

Electronic Supplementary Information (ESI)

First-in-class hydrazone-pyrazoline sensors for selective detection of
 Zn^{2+} , Cd^{2+} , and Hg^{2+} in aqueous environments

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General Experimental

Chemicals, solvents and reagents were purchased from commercial sources and used without further purification. PE refers to petroleum ether, bp 40-60 °C. Spectroscopy was performed with CHROMASOLV® gradient grade acetonitrile for HPLC, ≥99.9%, from Sigma-Aldrich.

The metal complexes used in this study were: LiCl, NaCl, KCl, CaCl₂, MgCl₂, CuCl₂, NiCl₂, ZnCl₂, CdCl₂, RuCl₃, CoCl₂, MnCl₂, PbCl₂, ZnCl₂ and HgI₂.

TLCs were carried out on Merck Aluminium backed TLC plates Silica Gel 60 F254 and viewed using UV light of wavelength 254 nm. Merck Silica Gel (0.040-0.063 mm) was used for column chromatography. Compounds were loaded as an oil, CH₂Cl₂ solution or dry loaded by adsorption onto silica.

NMR spectra were obtained on a Bruker Avance III (400 MHz) spectrometer and processed via TopSpin® software. The chemical shifts are recorded in parts per million (ppm) with reference to tetramethylsilane. The coupling constants J are quoted to the nearest 0.5 Hz and are not corrected.

High resolution Mass spectroscopy was performed on Bruker Quadrupole Time-of-Flight (qToF) mass spectrometer.

UV/Vis spectroscopy was performed on an Agilent Cary5000 in quartz cuvettes with a 1 cm pathlength using HPLC grade MeCN, 250-500 nm range with 0.2 sec dwell time. Detector switchover occurred at 350 nm.

FTIR spectroscopy was performed on a Bruker VERTEX 70 spectrometer.

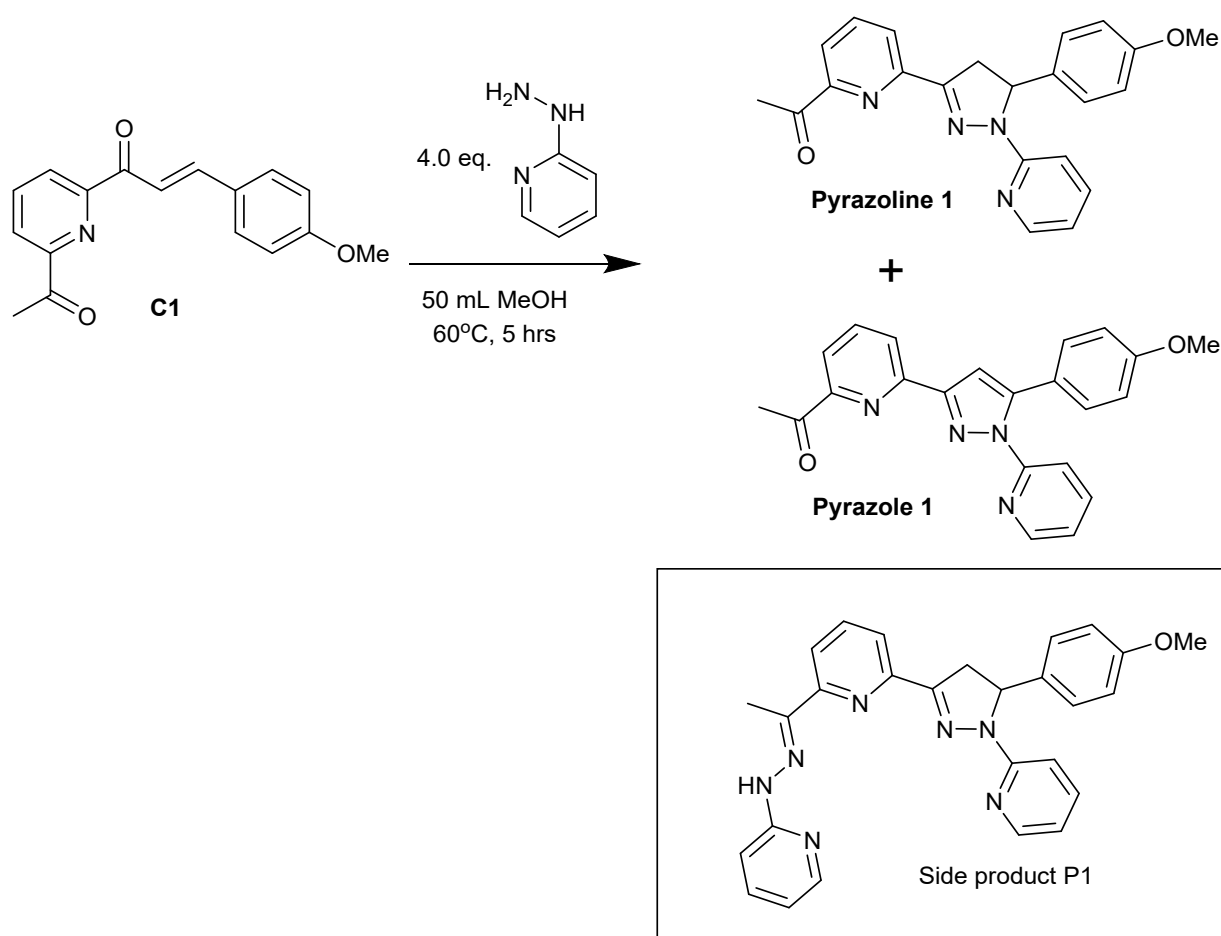
Fluorescence spectroscopy was performed on an Edinburgh Instruments FLS1000 with a xenon excitation source, 2 nm bandwidths for both excitation and emission monochromator, scan speed of 1 nm and dwell time of 0.2 sec. Fluorescence quartz cuvettes with a 1 cm pathlength were used throughout with HPLC grade MeCN.

A 100 Watt 365 nm Analytikjena High intensity UV lamp was used to image the sensors in cuvettes with 5.0 equivalent indicated metal, sensor concentration was 20 μM, solvent was MeCN.

All figures were plotted using SigmaPlot® 14.5 software.

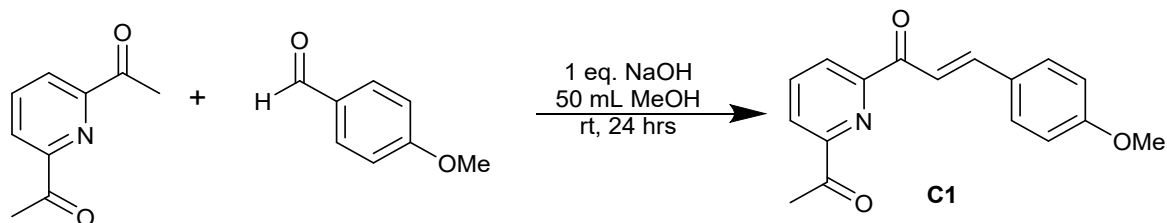
Preliminary Pyrazoline synthesis Screening (S1)

In our search for new pyrazoline and pyrazole fluorescent sensors, we synthesised pyrazoline 1 and pyrazole 2 below and discovered an unexpected 3rd product, the hydrazone pyrazoline (**P1**) which was isolated and discovered to have useful properties. While pyrazoline 1 and pyrazole did display “turn on” fluorescent properties, they are not the subject of this study and therefore will not be discussed further.



General Synthesis (S2)

Synthesis of chalcone **C1**



Using a method adapted from a previous synthesis (*RSC Adv.*, 2024, **14**, 3519-3524), 6 mmol 2,6-acetylpyridine was added to a stirred solution of 3.0 mmol aldehyde in MeOH followed by the addition of 3.0 mmol NaOH and stirring continued at room temperature. After 24 hours the precipitate was filtered, washed with copious amounts of cold H₂O and collected and dried to afford the desired chalcone without further purification.

Yield 0.442g (52%);

V_{max} (Solid)/cm⁻¹ 1701, 1666, 1216 and 952;

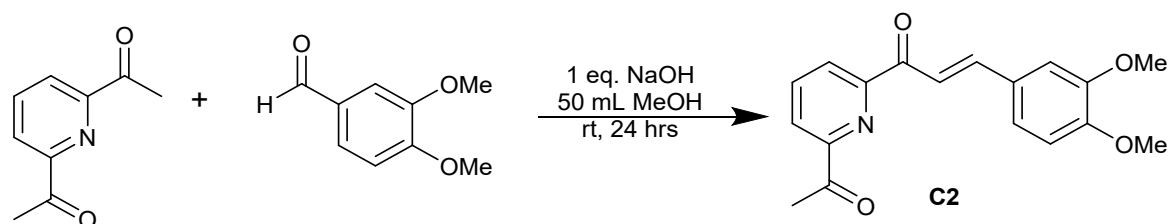
¹H NMR δ_H (400 MHz; CDCl₃) 2.90 (3 H, s, CH₃), 3.90 (3 H, s, OCH₃), 6.98-7.00 (2 H, m, CH), 7.70-7.72 (2 H, m, CH), 7.98-8.06 (3 H, m, CH=CH and CH), 8.24-8.28 (2 H, m, CH=CH and CH) and 8.38-8.40 (1 H, m, CH);

¹³C NMR δ_C (100 MHz; CDCl₃) 25.8, 55.5, 114.5, 118.0, 124.4, 125.6, 126.1, 127.8, 130.5, 138.1, 145.1, 152.6, 161.9, 188.5 and 199.5;

HRMS m/z (qToF) Found 282.1152 (M+H⁺). C₁₇H₁₆NO₃ requires 282.1130.

Above in agreement with previous data from *RSC Adv.*, 2024, **14**, 3519-3524.

Synthesis of chalcone **C2**



Using a method adapted from a previous synthesis (*RSC Adv.*, 2024, **14**, 3519-3524), 10 mmol 2,6-acetylpyridine was added to a stirred solution of 5.0 mmol aldehyde in MeOH followed by the addition of 5.0 mmol NaOH and stirring continued at room temperature. After 24 hours the precipitate was filtered, washed with copious amounts of cold H₂O and collected and dried to afford the desired chalcone without further purification.

Yield 0.65g (41%);

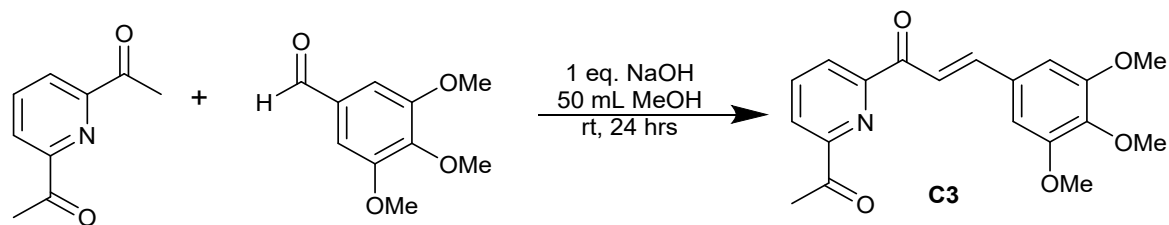
V_{max} (Solid)/cm⁻¹ 1593, 1512, 1429, 1162, 1072 and 991;

¹H NMR δ_H (400 MHz; CDCl₃) 2.88 (3 H, s, CH₃), 3.96 (3 H, s, CH₃), 4.00 (3 H, s, CH₃), 6.91-6.97 (1 H, m, CH), 7.24-7.27 (1 H, m, CH), 7.34-7.37 (1 H, m, CH), 7.98 (1 H, d, *J* = 16 Hz, CH=CH), 8.03-8.07 (1 H, m, CH), 8.21-8.26 (2 H, m, CH) and 8.38-8.40 (1 H, m, CH);

¹³C NMR δ_C (100 MHz; CDCl₃) 26.6, 55.9, 56.1, 110.6, 111.2, 118.3, 123.0, 124.5, 126.1, 128.1, 128.2, 145.4, 149.3, 151.7, 152.5, 153.6, 188.4 and 199.5;

HRMS *m/z* (qToF) Found 312.1356 (M+H⁺). C₁₈H₁₈NO₄ requires 312.1236.

Synthesis of chalcone **C3**



Using a method adapted from a previous synthesis (*RSC Adv.*, 2024, **14**, 3519-3524), 10 mmol 2,6-acetylpyridine was added to a stirred solution of 5.0 mmol aldehyde in MeOH followed by the addition of 5.0 mmol NaOH and stirring continued at room temperature. After 24 hours the precipitate was filtered, washed with copious amounts of cold H₂O and collected and dried to afford the desired chalcone without further purification.

Yield 1.02g (60%);

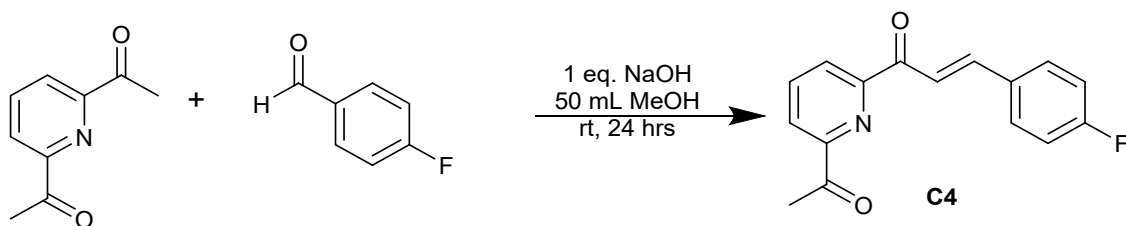
V_{max} (Solid)/cm⁻¹ 1699, 1593, 1430 and 1234;

¹H NMR δ_H (400 MHz; CDCl₃) 2.88 (3 H, s, CH₃), 3.94 (3 H, s, CH₃), 3.96 (6 H, s, CH₃), 6.98 (2 H, s, CH), 7.94 (1 H, d, *J* = 16 Hz, CH=CH), 8.06 (1 H, t, *J* = 7.6 Hz, CH), 8.24-8.28 (2 H, m, CH), 8.38-8.40 (1 H, m, CH);

¹³C NMR δ_C (100 MHz; CDCl₃) 25.6, 56.2, 61.1, 105.9, 119.7, 124.6, 126.1, 130.5, 138.2, 140.8, 145.3, 152.4, 153.4, 153.5, 188.4 and 199.3;

HRMS m/z (qToF) Found 342.1461 (M+H⁺). C₁₉H₂₀NO₅ requires 342.1341.

Synthesis of chalcone **C4**



Using a method adapted from a previous synthesis (*RSC Adv.*, 2024, **14**, 3519-3524), 6 mmol 2,6-acetylpyridine was added to a stirred solution of 3.0 mmol aldehyde in MeOH followed by the addition of 3.0 mmol NaOH and stirring continued at room temperature. After 24 hours the precipitate was filtered, washed with copious amounts of cold H₂O and collected and dried to afford the desired chalcone without further purification.

Yield 0.174g (22%);

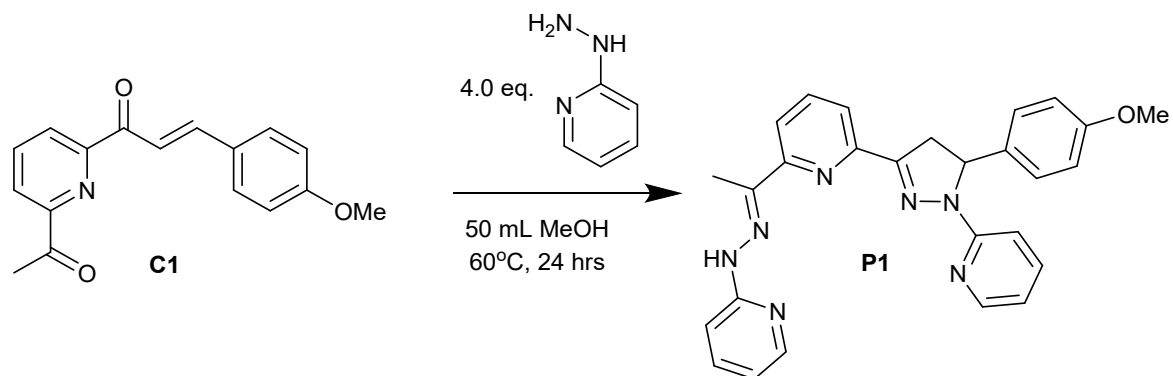
V_{max} (Solid)/cm⁻¹ 1593, 1439, 1257 and 1138;

¹H NMR δ_H (400 MHz; CDCl₃) 2.90 (3 H, s, CH₃), 7.15-7.19 (2 H, m, CH), 7.72-7.76 (2 H, m, CH), 7.98 (1 H, d, *J* = 16.0 Hz, CH=CH), 8.04-8.08 (1 H, m, CH), 8.26-8.32 (2 H, m, CH) and 8.38-8.41 (1 H, m, CH);

¹³C NMR δ_C (100 MHz; CDCl₃) 25.8, 116.1, 116.3, 120.0, 124.7, 126.2, 130.6, 138.2, 143.9, 143.9, 152.6, 153.3, 163.1, 165.5, 188.4 and 199.3;

HRMS m/z (qToF) Found 270.1068 (M+H⁺). C₁₆H₁₃FNO₂ requires 270.0930.

Synthesis of pyrazoline **P1**



4.0 mmol of 2-hydrazinopyridine was added to a stirred solution of 1.0 mmol **C1** in 50 mL MeOH and heated to 60 °C. After 24 hours the solvent was removed under reduced pressure, 100 mL H₂O added and extracted into 3 x 50 mL EtOAc. The EtOAc fractions were combined and the solvent removed under reduced pressure to give an oil which was then purified by column chromatography 6:4 EtOAc: PE to afford the desired product.

Yield 0.23g (50%);

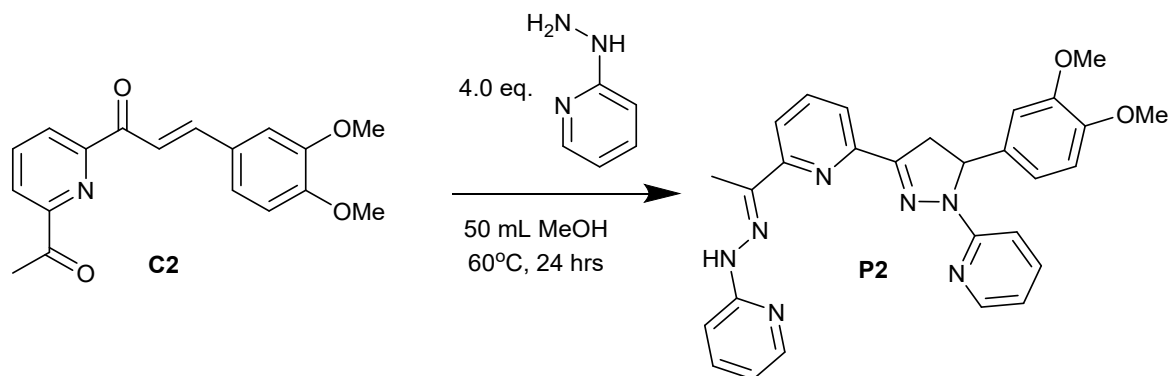
V_{max} (Solid)/cm⁻¹ 1494, 1454, 847 and 664;

¹H NMR δ_H (400 MHz; CDCl₃) 2.30 (3 H, s, CH₃), 3.32-3.38 (1 H, m, CH), 3.68 (3 H, s, OCH₃), 3.82-3.90 (1 H, m, CH), 5.67-5.72 (1 H, m, CH), 5.69-5.72 (1 H, m, CH), 6.56-6.60 (1 H, m, CH), 6.74-6.76 (3 H, Ar CH and CH), 7.15-7.18 (2 H, m, Ar CH and CH), 7.34-7.37 (2 H, m, CH), 7.42-7.44 (1 H, m, CH), 7.56-7.63 (3 H, m, CH), 7.96-8.02 (3 H, m, CH), 8.07-8.08 (1 H, m, CH), 8.21 (1 H, Br s, NH);

¹³C NMR δ_C (100 MHz; CDCl₃) 42.5, 55.2, 61.6, 107.7, 109.1, 114.0, 114.7, 116.3, 119.1, 119.5, 127.1, 130.5, 135.4, 136.0, 137.1, 138.3, 144.2, 147.7, 150.5, 151.2, 155.3, 156.6 and 158.6;

HRMS m/z (qToF) Found 464.2219 (M+H⁺). C₂₇H₂₆N₇O requires 464.2199.

Synthesis of pyrazoline **P2**



4.0 mmol of 2-hydrazinopyridine was added to a stirred solution of 1.0 mmol **C2** in 50 mL MeOH and heated to 60 °C. After 24 hours the solvent was removed under reduced pressure, 100 mL H₂O added and extracted into 3 x 50 mL EtOAc. The EtOAc fractions were combined and the solvent removed under reduced pressure to give an oil which was then purified by column chromatography 6:4 EtOAc: PE to afford the desired product.

Yield 0.21g (43%);

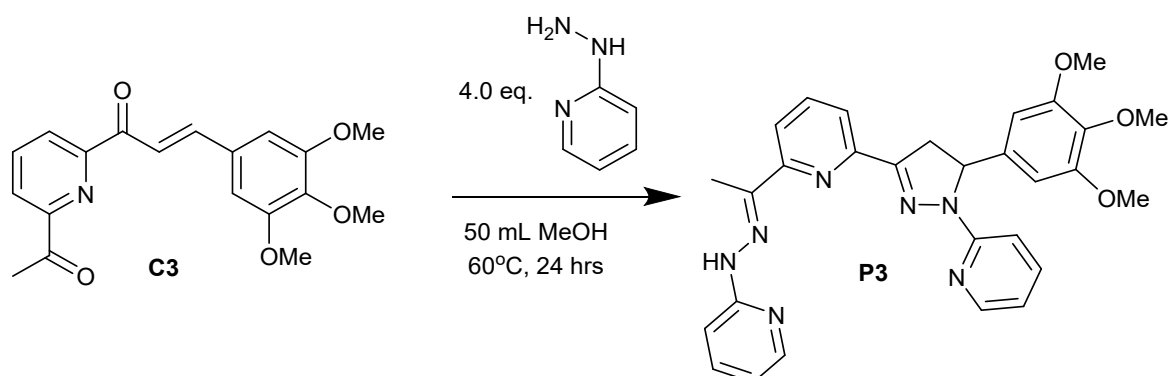
V_{max} (Solid)/cm⁻¹ 2926, 1592, 1511, 1432, 1257 and 1234;

¹H NMR δ_H (400 MHz; CDCl₃) 2.40 (3 H, s, CH₃), 3.43-3.49 (1 H, m, CH), 3.85 (6 H, s, CH₃), 3.99-4.01 (1 H, m, CH), 5.75-7.80 (1 H, m, CH), 6.69-6.80 (1 H, m, CH), 6.81-6.87 (5 H, m, CH), 7.45-7.47 (3 H, m, CH), 7.53-7.55 (1 H, m, CH), 7.66-7.74 (1 H, m, CH), 8.11-8.12 (3 H, m, CH), 8.18- 8.19 (1 H, m, CH), 8.25 (1 H, br s, NH);

¹³C NMR δ_C (100 MHz; CDCl₃) 42.5, 55.9, 62.0, 65.9, 107.7, 109.1, 109.2, 111.3, 114.85, 116.3, 117.8, 119.5, 121.9, 123.1, 135.9, 136.0, 137.1, 138.2, 144.0, 144.6, 147.8, 147.9, 148.0, 149.1, 150.5, 151.26, 155.3 and 156.6;

HRMS m/z (qToF) Found 494.2349 (M+H⁺). C₂₈H₂₈N₇O₂ requires 494.2304.

Synthesis of pyrazoline **P3**



4.0 mmol of 2-hydrazinopyridine was added to a stirred solution of 1.0 mmol **C3** in 50 mL MeOH and heated to 60 °C. After 24 hours the solvent was removed under reduced pressure, 100 mL H₂O added and extracted into 3 x 50 mL EtOAc. The EtOAc fractions were combined and the solvent removed under reduced pressure to give an oil which was then purified by column chromatography 6:4 EtOAc: PE to afford the desired product.

Yield 0.320g (61%);

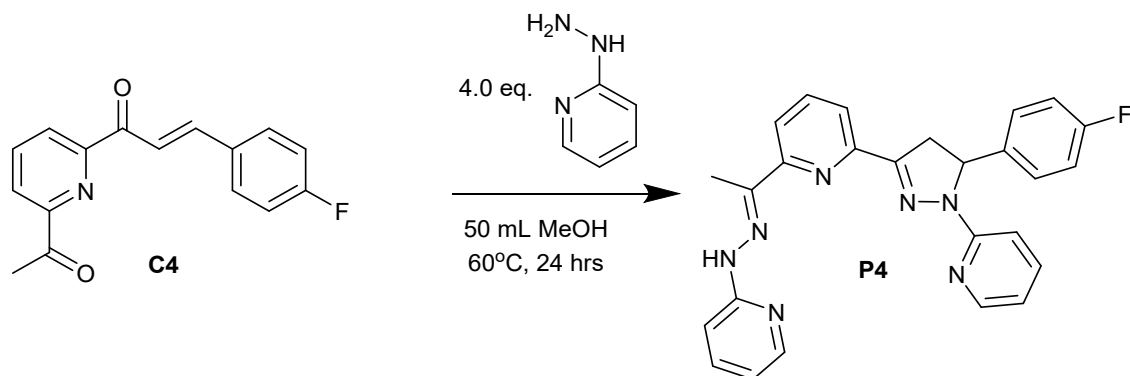
V_{max} (Solid)/cm⁻¹ 2928, 1592, 1432, 1257, 1161 and 1137;

¹H NMR δ_H (400 MHz; CDCl₃) 2.41 (3 H, s, CH₃), 3.42-3.48 (1 H, m, CH), 3.82 (9 H, s, CH₃), 3.99-4.15 (1 H, m, CH), 5.32-5.78 (1 H, m, CH), 6.53 (2 H, s, CH), 6.72-6.73 (1 H, m, CH), 6.83-6.86 (1 H, m, CH), 7.44-7.49 (2 H, m, CH), 7.55-7.59 (1 H, m, CH), 7.65-7.68 (1 H, m, CH), 7.72-7.76 (1 H, m, CH), 8.09-8.150 (3 H, m, CH), 8.18-8.20 (1 H, m, CH) and 8.25 (1 H, br s, NH);

¹³C NMR δ_C (100 MHz; CDCl₃) 42.6, 56.1, 60.8, 62.4, 102.5, 107.7, 109.2, 114.9, 116.4, 119.3, 119.5, 136.9, 137.2, 138.2, 139.0, 143.9, 147.8, 147.9, 150.4, 151.4, 153.5, 155.3, 155.4 and 156.6;

HRMS m/z (qToF) Found 524.6547 (M+H⁺). C₂₉H₃₀N₇O₃ requires 524.6050.

Synthesis of pyrazoline **P4**



2.2 mmol of hydrazine was added to a stirred solution of 0.55 mmol **C4** in 50 mL MeOH and heated to 60°C . After 24 hours the solvent was removed under reduced pressure, 100 mL H_2O added and extracted into 3 x 50 mL EtOAc. The EtOAc fractions were combined and the solvent removed under reduced pressure to give an oil which was then purified by column chromatography 6:4 EtOAc: PE to afford the desired product.

Yield 0.06g (24%);

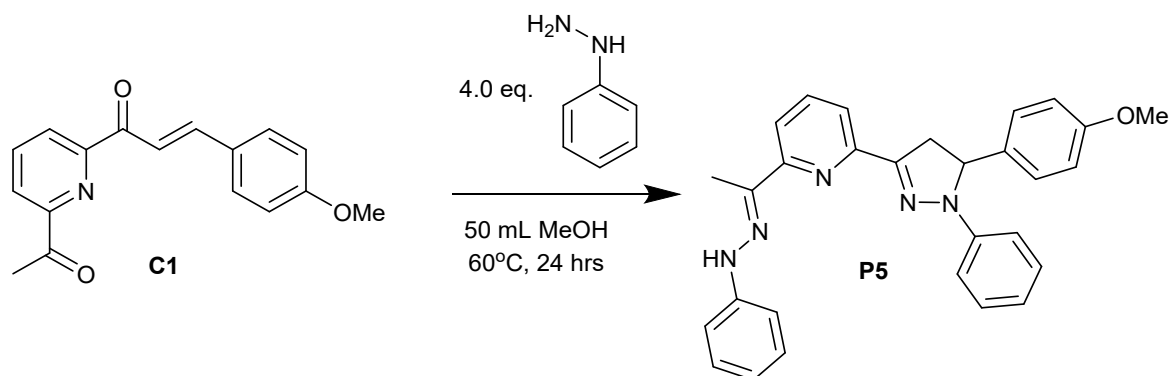
Vmax (Solid)/ cm^{-1} 2927, 1592, 1432, 1257 and 1138;

^1H NMR δ_{H} (400 MHz; CDCl_3) 2.30 (3 H, s, CH_3), 3.30-3.34 (1 H, m, CH), 3.85-3.92 (1 H, m, CH), 5.72-5.74 (1 H, m, CH), 6.57-6.59 (1 H, m, CH), 6.74-7.77 (1 H, m, CH), 6.87-8.91 (2 H, m, CH), 7.19-7.21 (1 H, m, CH), 7.33-7.39 (2 H, m, CH), 7.40-7.45 (1 H, m, CH), 7.55-7.56 (1 H, m, CH), 7.62-7.66 (1 H, m, CH), 7.96-8.09 (3 H, m, CH), 8.08-8.09 (1 H, m, CH) and 8.13 (1 H, br s, NH);

^{13}C NMR δ_{C} (100 MHz; CDCl_3); 42.4, 61.5, 107.7, 109.2, 114.9, 115.4, 115.6, 116.3, 119.3, 119.5, 127.5, 127.6, 136.0, 137.1, 138.2, 144.0, 147.7, 150.4, 151.1, 155.2, 155.3 and 156.6;

HRMS m/z (qToF) Found 452.2185 ($\text{M}+\text{H}^+$). $\text{C}_{26}\text{H}_{23}\text{FN}_2$ requires 452.5174.

Synthesis of pyrazoline **P5**



4.0 mmol of phenylhydrazine was added to a stirred solution of 1.0 mmol **C1** in 50 mL MeOH and heated to 60 °C. After 24 hours the solvent was removed under reduced pressure, 100 mL H₂O added and extracted into 3 x 50 mL EtOAc. The EtOAc fractions were combined and the solvent removed under reduced pressure to give an oil which was then purified by column chromatography 6:4 EtOAc: PE to afford the desired product.

Yield 0.09g (21%);

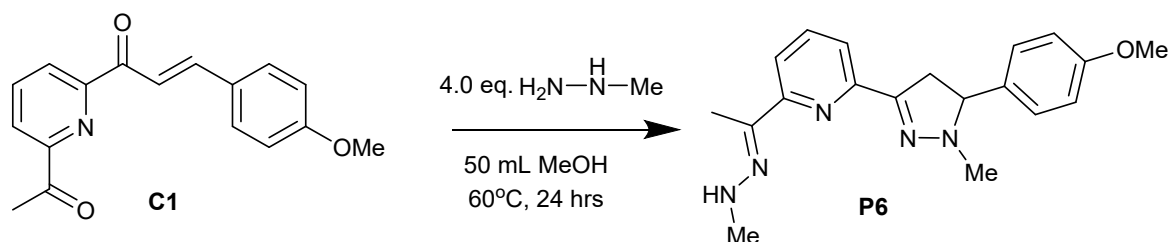
V_{max} (Solid)/cm⁻¹ 2928, 1595, 1434 and 1244;

¹H NMR δ_H (400 MHz; CDCl₃) 2.37 (3 H, s, CH₃), 3.35-3.41 (1 H, m, CH), 3.81 (3 H, s, CH₃), 3.99-4.06 (1 H, m, CH), 5.20-5.23 (1 H, m, CH), 6.83-6.93 (4 H, m, CH), 7.12-7.22 (2 H, m, CH), 7.23-7.24 (5 H, m, CH), 7.27-7.35 (3 H, m, CH), 7.54-7.57 (1 H, m, CH), 7.70-7.29 (1 H, m, CH), 8.04-8.10 (1 H, m, CH) and 8.10- 8.12 (1 H, m, CH);

¹³C NMR δ_C (100 MHz; CDCl₃); 43.5, 55.3, 64.2, 113.3, 113.6, 114.5, 118.7, 118.9, 119.4, 120.6, 122.4, 127.1, 128.9, 129.3, 134.8, 135.9, 142.1, 144.6, 148.7, 150.7, 155.6 and 159.0;

HRMS m/z (qToF) Found 432.5376 (M+H⁺). C₂₈H₂₆N₅ requires 432.5510.

Synthesis of pyrazoline **P6**



4.0 mmol of methylhydrazine was added to a stirred solution of 1.0 mmol **C1** in 50 mL MeOH and heated to 60 °C. After 24 hours the solvent was removed under reduced pressure, 100 mL H_2O added and extracted into 3 x 50 mL EtOAc. The EtOAc fractions were combined and the solvent removed under reduced pressure to give an oil which was then purified by column chromatography 6:4 EtOAc: PE to afford the desired product.

Yield 0.06g (18%);

Vmax (Solid)/ cm^{-1} 2921, 1165, 1341 and 1141;

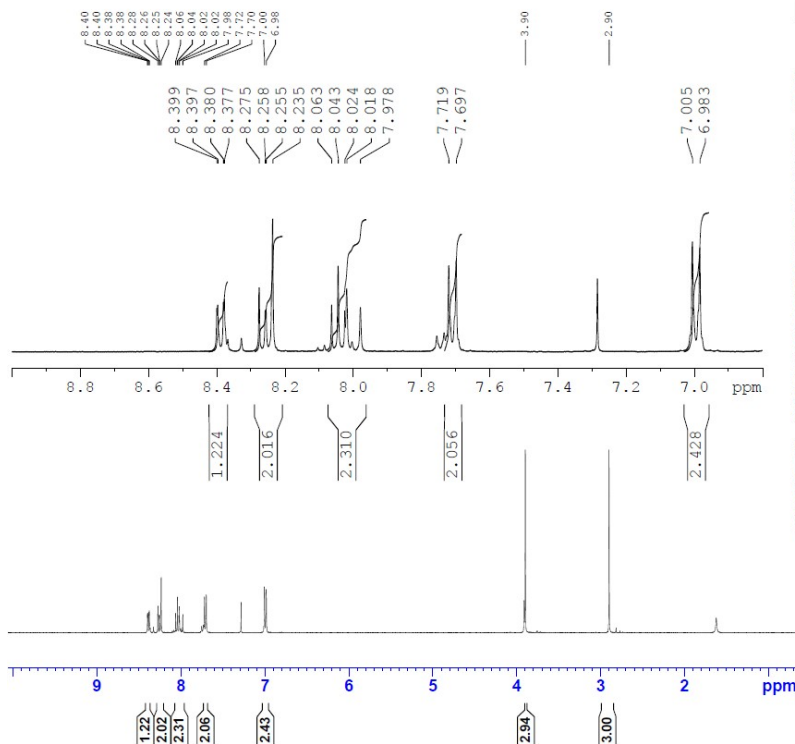
^1H NMR δ_{H} (400 MHz; CDCl_3) 2.20 (3 H, s, CH_3), 2.89 (3 H, s, CH_3), 1.90 (1 H, m, CH), 3.72 (3 H, s, CH_3), 3.85 (1 H, m, CH), 3.98 (1 H, m, CH), 5.20 (1 H, br s, NH), 6.93-6.95 (2 H, m, CH), 7.41-7.43 (2 H, m, CH), 7.62 (1 H, m, CH), 7.81-7.82 (1 H, m, CH) and 7.61-7.93 (1 H, br s, NH);

^{13}C NMR δ_{C} (100 MHz; CDCl_3); 41.2, 42.9, 55.3, 114.0, 114.1, 114.4, 118.4, 118.4, 118.5, 118.9, 120.3, 121.1, 122.6, 130.4, 132.4, 151.6 and 159.3;

HRMS m/z (qToF) Found 338.4387 ($\text{M}+\text{H}^+$). $\text{C}_{19}\text{H}_{24}\text{N}_5\text{O}$ requires 338.4350.

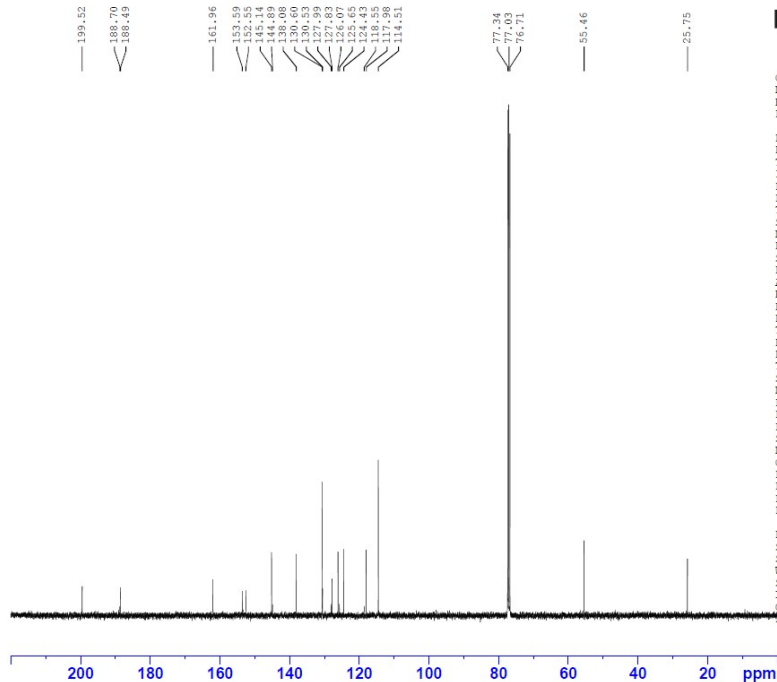
NMR Spectra (S3)

C1
PROTON16.CMDnp CDCl3 {C:\Bruker\TopSpin3.6.5} nmrsu 62



¹H NMR (400 Mhz) of C1 in CDCl₃

C1
C13CPD512.CMDnp CDCl3 {C:\Bruker\TopSpin3.6.5} nmrsu 62



Current Data Parameters
NAME Dec19-2024-Alex
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
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Time 8.30 h
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 5.1118078 sec
RG 209.43
DW 78.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SF01 400.1924011 MHz
NUC1 1H
FO 5.00 usec
PI 15.00 usec
PLW1 11.09700012 W

F2 - Processing parameters
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SF 400.1900000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

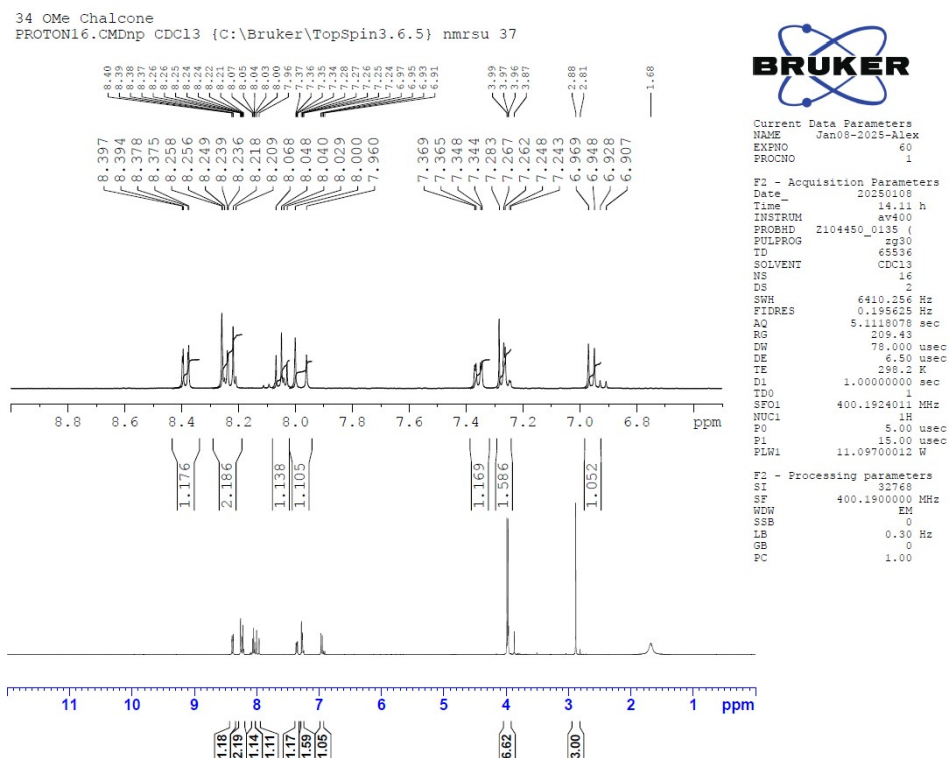


Current Data Parameters
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EXPNO 20
PROCNO 1

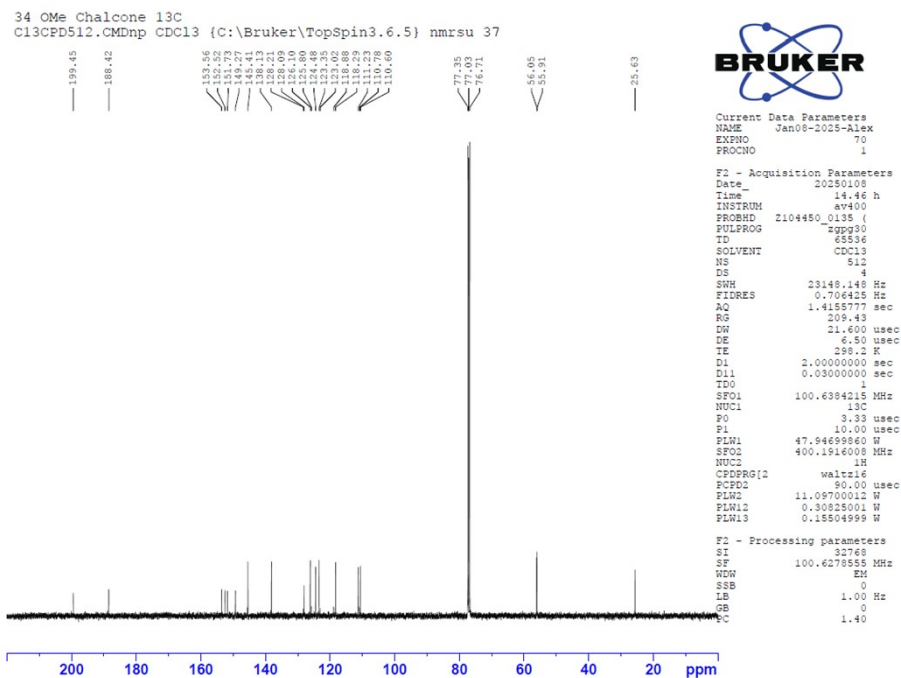
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SOLVENT CDCl3
NS 512
DS 4
SWH 23148.148 Hz
FIDRES 0.706425 Hz
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RG 209.43
DW 21.600 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SF01 100.6384215 MHz
NUC1 13C
FO 3.33 usec
PI 10.00 usec
PLW1 47.94699860 W
SF02 400.1916008 MHz
NUC2 1H
CPDPRG12 waltz16
PCPD2 90.00 usec
PLW2 11.09700012 W
PLW12 0.30825001 W
PLW13 0.15504999 W

F2 - Processing parameters
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WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹³C NMR (100 Mhz) of **C1** in CDCl₃

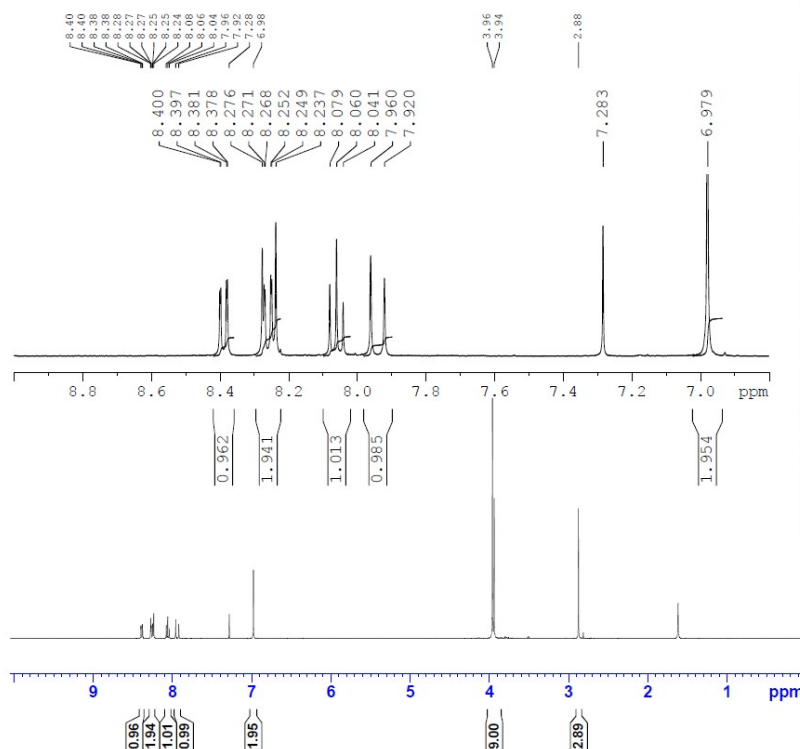


¹H NMR (400 Mhz) of **C2** in CDCl₃



¹³C NMR (100 Mhz) of **C2** in CDCl₃

345 OMe Chalcone
 PROTON16.CMDnp CDCl3 {C:\Bruker\TopSpin3.6.5} nmrsu 94



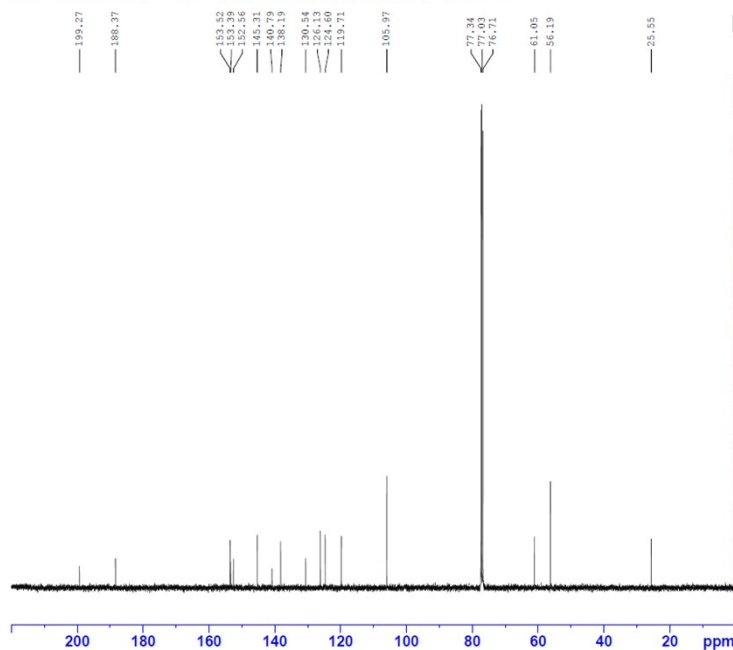
Current Data Parameters
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 EXPNO 10
 PROCNO 1

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 PROBHD Z104450_0135 ()
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 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6410.256 Hz
 FIDRES 0.195625 Hz
 AQ 5.1118078 sec
 RG 209.43
 DW 78.000 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.00000000 sec
 TDO 1
 SFO1 400.1924011 MHz
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 P0 5.00 usec
 P1 15.00 usec
 PLW1 11.09700012 W

F2 - Processing parameters
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 SF 400.1900000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹H NMR (400 Mhz) of **C3** in CDCl₃

345 OMe Chalcone 13C
 C13CPD512.CMDnp CDCl3 {C:\Bruker\TopSpin3.6.5} nmrsu 94



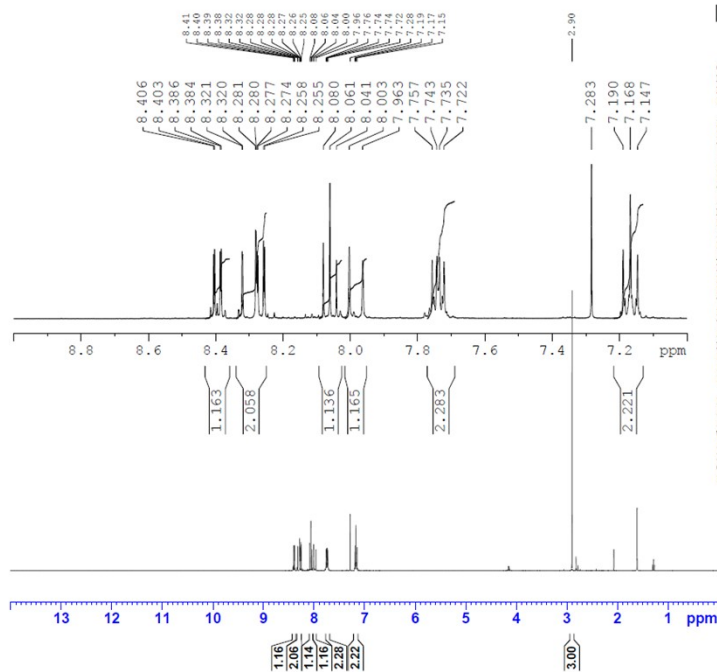
Current Data Parameters
 NAME Jan07-2025-Alex
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date 20250107
 Time 13.18 h
 INSTRUM av400
 PROBHD Z104450_0135 ()
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 23148.148 Hz
 FIDRES 0.706425 Hz
 AQ 1.4155777 sec
 RG 209.43
 DW 21.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1
 SFO1 100.6384215 MHz
 NUC1 13C
 P0 3.33 usec
 P1 10.00 usec
 PLW1 47.94699860 W
 SFO2 400.1916008 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 11.09700012 W
 PLW12 0.30825001 W
 PLW13 0.15504999 W

F2 - Processing parameters
 SI 32768
 SF 100.6278555 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹³C NMR (100 Mhz) of **C3** in CDCl₃

4F Chalcone
 PROTON16.CMDnp CDC13 {C:\Bruker\TopSpin3.6.5} nmrsu 91



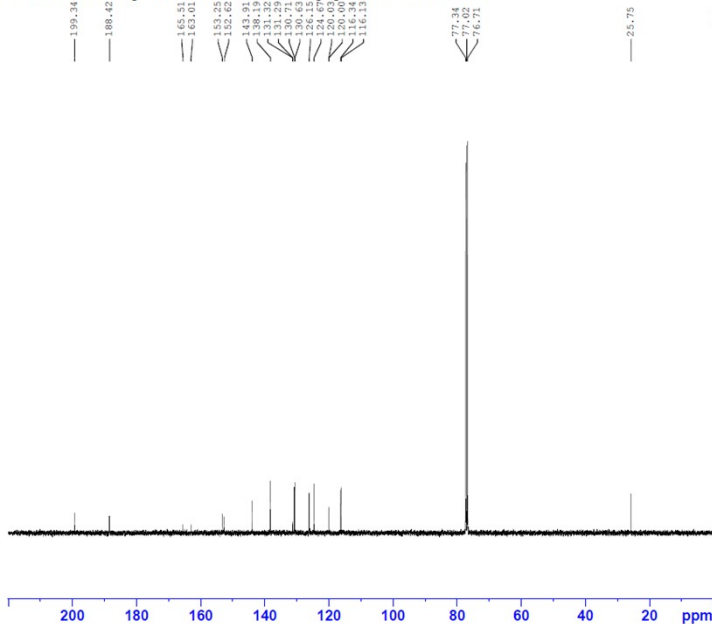
Current Data Parameters
 NAME Jan09-2025-Alex
 EXPNO 50
 PROCNO 1

F2 - Acquisition Parameters
 Date 20250109
 Time 12.48 h
 INSTRUM av400
 PROBHD Z104450_0135
 PULPROG zgpg30
 ID 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 6410.256 Hz
 FIDRES 0.195625 Hz
 AQ 5.1118078 sec
 RG 209.43
 DW 78.000 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.00000000 sec
 ID0 1
 SF01 400.1924011 MHz
 NUC1 1H
 P0 5.00 usec
 P1 15.00 usec
 PLW1 11.09700012 W

F2 - Processing parameters
 SI 32768
 SF 400.1900000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹H NMR (400 Mhz) of **C4** in CDCl₃

4F chalcone 13C
 C13CPD512.CMDnp CDC13 {C:\Bruker\TopSpin3.6.5} nmrsu 91



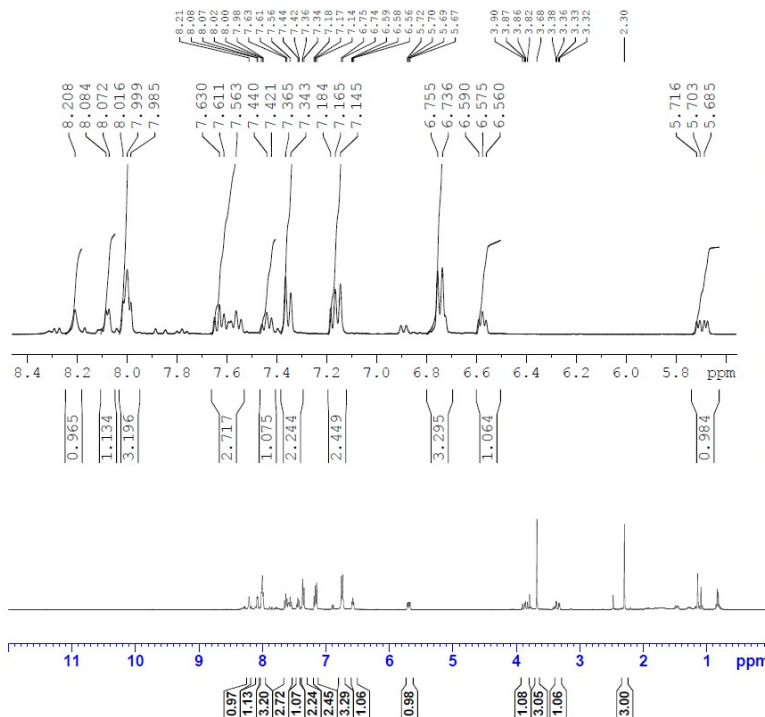
Current Data Parameters
 NAME Jan09-2025-Alex
 EXPNO 60
 PROCNO 1

F2 - Acquisition Parameters
 Date 20250109
 Time 13.27 h
 INSTRUM av400
 PROBHD Z104450_0135
 PULPROG zgpg30
 ID 65536
 SOLVENT CDC13
 NS 512
 DS 1
 SWH 23148.148 Hz
 FIDRES 0.706425 Hz
 AQ 1.4155777 sec
 RG 209.43
 DW 21.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 ID0 1
 SF01 100.6394215 MHz
 NUC1 13C
 P0 3.33 usec
 P1 10.00 usec
 PLW1 47.94699860 W
 SF02 400.1916008 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 11.09700012 W
 PLW12 0.30825001 W
 PLW13 0.15504999 W

F2 - Processing parameters
 SI 32768
 SF 100.6278555 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹³C NMR (100 Mhz) of **C4** in CDCl₃

PROTON16.CMDnp CDCl3 {C:\Bruker\TopSpin3.6.5} nmrsu 15



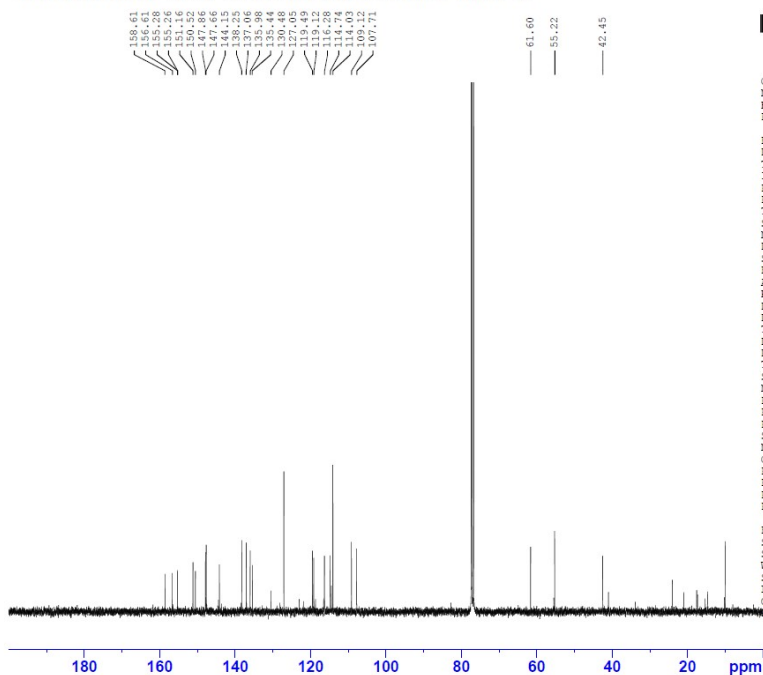
Current Data Parameters
NAME Dec17-2024-Alex
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20241217
Time 14.16 h
INSTRUM av400
PROBHD Z104450_0135 ()
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 5.1118078 sec
RG 209.43
DW 78.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SF01 400.1924011 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
PLW1 11.09700012 W

F2 - Processing parameters
SI 32768
SF 400.1900395 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H NMR (400 Mhz) of **P1** in CDCl₃

C13CPD512.CMDnp CDCl3 {C:\Bruker\TopSpin3.6.5} nmrsu 14



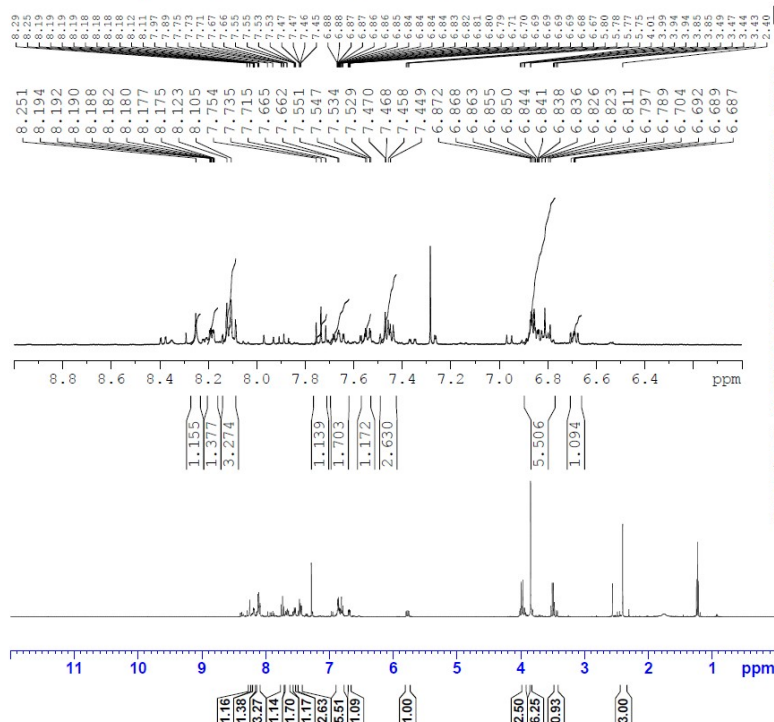
Current Data Parameters
NAME Dec17-2024-Alex
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20241217
Time 14.53 h
INSTRUM av400
PROBHD Z104450_0135 ()
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 1.4155777 sec
RG 209.43
DW 21.600 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SF01 100.6384215 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 47.94699860 W
SF02 400.1516008 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 11.09700012 W
PLW12 0.30825001 W
PLW13 0.15504999 W

F2 - Processing parameters
SI 32768
SF 100.6278555 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹³C NMR (100 Mhz) of **P1** in CDCl₃

3,4 OMe Hydrazone
 PROTON16.CMDnp CDCl3 {C:\Bruker\TopSpin3.6.5} nmrsu 11



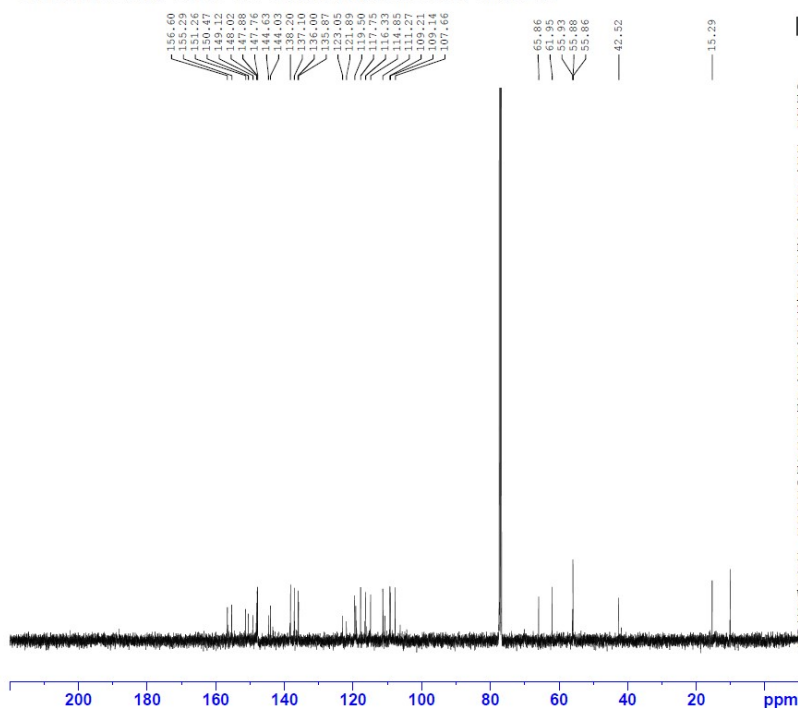
Current Data Parameters
 NAME Jan10-2025-Alex
 EXPNO 50
 PROCNO 1

F2 - Acquisition Parameters
 Date 20250110
 Time 15.24 h
 INSTRUM av400
 PROBHD Z104450_0135
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 6410.256 Hz
 FIDRES 0.195625 Hz
 AQ 5.1118078 sec
 RG 209.43
 DW 78.000 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.00000000 sec
 TD0 1
 SF01 400.1924011 MHz
 NUC1 1H
 P0 5.00 usec
 F1 15.00 usec
 PLW1 11.09700012 W

F2 - Processing parameters
 SI 32768
 SF 400.1900000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹H NMR (400 Mhz) of **P2** in CDCl₃

3,4 OMe Hydrazone
 C13CPD512.CMDnp CDCl3 {C:\Bruker\TopSpin3.6.5} nmrsu 11



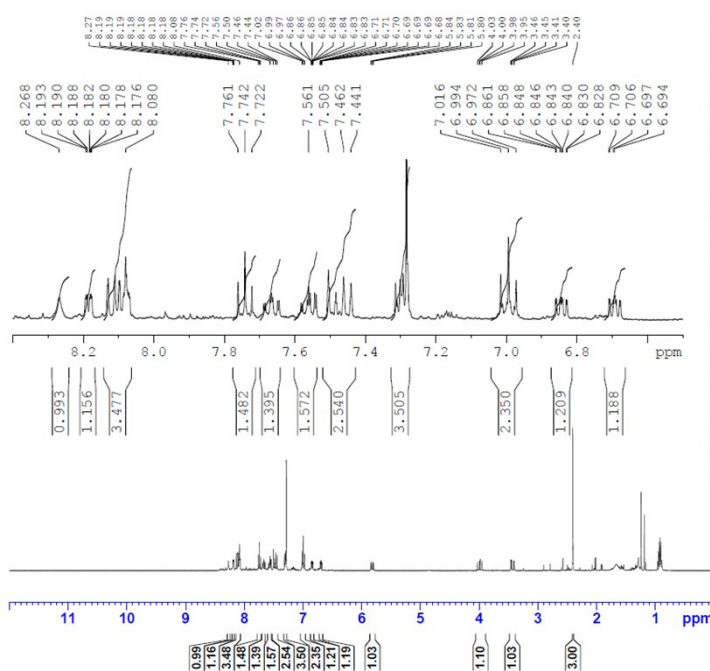
Current Data Parameters
 NAME Jan10-2025-Alex
 EXPNO 70
 PROCNO 1

F2 - Acquisition Parameters
 Date 20250110
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 INSTRUM av400
 PROBHD Z104450_0135
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 23148.148 Hz
 FIDRES 0.706425 Hz
 AQ 1.4155777 sec
 RG 209.43
 DW 21.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1
 SF01 100.6384215 MHz
 NUC1 13C
 P0 3.33 usec
 F1 10.00 usec
 PLW1 47.94699860 W
 SF02 400.1916008 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 11.09700012 W
 PLW12 0.30825001 W
 PLW13 0.15504999 W

F2 - Processing parameters
 SI 32768
 SF 100.6278555 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹³C NMR (100 Mhz) of **P2** in CDCl₃

4F Hydrazone
PROTON16.CMDnp CDCl3 {C:\Bruker\TopSpin3.6.5} nmrsu 10



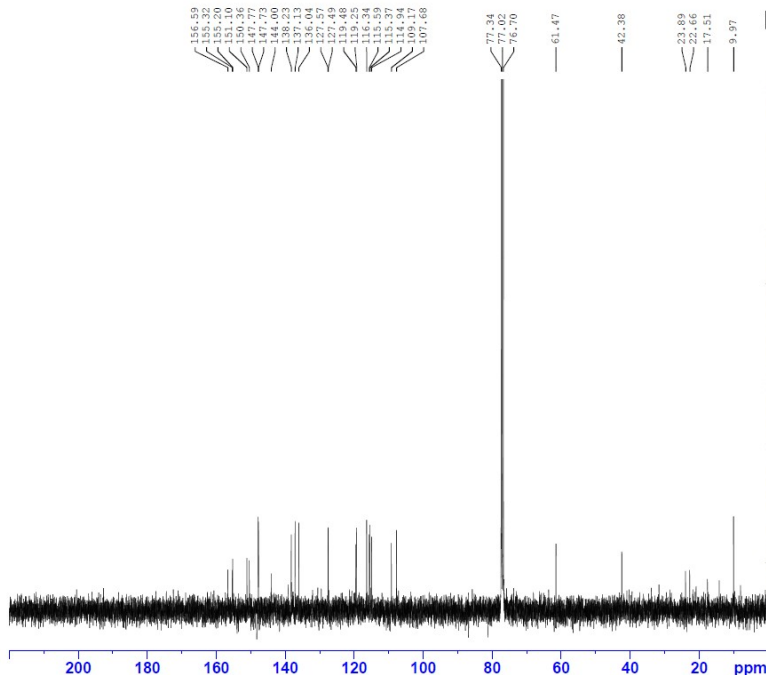
Current Data Parameters
NAME Jan10-2025-Alex
EXPNO 40
PROCNO 1

F2 - Acquisition Parameters
Date_ 20250110
Time_ 15.16 h
INSTRUM av400
PROBHD Z104450_0135 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 5.1118078 sec
RG 209.43
DW 76.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
D11 1
D12 1
SFO1 400.1924011 MHz
NUC1 1H
P0 5.00 usec
P1 15.00 usec
PLW1 11.09700012 W

F2 - Processing parameters
SI 32768
SF 400.1900000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹H NMR (400 Mhz) of **P4** in CDCl₃

4F Hydrazone 13C
C13CPD512.CMDnp CDCl3 {C:\Bruker\TopSpin3.6.5} nmrsu 10



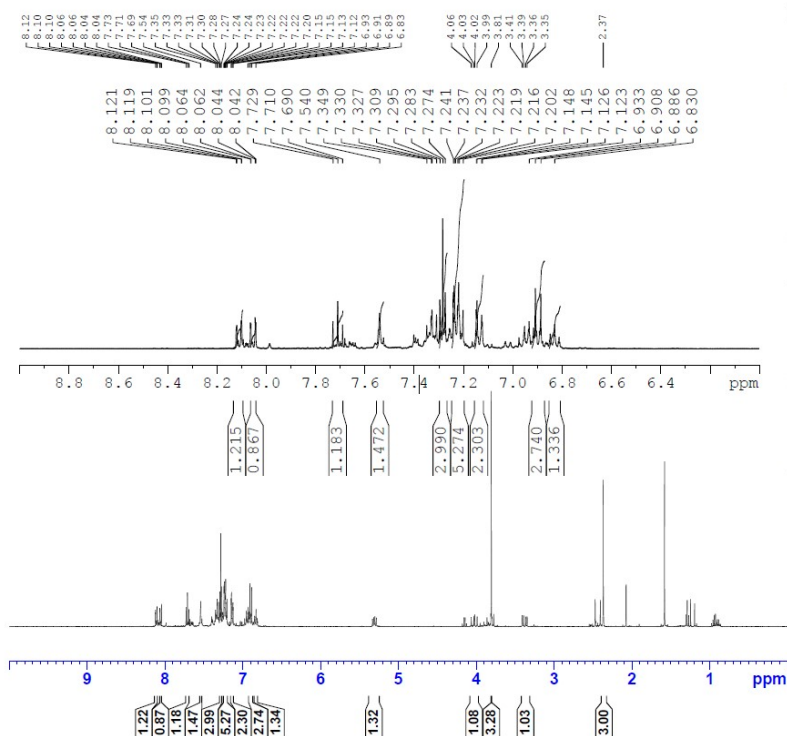
Current Data Parameters
NAME Jan10-2025-Alex
EXPNO 60
PROCNO 1

F2 - Acquisition Parameters
Date_ 20250110
Time_ 15.59 h
INSTRUM av400
PROBHD Z104450_0135 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 1.4155777 sec
RG 209.43
DW 21.600 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 1
D13 1
SFO1 100.6384215 MHz
NUC1 13C
P0 3.33 usec
P1 10.00 usec
PLW1 47.94699860 W
SFO2 400.1816008 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 11.09700013 W
PLW12 0.30825001 W
PLW13 0.15504999 W

F2 - Processing parameters
SI 32768
SF 100.6278555 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹³C NMR (100 Mhz) of **P4** in CDCl₃

Ph Hydrazone Lower spot
 PROTON16.CMDnp CDCl₃ (C:\Bruker\TopSpin3.6.5) nmrsu 53



¹H NMR (400 Mhz) of P5 in CDCl₃

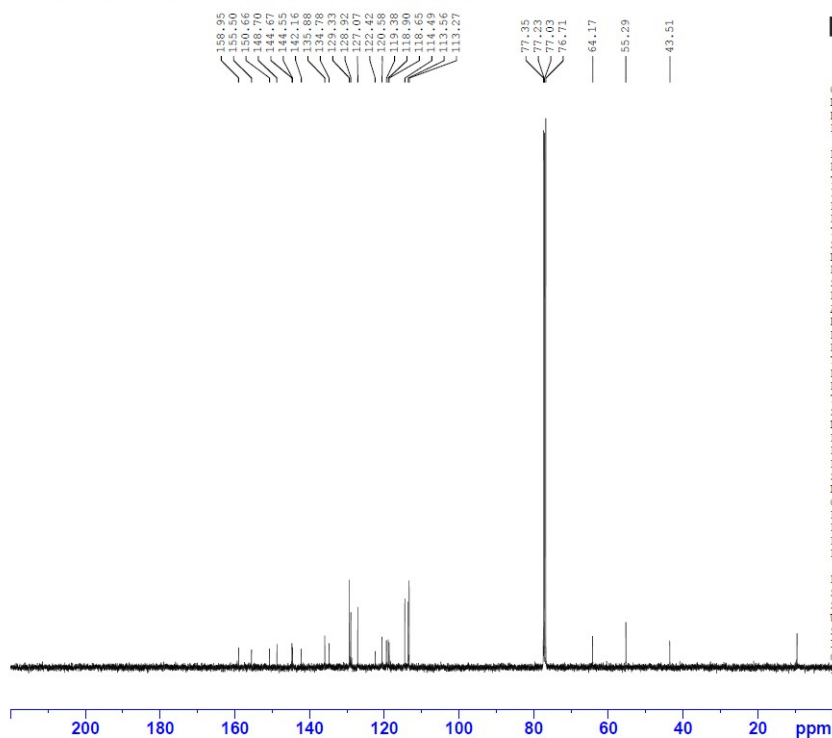


Current Data Parameters
 NAME Jan15-2025-Alex
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20250115
 Time 13.14 h
 INSTRUM av400
 PROBHD Z104450_0135 ()
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 6410.256 Hz
 FIDRES 0.195625 Hz
 AQ 5.1118078 sec
 RG 209.43
 DW 78.000 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.00000000 sec
 TD0 1
 SFO1 400.1924011 MHz
 NUC1 1H
 P0 5.00 usec
 PL1 15.00 usec
 PLW1 11.09700012 W

F2 - Processing parameters
 SI 32768
 SF 400.1900000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Ph Hydrazone
 C13CPD512.CMDnp CDCl₃ (C:\Bruker\TopSpin3.6.5) nmrsu 53



¹³C NMR (100 Mhz) of P5 in CDCl₃



Current Data Parameters
 NAME Jan15-2025-Alex
 EXPNO 40
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20250115
 Time 14.32 h
 INSTRUM av400
 PROBHD Z104450_0135 ()
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 512
 DS 4
 SWH 23149.148 Hz
 FIDRES 0.706425 Hz
 AQ 1.4155777 sec
 RG 209.43
 DW 21.600 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1
 SFO1 100.6384215 MHz
 NUC1 13C
 P0 3.33 usec
 PL1 10.00 usec
 PLW1 47.94699860 W
 SFO2 400.1916008 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 11.09700012 W
 PLW12 0.30825001 W
 PLW13 0.15504999 W

F2 - Processing parameters
 SI 32768
 SF 100.6278555 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Limit of Detection (LoD) Studies (S4)

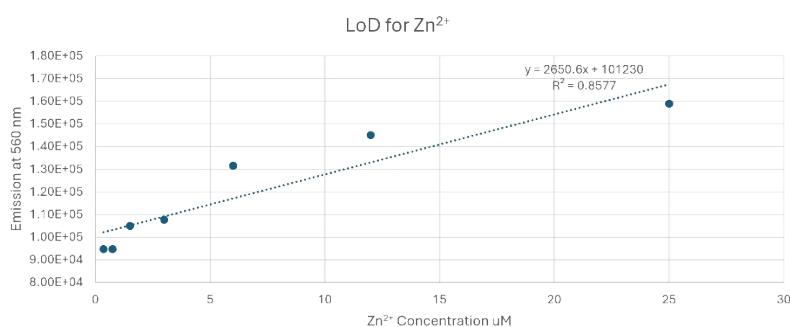
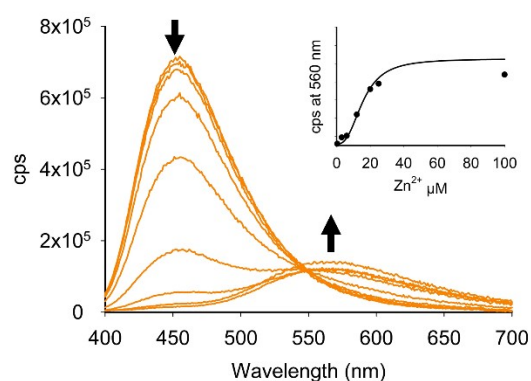
The method reported by Lee *et al* was used to calculate limit of detection (LoD) for **P1** (λ_{ex} 285 nm) in 7:3 MeCN:H₂O with the average from two replicates used.

$$\text{LoD} = 3\sigma_{\text{bi}}/m$$

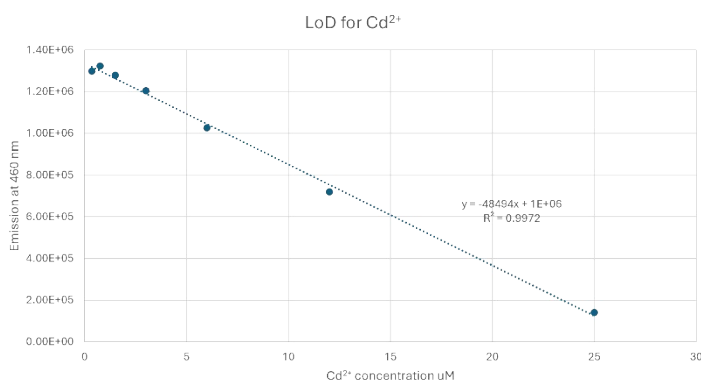
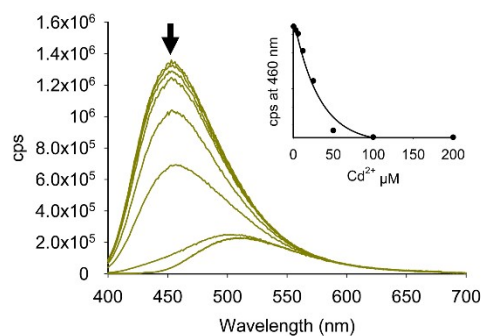
σ_{bi} = standard deviation of sensor only (n=10)

m = gradient of the slope

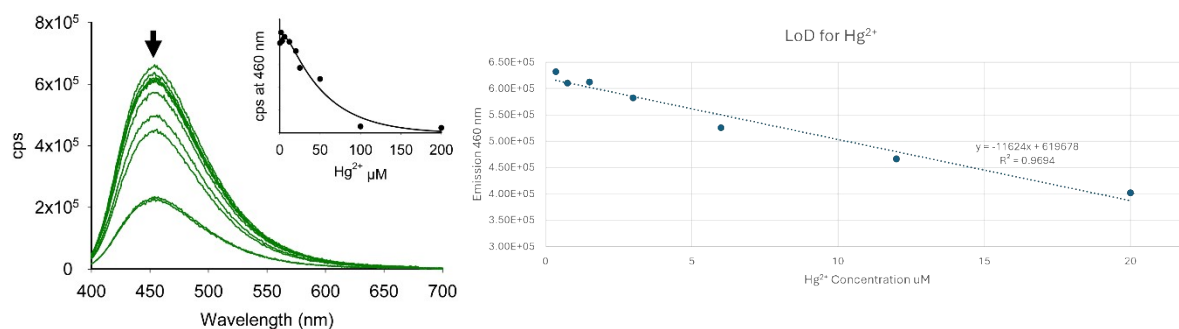
B. P. Joshi, J. Park, W. I. Lee and K.-H. Lee, Talanta, 2009, 78, 903.



Limit of detection study for **P1** with Zn²⁺ (0.35, 0.7, 1.5, 3, 6, 12, 25 μM) with λ_{em} 560 nm. Values are the average of two independent experiments. Standard deviation of blanks (n= 10) at 560 nm = 5705. Zn²⁺ LoD calculated at 6.4 μM.

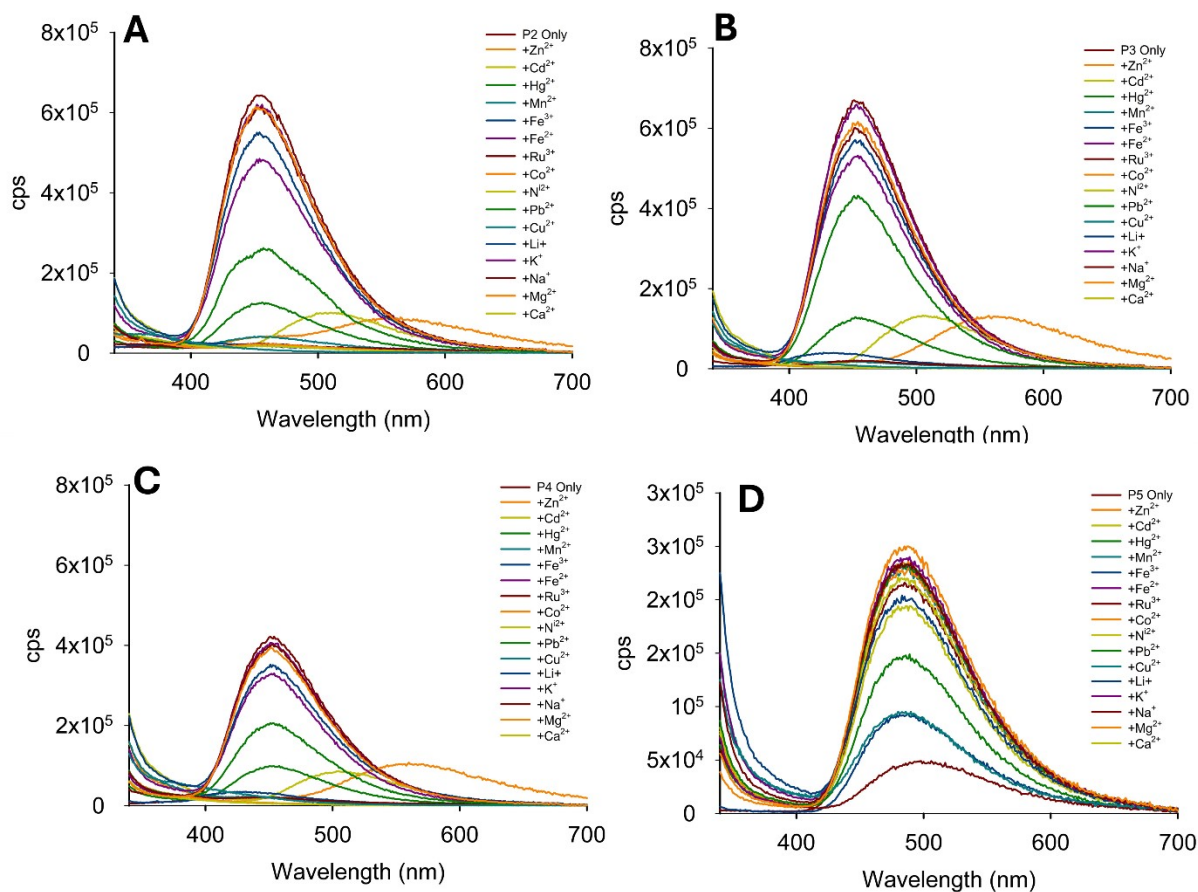


Limit of detection study for **P1** with Cd²⁺ (0.35, 0.7, 1.5, 3, 6, 12, 25 μM) with λ_{em} 460 nm. Values are the average of two independent experiments. Standard deviation of blanks (n= 10) at 460 nm = 39048. Cd²⁺ LoD calculated at 2.4 μM.

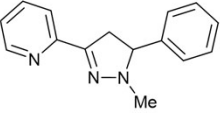
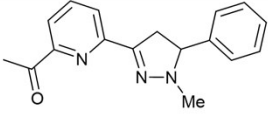
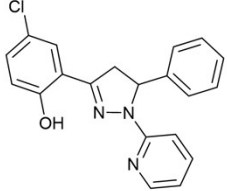
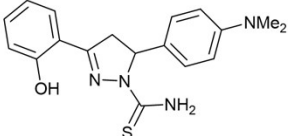
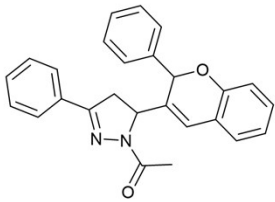
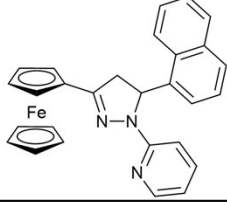
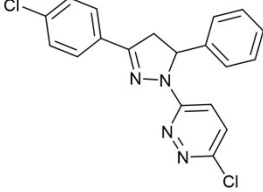
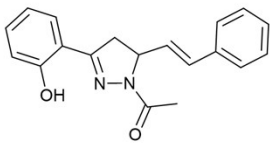


Limit of detection study for **P1** with Hg^{2+} (0.35, 0.7, 1.5, 3, 6, 12, 20 μM) with λ_{em} 460 nm. Values are the average of two independent experiments. Standard deviation of blanks ($n=10$) at 460 nm = 39048. Hg^{2+} LoD calculated at 10.0 μM .

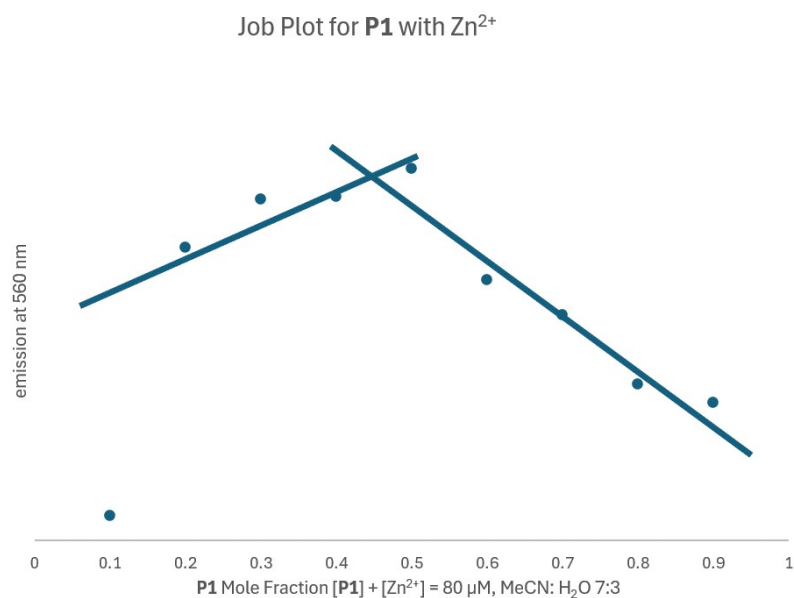
Metal Screen for P2-P5 (S5)



Limit of Detection (LoD) for literature pyrazolines (S6)

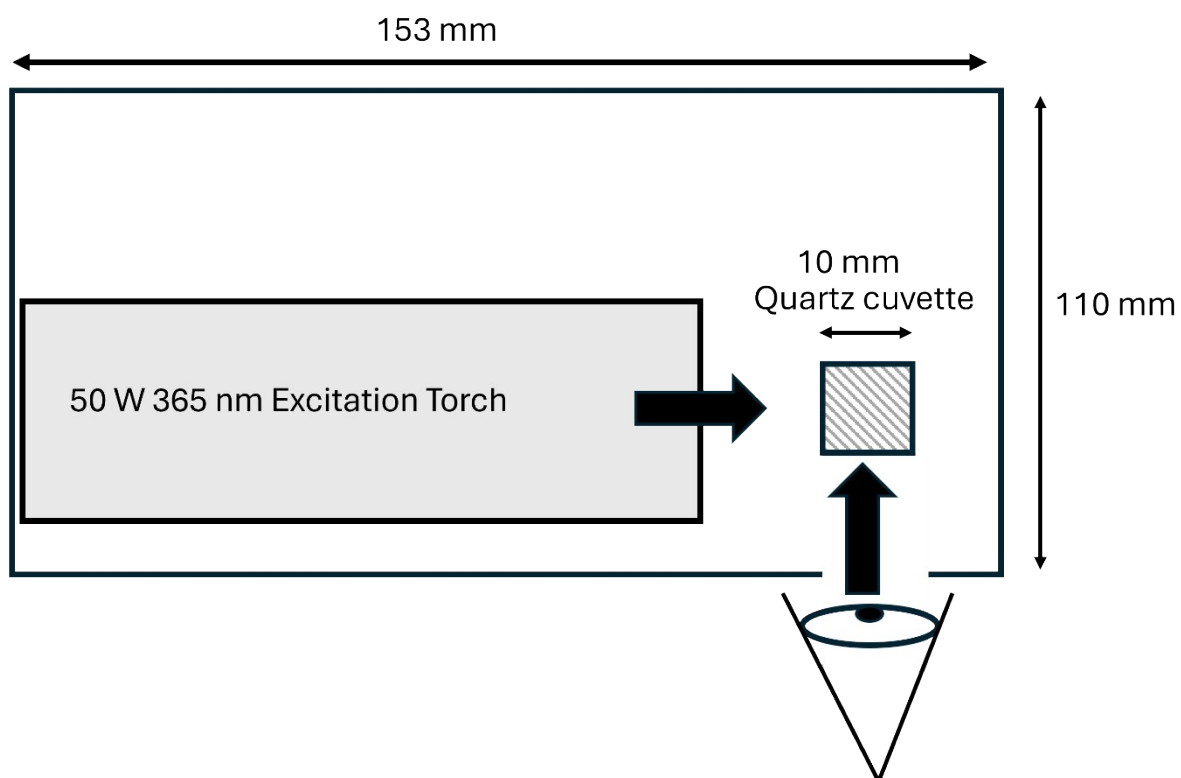
Sensor	Solvent	Zn ²⁺ LoD	Cd ²⁺ LoD	Reference
S1 	100% MeCN	0.2 μM	0.12 μM	<i>Org. Biomol. Chem.</i> , 2012, 10 , 8753
S2 	100% MeCN	0.03 μM	-	<i>RSC Adv.</i> , 2024, 14 , 3519
S3 	50% H ₂ O 50% MeCN	0.12 μM	-	<i>Sensors and Actuators B</i> , 2011, 159 , 148
S4 	50% H ₂ O 50% EtOH	0.93 μM	0.82 μM	<i>Inorganic Chemistry Communications</i> , 2021, 130 , 108735
S5 	50% H ₂ O 50% EtOH	0.16 μM	-	<i>Inorganica Chimica Acta</i> , 2018, 479 , 128
S6 	CH ₂ Cl ₂	1.4 μM	-	<i>Eur. J. Inorg. Chem.</i> 2013, 6019
S7 	90% MeCN 10% EtOH	0.4 μM	-	<i>Journal of Photochemistry and Photobiology A: Chemistry</i> , 2011, 218 , 6
S8 	50% H ₂ O 50% EtOH	0.03 μM	-	<i>Anal. Methods</i> , 2018, 10 , 1833

Job Plot for P1 with Zn²⁺ (S7)



Job Plot for P1 with Zn²⁺, λ_{ex} 295 nm, λ_{em} 560 nm, total concentration was 80 μM in a 7:3 MeCN: H₂O.

In situ monitoring using prototype portable device (S8):



Torch used:



Roll over image to zoom in



DARKBEAM UV Torch 365nm Flashlight Wood's lamp, Rechargeable Blacklight 50W Powerful, LED Ultraviolet USB-C Black Light Portable Pet Urine Detector for Resin Curing, Ore, Leak Detection [Energy Class A]

Brand: DARKBEAM

4.2 ★★★★★ (17)

1 sustainability feature

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Special feature	3D print, UV resin curing, Pet Dog Urine Detection, Scorpions, Uranium glass, invisible ink, Woods lamp for Cat Ringworm/ Pee Detector
Colour	Uv 365nm(bluish)
Power source	Battery Powered
Light source type	UV
Material	Aluminium

About this item

- Higher Power, more practical UV torch: This is a spotlight blacklight that integrates high purity, high intensity and long range. Filtered and pure uv 365nm ultraviolet, unlike ordinary unfiltered 365 UV light. 4 LEDs powerful uv light, with high power of 50W and 2150mW radiant intensity. More than 70 feet beam distance, easily find what you're looking for (No need to get too close or squat). It also has a USB-C direct charging function, Using rechargeable battery power
- Professional filtered uv 365nm light: Equipped with hard toughened black filter optical lens that can filter out disturbing visible light, and release purer invisible UV365 light, which is suitable for more professional detection needs.