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A FACILE ONE POT THREE-COMPONENT SYNTHESIS OF PYRAZOLYL NAPHTHOXAZIN-3-ONE DERIVATIVES USING AMBERLITE IRA 400-Cl RESIN

M. Vetriselvan, M. Pramesh✉, S. Jayanthi and K. Gunasundari
PG and Research Department of Chemistry, A.V.V.M. Sri Pushpam College, Poondi, Thanjavur-613503, Tamil Nadu, India.
✉Corresponding Author: mprameshchemistry@gmail.com

ABSTRACT
This report describes a synthesis of pyrazolylnaphthoxazin-3-one by a three-component one-pot reaction of pyrazole aldehyde, β-naphthol, urea/thiourea in the presence of an Amberlite 400Cl resin catalyst using dichloromethane as solvent. The catalyst is easily recoverable and reusable. The synthesized pyrazolylnaphthoxazin-3-one was purified and characterized by various spectral methods such as 1H-NMR, 13C-NMR, HRMass, and IR spectroscopy.
Keywords: Pyrazole aldehyde, β-naphthol, Amberlite IRA 400-Cl Resin, Naphthoxazin-3-one
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INTRODUCTION

Heterocyclic oxazinone core compounds have various therapeutic activities[1]. Due to their improved pharmacological properties, these heterocyclic scaffolds are essential among synthetic organic chemicals[2]. Medications in these proportions are used to cure inflammation, ulcers, high blood pressure, and fungal infections. Therefore, they become an integral part of pharmacologically important heterocyclic compounds[3-7]. The resin now plays a key role as an effective catalyst in synthesizing various heterocyclic compounds. These resins are easy to separate, recycle and reuse, making them an excellent heterogeneous green catalyst[8-12]. Resin catalysts such as Amberlite IRA 402 Cl, Ceralite IRA 410 Cl, ASF terpolymer resin, and Indion GS300 have been used in many organic conversions[13-15]. We present a novel, convenient, and more efficient method for the condensation of β-naphthol, pyrazole aldehyde, and urea in the presence of Amberlite IRA - 400 Cl to give pyrazolylnaphthoxazin-3-one derivatives. [16-18]

EXPERIMENTAL

Chemicals and Reagents

All chemicals were purchased from Sigma Aldrich USA. Analytical TLC was carried out on pre-coated aluminum foils made of CCM Silica Gel 60 F254 silica gel 0.2mm thick (Merck, Germany). Column chromatography was performed on silica gel (100-200 mesh, Merck, India).

Equipment and Analytical Instruments

Melting points were obtained on a Cambridge Instruments Galen III hot stage device. IR spectra were recorded on a Lambda FTIR scientific spectrophotometer. 1H and 13C NMR spectra were recorded DSMO using TMS as internal standard on a BRUKER spectrometer at 400 MHz and 100 MHz, respectively. Mass spectra were recorded on the JEOL GC MATEII HR mass 70 ev E1 spectrometer.

General Procedure for the Synthesis of Naphthoxin-3-one derivatives 4a-j

Pyrazole aldehyde (1.0 mmol), β-Naphthol (1.0 mmol) urea (1.2 mmol), and Amberlite IRA 400Cl anion exchange resin (10 mol%) are added in dichloromethane solvent. The reaction mixture was stirred for an appropriate time at 50 ° C (Table-1). TLC indicated completion of the reaction. The mixture was extracted with ethyl acetate; the catalyst was removed by filtration. The filtrate was concentrated and the residue was column chromatographed with 50% ethyl acetate-hexane to obtain pure pyrazolylnaphthoxazin-3-one in excellent yields.

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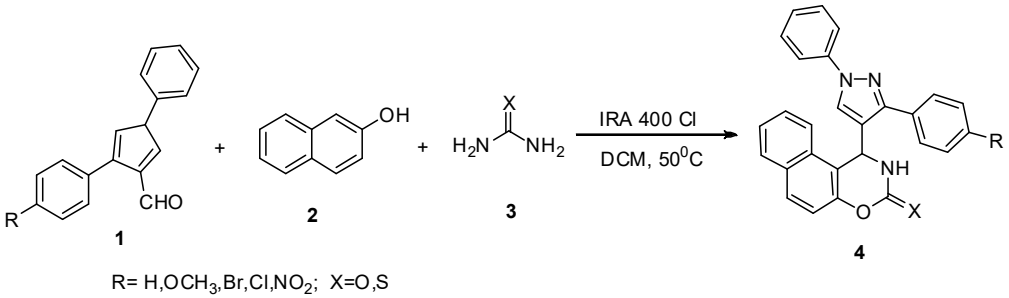
RESULTS AND DISCUSSION

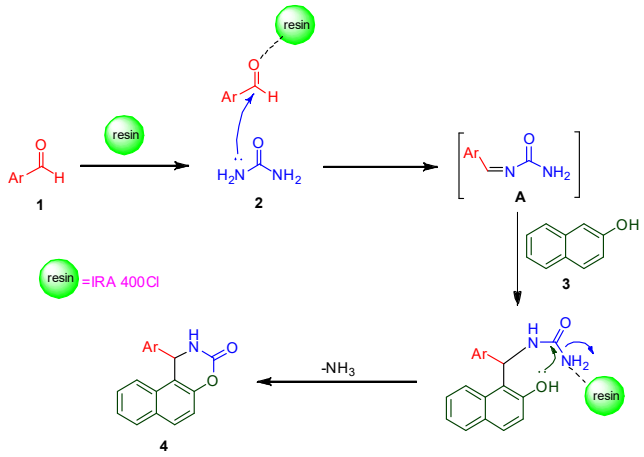
A mixture of pyrazole aldehyde 1, β-naphthol 2, urea 3, and Amberlite IRA 400Cl resin was mixed with DCM and the mixture was stirred for 2 hours. The mixture was then extracted with ethyl acetate and column chromatographed with a 50% ethyl acetate-hexane mixture to yield pyrazolylnaphthoxazin3one in good to excellent yields (Scheme-1). The structures of the products were thoroughly characterized using various spectroscopic techniques, such as 1Hand 13C-NMR spectroscopy and mass spectroscopy. The results are summarized in Table-3.



Scheme-1: Synthesis of Pyrazolyl naphthoxazinone (4a-j)

Mechanism of the Reaction

A plausible mechanism for forming naphthoxazin3ones (4) in the presence of Amberlite IRA400 Cl resin is suggested in Scheme-2. The reaction initially proceeds through acylimine intermediate (A) formed in situ by the reaction of aldehyde (1) with urea (2) in the presence of Amberlite IRA400 Cl resin. Then, the acylimine (A) intermediate reacts with *β*-naphthol (3) with cyclization and subsequent elimination of ammonia to give the product (4).



Scheme-2: Plausible Mechanism

Table-1: Screening of Catalyst

Entry	Catalyst (10 mol %)	Time (h)	Yield[a] %
1	Amberlite IRA 96	24	Trace
2	Amberlite HPR 4200 Cl	12	30
3	Amberlite IRA 402 Cl	8	60
4	Amberlite IRA 400 Cl	2	85
5	Amberlite IRA 900 Cl	8	55
6	Ceralite IRA 410 Cl	6	70
7	Indion GS-300	12	25

[a]Isolated yield.

We examined the effect of various catalysts for the synthesis of pyrazolyl naphthoxazin-3-one from pyrazole aldehyde, urea and β-naphthol. After the careful, systematic screening (Table-1), Amberlite IRA 400 Cl was found to be the catalyst of choice.

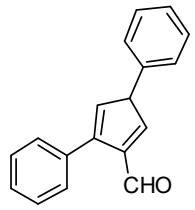
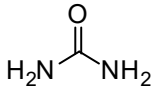
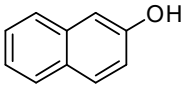
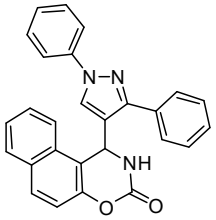
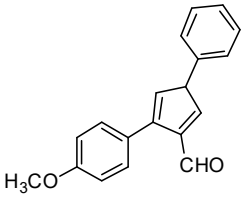
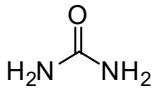
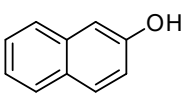
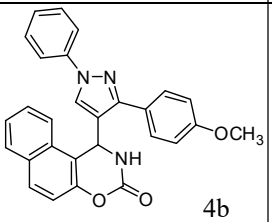
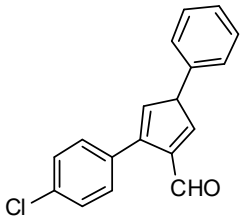
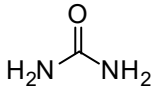
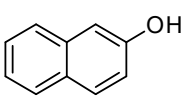
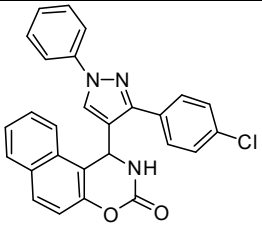
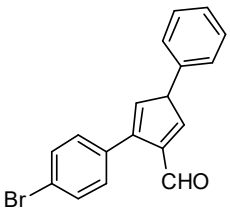
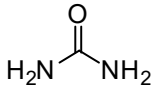
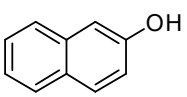
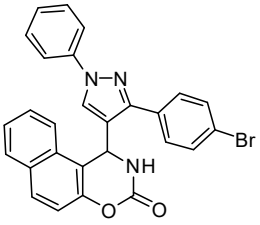
Table-2: Recycling and Reusability of the Catalyst

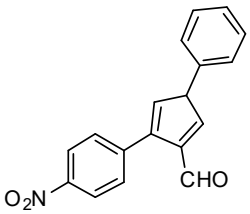
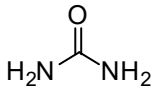
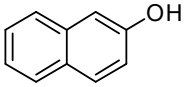
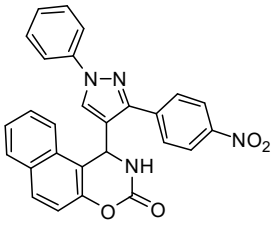
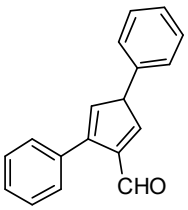
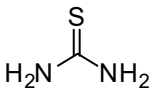
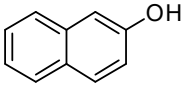
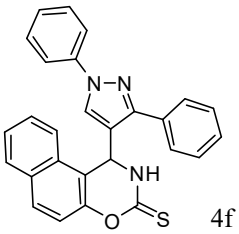
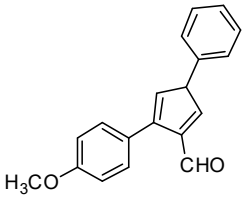
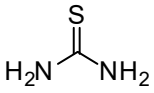
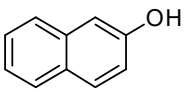
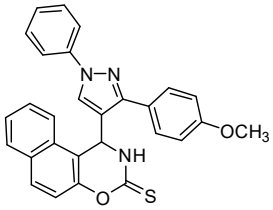
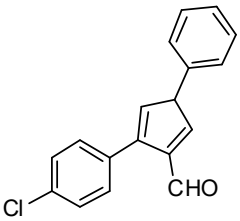
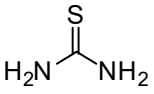
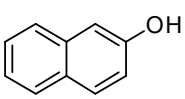
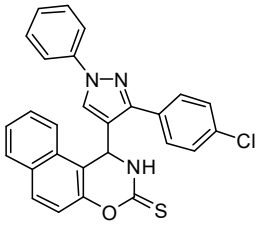
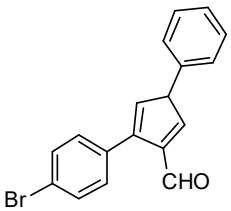
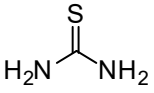
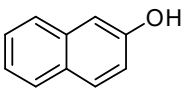
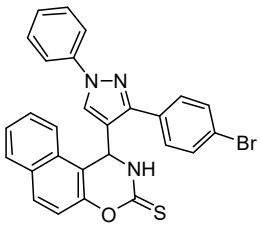
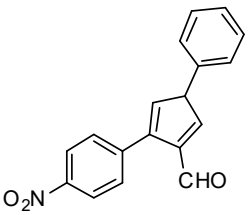
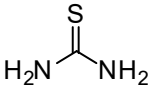
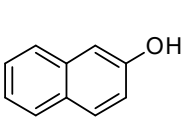
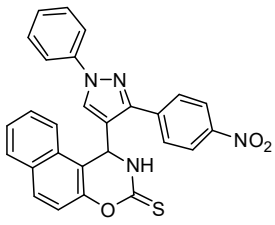
Entry	Run	Time (h)	Yield[a] %
1	1st	2	85
2	2nd	2	85
3	3rd	2	85
4	4th	2	80
5	5th	2	78

[a]Isolated yield.

We have studied the reusability of Amberlite IRA 400-Cl resin catalyst using similar reactants. The catalyst recovered from the 1st run can be reused up to three runs, but on further use, the catalytic activity decreased.

Table-3: Synthesis of Pyrazolyl naphthoxazinone (4a-j)

Entry	Pyrazole aldehyde (1)	Urea / Thio urea (2)	β – Naphthol (3)	Product (4)[a]	Time (h)	Yield[b] (%)
1				 4a	2	85
2				 4b	2.5	88
3				 4c	2	82
4				 4d	2	80

5				 4e	3	76
6				 4f	2	80
7				 4g	3	78
8				 4h	2.5	86
9				 4i	2.5	82
10				 4j	3	80

[a]All products were characterized by IR, [1]HNMR, [13]CNMR and Mass spectroscopy
[b]Isolated yield after column chromatography.

The [1]H NMR spectrum of compound 4a exhibited a one proton singlet at 8.22ppm, clearly shows that the presence of –NH group. The one proton singlet at 5.73ppm was presented to a naphthoxazinone ring (-

CH). The singlet at 7.72ppm was assigned to pyrazole ring proton. The 10.16ppm one proton singlet was attributed to naphthalene ring 8th carbon. The range of 7.80-7.06ppm was assigned to aromatic proton.
In the ^{13}C NMR spectra, the peak at 42.0ppm was attributed to methain carbon. The peak appeared at 124.0, 133.1 and 150.1ppm to a pyrazole ring carbon. The peak at 158ppm showed carbonyl carbon in naphthoxazinone ring. The range between 118-152ppm was assigned to aromatic carbon. The mass spectrum reveals the molecular ion peak $[M]^+$ at m/z 417.1499.

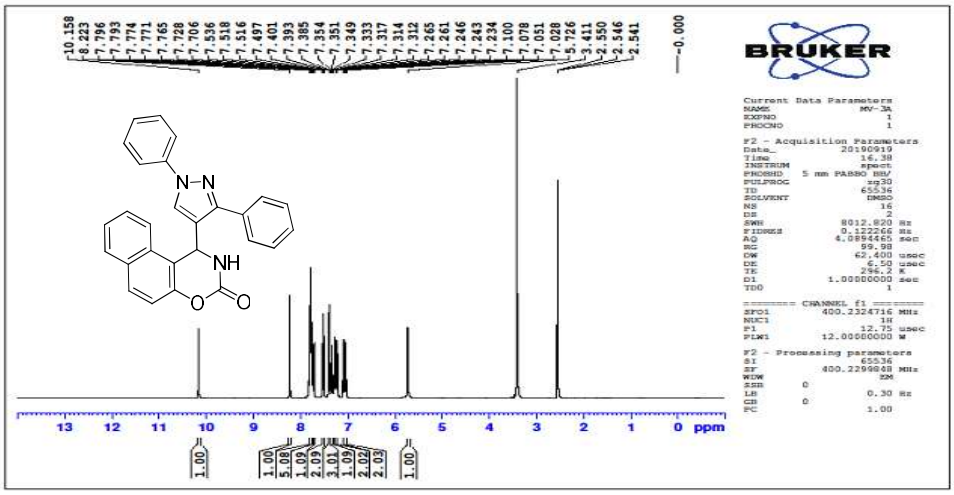


Fig.-1: ^1H NMR Spectrum of Compound 4a

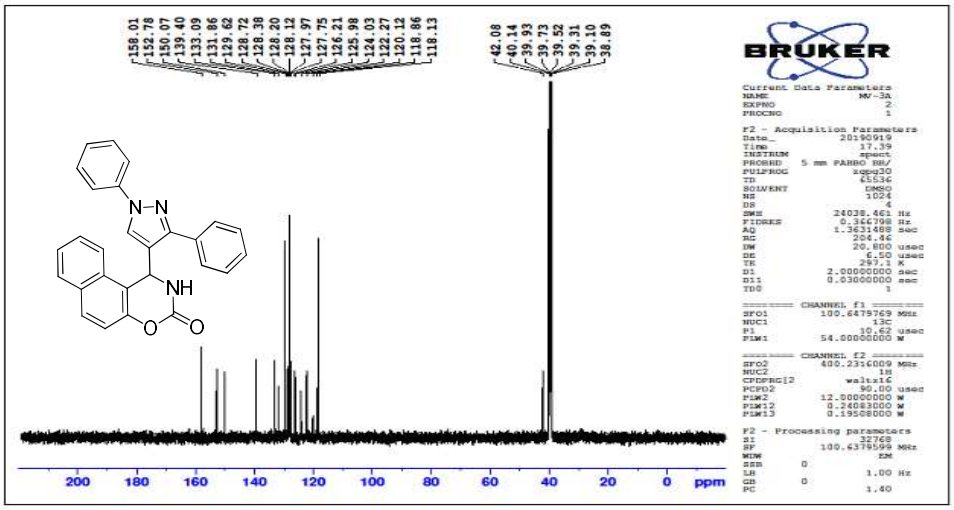


Fig.-2: ^{13}C NMR Spectrum of Compound 4a

Characterization of Compounds 4a-j

Compound 4a

1-(1,3-diphenyl-1H-pyrazol-4-yl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one, White powder; 85% yield; R_f (50% EtOAc-Hexane): 0.46, mp:248-252°C; ^1H NMR (400MHz, DMSO-D_6): δ 10.16 (s, 1H), 8.22 (s, 1H), 7.80 – 7.76 (m, 5H), 7.72 (s, 1H), 7.52 (dd, *J* = 8.6, 7.4 Hz, 2H), 7.39 (dd, *J* = 5.2, 2.1 Hz, 3H), 7.36 – 7.31 (m, 1H), 7.27 – 7.22 (m, 2H), 7.06 (q, *J* = 9.1 Hz, 2H), 5.73 (s, 1H).^{13}C NMR (100MHz, DMSO-D_6): δ 158.0, 152.8, 150.1, 139.4, 133.1, 131.9, 129.6, 128.7, 128.4, 128.2, 128.1, 128.0, 127.8, 126.2, 126.0, 124.0, 122.3, 120.1, 118.9, 118.1, 42.1; IR (KBr) ν_{max}: 3475, 3396, 3334, 3062, 2919, 2708, 1598, 1548, 1529, 1515, 1451, 1349 cm^{-1};HRMS-ESI $[M]^+$: m/z 417.1499.

Compound 4b

1-(3-(4-methoxyphenyl)-1-phenyl-1H-pyrazol-4-yl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one, yellow powder; 88% yield; R_f (50% EtOAc-Hexane): 0.52, mp: 257-261°C; ^{1}H NMR (400MHz, DMSO-D_6): δ 10.14 (s, 1H), 8.16 (s, 1H), 7.80 (s, 1H), 7.74 – 7.70 (m, 4H), 7.68 – 7.66 (m, 2H), 7.43 (dd, *J* = 8.2, 7.4 Hz, 2H), 7.36 – 7.14 (m, 4H), 7.04 (d, *J* = 8.5 Hz, 2H), 5.74 (s, 1H), 3.80 (s, 3H).^{13}C NMR (100MHz, DMSO-D_6): δ 160.2, 157.5, 151.8, 149.4, 139.8, 133.2, 129.3, 129.0, 128.8, 128.4, 128.0, 126.3, 126.0, 125.3, 123.4, 123.1, 119.7, 119.4, 117.5, 115.2, 114.6, 55.4, 42.2; IR (KBr) ν_{max}: 3460, 3372, 3320, 3018, 2902, 2708, 1596, 1540, 1510, 1460, 1344 cm^{-1};HRMS-ESI M]$^+$: m/z447.1598.

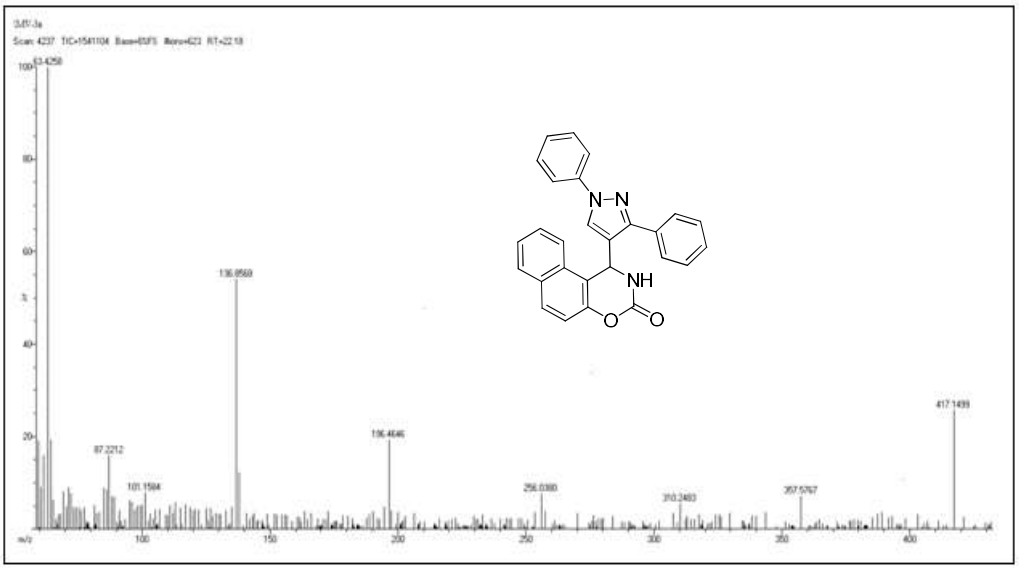


Fig.-3: HR Mass Spectrum of Compound 4a

Compound 4c

1-(3-(4-chlorophenyl)-1-phenyl-1H-pyrazol-4-yl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one, white powder; 82% yield; R_f (50% EtOAc-Hexane): 0.56, mp: 252-256°C; ^{1}H NMR (400MHz, DMSO-D_6): δ10.10 (s, 1H), 8.14 (s, 1H), 7.82 (s, 1H), 7.76 – 7.72 (m, 4H), 7.70 – 7.67 (m, 2H), 7.47 (dd, *J* = 8.6, 7.4 Hz, 2H), 7.36 (d, *J* = 8.5 Hz, 2H), 7.22 (ddd, *J* = 7.9, 6.8, 1.1 Hz, 1H), 7.17 (d, *J* = 8.8 Hz, 1H), 7.03 – 6.96 (m, 2H), 5.68 (s, 1H).^{13}C NMR (100MHz, DMSO-D_6): δ 158.1, 152.8, 148.9, 139.3, 132.4, 132.0, 131.8, 129.6, 128.8, 128.4, 128.2, 128.0, 126.3, 126.1, 124.2, 122.3, 119.7, 118.8, 118.2, 42.0; IR (KBr) ν_{max}: 3480, 3355, 3062, 2708, 2607, 1646, 1597, 1535, 1503, 1448, 1349 cm^{-1};HRMS-ESI [M]$^+$: m/z 451.1099.

Compound 4d

1-(3-(4-bromophenyl)-1-phenyl-1H-pyrazol-4-yl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one, brown powder; 80% yield; R_f (50% EtOAc-Hexane): 0.54, mp: 263-268 °C; ^{1}H NMR (400MHz, DMSO-D_6): δ 10.16 (s, 1H), 8.20 (s, 1H), 7.78 (s, 1H), 7.76 – 7.72 (m, 4H), 7.64 – 7.62 (m, 2H), 7.48 (dd, *J* = 7.8, 7.2 Hz, 2H), 7.34 – 7.16 (m, 4H), 7.08 (d, *J* = 8.1 Hz, 2H), 5.80 (s, 1H).^{13}C NMR (100MHz, DMSO-D_6): δ 158.2, 152.4, 151.0, 139.6, 132.9, 132.4, 132.0 131.5, 131.0, 129.4, 129.1, 128.8, 128.4, 128.2, 128.0, 126.8, 126.3, 123.5, 123.2, 120.4, 119.2, 117.6, 116.3, 42.4; IR (KBr) ν_{max}: 3478, 3346, 3055, 2712, 2606, 1654, 1597, 1540, 1501, 1449, 1355 cm^{-1};HRMS-ESI [M]$^+$: m/z495.0594.

Compound 4e

1-(3-(4-nitrophenyl)-1-phenyl-1H-pyrazol-4-yl)-1H-naphtho[1,2-e][1,3]oxazin-3(2H)-one, yellow solid; 76% yield; R_f (50% EtOAc-Hexane): 0.48, mp: 224-228°C; ^{1}H NMR (400MHz, DMSO-D_6): δ 10.12 (s, 1H), 8.18 (s, 1H), 7.82 (s, 1H), 7.78 – 7.74 (m, 4H), 7.66 – 7.64 (m, 2H), 7.42 (dd, *J* = 8.2, 7.4 Hz, 2H), 7.33 – 7.18 (m, 4H), 7.04 (d, *J* = 8.2 Hz, 2H), 5.74 (s, 1H).^{13}C NMR (100MHz, DMSO-D_6): δ 158.3, 151.5, 151.2, 149.1, 139.8, 139.2, 133.8, 129.4, 129.1, 128.6, 128.4, 128.0, 126.8, 126.4, 126.1, 124.7,

124.3, 123.8, 123.2, 123.0, 119.8, 119.5, 118.7, 118.5, 118.2, 43.1; IR (KBr) ν_{max}: 3464, 3352, 3055, 2706, 2610, 1654,1597, 1554, 1534, 1437, 1368, 1339 cm^{-1};HRMS-ESI $[M]^+$: m/z 462.1338.

Compound 4f

1-(1,3-diphenyl-1H-pyrazol-4-yl)-1H-naphtho[1,2-e][1,3]oxazine-3(2H)-thione, pale yellow solid; 80% yield; R_f(50% EtOAc-Hexane): 0.44, mp: 218-223 ºC; ^{1}H NMR (400MHz, DMSO-D_6): δ 10.16 (s, 1H), 8.22 (s, 1H), 7.80 – 7.76 (m, 4H), 7.72 (d, *J* = 8.8 Hz, 1H), 7.54 (dd, *J* = 8.6, 7.4 Hz, 2H), 7.42 (dd, *J* = 5.2, 2.1 Hz, 2H), 7.38 – 7.33 (m, 2H), 7.27 – 7.06 (m, 4H), 5.78 (s, 1H), 2.14 (s, 1H).^{13}C NMR (100MHz, DMSO-D_6): δ 152.3, 150.1, 147.3, 139.8, 133.7, 133.1, 129.6, 129.3, 129.1, 128.8, 128.6, 128.3, 128.0, 127.5, 127.3, 126.6, 126.4, 123,8, 123.4, 123.0, 119.9, 119.6, 118.8, 118.5, 117.1, 43.4; IR (KBr) ν_{max}: 3478, 3355, 3068, 2706, 2608, 2074, 1655, 1580, 1468, 1444, 1335 cm^{-1};HRMS-ESI$[M]^+$: m/z433.1257.

Compound 4g

1-(3-(4-methoxyphenyl)-1-phenyl-1H-pyrazol-4-yl)-1H-naphtho[1,2-e][1,3]oxazine-3(2H)-thione, yellow solid; 78% yield; R_f (50% EtOAc-Hexane): 0.48, mp: 243-248ºC; ^{1}H NMR (400MHz, DMSO-D_6): δ 10.18 (s, 1H), 8.22 (s, 1H), 7.80 (s, 1H), 7.74 – 7.70 (m, 4H), 7.66 – 7.64 (m, 2H), 7.44 (dd, *J* = 8.1, 7.6 Hz, 2H), 7.34 – 7.12 (m, 4H), 7.04 (d, *J* = 7.9 Hz, 1H), 5.82 (s, 1H), 3.78 (s, 3H), 2.16 (s, 1H).^{13}C NMR (100MHz, DMSO-D_6): δ 161.6, 152.1, 150.3, 147.1, 139.8, 133.7, 131.1, 129.8, 128.7, 128.5, 128.3, 128.0, 126.4, 125.7, 123.6, 123.3, 123.0, 119.7, 119.3, 118.9, 118.6, 117.3, 115.9, 55.4, 42.9; IR (KBr) ν_{max}: 3480, 3374, 3072, 2714, 2604, 2068, 1644, 1574, 1488, 1434, 1340 cm^{-1};HRMS-ESI$[M]^+$: m/z 463.1364.

Compound 4h

1-(3-(4-chlorophenyl)-1-phenyl-1H-pyrazol-4-yl)-1H-naphtho[1,2-e][1,3]oxazine-3(2H)-thione, white powder; 86% yield; R_f(50% EtOAc-Hexane): 0.52, mp: 234-239 ºC; ^{1}H NMR (400MHz, DMSO-D_6): δ 10.10 (s, 1H), 8.16 (s, 1H), 7.82 (s, 1H), 7.78 – 7.74 (m, 3H), 7.68 – 7.65 (m, 2H), 7.44 (dd, *J* = 8.4, 7.6 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.22 – 6.96 (m, 4H), 5.78 (s, 1H), 2.24 (s, 1H).^{13}C NMR (100MHz, DMSO-D_6): δ 151.9, 150.1, 147.2, 140.1, 134.4, 133.5, 131.4, 129.8, 129.6, 129.4, 129.1, 128.8, 128.4 128.1, 126.5, 126.3, 123.8, 123.2, 123.0, 119.9, 119.6, 118.8, 118.1, 117.6, 43.1; IR (KBr) ν_{max}: 3477, 3350, 3044, 2724, 2618, 2050, 1634, 1510, 1422, 1408, 1315 cm^{-1};HRMS-ESI$[M]^+$: m/z 467.0867.

Compound 4i

1-(3-(4-bromophenyl)-1-phenyl-1H-pyrazol-4-yl)-1H-naphtho[1,2-e][1,3]oxazine-3(2H)-thione, brown solid; 82% yield; R_f (50% EtOAc-Hexane): 0.44, mp: 212-217 ºC; ^{1}H NMR (400MHz, DMSO-D_6): δ 10.16 (s, 1H), 8.20 (s, 1H), 7.76 (s, 1H), 7.74 – 7.70 (m, 4H), 7.66 – 7.64 (m, 2H), 7.48 (dd, *J* = 8.1, 7.4 Hz, 2H), 7.34 – 7.16 (m, 3H), 7.08 (d, *J* = 7.8 Hz, 2H), 5.73 (s, 1H), 2.16 (s, 1H).^{13}C NMR (100MHz, DMSO-D_6): δ 152.4, 150.1, 146.8, 139.7, 133.5, 132.4, 132.0, 129.6, 129.1, 128.8, 128.6, 128.4, 126.3, 126.2, 123.8, 123.3, 123.0, 119.9, 118.8, 118.2, 117.4, 44.1; IR (KBr) ν_{max}: 3478, 3344, 3036, 2706, 2608, 2045, 1674, 1597, 1462, 1418, 1345 cm^{-1};HRMS-ESI$[M]^+$: m/z 511.0368.

Compound 4j

1-(3-(4-nitrophenyl)-1-phenyl-1H-pyrazol-4-yl)-1H-naphtho[1,2-e][1,3]oxazine-3(2H)-thione**,**pale yellow solid; 80% yield; R_f (50% EtOAc-Hexane): 0.46, mp: 204-210ºC; ^{1}H NMR (400MHz, DMSO-D_6): δ 10.12 (s, 1H), 8.22 (s, 1H), 7.80 (s, 1H), 7.78 – 7.74 (m, 4H), 7.68 – 7.66 (m, 2H), 7.44 (dd, *J* = 8.1, 7.6 Hz, 2H), 7.34 – 7.18 (m, 3H), 7.06 (d, *J* = 7.9 Hz, 2H), 5.74 (s, 1H), 2.24 (s, 1H).^{13}C NMR (100MHz, DMSO-D_6): δ 152.5, 151.2, 139.8, 139.2, 133.8, 129.4, 129.1, 128.6, 128.4, 128.0, 126.8, 126.4, 126.1, 124.7, 124.3, 123.8, 123.2, 123.0, 119.8, 119.5, 118.7, 118.5, 118.2, 43.1; IR (KBr) ν_{max}: 3484, 3366, 3056, 2708, 2610, 2040, 1674, 1594, 1472, 1422, 1368, 1335 cm^{-1};HRMS-ESI $[M]^+$: m/z 478.1122.

CONCLUSION

We demonstrate a three-component, one-pot reaction to synthesize a biologically important derivative of naphthoxazin3one from substituted pyrazole aldehyde, β-naphthol and urea/thiourea in DCM using the

Amberlite IRA 400Cl resin catalyst. This method offers several advantages such as mild reaction conditions, shorter reaction times, absence of toxic by-products, good yield, and simple experimental and isolation procedures, making it a methodical route for synthesizing naphthoxazin3one derivatives.

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