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Supplementary Material

4,4'-(Pyridin-4-ylmethylene)dibenzonitrile

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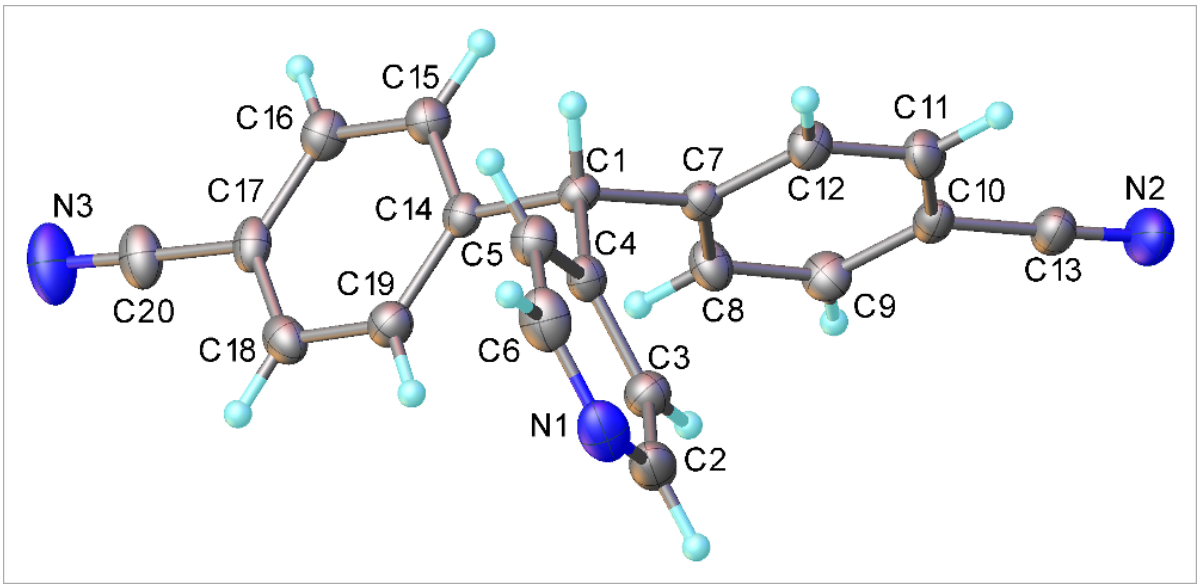
Contents

1. X-Ray Crystallographic Data of 3

X-ray diffraction experiments (Table X) were carried out on a Bruker 3-circle D8 Venture diffractometer with a PHOTON 100 CMOS area detector, using Mo-K_α radiation (λ=0.71073 Å) from Incoatec IμS microsource with focusing mirrors. The crystal was cooled using a Cryostream (Oxford Cryosystems) open-flow N_2 gas cryostat. The data were processed using APEX3 v.2017.3-0 and reflection intensities integrated using SAINT v8.38A software (Bruker AXS, 2017). The structures were solved by dual-space intrinsic phasing method using SHELXT 2018/2 program[1] (**3**) or direct methods using SHELXS 2013/1 program [2] (**4**) and refined by full-matrix least squares using SHELXL 2018/3 software[3] on OLEX2 platform.[4] Full crystallographic data including structure factors has been deposited with Cambridge Crystallographic Data Centre deposition no CCDC-2122916.

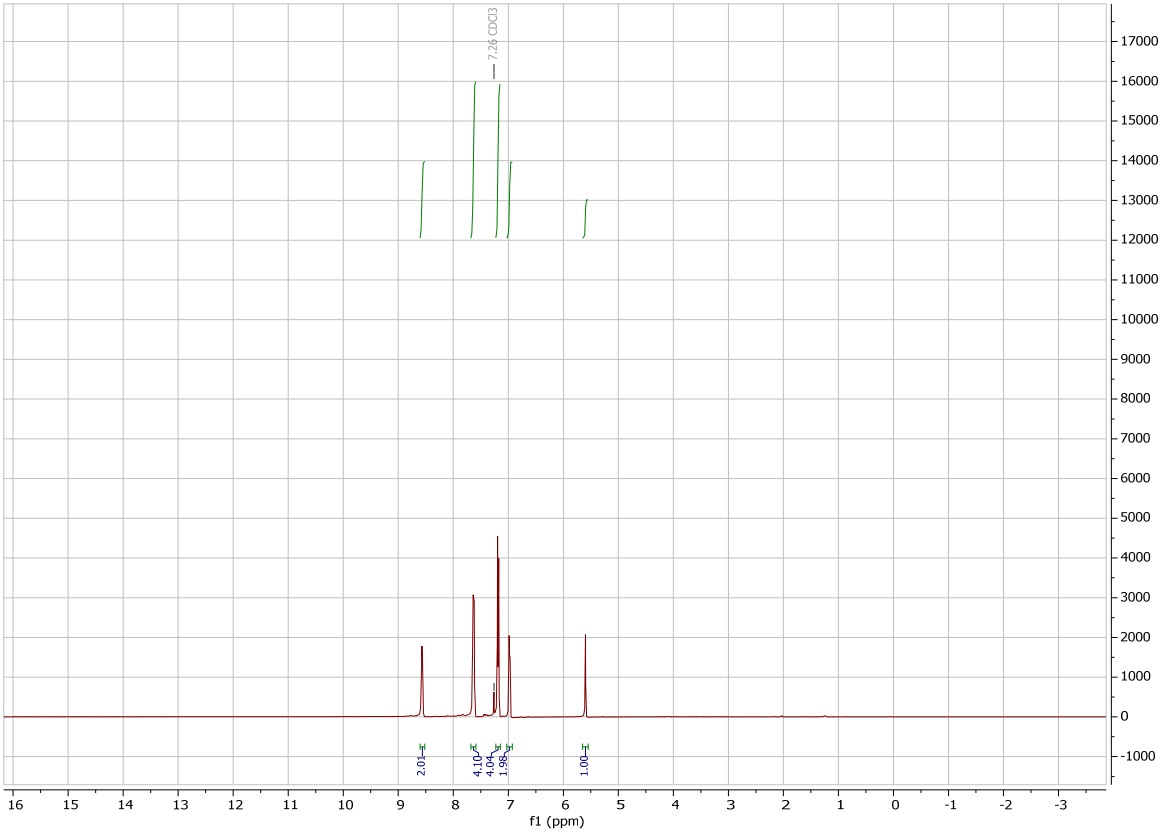


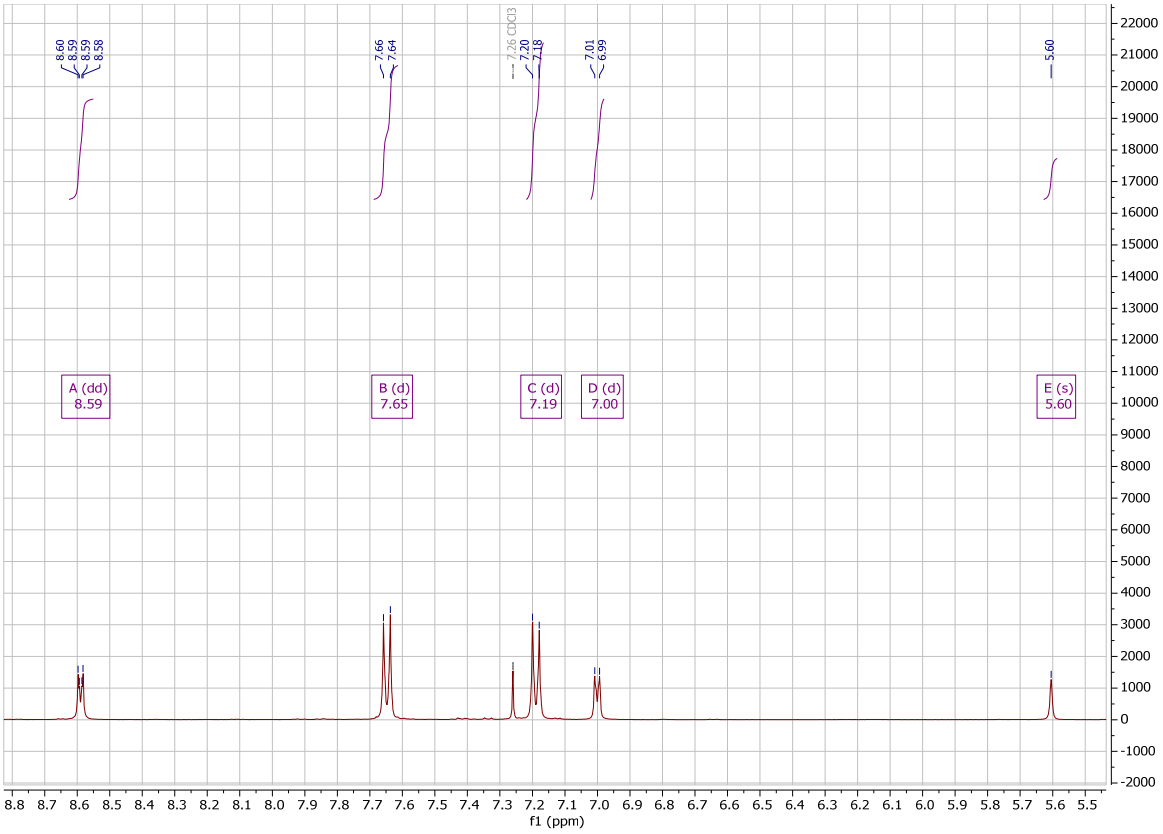
X-ray molecular structure of 3. Atomic displacement ellipsoids are drawn at 50% probability level.

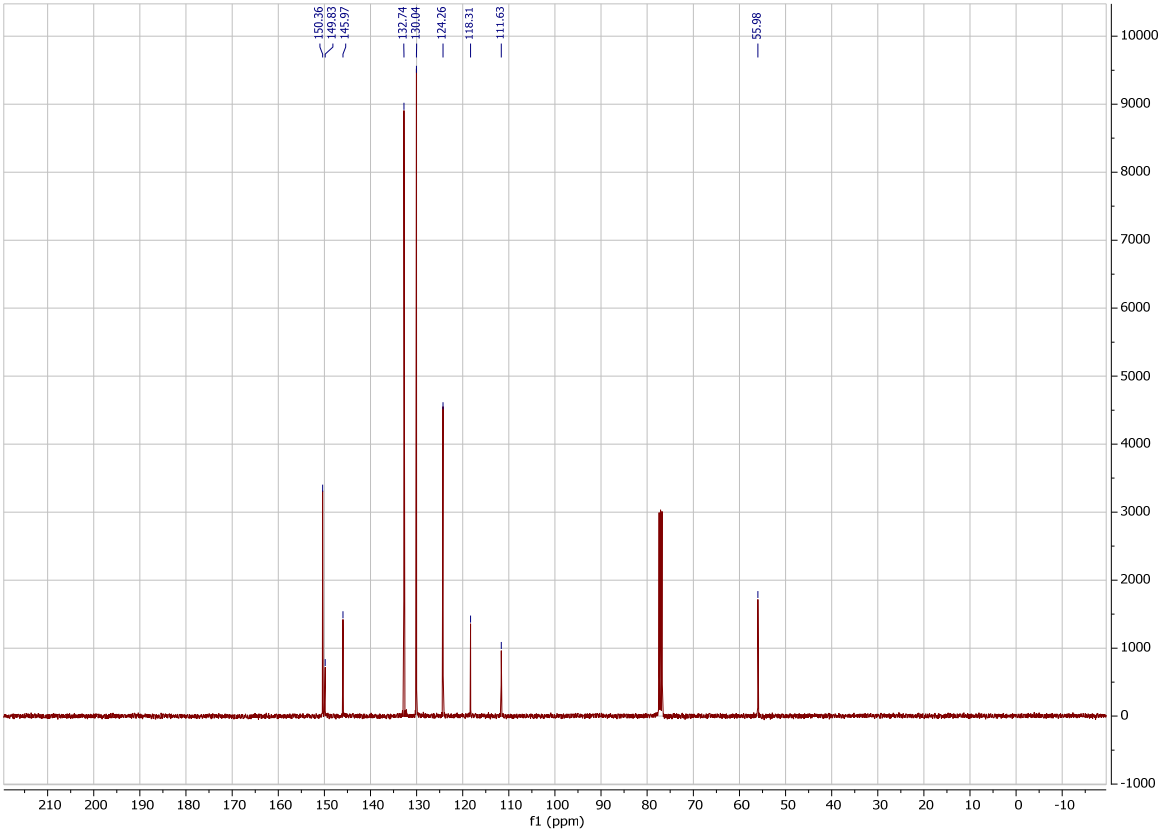
Table 1. Crystal data and structure refinement for CCDC-2122916.

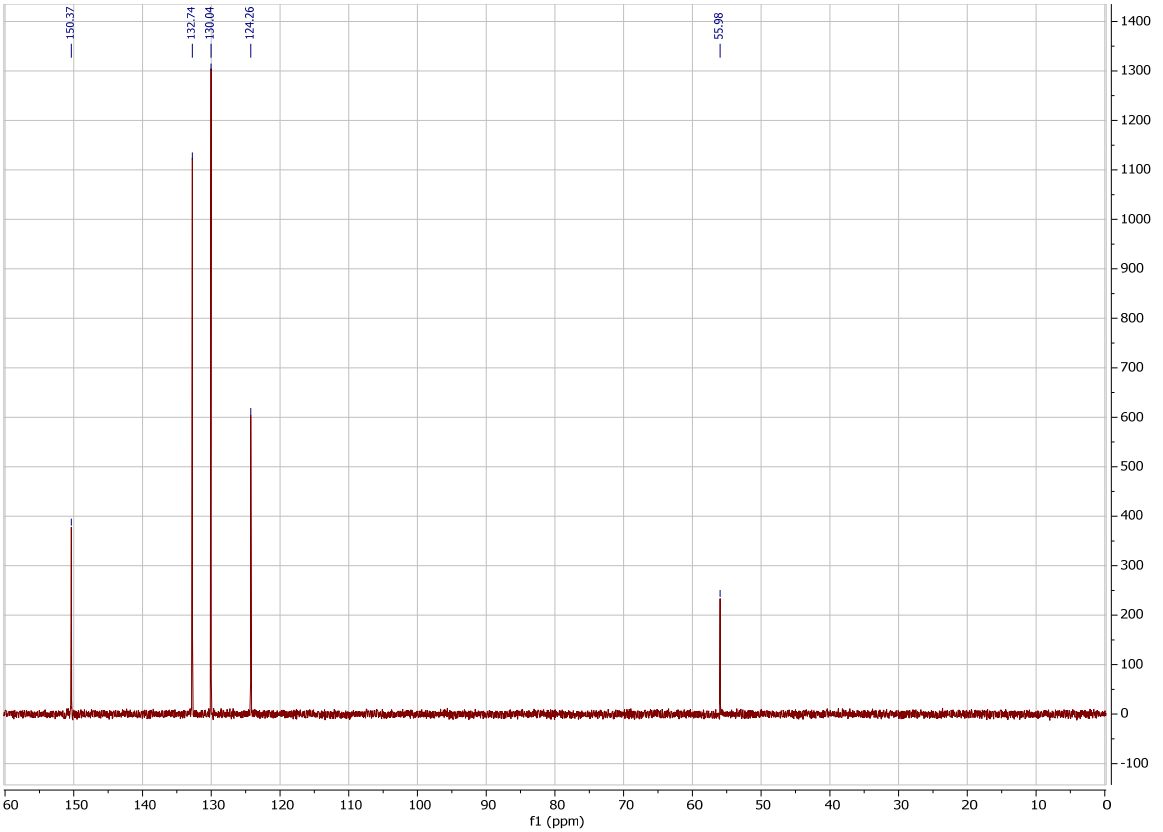
Empirical Formula	**$C_{20}H_{13}N_3$**
Formula weight	295.33
Temperature/K	120
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	9.2835(6)
b/Å	17.5421(11)
c/Å	9.6559(6)
α/°	90
β/°	90.090(3)
γ/°	90
Volume/Å^3	1572.48(17)
Z	4
ϱ_{calc}g/cm^3	1.247
μ/mm^{-1}	0.076
F(000)	616.0
Crystal size/mm^3	0.434 × 0.262 × 0.258
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.644 to 54.996
Index ranges	$-12 \le h \le 12$, $-22 \le k \le 22$, $-12 \le l \le 12$
Reflections collected	29095
Independent reflections	3612 [R_{int} = 0.0387, R_{sigma} = 0.0244]
Data/restraints/parameters	3612/0/261
Goodness-of-fit on F^2	1.029
Final R indexes [I>=2σ (I)]	R_1 = 0.0438, wR_2 = 0.1021
Final R indexes [all data]	R_1 = 0.0601, wR_2 = 0.1112
Largest diff. peak/hole/e Å^{-3}	0.35/−0.21

2. NMR Spectra of 3

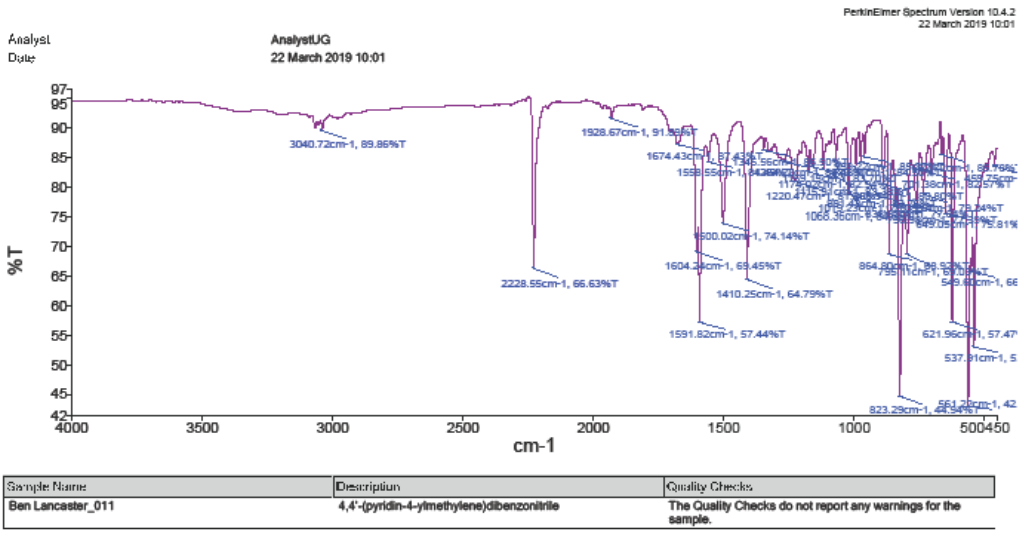

7.26 CDCl3
2.01
4.10
4.04
1.96
1.00
f1 (ppm)


8.60
8.59
8.59
8.58
7.66
7.64
7.26 CDCl3
7.20
7.18
7.01
6.99
5.60
A (dd)
8.59
B (d)
7.65
C (d)
7.19
D (d)
7.00
E (s)
5.60
f1 (ppm)





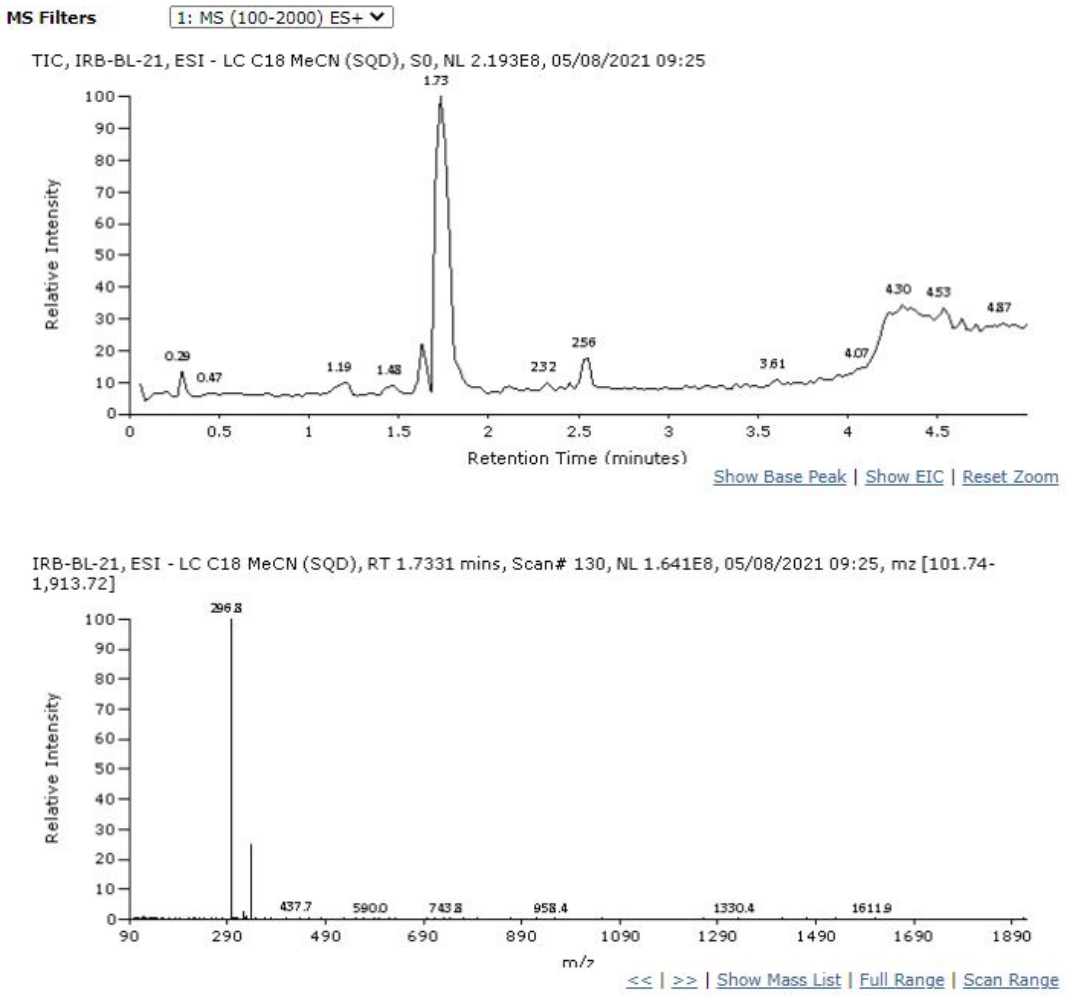
3. IR Spectrum of 3



4. Mass Spectrum of 3

Mass spectra were obtained using a TQD mass spectrometer and an Acquity UPLC (Waters Ltd, UK) for low-resolution ESI+ or ESI-. This instrument is setup for flow injection analysis (FIA) or ultra-performance liquid chromatography (UPLC); column dimensions Acquity UPLC BEH C18 1.7µm (2.1mm x 50mm). Accurate mass measurements were obtained using a QtoF Premier mass spectrometer with an Acquity UPLC (Waters Ltd, UK).

5. Low resolution LC-MS.


MS Filters
1: MS (100-2000) ES+
TIC, IRB-BL-21, ESI - LC C18 MeCN (SQD), S0, NL 2.193E8, 05/08/2021 09:25
Relative Intensity
Retention Time (minutes)
0.29
0.47
1.19
1.48
1.73
2.32
2.56
3.61
4.07
4.30
4.53
4.87
Show Base Peak | Show EIC | Reset Zoom
IRB-BL-21, ESI - LC C18 MeCN (SQD), RT 1.7331 mins, Scan# 130, NL 1.641E8, 05/08/2021 09:25, mz [101.74-1,913.72]
Relative Intensity
m/z
296.8
437.7
590.0
743.8
958.4
1330.4
1611.9
<< | >> | Show Mass List | Full Range | Scan Range

Elemental Composition Report **Page 1**

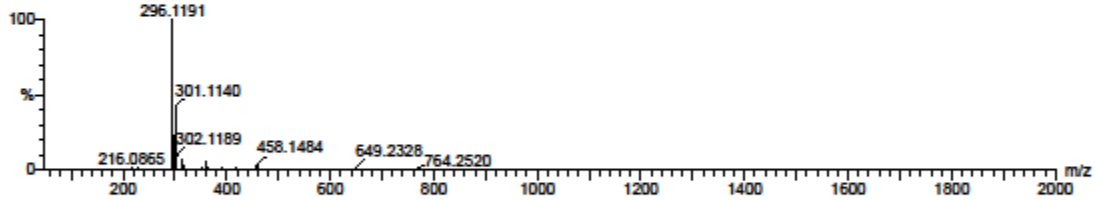
Single Mass Analysis
Tolerance = 3.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
1033 formula(e) evaluated with 7 results within limits (up to 500 closest results for each mass)
Elements Used:
C: 0-35 H: 0-60 N: 0-6 O: 0-10 F: 0-4

QToF Premier

22-Mar-2019
BL-21 354 (2.988) Cm (352:367)

1: TOF MS ES+
1.94e+005



Minimum:				-1.5			
Maximum:		3.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
296.1191	296.1188	0.3	1.0	15.5	1062.9	0.1	C20 H14 N3
	296.1199	-0.8	-2.7	11.5	1065.4	2.5	C17 H15 N3 O F
	296.1182	0.9	3.0	-0.5	1075.2	12.3	C6 H17 N5 O5 F3
	296.1206	-1.5	-5.1	2.5	1073.6	10.7	C8 H18 N5 O7
	296.1211	-2.0	-6.8	7.5	1068.8	6.0	C14 H16 N3 O2 F2
	296.1170	2.1	7.1	3.5	1073.4	10.6	C9 H16 N5 O4 F2
	296.1218	-2.7	-9.1	-1.5	1075.3	12.5	C5 H19 N5 O8 F

1-(4-Fluorophenyl)-2-(4-pyridinyl)ethenone [5–7] (4)

$C_{13}H_{10}FNO$, M_r = 215.23 g mol^{-1}, yellow crystals. δ_H (400 MHz, $CDCl_3$) 8.63–8.55 (2H, m), 8.08–8.00 (2H, m), 7.25–7.12 (4H, m), 4.28 (2H, s, CH_2). δ_C (101 MHz, $CDCl_3$) 194.3 (C=O), 166.0 (C, d, *J* 256.0, CF), 150.0 (2CH), 143.21 (C), 132.6 (C, d, *J* 3.1), 131.2 (2CH, d, *J* 9.4), 124.9 (2CH), 116.0 (2CH, d, *J* 22.0), 44.5 (CH_2). δ_F (376 MHz, $CDCl_3$) -104.0 (s). IR (neat) ν = 2923.5 (m), 1684.7 (s, C=O), 1593.2 (s), 1505.7 (m), 1415.6 (m), 1159.3 (s), 999.3 (m), 834.3 (s), 767.0 (s), 556.1 (s) cm^{-1}. GC-MS: R_t = 5.32 min, *m*/*z* 215.1 [M$^+$]. Melting point: 87.5–89.9 °C (EtOAc). R_f 0.31 (20% hexanes / 80% EtOAc).

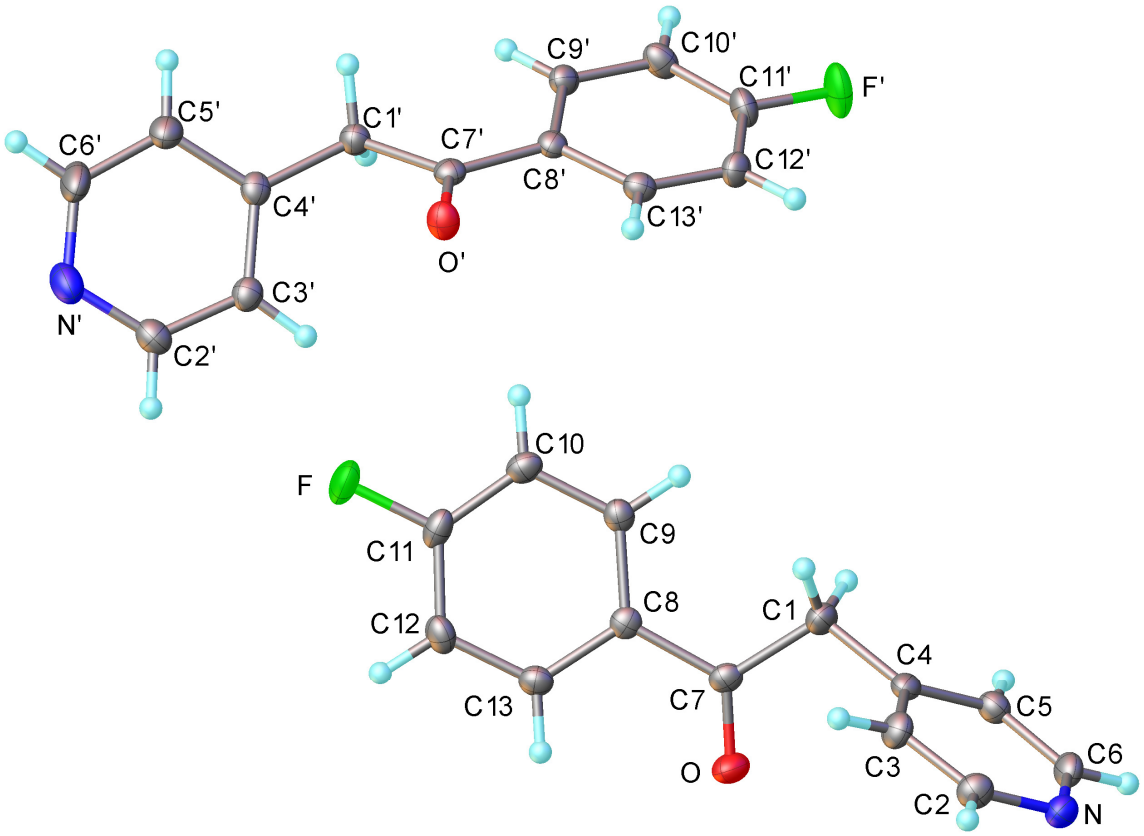


Table 2. Crystal data and structure refinement for CCDC-2124377.

Empirical Formula	**$C_{13}H_{10}FNO$**
Formula weight	215.22
Temperature/K	120
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	11.317(4)
b/Å	7.374(3)
c/Å	24.447(9)
α/°	90
β/°	97.012(13)
γ/°	90
Volume/Å^3	2024.8(13)
Z	8
ϱ_{calc}g/cm^3	1.412
μ/mm^{-1}	0.102
F(000)	896.0
Crystal size/mm^3	0.702 × 0.272 × 0.124
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.632 to 49.998
Index ranges	-13 ≤ h ≤ 13, -8 ≤ k ≤ 8, -29 ≤ l ≤ 28
Reflections collected	23163
Independent reflections	3571 [R_{int} = 0.0493, R_{sigma} = 0.0333]
Data/restraints/parameters	3571/0/290
Goodness-of-fit on F^2	1.025
Final R indexes [I>=2σ (I)]	R_1 = 0.0338, wR_2 = 0.0808
Final R indexes [all data]	R_1 = 0.0444, wR_2 = 0.0858

Largest diff. peak/hole / e Å^{-3}	0.22/-0.18

X-ray molecular structure of 4. Atomic displacement ellipsoids are drawn at 50% probability level.

References

1. Sheldrick, G.M. *Acta Crystallogr. A*, **2015**, *71*, 3–8.
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4. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J., Howard, J.A.K., Puschmann, H. *J. Appl. Crystallogr*. **2009**, *42*, 339–341.
5. Venturoni, F., Nikbin, N., Ley, S.V., Baxendale, I.R. *Org. Biomol. Chem*., **2010**, *8*, 1798–1806.
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