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## RECENT ACHIEVEMENTS IN THE SYNTHESIS OF HETEROCYCLIC COMPOUNDS BY PHTHALHYDRAZIDE-BASED MULTICOMPONENT REACTIONS

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**Abstract** – Phthalhydrazide is one of the useful starting material toward the synthesis of heterocyclic compounds, and considered as a valuable structural unit in organic synthesis due to its significant pharmacological and biological activities. There are many multicomponent reactions using phthalhydrazide under different conditions. This review article aims to give an overview of recent applications of phthalhydrazide in the multicomponent reactions during the period of 2006 to 2021.

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### 1. INTRODUCTION

In the last few decades, the synthesis of heterocyclic compounds containing nitrogen atoms in their ring system has become a subject of great interest, due to their widespread applications in organic synthesis and pharmaceutical chemistry.<sup>1-3</sup> Among a wide variety of *N*-heterocyclic compounds, heterocycles containing a phthalazine moiety with two nitrogen bridgehead atoms in a fused ring structure have gained considerable attention, because of their biological and pharmacological activities.<sup>4-6</sup> Phthalhydrazide **1** (2,3-dihydro-1,4-phthalazinedione) with two NH-nucleophilic groups is one the most widely used starting materials for the synthesis of phthalazine derivatives through multicomponent reactions. It has cytotoxic,<sup>7</sup>

antimicrobial,<sup>8</sup> anticonvulsant,<sup>9</sup> antifungal,<sup>10</sup> anticancer,<sup>11</sup> and anti-inflammatory activities.<sup>12</sup> Phthalazine-containing compounds are also very strong inhibitors of vascular endothelial growth factor receptor II (VEGFR-2).<sup>13</sup> Moreover, a series of tetrapeptide phthalhydrazide ketones like compound **5** are effective as SARS 3C-like protease (3CL<sup>PRO</sup>) inhibitors, and the phthalhydrazide and  $\gamma$ -lactam moieties are important structural factors for good inhibition.<sup>14</sup> Luminol **3** is another phthalhydrazide derivative, which is used as a potent antineoplastic agent and possesses hypolipidemic activities as shown in Figure 1.<sup>15,16</sup>

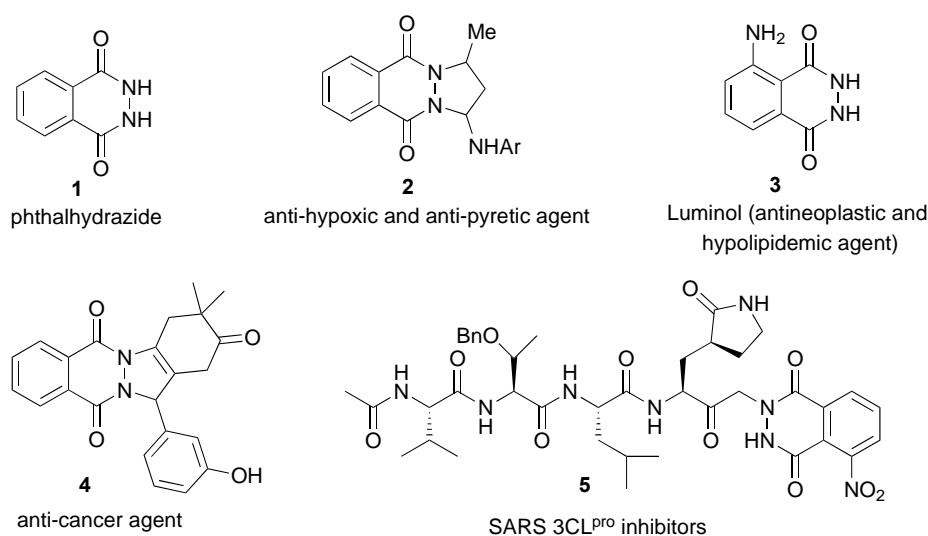


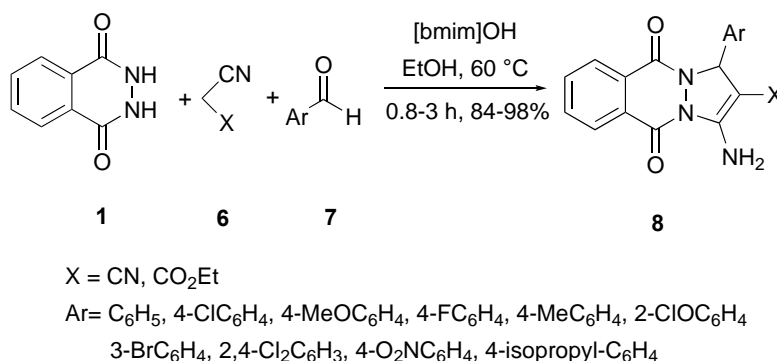
Figure 1. Examples of bioactive compounds containing phthalazine derivatives

These compounds also could be utilized as interesting materials for new fluorescence or luminescence probes.<sup>17</sup> One of the important tasks in organic synthesis is the creation of simple synthetic pathways for complex organic molecules from readily available reagents. Multicomponent reactions (MCRs) are the critical approach for the rapid and successful synthesis of heterocyclic compounds.<sup>18,19</sup> The MCR method is ideal for any type of synthesis, due to its ease of operation, low cost of product separation and purification, high yield and selectivity by the use of minimum synthetic starting materials. Therefore, synthetic strategies involving MCRs are valuable and powerful tools for the efficient and rapid synthesis of a wide variety of organic compounds.<sup>20,21</sup> In continuation of our previous studies on the synthesis of heterocyclic compounds and in MCRs,<sup>22-35</sup> it was decided to study the application of phthalhydrazide as a starting material in the MCRs based on different heterocyclic compounds.

## 2. THE SYNTHESIS OF PYRAZOLOPHthalAZINES

Guo and co-workers described a three-component reaction for the synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives **8** from phthalhydrazide **1** with malononitrile or ethyl cyanoacetate **6** and aromatic aldehydes **7** using of 1-butyl-3-methylimidazolium hydroxide

([bmim]OH) as catalyst in EtOH (Scheme 1).<sup>36</sup> This method applied on different aromatic aldehydes, which were shown higher reactivity with electron-withdrawing groups.



Scheme 1. The synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives **8**

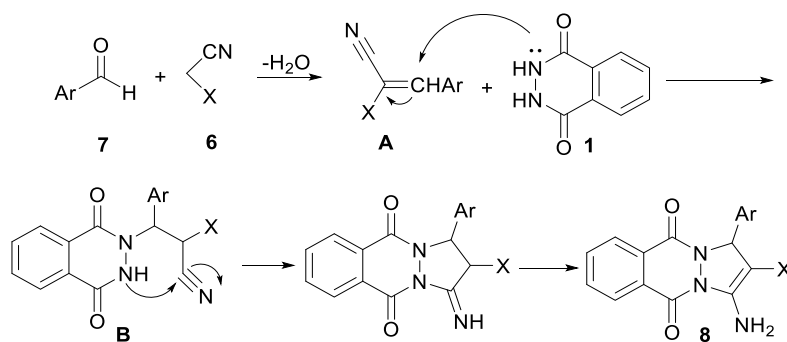
This reaction was also performed in the presence of different catalysts and under various conditions as shown in Table 1.

Table 1. The synthesis of products **8** under different conditions

Entry	Solvent	Catalyst	Condition	Time (min)	Yield (%)	Ref.
1	EtOH	[Bmim]OH	60 °C	0.8-3 h	84-98	36
2	—	Nano-ZnO	100 °C	8-45	86-93	37
3	glycerol	—	80 °C	45-75	85-94	38
4	—	NaHCO <sub>3</sub>	120 °C	15-50	85-98	39
5	EtOH	Cu(OTf) <sub>2</sub>	80 °C	120-180	86-93	40
6	—	SBA@BiPy <sup>2+</sup> 2Cl <sup>-</sup>	100 °C	30-60	86-96	41
7	[bmim]Br	<i>p</i> -toluenesulfonic acid	100 °C	2-5 h	73-97	42
8	EtOH	RH@[SiPrDABCO@BuSO <sub>3</sub> H]HS O <sub>4</sub>	reflux	3-5 h	84-94	43
9	—	TBBDA	100 °C	10-20	83-90	44
10	—	PBBS	100 °C	10-50	64-85	44
11	EtOH	Al-KIT-6	60 °C	240	79-93	45
12	—	CuFe <sub>2</sub> O <sub>4</sub> @SiO <sub>2</sub>	120 °C	10-15	88-95	46
13	—	[SiPMIM]OH@MNPs	rt	10-20	82-95	47
14	<i>n</i> -PrOH	Electrocatalytic	rt	4-8	85-98	48
15	<i>n</i> -PrOH/DMSO	Electrocatalytic	rt	60	75-92	49
16	PEG 400	CAN	45 °C	50-80	84-92	50
17	H <sub>2</sub> O	TBAF	ultrasound 75 °C	20-45	86-95	51
18	—	NZF@HAP-Cs	110 °C	10-30	76-98	52

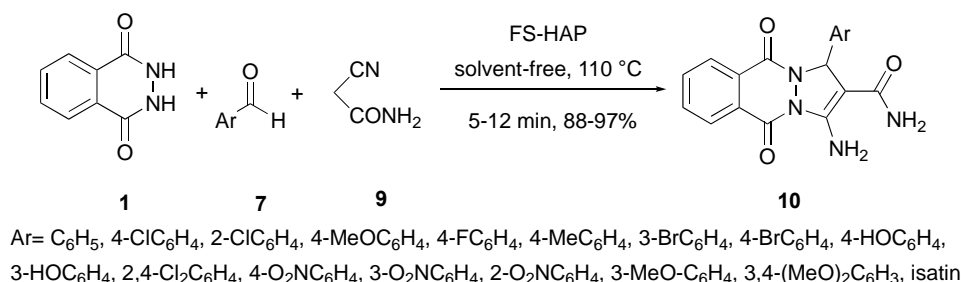
19	EtOH	Et <sub>3</sub> N	ultrasound 50 °C	60	85-98	53
20	—	CuO NPs	100 °C	15-35	86-94	54
21	—	ZrO <sub>2</sub> NPs	100 °C	35-45	87-96	55
22	—	[Bmim]OH	MW, 45 °C	4-5	89-98	56
23	—	STA	Neat, 70 °C	20-35	85-93	57
24	—	H <sub>5</sub> BW <sub>12</sub> O <sub>40</sub> -ILMNP	80 °C	10	58-97	58
25	—	[Bu <sub>3</sub> NH][HSO <sub>4</sub> ]	80 °C	9-20	85-94	59
26	H <sub>2</sub> O/EtOH	$\beta$ -cyclodextrin	100 °C	2.5-5 h	82-93	60
27	—	PbO NPs	80 °C	15	69-99	61
28	—	InCl <sub>3</sub>	80 °C	24-40	84-94	62
29	—	SBA-Pr-SO <sub>3</sub> H	80 °C	15-30	70-90	63
30	—	InCl <sub>3</sub>	80 °C	25-40	85-94	64
31	EtOH	WEMPA	MW	6-7	83-89	65
32	—	[Bn-DBU][TFA]	100 °C	8-25	76-93	66
33	—	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> @CPTES@Cu(II)schiff base	80 °C	10-15	90-98	67
34	EtOH	2-aminopyridine	reflux	4-10	86-97	68
35	—	Cu-doped ZnO hollow spheres	100 °C	8-35	81-93	69
36	PEG 400	Nano-NiFe <sub>2</sub> O <sub>4</sub> @TiO <sub>2</sub> -ILPip	90 °C	10-70	85-97	70
37	H <sub>2</sub> O	Fe <sub>3</sub> O <sub>4</sub> @GOQDs- <i>N</i> -( $\beta$ -alanine)	rt	10-20	90-98	71

A reasonable mechanism for the synthesis of product **8** was shown in Scheme 2. The reaction begins through the Knoevenagel condensation of malononitrile or ethyl cyanoacetate **6** with the aldehyde **7** to obtain the intermediate **A**, followed by removing of H<sub>2</sub>O. Then, the phthalhydrazide **1** reacted with intermediate **A** to yield intermediate **B** through the Michael addition, followed by cyclization and tautomerization to provide the corresponding products **8**.



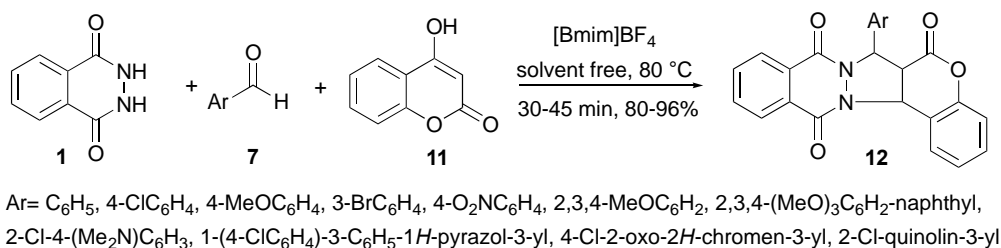
Scheme 2. Plausible reaction mechanism for the synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives **8**

Lalita *et al.*<sup>72</sup> reported the synthesis of various 3-amino-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carboxamide derivatives **10** *via* the reaction of phthalhydrazide **1** with aromatic aldehydes **7** and cyanoacetamide **9** using fish scale hydroxyapatite (FS-HAP) as heterogenous catalyst (Scheme 3). In another study, this reaction was also performed in catalyst free condition in refluxing EtOH.<sup>73</sup>



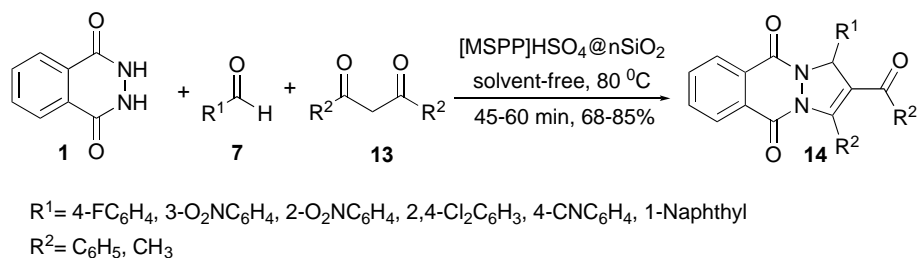
Scheme 3. The synthesis of 3-amino-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carboxamide derivatives **10**

A series of antifungal chromeno-pyrazolo[1,2-*b*]phthalazine-6,9,14(7*H*)-trione derivatives **12** were synthesized *via* a three-component reaction of phthalhydrazide **1**, aromatic aldehydes **7** and 4-hydroxy coumarin **11** using ionic liquid [Bmim]BF<sub>4</sub> as an efficient and reusable catalyst (Scheme 4).<sup>74</sup>



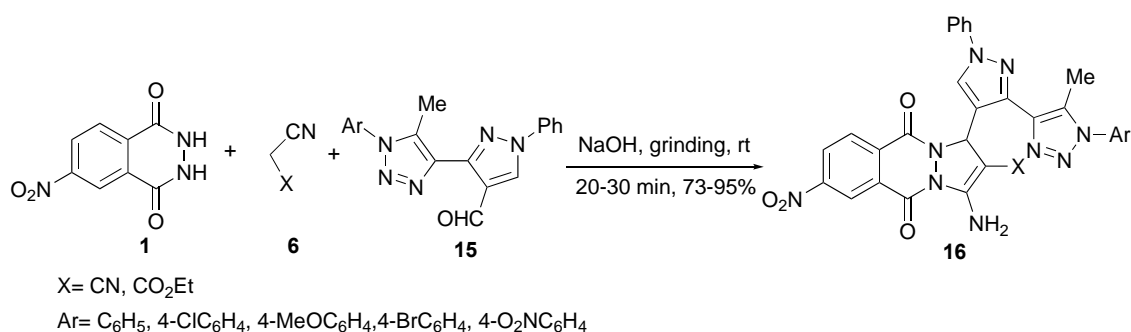
Scheme 4. The synthesis of chromeno-pyrazolo[1,2-*b*]phthalazine-6,9,14(7*H*)-trione derivatives **12**

4-Methyl-1-(3-sulfopropyl)pyridinium hydrogen sulfate as a new ionic liquid immobilized on silica nanoparticles [MSPP]HSO<sub>4</sub>@nSiO<sub>2</sub> prepared by Mohammadpoor-Baltork and his co-workers and investigated for the synthesis of various substituted phthalazine-ones *via* multicomponent reactions of phthalhydrazide **1**, aldehydes **7** and acyclic 1,3-dione **13** to produce the desired products **14** in high yield and short reaction times (Scheme 5).<sup>75</sup> This reaction was also reported in the presence of other catalysts such as wet 2,4,6-trichlorotriazine (TCT) under solvent-free condition,<sup>76</sup> [SBA-Im]HSO<sub>4</sub>,<sup>77</sup> Fe<sub>3</sub>O<sub>4</sub>@silica sulfuric acid nanoparticles,<sup>78</sup> (*S*)-camphorsulfonic acid,<sup>79</sup> ionic liquid [HDEA][ClAc],<sup>80</sup> and RHA@DABCO.<sup>81</sup> This reaction was also accomplished under various conditions *via* different catalysts as shown in Table 2.

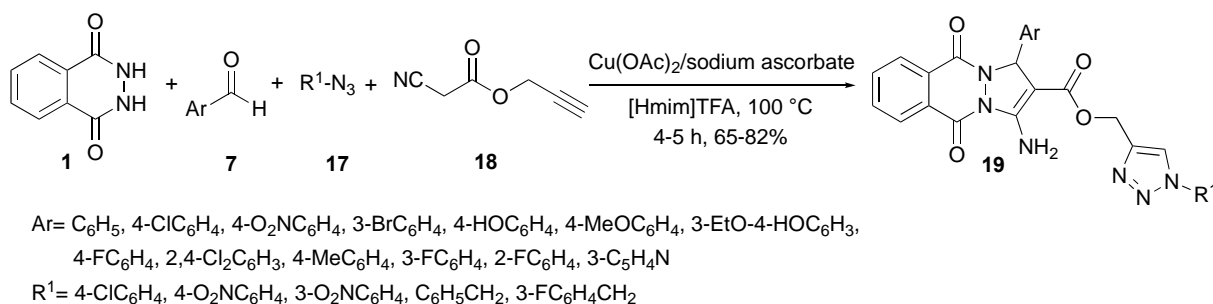
Scheme 5. The synthesis of substituted phthalazine-ones derivatives **14**Table 2. The synthesis of compound **14** under different conditions

Entry	Solvent	Catalyst	Condition	Time (min)	Yield (%)	Ref.
1	—	[MSPP]HSO <sub>4</sub> @nSiO <sub>2</sub>	80 °C	45-60	68-85	<sup>75</sup>
2	—	TCT	110 °C	15-40	40-92	<sup>76</sup>
3	EtOH	[SBA-Im]HSO <sub>4</sub>	reflux	30-35	86-88	<sup>77</sup>
4	—	Fe <sub>3</sub> O <sub>4</sub> @silica sulfuric acid	100 °C	35-45	84-88	<sup>78</sup>
5	—	(S)-camphorsulfonic acid	ultrasound, rt	40-70	45-82	<sup>79</sup>
6	—	(S)-camphorsulfonic acid	80 °C	35-65	50-85	<sup>79</sup>
7	—	[HDEA][ClAc]	140 °C	6 h	70-90	<sup>80</sup>
8	EtOH	RHA@DABCO	reflux	45	80-83	<sup>81</sup>

The grinding method for the synthesis of 1-(3-(1,2,3-triazol-5-yl)pyrazol-4-yl)pyrazolo[1,2-*b*]phthalazinedione derivatives **16** via a three component reaction of the 6-nitrophthalhydrazide **1**, 1,2,3-triazolyl pyrazolecarbaldehydes **15** with active methylene compounds **6** (such as malononitriles or ethyl cyanoacetate) in the presence of NaOH under solvent-free condition was reported by Gomha *et al.* (Scheme 6).<sup>82</sup> The newly synthesized compounds have shown high potency as anticancer agents, which inhibit cancer cells growth but have lower cytotoxic effects on normal cells in the concentration range used.

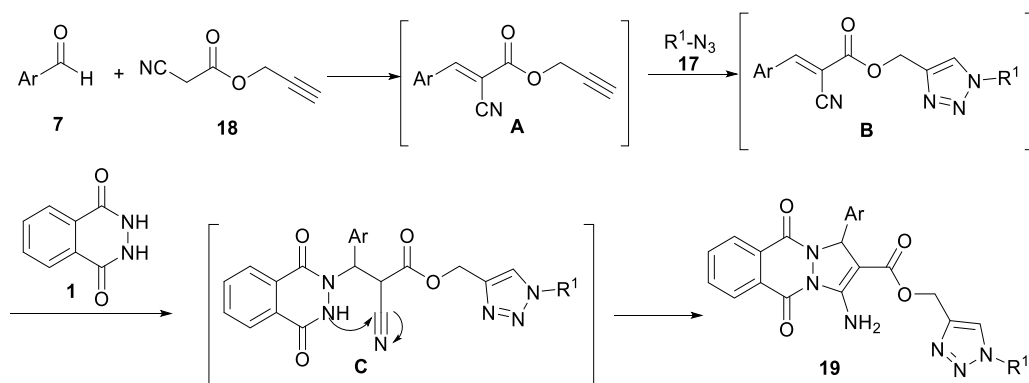
Scheme 6. The synthesis of 1-(3-(1,2,3-triazol-5-yl)pyrazol-4-yl)pyrazolo[1,2-*b*]phthalazinedione **16**

Dabiri *et al.*<sup>83</sup> developed a new approach *via* a one-pot multicomponent reaction of phthalhydrazide **1**, different aromatic aldehydes **7**, and prop-2-ynyl-2-cyanoacetate **18** as an active methylene reagent to produce 1,2,3-triazol-4-ylmethyl-3-amino-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carboxylate derivatives **19**. In this method, the Cu(OAc)<sub>2</sub>/sodium ascorbate was used as catalyst in 1-methyl-1*H*-imidazolium trifluoroacetate ([Hmim]TFA) as an ionic liquid medium (Scheme 7).



Scheme 7. The synthesis of 1,2,3-triazol-4-ylmethyl-3-amino-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carboxylate skeleton **19**

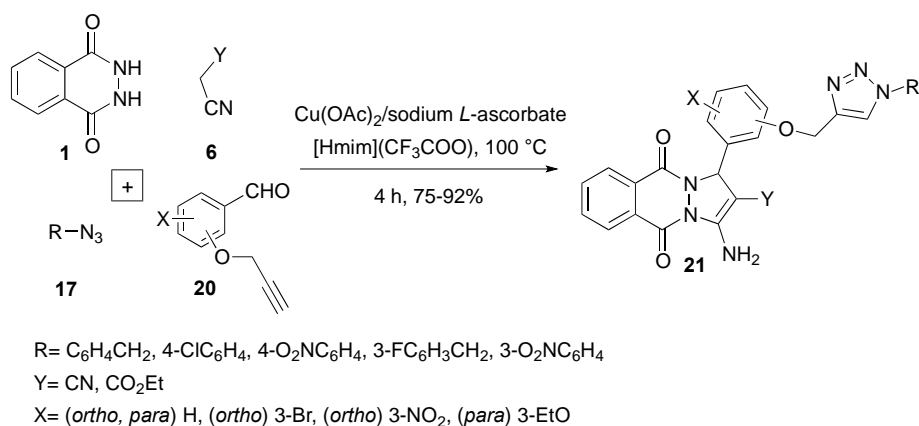
The plausible mechanism for the preparation of product **19** was outlined in scheme 8. The Knoevenagel condensation of aromatic aldehydes **7** and active methylene compound **18** produced the intermediate **A**, which was reacted with azide **17** *via* a 1,3-dipolar cycloaddition reaction to give the intermediate **B**. Then, it was reacted with phthalhydrazide **1** through the Michael-type addition to provide intermediate **C**, which was cyclized to afford the target product **19**.



Scheme 8. Proposed mechanism for the synthesis of 1,2,3-triazol-4-ylmethyl-3-amino-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carboxylate skeleton **19**

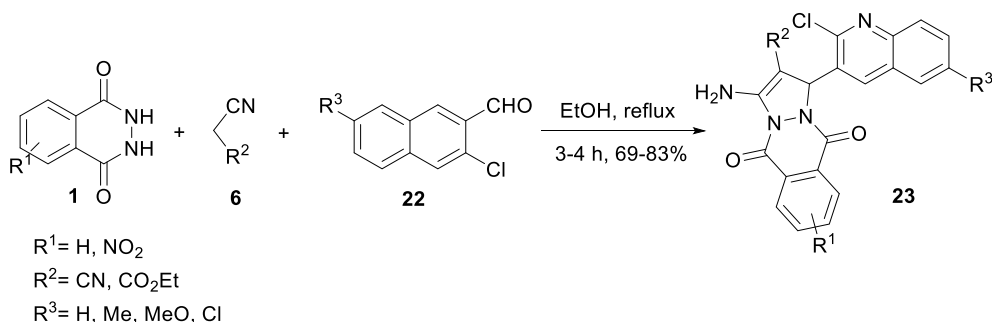
In another study, the synthesis of 1-[(triazolylmethoxy)phenyl]-1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives **21** *via* a four-component reaction of phthalhydrazide **1**, (propargyloxy)benzaldehyde **20**, an active methylene reagent **6** (malononitrile or ethyl

cyanoacetate), and an azide **17** using  $\text{Cu}(\text{OAc})_2/\text{sodium } L\text{-ascorbate}$  as catalyst and 1-methyl-1*H*-imidazolium trifluoroacetate ( $[\text{Hmim}](\text{CF}_3\text{COO})$ ) as an ionic liquid medium was reported by the same group (Scheme 9).<sup>84</sup> It is important to mention that this domino reaction involved the highly selective formation of one C–C and four C–N bonds and of two heterocyclic scaffolds, and could be widely applied in combination chemistry, diversity-oriented synthesis, and drug discovery.



Scheme 9. The synthesis of 1-[(triazolylmethoxy)phenyl]-1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives **21**

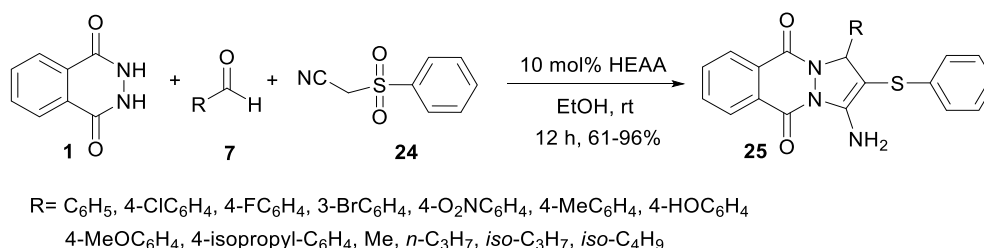
Patel and co-workers have demonstrated a simple and efficient method for the synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives **23** through the one-pot three-component condensation reaction of phthalhydrazide **1**, 2-chloro-3-formylquinolines **22**, and malononitrile or ethyl cyanoacetate **6** using piperidine as catalyst in refluxing EtOH. All the synthesized compounds were screened for their antibacterial activity against a panel of pathogenic strains of bacteria and fungi (Scheme 10).<sup>85</sup>



Scheme 10. Synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives **23**

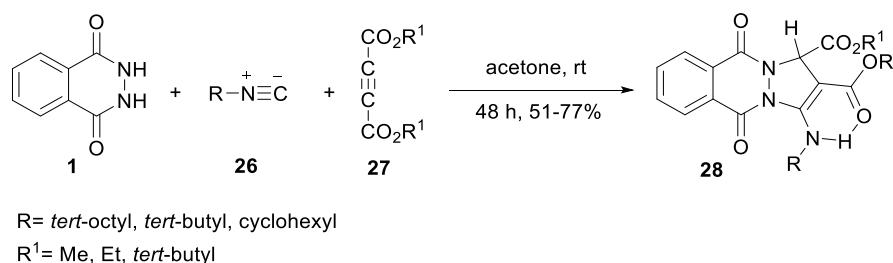
Guo and his co-workers reported the synthesis of 3-amino-2-benzenesulfonyl-1-alkyl/aryl-1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives **25** via one-pot, three-component condensation of phthalhydrazide **1**, aldehydes **7**, and

(phenylsulfonyl)acetonitrile **24** in the presence of 2-hydroxyethylammonium acetate (HEAA) as catalyst in EtOH (Scheme 11).<sup>86</sup> The advantages of this method include easy work-up, environmental friendliness, and good to excellent yields. Also, the photophysical properties of these products were studied, and showed a certain fluorescent structure-property relationship.



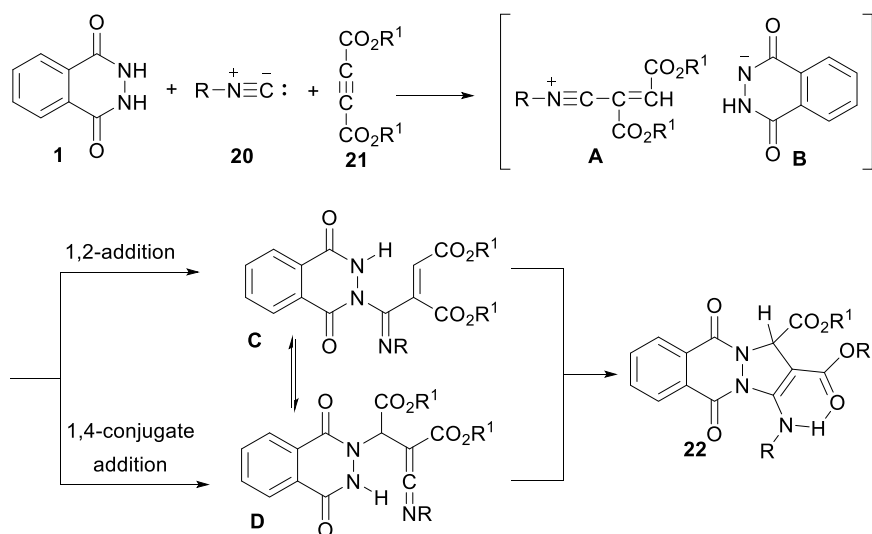
Scheme 11. The synthesis of 3-amino-2-benzenesulfonyl-1-alkyl/aryl-1*H*-pyrazolo[1,2-*b*]-phthalazine-5,10-dione derivatives **25**

In another study, the various of 3-(alkylamino)-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-1,2-dicarboxylate derivatives **28** were synthesized through the reaction of phthalhydrazide **1**, alkyl isocyanides **26** with dialkyl acetylenedicarboxylates **27** at room temperature in dry acetone to afford the desired products **28** (Scheme 12).<sup>87</sup>



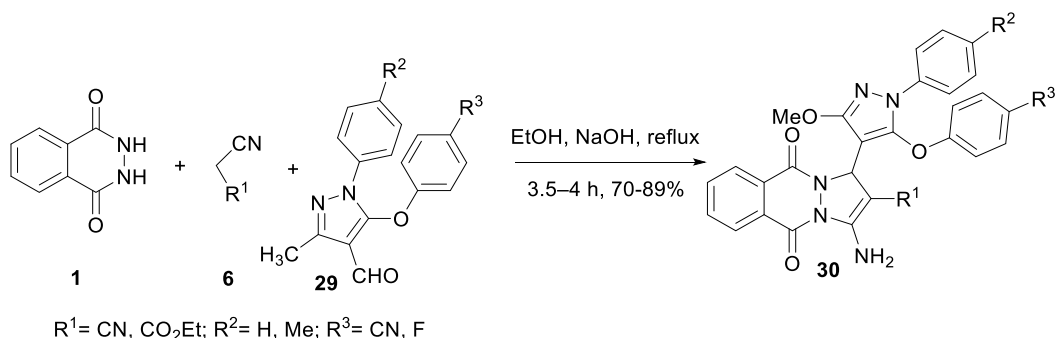
Scheme 12. The synthesis of dialkyl 3-(alkylamino)-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-1,2-dicarboxylate derivatives **28**

A plausible mechanism for the formation of compound **28** is proposed in Scheme 13. In the first step, nucleophilic addition of the isocyanide **26** to the acetylenic ester **27**, and subsequent protonation by NH-acid (phthalhydrazide) provided the vinylisonitrilium cation **A**, which underwent the addition reactions with the nitrogen atom of the conjugate base of the NH-acid **B** on the two possible electrophilic sites (1,2-addition and 1,4-conjugate addition) to produce two possible intermediates **C** and **D** in equilibrium with each other. Then, cyclization of these two intermediates under the reaction conditions provided the dialkyl 3-(alkylamino)-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-1,2-dicarboxylates **28**.



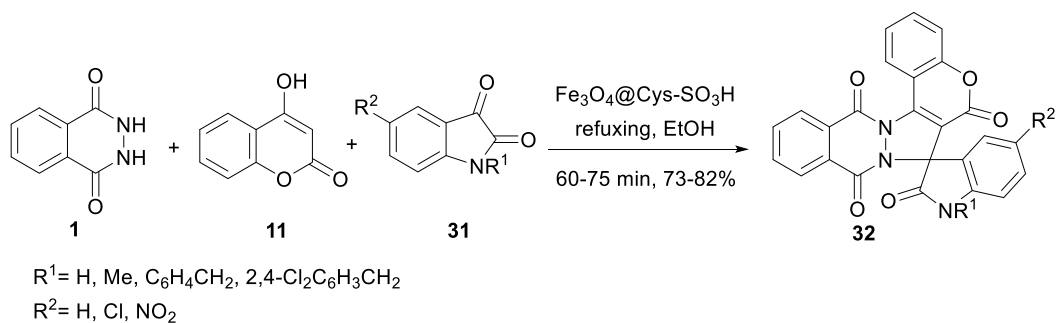
Scheme 13. Proposed mechanism for the formation of compound **28**

The synthesis of new series of pyrazolo[1,2-*b*]phthalazine derivatives **30** bearing the 5-aryloxy pyrazole moiety was developed *via* a one-pot, three-component cyclo-condensation reaction of phthalhydrazide **1**, malononitrile or ethyl cyanoacetate **6**, and 3-methyl-5-aryloxy-1-aryl-1*H*-pyrazole-4-carbaldehyde **29** in the presence of base catalyst in good to excellent yields (Scheme 14).<sup>88</sup> The required starting material of 1-aryl-5-chloro-3-methyl-1*H*-pyrazole-4-carbaldehyde **29** was prepared using reported methods.<sup>89</sup> All the products have been screened *in vitro* antibacterial, antitubercular, cytotoxicity and antioxidant activities.



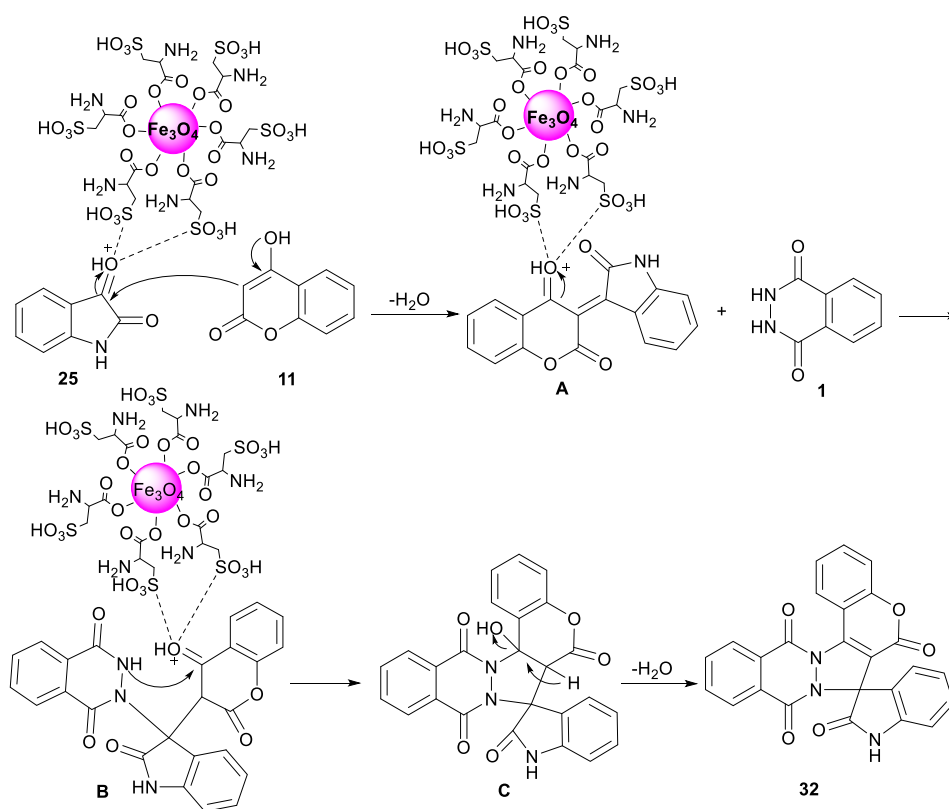
Scheme 14. The synthesis of novel series of pyrazolo[1,2-*b*]phthalazine derivatives **30**

Synthesis of *spiro*-[chromeno[4',3':3,4]pyrazolo[1,2-*b*]phthalazine-7,3'-indoline]-2',6,9,14-tetraone **32** *via* three-component condensation reaction of phthalhydrazide **1**, 4-hydroxycoumarin **11** and isatin derivatives **31** in refluxing EtOH in the presence of Fe<sub>3</sub>O<sub>4</sub>@Cys-SO<sub>3</sub>H magnetic nanocatalyst was reported by Kefayati *et al.* (Scheme 15).<sup>90</sup>



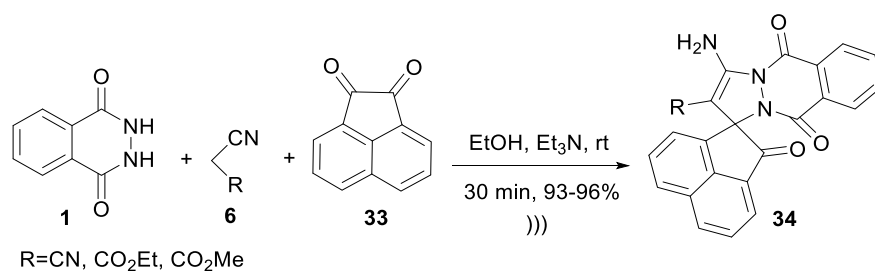
Scheme 15. The synthesis of *spiro*-[chromeno[4',3':3,4]pyrazolo[1,2-*b*]phthalazine-7,3'-indoline]-2',6,9,14-tetraone **32**

A proposed mechanism for the synthesis of compound **32** is shown in Scheme 16. The sulfonic groups (SO<sub>3</sub>H) on the catalyst activate the carbonyl groups of isatin **26**, which was reacted with 4-hydroxycoumarins **11** through the Knoevenagel condensation to give product **A**. Then, it was reacted with phthalhydrazide **1** via the Michael addition reaction to provide the intermediate **B**, followed by cyclization to give the intermediate **C** and dehydration to afford the *spiro*-[chromeno[4',3':3,4]pyrazolo[1,2-*b*]phthalazine-7,3'-indoline]-2',6,9,14-tetraone **32**.



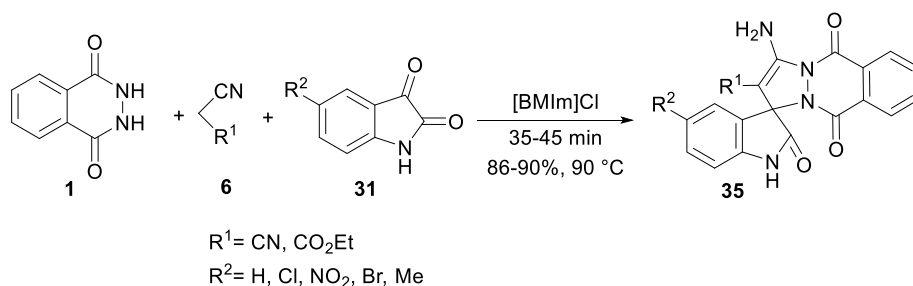
Scheme 16. Plausible reaction mechanism for the synthesis of *spiro*-[chromeno[4',3':3,4]pyrazolo[1,2-*b*]phthalazine-7,3'-indoline]-2',6,9,14-tetraone **32**

In another study, Nabid and his co-workers developed an efficient procedure under ultrasonic condition for the synthesis of a novel class of *spiro*-acenaphthylene-1,1'-pyrazolo[1,2-*b*]phthalazines **34** through the reaction of alkyl nitrile such as malononitrile **6**, acenaphthylene-1,2-dione **33**, and phthalhydrazide **1** in ethanol, and Et<sub>3</sub>N as catalyst at room temperature (Scheme 17).<sup>91</sup> Reduction in reaction times, employing a green solvent, and improved yields are the benefits of this technique under ultrasonic irradiation compared to traditional heating methods.



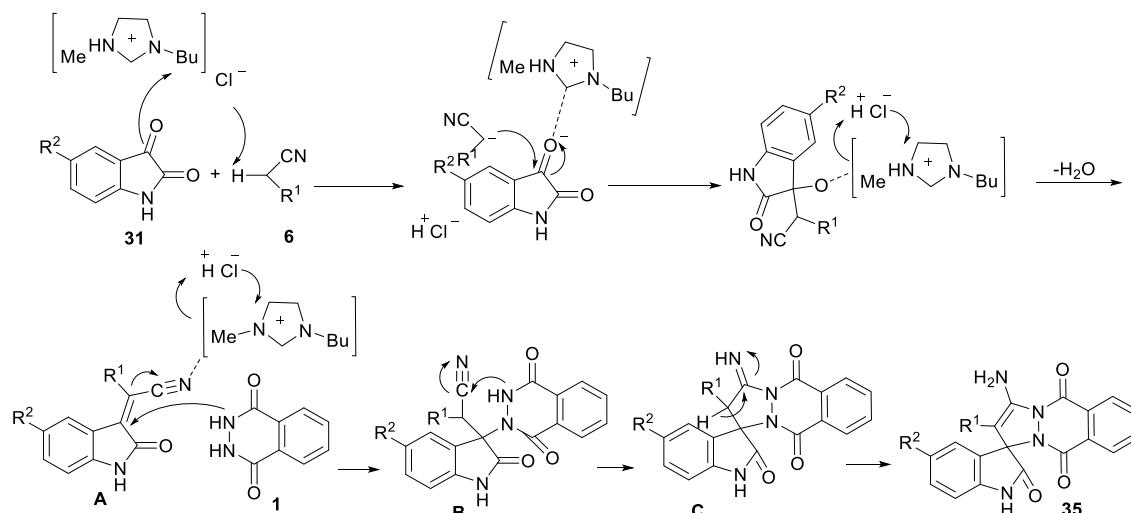
Scheme 17. The synthesis of *spiro*-acenaphthylene-1,1'-pyrazolo[1,2-*b*]phthalazines **34**

For the synthesis of *spiro*-annulated pyrazole-oxindole ring system **35**, a reaction between phthalhydrazide **1**, isatin **31**, and active methylene reagents **6** in the presence of ionic liquid ([BMIm]Cl) as a solvent catalyst was occurred (Scheme 18).<sup>92</sup>



Scheme 18. The synthesis of spirooxindoles derivatives **35**

A plausible mechanism for the formation of the desired product **35** is outlined in Scheme 19. The condensation of isatin **31**, malononitrile or ethyl cyanoacetate **6** and phthalhydrazide **1** was occurred by the Knoevenagel condensation, Michael addition, intramolecular cyclization, and isomerization reaction. Through the Knoevenagel condensation of isatin **25** and malononitrile or ethyl cyanoacetate **2** by the action of ionic liquid, the intermediate **A** was produced. Then, the nitrogen group of phthalhydrazide **1** was reacted with intermediate **A** via the Michael addition to afford the intermediate **B**, which was cyclized to provide the intermediate **C**, followed by tautomerization to afford the corresponding products **35**.

Scheme 19. Plausible mechanism for synthesis of spirooxindole systems **35**

Similar procedures were also described using  $I_2$ ,<sup>93</sup> *L*-proline,<sup>94</sup> piperidine,<sup>95,96</sup> silica bonded *N*-propyl-sulfamic acid (SBNPSA),<sup>97</sup> [DBU][AcOH],<sup>98</sup>  $NiCl_2$ ,<sup>99</sup> as catalysts in these reactions as summarized in Table 3.

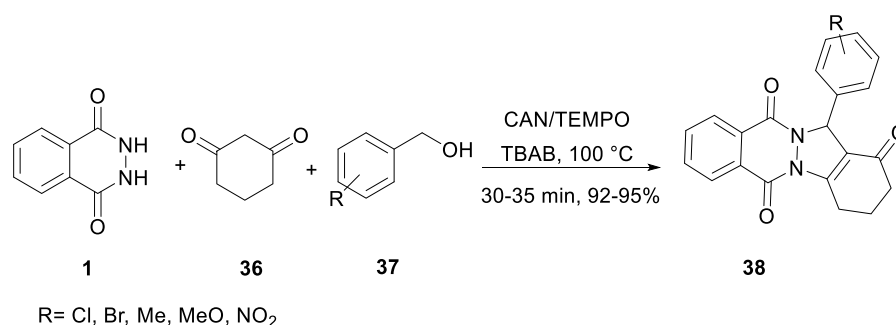
Table 3. Comparison of different conditions for the synthesis of products **35**

Entry	Solvent	Catalyst	Condition	Time	Yield(%)	Ref.
1	—	[BMIm]Cl	90 °C	35-45 min	86-90	92
2	EtOH	$I_2$	sonication	15 min	90	93
3	EtOH	<i>L</i> -proline	reflux	2-2.5 h	86-92	94
4	EtOH	piperidine	reflux	4-9 h	42-81	95
5	EtOH	piperidine	ultrasound, rt	0.5-2 h	69-93	95
6	MeCN	piperidine	reflux	4-8 h	74-89	96
7	EtOH	SBNPSA	MW	30-40 min	89-97	97
8	—	[DBU][AcOH]	80 °C	1-2 h	81-93	98
9	PEG 600	$NiCl_2$	100 °C	0.8-3.5 h	80-95	99

### 3. THE SYNTHESIS OF INDAZOLOPHthalAZINES

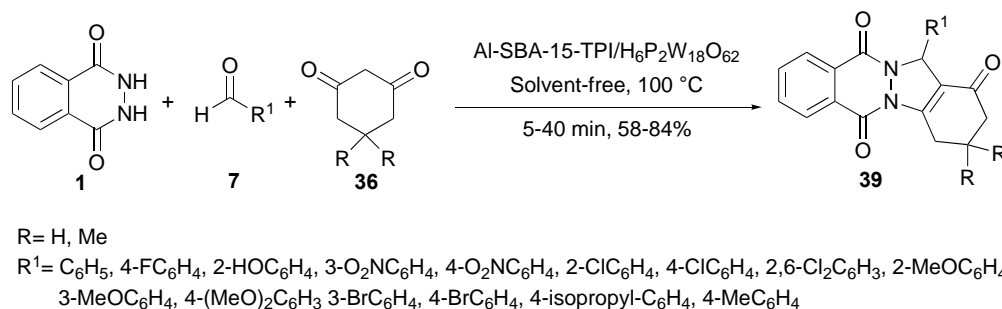
A metal-free synthesis of 2*H*-indazolo[1,2-*b*]phthalazine-triones derivatives **38** using phthalhydrazide **1** with 1,3-diketones **36** and benzyl alcohol **37** via 2,2,6,6-tetramethyl-piperidiny-1-oxy (TEMPO) as a radical initiator, and ceric ammonium nitrate (CAN) as an oxidation reagent in the presence of tetrabutylammonium bromide (TBAB) as catalyst was reported by Pavithra and Ethiraj. Also, this method

was applied for the synthesis of a number of pyrazolophthalazine derivatives (Scheme 20).<sup>100</sup>



Scheme 20. The synthesis of 2*H*-indazolo[1,2-*b*]phthalazine-triones derivatives **38**

In another study, Tayebee *et al.* designed a novel inorganic-organic hybrid material Al-SBA-15-TPI/H<sub>6</sub>P<sub>2</sub>W<sub>18</sub>O<sub>62</sub> as a useful heterogeneous catalyst to produce 2*H*-indazolo[2,1-*b*]phthalazine-1,6,11(13*H*)-trione derivatives **39** under solvent free conditions through the one-pot, three-component reaction by phthalhydrazide **1**, aromatic aldehydes **7** and cyclic diones **36** in short reaction times (Scheme 21).<sup>101</sup>



Scheme 21. The synthesis of 2*H*-indazolo[2,1-*b*] phthalazine-1,6,11(13*H*)-trione derivatives **39**

Several catalysts and various conditions in the synthesis of 2*H*-indazolophthalazine-trione derivatives **39** were investigated in Table 4. The best results were obtained under solvent-free conditions.

Table 4. Comparison of different conditions for synthesis of product **39**

Entry	Solvent	Catalyst	Condition	Time( min)	Yield (%)	Ref.
1	—	Al-SBA-15-TPI/H <sub>6</sub> P <sub>2</sub> W <sub>18</sub> O <sub>62</sub>	100 °C	5-40	58-84	101
2	—	phosphosulfonic acid	100 °C	4-15	75-98	102
3	—	silica-based sulfonic acid	100 °C	15-20	81-90	103
4	EtOH	I <sub>2</sub>	reflux	10-30	86-96	104

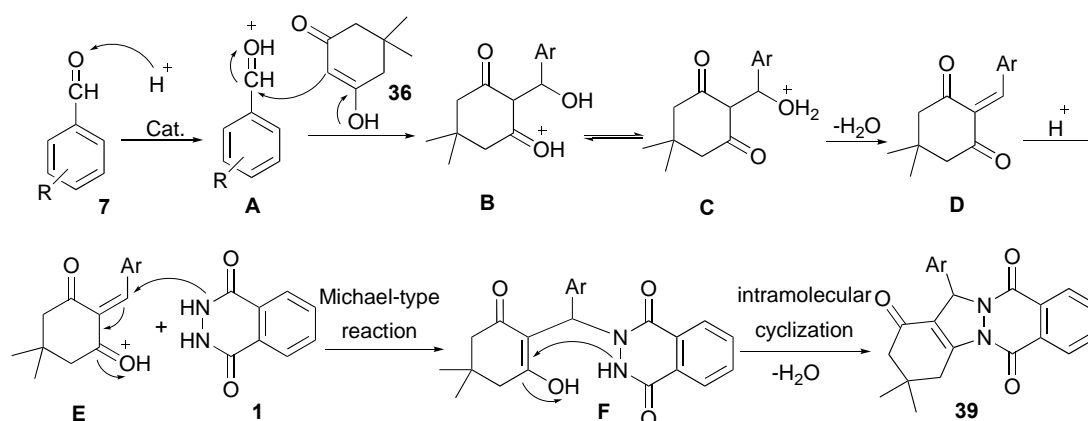
5	—	I <sub>2</sub>	ultrasound	10-15	87-95	105
6	glycerol	PSSA	80 °C	40-70	80-92	106
7	—	H <sub>14</sub> [NaP <sub>5</sub> W <sub>30</sub> O <sub>110</sub> ]/SiO <sub>2</sub>	reflux	6-15	80-94	107
8	—	MTSA	reflux, 100 °C	10-20	78-92	108
9	—	KHSO <sub>4</sub> -SiO <sub>2</sub>	80 °C	10	87-94	109
10	H <sub>2</sub> O/phenol	<i>O</i> -phosphoric acid	reflux, 70 °C	4-20	84-94	110
11	—	H <sub>4</sub> PMo <sub>11</sub> V <sub>1</sub> O <sub>40</sub> /K-10	100 °C	16-38	86-98	111
12	—	[Dsim][HSO <sub>4</sub> ]	100 °C	15-20	76-97	112
13	—	PPA-SiO <sub>2</sub>	100 °C	4-24	78-93	113
14	EtOH	Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> ·xH <sub>2</sub> O	reflux	2-4 h	80-93	114
15	—	[SuSA-H]HSO <sub>4</sub>	100 °C	5-70	88-97	115
16	—	SO <sub>3</sub> H-FMSM	110 °C	15-25	90-95	116
17	—	$\beta$ -cyclodextrine-SO <sub>3</sub> H	80 °C	10-30	85-95	117
18	—	MNPs-guanidine	70 °C	30-65	80-93	118
19	—	[Cu(CH <sub>3</sub> CN) <sub>4</sub> ]PF <sub>6</sub>	100 °C	10-15	79-92	119
20	—	[C <sub>4</sub> (mim) <sub>2</sub> ](FeCl <sub>4</sub> ) <sub>2</sub>	100 °C	10-15	86-91	120
21	EtOH	Fe <sub>3</sub> O <sub>4</sub> @GO-Pr-SO <sub>3</sub> H	reflux	2-3.5 h	90-95	121
22	—	pentaerythritol	80 °C	30-60	75-91	122
23	—	TBBDA	100 °C	10-15	80-91	123
24	—	PBBS	100 °C	60-25	51-87	123
25	—	[PVP-SO <sub>3</sub> H] HSO <sub>4</sub>	80 °C	6-30	85-96	124
26	—	[H-Suc]HSO <sub>4</sub>	80 °C	7-30	89-97	125
27	—	NS-[C <sub>4</sub> (DABCO-SO <sub>3</sub> H) <sub>2</sub> ].4Cl	80 °C	5-30	88-96	126
28	—	NiFe <sub>2</sub> O <sub>4</sub> @TiO <sub>2</sub> -SiO <sub>2</sub> -Pr-DEA-OS O <sub>3</sub> H	90 °C	5-30	83-97	127
29	—	NiFe <sub>2</sub> O <sub>4</sub> @TiO <sub>2</sub> -SO <sub>3</sub> H	80 °C	5-35	87-95	128
30	—	PTA@Fe <sub>3</sub> O <sub>4</sub> /EN-MIL-101	100 °C	20-80	50-94	129
31	H <sub>2</sub> O	[PhBS] <sub>3</sub> <sup>+</sup> PW <sub>12</sub> O <sub>40</sub> <sup>3-</sup>	reflux	5-20	80-97	130
32	—	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> -ZrCl <sub>2</sub> -MNPs	110 °C	9-30	85-95	131
33	H <sub>2</sub> O/EtOH	H <sub>2</sub> SO <sub>4</sub>	reflux	25-30	80-93	132

34	[bmim]BF <sub>4</sub>	H <sub>2</sub> SO <sub>4</sub>	80 °C	30-40	83-94	132
35	—	nano-alumina sulfuric acid	110 °C	10-20	70-98	133
36	—	dodecylphosphonic acid	80 °C	5-15	79-96	134
37	—	SnO <sub>2</sub> NPs	80 °C	12-22	84-96	135
	—	SBA-Pr-NH <sub>2</sub>	80 °C	5-25	70-92	136
38	—	<i>N</i> -propylsulfamic acid functionalized	90 °C	10-25	85-98	137
39		[PVPH]ClO <sub>4</sub>	100 °C	4-45	90-97	138
40	—	MnFe <sub>2</sub> O <sub>4</sub> @SiO <sub>2</sub> NH-NH <sub>2</sub> -PTA	80 °C	20-30	88-96	139
41	—	MnFe <sub>2</sub> O <sub>4</sub> @SiO <sub>2</sub> NH-NH <sub>2</sub> -PTA	ultrasound, 80 °C	6-12	86-95	139
42	MeCN/DMF	trimethylsilyl chloride	80 °C	30-60	86-95	140
43	—	Ni (II)-vanillin-Schiff base-MCM-41	100 °C	70-22 0	89-95	141
44	—	PEG-MDIL	100 °C	10-15	85-90	142
45	EtOH	SBA-15/SO <sub>3</sub> H	ultrasound, 45 °C	15-20	92-96	143
46	TFE	SBA-15	65 °C	140-1 80	89-94	144
47	—	[C <sub>4</sub> (H-DABCO) <sub>2</sub> ][HSO <sub>4</sub> ] <sub>4</sub>	80 °C	7-20	87-96	145
48	—	caffeine-H <sub>2</sub> SO <sub>4</sub>	100 °C	10-20	88-96	146
49	—	Ni NPs	80 °C	10-15	88-94	147
50	—	<i>p</i> -toluenesulfonic acid	80 °C	10-20	80-93	148
51	—	γ-Fe <sub>2</sub> O <sub>3</sub> @Starch-n-Butyl SO <sub>3</sub> H	100 °C	13-38	83-98	149
52	—	silica sulfuric acid	100 °C	7-35	80-91	150
53	—	H <sub>3</sub> PO <sub>4</sub> -Al <sub>2</sub> O <sub>3</sub>	100 °C	8-23	76-93	151
54	—	DABCO(HSO <sub>3</sub> ) <sub>2</sub> (HSO <sub>4</sub> ) <sub>2</sub>	80 °C	4-30	83-95	152
55	—	boron sulfonic acid	100 °C	8-20	86-96	153
56	—	[Simp]HSO <sub>4</sub>	100 °C	10-40	55-88	154

57	—	[Simp] <sub>3</sub> PW <sub>12</sub> O <sub>40</sub>	100 °C	15-80	66-92	155
58	EtOH	Y(OTf) <sub>3</sub>	80 °C	30-50	76-86	156
59	—	Montmorillonite K-10	MW, 80 °C	5-13	90-96	157
60	—	SBA-15-Ph-SO <sub>3</sub> H	80 °C	5-15	85-96	158
61	—	wet cyanuric chloride	100 °C	10-25	89-97	159
62	MeOH	ChCl/ <i>p</i> -TsOH	reflux	4 h	83-93	160
63	—	[(CH <sub>2</sub> ) <sub>4</sub> SO <sub>3</sub> HMIM] [HSO <sub>4</sub> ]	100 °C	10	55-86	161
64	—	citric acid	100 °C	10-30	81-92	162
65	—	ZrOCl <sub>2</sub> .8H <sub>2</sub> O	80 °C	60	80-88	163
66	—	[Et <sub>3</sub> N-SO <sub>3</sub> H]HSO <sub>4</sub>	90 °C	5-10	90-98	164
67	—	silica-supported tungstic acid	80 °C	30-50	87-94	165
68	—	SBA-Pr-SO <sub>3</sub> H	80 °C	5-15	70-90	166
69	—	H <sub>4</sub> SiW <sub>12</sub> O <sub>40</sub>	100 °C	10-25	83-96	167
70	PEG 600	[BSO <sub>3</sub> HmIm]HSO <sub>4</sub>	120 °C	30-40	80-95	168
71	—	IL@nano-SiO <sub>2</sub>	80 °C	10-20	77-96	169
72	—	(PMA)-SiO <sub>2</sub>	80 °C	30-40	80-90	170
73	EtOAc	<i>p</i> -sulfonic acid calix[4]arene	MW, 130 °C	10	51-94	171
74	—	Cu(II)-adenine-MCM-41	100 °C	105-1 50	85-95	172
75	—	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub>	120 °C	10-20	85-96	173
76	PEG 400	ceric ammonium nitrate	50 °C	2-3.5 h	88-94	174
77	—	HFIP	55 °C	8-15 h	75-92	175
78	—	phosphosulfonic acid	100 °C	4-15	75-98	176
79	—	Fe <sub>3</sub> O <sub>4</sub> @Ca(HSO <sub>4</sub> ) <sub>2</sub>	110 °C	2-8	74-87	177
80	EtOH	NiFe <sub>2</sub> O <sub>4</sub> @TiO <sub>2</sub> -Pr-DEA-OSO <sub>3</sub> H	reflux	25-75	70-95	178
81	—	SO <sub>3</sub> H-Fe <sub>3</sub> O <sub>4</sub> -SiO <sub>2</sub>	80 °C	20-37	88-98	179
82	EtOH	Y(OTf) <sub>3</sub>	80 °C	30-50	76-83	180

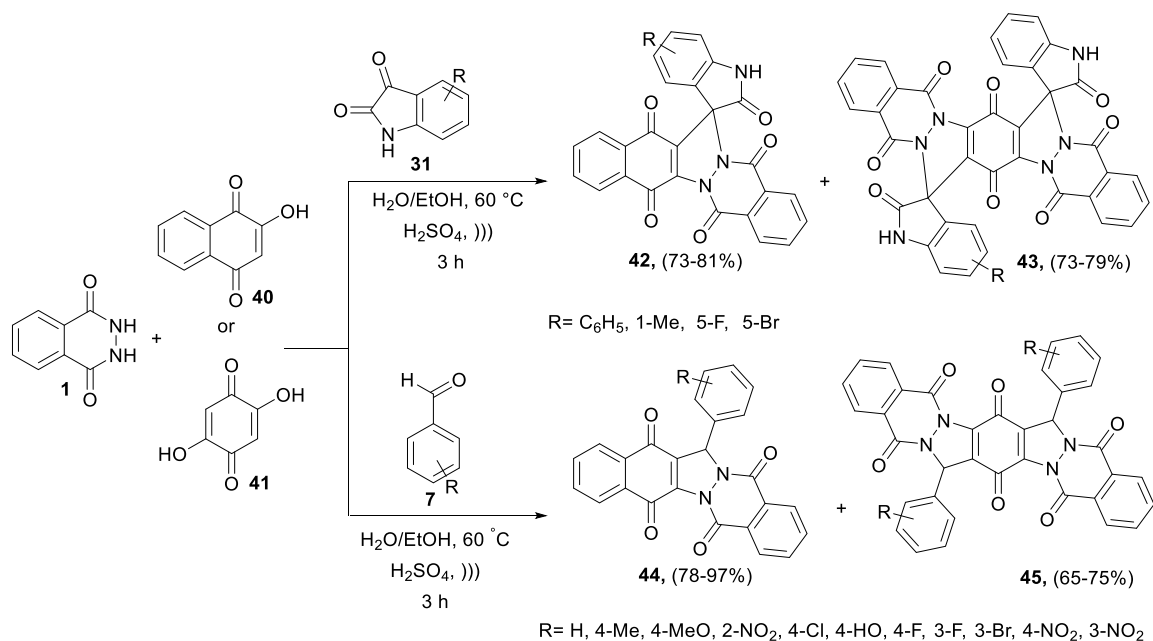
83	—	[PVP-SO <sub>3</sub> H]Cl	80 °C	6-26	85-96	<sup>181</sup>
84	—	5-sulphosalicylic acid	100 °C	8-15	76-92	<sup>182</sup>
85	—	BAILs	110 °C	6-25	86-96	<sup>183</sup>

A plausible mechanism for the formation of compound **39** is proposed in Scheme 22. First, the heteropolyacid increased its acidity through the binding between the H<sup>+</sup> (from heteropolyacid) and carbonyl oxygen of the aromatic aldehyde **7**. Then, the Knoevenagel condensation of dimedone **36** to the carbonyl group of aromatic aldehydes **7** afforded the intermediate **B** which is in equilibrium with intermediate **C**, followed by the loss of H<sub>2</sub>O to yield the intermediate **D**. Protonation of intermediate **D** provided intermediate **E**, which underwent 1,4-conjugate Michael-type addition with phthalhydrazide **1** to give intermediate **F**, followed by intramolecular cyclization to afford the corresponding product **39**.

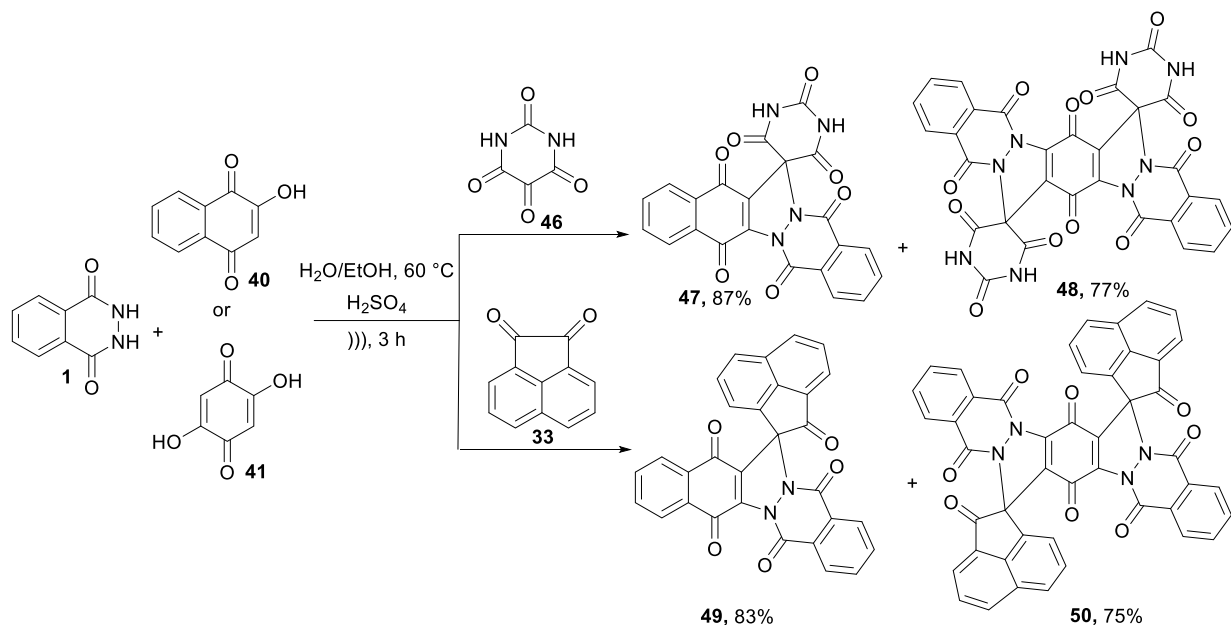


Scheme 22. Plausible mechanism for synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-trione derivatives **39**

In another work, Dabiri *et al.* reported a simple and efficient method through the multicomponent reaction of phthalhydrazide **1**, and 2-hydroxynaphthalene-1,4-dione **40** or 2,5-dihydroxycyclohexa-2,5-diene-1,4-dione **41** with active carbonyl compounds like isatin **31** and aromatic aldehydes **7** to produce *spiro*-[benzindazolophthalazine-indoline]pentaones **42**, *bis-spiro*-benzindazolophthalazine-indoline **43**, benzo[5,6]indazolo[2,1-*b*]phthalazine-tetraones **44** and *bis*-indazolophthalazine **45**, respectively (Scheme 23).

Scheme 23. Synthesis of new indazolophthalazine derivatives **42-45**

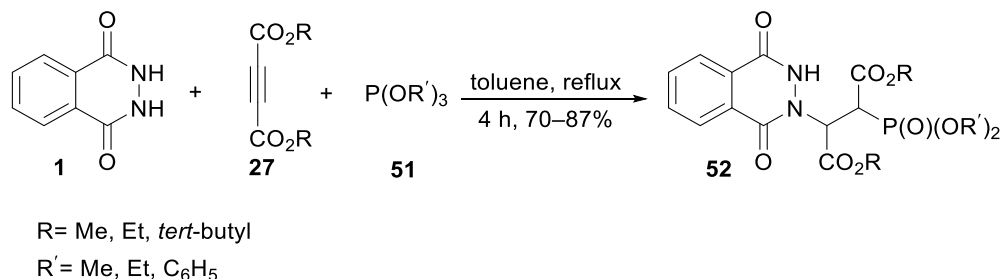
Also, they investigated the reaction of phthalhydrazide **1** and compounds **40** or **41** with acenaphthenequinone **33** or aloxane **47** using H<sub>2</sub>SO<sub>4</sub> under ultrasonic irradiation to provide the indazolophthalazine derivatives (Scheme 24).<sup>184</sup>

Scheme 24. Synthesis of indazolophthalazine **47-50**

#### 4. THE SYNTHESIS OF PHTHALAZINE-SUCCINATE DERIVATIVES

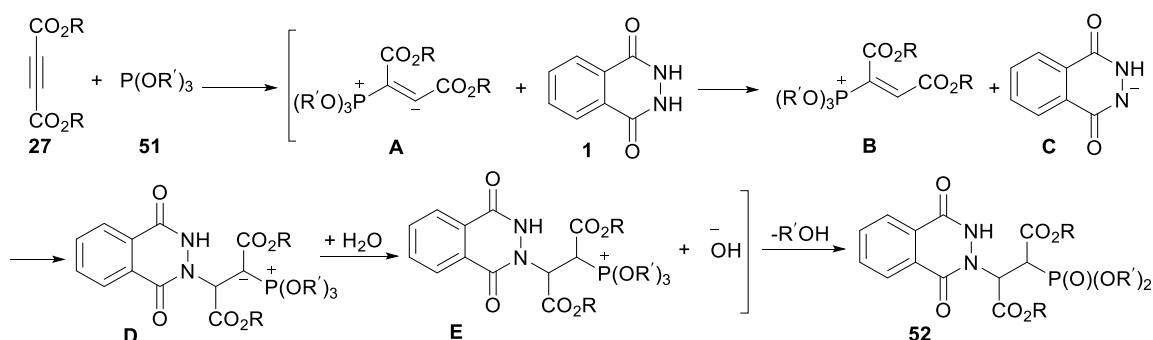
A series of dialkyl-2-(dialkoxyphosphoryl)-3-(1,4-dioxo-3,4-dihydrophthalazin-2(1*H*)-yl)succinate derivatives **52** were synthesized by Azizian and his co-workers in 2012. In this process, phthalhydrazide **1**

was treated with dialkyl acetylenedicarboxylates **27**, and trialkyl (aryl) phosphites **51** in refluxing toluene via the one-pot, three component reactions (Scheme 25).<sup>185</sup>



Scheme 25. The synthesis of dialkyl-2-(dialkoxyphosphoryl)-3-(1,4-dioxo-3,4-dihydrophthalazin-2(1*H*)-yl)succinate derivatives **52**

A plausible mechanism for the formation of compound **52** is proposed in Scheme 26. The addition of trialkyl (aryl) phosphite **51** to the acetylenic ester **27** gave the intermediate **A**, which was reacted with 2,3-dihydrophthalazine-1,4-dione **1** to afford the intermediate **B**, followed by the reaction with the anion of the NH-acid **C** to produce ylide **D**. Then, it was converted to intermediate **E**, which was hydrolyzed to give the compound **52**. Since the reactions were performed in the ordinary atmosphere, it is assumed that the conversion of **D** to **52** is achieved by the air humidity.

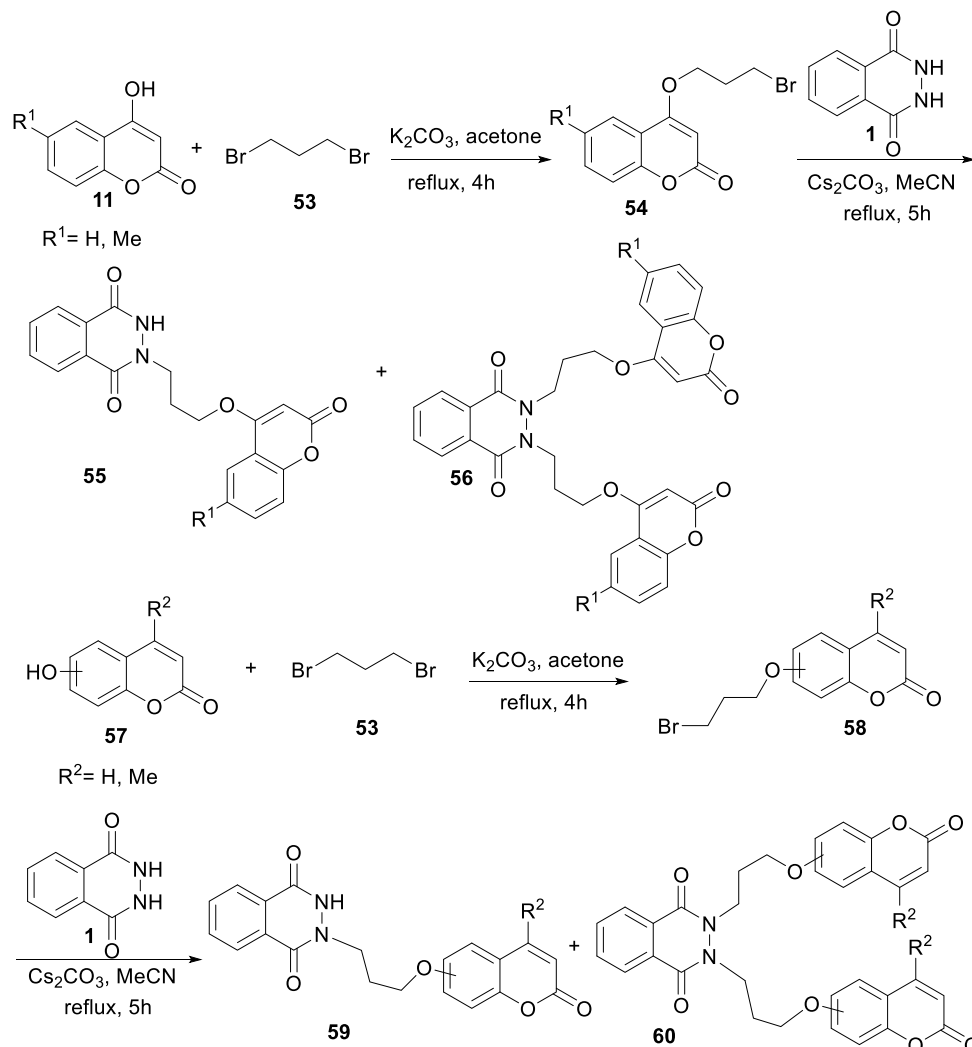


Scheme 26. Plausible mechanism for the synthesis of dialkyl-2-(dialkoxyphosphoryl)-3-(1,4-dioxo-3,4-dihydrophthalazin-2(1*H*)-yl)succinate derivatives **52**

## 5. MISCELLANEOUS REACTIONS

The reaction of phthalhydrazide **1** with bromopropoxycoumarin derivatives **54** in the presence of  $\text{Cs}_2\text{CO}_3$  as catalyst in MeCN as a solvent under reflux condition provided a series of novel phthalhydrazide-coumarin derivatives **55**, **56**, **59**, **60** in good yields. These new compounds were screened for their biological activities by online program PASS (Prediction of Activity Spectra for Substances), and the serotonin 2A receptor (5-HT<sub>2A</sub>R) was selected for molecular docking study. By comparing the

biological activity of these newly synthesized compounds with commercially available drug Risperidone, it was found that the potential activity of dialkylated compounds was better than Risperidone and could be used as potential new antipsychotics for further research (Scheme 27).<sup>186</sup>

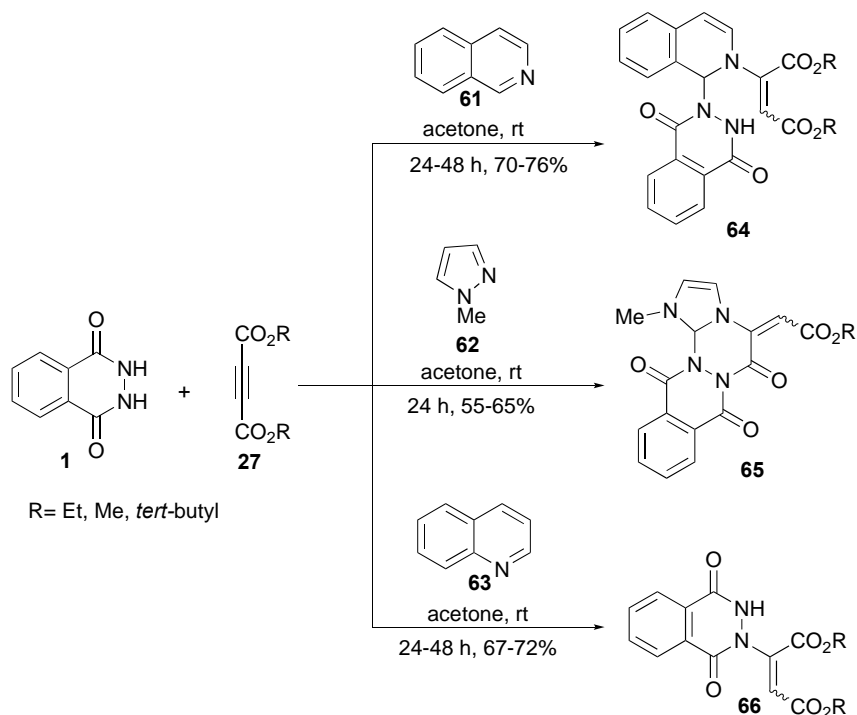


Scheme 27. The synthesis of phthalhydrazide-coumarin derivatives **55**, **56**, **59**, **60**

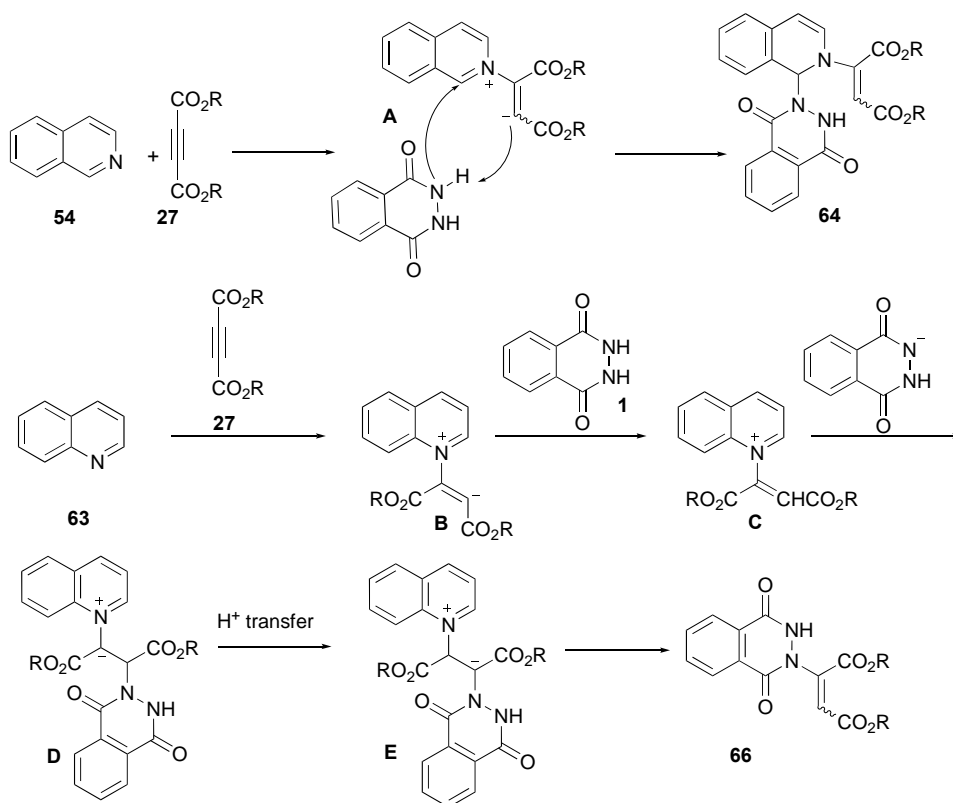
Ghahremanzadeh *et al.* prepared some new phthalazine derivatives through the reaction of phthalhydrazide **1** and dialkyl acetylenedicarboxylates **27** in the presence of isoquinoline **61**, *N*-methylimidazole **62**, and quinoline **63** as *N*-heterocycles compounds (Scheme 28).<sup>187</sup>

Through the initial addition of isoquinoline **61** to the dialkyl acetylenedicarboxylate **27**, a 1:1 zwitterionic intermediate **A** was provided. It was protonated by phthalhydrazide **1**, and then was attacked by the conjugate base of phthalhydrazide to produce compound **64**. Also, the desired compound **66** was produced through the addition of quinoline **63** to the dialkyl acetylenedicarboxylate **27** to give intermediate **B**, followed by protonation with phthalhydrazide **1** to obtain the positively charged ion **C**. Then, it was attacked by the conjugate base of phthalhydrazide to produce the nitrogen ylide **D**, which

underwent a proton-transfer reaction to produce intermediate **E**. Then, it was converted to target compound **66** by elimination of quinoline moiety (Scheme 29).

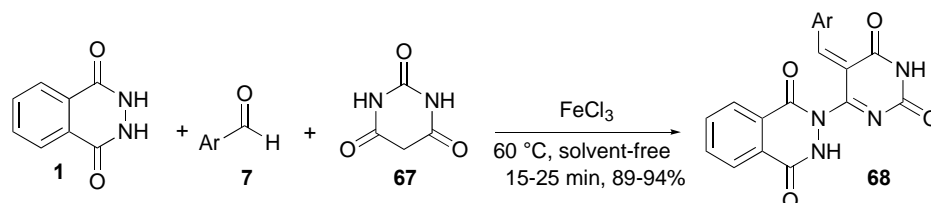


Scheme 28. Synthesis of some new phthalazine derivatives **64**, **65**, **66**



Scheme 29. Plausible mechanism for the synthesis of some new phthalazine derivatives

Yeon and co-workers reported the synthesis of phthalhydrazide derivatives through one-pot three-component coupling reaction in 2014. In this study, phthalhydrazide **1** reacted with aromatic aldehydes **7** and barbituric acid **67** in the presence of 15 mol% FeCl<sub>3</sub> under solvent-free conditions to provide (tetrahydro-2,6-dioxypyrimidin-4-yl)-2,3-dihydrophthalazine-1,4-dione derivatives **68** (Scheme 30).<sup>188</sup>



Ar = C<sub>6</sub>H<sub>5</sub>, 4-EtOC<sub>6</sub>H<sub>4</sub>, 4-MeOC<sub>6</sub>H<sub>4</sub>, 4-BrC<sub>6</sub>H<sub>4</sub>, 4-ClC<sub>6</sub>H<sub>4</sub>, 4-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, 4-MeC<sub>6</sub>H<sub>4</sub>, 4-FC<sub>6</sub>H<sub>4</sub>, 4-HOC<sub>6</sub>H<sub>4</sub>, 3-BrC<sub>6</sub>H<sub>4</sub>, 3-MeC<sub>6</sub>H<sub>4</sub>, 3-FC<sub>6</sub>H<sub>4</sub>, 3-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, 2-FC<sub>6</sub>H<sub>4</sub>, 2-BrC<sub>6</sub>H<sub>4</sub>, 2-MeC<sub>6</sub>H<sub>4</sub>, 3,4,5-(MeO)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, 2-thienyl, 4-*i*-PrC<sub>6</sub>H<sub>4</sub>, 1,3-Benzodioxole-5-carbaldehyde

Scheme 30. The synthesis of tetrahydro-2,6-dioxypyrimidin-4-yl)-2,3-dihydrophthalazine-1,4-dione derivatives **68**

This reaction in the presence of 1-ethyl-3-methylimidazolium tetrafluoroborate ([Emim][BF<sub>4</sub>]) IL as a solvent and as well as an efficient catalyst was also reported (Table 5).<sup>189</sup> The target compounds have been tested against human chronic myeloid leukemia cells (K 562) and human colon carcinoma cells (Colo 205) for their anticancer activity.

Table 5. The synthesis of compound **68** under different conditions

Entry	Solvent	Catalyst	Condition	Time (min)	Yield (%)	Ref.
1	—	FeCl <sub>3</sub>	60 °C	15-25	89-94	188
2	—	[Emim][BF <sub>4</sub> ]	70 °C	15-20	92-95	189

## 6. CONCLUSIONS

There is a broad variety of multicomponent reactions, which involve phthalhydrazide in the synthesis of different heterocyclic compounds. The biological activities of phthalhydrazide derivatives make these compounds versatile synthetic targets as well as important structural units in medicinal and synthetic organic chemistry. Further research and developments of this compound is expected to be published on the synthetic chemistry.

## ACKNOWLEDGEMENTS

The authors acknowledge the research council of the Alzahra University for support.

## ABBREVIATIONS

<b>[Bmim]OH</b>	1-butyl-3-methylimidazolium hydroxide
<b>BAILs</b>	brønsted acid ionic liquid
<b>[BMIm]Cl</b>	1-butyl-3-methylimidazolium chloride
<b>[BSO<sub>3</sub>HmIm]HSO<sub>4</sub></b>	4-(3-methyl-1-imidazolium)-1-butanefulfonic acid hydrogen sulfate
<b>CPTES</b>	3-chloropropyltriethoxysilane
<b>CAN</b>	ceric ammonium nitrate
<b>ChCl</b>	choline chloride
<b>[C<sub>4</sub>(mim)<sub>2</sub>](FeCl<sub>4</sub>)<sub>2</sub></b>	1,4'-(butane-1,4-diyl) <i>bis</i> (3-methylimidazolium) <i>bis</i> -[tetrachloroferrate(III)]
<b>DABCO</b>	1,4-diazabicyclo[2.2.2]octane
<b>Dsim</b>	1,3-disulfonic acid imidazolium
<b>DEA</b>	diethanolamine
<b>EN</b>	ethylenediamine
<b>FMSM</b>	functionalized mesoporous silica materials
<b>GOQDs</b>	graphene oxide quantum dots
<b>GO</b>	graphene oxide
<b>[HDEA][ClAc]</b>	diethanolammonium chloroacetate
<b>[Hmim]TFA</b>	1-methyl-1 <i>H</i> -imidazolium trifluoroacetate
<b>HFIP</b>	1,1,1,3,3,3-hexafluoro-2-propanol
<b>MTSA</b>	melaminetrisulfonic acid
<b>[MSPP]HSO<sub>4</sub></b>	4-methyl-1-(3-sulfopropyl)pyridinium hydrogen sulfate
<b>NZF@HAP-Cs</b>	caesiumcarbonate supported on hydroxyapatite-coated nickel zinc ferrite
<b>PPA</b>	polyphosphoric acid
<b>PVP</b>	poly(4-vinylpyrrolidonium)
<b>PEG-MDIL</b>	poly(ethylene glycol) <i>bis</i> (methylimidazolium dichloride)
<b>PTA</b>	phosphotungstic acid
<b>PMA</b>	phosphomolybdic acid
<b>[PhBS]</b>	phenanthrolinebutane sulfonate
<b>PBBS</b>	poly( <i>N</i> -bromo- <i>N</i> -ethylbenzene-1,3-disulfonamide)
<b>RHA</b>	rice husk ash silica
<b>SiPMIM</b>	(3-propyltrimethoxysilane)imidazolium
<b>SBNPSA</b>	silica bonded <i>N</i> -propylsulfamic acid
<b>SuSA-H</b>	succinimidinium <i>N</i> -sulfonic acid

<b>Simp</b>	3-sulfonic acid 1-imidazolopyridinium
<b>TBBDA</b>	tetrabromobenzene-1,3-disulfonamide
<b>TBAF</b>	tetrabutylammonium fluoride
<b>TPI</b>	<i>N</i> -[3-(triethoxysilyl)propyl]isonicotinamide
<b>TEMPO</b>	2,2,6,6-tetramethylpiperidinyl-1-oxy
<b>TCT</b>	trichlorotriazine
<b>WEMPA</b>	water extract of mango peel ash

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