Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Chemoselective, Osmium-Free, Dihydroxylation / Oxidative Cleavage of Heteroaryl Isoprenes by a Contemporary Malaprade Reaction

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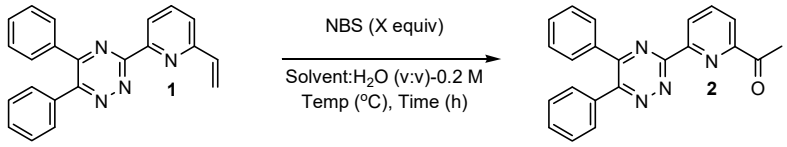
Experimental Section

General Considerations: All reagents were purchased from U.S. chemical suppliers, stored according to published protocols, and used as received unless indicated otherwise. All experiments were performed in oven-dried glassware. Reaction progress was monitored using thin-layer chromatography on glass-backed silica gel plates and/or ^{1}H NMR analysis of crude reaction mixtures. R_f values for compounds that resulted in a concentrically observed spot on normal phase silica gel, or alumina are reported using the conditions listed. Melting point data listed is for a single, uncorrected experiment. All reported yields listed are for pure compounds and corrected for residual solvent, if applicable, from ^{1}H NMR spectroscopy. Infrared spectral data was acquired from the (form) listed. All ^{1}H and ^{13}C NMR data was acquired from a 500 MHz multinuclear spectrometer with broad-band N_2 cryoprobe. Chemical shifts are reported using the δ scale and are referenced to the residual solvent signal: $CDCl_3$ (δ 7.26), $(CD_3)_2C{=}O$ (δ 2.05), and $(CD_3)_2S{=}O$ (δ 2.50) for ^{1}H NMR and $CDCl_3$ (δ 77.15), $(CD_3)_2C{=}O$ (δ 29.84), $(CD_3)_2S{=}O$ (δ 39.52), and CD_3CN (δ 1.32) for ^{13}C NMR.[1] Splittings are reported as follows: (br) = broad, (s) = singlet, (d) = doublet, (t) = triplet, (dd) = doublet of doublets, and (m) = multiplet. ^{13}C NMR spectra were corrected for ring down using linear back prediction. High resolution mass spectrometry (HRMS) data was obtained utilizing electron impact ionization (EI) with a magnetic sector (EBE trisector), double focusing-geometry mass analyzer. In regards to analysis of samples via Qtof in positive ion mode, the following parameters were utilized: Capillary voltage 0.5–1.5 KV, Sampling cone 40 V, Source offset 60, Source temperature 80 °C, Desolvation 350 °C, Cone gas 30 L/h, Desolvation gas 600 L/h, lock spray capillary voltage 0.2KV, lock mass: 556.2771 in positive mode (leucine enkephalin).

General Procedure for the Synthesis of Methyl MTPhen Derivatives

Table S1. Summary of NBS-mediated Oxidation Conditions



Entry	NBS (equiv)	Solvent	Temp (°C)	Time (h)	Conversion (%)[a]
1	1.2	EtOAc (10:1)	75	18	56 (**38**)[b]
2	1.2	THF (10:1)	66	18	0
3	1.2	MeCN (10:1)	82	18	31
4	1.2	MeOH (10:1)	65	18	0
5	1.2	$CHCl_3$ (10:1)	62	18	trace
6[c]	1.2	EtOAc (10:1)	75	18	50
7[d]	1.2	EtOAc (10:1)	75	18	81 (**42**)[b]

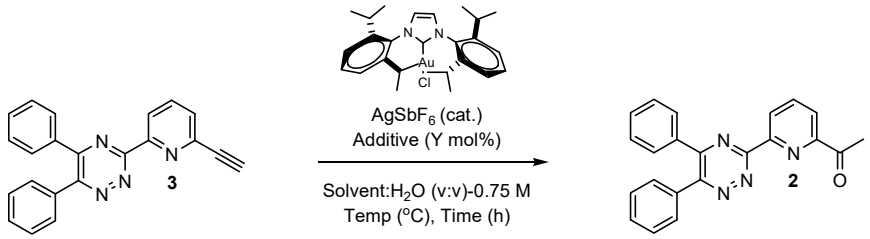
[a]Determined via integration of crude 1H-NMR spectrum without internal standard

[b]Purified, isolated yield (average of 3 experiments)

[c]Drop-wise addition of NBS over 2 hours as a solution in MeCN

[d]Brand-new NBS utilized

Table S2. Summary of Au-mediated Alkyne Hydration Strategies



Entry	[(iPr)AuCl]	Additive (mol%)	Solvent	Temp (°C)	Time (h)	Conversion (%)[b]
1	100 ppm	-----	MeOH (100:0)	65	20	0
2	500 ppm	-----	MeOH (2:1)	65	20	0
3	5 mol%	-----	MeOH (2:1)	65	20	15
4	5 mol%	-----	1,4-dioxane (3:1)	120[c]	16	15
5	5 mol%	*p*-TsOH (5 mol%)	MeOH (2:1)	80[c]	20	48
6	5 mol%	*p*-TsOH (5 mol%)	MeOH (10:1)	80[c]	20	50
7	5 mol%	*p*-TsOH (5 mol%)	MeOH (10:1)	80[c]	40	50
8	5 mol%	*p*-TsOH (5 mol%)	MeOH (10:1)	85[c]	20	50 (**38**)[d]
9	5 mol%	*p*-TsOH (5 mol%)	Toluene (10:1)	85[c]	20	0
10	5 mol%	*p*-TsOH (10 mol%)	MeOH (10:1)	85[c]	20	37
11	10 mol%	*p*-TsOH (10 mol%)	MeOH (10:1)	85[c]	20	42

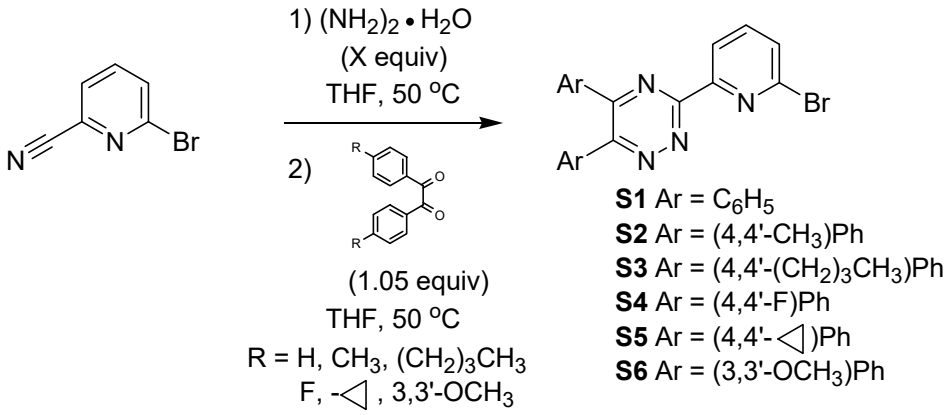
[a]Tip of spatula amount used

[b]Determined via integration of crude ^{1}H-NMR spectrum without internal standard

[c]Reaction performed in a tightly sealed vessel

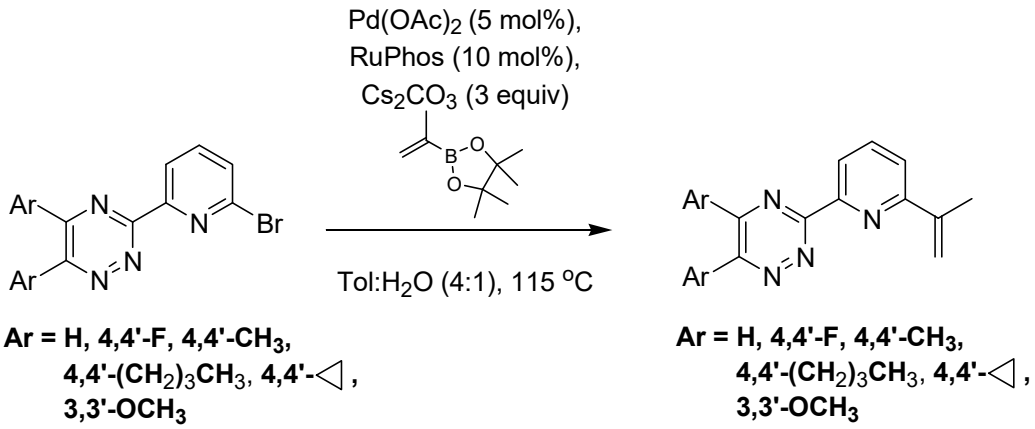
[d]Purified, isolated yield

Scheme S1. Preparation of Br-MTP Starting Materials

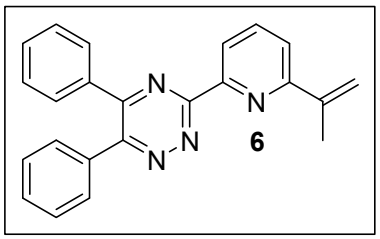


For a general procedure for the construction of **S1**–**S6** above, an adaptation of a literature procedure was employed.[2] The requisite Suzuki-Miyaura cross-coupling of 4,4'-dibromobenzil with potassium butyltrifluoroborate afforded the 4,4'-dibutylbenzyl used in the condensation sequence described above for accessing **S3**.

Scheme S2. General procedure for the Suzuki-Miyaura Cross-Coupling of Scaffolds

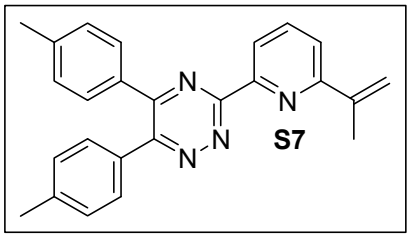


An adaptation of a literature procedure was employed.[3] To a 100 mL round-bottom flask equipped with a magnetic stirring bar at ambient temperature was charged the requisite functionalized bromopyridine (2.0000 g, 5.144 mmol, 1.00 equiv), followed by palladium(II) acetate (0.0577 g, 0.257 mmol, 0.05 equiv), 2-dicyclohxylphosphino-2',6'-diisopropoxybiphenyl (RuPhos) (0.2400 g, 0.514 mmol, 0.10 equiv), and Cs_2CO_3 (5.0280 g, 15.432 mmol, 3.00 equiv). The flask was then fitted with a rubber septum and purged with dry nitrogen for five minutes using a gas bubbler. After purging with inert atmosphere, the solids were slurried in a 4:1 (v:v) mixture of toluene:H_2O (25.72 mL, 0.20 M final concentration) and stirred for 10 minutes at ambient temperature. 2-Isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane, or analogous coupling reagent, (1.037 g, 6.172 mmol, 1.20 equiv) was then added under a stream of nitrogen and the contents heated to 110 °C for 16 hours. TLC results indicating consumption of starting material were subsequently benchmarked by ^{1}H NMR spectroscopy to determine relative ratios of starting material to product, as well as to assess the relevant impurity profile for purification. The crude reaction mixtures were directly adsorbed on silica gel (following removal of aqueous phase) under reduced pressure and purified using automated flash column chromatography with normal phase silica gel columns to afford the title compounds in the morphologies indicated after concentration *in vacuo* at ambient temperature.

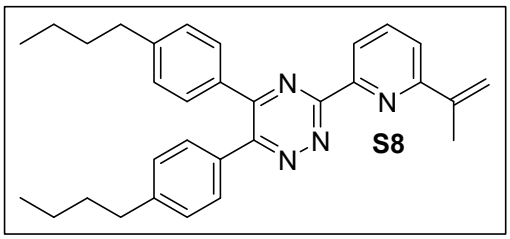


3-(6-Isopropenyl-pyridin-2-yl)-6-(1-methylene-but-2-enyl)-5-phenyl-[1,2,4]triazine (**6**). Prepared according to the general procedure discussed above input of **S1** (2.0080 g, 5.165 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0579 g, 0.2585 mmol, 0.05 equiv), RuPhos (0.2410 g, 0.5165 mmol, 0.10 equiv), Cs_2CO_3 (5.0486 g, 15.495 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.0414 g, 6.198 mmol, 1.20 equiv), R_f = 0.35, 20% ethylacetate:hexanes; purified using automated flash column chromatography on a normal phase silica gel column with a methyl-*tert*-butyl ether:hexanes gradient mobile phase; isolated yield 1.7180 g, 95%; yellow solid; melting point = 165.9–177.3 °C; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 8.50 (d, *J* = 7.7 Hz, 1H), 8.04 (t, *J* = 7.8 Hz, 1H), 7.86 (d, *J* = 7.8 Hz, 1H), 7.76–7.72 (m, 2H), 7.70–7.66 (m, 2H), 7.53–7.48 (m, 2H), 7.47–7.41 (m, 4H), 6.14 (s, 1H), 5.44–5.41 (m, 1H), 2.33 (s, 3H) ; ^{13}C{^{1}H} NMR (125 MHz, $(CD_3)_2CO$): δ = 162.0, 159.2, 157.4, 156.6, 153.4, 144.2, 138.4, 137.1, 137.0, 131.5, 130.9, 130.5, 130.4, 129.4, 129.3, 123.3, 121.9, 116.5, 20.6 ; IR (ATR-solid):

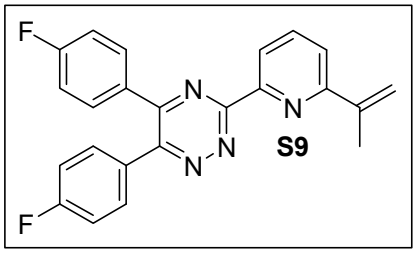
$\bar{\upsilon}_{max}$ = 3048, 2976, 2955, 2921, 1581, 1565, 1491, 1440, 1358, 775, 763, 697 cm^{-1}; HRMS (EI): m/z: $[M]^+$ Calcd for $C_{23}H_{18}N_4$ 350.1531; Found: 350.1519.



3-(6-Isopropenyl-pyridin-2-yl)-5,6-di-p-tolyl-[1,2,4]triazine (**S7**). Prepared according to the general procedure discussed above input of **S2** (1.1080 g, 2.663 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0299 g, 0.1331 mmol, 0.05 equiv), RuPhos (0.1243 g, 0.2663 mmol, 0.10 equiv), Cs_2CO_3 (2.6030 g, 7.989 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.0414 g, 3.1956 mmol, 1.20 equiv), R_f = 0.40, 20% ethylacetate:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with an ethyl acetate:hexanes gradient mobile phase ; isolated yield 0.5640 g, 56%; peach colored solid ; melting point = 154,2–155.6 °C; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 8.49 (d, *J* = 7.7 Hz, 1H), 8.03 (t, *J* = 7.8 Hz, 1H), 7.84 (d, *J* = 7.9 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 2H), 7.58 (d, *J* = 7.9 Hz, 2H), 7.28 (d, *J* = 7.8 Hz, 2H), 7.25 (d, *J* = 7.9 Hz, 2H), 6.14 (s, 1H), 5.42 (s, 1H), 2.40 (s, 3H), 2.38 (s, 3H), 2.33 (s, 3H), ; $^{13}C\{^1H\}$ NMR (125 MHz, $(CD_3)_2CO$): δ = 161.7, 159.1, 157.1, 156.3, 153.5, 144.2, 141.9, 140.5, 138.3, 134.27, 134.25, 130.8, 130.3, 130.1, 130.0, 123.2, 121.8, 116.5, 21.4, 21.3, 20.6 ; IR (ATR-solid): $\bar{\upsilon}_{max}$ = 3032, 2921, 1699, 1609, 1583, 1493, 1384, 1360, 1241, 1185, 1091, 911, 820, 733, 592, 538 cm^{-1}; HRMS (EI): m/z: $[M]^+$ Calcd for $C_{25}H_{22}N_4$ 378.1844; Found: 378.1847.

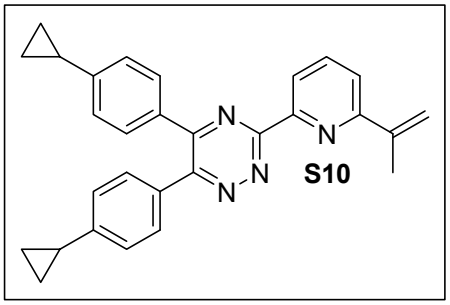


5,6-Bis-(4-butyl-phenyl)-3-(6-isopropenyl-pyridin-2-yl)-[1,2,4]triazine (**S8**). Prepared according to the general procedure discussed above input of **S3** (0.6169 g, 1.233 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0138 g, 0.0616 mmol, 0.05 equiv), RuPhos (0.0575 g, 0.1233 mmol, 0.10 equiv), Cs_2CO_3 (1.2052 g, 3.699 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.2487 g, 1.480 mmol, 1.20 equiv), R_f = 0.53, 20% ethyl acetate:hexanes; purified using automated flash column chromatography on a normal phase silica gel column with an ethyl acetate:methyl-*tert*-butyl ether (1:1):hexanes gradient mobile phase ; isolated yield 0.302 g g, 53%; yellow crystalline solid; melting point = 50.1–51.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.53 (d, *J* = 7.8 Hz, 1H), 7.88 (t, *J* = 7.8 Hz, 1H), 7.69–7.64 (m, 3H), 7.58 (d, *J* = 7.5 Hz, 2H), 7.21 (d, *J* = 7.5 Hz, 1H), 7.17 (d, *J* = 7.6 Hz, 2H), 6.10 (s, 1H), 5.42 (s, 1H), 2.66 (t, *J* = 8.8 Hz, 2H), 2.64 (t, *J* = 8.8 Hz, 2H), 2.36 (s, 3H), 1.67–1.56 (m, 4H), 0.98–0.86 (m, 6H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): δ = 160.7, 158.9, 156.2, 155.8, 152.3, 146.3, 145.0, 143.1, 137.4, 133.3, 133.0, 130.0, 129.5, 128.8, 128.7, 122.5, 121.3, 116.7, 35.67, 35.63, 33.44, 33.35, 22.47, 22.42, 20.6, 14.08, 14.06; IR (ATR-solid): $\bar{\upsilon}_{max}$ = 2956, 2929, 2858, 1609, 1582, 1492, 1382, 1360, 831, 733 cm^{-1}; HRMS (EI): m/z: $[M]^+$ Calcd for $C_{31}H_{34}N_4$ 462.2783; Found: 462.2795.

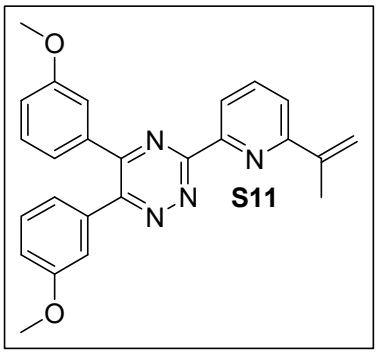


5,6-Bis-(4-fluoro-phenyl)-3-(6-isopropenyl-pyridin-2-yl)-[1,2,4]triazine (**S9**). Prepared according to the general procedure discussed above input of **S4** (0.6906 g, 1.629 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0183 g, 0.0814 mmol, 0.05 equiv), RuPhos (0.0760 g, 0.163 mmol, 0.10 equiv), Cs_2CO_3 (1.5923 g, 4.887 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.3285 g, 1.955 mmol, 1.20

equiv), R_f = 0.33, 20% ethyl acetate:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with an ethyl acetate:methyl-*tert*-butyl ether (1:1):hexanes gradient mobile phase ; isolated yield 0.192 g, 64%; yellow solid; melting point = 163.5–164.9 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.55 (d, *J* = 7.8 Hz, 1H), 7.93 (t, *J* = 7.8 Hz, 1H), 7.78–7.73 (m, 2H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.69–7.64 (m, 2H), 7.15–7.06 (m, 4H), 6.11 (s, 1H), 5.44 (m, 1H), 2.36 (s, 3H) ; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ = 165.3 (*J* = 330.6 Hz), 163.3, 160.9, 159.0, 155.2, 154.9, 151.8, 142.9, 137.7, 132.3 (*J* = 34.3 Hz), 131.7 (*J* = 12.9 Hz), 131.6 (*J* = 34.5 Hz), 131.5 (*J* = 13.7 Hz), 122.7, 121.7, 117.1, 116.1, 20.6 ; ^{19}F NMR (471 MHz, $(CD_3)_2SO$): δ = −109.9– −110.0 (m, 1F), −111.8– −111.9 (m, 1F) ; IR (ATR-solid): $\bar{\upsilon}_{max}$ = 3072, 2922, 2863, 1603, 1583, 1514, 1492, 1383, 1362, 1236, 1160, 841, 728, 668 cm^{-1}; HRMS (EI): m/z: $[M]^+$ Calcd for $C_{23}H_{16}F_2N_4$ 386.1343; Found: 386.1333.



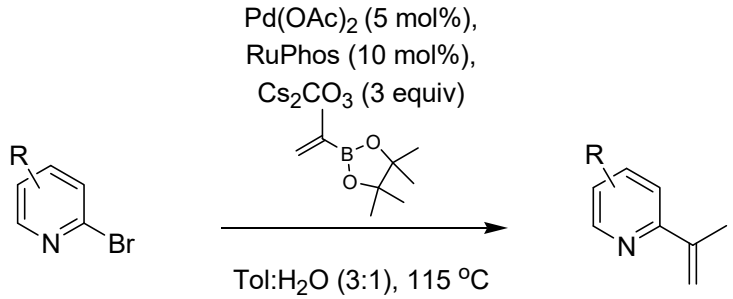
5,6-Bis-(4-cyclopropyl-phenyl)-3-(6-isopropenyl-pyridin-2-yl)-[1,2,4]triazine (**S10**). Prepared according to the general procedure discussed above input of **S5** (0.0981 g, 0.210 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0024 g, 0.001 mmol, 0.05 equiv), RuPhos (0.0098 g, 0.002 mmol, 0.10 equiv), Cs_2CO_3 (0.205 g, 0.629 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.0414 g, 6.198 mmol, 1.20 equiv), R_f = 0.38, 20% ethyl acetate:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with an ethyl acetate:methyl-*tert*-butyl ether (1:1):hexanes gradient mobile phase ; isolated yield 0.0514 g, 57%; yellow crystalline solid; melting point = 112.3–113.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.52 (d, *J* = 7.7 Hz, 1H), 7.88 (t, *J* = 7.8 Hz, 1H), 7.68–7.63 (m, 3H), 7.57 (d, *J* = 7.6 Hz, 2H), 7.08 (d, *J* = 7.7 Hz, 2H), 7.05 (d, *J* = 7.7 Hz, 2H), 6.10 (s, 1H), 5.41 (s, 1H), 2.35 (s, 3H0, 1.97–1.87 (m, 2H), 1.07–1.00 (m, 4H), 0.79–0.73 (m, 4H) ; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ = 160.6, 158.9, 155.9, 155.6, 152.3, 147.9, 146.3, 143.2, 137.4, 133.0, 132.7, 130.1, 129.5, 125.9, 125.7, 122.5, 121.3, 116.6, 20.6, 15.7, 15.6, 10.3, 10.0 ; IR (ATR-solid): $\bar{\upsilon}_{max}$ = 3081, 3004, 1699, 1609, 1492, 1384, 1362, 902, 826, 733 cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for $C_{29}H_{27}N_4$ 431.2236 ; Found: 431.2233.

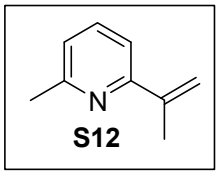


3-(6-Isopropenyl-pyridin-2-yl)-5,6-bis-(3-methoxy-phenyl)-[1,2,4]triazine (**S11**). Prepared according to the general procedure discussed above input of **S6** (0.5151 g, 1.1500 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0129 g, 0.0575 mmol, 0.05 equiv), RuPhos (0.0537 g, 0.115 mmol, 0.10 equiv), Cs_2CO_3 (1.1241 g, 3.450 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.0414 g, 6.198 mmol, 1.20 equiv) R_f = 0.18, 20% ethyl acetate:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with an ethyl acetate:methyl-*tert*-butyl ether (1:1):hexanes gradient mobile phase ; isolated yield 0.3442 g, 73%; yellow solid; melting point = 99.5–100.8 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.56 (dd, *J* = 0.7, 7.8 Hz, 1H), 7.91 (t, *J* = 7.9 Hz, 1H), 7.68 (dd, *J* = 0.8, 7.9 Hz, 1H), 7.37 (br-s, 1H), 7.31–7.30 (m, 1H), 7.27–7.24 (m, 3H), 7.17–7.14 (m, 1H), 7.02–6.96 (m, 2H), 6.10 (br-s, 1H), 5.44–5.41 (m, 1H), 3.78 (s, 3H), 3.75 (s, 3H), 2.36 (s, 3H) ; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ = 161.0, 159.9, 159.7, 158.9, 156.1, 155.8, 152.0, 143.1, 137.5, 137.0, 136.8, 129.8, 129.7, 122.6, 122.2, 121.5, 117.5, 116.7X (overlaps with 116.7), 116.7, 116.3, 114.7, 114.4,

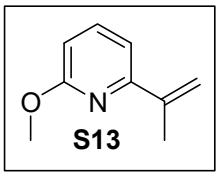
55.5, 55.4, 20.6 ; IR (ATR-solid): $\bar{\upsilon}_{max}$ = 3073, 3002, 2939, 2835, 1599, 1582, 1504, 1487, 1463, 1381, 1290, 1249, 1044, 817, 790, 703 cm^{-1}; HRMS (EI): m/z: $[M]^+$ Calcd for $C_{25}H_{22}N_4O_2$ 410.1743; Found: 410.1731.

Scheme S3. Suzuki-Miyaura Cross-Coupling of Functionalized Pyridines Utilized

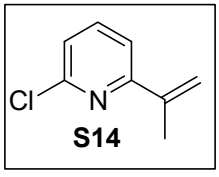




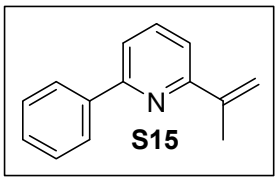
2-Isoprenyl-6-methylpyridine (**S12**). Prepared according to the general procedure discussed above input of 2-bromo-6-methylpyridine (1.427 g, 8.346 mmol). Spectroscopic data obtained was commensurate with that previously reported.[4]



2-Isopropenyl-6-methoxypyridine (**S13**). Prepared according to the general procedure discussed above via input of 2-bromo-6-methoxypyridine (0.2510 g, 1.343 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0150 g, 0.067 mmol, 0.05 equiv), RuPhos (0.0630 g, 0.135 mmol, 0.10 equiv), Cs_2CO_3 (1.312 g, 4.027 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.271 g, 1.613 mmol, 1.20 equiv), R_f = 0.70, 20% methyl *tert*-butyl ether:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with a methyl-*tert*-butyl ether:hexanes gradient mobile phase ; isolated yield 0.0852 g, 43%; colorless oil; ^1H NMR (500 MHz, $(CD_3)_2CO$): δ = 7.64 (dd, *J* = 7.6, 7.9 Hz, 1H), 7.13 (d, *J* = 7.4 Hz, 1H), 6.66 (d, *J* = 8.2 Hz, 1H), 6.01–5.99 (m, 1H), 5.25–5.23 (m, 1H), 3.91 (s, 3H), 2.16 (s, 3H) ; $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$): δ = 163.1, 155.5, 142.6, 139.0, 115.5, 112.5, 109.5, 53.2, 20.4 ; IR (ATR-liquid): $\bar{\upsilon}_{max}$ = 3091, 2977, 2949, 2926, 2849, 1463, 1413, 1576, 1318, 1255, 1030, 808 cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for $C_9H_{12}NO$ 150.0919; Found: 150.0917.

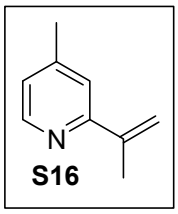


2-Isoprenyl-6-methylpyridine (**S14**). Prepared according to the general procedure discussed above input of 2-bromo-6-chloropyridine (0.6018 g, 3.152 mmol). Spectroscopic data obtained was commensurate with that reported.[5]



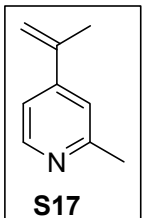
2-Isopropenyl-6-phenyl-pyridine (**S15**). Prepared according to the general procedure discussed above via input of 2-bromo-6-phenylpyridine (0.1200 g, 0.515 mmol), $Pd(OAc)_2$ (0.0060 g, 0.0267 mmol, 0.05 equiv), RuPhos (0.024 g, 0.051 mmol, 0.10 equiv), Cs_2CO_3 (0.5000 g, 1.535 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.1030 g, 0.613 mmol, 1.20 equiv), R_f = 0.50, 2% methyl-*tert*-butyl ether ; purified using

automated flash column chromatography on a normal phase silica gel column with a methyl-*tert*-butyl ether:hexanes gradient mobile phase ; isolated yield 0.0450 g, 45%; light brown oil; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 8.18–8.15 (m, 2H), 7.86–7.80 (m, 2H), 7.58 (dd, *J* = 1.8, 6.7 Hz, 1H), 7.51–7.47 (m, 2H), 7.45–7.39 (m, 1H), 6.04–6.01 (m, 1H), 5.36–5.34 (m, 1H), 2.29–2.27 (m, 3H) ; ^{13}C{^{1}H} NMR (125 MHz, $(CD_3)_2CO$): δ = 158.4, 156.4, 144.5, 140.3, 138.2, 129.7, 129.5, 127.5, 119.3, 118.9, 115.7, 20.6 ; IR (ATR-liquid): $\bar{\upsilon}_{max}$ = 3064, 2976, 2921, 2853, 1372, 1567, 1458, 1444, 904, 822, 769, 747, 695 cm^{-1}; HRMS (EI): m/z: $[M]^+$ Calcd for $C_{14}H_{14}N$ 196.1126; Found: 196.1129.



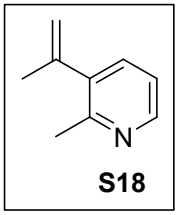
S16

2-Isopropenyl-4-methyl-pyridine (**S16**). Prepared according to the general procedure discussed above via input of 2-bromo-4-methylpyridine (0.257 g, 1.503 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.017 g, 0.076 mmol, 0.05 equiv), RuPhos (0.070 g, 0.150 mmol, 0.10 equiv), Cs_2CO_3 (1.469 g, 4.509 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.3030 g, 1.803 mmol, 1.20 equiv), R_f = 0.50, 15% methyl *tert*-butyl ether:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with a methyl-*tert*-butyl ether :hexanes gradient mobile phase ; isolated yield 0.1281 g, 64%; yellow oil; ^{1}H NMR (500 MHz, CD_3CN): δ = 8.29 (d, *J* = 5.0 Hz, 1H), 7.37 (s, 1H), 7.01 (d, *J* = 4.6 Hz, 1H), 5.83 (s, 1H), 5.19–5.16 (m, 1H), 2.26 (s, 3H), 2.08–2.06 (m, 3H) ; ^{13}C{^{1}H} NMR (125 MHz, $(CD_3)_2CO$): δ = 158.1, 149.0, 148.9, 143.8, 124.1, 121.5, 116.0, 21.0, 20.6 ; IR (ATR-liquid): $\bar{\upsilon}_{max}$ = 3088, 3052, 2977, 2921, 1596, 1558, 1467, 1451, 901, 827 cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for $C_9H_{12}N$ 134.0970; Found: 134.0965.



S17

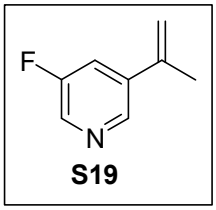
4-Isopropenyl-2-methyl-pyridine (**S17**). Prepared according to the general procedure discussed above via input of 4-bromo-2-methylpyridine (0.257 g, 1.503 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.017 g, 0.076 mmol, 0.05 equiv), RuPhos (0.070 g, 0.150 mmol, 0.10 equiv), Cs_2CO_3 (1.469 g, 4.509 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.3030 g, 1.803 mmol, 1.20 equiv); R_f = 0.40, 35% ethyl acetate:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with an ethyl acetate:hexanes gradient mobile phase ; isolated yield 0.146 g, 73%; amber oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.46–8.42 (m, 1H), 7.25–7.16 (m, 2H), 5.56 (br-d, *J* = 4.3 Hz, 1H), 5.30–5.25 (m, 1H), 2.60 (d, *J* = 6.4 Hz, 3H), 2.13 (s, 3H) ; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ = 157.9, 149.7, 148.1, 141.0, 120.1, 117.7, 116.3, 23.9, 20.9 ; IR (ATR-liquid): $\bar{\upsilon}_{max}$ = 3087, 2975, 2923, 2857, 1598, 1542, 1442, 1406, 1381, 905, 840 cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for $C_9H_{12}N$ 134.0970; Found: 134.0965.



S18

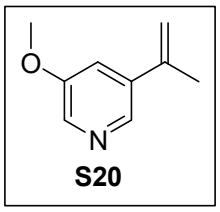
3-Isopropenyl-2-methyl-pyridine (**S18**). Prepared according to the general procedure discussed above via input of 3-bromo-2-methylpyridine pyridine (0.257 g, 1.503 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.017 g, 0.076 mmol, 0.05 equiv), RuPhos (0.070 g, 0.150 mmol, 0.10 equiv), Cs_2CO_3 (1.469 g, 4.509 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.3030 g, 1.803 mmol, 1.20 equiv), R_f = 0.33, 5% methanol:dichloromethane ; purified using automated flash column chromatography on a normal phase silica gel column with a methyl-*tert*-butyl ether pentane gradient mobile phase ; isolated yield 0.416 g, 57%; yellow powder; melting point = 175.6–176.0 °C; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 8.35 (d, *J* = 4.6 Hz, 1H), 7.46–7.42 (m, 1H), 7.14 (dd, *J* = 4.9, 7.5 Hz, 1H), 5.26 (s, 1H), 4.89 (s, 1H), 2.47 (s, 3H), 2.05 (s, 3H)-overlaps with residual $(CH_3)_2CO$; ^{13}C{^{1}H} NMR (125 MHz, $(CD_3)_2CO$): δ = 155.5, 148.4, 145.2, 139.2, 135.9,

121.7, 116.2, 24.1, 23.0 ; IR (ATR-solid): $\bar{\upsilon}_{max}$ = 3083, 3046, 2974, 2921, 2854, 1638, 1567, 1458, 1432, 1373, 1095, 901, 808, 742, cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for $C_9H_{12}N$ 134.0970; Found: 134.00967.



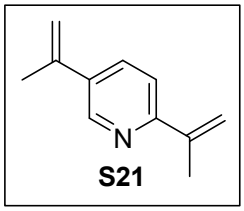
S19

3-Fluoro-5-isopropenyl-pyridine (**S19**). Prepared according to the general procedure discussed above via input of 3-bromo-5-fluoro-pyridine (0.3000 g, 1.712 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0193 g, 0.086 mmol, 0.05 equiv), RuPhos (0.0798 g, 0.171 mmol, 0.10 equiv), Cs_2CO_3 (1.6734 g, 5.136 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.3452 g, 2.054 mmol, 1.20 equiv), R_f = 0.48, 10% methyl *tert*-butylether:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with a methyl-*tert*-butyl ether:hexanes gradient mobile phase ;isolated yield 0.1904 g, 69%; clear oil; ^1H NMR (500 MHz, CD_3CN): δ = 8.59–8.55 (m, 1H), 8.38 (d, *J* = 2.7 Hz, 1H), 7.62 (ddd, *J* = 1.9, 2.6, 10.5 Hz, 1H), 5.54 (s, 1H), 5.29–5.25 (m, 1H), 2.16, (br-s, 3H) ; $^{13}C\{^1H\}$ NMR (125 MHz, CD_3CN): δ = 160.6 (d, *J* = 252.2 Hz), 144.0 (d, *J* = 2.4 Hz), 140.7, 139.2 (d, *J* = 3.6 Hz), 137.4 (d, *J* = 23.1 Hz), 120.3 (d, *J* = 18.6 Hz), 116.1, 21.5 ; ^{19}F NMR (471 MHz, CD_3CN): δ = −130.0 (d, *J* = −5.6 Hz) ; IR (ATR-oil): $\bar{\upsilon}_{max}$ = 3733, 3627, 2598, 2923, 2853, 1457, 668 cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for C_8H_9FN 138.0719; Found: 138.0714.



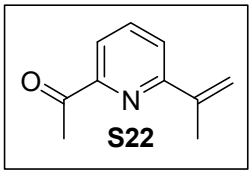
S20

3-Isopropenyl-5-methoxy-pyridine (**S20**). Prepared according to the general procedure discussed above via input of 3-bromo-5-methoxy-pyridine (0.2510 g, 1.343 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0150 g, 0.067 mmol, 0.05 equiv), RuPhos (0.0630 g, 0.135 mmol, 0.10 equiv), Cs_2CO_3 (1.312 g, 4.027 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.271 g, 1.613 mmol, 1.20 equiv), R_f = 0.40, 20% ethyl acetate:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with an ethyl acetate:hexanes gradient mobile phase ; isolated yield 0.1863 g, 93%; yellow oil; ^1H NMR (500 MHz, $(CD_3)_2CO$): δ = 8.33 (d, *J* = 1.3 Hz, 1H), 8.21 (d, *J* = 2.6 Hz, 1H), 7.41–7.39 (m, 1H), 5.51 (br-s, 1H), 5.22–5.20 (m, 1H), 3.92 (s, 3H), 2.19–2.17 (m, 3H) ; $^{13}C\{^1H\}$ NMR (125 MHz, $(CD_3)_2CO$): δ = 156.5, 141.6, 140.1, 137.9, 137.4, 117.6, 114.7, 55.9, 21.7 ; IR (ATR-liquid): $\bar{\upsilon}_{max}$ = 3093, 3044, 2968, 2941, 2841, 1629, 1585, 1567, 1453, 1417, 1305, 1254, 1048, 896, 871, 690 cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for $C_9H_{12}NO$ 150.0919; Found: 150.0922.

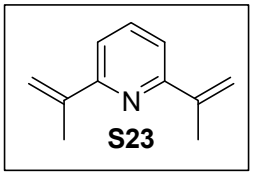


S21

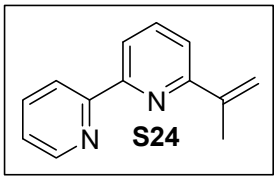
2,5-Diisopropenyl-pyridine (**S21**). Prepared according to the general procedure discussed above input of 2,5-dibromopyridine (0.2977 g, 1.257 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0281 g, 0.126 mmol, 0.05 equiv), RuPhos (0.1172 g, 0.251 mmol, 0.10 equiv), Cs_2CO_3 (1.2286 g, 3.771 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.423 g, 2.51 mmol, 1.20 equiv), R_f = 0.40, 1% methyl *tert*-butyl ether:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with a methyl-*tert*-butyl ether:hexanes gradient mobile phase ;isolated yield 0.1285 g, 61% over two steps from 2,5-dibromopyridine; red-orange oil; ^1H NMR (500 MHz, $CDCl_3$): δ = 8.72 (s, 1H), 7.77–7.71 (br-m, 1H), 7.49–7.45 (br-m, 1H0, 5.93–5.88 (br-m, 1H), 5.44 (s, 1H), 5.35–5.30 (br-m, 1H), 5.17 (s, 1H), 2.30 (s, 3H), 2.17 (s, 3H) ; $^{13}C\{^1H\}$ NMR (125 MHz, $(CD_3)_2CO$): δ = 157.6, 146.8, 144.0, 141.4, 135.5, 133.7, 119.7, 115.4, 114.0, 21.5, 20.5 ; IR (ATR-liquid): $\bar{\upsilon}_{max}$ = 3088, 2973, 2921, 2853, 1628, 1589, 1547, 1479, 1456, 1380 cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for $C_{11}H_{14}N$ 160.1126; Found: 160.1133.



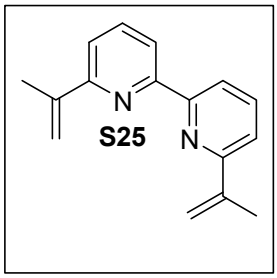
1-(6-Isopropenyl-pyridin-2-yl)-ethanone (**S22**). Prepared according to the general procedure discussed above via input of 1-(6-bromo-pyridin-2-yl)-ethanone (0.2470 g, 1.241 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0139 g, 0.062 mmol, 0.05 equiv), RuPhos (0.0579 g, 0.124 mmol, 0.10 equiv), Cs_2CO_3 (1.214 g, 3.726 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.2500 g, 1.488 mmol, 1.20 equiv), R_f = 0.60, 7% methyl-*tert*-butyl ether:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with a methyl-*tert*-butyl ether:hexanes gradient mobile phase ;isolated yield 0.1825 g, 92%; amber oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.90 (d, *J* = 7.7 Hz, 1H), 7.78 (t, *J* = 7.8 Hz, 1H), 7.66 (d, *J* = 7.9 Hz, 1H), 5.97 (br-s, 1H), 5.38 (br-s, 1H), 2.75 (s, 3H), 2.26 (s, 3H) ; ^{13}C{^{1}H} NMR (125 MHz, $(CD_3)_2CO$): δ = 200.0, 158.1, 153.6, 143.7, 138.4, 123.8, 120.2, 116.6, 25.5, 20.4, one ^{13}C resonance, either overlapped, or was phased out during acquisition and not observed ; IR (ATR-liquid): $\bar{\upsilon}_{max}$ = 3097, 2975, 2923, 1698, 1581, 1454, 1356, 1241, 1108, 822, 598 cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for $C_{10}H_{11}NO$ 162.0919 ; Found: 162.0915.



2,6-Diisopropenyl-pyridine (**S23**). Prepared according to the general procedure discussed above via input of 2,6-dibromopyridine (0.2977 g, 1.257 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0281 g, 0.126 mmol, 0.05 equiv), RuPhos (0.1172 g, 0.251 mmol, 0.10 equiv), Cs_2CO_3 (1.2286 g, 3.771 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.423 g, 2.51 mmol, 1.20 equiv), R_f = 0.60, 1% methyl *tert*-butyl ether:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with a methyl-*tert*-butyl ether (1:1):hexanes gradient mobile phase ;isolated yield 0.1326 g, 66% over two steps from 2,6-dibromopyridine; yellow oil; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 7.73 (t, *J* = 7.9Hz, 1H), 7.51 (d, *J* = 7.8 Hz, 2H), 5.96–5.94 (m, 2H), 5.31–5.29 (m, 2H), 2.22–2.20 (m, 6H) ; ^{13}C{^{1}H} NMR (125 MHz, $(CD_3)_2CO$): δ = 157.5, 144.5, 137.6, 118.8, 115.4, 20.5 ; IR (ATR-liquid): $\bar{\upsilon}_{max}$ = 3088, 2975, 2922, 1632, 1566, 1455, 1370, 1175, 897, 822, 747 cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for $C_{11}H_{14}N$ 160.1126; Found: 160.1120.

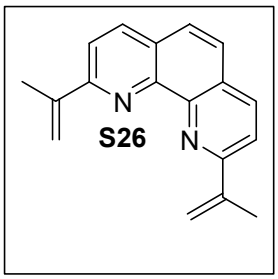


6-Isoprenyl-[2,2']bipyridinyl (**S24**). Prepared according to the general procedure discussed above input of 6-bromo-2,2'-bipyridine (0.3000 g, 1.282 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0144 g, 0.0641 mmol, 0.05 equiv), RuPhos (0.0598 g, 0.1282 mmol, 0.10 equiv), Cs_2CO_3 (1.2531 g, 3.846 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.2586 g, 1.539 mmol, 1.20 equiv), R_f = 0.65, 10% (9:1) ethyl acetate:methyl *tert*-butyl ether:hexanes ; purified using automated flash column chromatography on a normal phase pH 7 Al_2O_3 column with an ethyl acetate:methyl-*tert*-butyl ether (1:1):hexanes gradient mobile phase ; isolated yield 0.1416 g, 56%-average yield over three experiments ; yellow oil; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 8.70–8.64 (m, 1H), 8.55 (d, *J* = 7.9 Hz, 1H), 8.38 (d, *J* = 7.7 Hz, 1H), 7.95–7.86 (m, 2H), 7.68 (d, *J* = 7.8 Hz, 1H), 7.43–7.38 (m, 1H), 6.05 (s, 1H), 5.37 (s, 1H), 2.29 (s, 3H) ; ^{13}C NMR (125 MHz, $(CD_3)_2CO$): δ = 158.0, 157.0, 155.8, 150.0, 144.3, 138.2, 137.7, 124.7, 121.4, 120.5, 119.9, 115.8, 20.6 ; IR (ATR-solid): $\bar{\upsilon}_{max}$ = 3060, 2976, 2921, 2857, 1634, 1579, 1563, 1472, 1456, 1428, 902, 830, 787, 753 cm^{-1}; HRMS (EI): m/z: $[M]^+$ Calcd for $C_{13}H_{13}N_2$ 197.1079; Found: 197.1072.



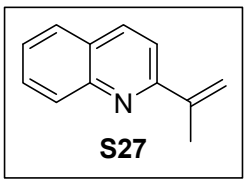
S25

6,6'-Diisopropenyl-[2,2']bipyridine (**S25**). Prepared according to the general procedure discussed above via input of 6,6'-dibromo-[2,2']bipyridine (0.3000 g, 0.962 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0108 g, 0.048 mmol, 0.05 equiv), RuPhos (0.0449 g, 0.0962 mmol, 0.10 equiv), Cs_2CO_3 (0.9403 g, 2.886 mmol, 3.00 equiv), and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.3880 g, 2.308 mmol, 2.40 equiv), R_f = 0.78, 15% acetone:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with an acetone:hexanes gradient mobile phase ; isolated yield 0.1070 g, 89% over two steps from 6,6'-dibromo-[2,2']bipyridine; white solid; melting point = 167.8–169.1 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.43 (dd, *J* = 0.8, 7.9 Hz, 2H), 7.78 (t, *J* = 7.8 Hz, 2H), 7.50 (dd, *J* = 0.9, 7.8 Hz, 2H), 6.01–5.99 (m, 2H), 5.36–5.33 (m, 2H), 2.32–2.29 (m, 6H) ; ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ = 157.3, 155.3, 143.6, 137.2, 119.6, 119.5, 115.5, 20.6 ; IR (ATR-solid): $\bar{\upsilon}_{max}$ = 3033, 2959, 2922, 1608, 1584, 1568, 1487, 1382, 820, 536 cm^{-1}; HRMS (EI): m/z: $[M+H]^+$ Calcd for $C_{16}H_{17}N_2$ 237.1392; Found: 237.1387.



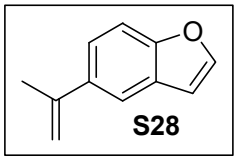
S26

2,9-Diisoprenyl-[1,10]phenanthroline (**S26**). Prepared according to the general procedure discussed above via input of 2,9-dibromo-1,10-phenanthroline (0.3000 g, 0.893 mmol, 1.00 equiv), $Pd(OAc)_2$ (0.0100 g, 0.0447 mmol, 0.05 equiv), RuPhos (0.0417 g, 0.0893 mmol, 0.10 equiv), Cs_2CO_3 (0.8729 g, 2.679 mmol, 3.00 equiv, and 2-isopropenyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.3602 g, 2.144 mmol, 2.40 equiv), R_f = 0.19, on C18 reverse-phase TLC plates MeCN:H_2O (3:1) ; isolated yield 0.2052 g, 87% over two steps from 2,9-dibromo-1,10-phenanthroline ; yellow gum ; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 8.37 (d, *J* = 8.4 Hz, 2H), 8.06 (d, *J* = 8.4 Hz, 2H), 7.89 (s, 2H), 6.20–6.19 (m, 2H), 5.56–5.53 (m, 2H), 2.50–2.47 (m, 6H) ; ^{13}C{^{1}H} NMR $(CD_3)_2CO$): δ = 158.1, 146.3, 145.6, 137.0, 128.7, 126.9, 120.0, 116.8, 20.6 ; IR (ATR- $CDCl_3$): $\bar{\upsilon}_{max}$ = 3727, 2676, 2629, 2923, 2853, 1654, 1584, 1498, 1457, 1033, 854, 669 cm^{-1} ; HRMS (EI): m/z: Calcd for $C_{18}H_{17}N_2$ $[M+H]^+$ 261.1392; Found: 261.1400.



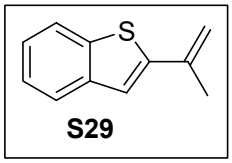
S27

2-Isoprenylquinoline (**S27**). Prepared according to the general procedure discussed above with 2-bromoquinoline (0.3000 g, 1.449 mmol). Spectroscopic data obtained was commensurate with that previously reported.[6]



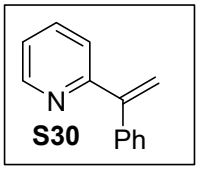
S28

5-isoprenylbenzofuran (**S28**). Prepared to the general procedure discussed above with 5-bromobenzofuran (0.2961 g, 1.503 mmol). Spectroscopic data obtained was commensurate with that previously reported.[7]



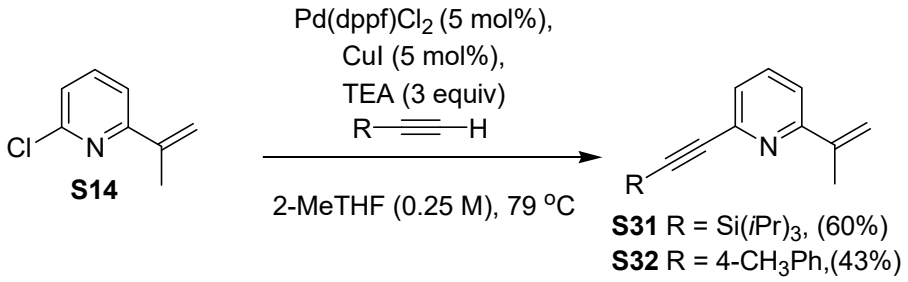
S29

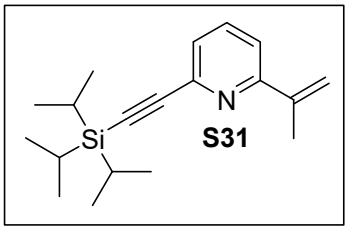
2-Isoprenylbenzothiophene (**S29**). Prepared to the general procedure discussed above with 2-bromobenzothiophene (0.3203 g, 1.503 mmol). Spectroscopic data obtained was commensurate with that previously reported.[8]



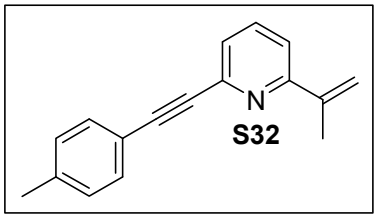
2-(1-Phenylvinyl)pyridine (**S30**). Spectroscopic data obtained was commensurate with that previously reported.[9]

Scheme S4. General procedure for Sonogashira Cross-Coupling of Scaffolds





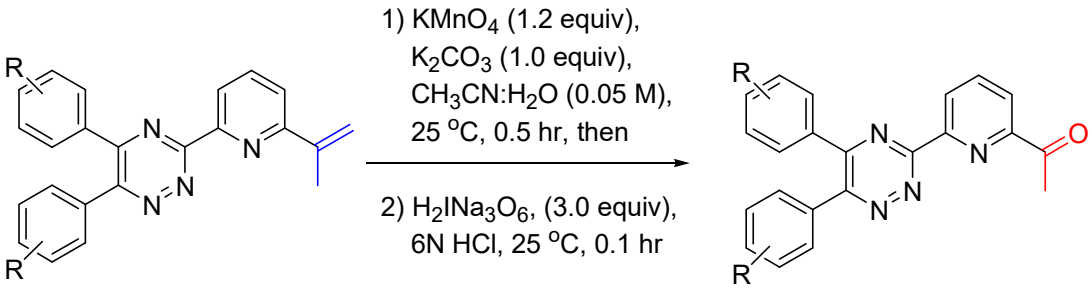
2-Isopropenyl-6-[(triisopropylsilanyl)-ethynyl]-pyridine (**S31**). An adaptation of a literature procedure was helpful.[10] To an 8 mL reaction vial equipped with a magnetic stirring bar at ambient temperature was charged **S14** (0.0912 g, 0.596 mmol, 1.00 equiv) in anhydrous 2-methyltetrahydrofuran (2.38 mL, 0.25 M). The resulting clear solution was charged successively with $Pd(dppf)Cl_2$ (0.0218 g, 0.0298 mmol, 0.05 equiv), CuI (0.0057 g, 0.0298 mmol, 0.05 equiv), triethylamine (0.1806 g, 1.288 mmol, 3.00 equiv), and triisopropylsilylethyne (0.1628 g, 0.8939 mmol, 1.20 equiv) then heated to 79 °C for 12 hours until consumption of the starting material was observed on TLC then benchmarked with ^{1}H NMR spectroscopy. The crude reaction mixture was worked up as described in the reference and purified using automated flash column chromatography with a normal phase silica gel column using a ethyl acetate:hexanes gradient mobile phase to afford the title compound, R_f = 0.55, 5% ethyl acetate:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with an ethyl acetate:hexanes gradient mobile phase ; isolated yield 0.1042 g, 60%; amber oil; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 7.77 (t, *J* = 7.8 Hz, 1H), 7.61 (dd, *J* = 0.7, 8.0 Hz, 1H), 7.44 (dd, *J* = 0.7, 7.6 Hz, 1H), 5.94 (br-m, 1H), 5.34–5.32 (br-m, 1H), 2.18 (s, 3H), 1.23–1.14 (m, 21H) ; $^{13}C\{^1H\}$ NMR (125 MHz, $(CD_3)_2CO$): δ = 159.1, 143.9, 143.0, 137.6, 127.0, 120.2, 116.4, 108.0, 90.1, 20.4, 19.0, 12.0 ; IR (ATR-liquid): $\bar{\upsilon}_{max}$ = 2943, 2864, 2161, 1574, 1562, 1463, 1445, 884, 818, 727, 677, 663 cm^{-1}; HRMS (EI): m/z $C_{19}H_{30}NSi$ [M+H] 300.2148; Found: 300.2146.



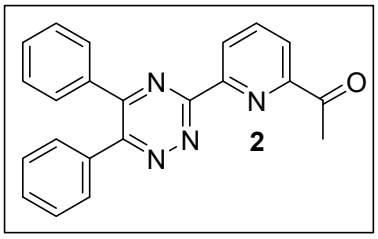
2-Isoprenyl-6-p-tolylethynyl-pyridine (**S32**). To an 8 mL reaction vial equipped with a magnetic stirring bar at ambient temperature was charged **S14** (0.1480 g, 0.967 mmol, 1.00 equiv) in anhydrous 2-methyltetrahydrofuran (3.87 mL, 0.25 M). The resulting clear solution was charged successively with $Pd(dppf)Cl_2$ (0.0353 g, 0.0484 mmol, 0.05 equiv), CuI (0.0092 g, 0.0484 mmol, 0.05 equiv), triethylamine (0.2933 g, 2.901 mmol, 3.00 equiv), and 4-methylphenylethyne (0.1178 g, 1.015 mmol, 1.05 equiv) then heated to 79 °C for 16 hours until consumption of the starting material was observed on TLC then benchmarked with ^{1}H NMR spectroscopy. The crude reaction

mixture was worked up as described in the reference and purified using automated flash column chromatography with a normal phase silica gel column using an ethyl acetate:hexanes gradient mobile phase to afford the title compound, R_f = 0.50, 5% ethyl acetate:hexanes ; purified using automated flash column chromatography on a normal phase silica gel column with an ethyl acetate:hexanes gradient mobile phase ;isolated yield 0.1110 g, 43%; amber liquid; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 7.79 (t, *J* = 7.8 Hz, 1H), 7.61 (dd, *J* = 0.7, 8.0 Hz, 1H), 7.51–7.47 (m, 3H), 7.27 (d, *J* = 7.8 Hz, 2H), 5.96–5.93 (m, 1H), 5.33–5.36 (m, 1H), 2.37 (s, 3H), 2.22–2.18 (m, 3H), ; ^{13}C NMR (125 MHz, $(CD_3)_2CO$): δ = 159.2, 144.0, 143.4, 140.3, 137.7, 132.6, 130.2, 126.5, 120.2, 119.8, 116.4, 89.5, 88.9, 21.5, 20.5 ; IR (ATR-$(CD_3)_2CO$): $\bar{\upsilon}_{max}$ = 3081, 3055, 2978, 2954, 2923, 2213, 1575, 1561, 1508, 1446, 1167, 814, 748, 530, cm^{-1}; HRMS (EI): m/z: Calcd for $C_{17}H_{16}N$ $[M+H]^+$ 234.1283; Found: 234.1283.

Scheme S5. Preparation of Novel Pyridinyl Methyl Ketones

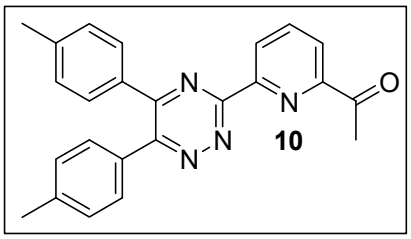


General Procedure for Oxidative Isoprene Cleavage. To an 8 mL reaction vial equipped with a magnetic stirring bar at ambient temperature were successively charged: the required substrate (0.285 mmol, 1.00 equiv), sodium paraperiodate (0.855 mmol, 3.00 equiv), and potassium carbonate (0.285 mmol, 1.00 equiv). The resulting mixture was slurried in a 4:1 (v:v) acetonitrile:water (0.05 M) solution followed by addition of potassium permanganate (0.342 mmol, 1.20 equiv) in one portion. The resulting mixture which possessed a pH of approximately 8–9 was continued at ambient temperature until conversion of the isoprene to the vicinal-1,2-diol was confirmed by thin layer chromatography. The reaction mixture was subsequently acidified to a pH of 3–4 with a 6.0 N aqueous hydrochloric acid solution, diluted with anhydrous dichloromethane, and subsequently quenched with a saturated aqueous sodium thiosulfate solution (3.0 mL). The biphasic mixture was filtered through a pad of Celite over normal phase silica gel flash chromatography column to remove residual manganese dioxide. The resulting filtrate was washed successively with a saturated potassium carbonate solution (2.0 mL), followed by a saturated sodium chloride solution, dried over anhydrous sodium sulfate, filtered, and then concentrated under reduced pressure at ambient temperature to afford the title compounds in the morphologies listed.

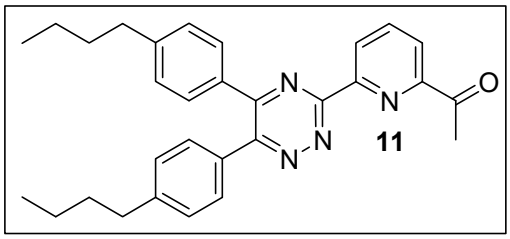


1-[6-(5,6-Diphenyl-[1,2,4]triazin-3-yl)-pyridin-2-yl]-ethanone (**2**). Prepared according to the general procedure discussed above with input of **6** (0.1000 g, 0.285 mmol), R_f = 0.22, 25% ethyl acetate:hexanes; isolated yield 0.0823 g, 82%; yellow solid; melting point = 147.5–148.9 °C; ^{1}H NMR (500 MHz, $CDCl_3)_2$: δ = 8.84 (d, *J* = 7.8 Hz, 1H), 8.23 (d, *J* = 7.7 Hz, 1H), 8.09 (t, *J* = 7.7 Hz, 1H), 7.73 (d, *J* = 7.8 Hz, 2H), 7.67 (d, *J* = 7.7 Hz, 2H), 7.51–7.45 (m, 2H), 7.44–7.37 (m, 4H), 2.93 (s, 3H) ; ^{13}C{^{1}H} NMR (125 MHz, $(CD_3)_2SO$): δ = 200.4,

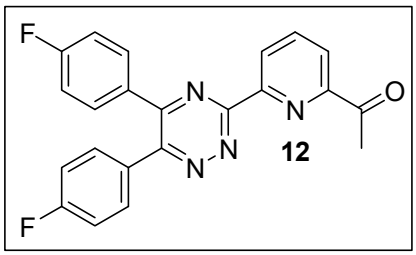
160.3, 156.7, 156.2, 154.2, 152.5, 138.2, 135.6, 135.3, 131.2, 130.13, 130.08, 129.7, 128.9, 128.8, 127.5, 123.2, 25.9; IR (ATR-solid): $\bar{\upsilon}_{max}$ = 3060, 2956, 2924, 1699, 1581, 1493, 1446, 1385, 1362, 1242, 771, 699 cm^{-1}; HRMS (EI): m/z: $[M]^+$ Calcd for $C_{22}H_{16}N_4O$ 352.1324; Found: 352.1324.



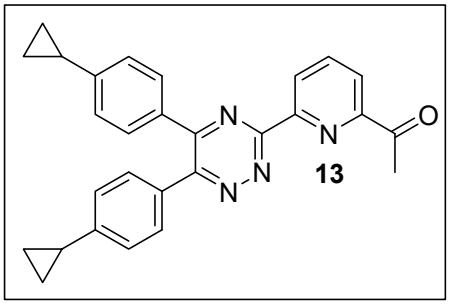
1-[6-(5,6-Di-p-tolyl-[1,2,4]triazin-3-yl)-pyridin-2-yl]-ethanone (**10**). Prepared according to the general procedure discussed above with input of **S7** (0.5640 g, 1.491 mmol), R_f = 0.29, 25% ethyl acetate:hexanes; isolated yield 0.4263 g, 74%; yellow solid; melting point = 185.1–186.8 °C; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 8.82 (dd, *J* = 1.2, 7.7 Hz, 1H), 8.26 (t, *J* = 7.8 Hz, 1H), 8.19 (dd, *J* = 1.1, 7.7 Hz, 1H), 7.69–7.66 (m, 2H), 7.61–7.57 (m, 2H), 7.31–7.27 (m, 2H), 7.26–7.24 (m, 2H), 2.81 (s, 3H), 2.41 (s, 3H), 2.39 (s, 3H) ; $^{13}C\{^1H\}$ NMR (125 MHz, $(CD_3)_2CO$): δ = 199.8, 161.0, 157.4, 156.5, 154.9, 153.9, 142.1, 140.6, 139.2, 134.1, 134.X (overlaps with 134.1), 130.8, 130.3, 130.1, 130.0, 128.0, 123.2, 25.6, 21.4, 21.3; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 3032, 2921, 1699, 1609, 1583, 1493, 1384, 1360, 1241, 1185, 1091, 911, 820, 733, 592, 538 cm^{-1}; HRMS (EI): m/z: $[M+Na]^+$ Calcd for $C_{24}H_{20}N_4ONa$ 403.1535; Found: 403.1529.



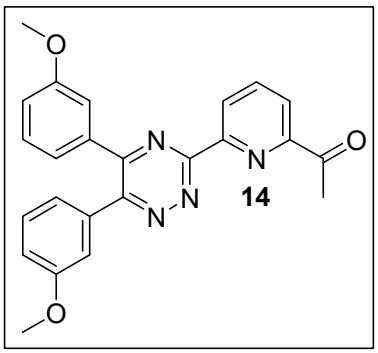
1-{6-[5,6-Bis-(4-butyl-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (**11**). Prepared according to the general procedure discussed above with input of **S8** (0.3015 g, 652 mmol), R_f = 0.37, 25% ethyl acetate:hexanes; isolated yield 0.1578 g, 52%; yellow solid; melting point = 79.8–81.3 °C; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 8.82 (dd, *J* = 0.8, 7.7 Hz, 1H), 8.25 (t, *J* = 7.7 Hz, 1H), 8.18 (dd, *J* = 0.9, 7.7 Hz, 1H), 7.71–7.67 (m, 2H), 7.62–7.59 (m, 2H), 7.32–7.28 (m, 2H), 7.27–7.25 (m, 2H), 2.80 (s, 3H), 2.71–2.65 (m, 4H), 1.69–1.57 (m, 4H), 1.42–1.31 (m, 4H), 0.97–0.90 (m, 6H) ; $^{13}C\{^1H\}$ NMR (125 MHz, $(CD_3)_2CO$): δ = 199.9, 161.0, 157.5, 156.5, 154.9, 153.9, 147.0, 145.6, 139.2, 134.32, 134.30, 130.9, 130.4, 129.5, 129.4, 128.0, 123.3, 36.02, 36.00, 34.2, 34.1, 25.7, 23.0, 22.9X (overlaps with 23.0), 14.20, 14.16 ; IR (ATR-solid): $\bar{\upsilon}_{max}$ = 3174, cm^{-1}; HRMS (EI): m/z: $[M+Na]^+$ Calcd for $C_{30}H_{32}N_4ONa$ 487.2474; Found: 487.2471.



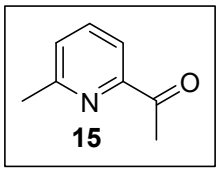
1-{6-[5,6-Bis-(4-fluoro-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (**12**). Prepared according to the general procedure discussed above with input of **S9** (0.6100 g, 1.990 mmol), R_f = 0.20, 25% ethyl acetate:hexanes; isolated yield 0.0373 g, 48%; yellow solid; melting point = 186.8–188.4 °C; ^{1}H NMR (500 MHz, $(CD_3)_2SO$): δ = 8.78 (dd, *J* = 0.9, 7.8 Hz, 1H), 8.29 (t, *J* = 7.8 Hz, 1H), 8.19 (dd, *J* = 1.0, 7.7 Hz, 1H), 7.73–7.69 (m, 2H), 7.36–7.30 (m, 4H), 2.78 (s, 3H) ; $^{13}C\{^1H\}$ NMR (125 MHz, $(CD_3)_2SO$): δ = 199.3, 164.3 (*J* = 289.4 Hz), 162.3 (*J* = 283.8 Hz), 159.9, 155.8, 155.1, 153.4, 152.1, 139.0, 132.4 (*J* = 35.8 Hz), 131.8 (*J* = 34.8 Hz), 131.7 (*J* = 13.0 Hz), 131.67 (*J* = 12.1 Hz), 127.5, 122.8, 115.8 (*J* = 17.7 Hz), 115.7 (*J* = 17.9 Hz), 25.5 ; ^{19}F NMR (471 MHz, $(CD_3)_2SO$): δ = −109.2– –109.4 (m, 1F), –111.1– –111.3 (m, 1F) ; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 3072, 2922, 2863, 1603, 1513, 1492, 1383, 1362, 1236, 1160, 1097, 1008, 910, 841, 728, 668, 567, 546 cm^{-1}; HRMS (EI): m/z: $[M+Na]^+$ Calcd for $C_{22}H_{14}F_2N_4ONa$ 411.1033; Found: 411.1031.



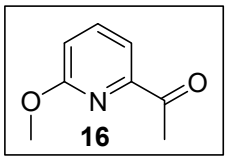
1-{6-[5,6-Bis-(4-cyclopropyl-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (**13**). Prepared according to the general procedure discussed above with input of **S10** (0.6200 g, 1.441 mmol), R_f = 0.15, 25% ethyl acetate:hexanes; isolated yield 0.2081 g, 33%; yellow solid; melting point = 112.3–113.7 °C; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 8.09 (d, *J* = 7.7 Hz, 1H), 8.24 (t, *J* = 7.7 Hz, 1H), 8.17 (d, *J* = 7.7 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 2H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 2.79 (s, 3H), 2.08–1.89 (m, 2H)-overlaps with residual (CH3)2CO resonance, 1.06–1.02 (m, 4H), 0.80–0.73 (m, 4H) ; ^{13}C{^{1}H} NMR (125 MHz, $(CD_3)_2CO$): δ = 199.9, 160.4, 157.3, 156.3, 154.9, 153.9, 148.8, 147.3, 139.2, 133.9, 133.8, 130.8, 130.3, 128.0, 126.4, 126.3, 123.3, 25.7, 16.0, 15.9, 10.6, 10.4 ; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 3086, 2992, 2959, 2921, 2853, 1695, 1610, 1493, 1358, 892, 828, 802, 598, 546 cm^{-1}; HRMS (EI): m/z: Calcd for $C_{28}H_{25}N_4O$ $[M+H]^+$ 433.2028; Found: 433.2023.



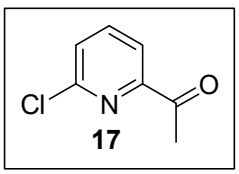
1-{6-[5,6-Bis-(3-methoxy-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (**14**). Prepared according to the general procedure discussed above with input of **S11** (0.6111 g, 1.490 mmol), R_f = 0.15, 25% ethyl acetate:hexanes; isolated yield 0.1780 g, 87%; yellow solid; melting point = 139.7–141.9 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.84 (d, *J* = 7.7 Hz, 1H), 8.23 (d, *J* = 7.7 Hz, 1H), 8.08 (t, *J* = 7.7 Hz, 1H), 7.37–7.27 (m, 5H), 7.19–7.15 (m, 1H), 7.05–6.98 (m, 2H), 3.79 (s, 3H), 3.74 (s, 3H), 2.92 (s, 3H); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ = 200.3, 160.3, 160.0, 159.8, 156.5, 156.0, 154.2, 152.5, 138.2, 136.8, 136.5, 129.8, 127.4, 123.2, 122.5, 122.2, 117.4, 116.4, 114.9, 114.5, 55.5, 55.4, 25.9, one ^{13}C resonance overlapped, or was phased out during acquisition and was not observed; IR (ATR-film): $\bar{\upsilon}_{max}$ = 3004, 2938, 2835, 1699, 1600, 1582, 1503, 1361, 1249, 1043, 912, 734, 703 cm^{-1}; HRMS (EI): m/z: $[M]^+$ Calcd for $C_{24}H_{20}N_4O_3$ 412.1535; Found: 412.1530.



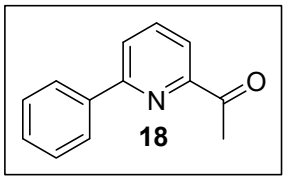
1-(6-Methylpyridin-2-yl)-ethanone (**15**). Prepared according to the general procedure discussed above with input of **S12** (0.0985 g, 0.740 mmol). Spectroscopic data obtained was commensurate with that previously reported.[11]



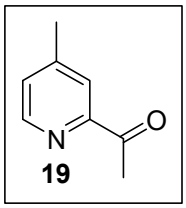
1-(6-Methoxypyridin-2-yl)-ethanone (**16**). Prepared according to the general procedure discussed above with input of **S13** (0.0745 g, 0.740 mmol). Spectroscopic data obtained was commensurate with that previously reported.[12]



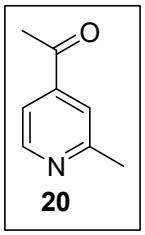
1-(6-Chloropyridin-2-yl)-ethanone (**17**). Prepared according to the general procedure discussed above with input of **S14** (0.0765 g, 0.500 mmol). Spectroscopic data obtained was commensurate with that previously reported.[13]



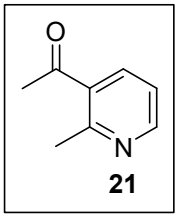
1-(6-Phenylpyridin-2-yl)-ethanone (**18**). Prepared according to the general procedure discussed above with input of **S15** (0.0976 g, 0.500 mmol). Spectroscopic data obtained was commensurate with that previously reported.[14]



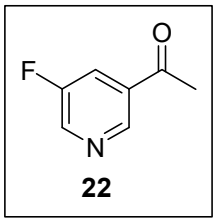
1-(4-Methylpyridin-2-yl)-ethanone (**19**). Prepared according to the general procedure discussed above with input of **S16** (0.0665 g, 0.500 mmol). Spectroscopic data obtained was commensurate with that previously reported.[15]



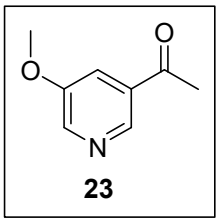
1-(2-Methylpyridin-4-yl)-ethanone (**20**). Prepared according to the general procedure discussed above with input of **S17** (0.0665 g, 0.500 mmol). Spectroscopic data obtained was commensurate with that previously reported.[16]



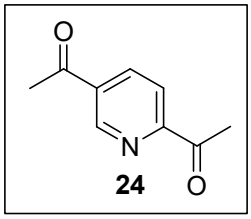
1-(2-Methylpyridin-3-yl)-ethanone (**21**). Prepared according to the general procedure discussed above with input of **S18** (0.180 g, 1.350 mmol). Spectroscopic data obtained was commensurate with that previously reported.[17]



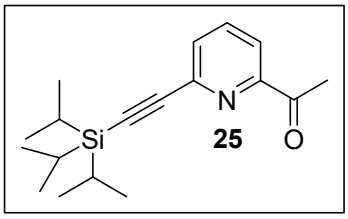
1-(5-Fluoro-pyridin-3-yl)-ethanone (**22**). Prepared according to the general procedure discussed above with input of **S19** (0.2297 g, 1.676 mmol). Spectroscopic data obtained was commensurate with that previously reported.[18]



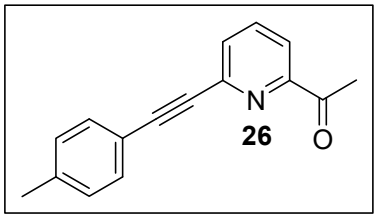
1-(5-Methoxy-pyridin-3-yl)-ethanone (**23**). Prepared according to the general procedure discussed above with input of **S20** (0.0754 g, 0.500 mmol). Spectroscopic data obtained was commensurate with that reported.[19]



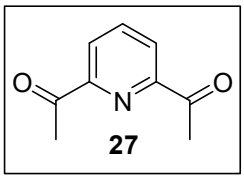
1-(6-Acetyl-pyridin-3-yl)-ethanone (**24**). Prepared according to the general procedure discussed above with input of **S21** (0.0796 g, 0.500 mmol). This compound is commercially available.



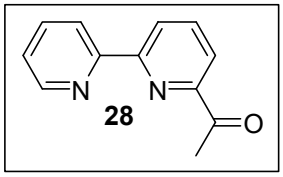
1-{6-[(Triisopropylsilanyl)-ethynyl]-pyridin-2-yl}-ethanone (**25**). Prepared according to the general procedure discussed above with input of **S31** (0.1047 g, 0.357 mmol), R_f = 0.41, 5% ethyl acetate:hexanes; isolated yield 0.0600 g, 56%; yellow oil; ^{1}H NMR (500 MHz, $(CD_3)_2CO$): δ = 7.99 (t, *J* = 7.7 Hz, 1H), 7.96 (dt, *J* = 1.2, 7.8 Hz, 1H), 7.78 (dt, *J* = 1.2, 7.5 Hz, 1H), 2.63 (s, 3H), 1.22–1.14 (m, 21H) ; $^{13}C\{^1H\}$ NMR (125 MHz, $(CD_3)_2CO$): δ = 199.2, 154.8, 143.3, 138.6, 131.8, 121.4, 106.9, 91.9, 25.5, 18.9, 12.0 ; IR (ATR-liquid): $\tilde{\upsilon}_{max}$ = 2943, 2893, 2865, 1703, 1576, 1463, 1445, 1357, 1295, 1254, 1222, 996, 903, 883, 815 cm^{-1}; HRMS (EI): m/z: $[M]^+$ Calcd for $C_{18}H_{28}NOSi$ [M+H] 302.1940; Found: 302.1939.



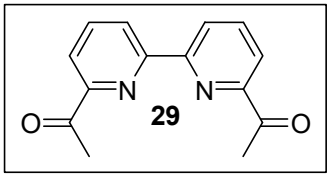
1-(6-p-Tolylethynyl-pyridin-2-yl)-ethanone (**26**). Prepared according to the general procedure discussed above with input of **S32** (0.1064 g, 0.456 mmol). Spectroscopic data obtained was commensurate with that reported.[20]



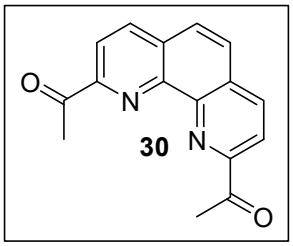
1-(6-Acetyl-pyridin-2-yl)-ethanone (**27**). *Procedure (A)*: Prepared according to the general procedure discussed above input of **S22** (0.0796 g, 0.500 mmol). *Procedure (B)*: Prepared according to the general procedure discussed above with input of **S23** (0.0805 g, 0.500 mmol). Spectroscopic data obtained was commensurate with that previously reported.[21]



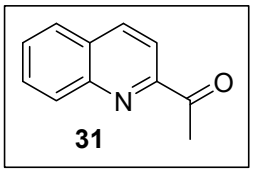
1-[2,2']Bipyridinyl-6-yl-ethanone (**28**). Prepared according to the general procedure discussed above with input of **S24** (0.1000 g, 0.510 mmol). Spectroscopic data obtained was commensurate with that reported.[22]



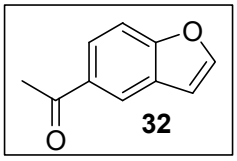
1-(6'-Acetyl-[2,2']bipyridinyl-6-yl)-ethanone (**29**). Prepared according to the general procedure discussed above with input of **S25** (0.2457 g, 0.934 mmol). Spectroscopic data obtained was commensurate with that reported.[23]



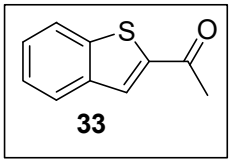
1-(9-Acetyl-[1,10]phenanthrolin-2-yl)-ethanone (**30**). Prepared according to the general procedure discussed above with input of **S26** (0.1701 g, 0.654 mmol). Spectroscopic data obtained was commensurate with that previously reported.[24]



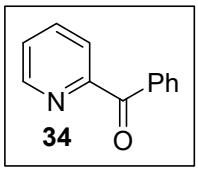
1-Quinolin-2-yl-ethanone (**31**). Prepared according to the general procedure discussed above with input of **S27** (0.0840 g, 0.498 mmol). Spectroscopic data obtained was commensurate with that previously reported.[25]



1-Benzofuran-5-yl-ethanone (**32**). Prepared according to the general procedure discussed above with input of **S28** (0.0450 g, 0.285 mmol). Spectroscopic data obtained was commensurate with that previously reported.[26]



1-Benzothiophen-2-ylethanone (**33**). Prepared according to the general procedure discussed above with input of **S29** (0.0450 g, 0.285 mmol). Spectroscopic data obtained was commensurate with that previously reported.[27]



Phenyl-pyridin-2-yl-methanone (**34**). Prepared according to the general procedure discussed above input of **S30**. Spectroscopic data obtained was commensurate with that previously reported.[28]

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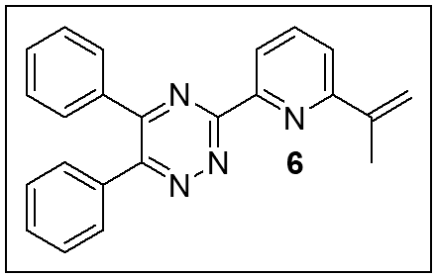
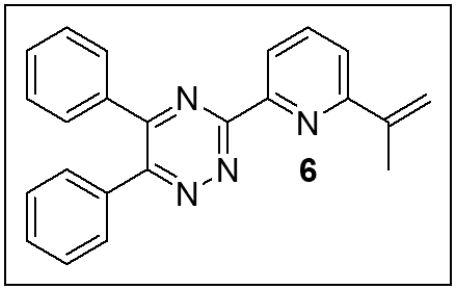
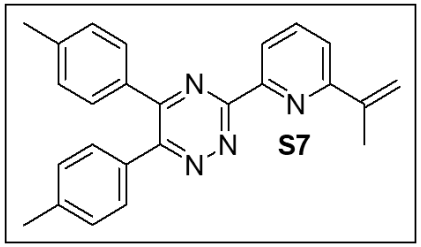
3-(6-Isopropenyl-pyridin-2-yl)-6-(1-methylene-but-2-enyl)-5-phenyl-[1,2,4]triazine (6)
AMS-A-193(7)



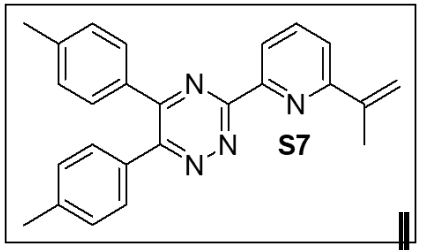
3-(6-Isopropenyl-pyridin-2-yl)-6-(1-methylene-but-2-enyl)-5-phenyl-[1,2,4]triazine (6)
AMS-A-193(8)



3-(6-Isopropenyl-pyridin-2-yl)-5,6-di-p-tolyl-[1,2,4]triazine (S7)
AMS-A-231(3)

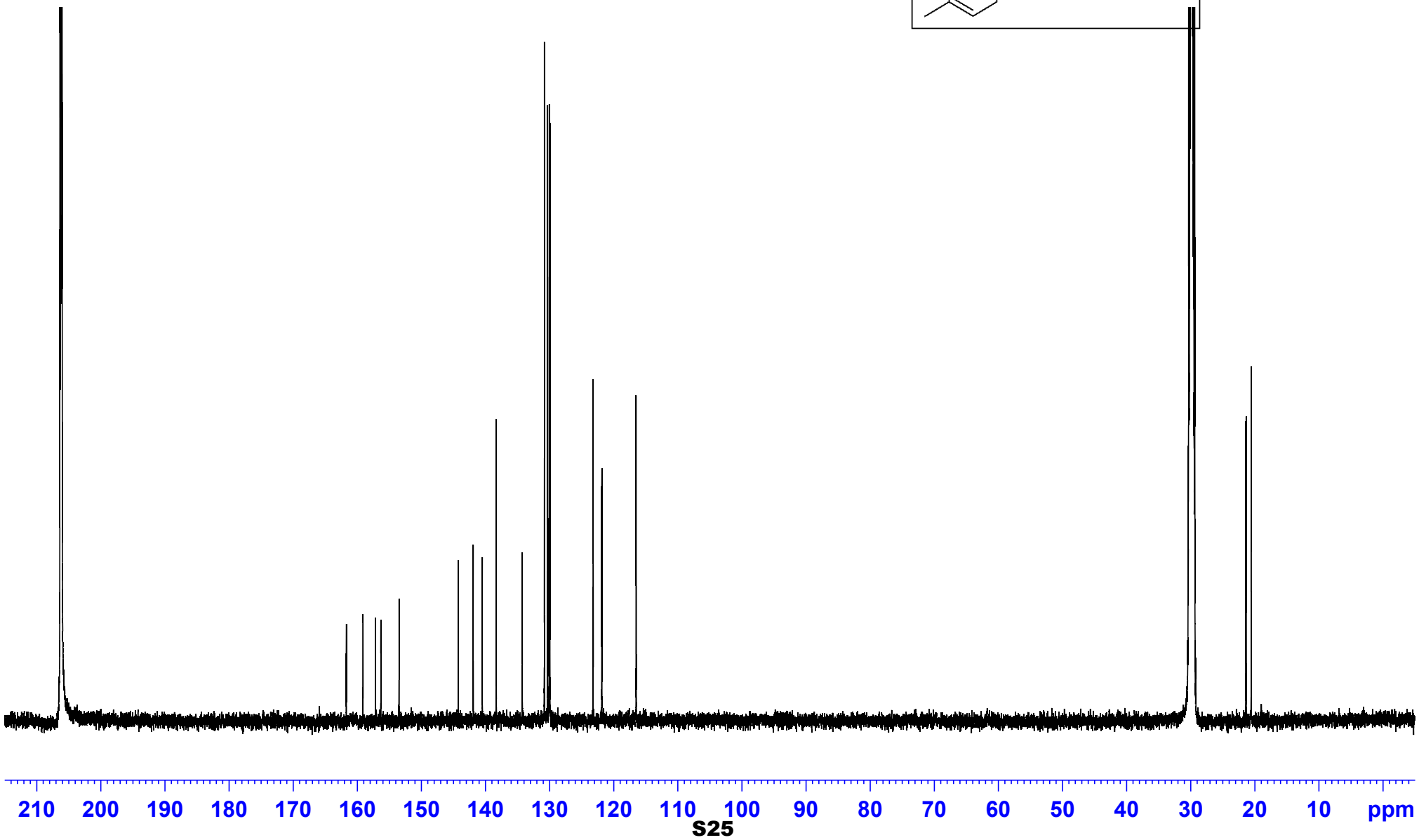


3-(6-Isopropenyl-pyridin-2-yl)-5,6-di-p-tolyl-[1,2,4]triazine (S7)
AMS-A-231(3)

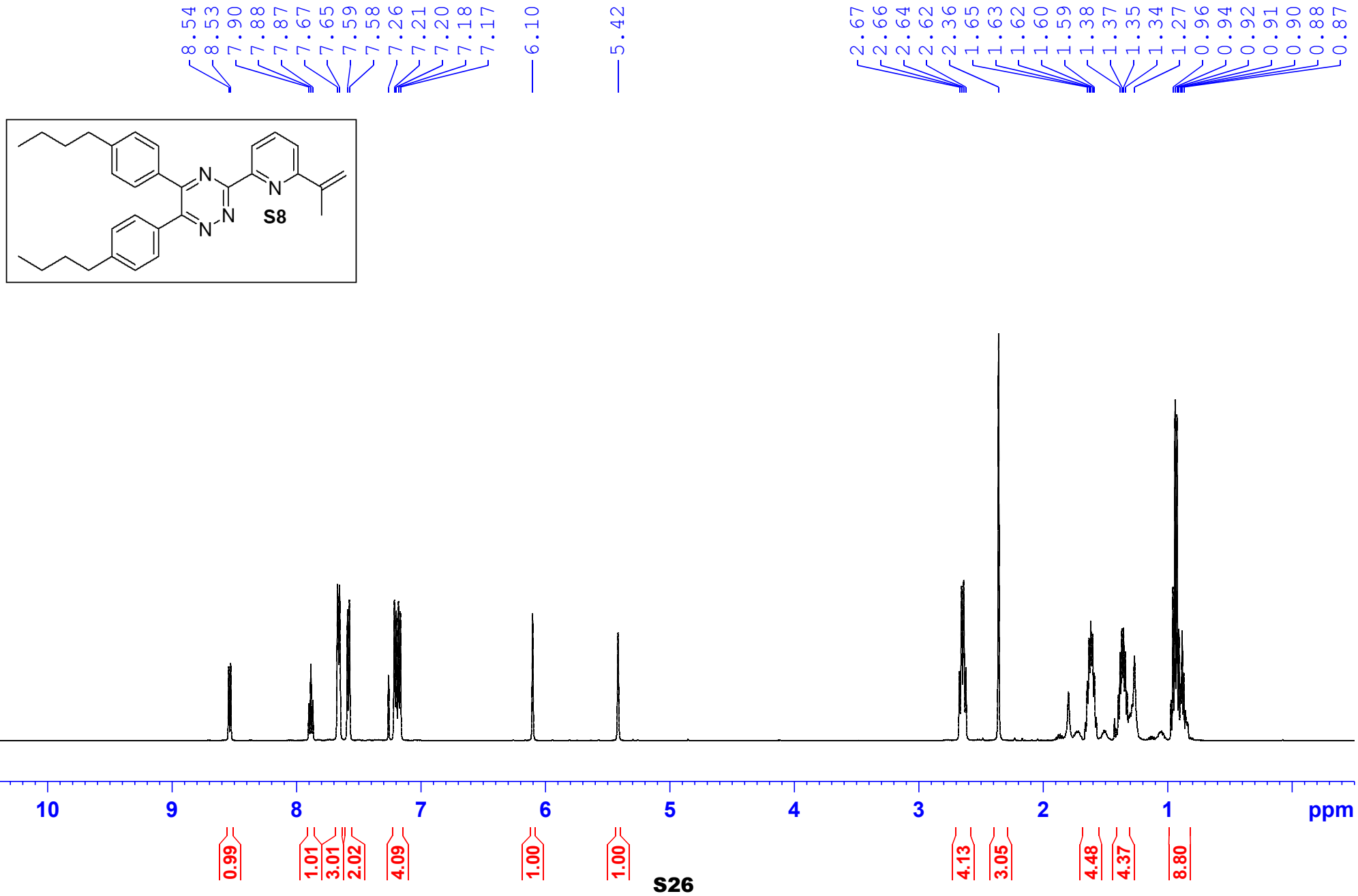


161.7
159.1
157.2
156.3
153.5
144.2
141.9
140.5
138.3
134.3
134.3
130.8
130.3
130.1
130.0
123.2
121.8
116.5

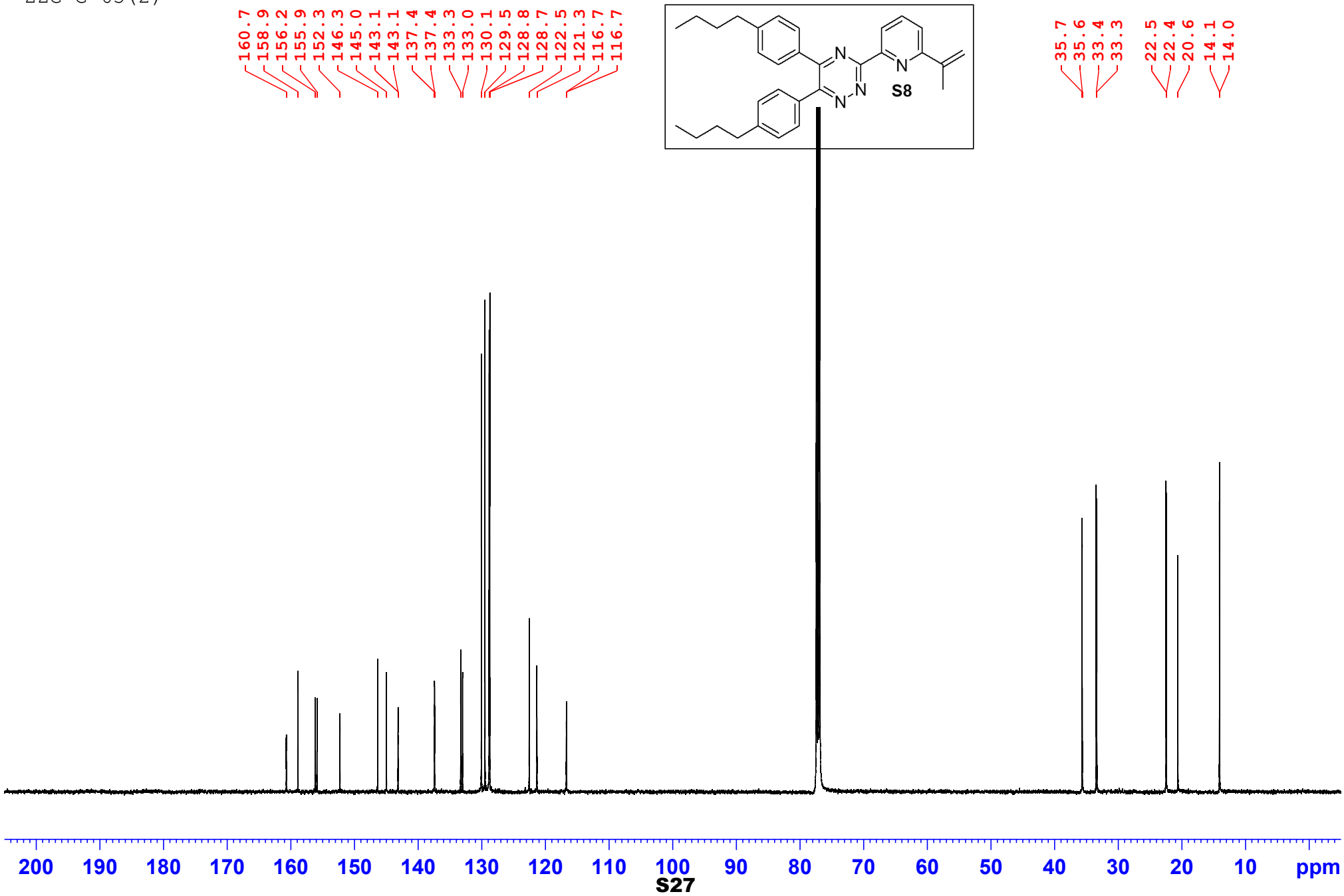
21.4
21.3
20.6



5,6-Bis-(4-butyl-phenyl)-3-(6-isopropenyl-pyridin-2-yl)-[1,2,4]triazine (S8)
ZZG-G-63(2)

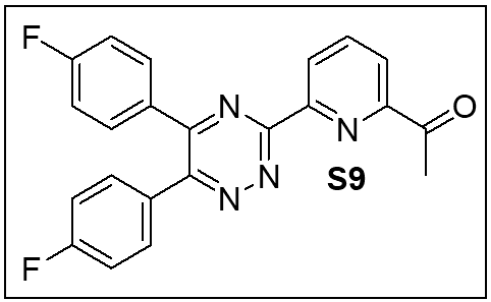


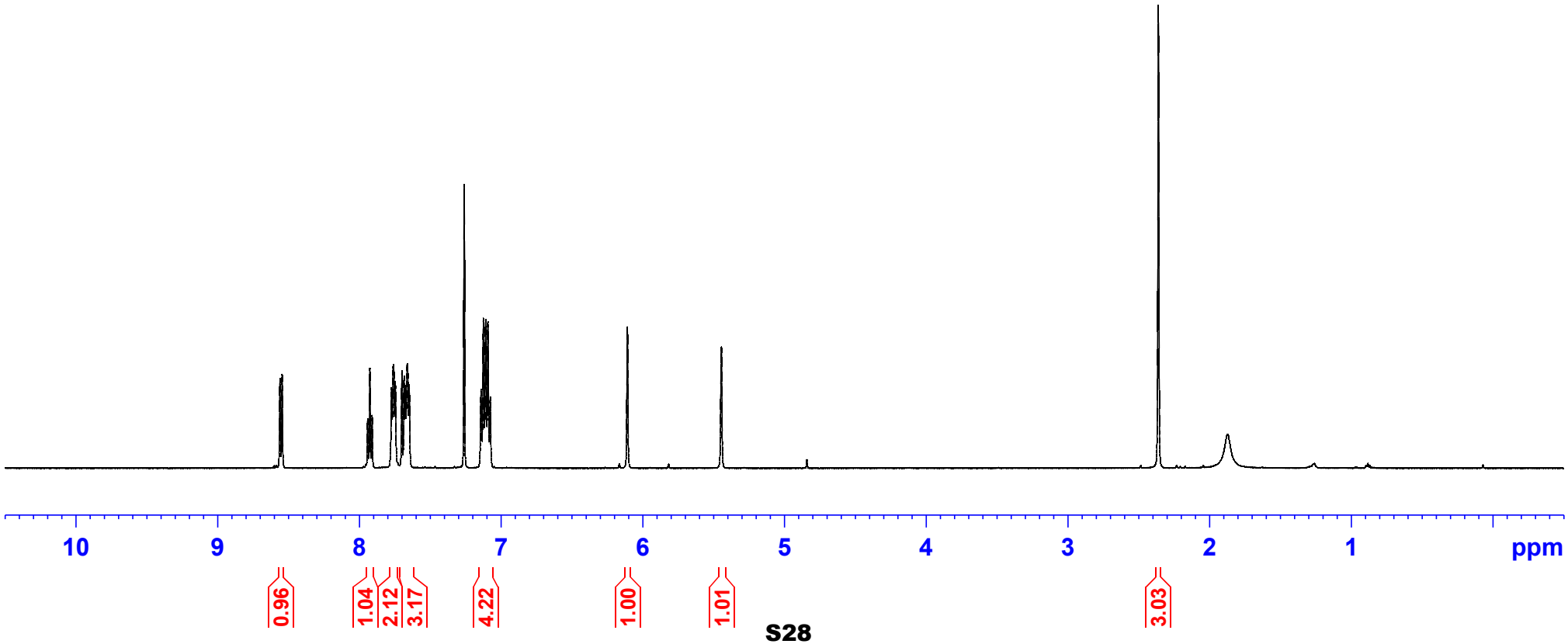
5,6-Bis-(4-butyl-phenyl)-3-(6-isopropenyl-pyridin-2-yl)-[1,2,4]triazine (S8)
ZZG-G-63(2)



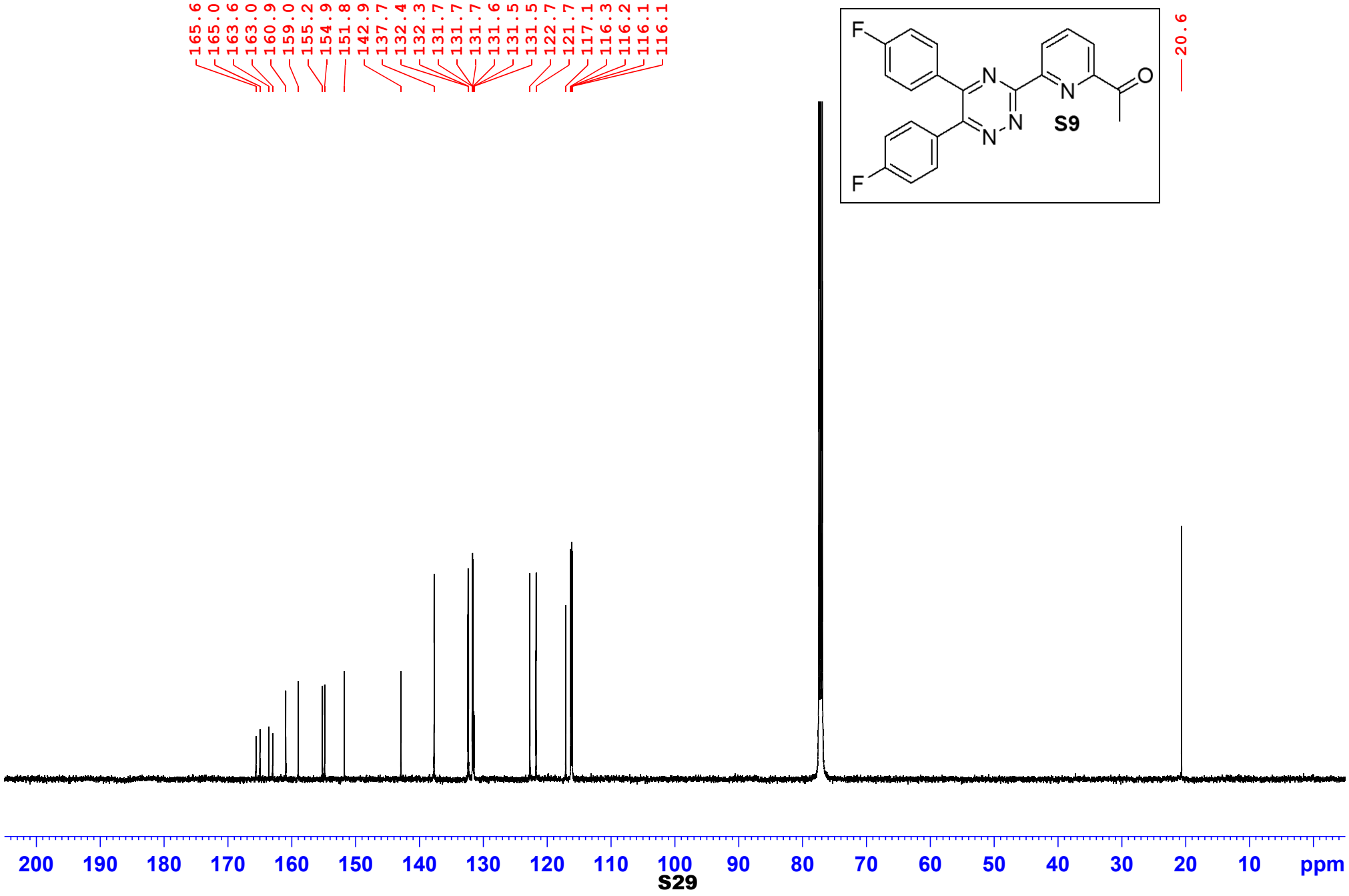
5,6-Bis-(4-fluoro-phenyl)-3-(6-isopropenyl-pyridin-2-yl)-[1,2,4]triazine (S9)
ZZG-G-51(2)

8.56 8.55 7.94 7.93 7.91 7.77 7.76 7.76 7.75 7.70 7.68 7.67 7.66 7.66 7.65 7.14 7.12 7.11 7.09 7.08 6.11 5.45 2.36

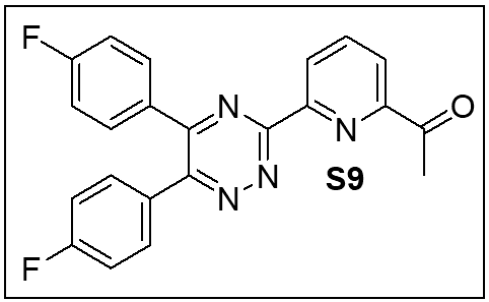




5,6-Bis-(4-fluoro-phenyl)-3-(6-isopropenyl-pyridin-2-yl)-[1,2,4]triazine (S9)
ZZG-G-51(2)

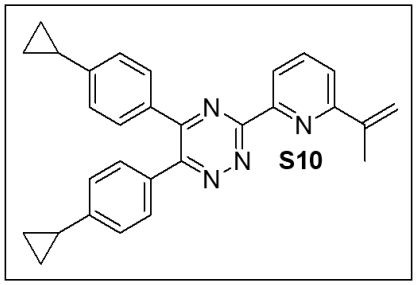


5,6-Bis-(4-fluoro-phenyl)-3-(6-isopropenyl-pyridin-2-yl)-[1,2,4]triazine (S9)
ZZG-G-51

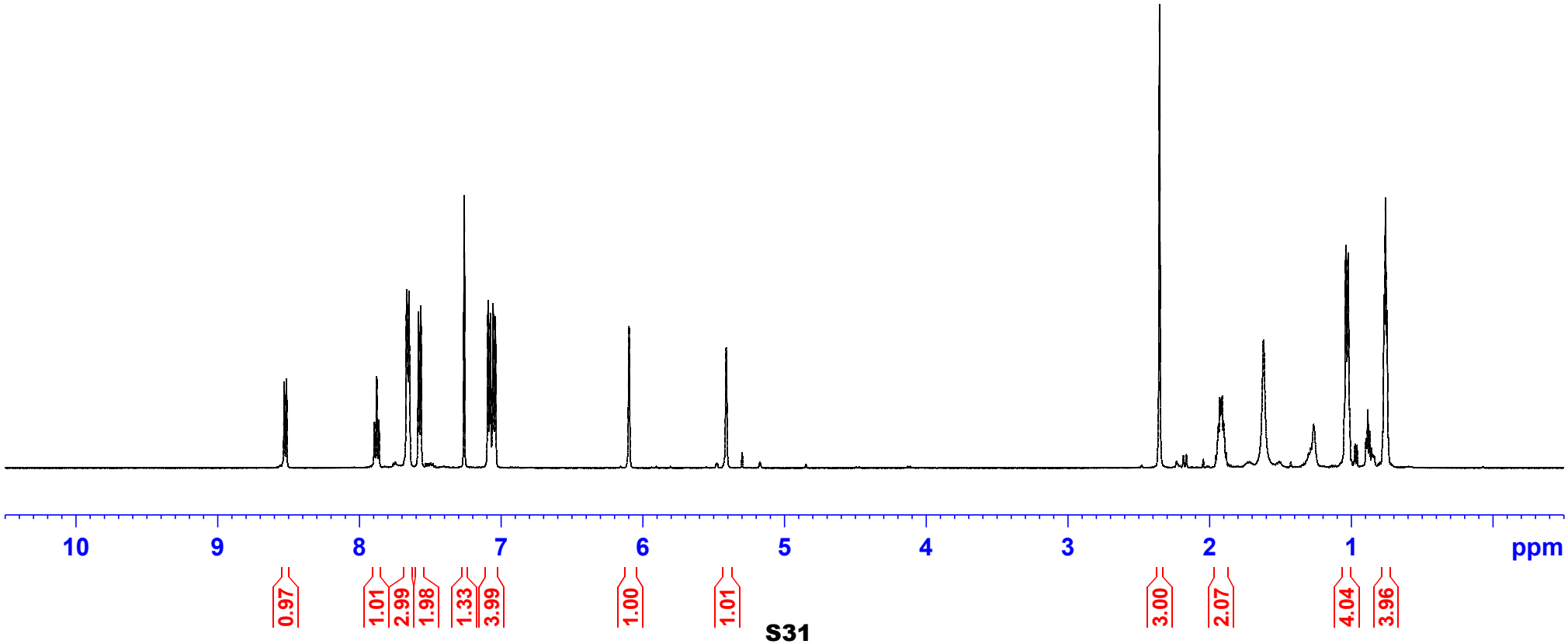


5,6-Bis-(4-cyclopropyl-phenyl)-3-(6-isopropenyl-pyridin-2-yl)-[1,2,4]triazine (S10)
ZZG-G-11(2)

8.53 8.51 7.89 7.88 7.86 7.67 7.65 7.58 7.57 7.09 7.08 7.06 7.04 6.10 5.41

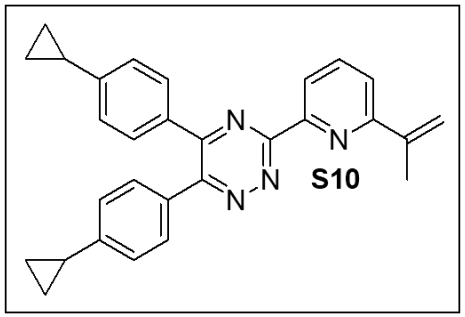


2.35 1.96 1.94 1.93 1.92 1.91 1.90 1.89 1.88 1.04 1.02 0.77 0.76 0.75

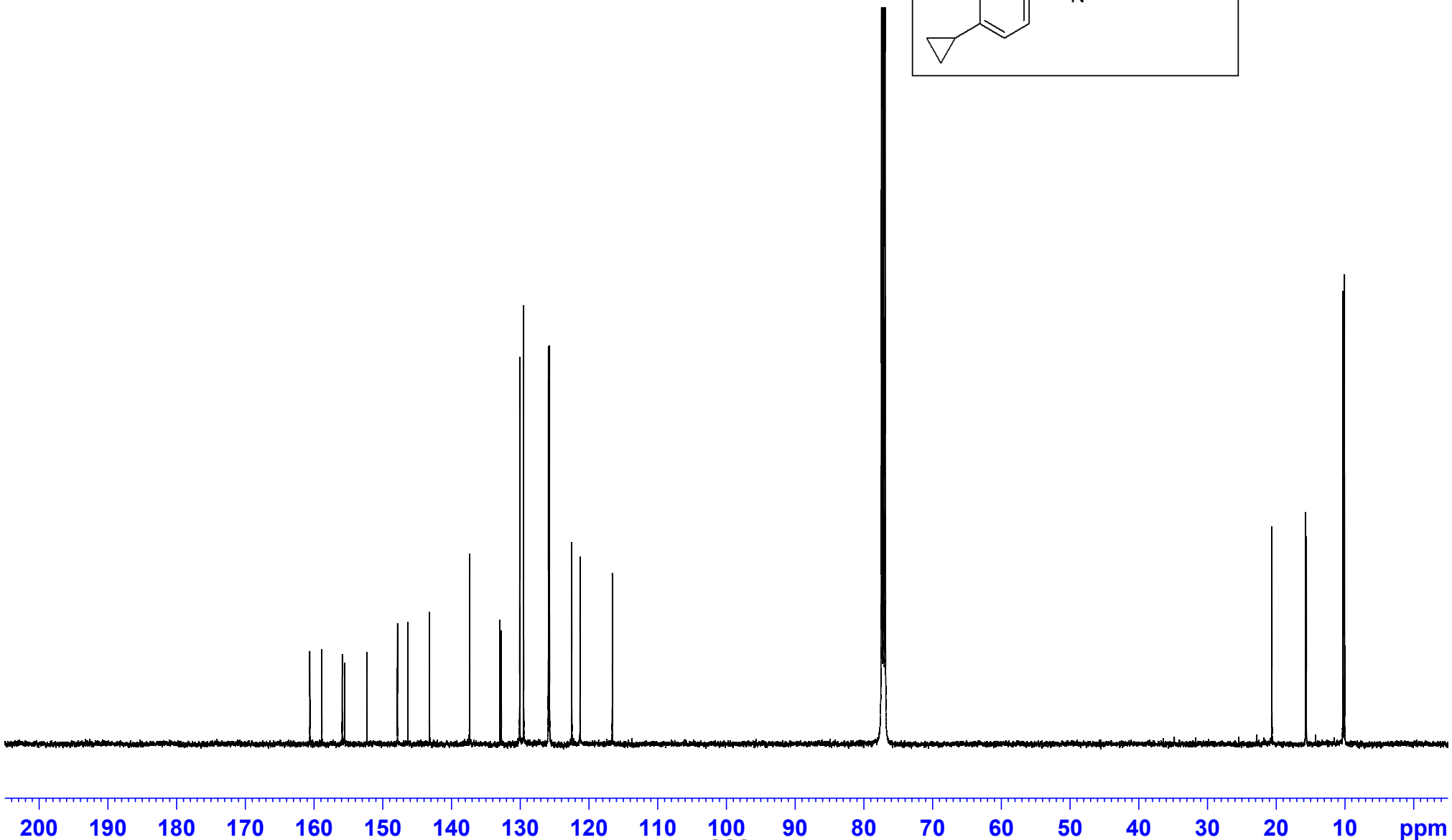


5,6-Bis-(4-cyclopropyl-phenyl)-3-(6-isopropenyl-pyridin-2-yl)-[1,2,4]triazine (S10)
ZZG-G-11(2)

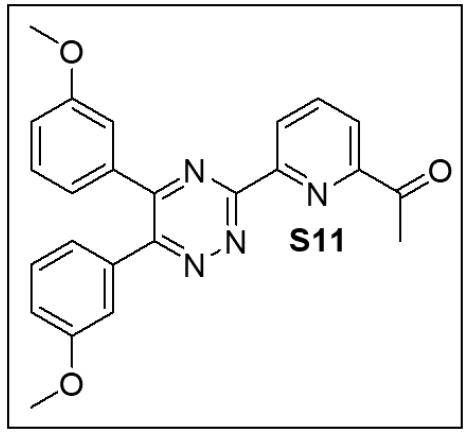
160.6
158.9
155.9
155.6
152.3
147.9
146.3
143.2
137.4
133.0
132.7
130.1
129.5
125.9
125.7
122.5
121.3
116.6



20.6
15.7
15.6
10.3
10.0



3-(6-Isopropenyl-pyridin-2-yl)-5,6-bis-(3-methoxy-phenyl)-[1,2,4]triazine (S11)
ZZG-G-61(3)

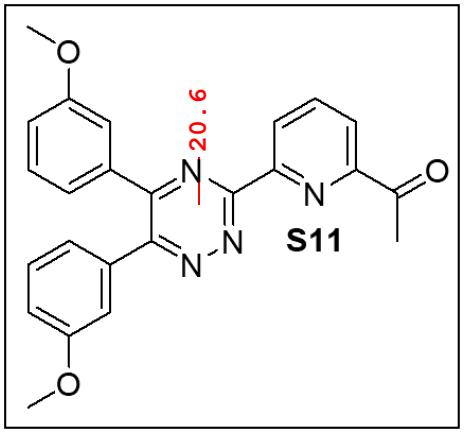


7.91 7.89 7.69 7.69 7.68 7.67 7.37 7.37 7.31 7.31 7.31 7.30 7.29 7.27 7.27 7.27 7.17 7.16 7.16 7.15 7.15 7.15 7.15 7.00 7.00 7.00 6.99 6.99 6.99 6.98 6.98 6.98 6.98 6.10 6.10 5.43 5.43 5.42 3.78 3.75 2.36

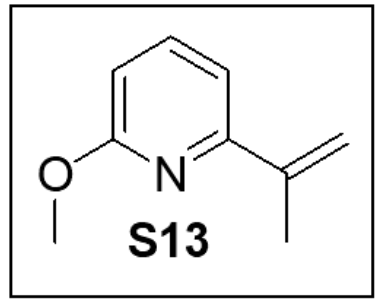
10 9 8 7 6 5 4 3 2 1 ppm

1.00 1.03 1.00 0.99 1.79 4.68 1.05 2.09 1.00 1.01 3.16 3.10 3.10

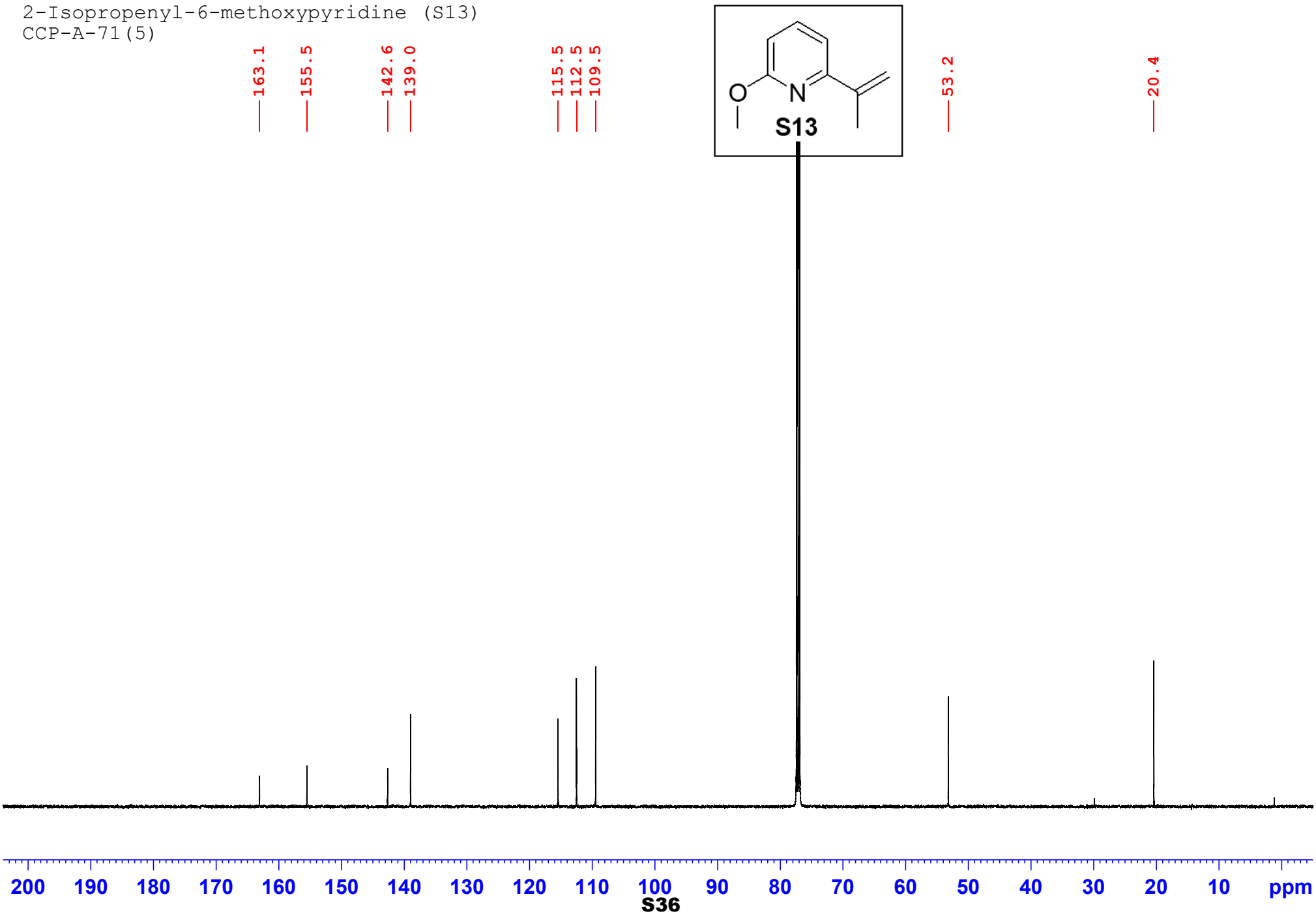
3-(6-Isopropenyl-pyridin-2-yl)-5,6-bis-(3-methoxy-phenyl)-[1,2,4]triazine (S11)
ZZG-G-61(2)



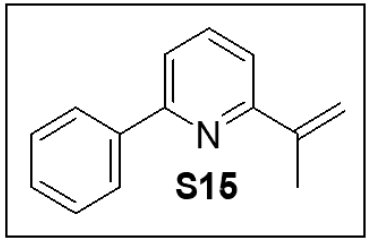
2-Isopropenyl-6-methoxypyridine (S13)
CCP-A-71(6)



2-Isopropenyl-6-methoxypyridine (S13)
CCP-A-71(5)



2-Isopropenyl-6-phenyl-pyridine (S15)
CCP-A-159(2)



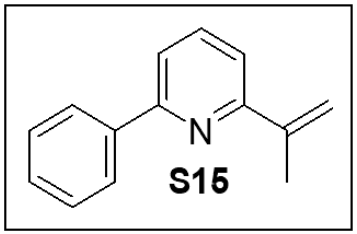
8.18 8.18 8.18 8.17 7.86 7.84 7.83 7.81 7.59 7.59 7.58 7.58 7.51 7.50 7.49 7.48 7.44 7.43 7.43 7.42 7.41 6.03 6.03 5.36 5.35 5.35

2.28 2.28

10 9 8 7 6 5 4 3 2 1 ppm

1.96 1.97 0.96 1.95 0.97 1.00 1.01 3.06

2-Isopropenyl-6-phenyl-pyridine (S15)
CCP-A-159(3)



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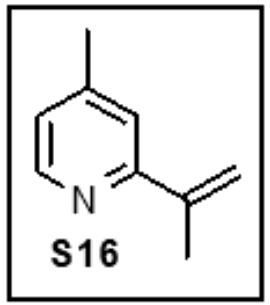
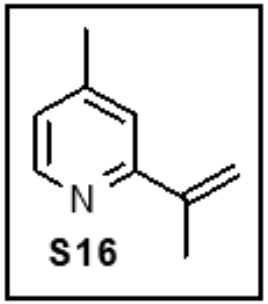


2-Isopropenyl-4-methyl-pyridine (S16)
CCP-A-81(10)



4-Isopropenyl-2-methyl-pyridine (S17)
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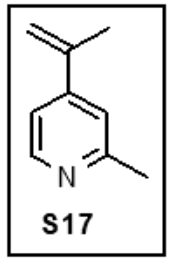

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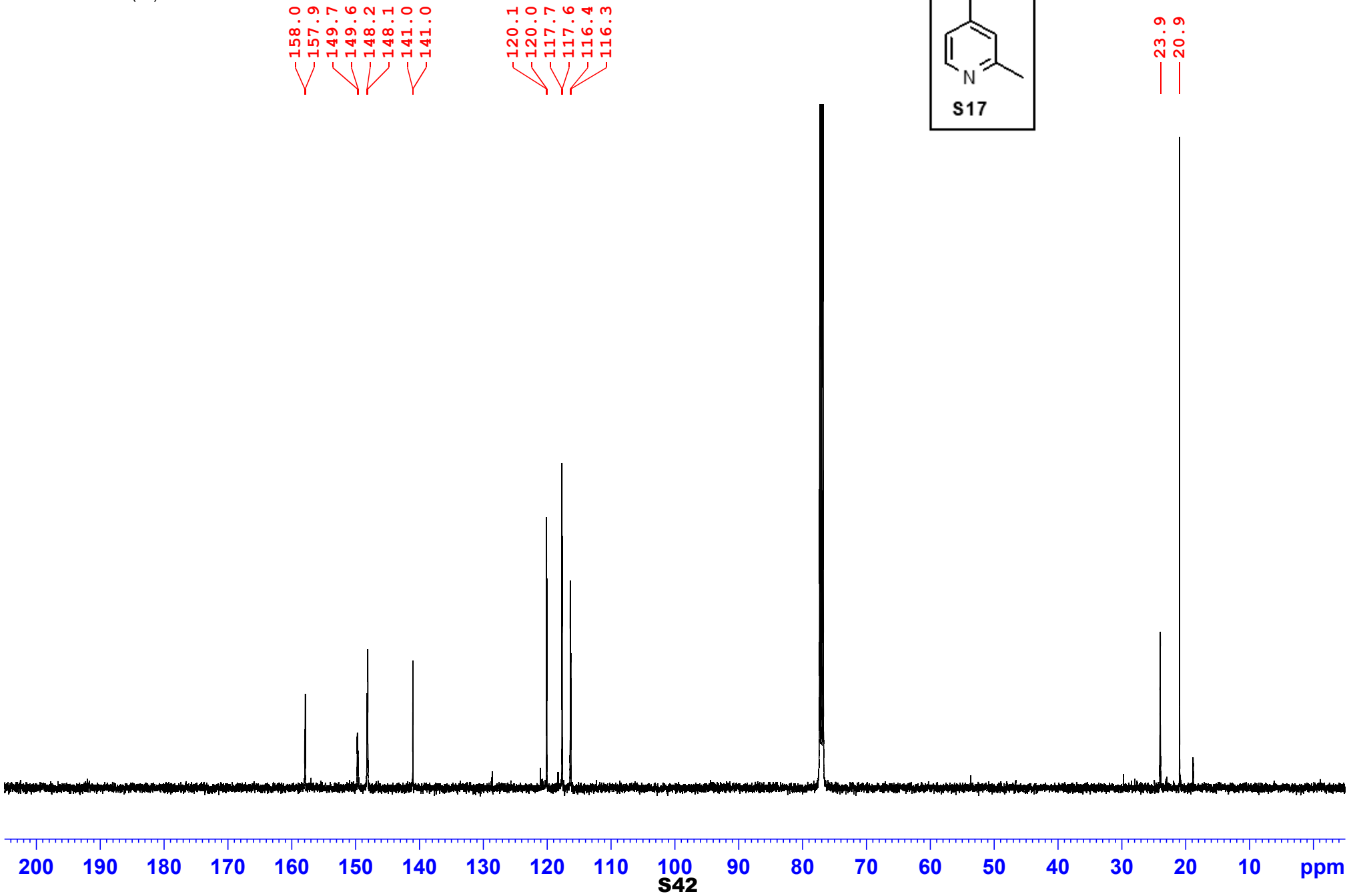
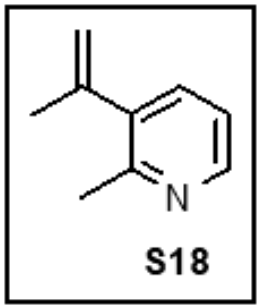
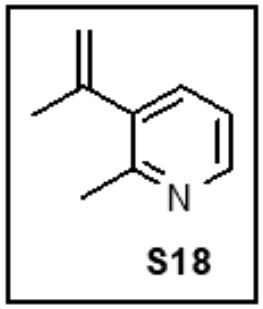


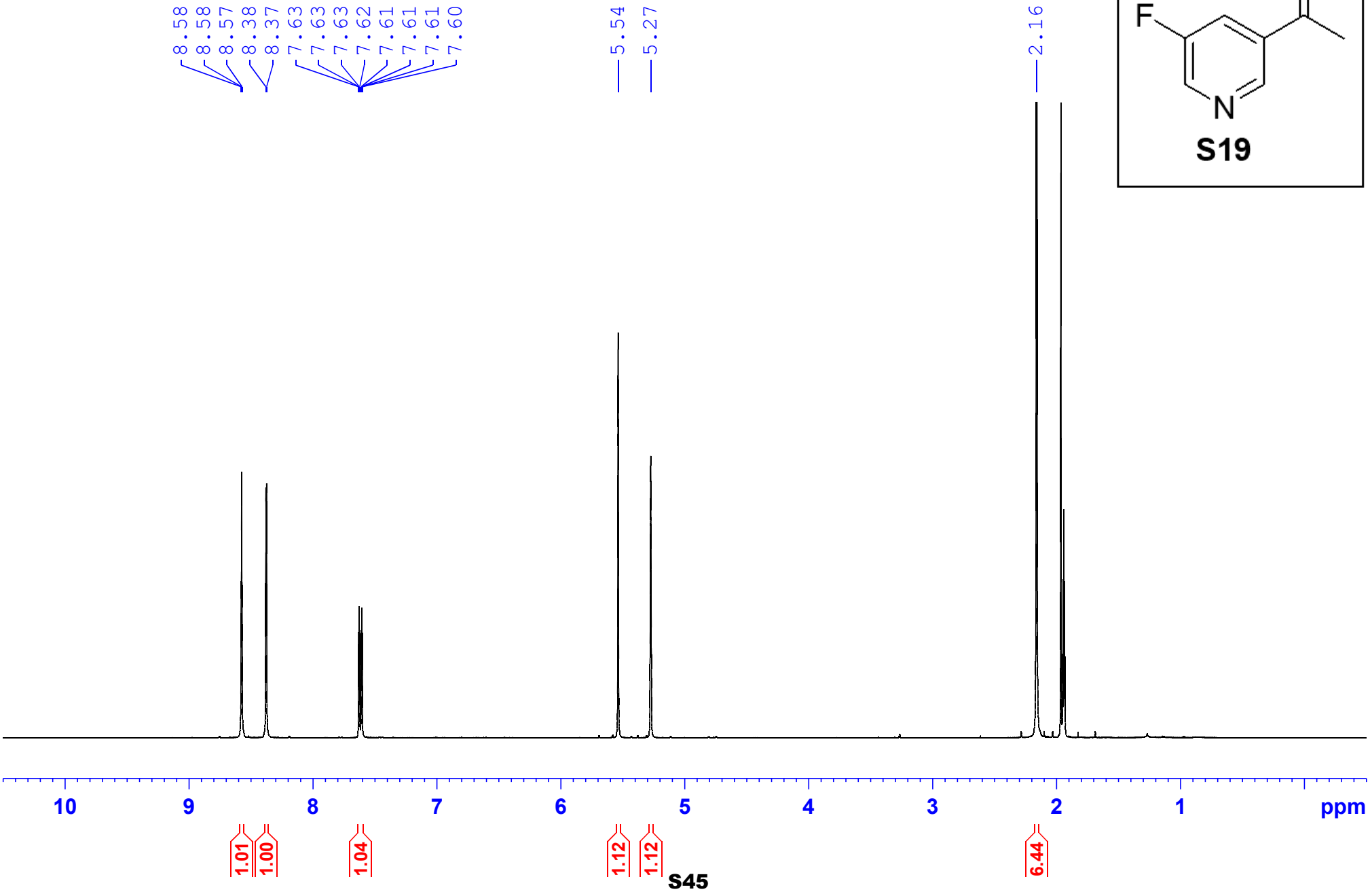
3-Isopropenyl-2-methyl-pyridine (S18)
CCP-A-163(1)



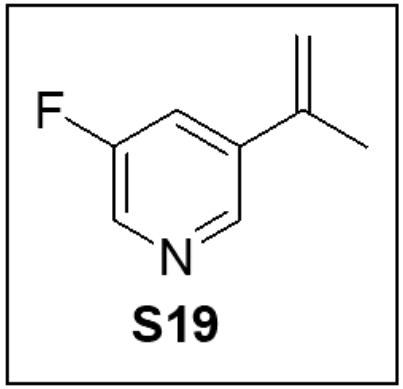
3-Isopropenyl-2-methyl-pyridine (S18)
CCP-A-163(1)



3-Fluoro-5-isopropenyl-pyridine (S19)
ZZG-J-19(4)

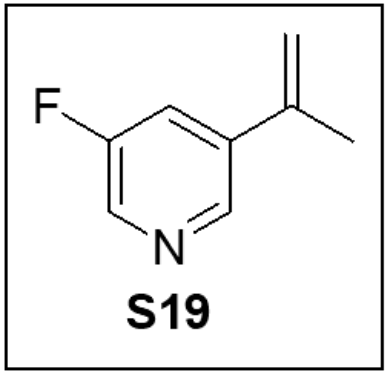


3-Fluoro-5-isopropenyl-pyridine (S19)
ZZG-J-19(4)



S19

3-Fluoro-5-isopropenyl-pyridine (S19)
ZZG-J-19(4)



3-Isopropenyl-5-methoxy-pyridine (S20)
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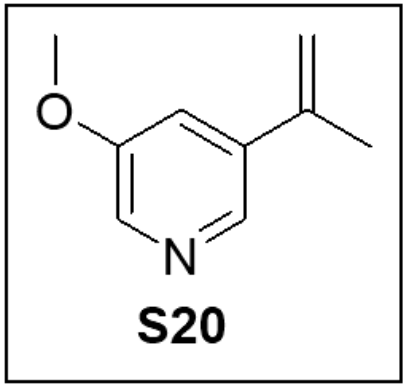

3-Isopropenyl-5-methoxy-pyridine (S20)
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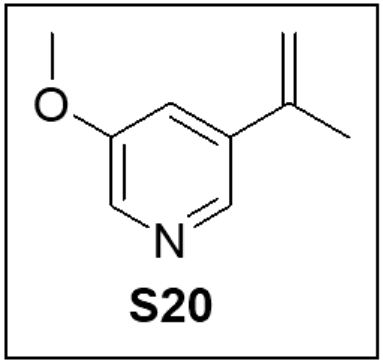

2,5-Diisopropenyl-pyridine (S21)
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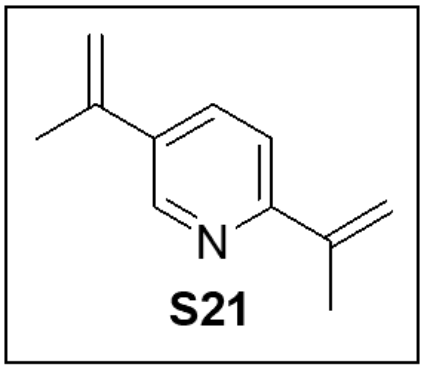
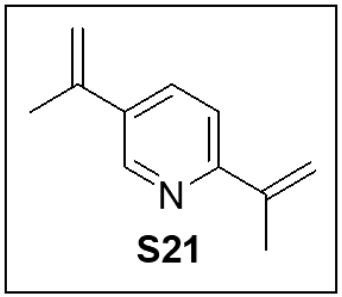
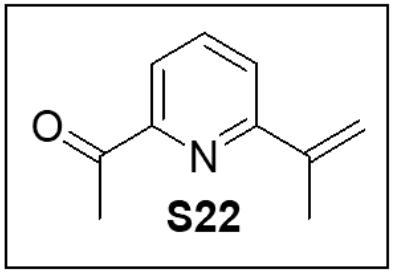


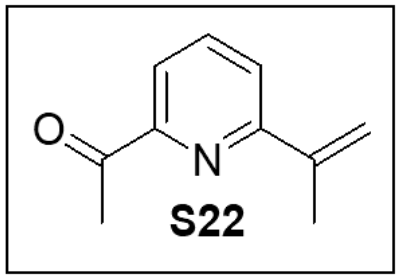
2,5-Diisopropenyl-pyridine (S21)
CCP-A-93(4)



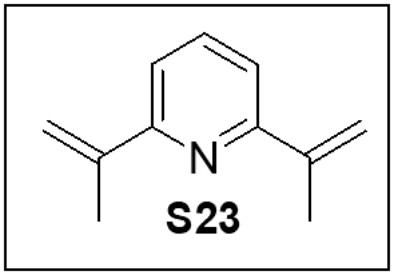
1-(6-Isopropenyl-pyridin-2-yl)-ethanone (S22)
CCP-A-101(3)



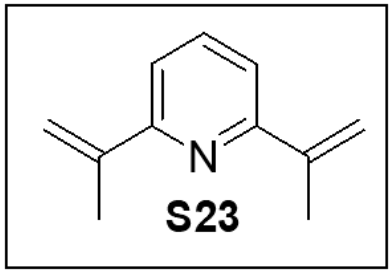
1-(6-Isopropenyl-pyridin-2-yl)-ethanone (S22)
CCP-A-101(4)



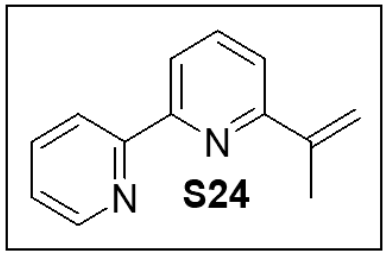
2,6-Diisopropenyl-pyridine (S23)
CCP-A-95(3)



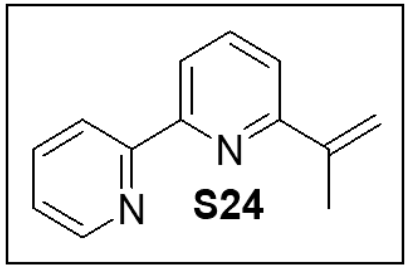
2,6-Diisopropenyl-pyridine (S23)
CCP-A-95(3)



6-Isoprenyl-[2,2']bipyridinyl (S24)
AMS-A-247(3)



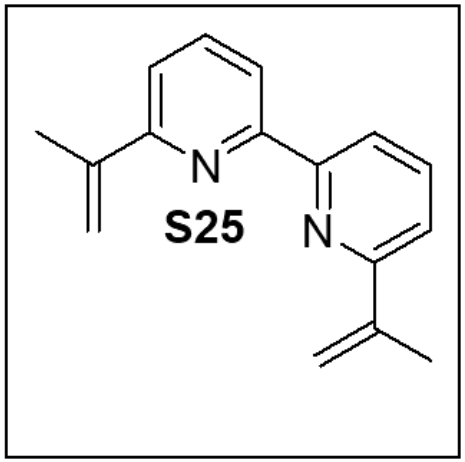
6-Isoprenyl-[2,2]bipyridinyl (S24)
AMS-A-247(3)



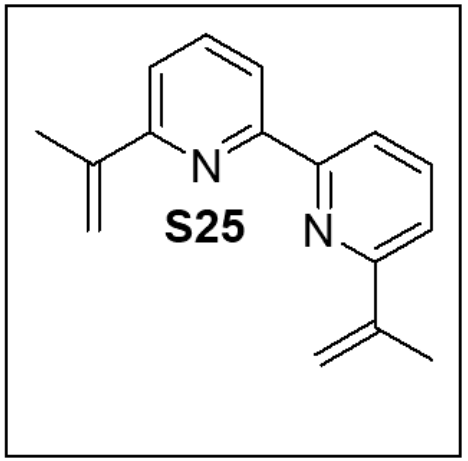
158.0
157.0
155.8
150.0
144.3
138.2
137.7
124.7
121.5
120.5
119.9
115.8
20.6

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

6,6'-Diisopropenyl-[2,2']bipyridine (S25)



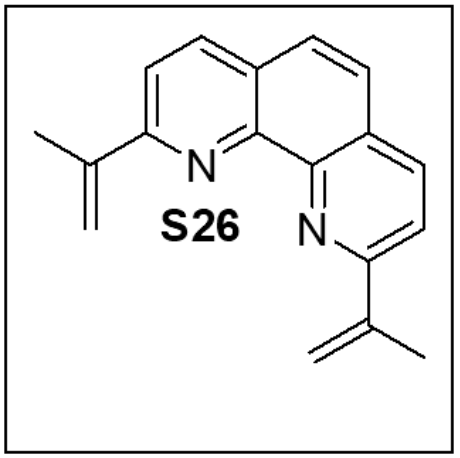
6,6'-Diisopropenyl-[2,2']bipyridine (S25)



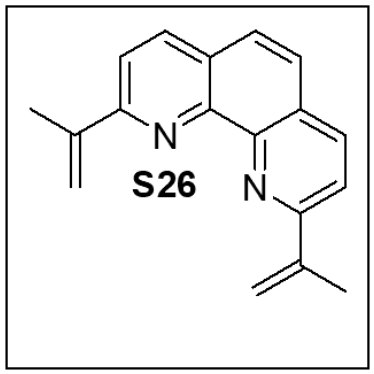
157.3
155.3
143.6
137.2
119.6
119.5
115.5
20.6

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

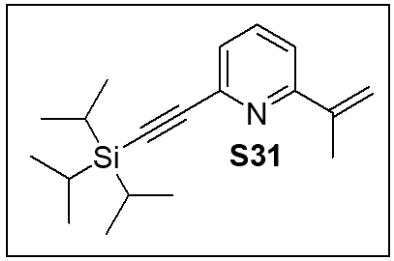
2,9-Diisoprenyl-[1,10]phenanthroline (S26)
AMS-B-17(3)



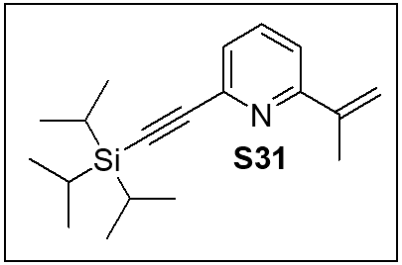
2,9-Diisoprenyl-[1,10]phenanthroline (S26)
AMS-B-17(5)



2-Isopropenyl-6-[(triisopropylsilanyl)-ethynyl]-pyridine (S31)
AMS-A-117(6)



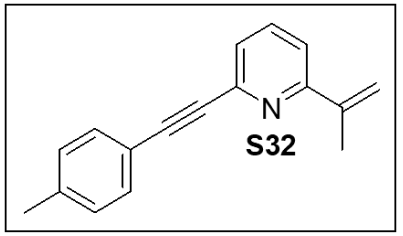
2-Isopropenyl-6-[(triisopropylsilanyl)-ethynyl]-pyridine (S31)
AMS-A-117(7)

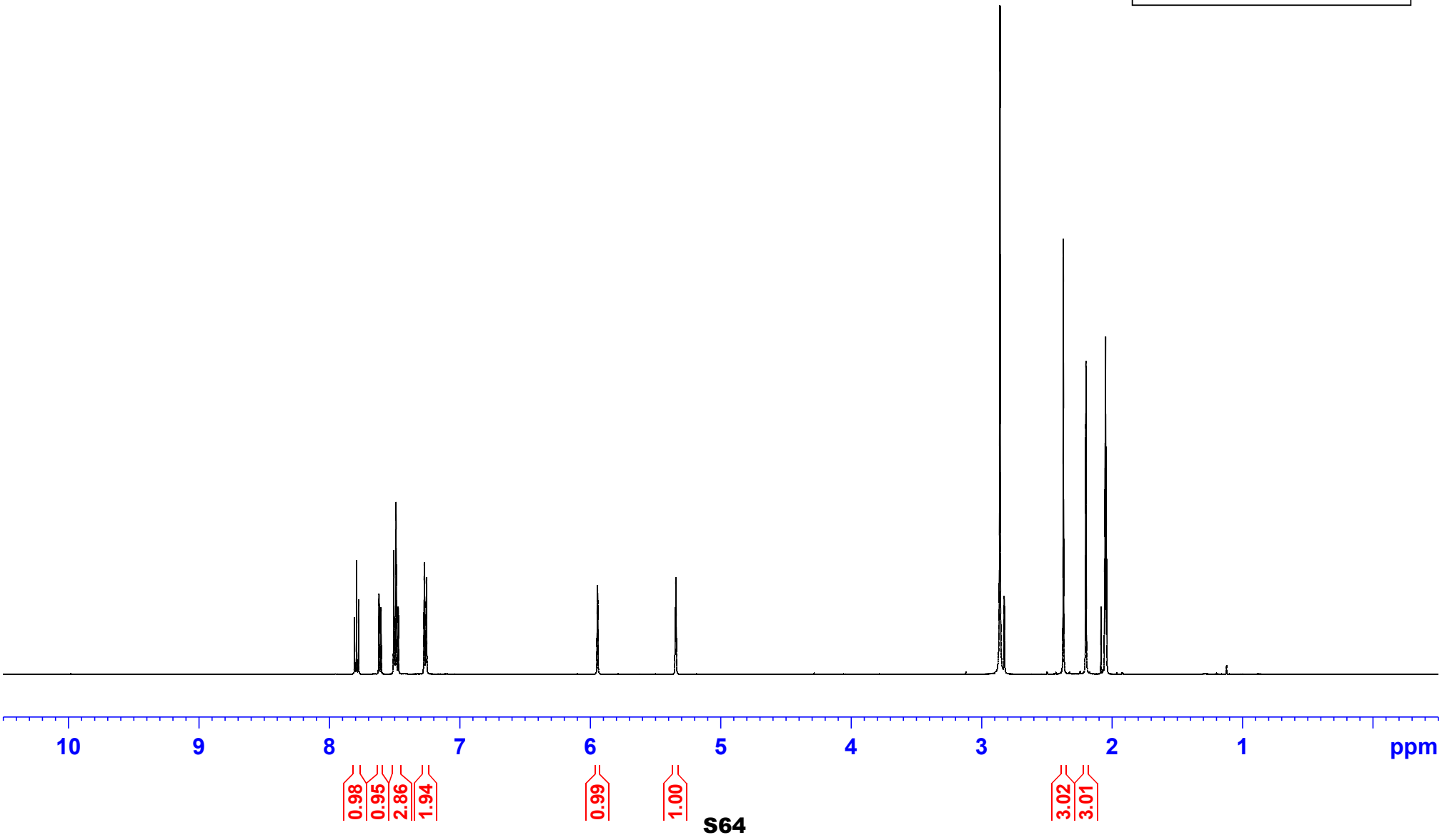


159.1
143.9
143.0
137.6
127.0
120.2
116.4
108.0
90.1
20.4
19.0
12.0

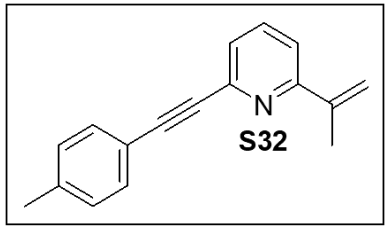
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

2-Isoprenyl-6-p-tolylethynyl-pyridine (S32)
AMS-B-25(2)

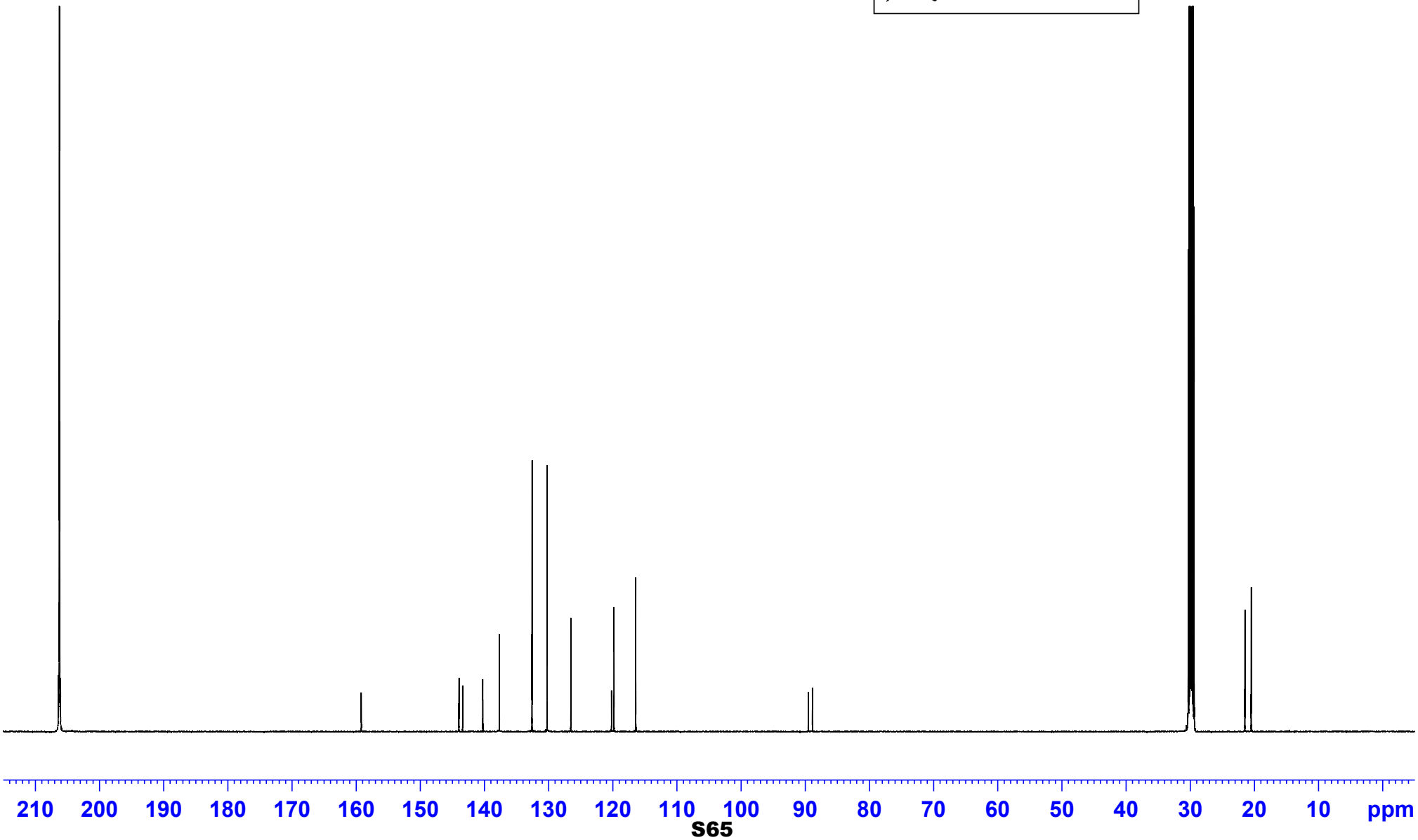




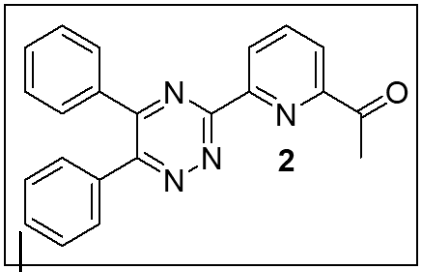
2-Isoprenyl-6-p-tolylethynyl-pyridine (S32)
AMS-B-25(2)



159.2
144.0
143.4
140.3
137.7
132.6
130.2
126.5
120.2
119.8
116.4
89.5
88.9
21.5
20.5



1-[6-(5,6-Diphenyl-[1,2,4]triazin-3-yl)-pyridin-2-yl]-ethanone (2)
ZZG-F-279(4)



8.85
8.83
8.24
8.23
8.10
8.09
8.07
7.74
7.72
7.68
7.67
7.48
7.47
7.46
7.43
7.42
7.40
7.38

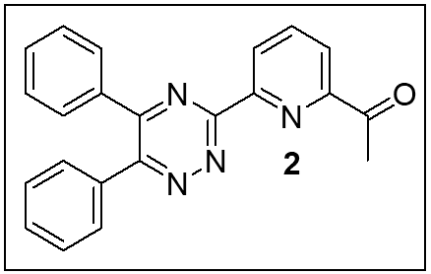
2.93

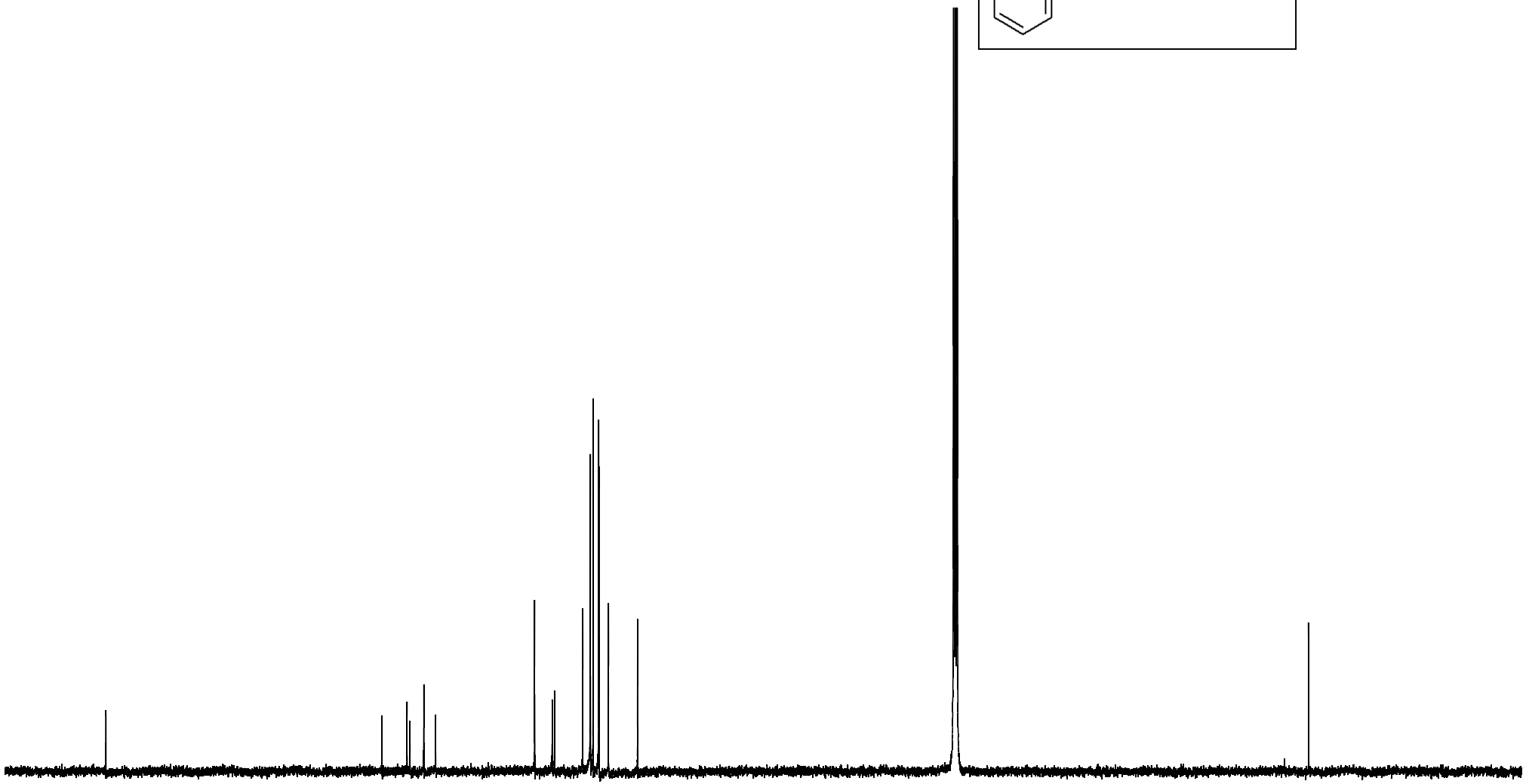
10 9 8 7 6 5 4 3 2 1 ppm

1.00 0.99 1.03 2.00 2.00 2.07 4.19 3.07

1-[6-(5,6-Diphenyl-[1,2,4]triazin-3-yl)-pyridin-2-yl]-ethanone (2)
ZZG-F-279(3)

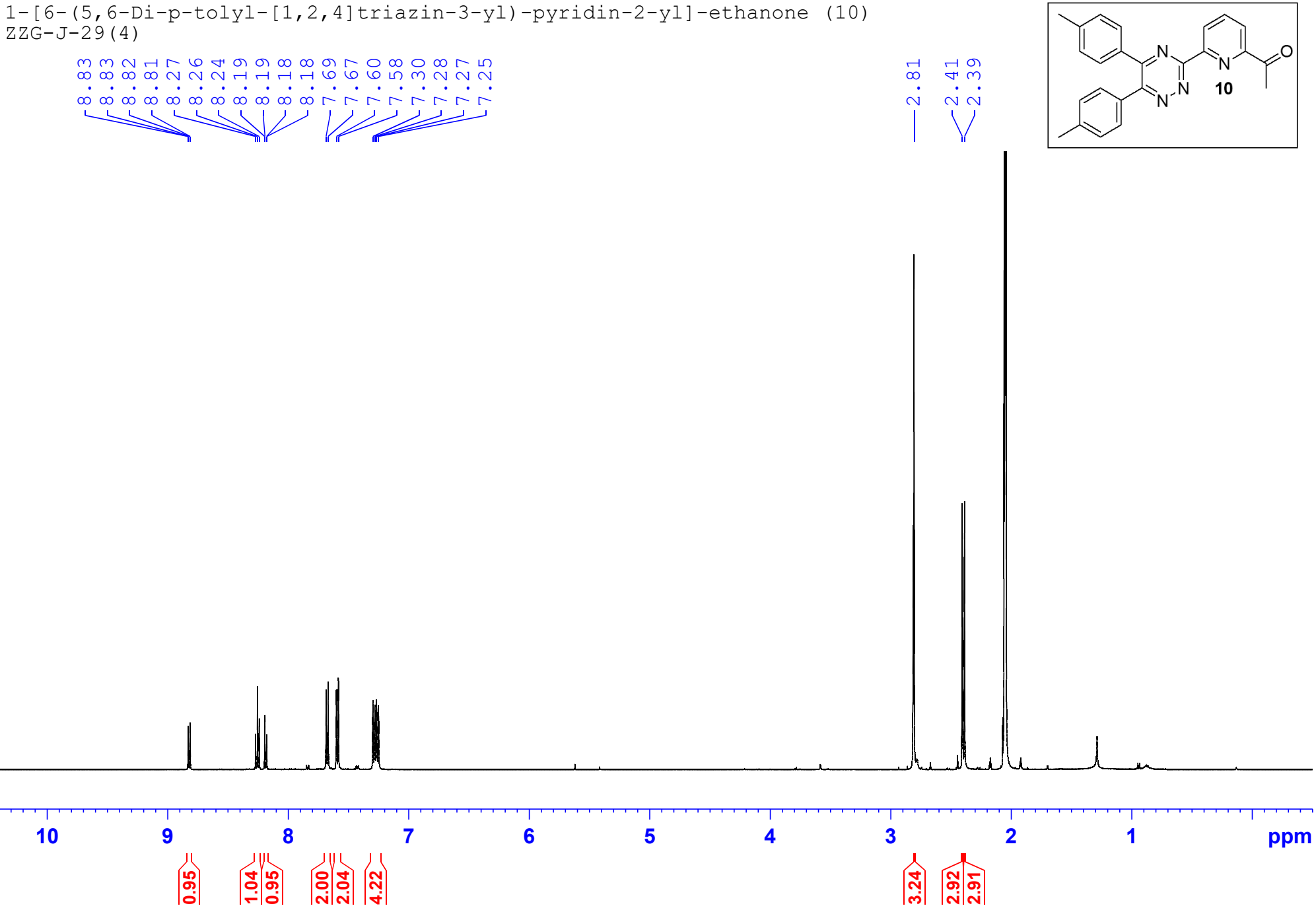
200.4
160.3
156.7
156.2
154.2
152.5
138.2
135.6
135.3
131.2
130.1
130.1
129.7
128.9
128.8
127.5
123.2
25.9



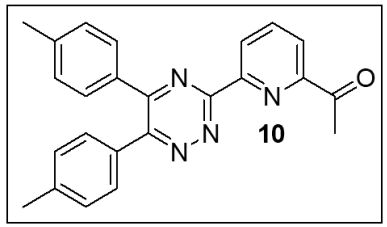


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

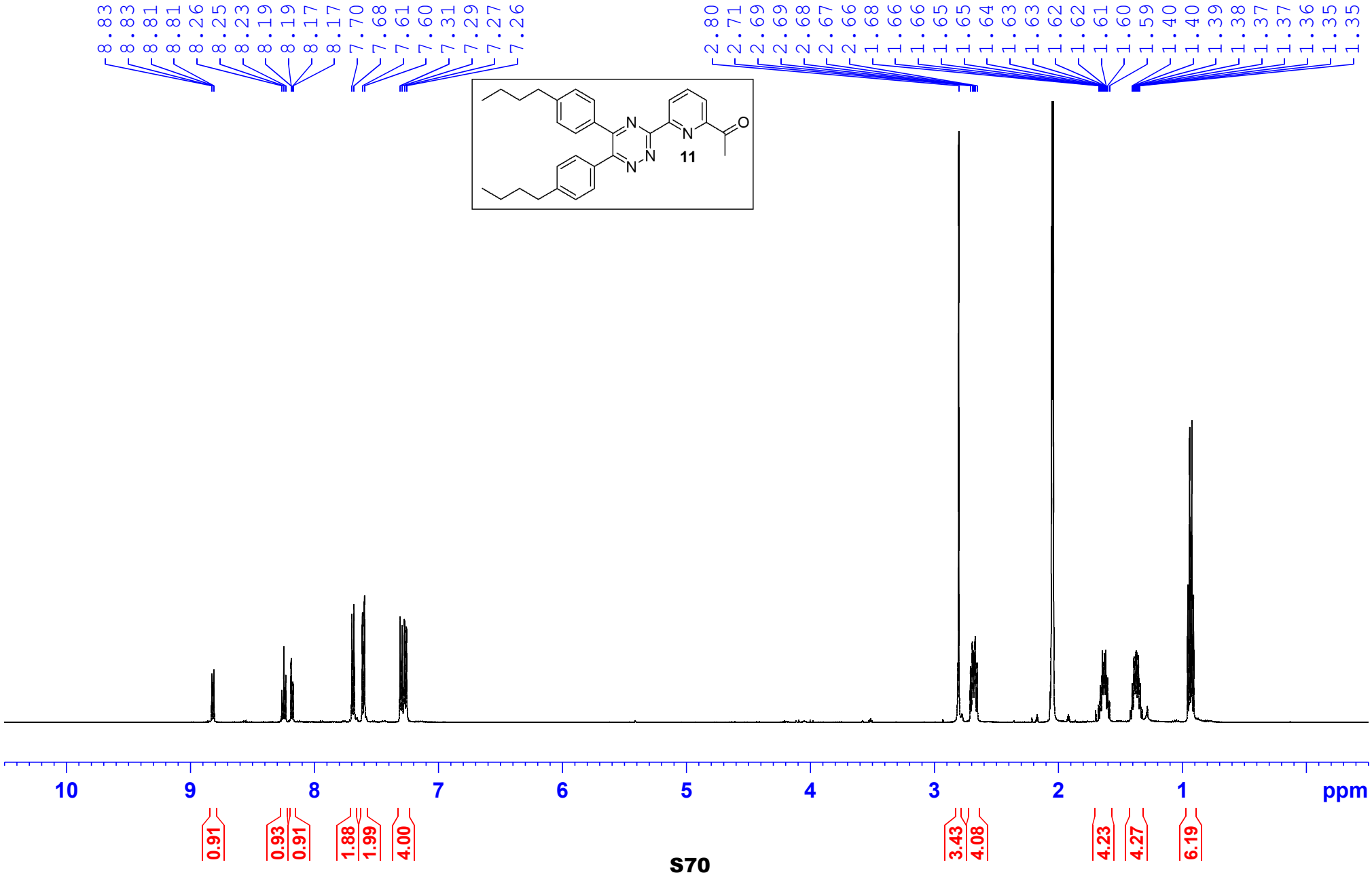
1-[6-(5,6-Di-p-tolyl-[1,2,4]triazin-3-yl)-pyridin-2-yl]-ethanone (10)
ZZG-J-29(4)



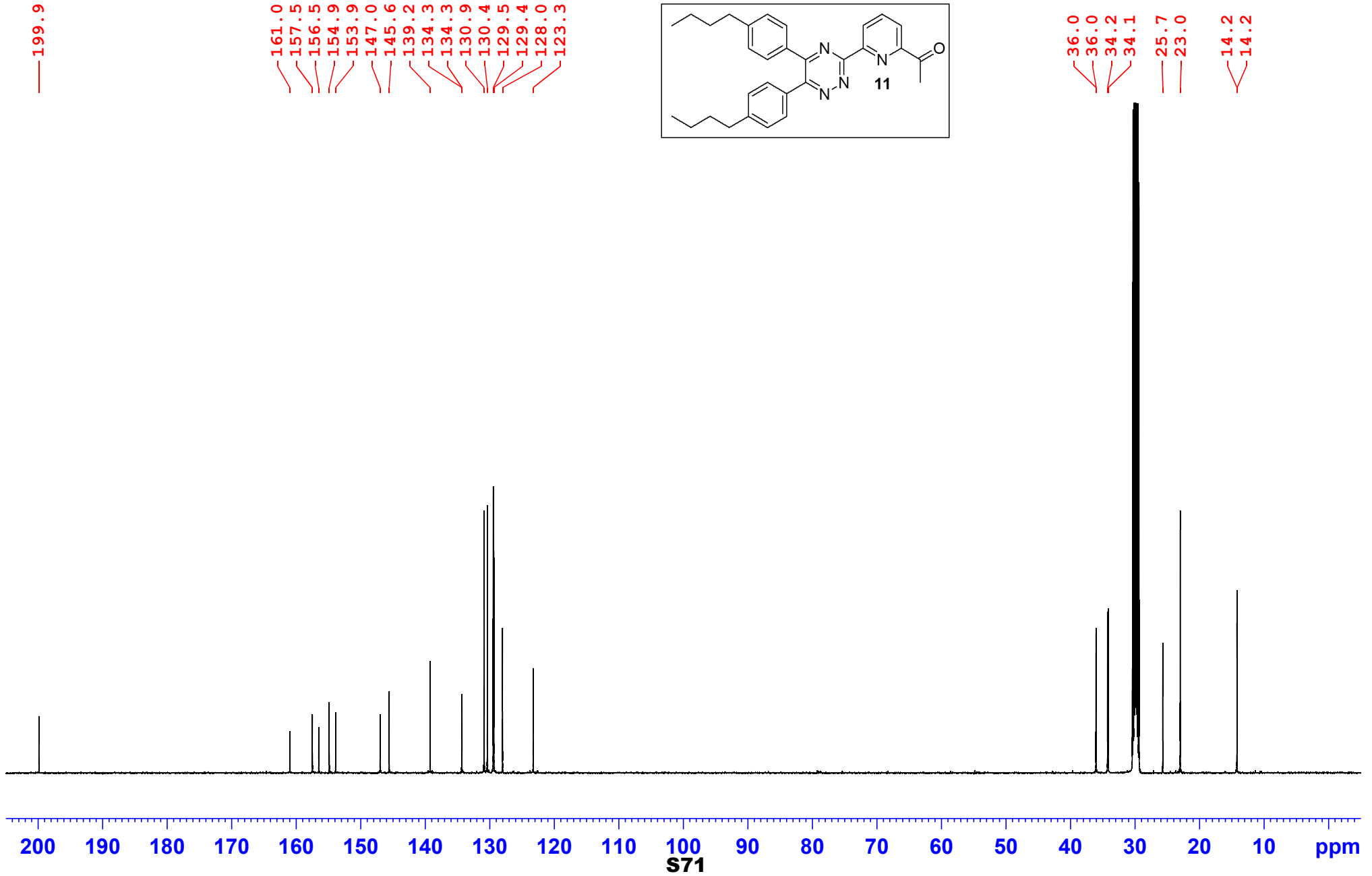
1-[6-(5,6-Di-p-tolyl-[1,2,4]triazin-3-yl)-pyridin-2-yl]-ethanone (10)
ZZG-J-29(5)



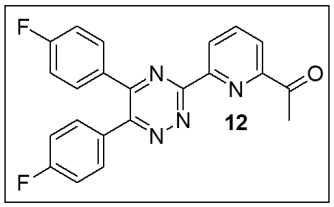
1-{6-[5,6-Bis-(4-butyl-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (11)
ZZG-J-31(4)



1-{6-[5,6-Bis-(4-butyl-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (11)
ZZG-J-31(5)

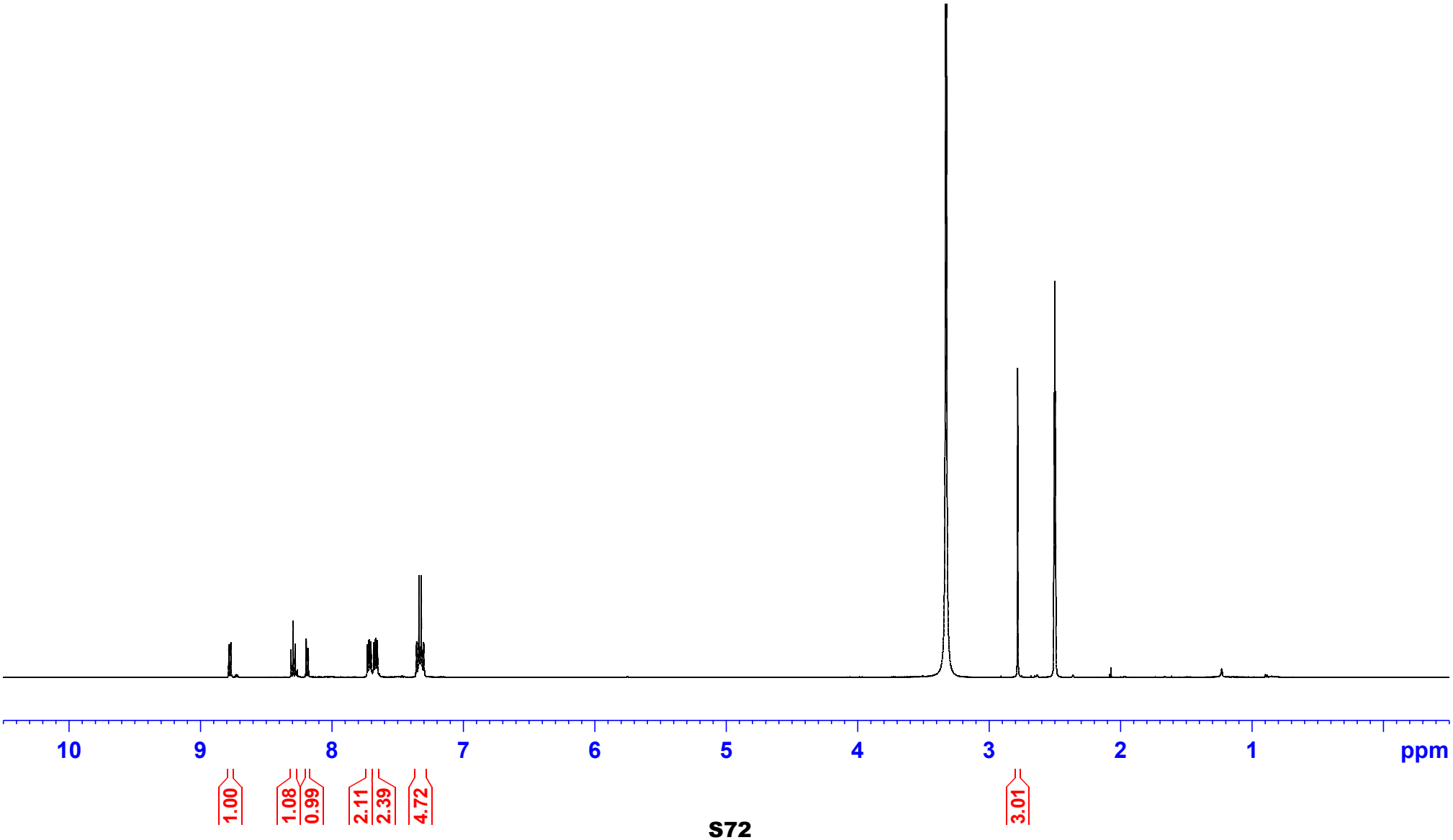


1-{6-[5,6-Bis-(4-fluoro-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (12)
ZZG-J-35(4)



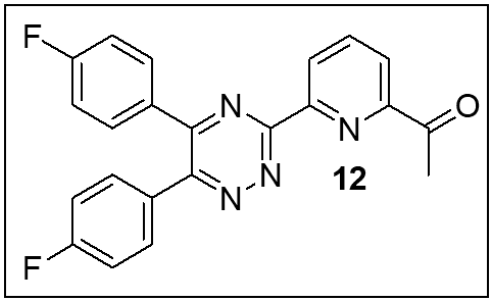
8.78 8.77 8.77 8.31 8.29 8.28 8.20 8.19 8.18 8.18 7.73 7.72 7.71 7.71 7.70 7.68 7.68 7.67 7.66 7.66 7.65 7.36 7.34 7.34 7.33 7.32 7.32 7.32 7.30

2.78



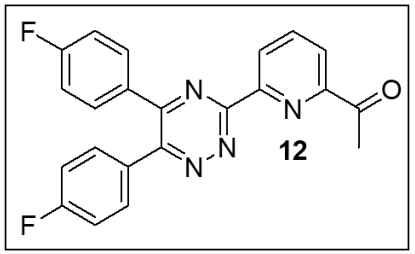
1-{6-[5,6-Bis-(4-fluoro-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (12)
ZZG-J-35(6)

199.3
164.5
164.0
162.6
162.0
159.9
155.8
155.1
153.4
152.1
139.0
132.4
132.4
131.9
131.8
131.7
131.7
131.7
131.7
127.5
122.8
115.8
115.8
115.7
115.6
25.5

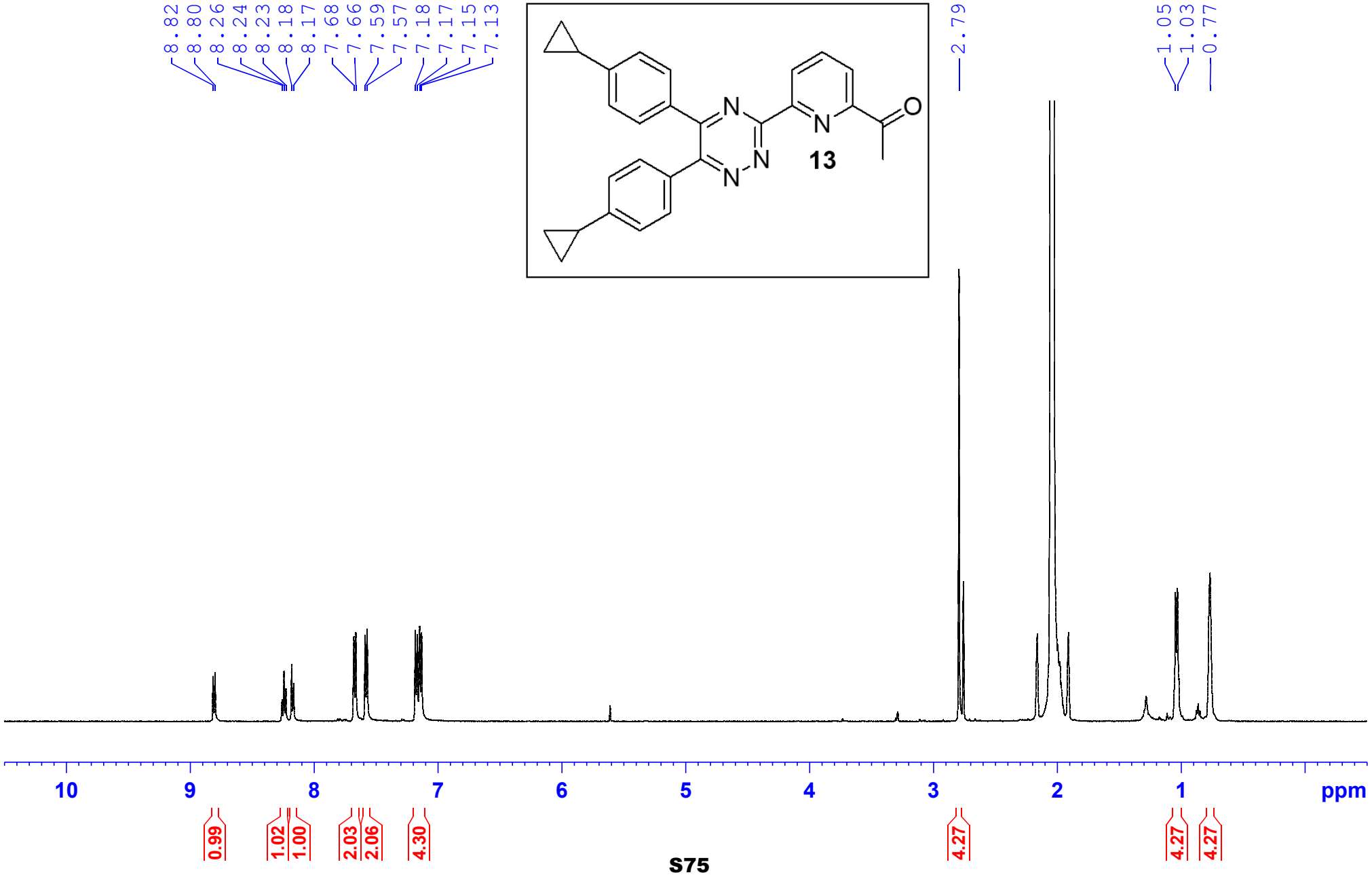


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

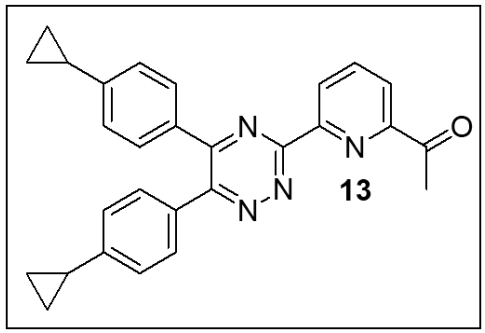
1-{6-[5,6-Bis-(4-fluoro-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (12)
ZZG-J-35(6)



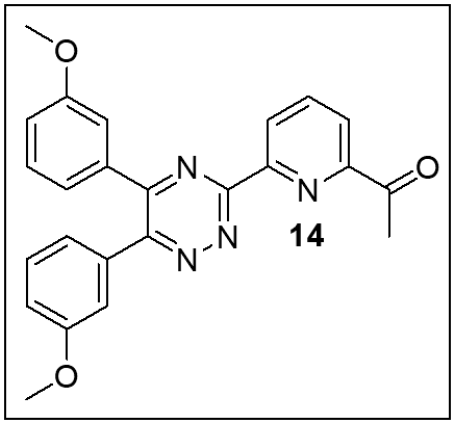
1-{6-[5,6-Bis-(4-cyclopropyl-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (13)
AMS-A-251(4)



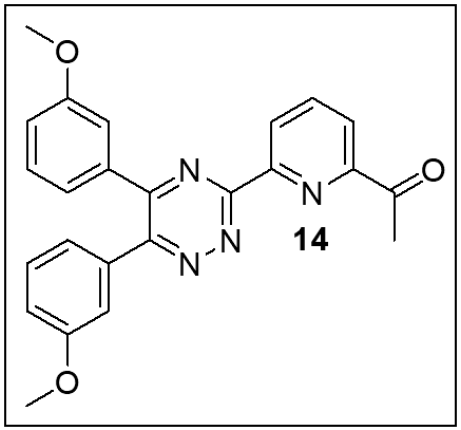
1-{6-[5,6-Bis-(4-cyclopropyl-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (13)
AMS-A-241(4)



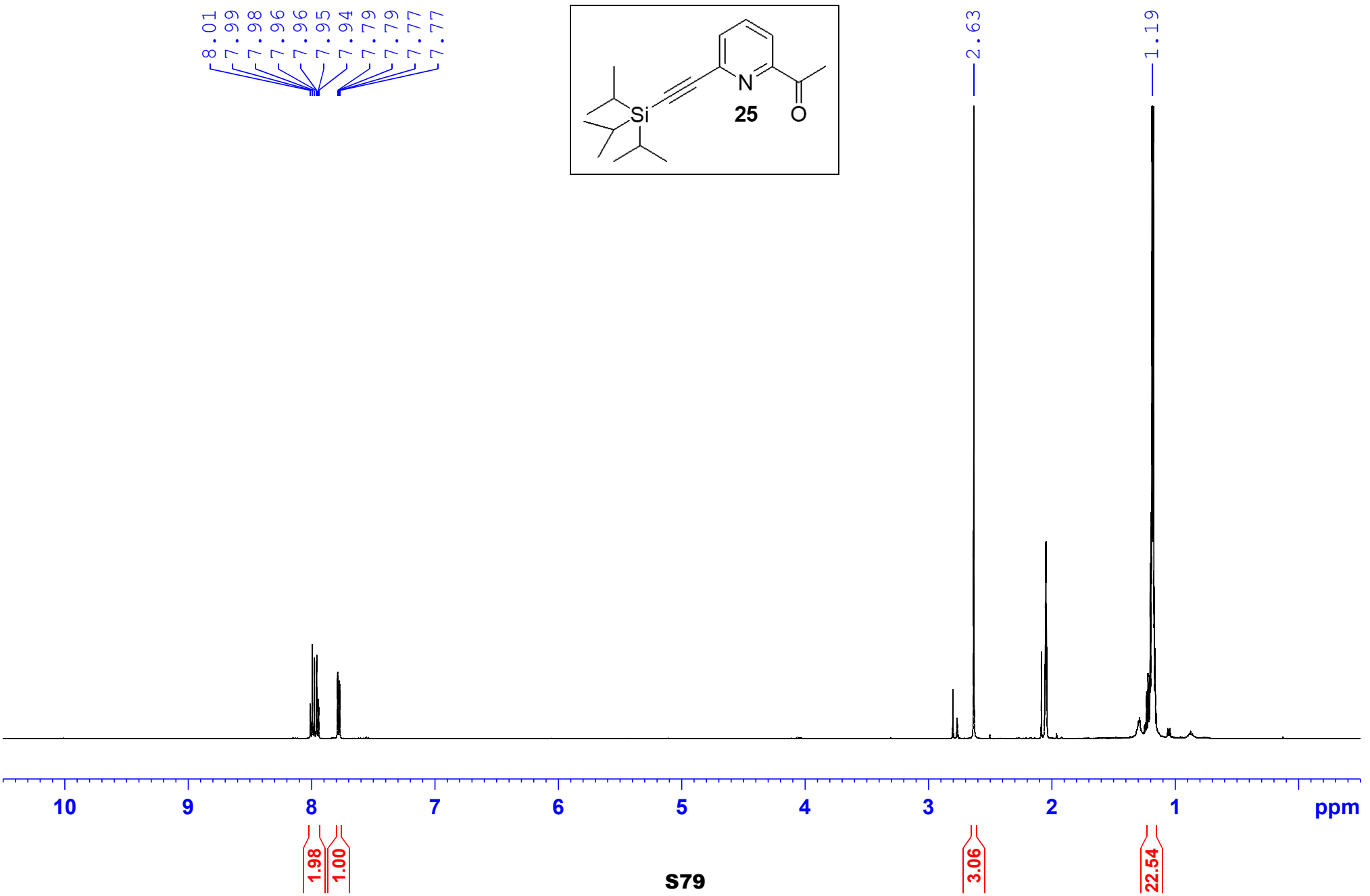
1-{6-[5,6-Bis-(3-methoxy-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (14)
ZZG-G-43(1)



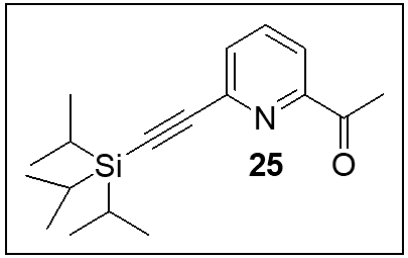
1-{6-[5,6-Bis-(3-methoxy-phenyl)-[1,2,4]triazin-3-yl]-pyridin-2-yl}-ethanone (14)
ZZG-G-43(1)



1-{6-[(Triisopropylsilanyl)-ethynyl]-pyridin-2-yl}-ethanone (25)
AMS-A-203(17)



1-{6-[(Triisopropylsilanyl)-ethynyl]-pyridin-2-yl}-ethanone (25)
AMS-A-203(17)



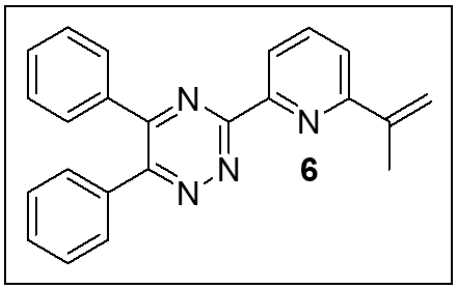
199.2
154.8
143.3
138.6
131.8
121.5
106.9
91.9
25.5
19.0
12.0

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

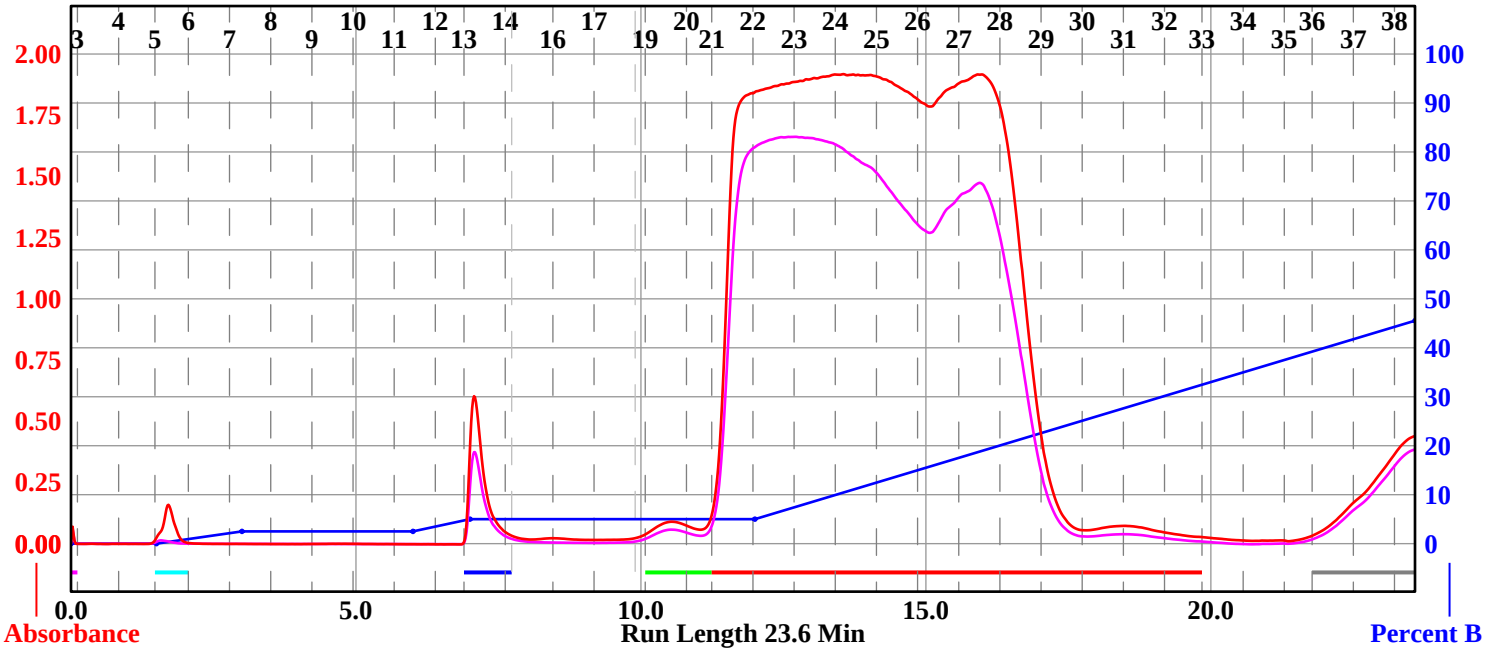
Sample: ZZG-G-63 **Rf+** **Wednesday 09 September 2020 06:50AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 1.0 min
Solvent A: Hexane
Solvent B: MTBE

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:5	A:5
3	A:13	A:14
4	A:19	A:20
5	A:21	A:32
6	A:36	A:38

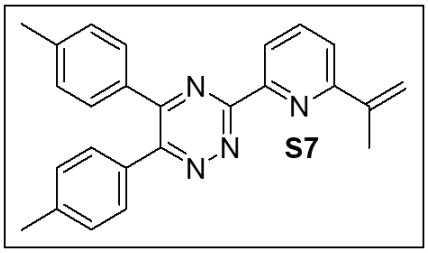
Duration	%B	Solvent A	Solvent B
0.0	0.0	Hexane	MTBE
1.5	0.0	Hexane	MTBE
1.5	2.5	Hexane	MTBE
3.0	2.5	Hexane	MTBE
1.0	5.0	Hexane	MTBE
5.0	5.0	Hexane	MTBE
11.6	45.5	Hexane	MTBE

S81

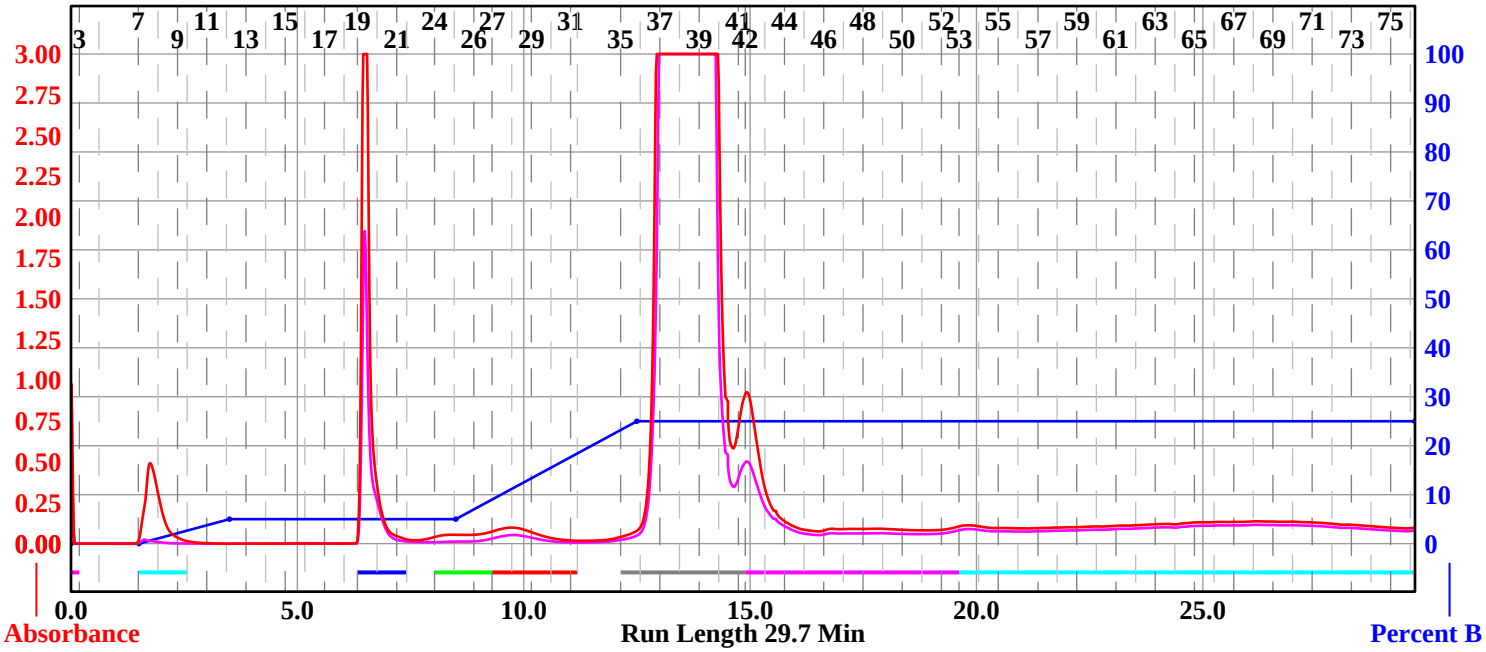
Sample: ZZG-F-251 **Rf+** **Wednesday 05 August 2020 11:25AM**

RediSep Column: Silica 12g
SN: E04150D81BD3C Lot: 301341205X
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent A: Hexane
Solvent B: Ethyl Acetate

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:7	A:9
3	A:19	A:21
4	A:24	A:26
5	A:27	A:31
6	A:35	A:41
7	A:42	A:52
8	A:53	A:1

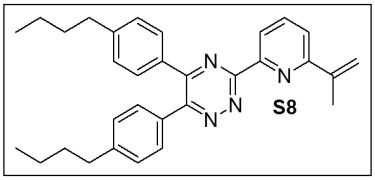
Duration	%B	Solvent A	Solvent B
0.0	0.0	Hexane	Ethyl Acetate
1.5	0.0	Hexane	Ethyl Acetate
2.0	5.0	Hexane	Ethyl Acetate
5.0	5.0	Hexane	Ethyl Acetate
4.0	25.0	Hexane	Ethyl Acetate
17.2	25.0	Hexane	Ethyl Acetate

S82

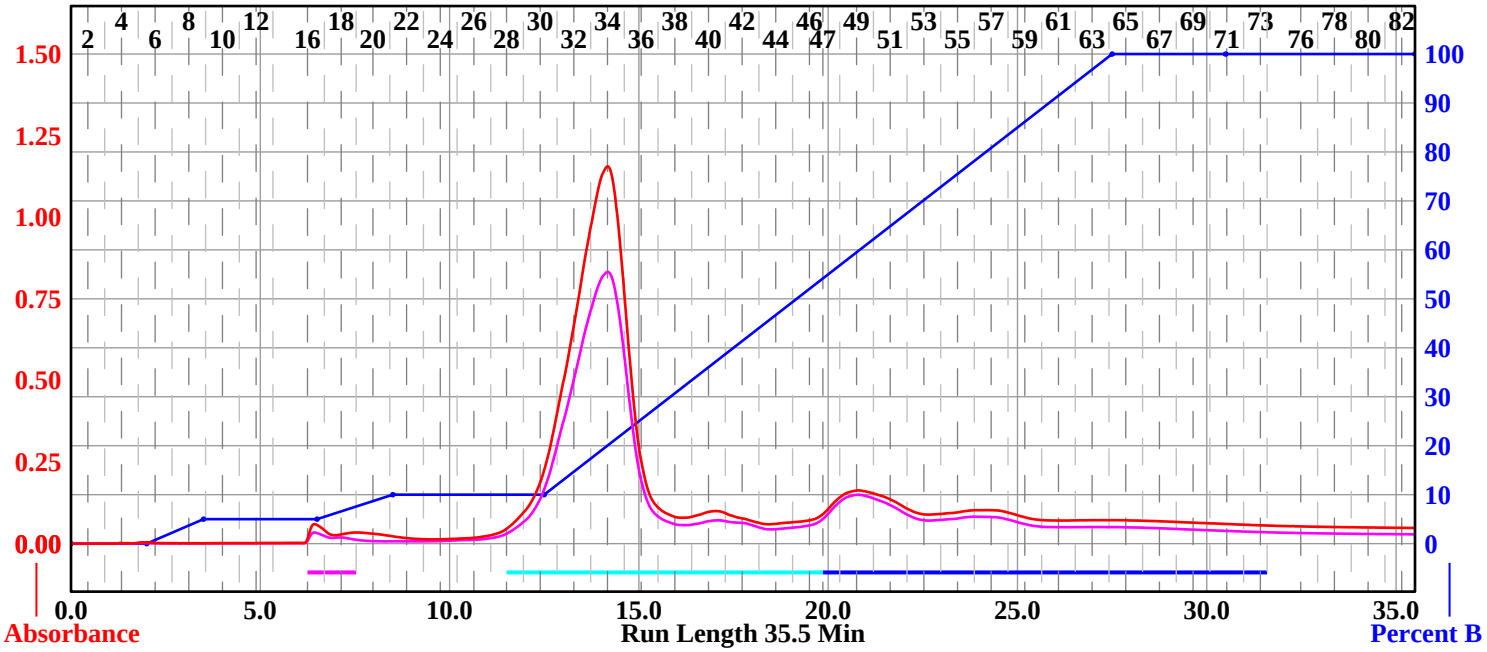
Sample: ZZG-G-11 **Rf+** **Friday 28 August 2020 07:42AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent A: Hexane
Solvent B: 1:1 MTBE:EtOAc

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

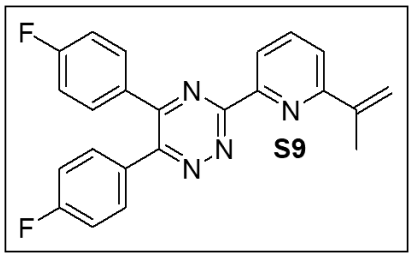
Peak #	Start Tube	End Tube
1	A:16	A:18
2	A:28	A:46
3	A:47	A:73

Duration	%B	Solvent A	Solvent B
0.0	0.0	Hexane	1:1 MTBE:EtOAc
2.0	0.0	Hexane	1:1 MTBE:EtOAc
1.5	5.0	Hexane	1:1 MTBE:EtOAc
3.0	5.0	Hexane	1:1 MTBE:EtOAc
2.0	10.0	Hexane	1:1 MTBE:EtOAc
4.0	10.0	Hexane	1:1 MTBE:EtOAc
15.0	100.0	Hexane	1:1 MTBE:EtOAc
3.0	100.0	Hexane	1:1 MTBE:EtOAc
5.0	100.0	Hexane	1:1 MTBE:EtOAc

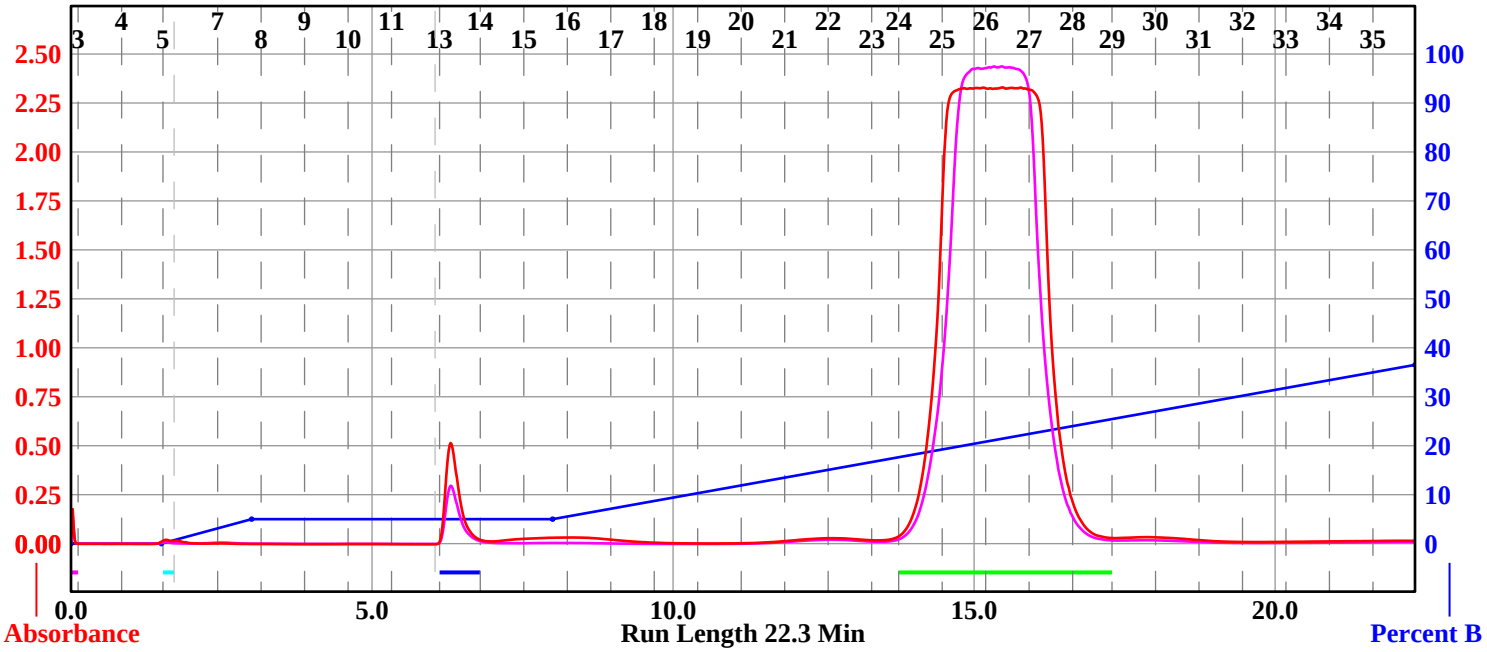
Sample: ZZG-G-51 | **Rf+** | **Friday 04 September 2020 12:44PM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent A: Hexane
Solvent B: 1:1 MTBE:EtOAc

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

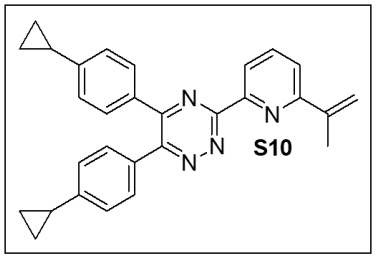
Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:5	A:5
3	A:13	A:13
4	A:24	A:28

Duration	%B	Solvent A	Solvent B
0.0	0.0	Hexane	1:1 MTBE:EtOAc
1.5	0.0	Hexane	1:1 MTBE:EtOAc
1.5	5.0	Hexane	1:1 MTBE:EtOAc
5.0	5.0	Hexane	1:1 MTBE:EtOAc
14.3	36.5	Hexane	1:1 MTBE:EtOAc

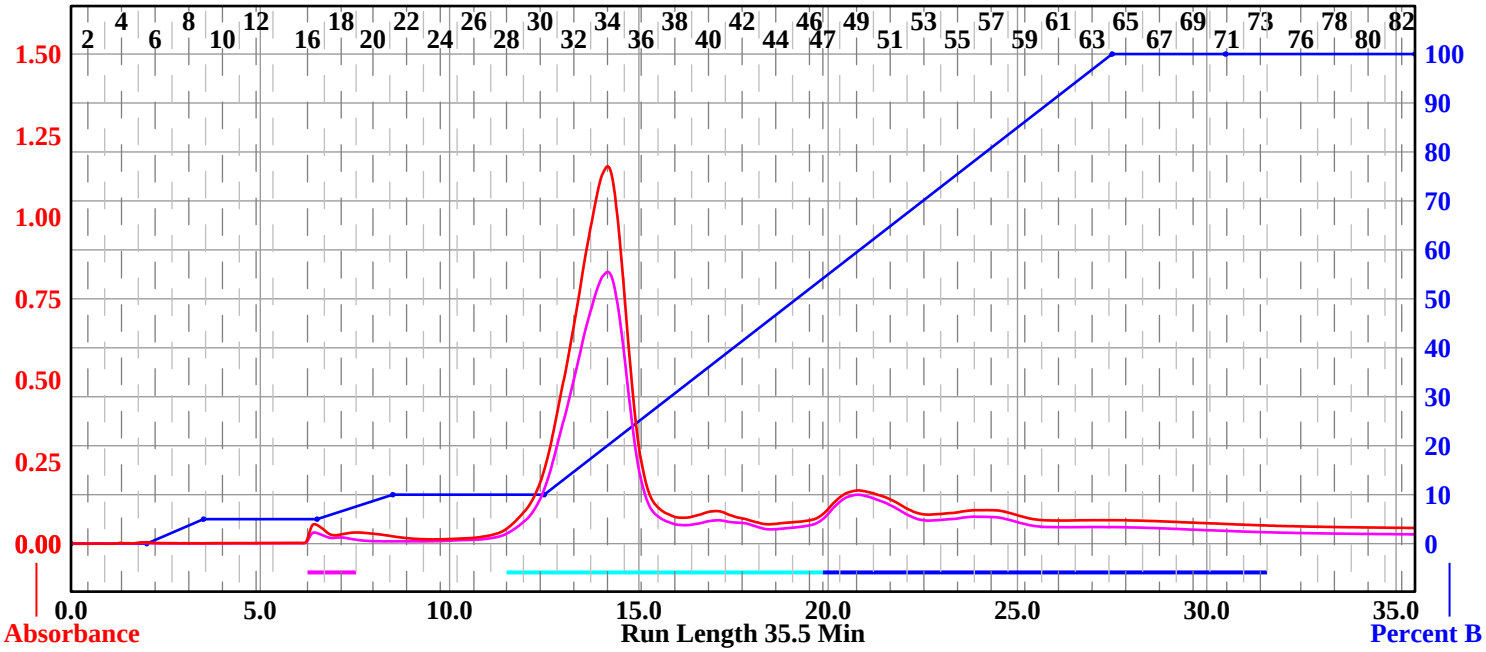
Sample: ZZG-G-11 | **Rf+** | **Friday 28 August 2020 07:42AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent A: Hexane
Solvent B: 1:1 MTBE:EtOAc

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:16	A:18
2	A:28	A:46
3	A:47	A:73

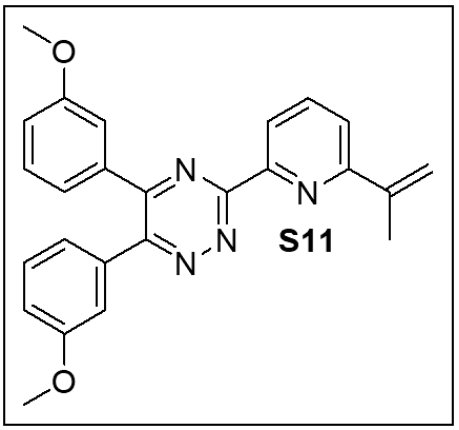
Duration	%B	Solvent A	Solvent B
0.0	0.0	Hexane	1:1 MTBE:EtOAc
2.0	0.0	Hexane	1:1 MTBE:EtOAc
1.5	5.0	Hexane	1:1 MTBE:EtOAc
3.0	5.0	Hexane	1:1 MTBE:EtOAc
2.0	10.0	Hexane	1:1 MTBE:EtOAc
4.0	10.0	Hexane	1:1 MTBE:EtOAc
15.0	100.0	Hexane	1:1 MTBE:EtOAc
3.0	100.0	Hexane	1:1 MTBE:EtOAc
5.0	100.0	Hexane	1:1 MTBE:EtOAc

S85

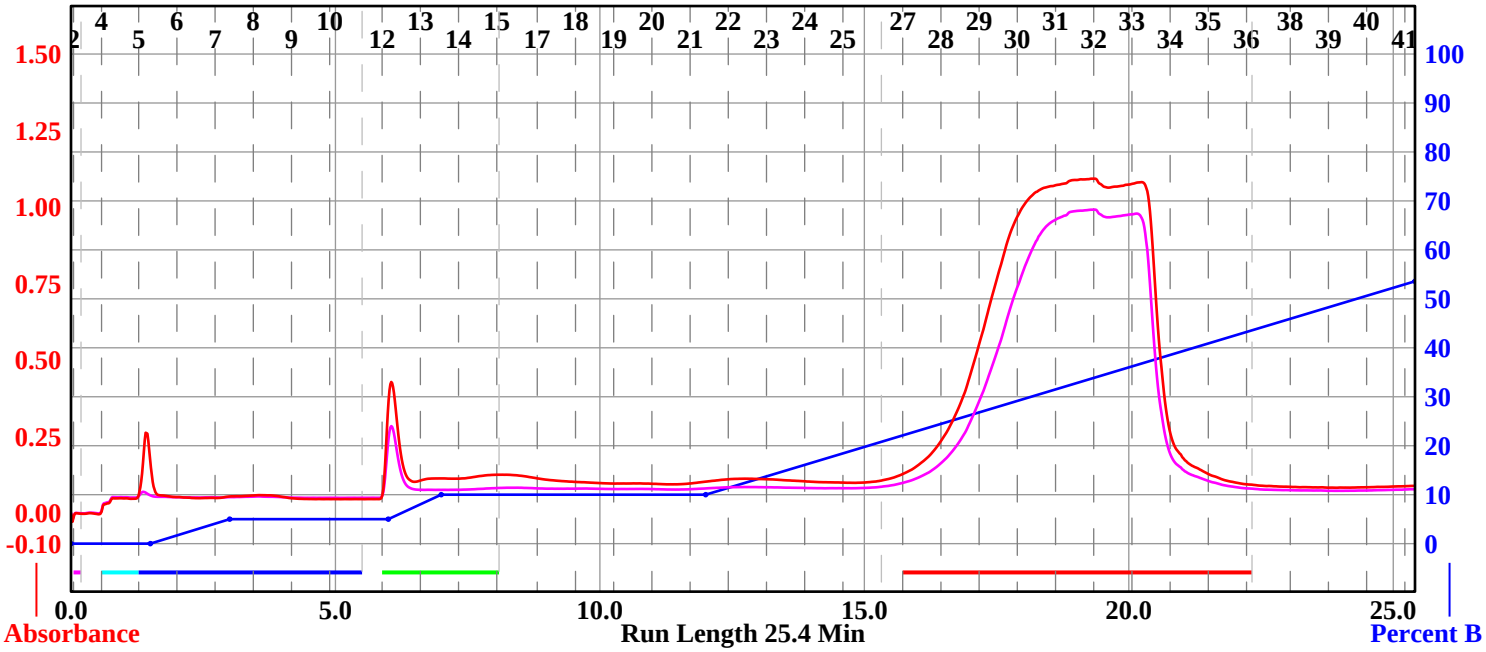
Sample: ZZG-G-61 **Rf+** **Wednesday 09 September 2020 06:15AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 1.0 min
Solvent A: Hexane
Solvent B: 1:1 MTBE:EtOAc

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:4	A:4
3	A:5	A:10
4	A:12	A:15
5	A:27	A:36

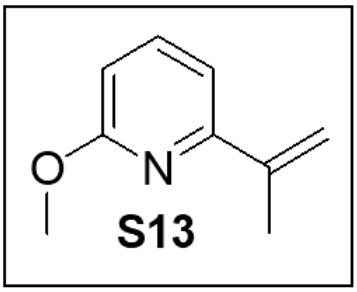
Duration	%B	Solvent A	Solvent B
0.0	0.0	Hexane	1:1 MTBE:EtOAc
1.5	0.0	Hexane	1:1 MTBE:EtOAc
1.5	5.0	Hexane	1:1 MTBE:EtOAc
3.0	5.0	Hexane	1:1 MTBE:EtOAc
1.0	10.0	Hexane	1:1 MTBE:EtOAc
5.0	10.0	Hexane	1:1 MTBE:EtOAc
13.4	53.6	Hexane	1:1 MTBE:EtOAc

S86

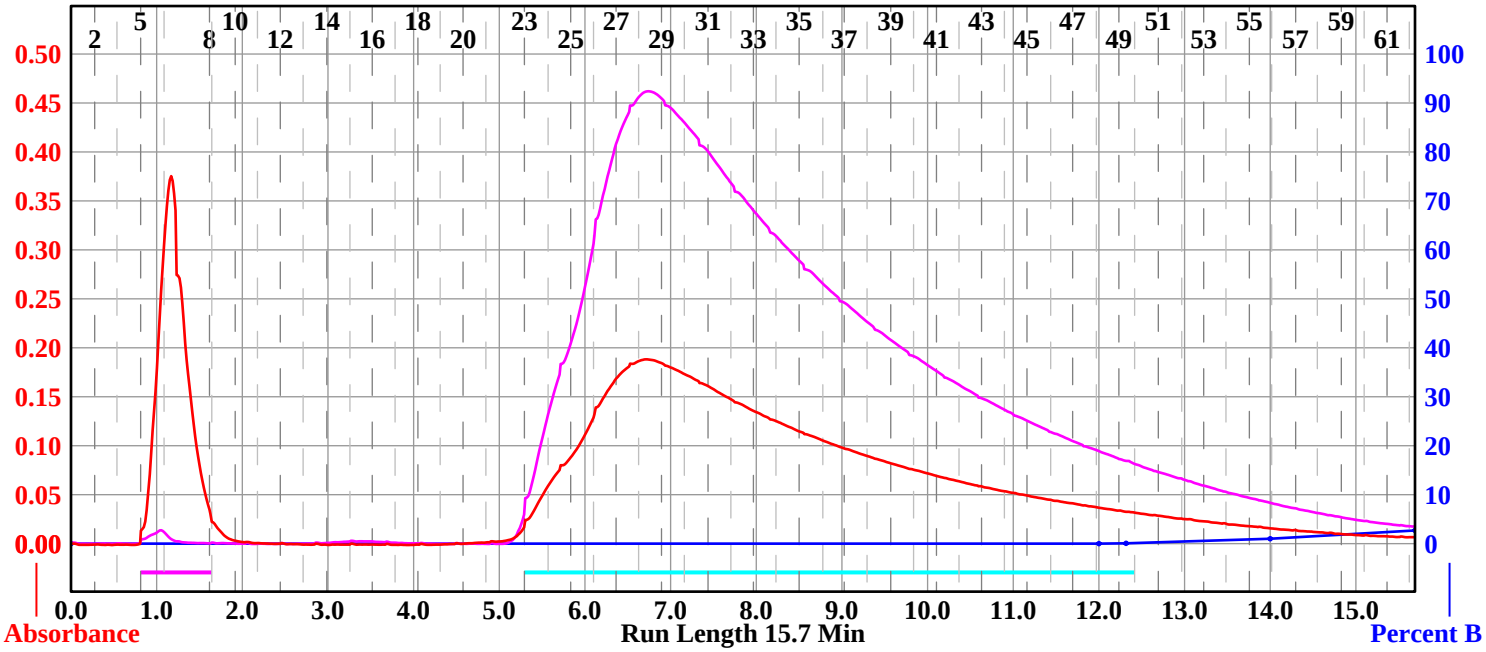
Sample: ccp-a-71 | **Rf 200** | **Tuesday 15 June 2021 06:47AM**

RediSep Column: Silica 12g
SN: E04150E8066AB Lot: 302224502X
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B2 MTBE

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:5	A:8
2	A:23	A:49

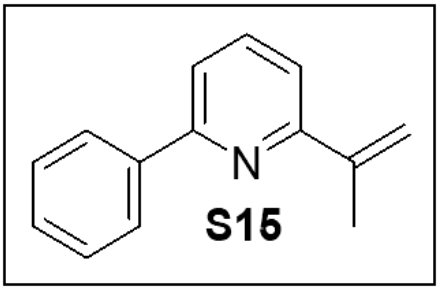
Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B2 MTBE
12.0	0.0	A1 Hexane	B2 MTBE
0.3	0.1	A1 Hexane	B2 MTBE
1.7	1.0	A1 Hexane	B2 MTBE
1.7	2.7	A1 Hexane	B2 MTBE

S87

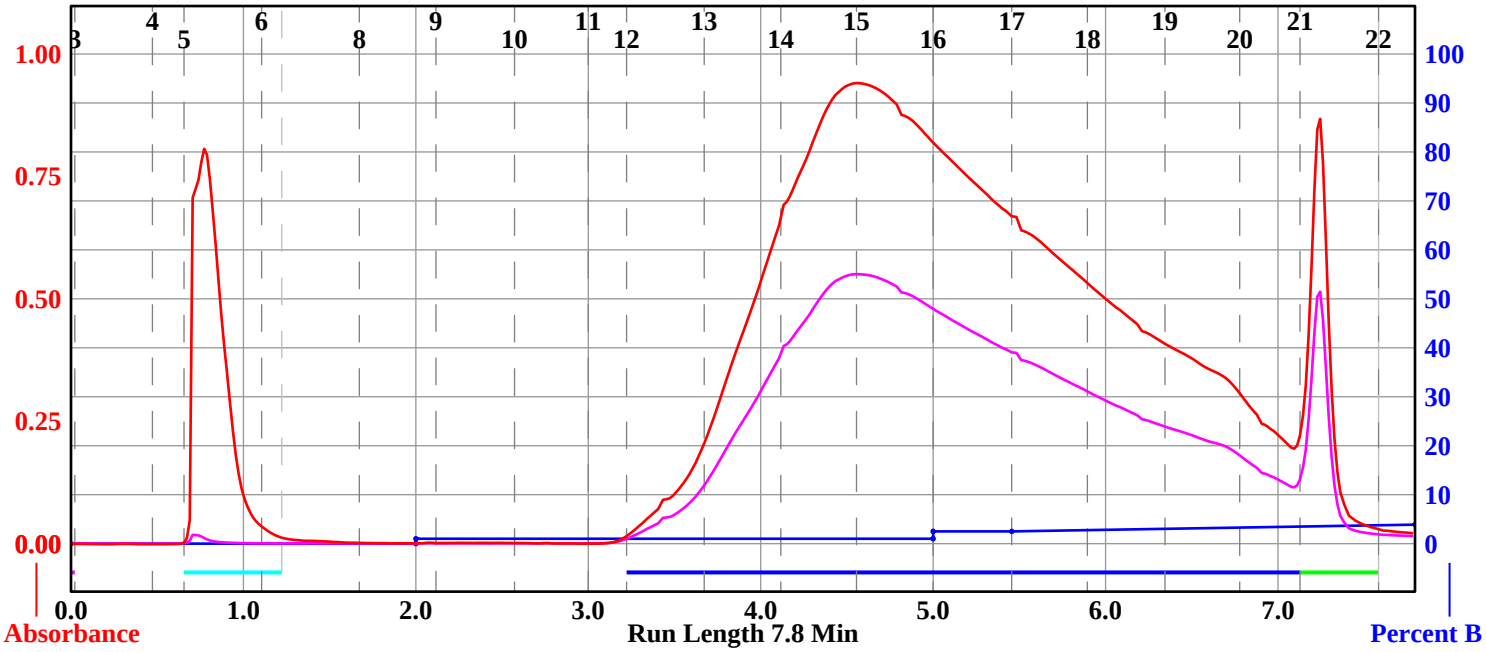
Sample: ccp-a-159 **Rf 200** **Thursday 23 September 2021 09:51AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B2 MTBE

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:5	A:6
3	A:12	A:20
4	A:21	A:22

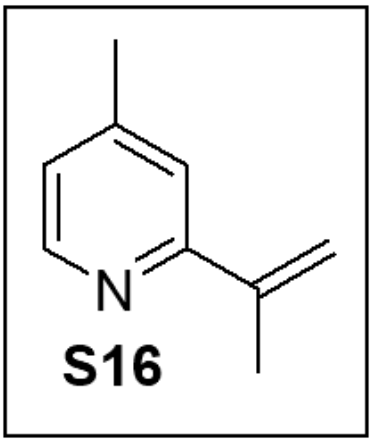
Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B2 MTBE
2.0	0.0	A1 Hexane	B2 MTBE
0.0	1.0	A1 Hexane	B2 MTBE
3.0	1.0	A1 Hexane	B2 MTBE
0.0	2.5	A1 Hexane	B2 MTBE
0.5	2.5	A1 Hexane	B2 MTBE
2.3	3.9	A1 Hexane	B2 MTBE

S88

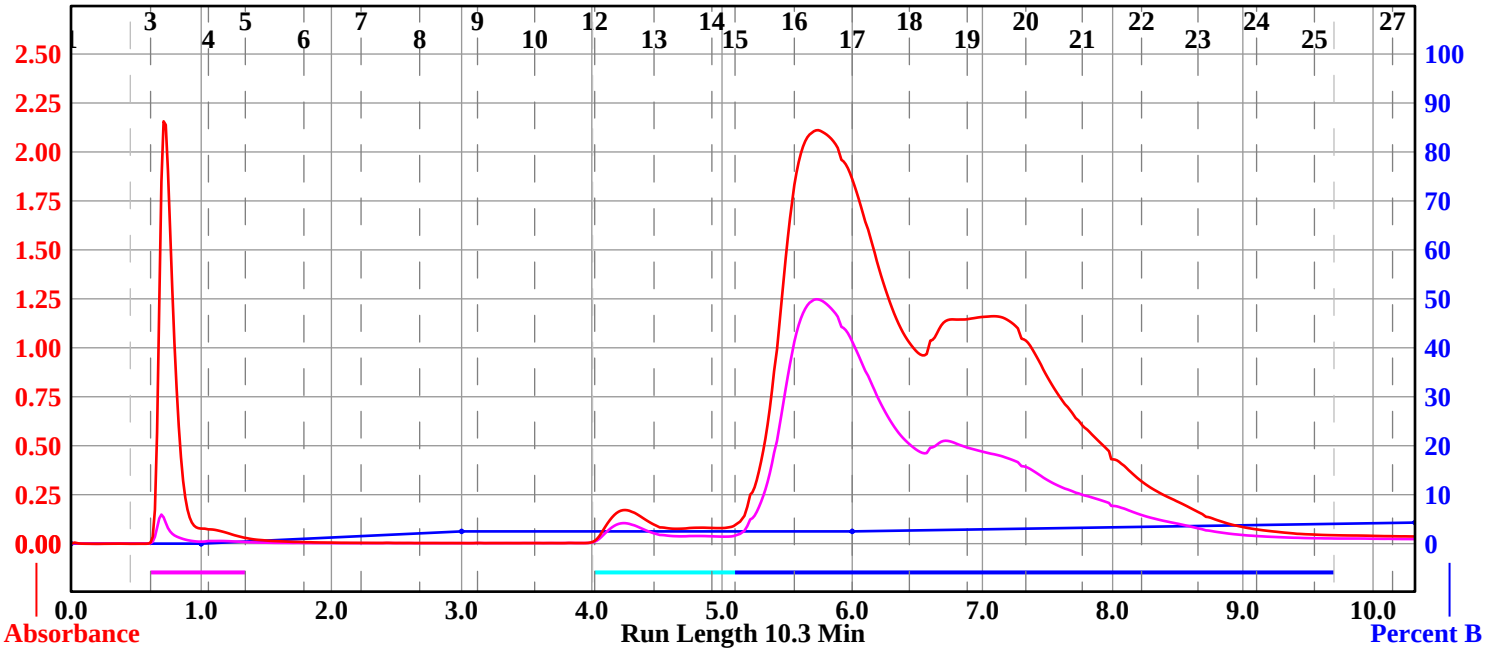
Sample: ccp-a-81 | **Rf 200** | **Thursday 24 June 2021 04:51AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B2 MTBE

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:4
2	A:12	A:14
3	A:15	A:25

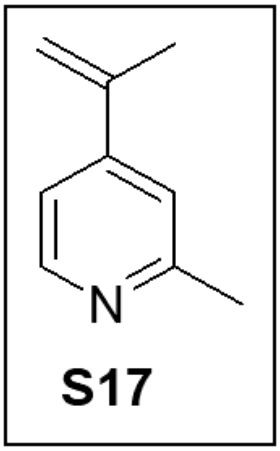
Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B2 MTBE
1.0	0.0	A1 Hexane	B2 MTBE
0.0	0.0	A1 Hexane	B2 MTBE
2.0	2.5	A1 Hexane	B2 MTBE
3.0	2.5	A1 Hexane	B2 MTBE
4.3	4.3	A1 Hexane	B2 MTBE

S89

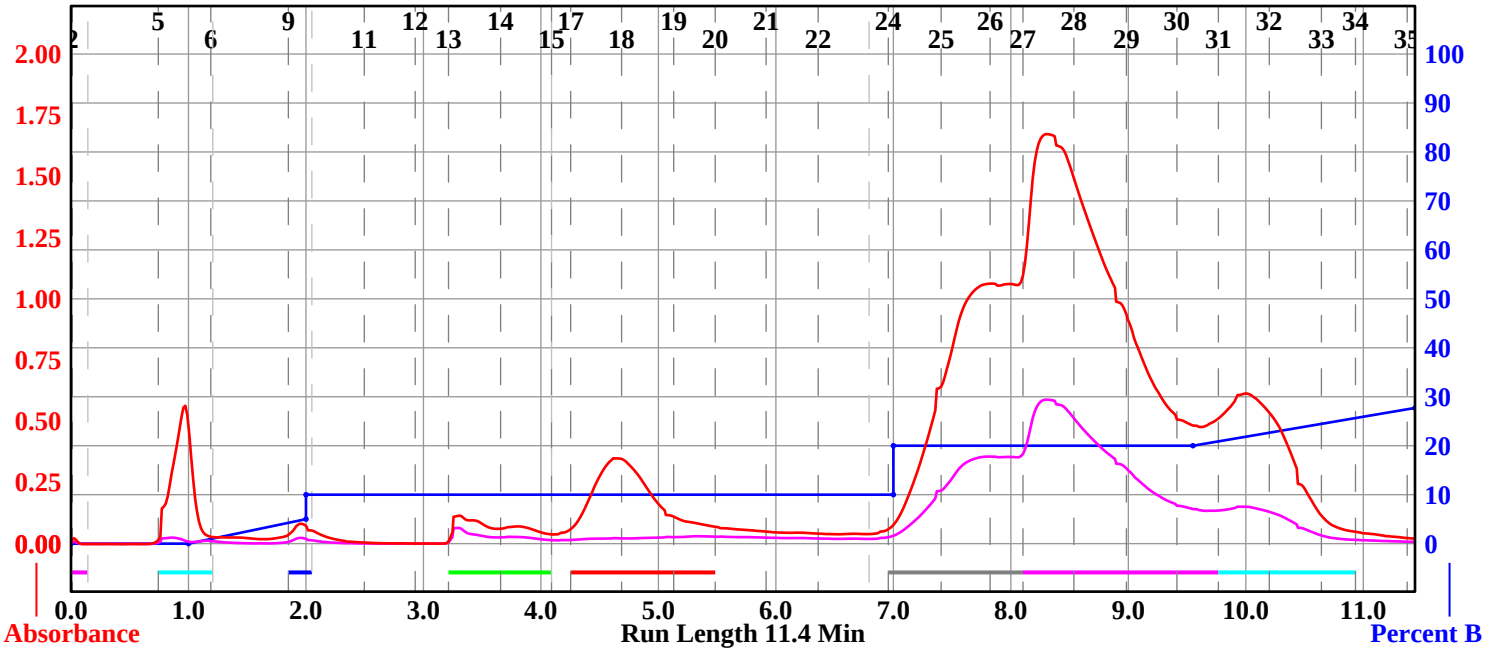
Sample: ccp-a-83 **Rf 200** **Tuesday 22 June 2021 09:27AM**

RediSep Column: Silica 12g
SN: E04150E63D749A Lot: 311537906Z
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B1 Ethyl Acetate

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:5	A:6
3	A:9	A:9
4	A:13	A:15
5	A:17	A:19
6	A:24	A:26
7	A:27	A:30
8	A:31	A:33

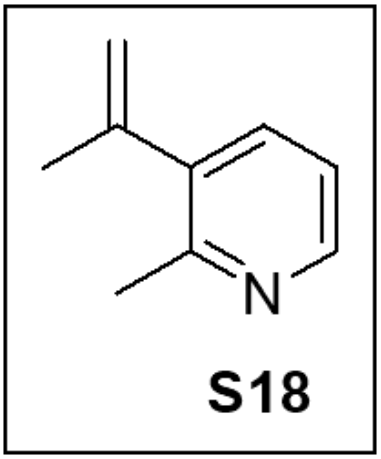
Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B1 Ethyl Acetate
1.0	0.0	A1 Hexane	B1 Ethyl Acetate
1.0	5.0	A1 Hexane	B1 Ethyl Acetate
0.0	10.0	A1 Hexane	B1 Ethyl Acetate
5.0	10.0	A1 Hexane	B1 Ethyl Acetate
0.0	20.0	A1 Hexane	B1 Ethyl Acetate
2.5	20.0	A1 Hexane	B1 Ethyl Acetate
1.9	27.7	A1 Hexane	B1 Ethyl Acetate

S90

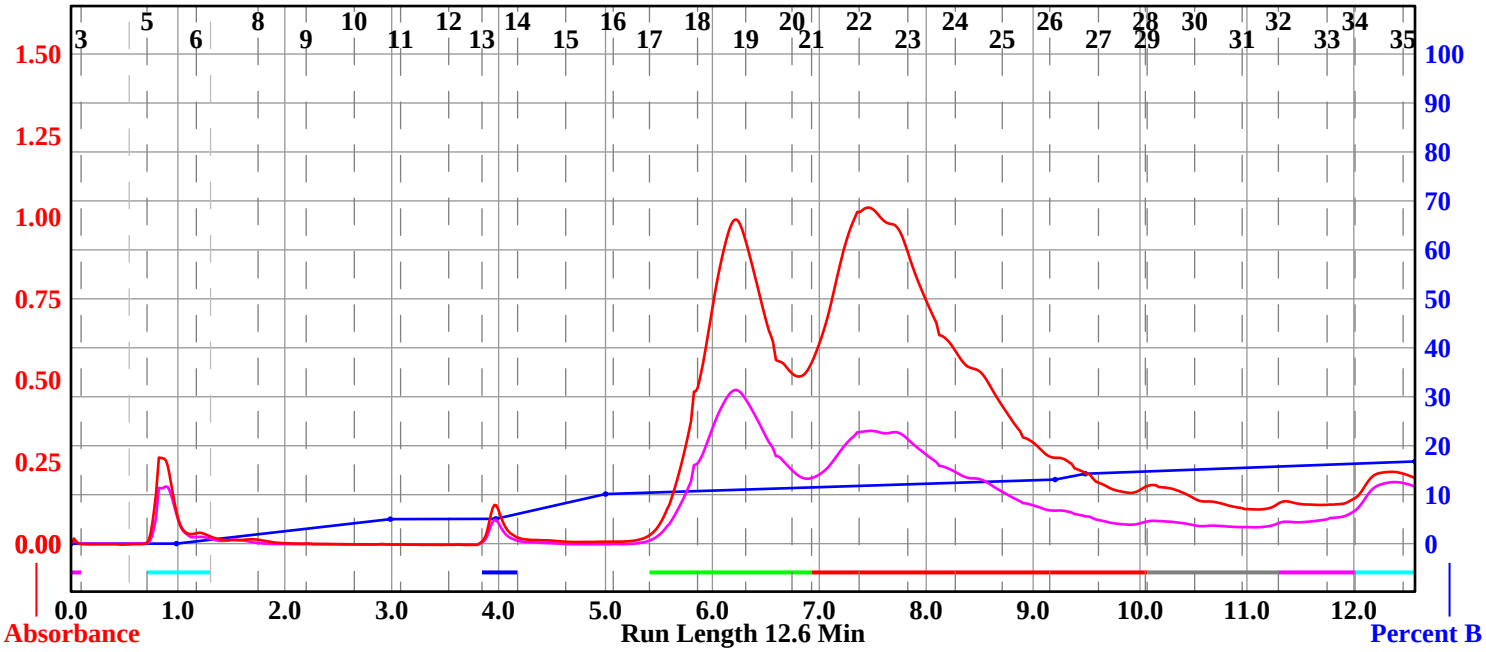
Sample: ccp-a-163 **Rf 200** **Thursday 14 October 2021 09:40AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 pentane
Solvent: B2 MTBE

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:5	A:6
3	A:13	A:13
4	A:17	A:20
5	A:21	A:28
6	A:29	A:31
7	A:32	A:33
8	A:34	A:1

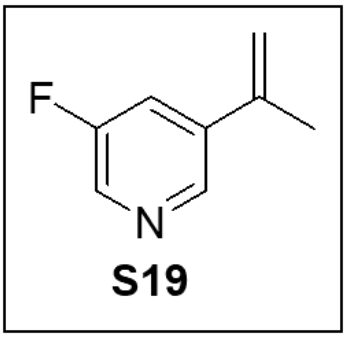
Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 pentane	B2 MTBE
1.0	0.0	A1 pentane	B2 MTBE
2.0	5.0	A1 pentane	B2 MTBE
1.0	5.1	A1 pentane	B2 MTBE
1.0	10.1	A1 pentane	B2 MTBE
4.2	13.1	A1 pentane	B2 MTBE
0.3	14.3	A1 pentane	B2 MTBE
3.1	16.8	A1 pentane	B2 MTBE

S91

Sample: ZZG-I-99 **Rf 200** **Monday 17 May 2021 12:01PM**

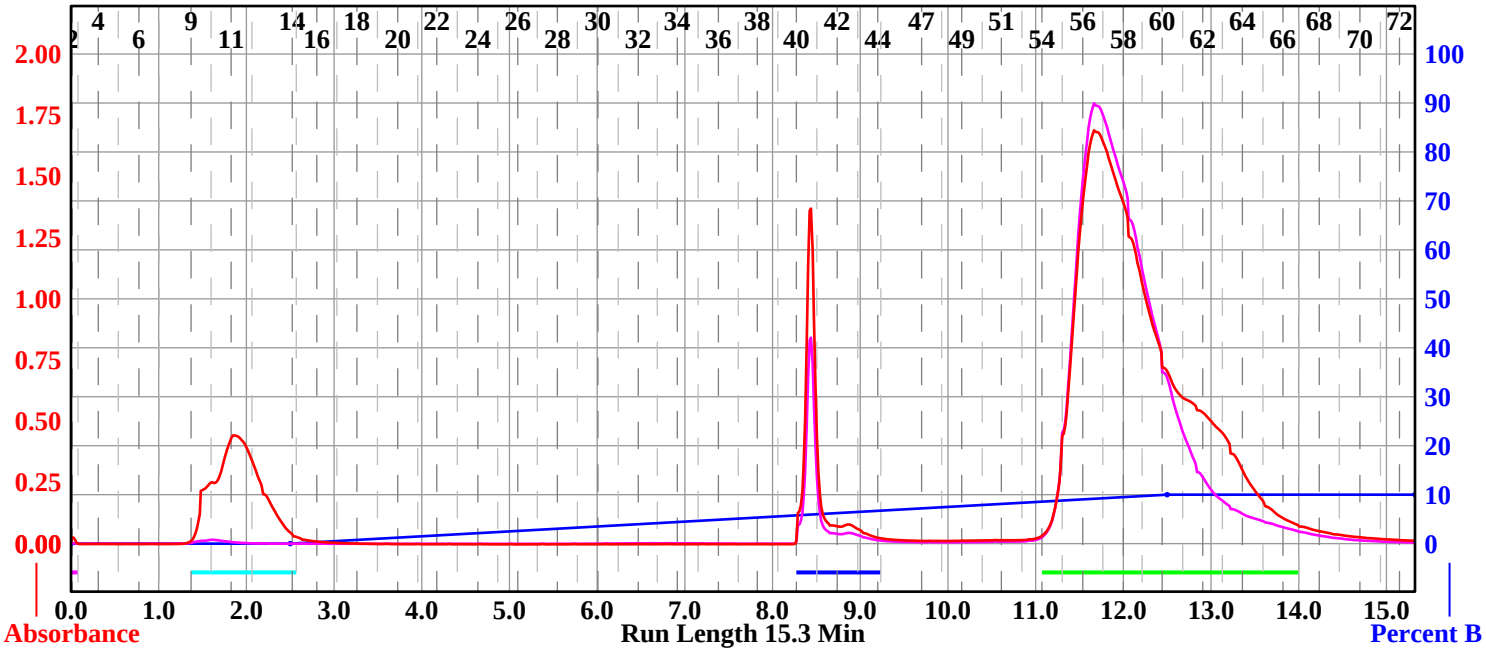
RediSep Column: Silica 24g
SN: E04150D74CBF37 Lot: 301334205X
Flow Rate: 35 ml/min
Equilibration Volume: 168.0 ml
Initial Waste: 0.0 ml
Air Purge: 1.0 min
Solvent: A1 Hexane
Solvent: B1 MTBE

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

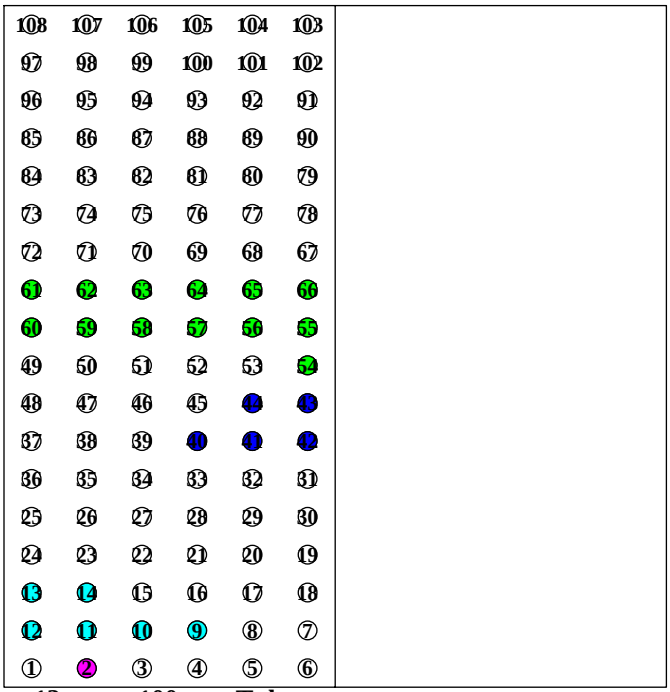


S19

Run Notes:



Rack A



13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:9	A:14
3	A:40	A:44
4	A:54	A:66

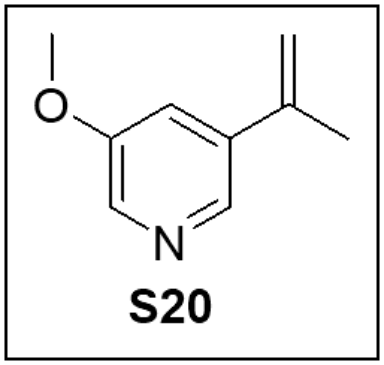
Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B1 MTBE
2.5	0.0	A1 Hexane	B1 MTBE
10.0	10.0	A1 Hexane	B1 MTBE
2.8	10.0	A1 Hexane	B1 MTBE

S92

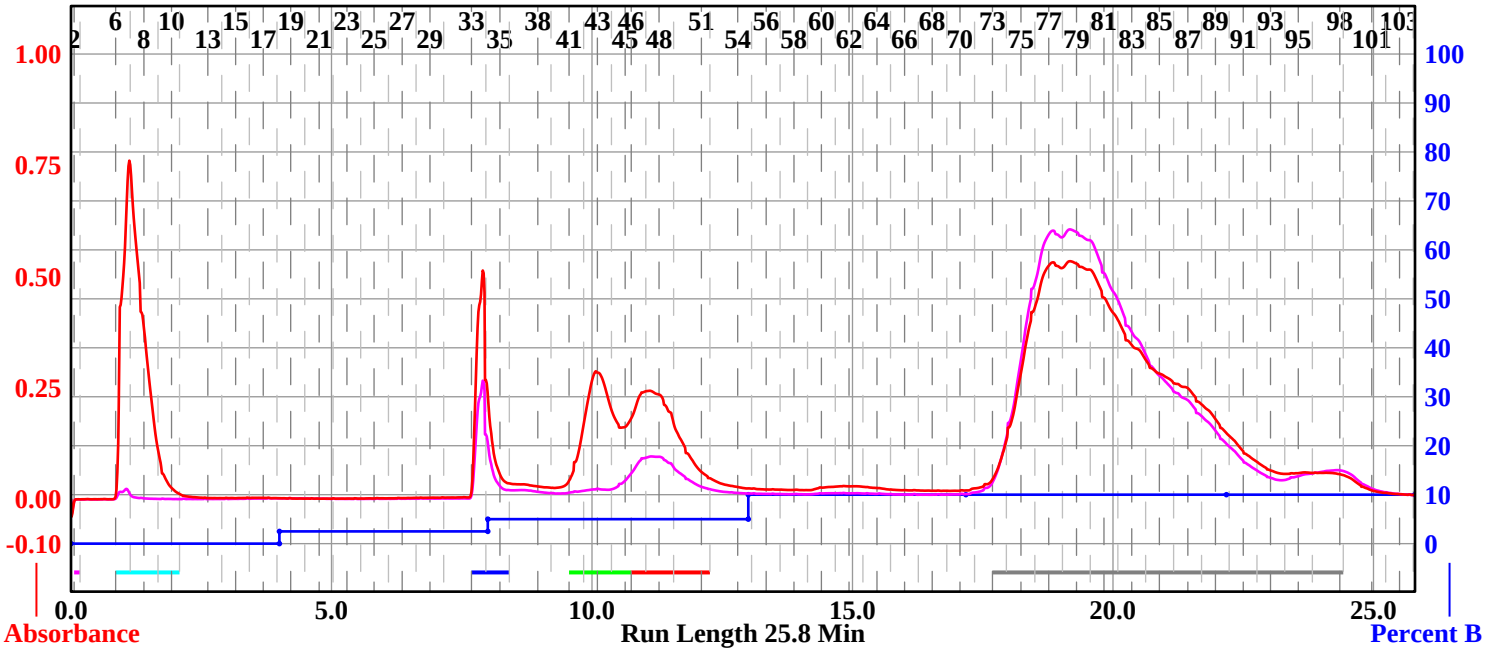
Sample: ccp-a-55 | **Rf 200** | **Thursday 03 June 2021 07:00AM**

RediSep Column: Silica 12g
SN: E04150E8067D5 Lot: 302224502X
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B1 Ethyl Acetate

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

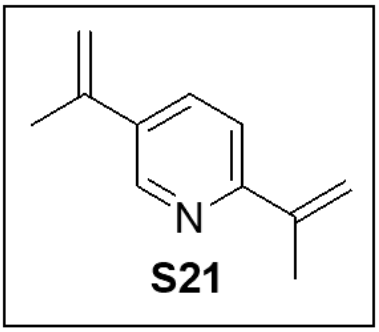
Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:6	A:10
3	A:33	A:35
4	A:41	A:45
5	A:46	A:51
6	A:73	A:98

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B1 Ethyl Acetate
4.0	0.0	A1 Hexane	B1 Ethyl Acetate
0.0	2.5	A1 Hexane	B1 Ethyl Acetate
4.0	2.5	A1 Hexane	B1 Ethyl Acetate
0.0	5.0	A1 Hexane	B1 Ethyl Acetate
5.0	5.0	A1 Hexane	B1 Ethyl Acetate
0.0	10.0	A1 Hexane	B1 Ethyl Acetate
4.2	10.0	A1 Hexane	B1 Ethyl Acetate
5.0	10.0	A1 Hexane	B1 Ethyl Acetate
3.6	10.0	A1 Hexane	B1 Ethyl Acetate

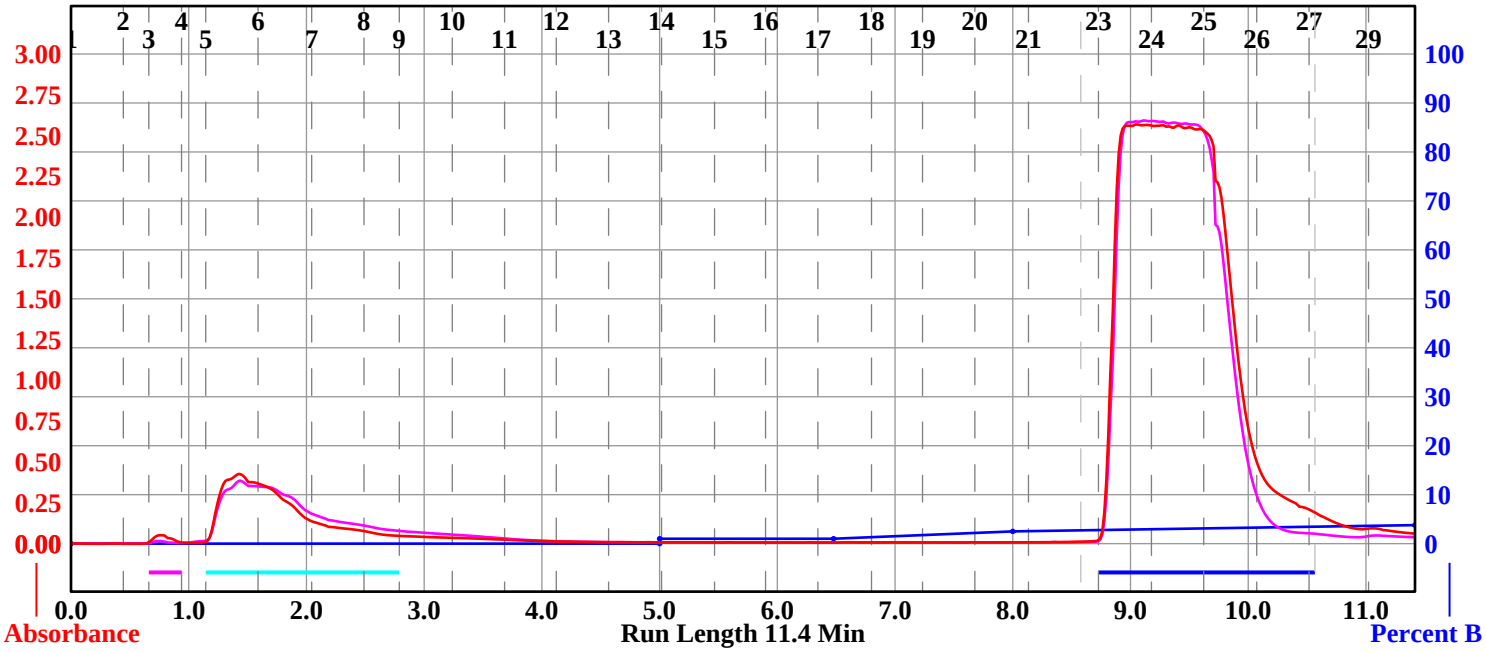
Sample: ccp-a-93 | **Rf 200** | **Wednesday 07 July 2021 05:16AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B2 MTBE

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

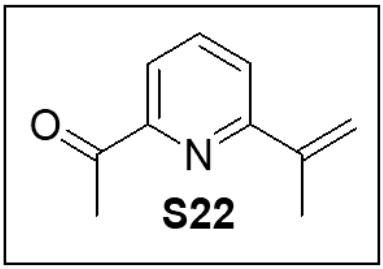
Peak #	Start Tube	End Tube
1	A:3	A:3
2	A:5	A:8
3	A:23	A:27

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B2 MTBE
5.0	0.0	A1 Hexane	B2 MTBE
0.0	1.0	A1 Hexane	B2 MTBE
1.5	1.0	A1 Hexane	B2 MTBE
1.5	2.5	A1 Hexane	B2 MTBE
3.4	3.8	A1 Hexane	B2 MTBE

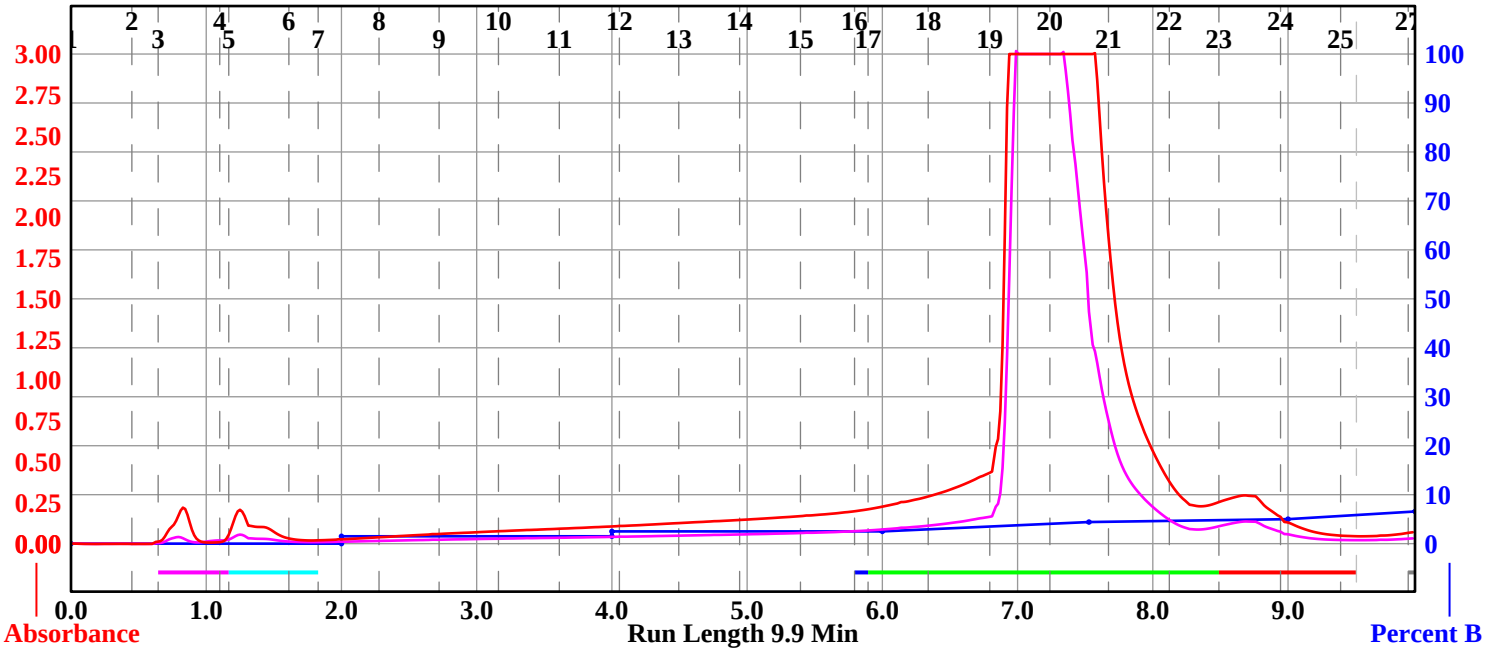
Sample: ccp-a-101 | **Rf 200** | **Wednesday 14 July 2021 05:35AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B2 MTBE

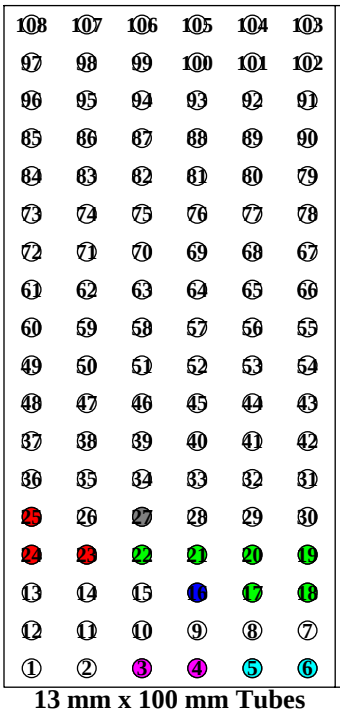
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A



13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:4
2	A:5	A:6
3	A:16	A:16
4	A:17	A:22
5	A:23	A:25
6	A:27	A:27

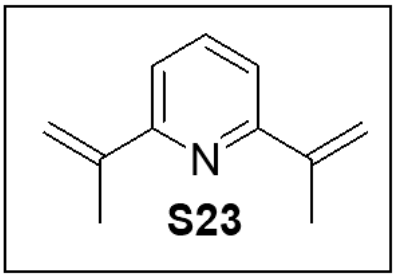
Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B2 MTBE
2.0	0.0	A1 Hexane	B2 MTBE
0.0	1.5	A1 Hexane	B2 MTBE
2.0	1.5	A1 Hexane	B2 MTBE
0.0	2.5	A1 Hexane	B2 MTBE
2.0	2.5	A1 Hexane	B2 MTBE
1.5	4.4	A1 Hexane	B2 MTBE
1.5	5.0	A1 Hexane	B2 MTBE
0.9	6.6	A1 Hexane	B2 MTBE

S95

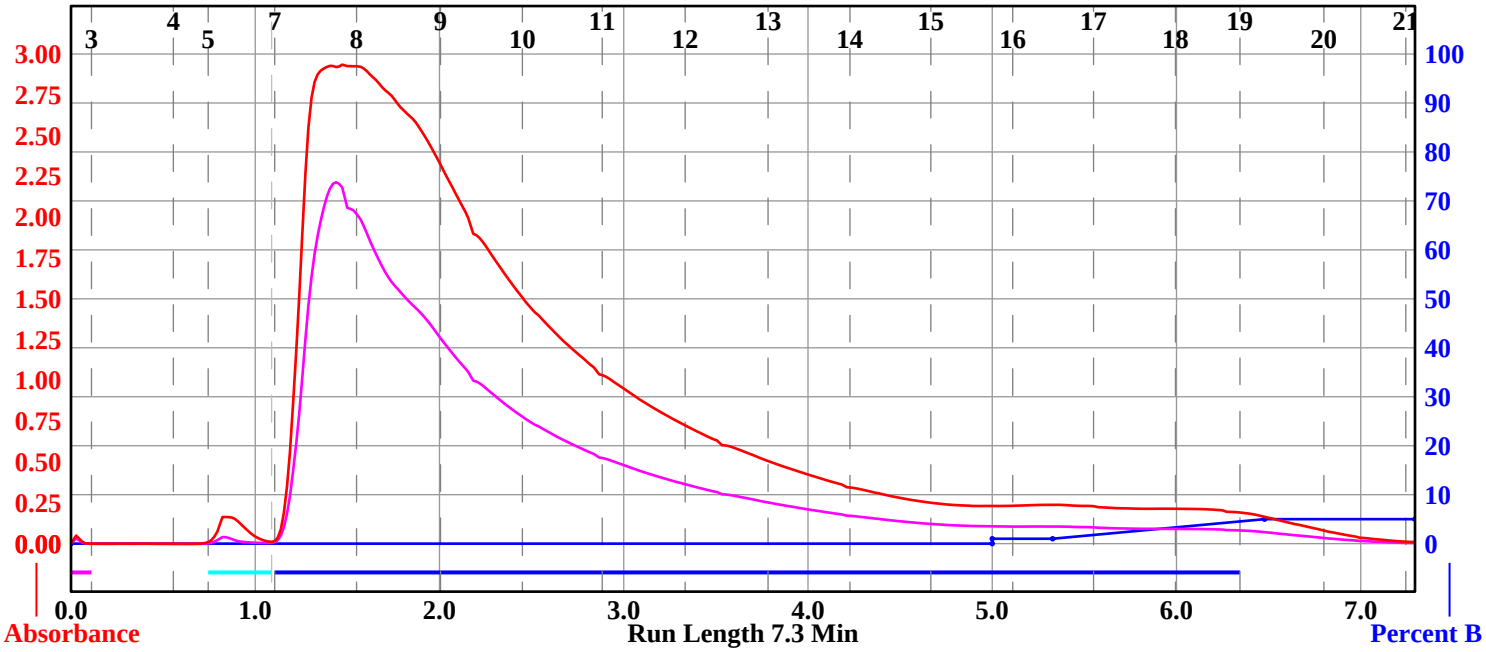
Sample: ccp-a-95 | **Rf 200** | **Wednesday 07 July 2021 10:12AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B2 MTBE

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes: 2,6-Diisopropenyl-pyridine (S24)



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
①	②	③	④	⑤	⑥

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:5	A:5
3	A:7	A:18

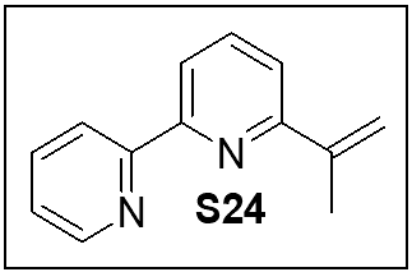
Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B2 MTBE
5.0	0.0	A1 Hexane	B2 MTBE
0.0	1.0	A1 Hexane	B2 MTBE
0.3	1.0	A1 Hexane	B2 MTBE
1.2	5.0	A1 Hexane	B2 MTBE
0.8	5.0	A1 Hexane	B2 MTBE

S96

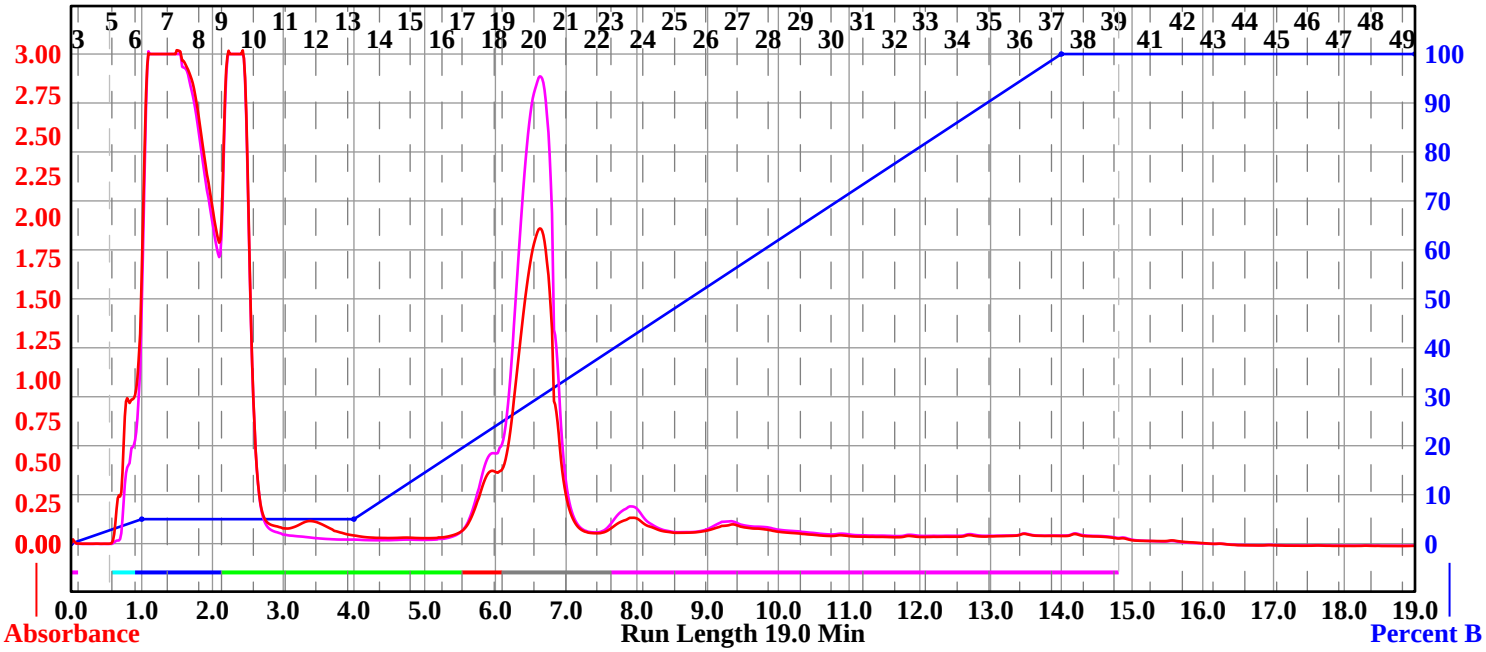
Sample: AMS-A-211 **Rf 200** **Thursday 21 October 2021 02:05PM**

RediSep Column: Al2O3 pH=7 8g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B1 1:1 MTBE:EtOAc

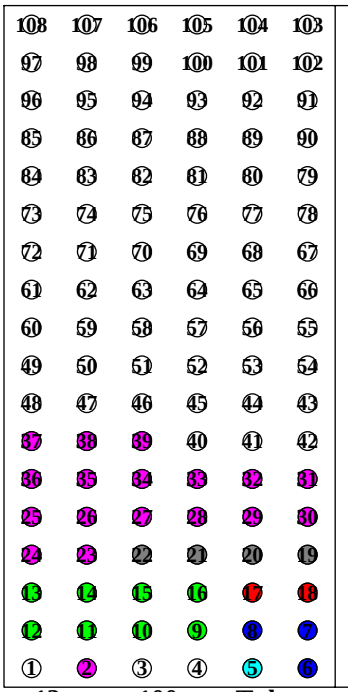
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A



13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:5	A:5
3	A:6	A:8
4	A:9	A:16
5	A:17	A:18
6	A:19	A:22
7	A:23	A:39

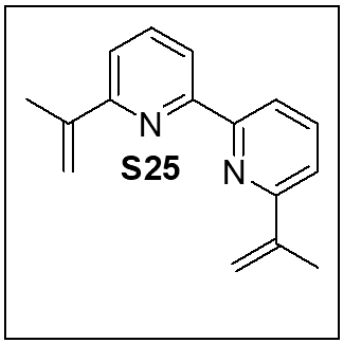
Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B1 1:1 MTBE:EtO
1.0	5.0	A1 Hexane	B1 1:1 MTBE:EtO
3.0	5.0	A1 Hexane	B1 1:1 MTBE:EtO
10.0	100.0	A1 Hexane	B1 1:1 MTBE:EtO
5.0	100.0	A1 Hexane	B1 1:1 MTBE:EtO

S97

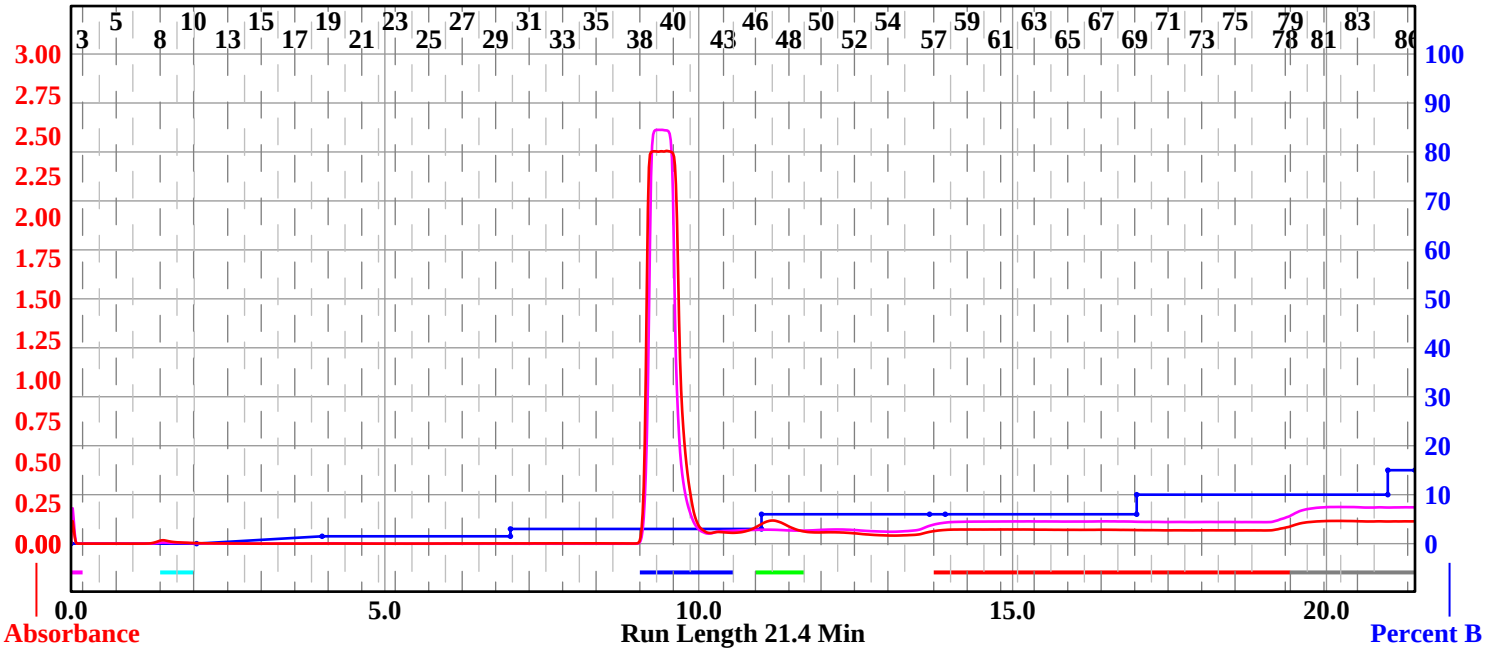
Sample: bis-isoprenylBIPY-ACTUAL **Rf+** **Sunday 04 July 2021 02:33PM**

RediSep Column: Silica 12g
SN: E04150E63D52C Lot: 311537906Z
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent A: Hexane
Solvent B: Acetone

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:8	A:10
3	A:38	A:43
4	A:46	A:48
5	A:57	A:78
6	A:79	A:86

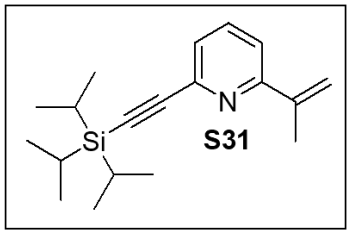
Duration	%B	Solvent A	Solvent B
0.0	0.0	Hexane	Acetone
2.0	0.0	Hexane	Acetone
2.0	1.5	Hexane	Acetone
3.0	1.5	Hexane	Acetone
0.0	3.0	Hexane	Acetone
4.0	3.0	Hexane	Acetone
0.0	6.0	Hexane	Acetone
2.7	6.0	Hexane	Acetone
0.3	6.0	Hexane	Acetone
3.0	6.0	Hexane	Acetone
...	...	...	...

S98

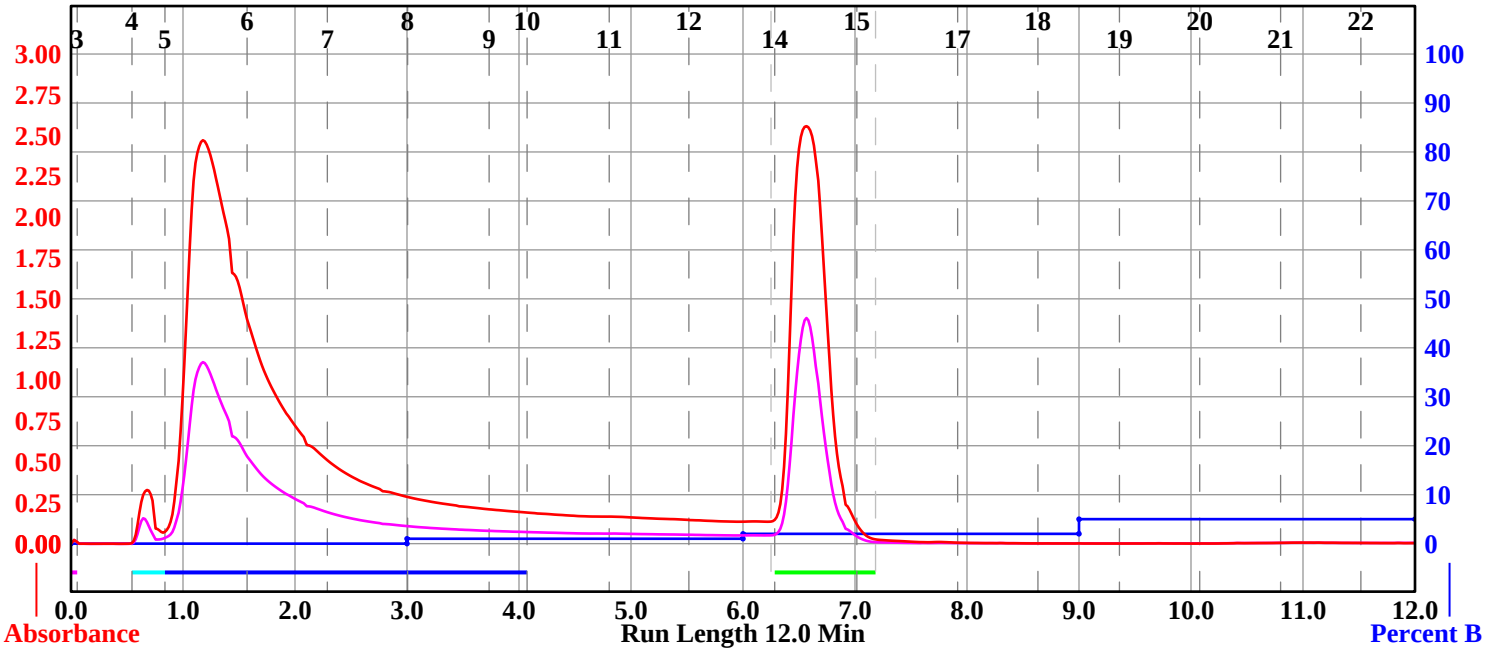
Sample: AMS-A-117 | **Rf 200** | **Friday 23 July 2021 07:01AM**

RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B1 Ethyl Acetate

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:2
2	A:4	A:4
3	A:5	A:9
4	A:14	A:15

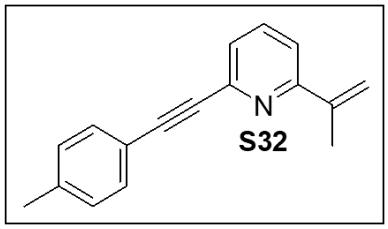
Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B1 Ethyl Acetate
3.0	0.0	A1 Hexane	B1 Ethyl Acetate
0.0	1.0	A1 Hexane	B1 Ethyl Acetate
3.0	1.0	A1 Hexane	B1 Ethyl Acetate
0.0	2.0	A1 Hexane	B1 Ethyl Acetate
3.0	2.0	A1 Hexane	B1 Ethyl Acetate
0.0	5.0	A1 Hexane	B1 Ethyl Acetate
3.0	5.0	A1 Hexane	B1 Ethyl Acetate

S99

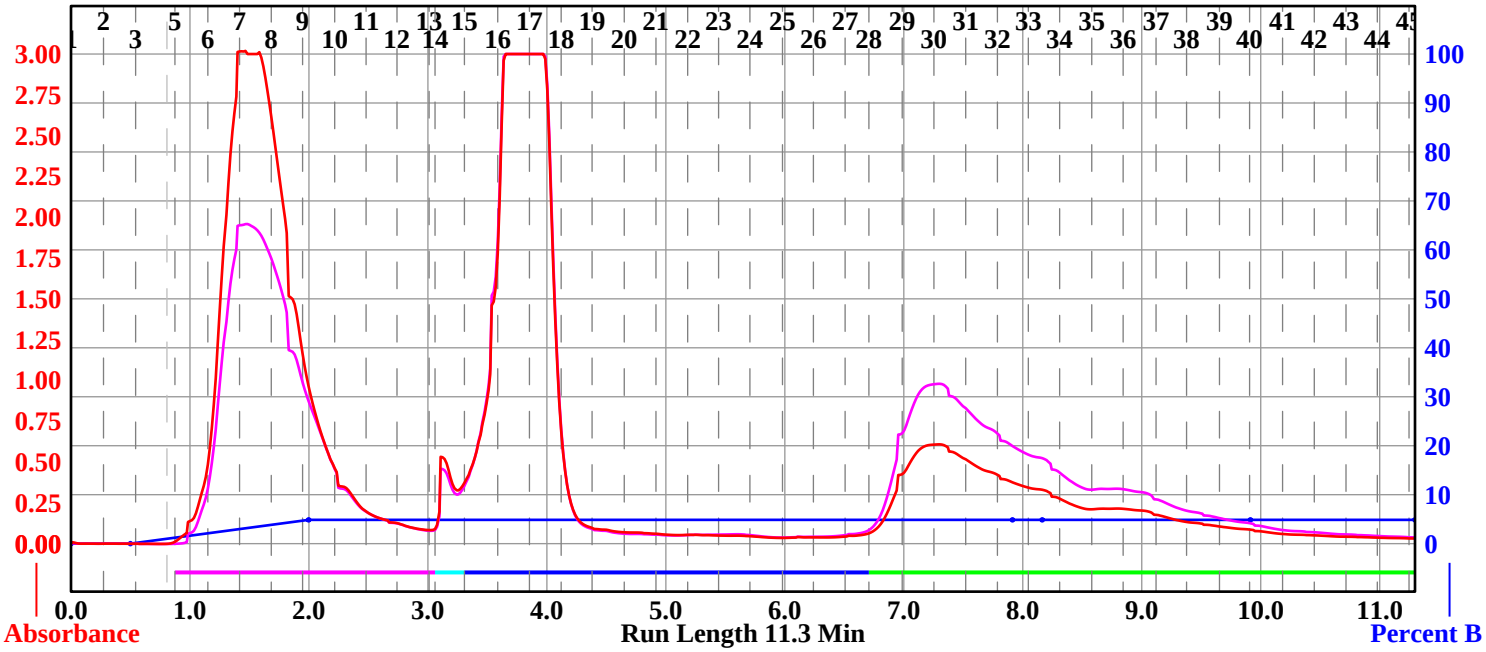
Sample: AMS-B-25 **Rf 200** **Tuesday 29 March 2022 12:27PM**

RediSep Column: Silica 12g Gold
SN: E04150AA31743 Lot: 312220702W
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 0.5 min
Solvent: A1 Hexane
Solvent: B1 Ethyl Acetate

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm



Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
①	②	③	④	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:5	A:13
2	A:14	A:14
3	A:15	A:27
4	A:28	A:45

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 Hexane	B1 Ethyl Acetate
0.5	0.0	A1 Hexane	B1 Ethyl Acetate
1.5	4.9	A1 Hexane	B1 Ethyl Acetate
5.9	4.9	A1 Hexane	B1 Ethyl Acetate
0.2	4.9	A1 Hexane	B1 Ethyl Acetate
1.8	4.9	A1 Hexane	B1 Ethyl Acetate
1.4	4.9	A1 Hexane	B1 Ethyl Acetate

S100