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An Efficient Nano-Catalyzed Green Synthesis, Characterization of Substituted Pyrazoles & Study of Their Fluorescence Characteristics

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ABSTRACT

Different derivatives of Cinnamaldehydeswere prepared by Claisen-Schmidt condensation (by using Strong basic reagent).The prepared Cinnamaldehydes were treated with Hydrazine hydrate in presence of ZnO Nano-catalyst under Microwave assisted solvent-free conditions to afford different substituted Pyrazoles. Green chemistry was employed. Comparisons of both microwave & conventional methods were studied & found that the first was more potent than the later. Compounds feature a donor-π-conjugated acceptor fluorescent activity system, and the fluorescence wavelength maxima were measured in 3 different solvents.The synthesized compounds were characterized by FT-IR,1HNMR& elemental analysis.

Keywords: *Cinnamaldehydes, ZnO Nano-Catalyst, Microwave assisted Synthesis, Absorption maxima, Fluorescence maxima & Fluorescence spectrometer.*

INTRODUCTION

[1,2,3,4]Different substituted Pyrazoles prepared bymicrowave method via Cinnamaldehydes with Hydrazine hydrate. ZnO catalyst with solvent free conditions was applied for both Microwave & conventional methods. Comparison studies were studied & found that microwave method was good in yield of compounds as well as in time completion of reactions than convention (sees table-1). Green synthesis was employed by using Nano-catalyst under solvent free conditions**.** Synthesized compounds exhibited good fluorescence characteristics. Blue shifts & Red shifts occurred by the influence of electro withdrawing groups as well as electron donating groups present on Cinnamaldehydes. Also, we reported shifting of wavelengths of all compounds influenced by three different solvent's polarity.[5,6,7]

We found that wavelengths of fluorescence shifts depend on the types different substituent's of Pyrazoles. Both ERGs & EWGs attached to the phenyl group of Pyrazoles were reported distinct fluorescence & absorbance.

ERGs shifted their electrons to the Π-conjugated phenyl group & then to the Pyrazole ring. Similarly, EWGs accepted electrons from phenyl group which in turn received those from Pyrazole ring.

Instruments Used

Required chemicals were purchased by SDFCL Company & Nano-ZnO was facilitated by Dept. Of Physics, Govt. Science College, Chitradurga. Melting point was determined in open capillary tubes in Buchi B-540 melting point apparatus. The reaction was monitored by thin layer chromatography using silica gel glass plates. The reaction was visualized by short Ultraviolet lamp & isolated in iodine chamber.

FT-IR spectrometer (Vertex series from Bruker), ^{1}HNMR (400MHz) & Thermo Fischer elemental analyzer (BR422710716) were used. Fluorescence spectrometer was used (Source - Xenon Lamp 450W, Range 180-850 nm, Resolution 0.2 nm (maximum at specific wave lengths& Software DATA MAX / GRAMS/31).

EXPERIMENTAL SECTION

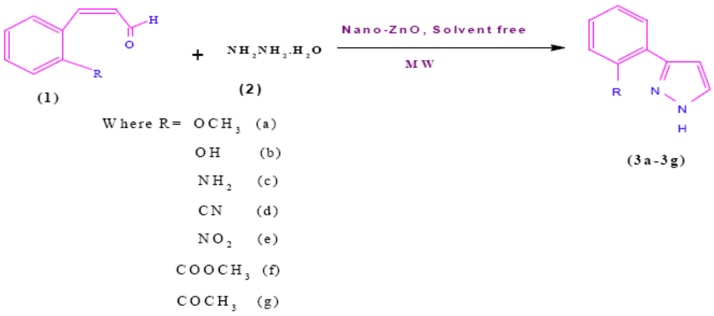
General Procedure for the Synthesis of 2-Substituted-3-Phenyl-1H-Pyrazoles:

To a solution of Cinnamaldehydes (0.01M), 0.5 g of Hydrazine hydrate (0.01M) was added & 2 equiv. of Nano-ZnO was mixed to the solution. The reaction mixture was kept under microwave oven without solvents used **(Scheme.1).** The reaction was monitored by TLC & iodine chamber.

The crude compound along with Nano-ZnO powder was washed with very hot ethanol/water.

The compound obtained was filtered, recrystallized by ethanol & finally dried.

Reaction Scheme



Scheme1. *Synthesis of different {3-(2-substituted)-Phenyl}-1H-Pyrazoles*

3-{(2-methoxy)}-Phenyl-1H-Pyrazole(3a):

Orange solid, %yield= 88.00, m p: 146^0c, IR (KBr): 3412 cm^{-1} (N-H stretch), 3060 cm^{-1}(=C-H, stretch), 2867 cm^{-1} (-CH, stretch), 1175 cm^{-1}(C-O-C), 1212cm^{-1} (C=N, stretch); 2.509-2.527 (CH=N, α to nitrogen), 3.333 (OCH_3), 7.486 -7.644 (Ar-H),.^{1}H NMR (400MHz, DMSO-d_6): δ 2.509- 2.527 (1H, d, CH=N, α to nitrogen), 3.33 (3H, s), 7.45 (1H, d, *J* = 2.5 Hz), 7.57 (1H, ddd, *J* = 8.2, 1.2, 0.5 Hz), 7.58-7.59 (2H, 7.14 (td, *J* = 8.2, 1.5 Hz), 7.6(ddd, *J* = 8.2, 8.0, 1.2 Hz)), 7.61-7.62 (2H, 7.52 (d, *J* = 2.5 Hz), 7.64 (ddd, *J* = 8.0, 1.5, 0.5 Hz), 8.169 - 8.19 (1H, s, N-NH-CH).

Anal.Calcd. For $\mathbf{C_{10}H_{10}N_2O}$(174.00%): C, 68.97; N, 16.1; O, 9.19; H, 5.74; Found: C, 69.14; N, 15.89; O, 9.14; H, 5.83.

3-{(2-hydroxy)}-Phenyl-1H-Pyrazole(3b):

Brown solid, %yield= 94.00, m p: 171^0c IR (KBr): 3432 cm^{-1} (N-H, stretch), 2919 cm^{-1} (-C-H, stretch), 1601 $cm^{-}1$ (C=C, aromatic), 1180 cm^{-1} (C-OH, stretch); HNMR (σ ppm): 1.926-3.335 (-CH=N, α to nitrogen), 4.717 (Ar-OH),^{1}H NMR (400MHz, DMSO-d_6): δ 6.77 (1H, d, *J* = 2.5 Hz), 6.96-6.94 (1H, ddd, *J* = 8.2, 1.5, 1.4 Hz), 7.28 (1H, ddd, *J* = 7.6, 1.5, 1.5 Hz), 7.4 (1H, td, *J* = 1.4, 0.5 Hz), 7.77 (1H, ddd, *J* = 8.2, 7.6, 0.5 Hz), 8.1 (1H, d, *J* = 2.5 Hz)., 9.687(=N-NH). Anal.Calcd. For $\mathbf{C_9H_8N_2O}$(160.00%): C, 67.50; N, 17.5; O, 10.00; H, 5.00; Found: C, 68.14; N, 17.59; O, 9.44; H, 4.83.

3-{(2-amino)}-Phenyl-1H-Pyrazole(3c):

Yellow solid, % yield= 92.00, m p: 149^0c, IR (KBr): 3432 cm^{-1} (N-H, stretch), 3251 cm^{-1} (NH_2,stretch), 3098 cm^{-1} (=C-H, stretch), 1422 $cm^{-}1$ (C=C, aromatic); ^{1}H NMR (400MHz, DMSO-d_6): δ 3.715-3.883 (2H, NH_2), 2.009-2.517 (-CH=N, stretch), 6.82 (1H, d, *J* = 2.5 Hz), 7.04 (1H, td, *J* = 8.1, 1.1 Hz), 7.11 (1H, ddd, *J* = 8.3, 8.1, 1.4 Hz), 7.48(1H, ddd, *J* = 8.3, 1.1, 0.5 Hz), 7.50-7.81 (2H, 7.48 (d, *J* = 2.5 Hz), 7.84 (ddd, *J* = 8.1, 1.4, 0.5 Hz), 8.64 (=CH-NH).

Anal.Calcd. For $\mathbf{C_9H_9N_3}$(159.00%): C, 67.92; N, 26.42; H, 5.66; Found: C, 68.12; N, 26.79; H, 5.09.

3-{(2-cyano)}-Phenyl-1H-Pyrazole(3d):

Yellow solid, %yield= 82.00, m p: 168^0c, IR (KBr): 3390 cm^{-1}(N-H, stretch), 2191 cm^{-1} (CN, stretch), 1597 cm^{-1} (C=C, aromatic).^{1}H NMR (400MHz, DMSO-d_6): δ 2.506 (-CH=N), 7.50 (1H, d, *J* = 2.4 Hz), 7.51-7.54 (2H, 7.58 (ddd, *J* = 8.4, 7.6, 1.6 Hz), 7.8 (d, *J* = 2.4 Hz)), 7.89 (1H, ddd, *J* = 7.6, 1.6, 0.5 Hz), 7.9-7.91 (2H, 7.76 (ddd, *J* = 7.6, 7.6, 1.2 Hz), 7.78 (ddd, *J* = 8.4, 1.2, 0.5 Hz), 8.72 (s, =CH-NH).

Anal.Calcd. For $\mathbf{C_{10}H_9N_3}$(171.00%): C, 70.17; N, 24.56; H, 5.26; Found: C, 71.12; N, 24.69; H, 4.19.

3-{(2-Nitro)}-Phenyl-1H-Pyrazole(3e):

Brown solid, %yield= 89.00, m p: 164^0c, IR (KBr): 3439 cm^{-1} (N-H, stretch), 3081 cm-1 (=C-H), 1630 cm^{-1} (), 1596 cm^{-1} (NO_2).^{1}H NMR (400MHz, DMSO-d_6): δ. 2.503-3.317 (=CH-N),7.18 (1H, d, *J* = 2.1 Hz), 7.33 (1H, ddd, *J* = 8.5, 8.1, 1.6 Hz), 7.49 (1H, d, *J* = 2.1 Hz), 7.62-7.75 (2H, 7.77 (ddd, *J* = 8.1, 7.5, 1.6 Hz), 7.8 (ddd, *J* = 7.5, 1.6, 0.5 Hz)), 8.4(1H, ddd, J = 8.5, 1.6, 0.5 Hz), 9.7 (1H, =CH-NH). Anal.Calcd. For C9H7N3O2(189.00%):

C,57.14; N, 22.23; O, 16.93, H, 3.70; Found: C, 58.02; N, 21.89;O, 16.97 H, 3.2.

3-{(2-methyl-carboxylate}-Phenyl-1H Pyrazole(3f):

Yellow solid, %yield= 79.00. m p: 192^{0}c, IR (KBr): 3333 cm^{-1} (N-H, stretch), 2921 cm^{-1} (-C-H, stretch), 1732 cm^{-1}(C=O, ester).^{1}H NMR (400MHz, DMSO-d$_6$): δ 3.88 (3H, CH$_3$), 6.82 (1H, d, *J* = 2.4 Hz), 7.04 (1H, ddd, *J* = 8.2, 7.7, 1.4 Hz), 7.16 (1H, d, *J* = 2.4 Hz), 7.48-7.50 (2H, 7.77 (ddd, *J* = 7.7, 7.6, 1.3 Hz), 7.81 (ddd, *J* = 7.6, 1.4, 0.4 Hz)), 7.86 (1H, ddd, *J* = 8.2, 1.3, 0.4 Hz), 8.64 (s, =CH-NH). Anal.Calcd. For **$C_{11}H_{10}N_2O_2$**(202.00%): C, 65.34; N, 13.86; O, 15.84, H, 4.96; Found: C, 66.02; N, 14.29; O, 15.27 H, 4.33.

Table4.1. *Details of Comparison for MW & a Convention method*

Compounds	Microwave method	Yield (%)	Convention method	Yield (%)
3a	30 sec	88.00	4 min	74.00
3b	60 sec	94.00	6 min	78.00
3c	45 sec	92.00	7 min	79.00
3d	60 sec	85.00	5 min	82.00
3e	75 sec	89.00	8 min	68.00
3f	40 sec	79.00	6 min	68.00
3g	60 sec	81.00	7 min	72.00

3-{(2-methyl-one)}-Phenyl-1H-Pyrazole (3g): Yellow solid, %yield= 81.00, m p:138^{0}c**,** IR (KBr): 3372 cm^{-1} (-NH, stretch), 3147 cm^{-1} (=CH, stretch),2942 cm-1(-CH, stretch), 1718 cm^{-1} (C=O, stretch), 1605 cm^{-1} =C, r, weak), 1438 cm^{-1} (CH$_3$, bend); ^{1}H NMR (400MHz, DMSO-d$_6$): δ 3.71 (3H, s), 2.315 (=CH-N), 6.82 (1H, d, *J* = 2.4Hz), 7.04 (1H, ddd, *J* = 7.9, 7.7, 1.5 Hz), 7.59-7.65 (2H, 7.64 (d, *J* = 2.4 Hz), 7.62 (ddd, *J* = 7.9, 1.3, 0.4), 7.48-7.51 (2H, 7.76 (td, *J* = 7.7, 1.3 Hz), 7.84 (ddd, *J* = 7.7, 1.5, 0.4 Hz, 8.64 (=CH-NH).

Anal.Calcd. For **$C_{11}H_{10}N_2O$** (186.00%): C,70.97; N, 15.05; O, 8.60, H, 5.37; Found: C, 71.46; N, 15.55; O, 7.78 H, 5.21.

RESULTS & DISCUSSION

Compounds were confirmed by FT-IR , 3043 cm-1 for N-H stretch, 1284, 1344, 1384 & 1441 cm-1 (C-N stretch), 1583 cm-1 (aromatic C=C) & HNMR confirmed by 8.0-11.5 for C-N=NH, 6.3-7.8 (Ar-H) (see data as separate file). Comparisons of Microwave method & Conventional method were studied & found that first one was more efficient in quick reactions & afforded more yield than the later one (See table-4.1

Table4.2. *Data of Absorption spectra & Fluorescence spectra for different substituted Compounds (3a-3g) & their Variations with Solvent polarity.*

Compounds	Solvents		λ_{amax} (nm)		λ_{fmax} (nm)
3a	Hexane		390		422
	Methanol		397		429
	DMSO		400		438
3b	Hexane		392		414
	Methanol		400		421
	DMSO		403		434
3c	Hexane		393		408
	Methanol		402		407
	DMSO		405		432
3d	Hexane		395		398
	Methanol		404		392
	DMSO		406		423
3e	Hexane		393		490
	Methanol		408		493
	DMSO		410		504
3f	Hexane		392		519
	Methanol		403		523
	DMSO		410		539
3g	Hexane		397		548
	Methanol		405		533
	DMSO		408		548

[8]The fluorescence maxima of Compounds (3a-3g) were studied in 3 different solvents [see table-4.2].The variation of fluorescence maxima and absorption maxima is attributed to varying contributions of charge transfer that are dependent on both the identity of the substituent & solvent polarity. As a result, competitive excited-state proton/ charge transfer properties have been observed in the Pyrazoles studied, of which the spectral properties can be fine-tuned by substituents. A small shift towards longer wavelength (4-6nm) was occurred in absorption spectra of 3a-3g. [8,9] [10,11]The increase in wavelength values of absorption spectra depends on solvent polarity (see table-4.2).In fluorescence spectra, there was decrease/ blue shift of wavelengths by ERG of phenyl substituent of Pyrazoles.[12,13] There is subsequent shifts towards shorter wavelengths **λ_{fmax} (nm)**along the vertical line for the compounds (3a-3d), **see Fig. 4.3 & 4.4.**

Electron releasing groups attached to the phenyl group acts as π-conjugated donor & N-atom of Pyrazoles act as π-conjugated acceptor. Because of lone pair of electrons on N-atom, it is difficult to accept π-electrons. Electron withdrawing groups of (3e-3g) shifted towards longer wavelengths **λ_{fmax} (nm)** along the vertical lines**(see Fig. 4.5 & 4.6).**[14,15]The lone pair of electrons on N-atom was excited to the π-conjugated ring of Pyrazoles & then to the π-conjugated phenyl ring. EWGs on phenyl group readily accept those electrons & shifted fluorescence towards longer wavelength.

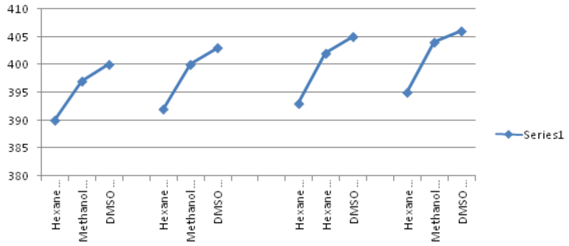


Fig4.3. Absorbance spectra of ERG substituents of Pyrazoles (3a-3d) with solvent polarity of hexane, methanol & DMSO are shown above

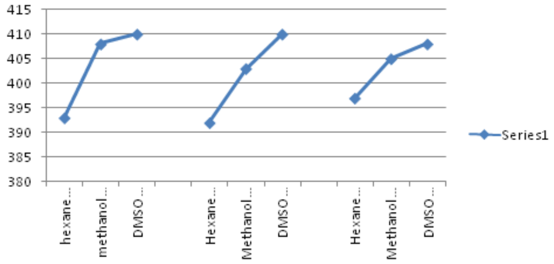


Fig4.4. Absorbance spectra of EWG substituents of Pyrazoles (3e-3g) with solvent polarity of hexane, methanol & DMSO are shown above.

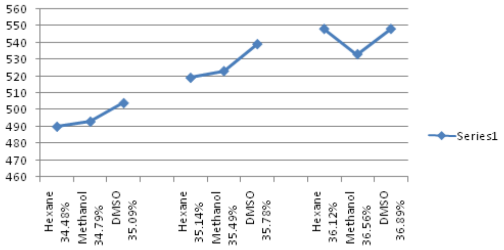


Fig4.5. Fluorescence spectra of EWG substituents of Pyrazoles (3e-3g) with solvent polarity of hexane, methanol & DMSO are shown above.

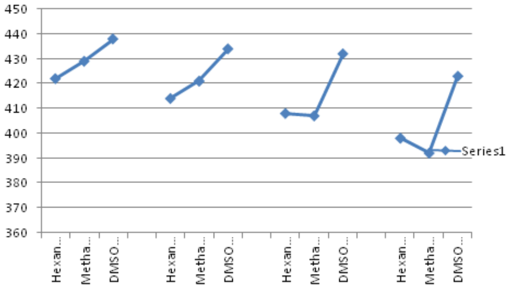


Fig4.6. Fluorescence spectra of ERG substituents of Pyrazoles (3a-3d) with solvent polarity of hexane, methanol & DMSO are shown above.

CONCLUSIONS

We studied that heterocyclic compounds exhibit fluorescence which is basis for the advancement of Optoelectronics. In this review, different derivatives of Pyrazoles showed different fluorescence maxima which made more interesting to study of both EWGs & ERGs were effect in wavelength shifts (Red & Blue shift). Moreover, we followed a microwave method in comparison with Conventional route & justified that the previous one is more potent than the later. Green chemistry, being ecofriendly, was employed to synthesize the compounds.

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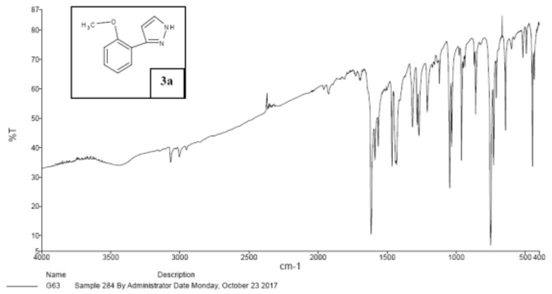
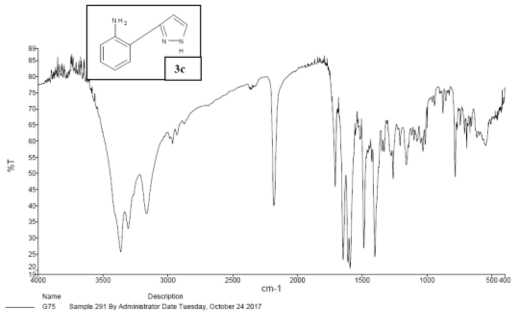
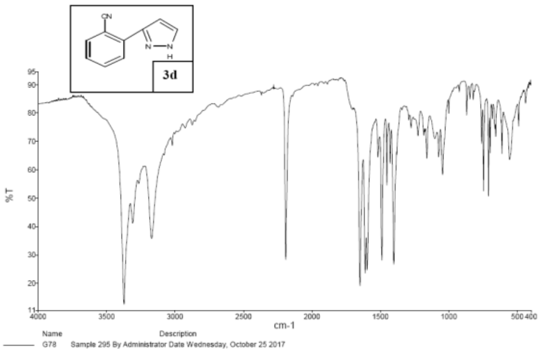
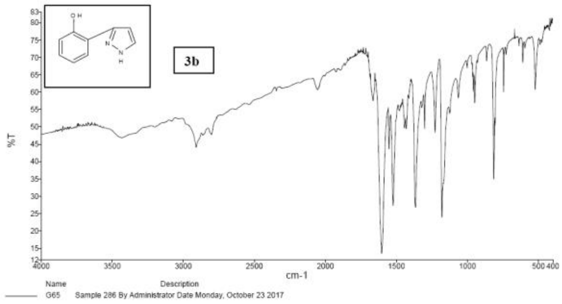
ANALYTICAL DATA

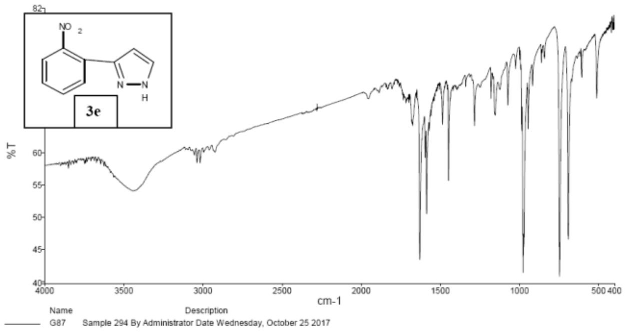
FT-IR Analysis (3a-3g)

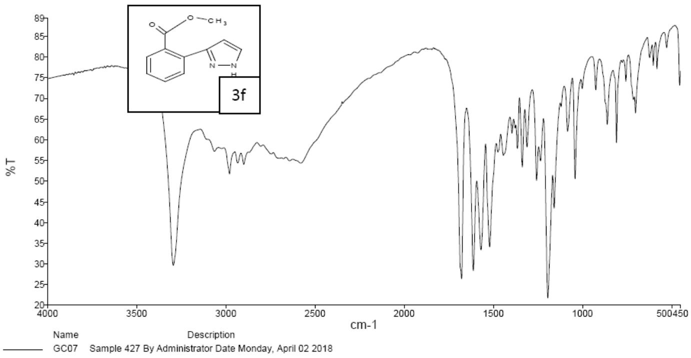


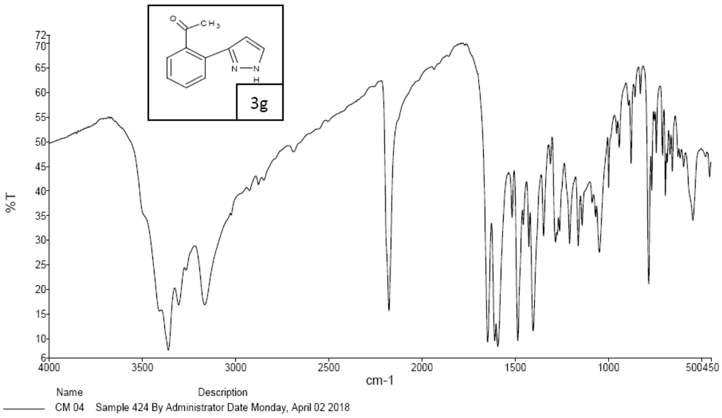




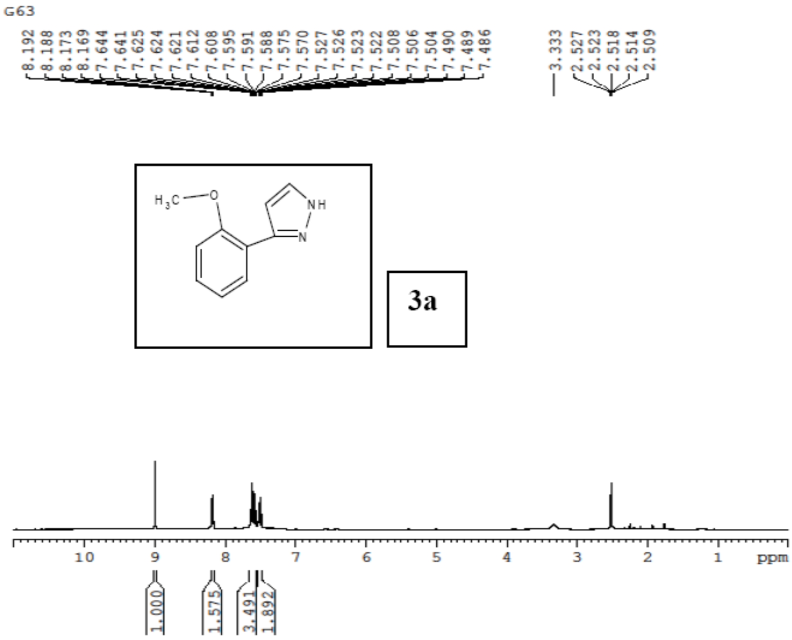








^{1}HNMR Analysis: (3a-3g)



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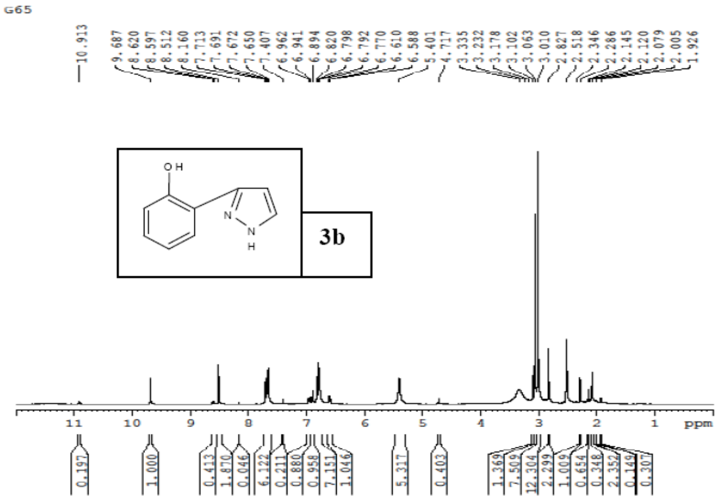

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PC                    1.00
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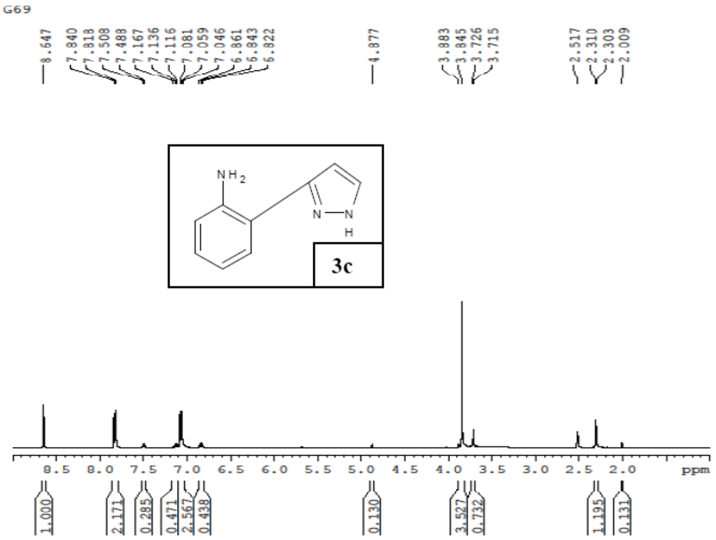
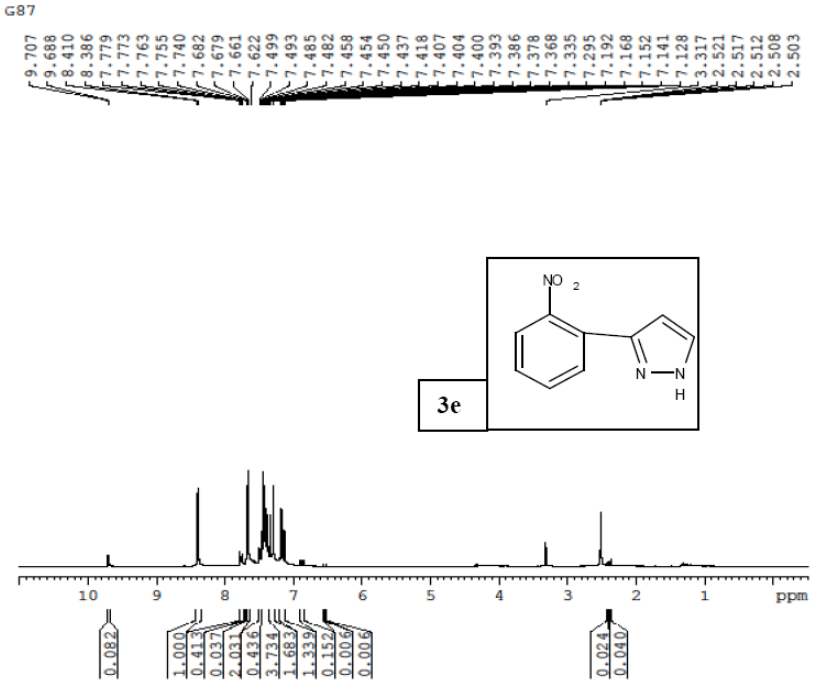


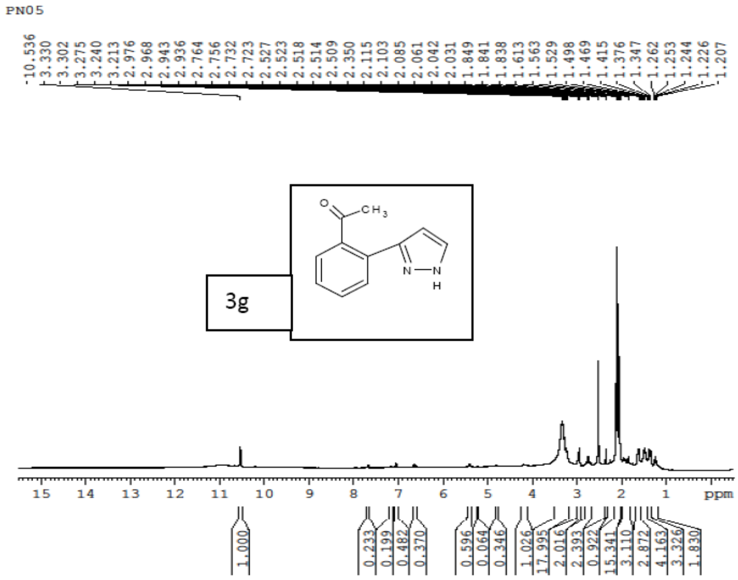
3c

```
Current Data Parameters
NAME             29_ganesh
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20171229
Time                 16.05
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg
TD                   32050
SOLVENT               DMSO
NS                      32
DS                       0
SWH               6410.256 Hz
FIDRES            0.200008 Hz
AQ               2.4999001 sec
RG                     181
DW                  78.000 usec
DE                    6.50 usec
TE                   298.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.50 usec
PL1                  -3.00 dB
PL1W           13.42244530 W
SFO1           400.2330017 MHz

F2 - Processing parameters
SI                  131072
SF             400.2300000 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



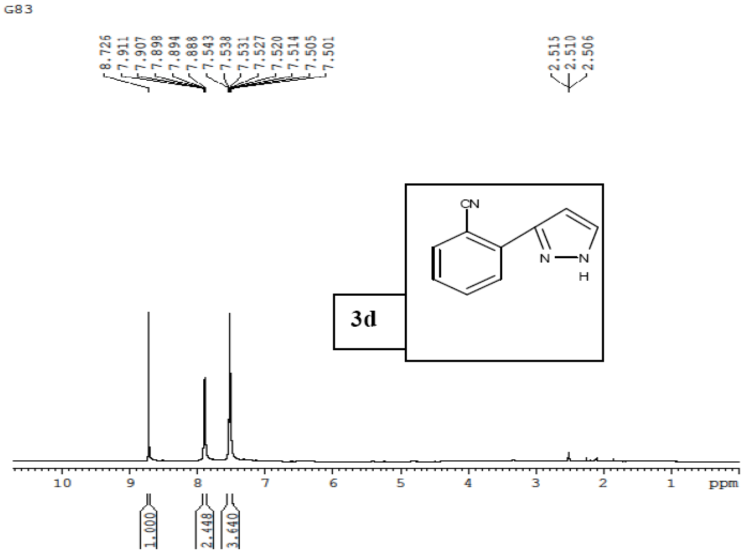
3e

```
Current Data Parameters
NAME             29_ganesh
EXPNO                   10
PROCNO                   2

F2 - Acquisition Parameters
Date_             20171229
Time                 17.00
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg
TD                   32050
SOLVENT               DMSO
NS                      32
DS                       0
SWH               6410.256 Hz
FIDRES            0.200008 Hz
AQ               2.4999001 sec
RG                     203
DW                  78.000 usec
DE                    6.50 usec
TE                   298.1 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.50 usec
PL1                  -3.00 dB
PL1W           13.42244530 W
SFO1           400.2330017 MHz

F2 - Processing parameters
SI                  131072
SF             400.2300022 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



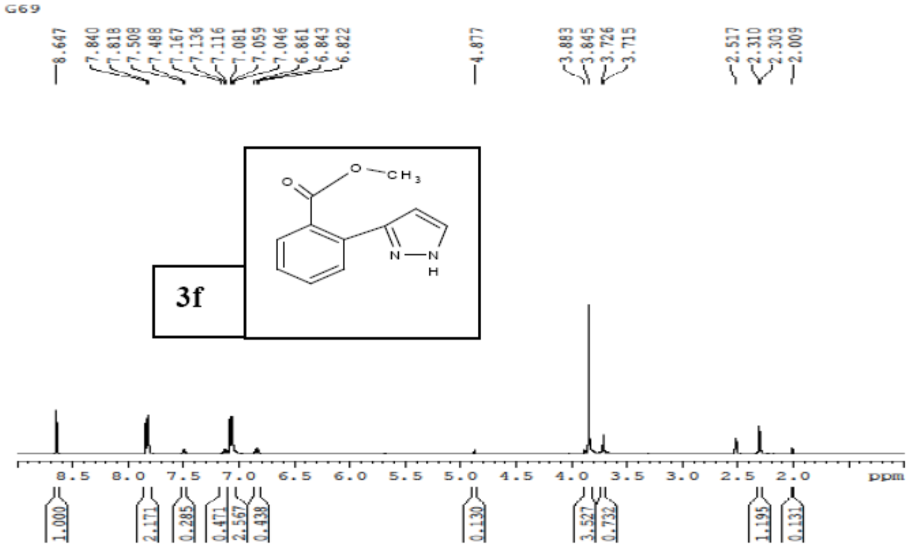
3g

```
Current Data Parameters
NAME            29_ganesh
EXPNO                  12
PROCNO                  2

F2 - Acquisition Parameters
Date_            20171229
Time                17.11
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG                zg
TD                  32050
SOLVENT              DMSO
NS                     32
DS                      0
SWH              6410.256 Hz
FIDRES           0.200008 Hz
AQ              2.4999001 sec
RG                    203
DW                 78.000 usec
DE                   6.50 usec
TE                  298.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.50 usec
PL1                 -3.00 dB
PL1W          13.42244530 W
SFO1          400.2330017 MHz

F2 - Processing parameters
SI                 131072
SF            400.2300000 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



3d

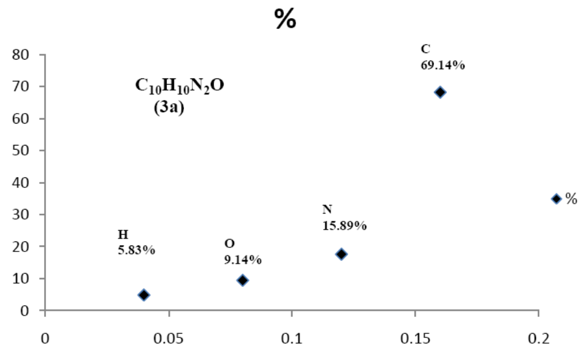
```
Current Data Parameters
NAME            29_ganesh
EXPNO                   9
PROCNO                  2

F2 - Acquisition Parameters
Date_            20171229
Time                16.53
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG                zg
TD                  32050
SOLVENT             CDCl3
NS                     32
DS                      0
SWH              6410.256 Hz
FIDRES           0.200008 Hz
AQ              2.4999001 sec
RG                    101
DW                 78.000 usec
DE                   6.50 usec
TE                  298.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.50 usec
PL1                 -3.00 dB
PL1W          13.42244530 W
SFO1          400.2330017 MHz

F2 - Processing parameters
SI                 131072
SF            400.2319121 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```

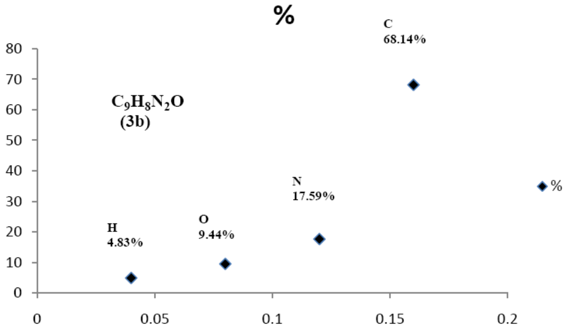


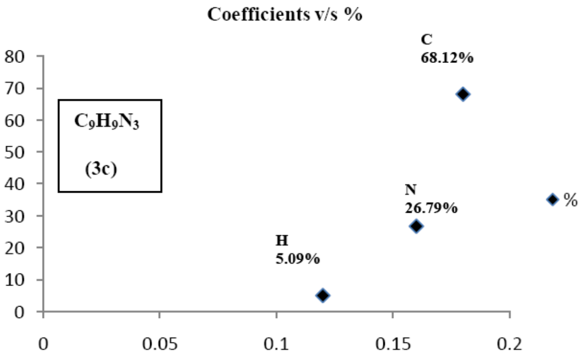
3f

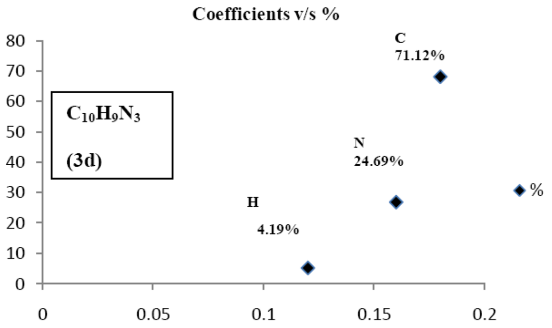
```
Current Data Parameters
NAME            29_ganesh
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20171229
Time                16.05
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG                zg
TD                  32050
SOLVENT              DMSO
NS                     32
DS                      0
SWH              6410.256 Hz
FIDRES           0.200008 Hz
AQ              2.4999001 sec
RG                    181
DW                 78.000 usec
DE                   6.50 usec
TE                  298.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.50 usec
PL1                 -3.00 dB
PL1W          13.42244530 W
SFO1          400.2330017 MHz

F2 - Processing parameters
SI                 131072
SF            400.2300000 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```

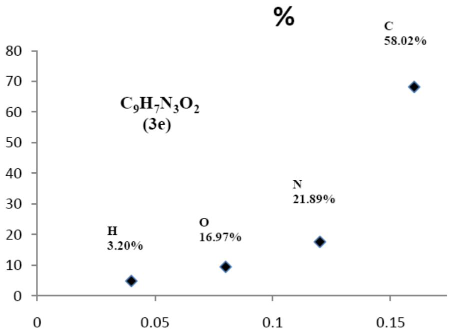
Elemental Analysis

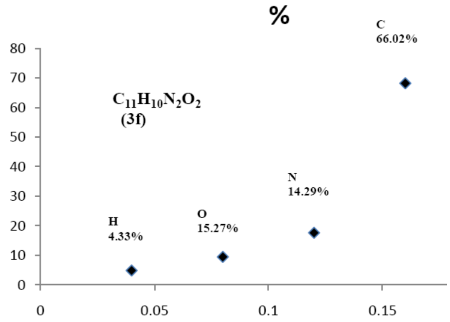












Citation: *Ganesh N Yallappa, D.Nagaraja & U. Chandrashekhara" An Efficient Nano-Catalyzed Green Synthesis, Characterization of Substituted Pyrazoles & Study of Their Fluorescence Characteristics", Research Journal of Nanoscience and Engineering, vol. 2, no. 4, pp.01-09, 2018.*