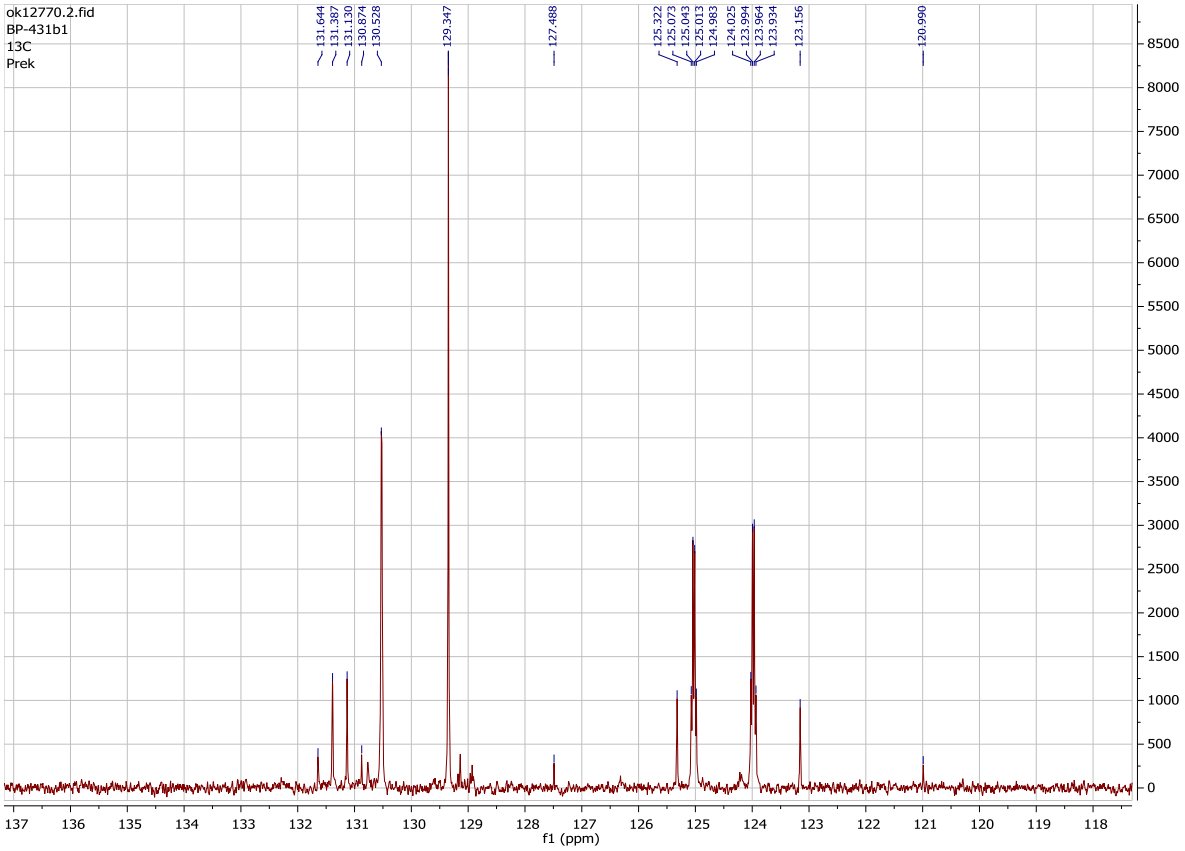
BP-431b1
13C
Prek
159.843
157.598
148.797
141.232
131.644
131.387
131.130
130.874
130.528
129.347
127.488
125.322
125.073
125.043
125.013
124.983
124.025
123.994
123.964
123.934
123.156
120.990
109.606
100.699
38.218
24.878
f1 (ppm)


ok12770.2.fid
BP-431b1
13C
Prek
131.644
131.387
131.130
130.874
130.528
129.347
127.488
125.322
125.073
125.043
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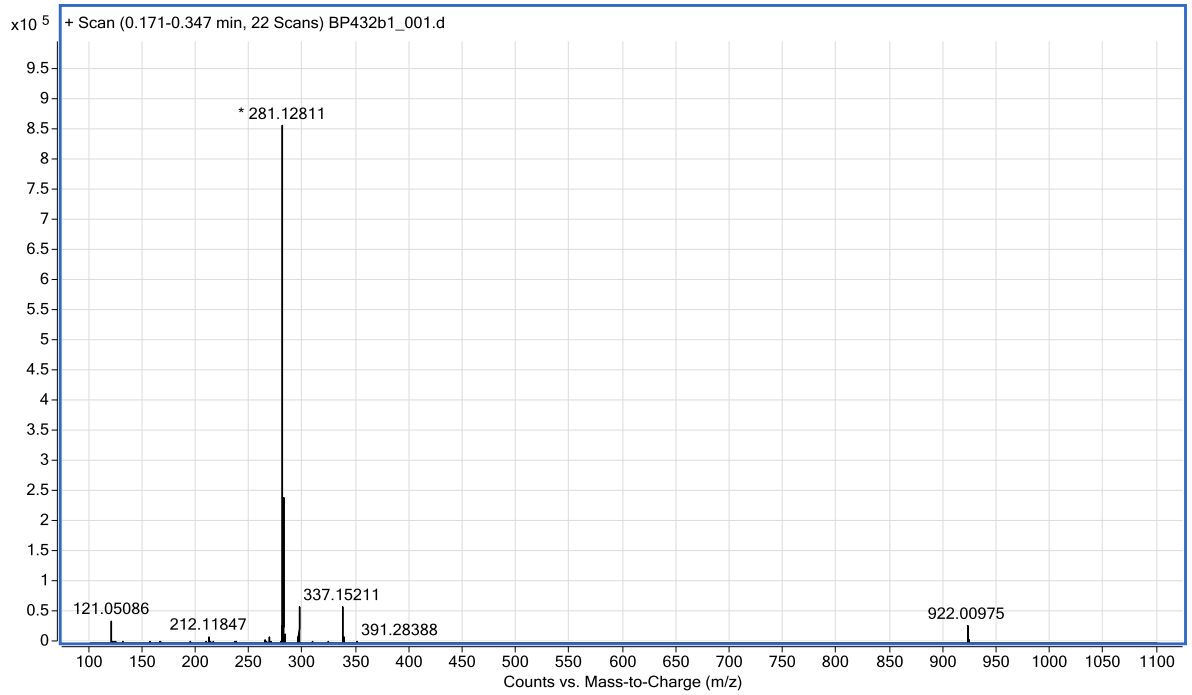
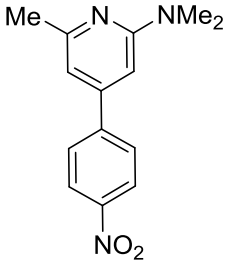
124.983
124.025
123.994
123.964
123.934
123.156
120.990
f1 (ppm)

MS:

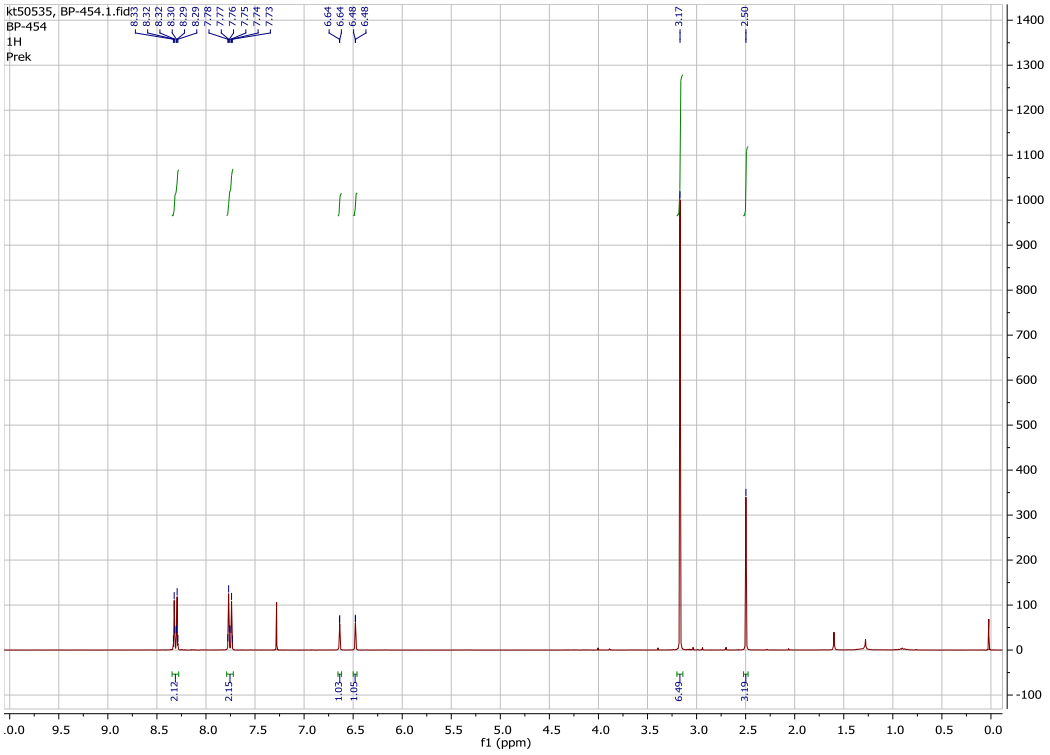

x10 5
+ Scan (0.171-0.347 min, 22 Scans) BP432b1_001.d
* 281.12811
337.15211
121.05086
212.11847
391.28388
922.00975
Counts vs. Mass-to-Charge (m/z)

N,N,6-trimethyl-4-(4-nitrophenyl)pyridin-2-amine (10d)

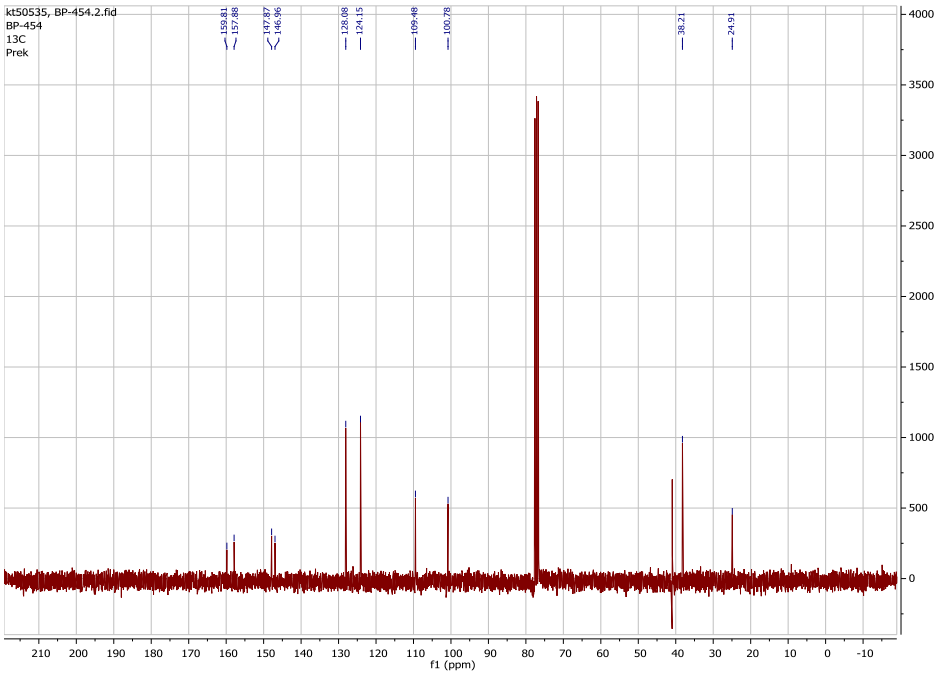


Prepared from (*Z*)-3-amino-1-(4-nitrophenyl)but-2-en-1-one (226 mg, 1.10 mmol) and DMADMA (0.321 mL, 2.20 mmol), 130 °C, 8 min. Column chromatography, Ethyl acetate : Petroleum ether = 1 : 5. Yield: 18 % (52 mg).

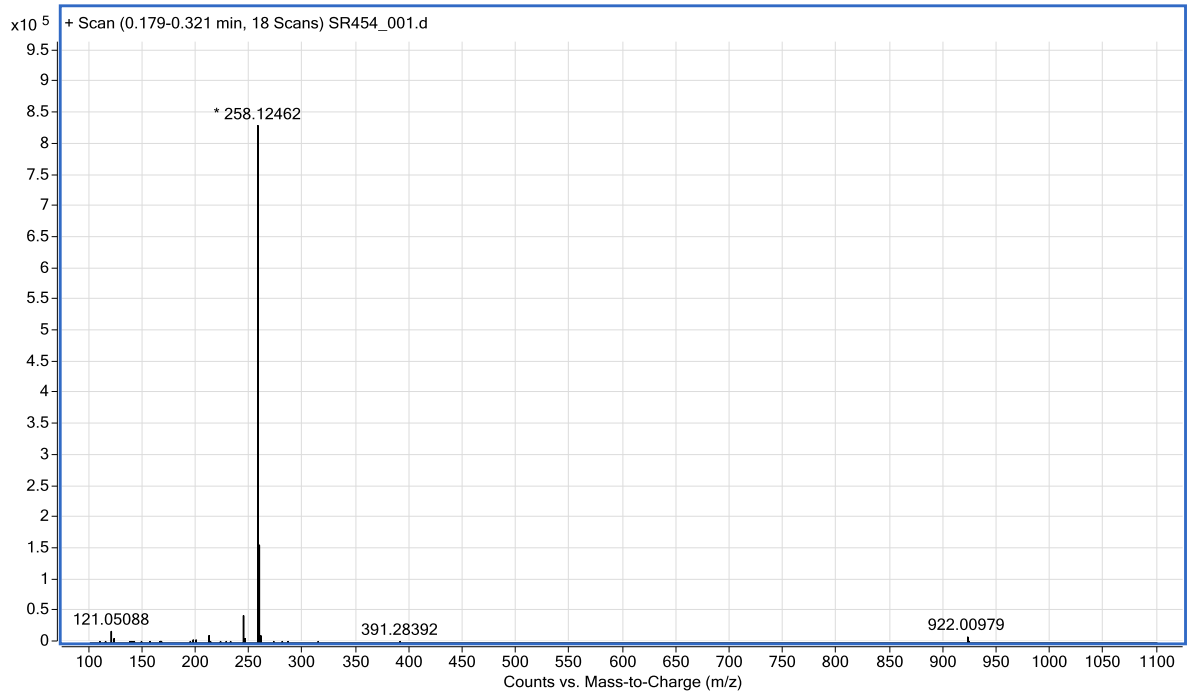
^{1}H NMR:



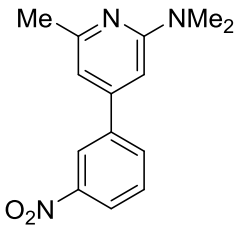
^{13}C NMR:


kt50535, BP-454.2.fid
BP-454
13C
Prek
159.81
157.88
147.87
146.96
128.06
124.15
109.48
100.78
38.21
24.91
f1 (ppm)

MS:

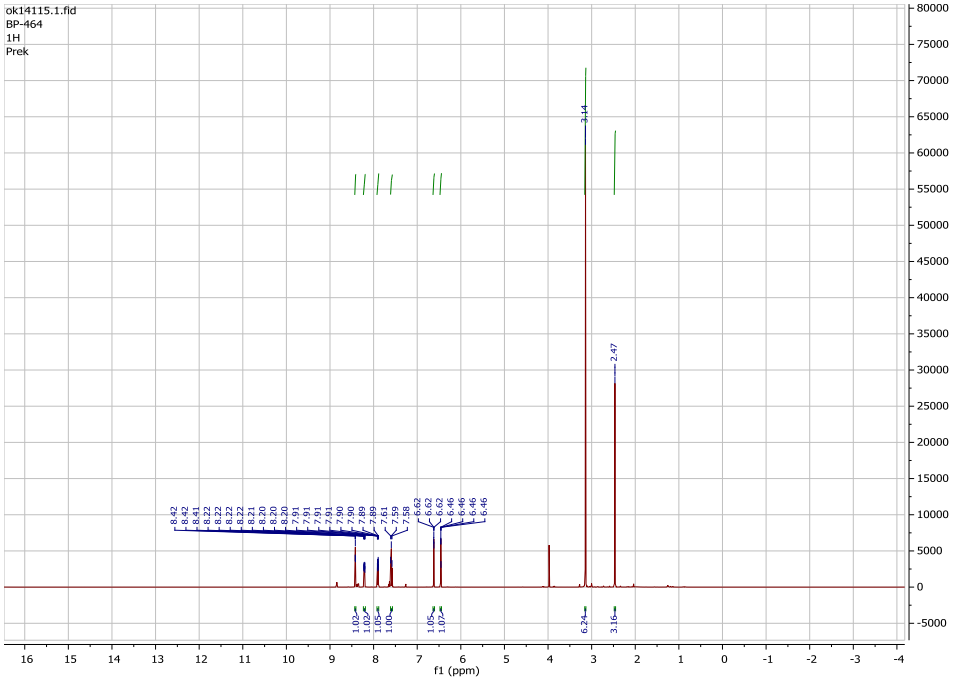

x10 5
+ Scan (0.179-0.321 min, 18 Scans) SR454_001.d
* 258.12462
121.05088
391.28392
922.00979
Counts vs. Mass-to-Charge (m/z)

N,N,6-trimethyl-4-(3-nitrophenyl)pyridin-2-amine (10e)

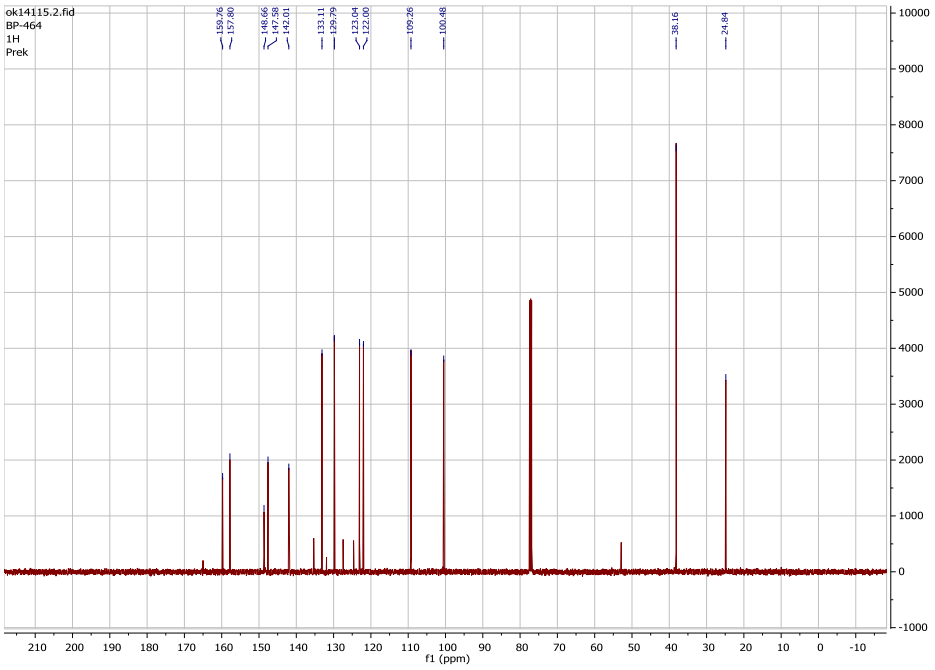


Prepared from (Z)-3-amino-1-(3-nitrophenyl)but-2-en-1-one (216 mg, 1.05 mmol) and DMADMA (0.306 mL, 2.10 mmol), 138 °C, 8 min. Column chromatography, Ethyl acetate : Petroleum ether = 1 : 3. Yield: 32 % (87 mg).

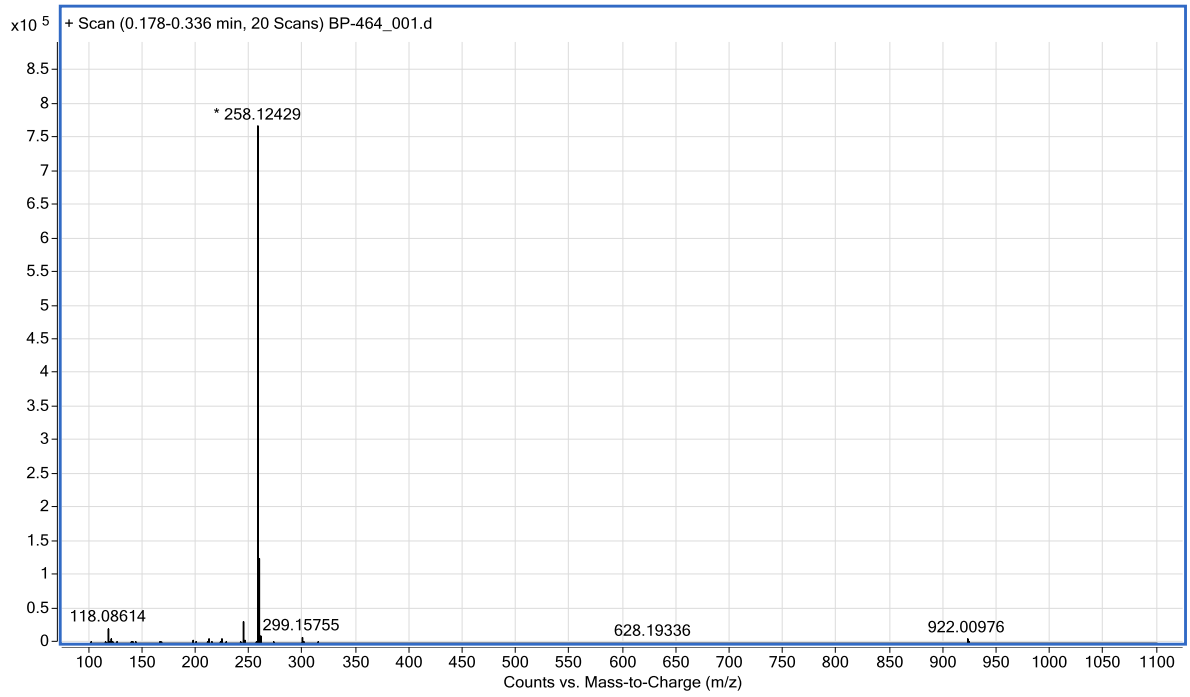
^{1}H NMR:



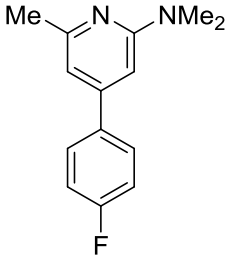
^{13}C NMR:


ok14115.2.fid
BP-464
1H
Prek
159.76
157.80
148.66
147.88
142.01
133.11
129.79
123.04
122.00
109.26
100.48
38.16
24.84
f1 (ppm)

MS:

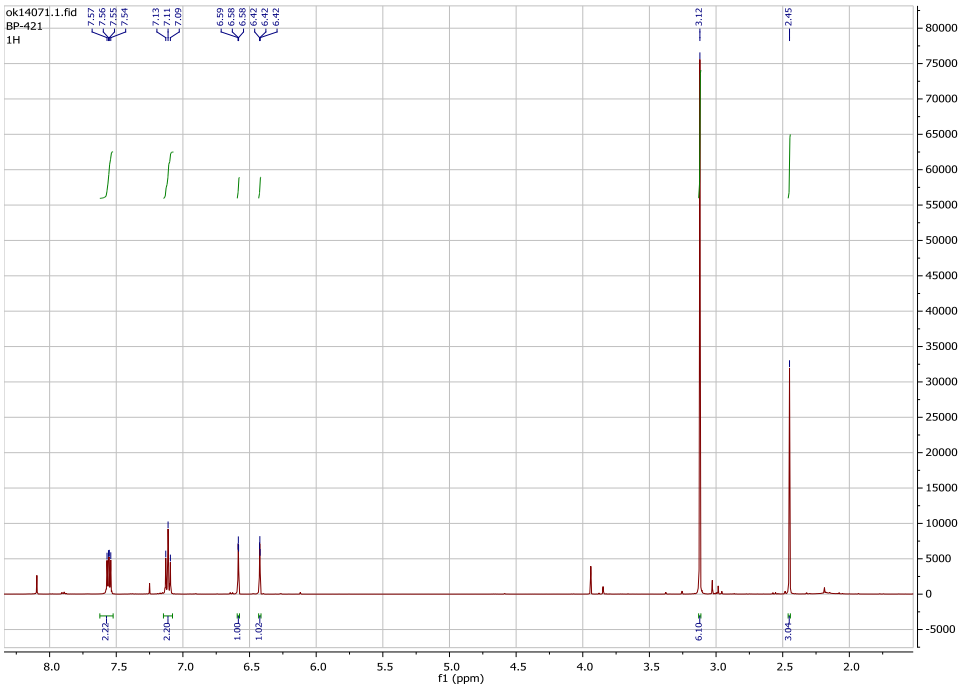

x10 5
+ Scan (0.178-0.336 min, 20 Scans) BP-464_001.d
* 258.12429
118.08614
299.15755
628.19336
922.00976
Counts vs. Mass-to-Charge (m/z)

4-(4-fluorophenyl)-N,N,6-trimethylpyridin-2-amine (10f)

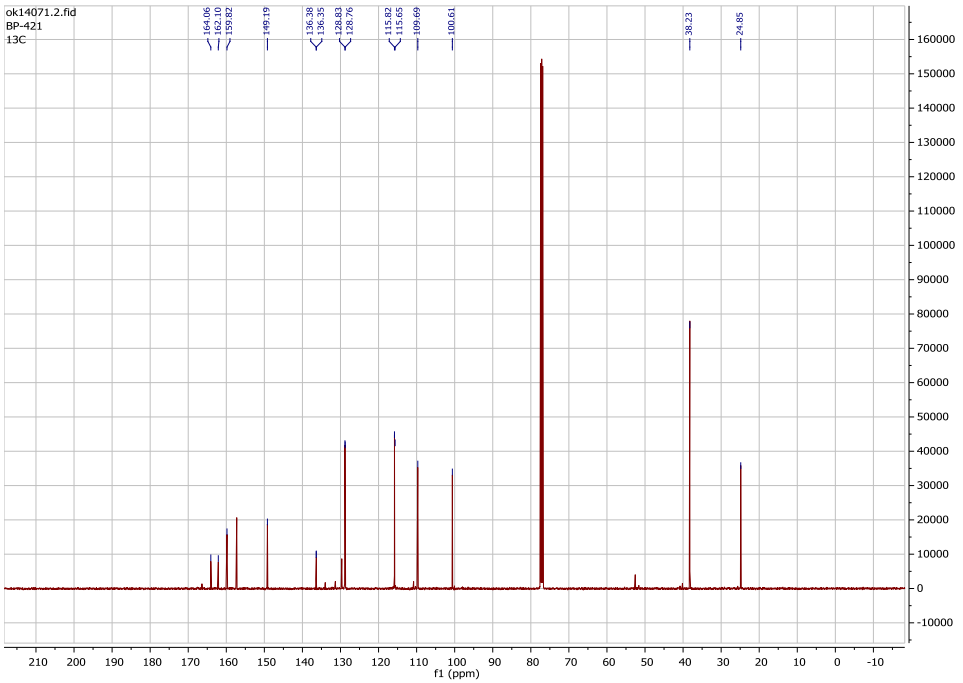


Prepared from (*Z*)-3-amino-1-(4-fluorophenyl)but-2-en-1-one (301 mg, 1.68 mmol) and DMADMA (0.491 mL, 3.36 mmol), 130 °C, 7 min. Column chromatography, Ethyl acetate : Petroleum ether = 1 : 10. Yield: 28 % (110 mg).

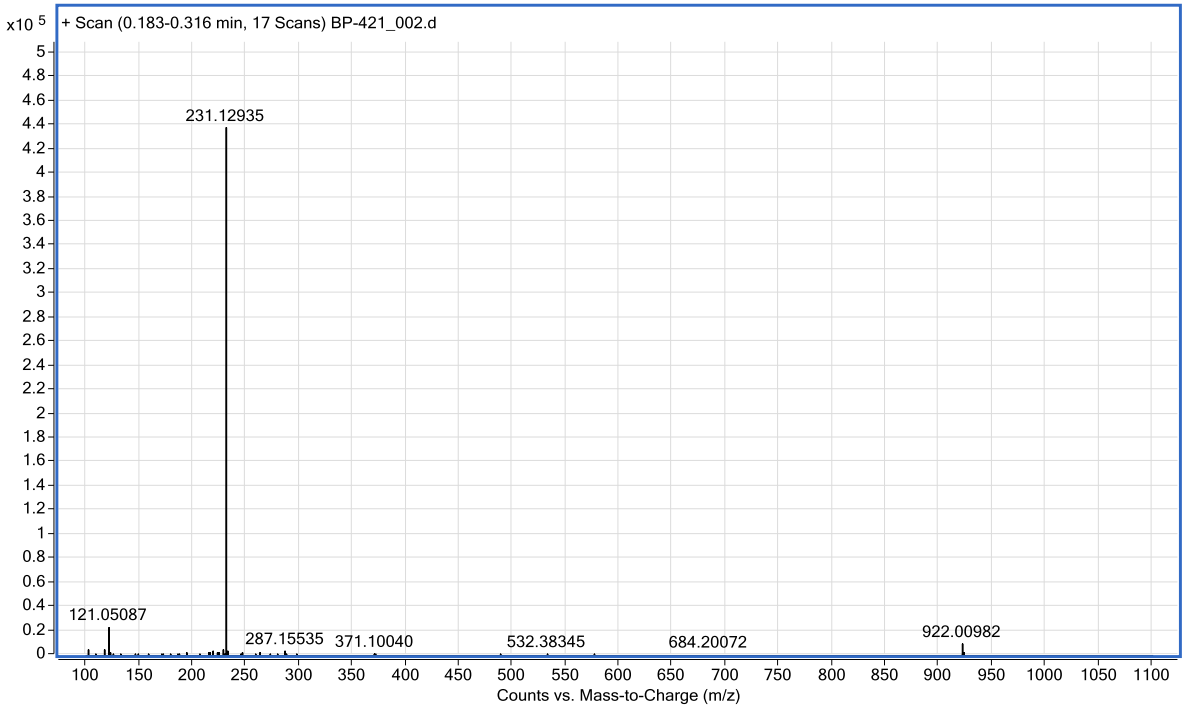
^{1}H NMR:



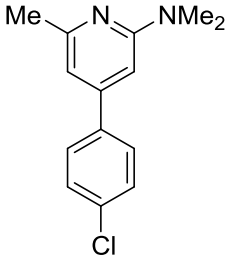
^{13}C NMR:


ok14071.2.fid
BP-421
13C
164.06
162.10
159.82
149.19
136.38
136.35
128.83
128.76
115.82
115.65
109.69
100.61
38.23
24.85
f1 (ppm)

MS:

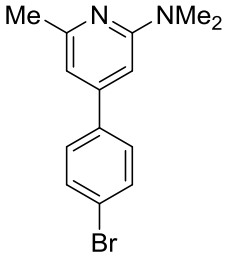

x10 5
+ Scan (0.183-0.316 min, 17 Scans) BP-421_002.d
231.12935
121.05087
287.15535
371.10040
532.38345
684.20072
922.00982
Counts vs. Mass-to-Charge (m/z)

4-(4-chlorophenyl)-N,N,6-trimethylpyridin-2-amine (10g)



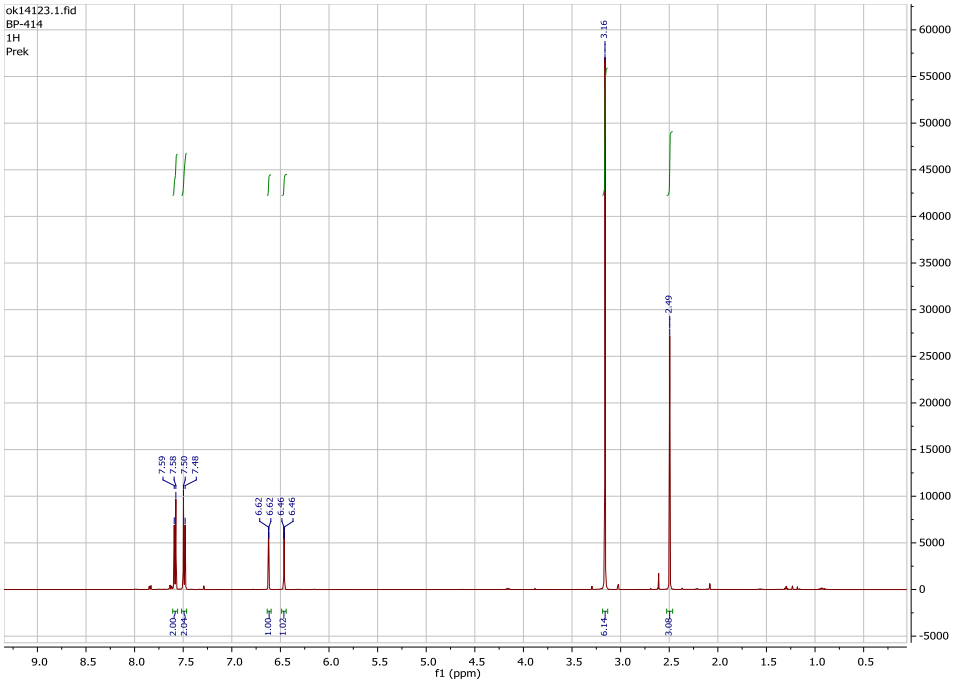
Prepared from (*Z*)-3-amino-1-(4-chlorophenyl)but-2-en-1-one (365 mg, 1.90 mmol) and DMADMA (0.800 mL, 5.48 mmol), 130 °C, 8 min. A larger excess of DMADMA was added due to low solubility of the starting compound. Column chromatography, Ethyl acetate : Hexane = 1:10. Yield: 22 % (104 mg).

4-(4-bromophenyl)-N,N,6-trimethylpyridin-2-amine (10h)

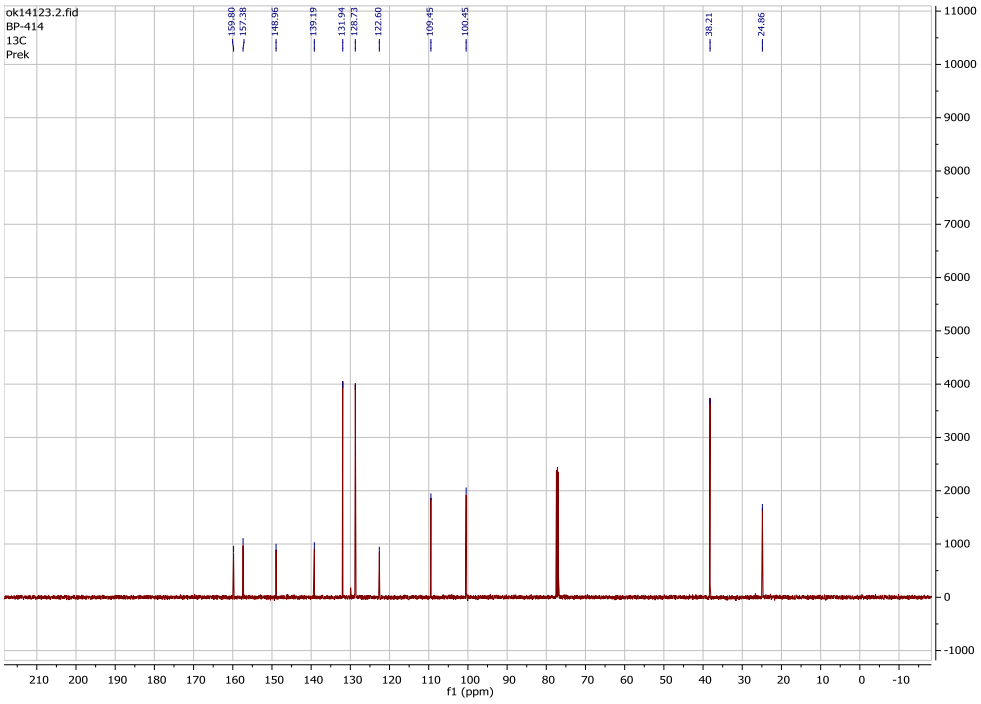


Prepared from (*Z*)-3-amino-1-(4-bromophenyl)but-2-en-1-one (307 mg, 1.30 mmol) and DMADMA (1.0 mL, 6.85 mmol), 130 °C, 7 min. A larger excess of DMADMA was added due to low solubility of the starting compound. Column chromatography, Ethyl acetate : Petroleum ether = 1 : 5. Yield: 13 % (50 mg).

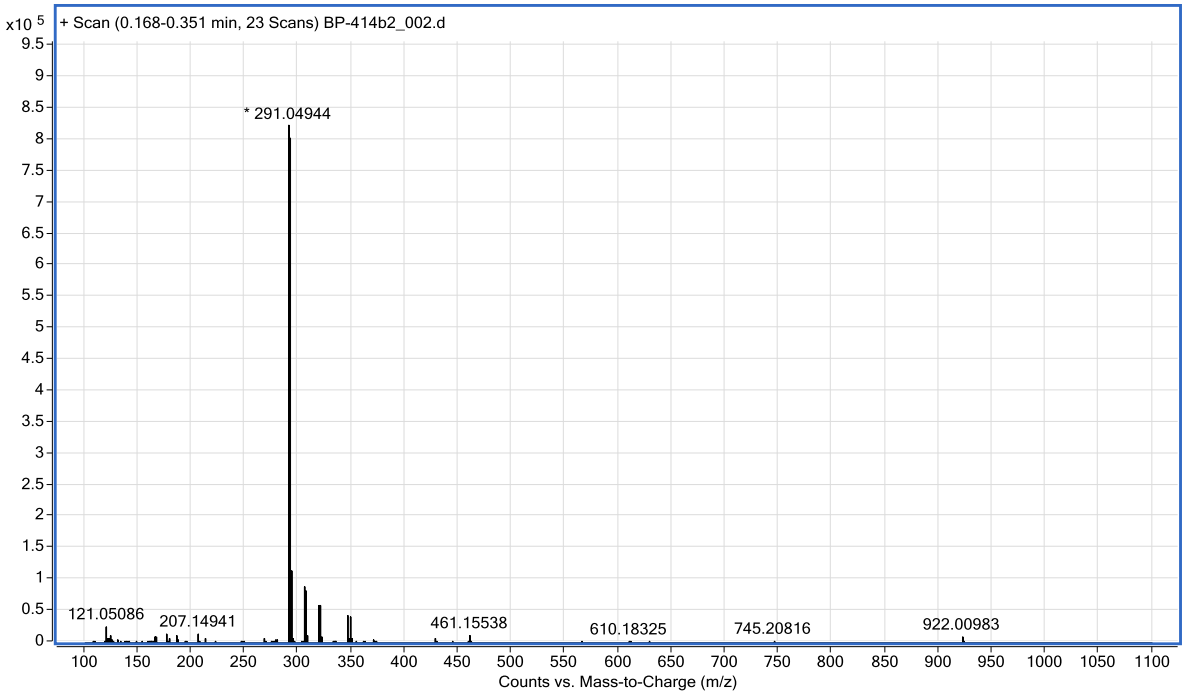
^{1}H NMR:



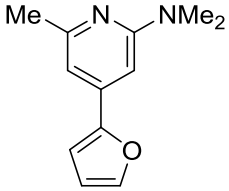
^{13}C NMR:


ok14123.2.fid
BP-414
13C
Prek
159.80
157.38
148.95
139.19
131.94
128.73
122.60
109.45
100.45
38.21
24.86
f1 (ppm)

MS:

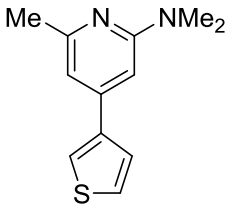

x10 5
+ Scan (0.168-0.351 min, 23 Scans) BP-414b2_002.d
* 291.04944
121.05086
207.14941
461.15538
610.18325
745.20816
922.00983
Counts vs. Mass-to-Charge (m/z)

4-(Furan-2-yl)-N,N,6-trimethylpyridin-2-amine (10i)



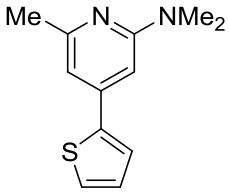
Prepared from (*Z*)-3-amino-1-(furan-2-yl)but-2-en-1-one (329 mg, 2.18 mmol) and DMADMA (0.642 mL, 4.40 mmol), 130 °C, 8 min. Column chromatography, Ethyl acetate : Petroleum ether = 1 : 5. Yield: 33 % (145 mg).

N,N,6-trimethyl-4-(thiophen-3-yl)pyridin-2-amine (10j)



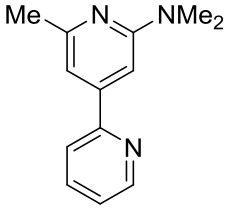
Prepared from (*Z*)-3-amino-1-(thiophen-3-yl)but-2-en-1-one (218 mg, 1.30 mmol), and DMADMA (0.380 mL, 2.60 mmol), 130 °C, 8 min. Column chromatography, Ethyl acetate : Hexane = 1 : 10. Yield: 42 % (120 mg).

N,N,6-trimethyl-4-(thiophen-2-yl)pyridin-2-amine (10k)



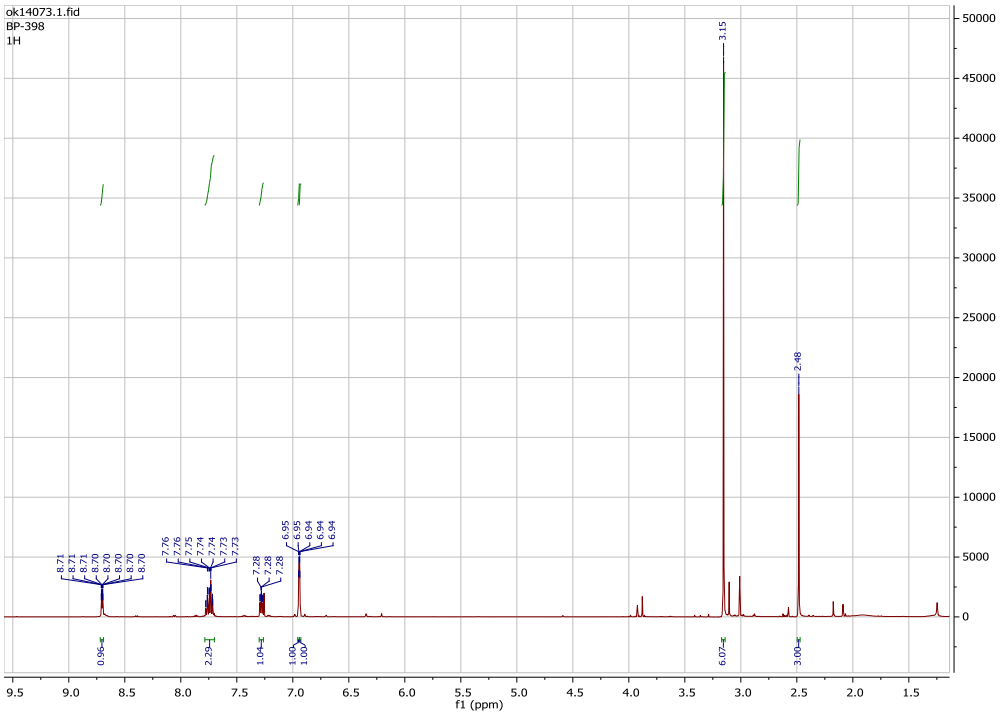
Prepared from (*Z*)-3-amino-1-(thiophen-2-yl)but-2-en-1-one (254 mg, 1.50 mmol) and DMADMA (0.438 mL, 3.00 mmol), 138 °C, 8 min. Column chromatography, Ethyl acetate : Petroleum ether = 1 : 5. Yield: 30 % (100 mg).

N,N,6'-trimethyl-[2,4'-bipyridin]-2'-amine (10l)

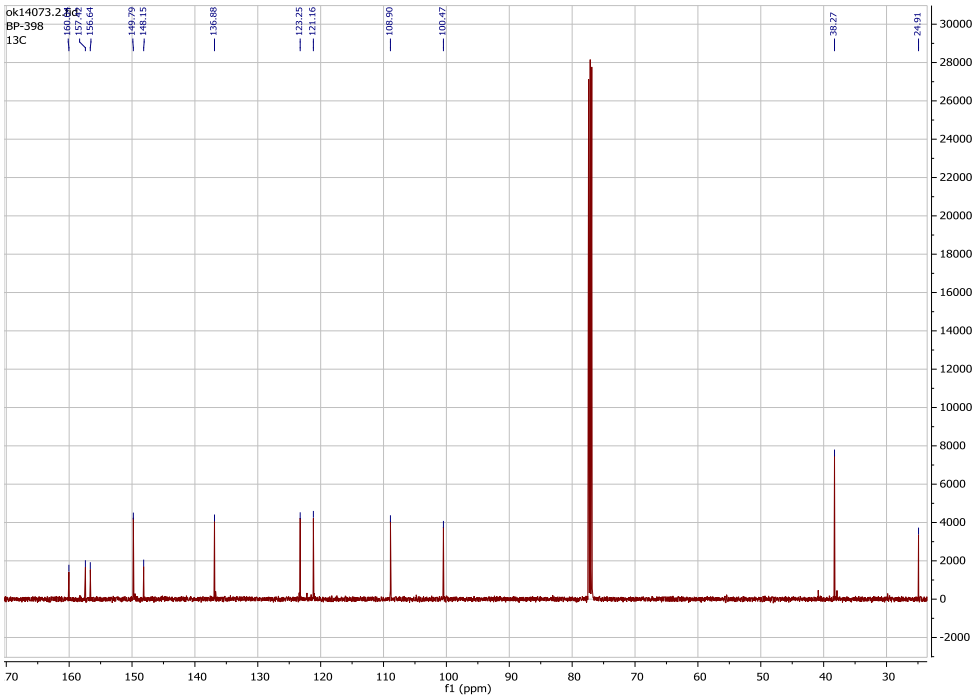


Prepared from (*Z*)-3-amino-1-(pyridin-2-yl)but-2-en-1-one (406 mg, 2.50 mmol) and DMADMA (0.730 mL, 5.00 mmol), 140 °C, 10 min. Column chromatography, Ethyl acetate : Petroleum ether = 1:3. Yield: 27 % (144 mg).

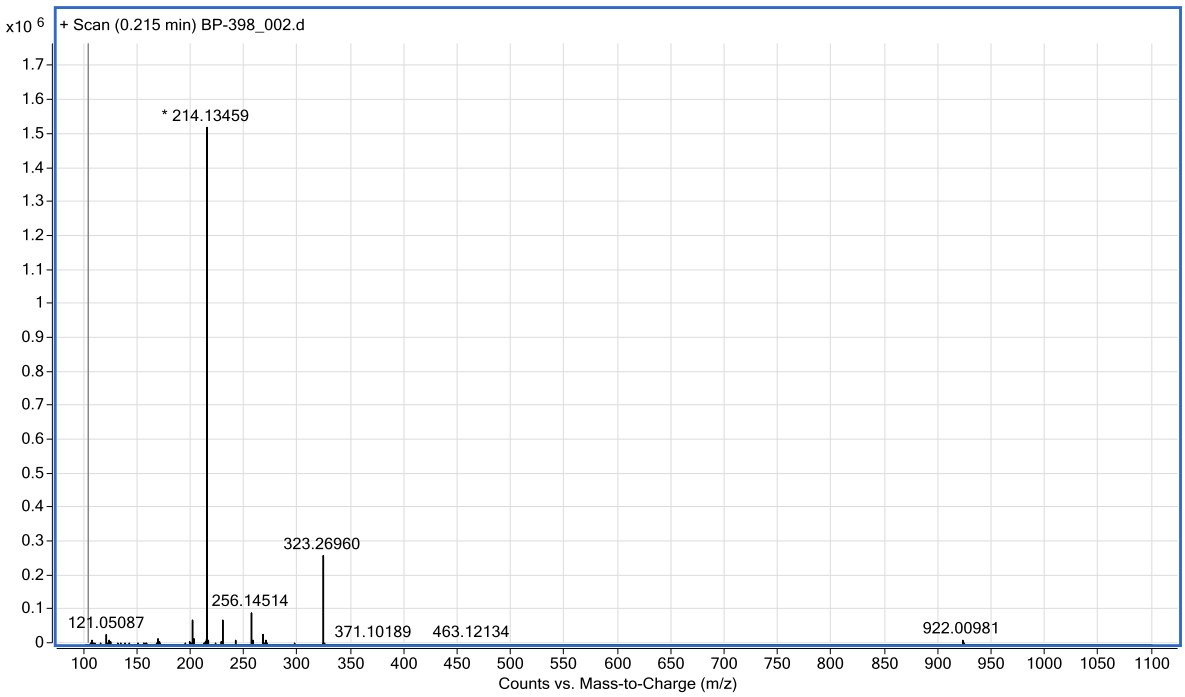
^{1}H NMR:



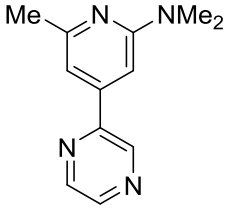
^{13}C NMR:


ok14073.2.fid
BP-398
13C
160.47
157.47
156.64
149.79
148.15
136.88
123.25
121.16
108.90
100.47
38.27
24.91
f1 (ppm)

MS:

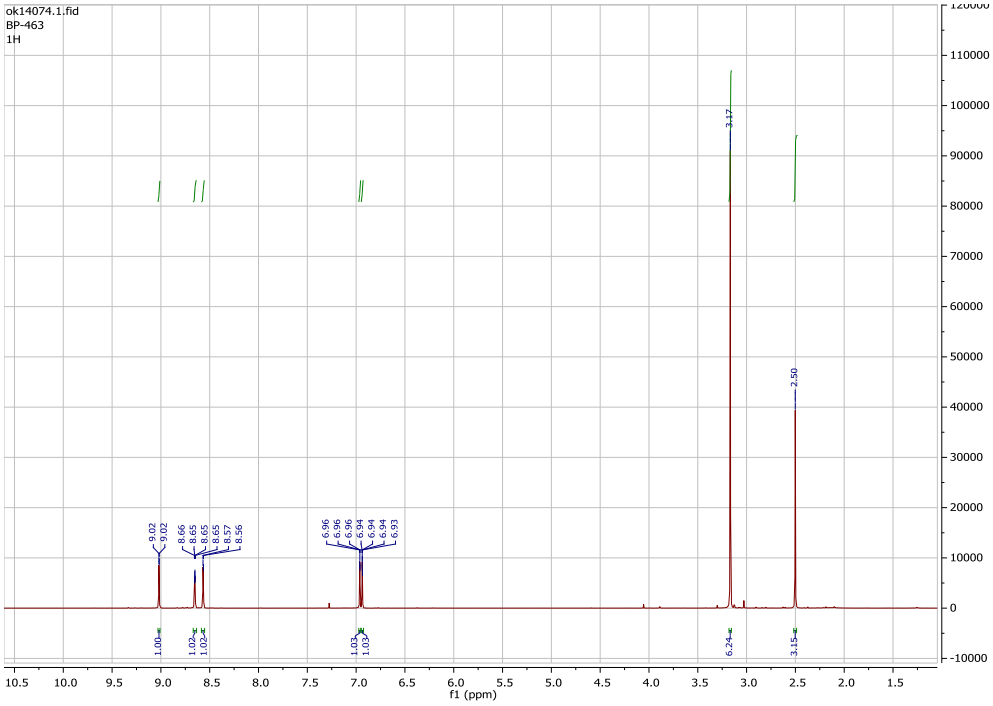

x10 6
+ Scan (0.215 min) BP-398_002.d
* 214.13459
323.26960
256.14514
121.05087
371.10189
463.12134
922.00981
Counts vs. Mass-to-Charge (m/z)

N,N,6-trimethyl-4-(pyrazin-2-yl)pyridin-2-amine (10m)

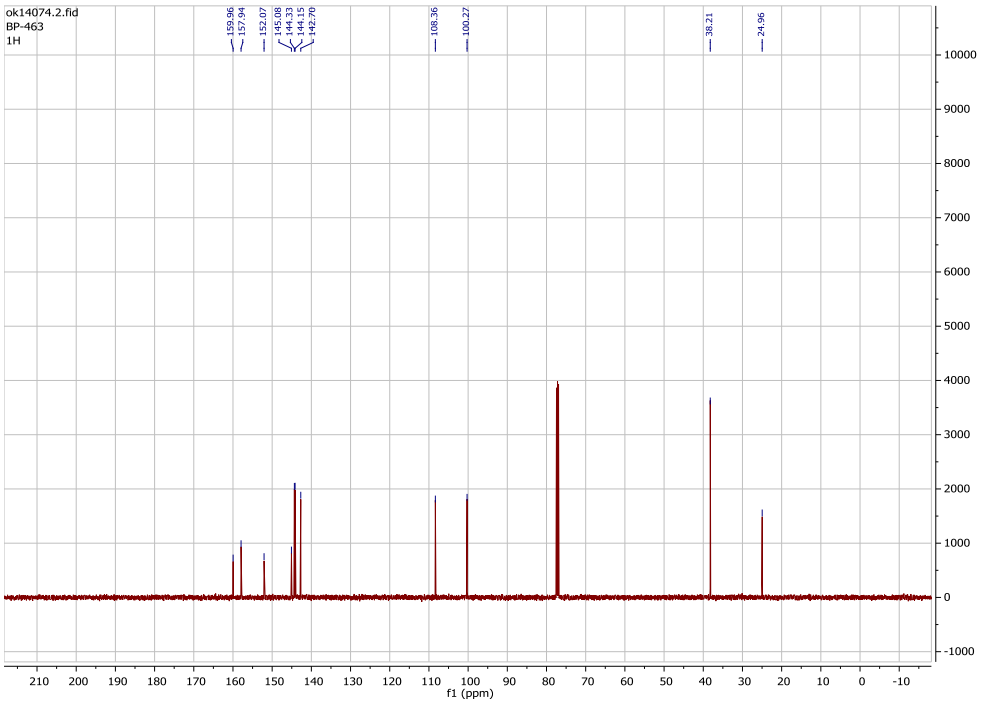


Prepared from (*Z*)-3-amino-1-(pyrazin-2-yl)but-2-en-1-one (322 mg, 2.00 mmol) and DMADMA (0.583 mL, 4.00 mmol), 138 °C, 8 min. Column chromatography, Ethyl acetate : Petroleum ether = 1 : 3. Yield: 24 % (104 mg).

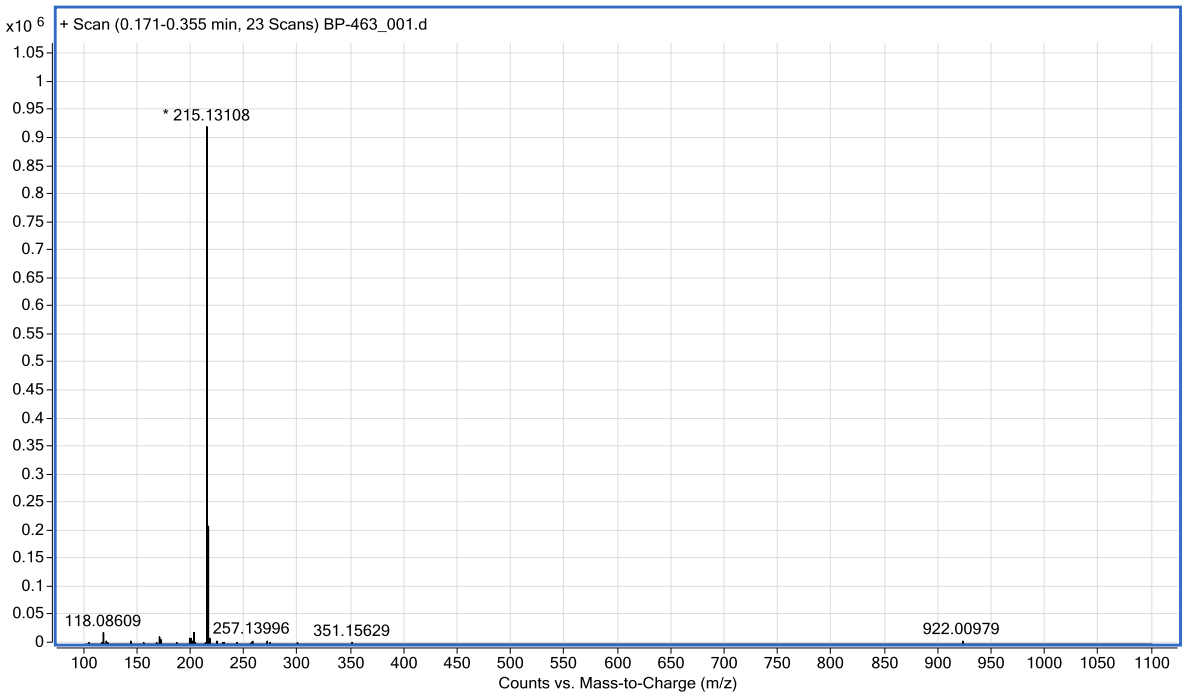
^{1}H NMR:



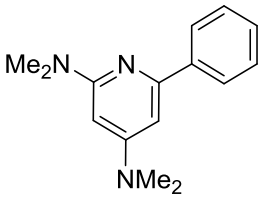
^{13}C NMR:


ok14074.2.fid
BP-463
1H
159.96
157.94
152.07
145.08
144.33
144.15
142.70
108.36
100.27
38.21
24.96
f1 (ppm)

MS:

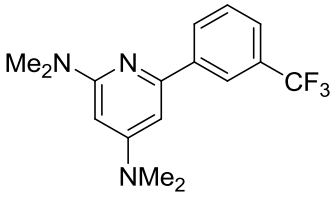

x10 6
+ Scan (0.171-0.355 min, 23 Scans) BP-463_001.d
* 215.13108
118.08609
257.13996
351.15629
922.00979
Counts vs. Mass-to-Charge (m/z)

N^2,N^2,N^4,N^4-tetramethyl-6-phenylpyridine-2,4-diamine (16a)



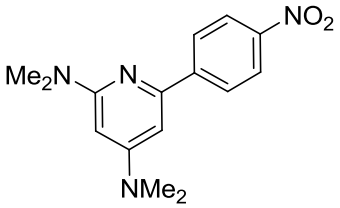
The product was prepared from benzamide (188 mg, 1.55 mmol), 140 °C, 10 min, column chromatography (Ethyl acetate : Petroleum ether = 1 : 1). Yield: 20 % (76 mg).

N^2,N^2,N^4,N^4-tetramethyl-6-(3-(trifluoromethyl)phenyl)pyridine-2,4-diamine (16b)



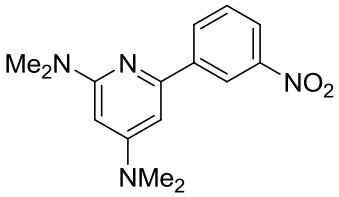
The product was prepared from 3-(trifluoromethyl)benzamide (379 mg, 2.00 mmol), 100 °C, 10 min, column chromatography (Ethyl acetate : Petroleum ether = 1 : 5). Yield: 31 % (193 mg).

N^2,N^2,N^4,N^4-tetramethyl-6-(4-nitrophenyl)pyridine-2,4-diamine (16c)



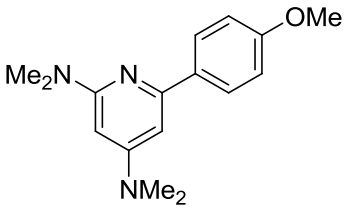
The product was prepared from 4-nitrobenzamide (498 mg, 3.00 mmol), 130 °C, 10 min, column chromatography (Ethyl acetate : Petroleum ether = 1 : 5). Yield: 19 % (160 mg).

N^2,N^2,N^4,N^4-tetramethyl-6-(3-nitrophenyl)pyridine-2,4-diamine (16d)



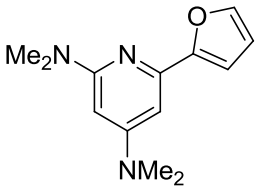
The product was prepared from 3-nitrobenzamide (498 mg, 3.00 mmol), 100 °C, 10 min, column chromatography (Ethyl acetate : Petroleum ether = 1 : 5). Yield: 26 % (223 mg).

6-(4-Methoxyphenyl)-N^2,N^2,N^4,N^4-tetramethylpyridine-2,4-diamine (16e)



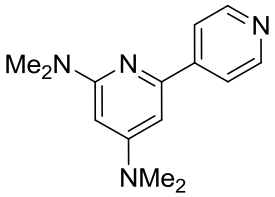
The product was prepared from 4-methoxybenzamide (453 mg, 3.00 mmol), 100 °C, 10 min, column chromatography (Ethyl acetate : Petroleum ether = 1 : 5). Yield: 19 % (154 mg).

6-(Furan-2-yl)-N^2,N^2,N^4,N^4-tetramethylpyridine-2,4-diamine (16f)



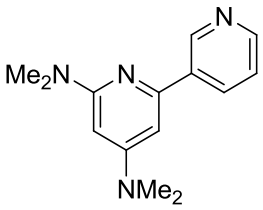
The product was prepared from furan-2-carboxamide (140 mg, 0.80 mmol), 140 °C, 10 min, column chromatography (Ethyl acetate : Petroleum ether = 1 : 5). Yield: 29 % (247 mg).

N^4,N^4,N^6,N^6-tetramethyl-2,4'-bipyridine-4,6-diamine (16g)



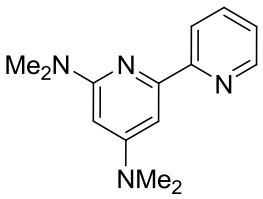
The product was prepared from isonicotinamide (366 mg, 3.00 mmol), 100 °C, 10 min, column chromatography (Ethyl acetate : Petroleum ether = 1 : 4). Yield: 38 % (276 mg).

N^4,N^4,N^6,N^6-tetramethyl-2,3'-bipyridine-4,6-diamine (16h)



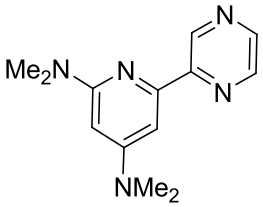
The product was prepared from nicotinamide (424 mg, 3.00 mmol), 130 °C, 10 min, column chromatography (Ethyl acetate). Yield: 24 % (200 mg).

N^4,N^4,N^6,N^6-tetramethyl-2,2'-bipyridine-4,6-diamine (16i)



The product was prepared from 2-pyridinecarboxamide (366 mg, 3.00 mmol), 110 °C, 10 min, column chromatography (Ethyl acetate : Petroleum ether = 1 : 1). Yield: 14 % (102 mg).

N^2,N^2,N^4,N^4-tetramethyl-6-(pyrazin-2-yl)pyridine-2,4-diamine (16j)



The product was prepared from pyrazinecarboxamide (369 mg, 3.00 mmol), 110 °C, 10 min, column chromatography (Ethyl acetate : Petroleum ether = 1 : 2). Yield: 36 % (266 mg).

Crystalographic data for Pyridines

Crystal data	**16a**	**16b**	**16g**	**16i**	**16j**
Formula	$C_{15}H_{19}N_3$	$C_{16}H_{18}F_3N_3$	$C_{14}H_{18}N_4$	$C_{14}H_{18}N_4$	$C_{13}H_{17}N_5$
M_r	241.33	309.33	242.32	242.32	243.32
Cell setting, space group	Monoclinic, $P12_1/c1$	Triclinic, $P-1$	Orthorhombic, $Fdd2$	Monoclinic, $P12_1/c1$	Orthorhombic, $Cmcm$
a (Å)	15.3125(4)	5.4406(2)	16.4792(10)	6.1248(2)	6.7919(4)
b (Å)	7.2109(2)	12.2175(7)	14.0329(7)	28.0478(11)	11.1592(4)
c (Å)	24.5888(6)	12.7096(7)	10.9681(6)	7.9067(3)	16.7932(8)
α (°)	90	104.016(5)	90	90	90
β (°)	102.956(2)	101.955(4)	90	107.795(4)	90
γ (°)	90	94.097(4)	90	90	90
V (Å^3)	2645.90(12)	795.27(7)	2536.4(2)	1293.28(8)	1272.79(11)
Z	8	2	8	4	4
D_x (Mg m^{-3})	1.212	1.292	1.269	1.245	1.270
μ (mm^{-1})	0.074	0.103	0.079	0.078	0.081
F(000)	1040	324	1040	520	520
Crystal form, colour	Prism, brown	Prism, colourless	Prism, yellow	Prism, brown	Platelet, yellow
Crystal size (mm)	0.45×0.35×0.20	0.50×0.40×0.30	0.40×0.30×0.25	0.40×0.25×0.20	0.30×0.20×0.03
Data collection					
T (K)	150	293	150	150	293

Radiation type, wavelength	Mo Kα, λ=0.7107 Å	Mo Kα, λ=0.7107 Å	Mo Kα, λ=0.7107 Å	Mo Kα, λ=0.7107 Å	Mo Kα, λ=0.7107 Å
Diffractometer	SuperNova-Atlas	SuperNova-Atlas	SuperNova-Atlas	SuperNova-Atlas	SuperNova-Atlas
Data collection method	ω scans	ω scans	ω scans	ω scans	ω scans
Absorption correction	Multi-scan	Multi-scan	Multi-scan	Multi-scan	Multi-scan
No. of measured, independent and observed reflections	15804, 6066, 4781	6214, 3246, 1912	12835, 1452, 1348	6959, 2960, 2355	6827, 820, 619
Criterion for observed reflections	>2sigma(I)	>2sigma(I)	>2sigma(I)	>2sigma(I)	>2sigma(I)
R_{int}	0.0237	0.0177	0.0387	0.0182	0.0240
θ range (°)	2.87−27.48	3.38−26.37	3.81−27.48	2.80−27.48	3.51−27.48
h range	−19 → 15	−6 → 6	−21 → 21	−7 → 5	−8 → 8
k range	−8 → 9	−15 → 15	−18 → 18	−36 → 33	−14 → 14
l range	−31 → 31	−12 → 15	−14 → 14	−10 → 10	−21 → 21
Refinement					
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
R (on F_{obs}), wR (on F_{obs}), S	0.0523, 0.1324, 1.020	0.1018, 0.3035, 1.111	0.0430, 0.1174, 0.975	0.0476, 0.1338, 1.097	0.0528, 0.1489, 1.122
No. of contributing reflections	6066	3246	1452	2960	820

No. of parameters	333	197	86	167	61
No. of restraints	0	6	1	0	0
$(\Delta/\sigma)_{max}$, $(\Delta/\sigma)_{ave}$	0.006, 0.000	0.003, 0.000	0.026, 0.002	0.000, 0.000	0.000, 0.000
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (eÅ^{-3})	0.235, −0.245	0.441, −0.394	0.260, −0.216	0.266, −0.196	0.131, −0.220