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

Ghodsi Mohammadi Ziarani,* Negar Jamasbi, and Fatemeh Mohajer

Department of Chemistry, Faculty of Physic and Chemistry, Alzahra University, Tehran, Iran. P. O. Box 1993893973. gmohammadi@alzahra.ac.ir

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.

CONTENTS

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.[1-3] 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.[4-6] 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,[7]

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

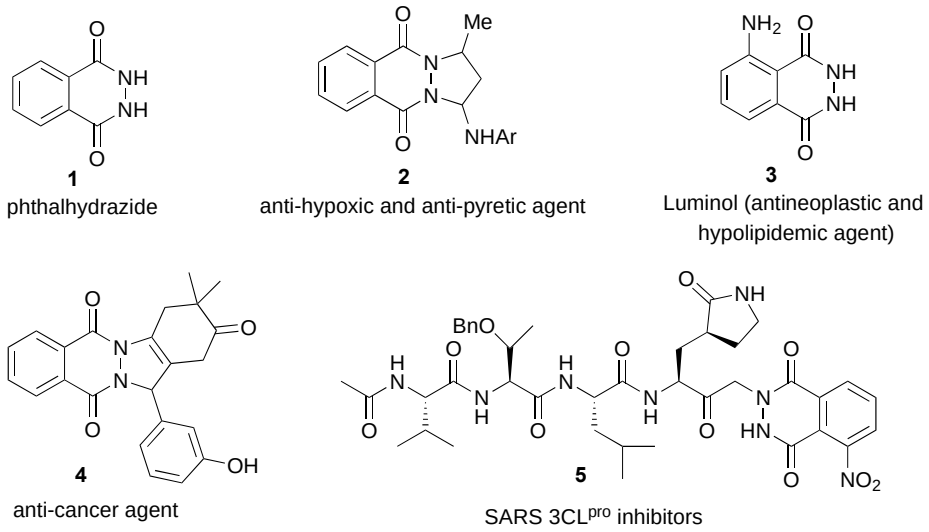


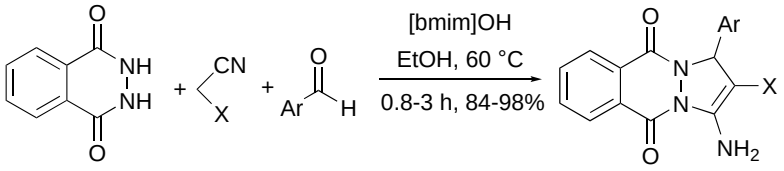
Figure 1. Examples of bioactive compounds containing phthalazine derivatives

These compounds also could be utilized as interesting materials for new fluorescence or luminescence probes.[17] 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.[18,19] 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.[20,21] In continuous of our previous studies on the synthesis of heterocyclic compounds and in MCRs,[22-35] 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).[36] This method applied on different aromatic aldehydes, which were shown higher reactivity with electron-withdrawing groups.



1 **6** **7** **8**

X = CN, CO_2Et

Ar= C_6H_5, 4-ClC_6H_4, 4-$MeOC_6H_4$, 4-FC_6H_4, 4-MeC_6H_4, 2-$ClOC_6H_4$
3-BrC_6H_4, 2,4-$Cl_2C_6H_3$, 4-$O_2NC_6H_4$, 4-isopropyl-C_6H_4

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

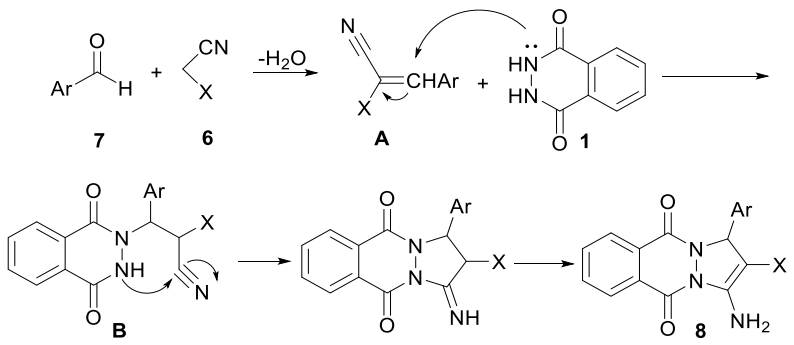
This reaction was also performed in the presence of different catalysts and under various conditions as shown in Table 1.

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	—	$NaHCO_3$	120 °C	15-50	85-98	39
5	EtOH	$Cu(OTf)_2$	80 °C	120-180	86-93	40
6	—	SBA@$BiPy^{2+}$ $2Cl^-$	100 °C	30-60	86-96	41
7	[bmim]Br	*p*-toluenesulfonic acid	100 °C	2-5 h	73-97	42
8	EtOH	RH@[SiPrDABCO@$BuSO_3H$]HSO_4	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	—	$CuFe_2O_4$@SiO_2	120 °C	10-15	88-95	46
13	—	[SiPMIM]OH@MNPs	rt	10-20	82-95	47
14	*n*-PrOH	Electrocatalytic	rt	4-8	85-98	48
15	*n*-PrOH/DMSO	Electrocatalytic	rt	60	75-92	49
16	PEG 400	CAN	45 °C	50-80	84-92	50
17	H_2O	TBAF	ultrasound 75 °C	20-45	86-95	51
18	—	NZF@HAP-Cs	110 °C	10-30	76-98	52

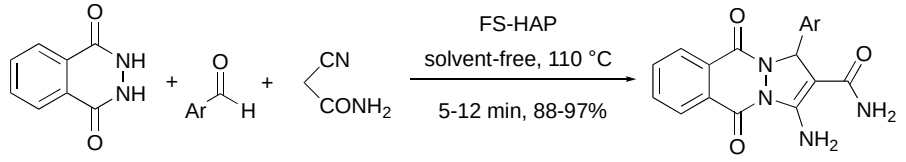
19	EtOH	Et_3N	ultrasound 50 °C	60	85-98	[53]
20	—	CuO NPs	100 °C	15-35	86-94	[54]
21	—	ZrO_2 NPs	100 °C	35-45	87-96	[55]
22	—	[Bmim]OH	MW, 45 °C	4-5	89-98	[56]
23	—	STA	Neat, 70 °C	20-35	85-93	[57]
24	—	$H_5BW_{12}O_{40}$-ILMNPs	80 °C	10	58-97	[58]
25	—	$[Bu_3NH][HSO_4]$	80 °C	9-20	85-94	[59]
26	H_2O/EtOH	*β*-cyclodextrin	100 °C	2.5-5 h	82-93	[60]
27	—	PbO NPs	80 °C	15	69-99	[61]
28	—	$InCl_3$	80 °C	24-40	84-94	[62]
29	—	SBA-Pr-SO_3H	80 °C	15-30	70-90	[63]
30	—	$InCl_3$	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_3O_4@SiO_2@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 hallow spheres	100 °C	8-35	81-93	[69]
36	PEG 400	Nano-$NiFe_2O_4$@TiO_2-ILPip	90 °C	10-70	85-97	[70]
37	H_2O	Fe_3O_4@GOQDs-*N*-(*β*-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_2O. 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.*[72] 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.[73]

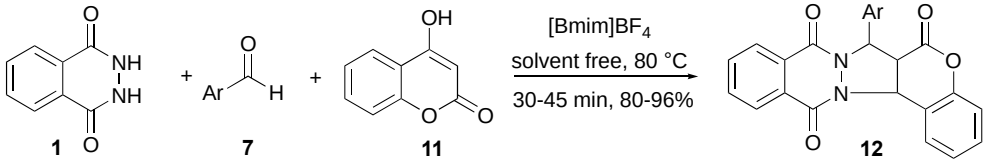


1 **7** **9** **10**

Ar= C_6H_5, 4-ClC_6H_4, 2-ClC_6H_4, 4-$MeOC_6H_4$, 4-FC_6H_4, 4-MeC_6H_4, 3-BrC_6H_4, 4-BrC_6H_4, 4-HOC_6H_4, 3-HOC_6H_4, 2,4-$Cl_2C_6H_4$, 4-$O_2NC_6H_4$, 3-$O_2NC_6H_4$, 2-$O_2NC_6H_4$, 3-MeO-C_6H_4, 3,4-$(MeO)_2C_6H_3$, isatin

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(*7H*)-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_4 as an efficient and reusable catalyst (Scheme 4).[74]

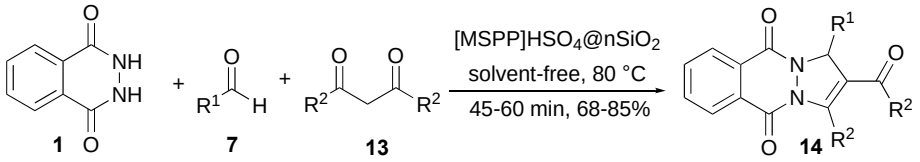


1 **7** **11** **12**

Ar= C_6H_5, 4-ClC_6H_4, 4-$MeOC_6H_4$, 3-BrC_6H_4, 4-$O_2NC_6H_4$, 2,3,4-$MeOC_6H_2$, 2,3,4-$(MeO)_3C_6H_2$-naphthyl, 2-Cl-4-$(Me_2N)C_6H_3$, 1-(4-ClC_6H_4)-3-C_6H_5-1*H*-pyrazol-3-yl, 4-Cl-2-oxo-2*H*-chromen-3-yl, 2-Cl-quinolin-3-yl

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

4-Methyl-1-(3-sulfopropyl)pyridinium hydrogen sulfate as a new ionic liquid immobilized on silica nanoparticles [MSPP]HSO_4@nSiO_2 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).[75] This reaction was also reported in the presence of other catalysts such as wet 2,4,6-trichlorotriazine (TCT) under solvent-free condition,[76] [SBA-Im]HSO_4,[77] Fe_3O_4@silica sulfuric acid nanoparticles,[78] (*S*)-camphorsulfonic acid,[79] ionic liquid [HDEA][ClAc],[80] and RHA@DABCO.[81] This reaction was also accomplished under various conditions *via* different catalysts as shown in Table 2.



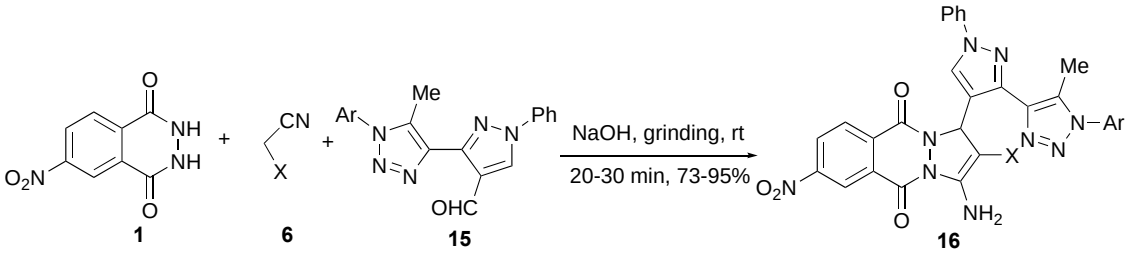
R^1= 4-FC_6H_4, 3-$O_2NC_6H_4$, 2-$O_2NC_6H_4$, 2,4-$Cl_2C_6H_3$, 4-CNC_6H_4, 1-Naphthyl
R^2= C_6H_5, Me

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_4@nSiO_2	80 °C	45-60	68-85	[75]
2	—	TCT	110 °C	15-40	40-92	[76]
3	EtOH	[SBA-Im]HSO_4	reflux	30-35	86-88	[77]
4	—	Fe_3O_4@silica sulfuric acid	100 °C	35-45	84-88	[78]
5	—	(*S*)-camphorsulfonic acid	ultrasound, rt	40-70	45-82	[79]
6	—	(*S*)-camphorsulfonic acid	80 °C	35-65	50-85	[79]
7	—	[HDEA][ClAc]	140 °C	6 h	70-90	[80]
8	EtOH	RHA@DABCO	reflux	45	80-83	[81]

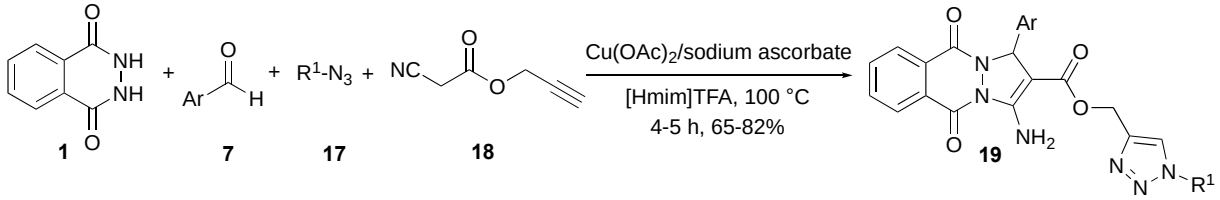
The grinding method for the synthesis of 1-(3-(1,2,3-triazol-5-yl)pyrazol-4-yl)pyrazolo[1,2-*b*]phthalazinedione derivatives **16** *via* a three component reaction of the 6-nitrophthalhydrazide **1**, 1,2,3-triazolyl pyrazolecarbaldehydes **15** with active methylene compounds **6** (such as malononitriles or ethyl cyanoacetate) in the presence of NaOH under solvent-free condition was reported by Gomha *et al.* (Scheme 6).[82] 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.



X= CN, CO_2Et
Ar= C_6H_5, 4-ClC_6H_4, 4-$MeOC_6H_4$,4-BrC_6H_4, 4-$O_2NC_6H_4$

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.*[83] 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 produce1,2,3-triazol-4-ylmethyl-3-amino-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carb oxylate derivatives **19**. In this method, the $Cu(OAc)_2$/sodium ascorbate was used as catalyst in 1-methyl-1*H*-imidazolium trifluoroacetate ([Hmim]TFA) as an ionic liquid medium (Scheme 7).

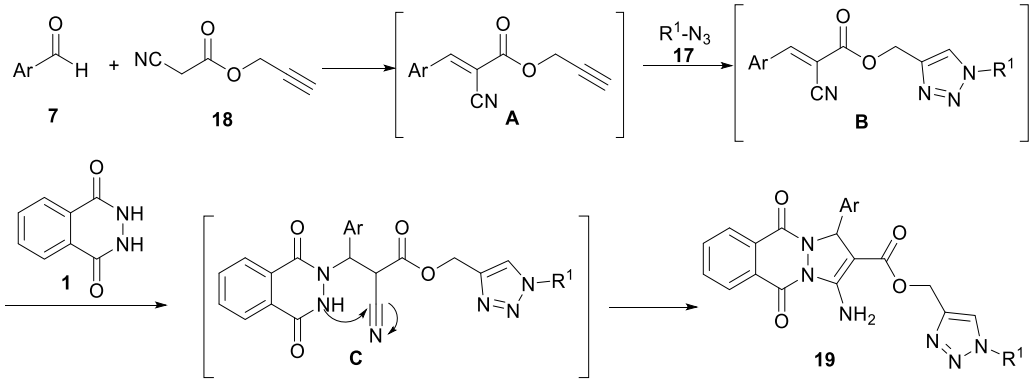


Ar= C_6H_5, 4-ClC_6H_4, 4-$O_2NC_6H_4$, 3-BrC_6H_4, 4-HOC_6H_4, 4-$MeOC_6H_4$, 3-EtO-4-HOC_6H_3, 4-FC_6H_4, 2,4-$Cl_2C_6H_3$, 4-MeC_6H_4, 3-FC_6H_4, 2-FC_6H_4, 3-C_5H_4N

R^1= 4-ClC_6H_4, 4-$O_2NC_6H_4$, 3-$O_2NC_6H_4$, $C_6H_5CH_2$, 3-$FC_6H_4CH_2$

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**

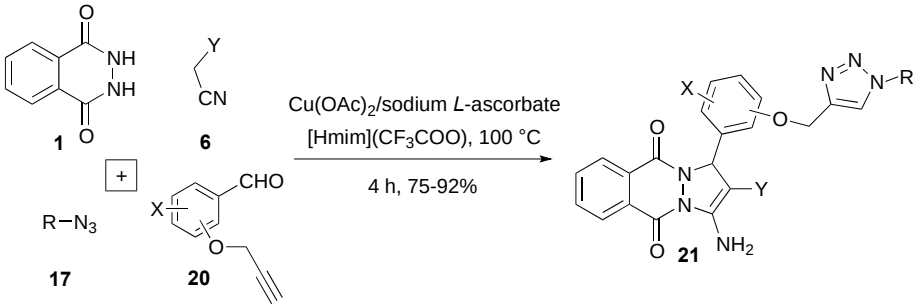
The plausible mechanism for the preparation of product **19** was outlined in scheme 8. The Knoevenagel condensation of aromatic aldehydes **7** and active methylene compound **18** produced the intermediate **A**, which was reacted with azide **17** *via* a 1,3-dipolar cycloaddition reaction to give 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**

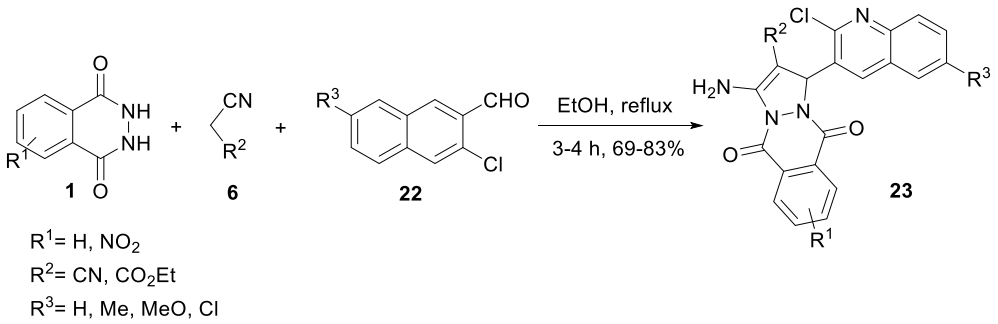
In another study, the synthesis of 1-[(triazolylmethoxy)phenyl]-1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives **21** *via* a four-component reaction of phthalhydrazide **1**, (propargyloxy)benzaldehyde **20**, an active methylene reagent **6** (malononitrile or ethyl

cyanoacetate), and an azide **17** using $Cu(OAc)_2$/sodium *L*-ascorbate as catalyst and 1-methyl-1*H*-imidazolium trifluoroacetate ([Hmim](CF_3COO)) as an ionic liquid medium was reported by the same group (Scheme 9).[84] 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**

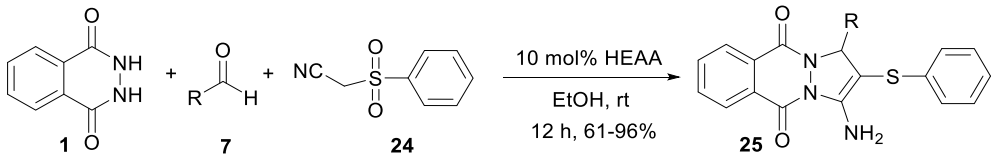
Patel and co-workers have demonstrated a simple and efficient method for the synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives **23** through the one-pot three-component condensation reaction of phthalhydrazide **1**, 2-chloro-3-formylquinolines **22**, and malononitrile or ethyl 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).[85]



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

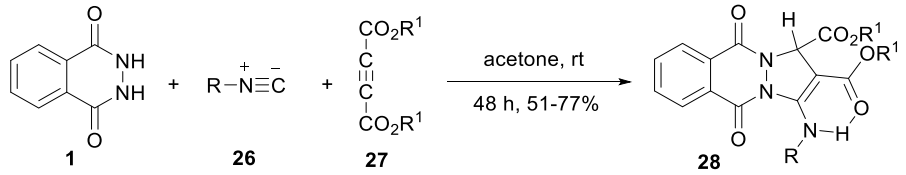
(phenylsulfonyl)acetonitrile **24** in the presence of 2-hydroxyethylammonium acetate (HEAA) as catalyst in EtOH (Scheme 11).[86] The advantages of this method include easy work-up, environmental friendliness, and good to excellent yields. Also, the photophysical properties of these products were studied,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



R= C_6H_5, 4-ClC_6H_4, 4-FC_6H_4, 3-BrC_6H_4, 4-$O_2NC_6H_4$, 4-MeC_6H_4, 4-HOC_6H_4
4-$MeOC_6H_4$, 4-isopropyl-C_6H_4, Me, *n*-C_3H_7, *iso*-C_3H_7, *iso*-C_4H_9

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

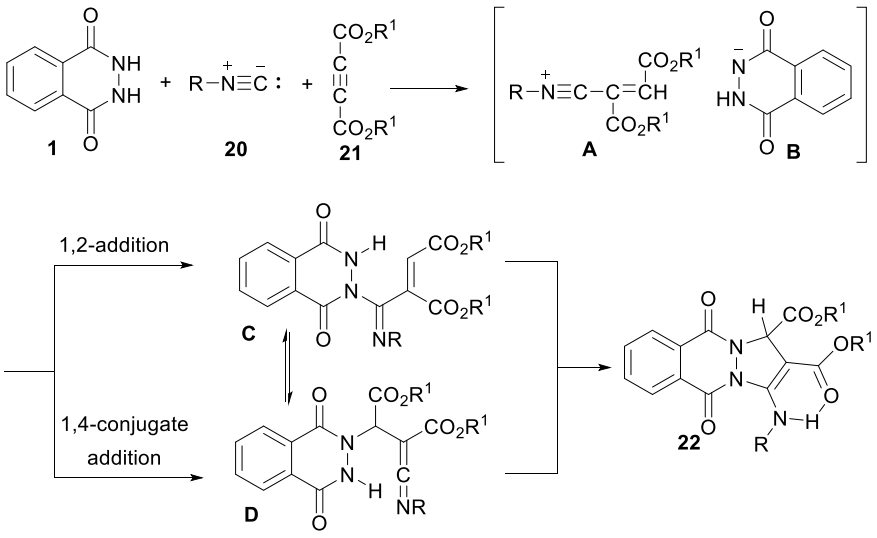
In another study, the various of 3-(alkylamino)-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-1,2-dicarboxylate derivatives **28** were synthesized through the reaction of phthalhydrazide **1**, alkyl isocyanides **26** with dialkyl acetylenedicarboxylates **27** at room temperature in dry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



R= *tert*-octyl, *tert*-butyl, cyclohexyl
R^1= Me, Et, *tert*-butyl

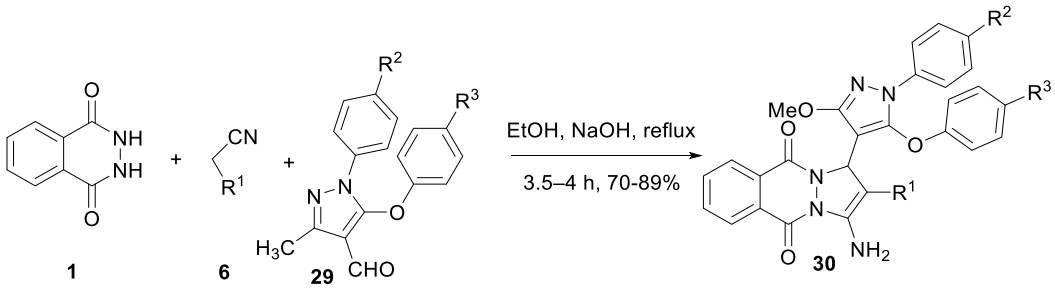
Scheme 12. The synthesis of dialkyl 3-(alkylamino)-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-1,2-dicarboxylate derivatives **28**

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**.



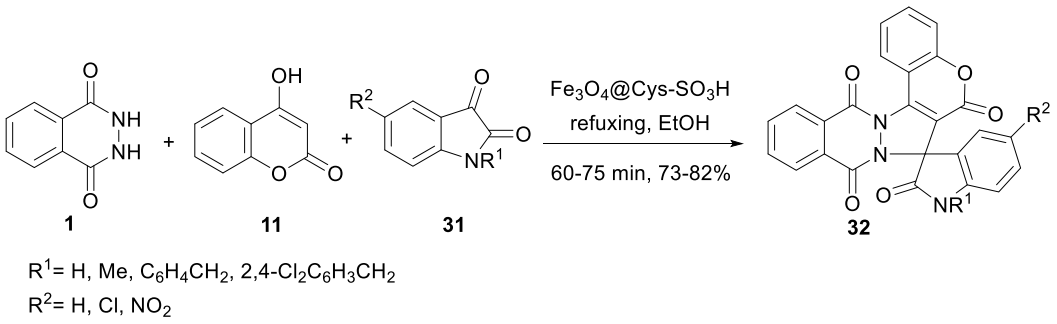
Scheme 13. Proposed mechanism for the formation of compound **28**

The synthesis of new series of pyrazolo[1,2-*b*]phthalazine derivatives **30** bearing the 5-aryloxypyrazole 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).[88] The required starting material of 1-aryl-5-chloro-3-methyl-1*H*-pyrazole-4-carbaldehyde **29** was prepared using reported methods.[89] All the products have been screened *in vitro* antibacterial, antitubercular, cytotoxicity and antioxidant activities.



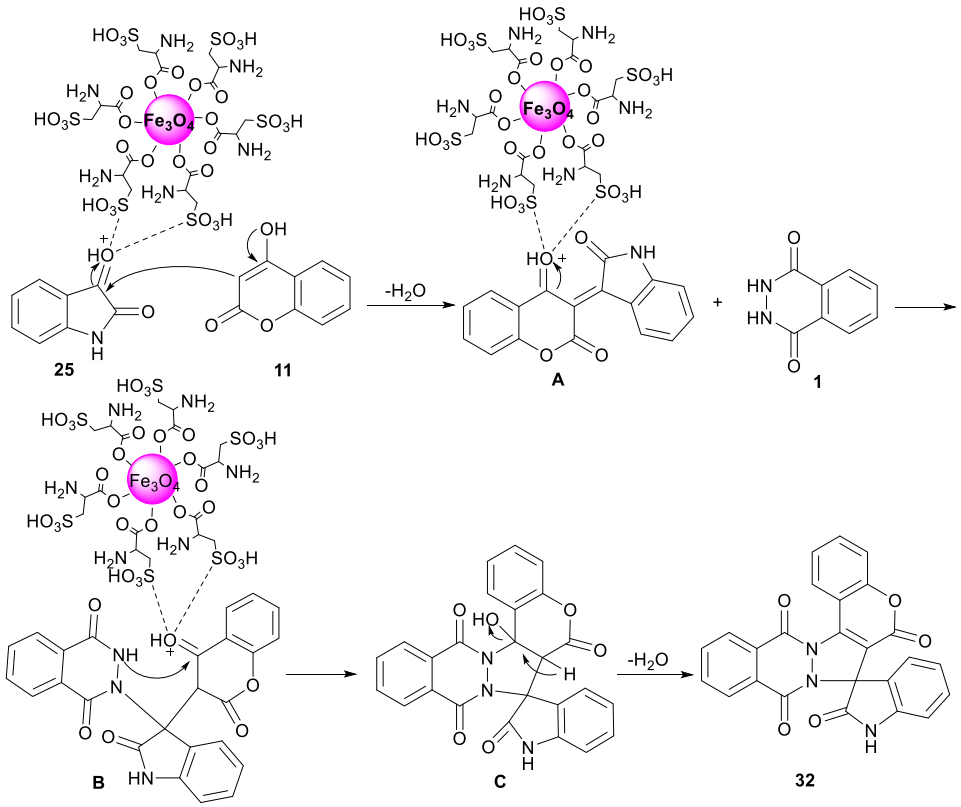
Scheme 14. The synthesis of novel series of pyrazolo[1,2-*b*]phthalazine derivatives **30**

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 refuxing EtOH in the presence of Fe_3O_4@Cys-SO_3H magnetic nanocatalyst was reported by Kefayati *et al*. (Scheme 15).[90]



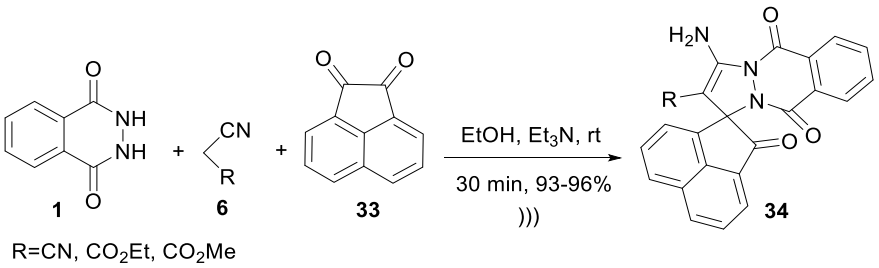
Scheme 15. The synthesis of *spiro*-[chromeno[4′,3′:3,4]pyrazolo[1,2-*b*]phthalazine-7,3′-indoline]-2′,6,9,14-tetraone **32**

A proposed mechanism for the synthesis of compound **32** is shown in Scheme 16. The sulfonic groups (SO_3H) 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**.



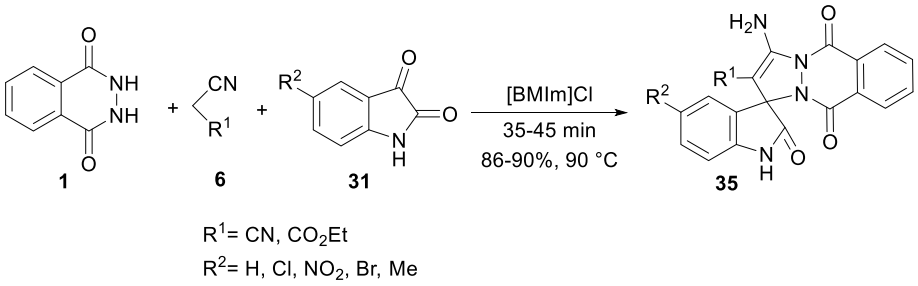
Scheme 16. Plausible reaction mechanism for the synthesis *spiro*-[chromeno[4′,3′:3,4]pyrazolo[1,2-*b*]phthalazine-7,3′-indoline]-2′,6,9,14-tetraone **32**

In another study, Nabid and his co-workers developed an efficient procedures 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_3N as catalyst at room temperature (Scheme 17).[91] 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.



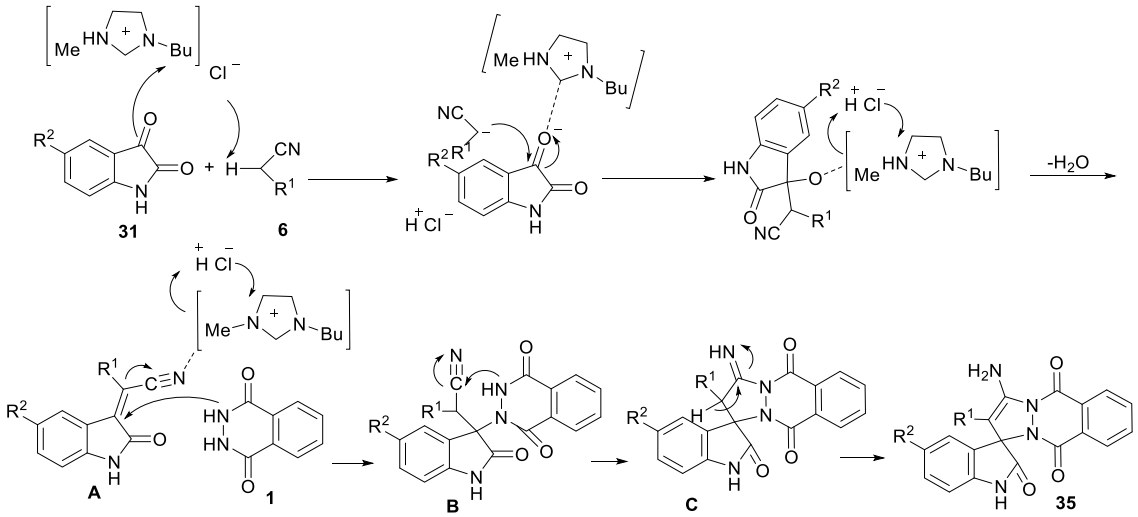
Scheme 17. The synthesis of *spiro*-acenaphthylene-1,1′-pyrazolo[1,2-*b*]phthalazines **34**

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).[92]



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,[93] *L*-proline,[94] piperidine,[95,96] silica bonded *N*-propyl-sulfamic acid (SBNPSA),[97] [DBU][AcOH],[98] $NiCl_2$,[99] as catalysts in these reactions as summarized in Table 3.

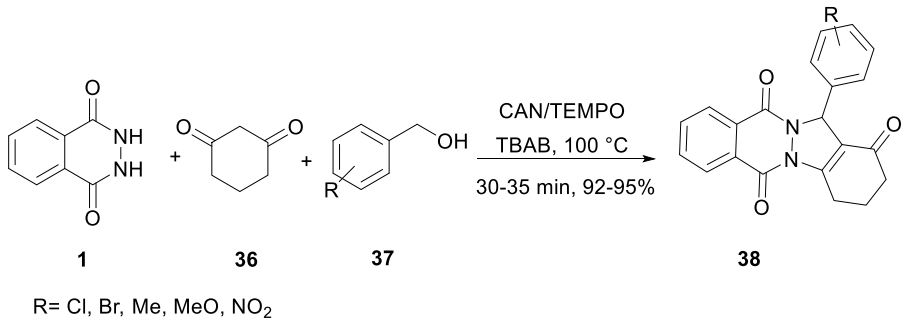
Table 3. Comparison of different conditions for the synthesis of products **35**

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	*L*-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

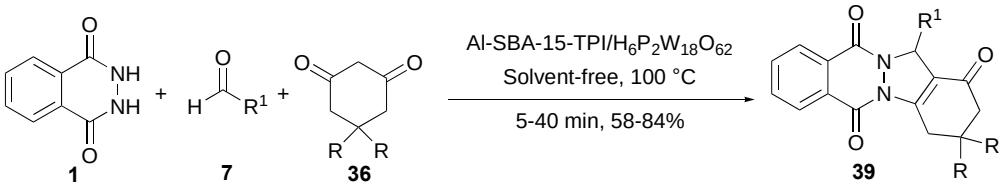
A metal-free synthesis of 2*H*-indazolo[1,2-*b*]phthalazine-triones derivatives **38** using phthalhydrazide **1** with 1,3-diketones **36** and benzyl alcohol **37** *via* 2,2,6,6-tetramethyl-piperidinyl-1-oxy (TEMPO) as a radical initiator, and ceric ammonium nitrate (CAN) as an oxidation reagent in 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).[100]



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/$H_6P_2W_{18}O_{62}$ 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).[101]



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/$H_6P_2W_{18}O_{62}$	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_2	reflux	10-30	86-96	[104]

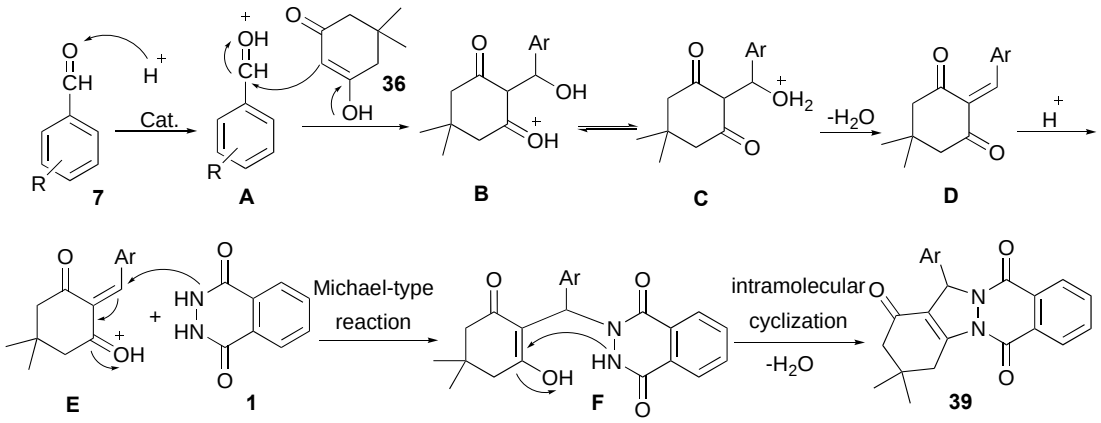
5	—	I_2	ultrasound	10-15	87-95	[105]
6	glycerol	PSSA	80 °C	40-70	80-92	[106]
7	—	$H_{14}[NaP_5W_{30}O_{110}]/SiO_2$	reflux	6-15	80-94	[107]
8	—	MTSA	reflux, 100 °C	10-20	78-92	[108]
9	—	$KHSO_4$-SiO_2	80 °C	10	87-94	[109]
10	H_2O/phenol	*O*-phosphoric acid	reflux,70 °C	4-20	84-94	[110]
11	—	$H_4PMo_{11}V_1O_{40}$/K-10	100 °C	16-38	86-98	[111]
12	—	[Dsim][HSO_4]	100 °C	15-20	76-97	[112]
13	—	PPA–SiO_2	100 °C	4-24	78-93	[113]
14	EtOH	$Fe_2(SO_4)_3 \cdot xH_2O$	reflux	2-4 h	80-93	[114]
15	—	[SuSA-H]HSO_4	100 °C	5-70	88-97	[115]
16	—	SO_3H-FMSM	110 °C	15-25	90-95	[116]
17	—	β-cyclodextrine-SO_3H	80 °C	10-30	85-95	[117]
18	—	MNPs-guanidine	70 °C	30-65	80-93	[118]
19	—	$[Cu(CH_3CN)_4]PF_6$	100 °C	10-15	79-92	[119]
20	—	$[C_4(mim)_2](FeCl_4)_2$	100 °C	10-15	86-91	[120]
21	EtOH	Fe_3O_4@GO-Pr-SO_3H	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_3H] HSO_4	80 °C	6-30	85-96	[124]
26	—	[H-Suc]HSO_4	80 °C	7-30	89-97	[125]
27	—	NS-[C_4(DABCO-$SO_3H)_2$]·4Cl	80 °C	5-30	88-96	[126]
28	—	$NiFe_2O_4$@TiO_2-SiO_2-Pr-DEA-OSO$_3$H	90 °C	5-30	83-97	[127]
29	—	$NiFe_2O_4$@TiO_2-SO_3H	80 °C	5-35	87-95	[128]
30	—	PTA@Fe_3O_4/EN-MIL-101	100 °C	20-80	50-94	[129]
31	H_2O	$[PhBS]_3^+PW_{12}O_{40}^{3-}$	reflux	5-20	80-97	[130]
32	—	Fe_3O_4@SiO_2–$ZrCl_2$-MNPs	110 °C	9-30	85-95	[131]
33	H_2O/EtOH	H_2SO_4	reflux	25-30	80-93	[132]

34	[bmim]BF_4	H_2SO_4	80 °C	30-40	83-94	[132]
35	—	nano-alumina sulforic acid	110 °C	10-20	70-98	[133]
36	—	dodecylphosphonic acid	80 °C	5-15	79-96	[134]
37	—	SnO_2 NPs	80 °C	12-22	84-96	[135]
	—	SBA-Pr-NH_2	80 °C	5-25	70-92	[136]
38	—	*N*-propylsulfamic acid functionalized	90 °C	10-25	85-98	[137]
39		[PVPH]ClO_4	100 °C	4-45	90-97	[138]
40	—	$MnFe_2O_4$@SiO_2NH–NH_2–PTA	80 °C	20-30	88-96	[139]
41	—	$MnFe_2O_4$@SiO_2NH–NH_2–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	70-220	89-95	[141]
44	—	PEG-MDIL	100 °C	10-15	85-90	[142]
45	EtOH	SBA-15/SO_3H	ultrasound, 45 °C	15-20	92-96	[143]
46	TFE	SBA-15	65 °C	140-180	89-94	[144]
47	—	$[C_4(H\text{-}DABCO)_2][HSO_4]_4$	80 °C	7-20	87-96	[145]
48	—	caffeine-H_2SO_4	100 °C	10-20	88-96	[146]
49	—	Ni NPs	80 °C	10-15	88-94	[147]
50	—	*p*-toluenesulfonic acid	80 °C	10-20	80-93	[148]
51	—	γ-Fe_2O_3@Starch-n-Butyl SO_3H	100 °C	13-38	83-98	[149]
52	—	silica sulfuric acid	100 °C	7-35	80-91	[150]
53	—	H_3PO_4-Al_2O_3	100 °C	8-23	76-93	[151]
54	—	$DABCO(HSO_3)_2(HSO_4)_2$	80 °C	4-30	83-95	[152]
55	—	boron sulfonic acid	100 °C	8-20	86-96	[153]
56	—	[Simp]HSO_4	100 °C	10-40	55-88	[154]

57	—	$[Simp]_3PW_{12}O_{40}$	100 °C	15-80	66-92	[155]
58	EtOH	$Y(OTf)_3$	80 °C	30-50	76-86	[156]
59	—	Montmorillonite K-10	MW, 80 °C	5-13	90-96	[157]
60	—	SBA-15-Ph-SO_3H	80 °C	5-15	85-96	[158]
61	—	wet cyanuric chloride	100 °C	10-25	89-97	[159]
62	MeOH	ChCl/*p*-TsOH	reflux	4 h	83-93	[160]
63	—	$[(CH_2)_4SO_3HMIM]$ $[HSO_4]$	100 °C	10	55-86	[161]
64	—	citric acid	100 °C	10-30	81-92	[162]
65	—	$ZrOCl_2.8H_2O$	80 °C	60	80-88	[163]
66	—	$[Et_3N\text{-}SO_3H]HSO_4$	90 °C	5-10	90-98	[164]
67	—	silica-supported tungstic acid	80 °C	30-50	87-94	[165]
68	—	SBA-Pr-SO_3H	80 °C	5-15	70-90	[166]
69	—	$H_4SiW_{12}O_{40}$	100 °C	10-25	83-96	[167]
70	PEG 600	$[BSO_3HmIm]HSO_4$	120 °C	30-40	80-95	[168]
71	—	IL@nano-SiO_2	80 °C	10-20	77-96	[169]
72	—	(PMA)-SiO_2	80 °C	30-40	80-90	[170]
73	EtOAc	*p*-sulfonic acid calix[4]arene	MW, 130 °C	10	51-94	[171]
74	—	Cu(II)-adenine-MCM-41	100 °C	105-150	85-95	[172]
75	—	$Fe_3O_4@SiO_2$	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	—	$Fe_3O_4@Ca(HSO_4)_2$	110 °C	2-8	74-87	[177]
80	EtOH	$NiFe_2O_4@TiO_2$-Pr-DEA-OSO_3H	reflux	25-75	70-95	[178]
81	—	SO_3H-Fe_3O_4-SiO_2	80 °C	20-37	88-98	[179]
82	EtOH	$Y(OTf)_3$	80 °C	30-50	76-83	[180]

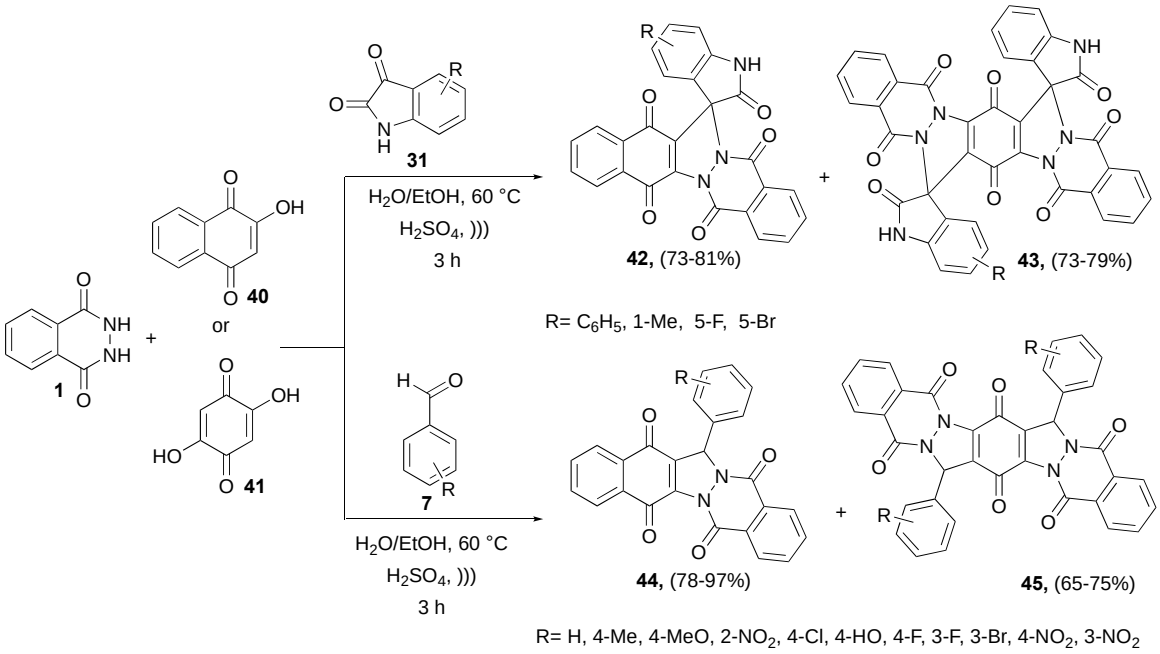
83	—	[PVP-SO_3H]Cl	80 °C	6-26	85-96	[181]
84	—	5-sulphosalicylic acid	100 °C	8-15	76-92	[182]
85	—	BAILs	110 °C	6-25	86-96	[183]

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^+ (from heteropolyacid) and carbonyl oxygen of the aromatic aldehyde **3**. 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_2O 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**.



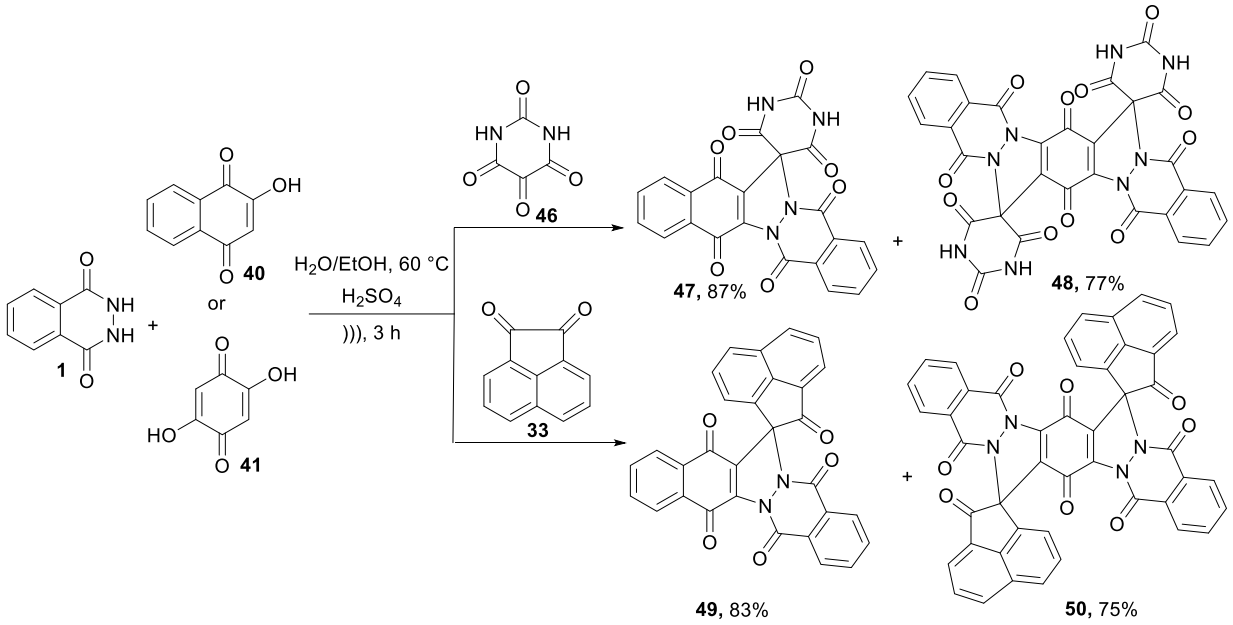
Scheme 22. Plausible mechanism for synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-trione derivatives **39**

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*-[benzoindazolophthalazine-indoline]pentaones **42**, *bis*-*spiro*-benzoindazolophthalazine-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_2SO_4 under ultrasonic irradiation to provide the indazolophthalazine derivatives (Scheme 24).[184]

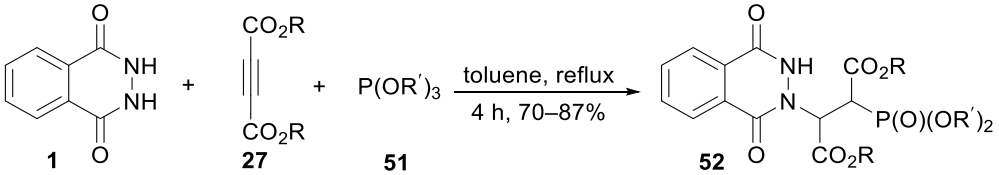


Scheme 24. Synthesis of indazolophthalazine **47-50**

4. THE SYNTHESIS OF PHTHALAZINE-SUCCINATE DERIVATIVES

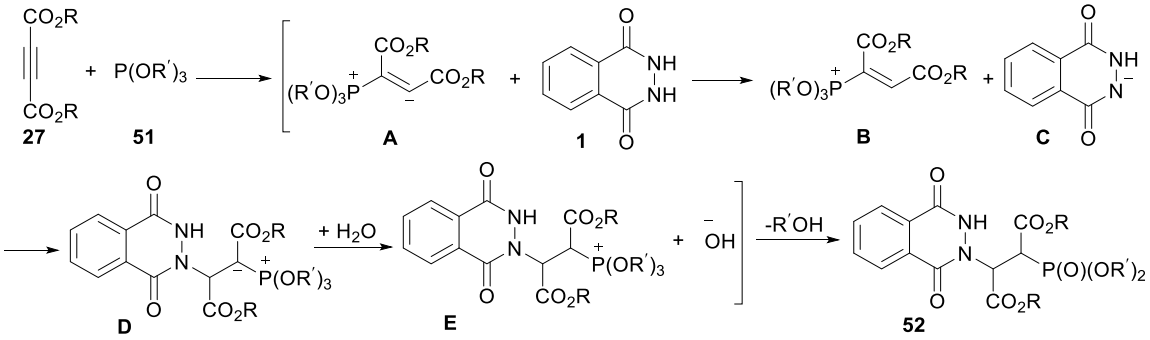
A series of dialkyl-2-(dialkoxyphosphoryl)-3-(1,4-dioxo-3,4-dihydrophthalazin-2(1*H*)-yl)succinate derivatives **52** were synthesized by Azizian and his co-workers in 2012. In this process, phthalhydrazide **1**

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



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.

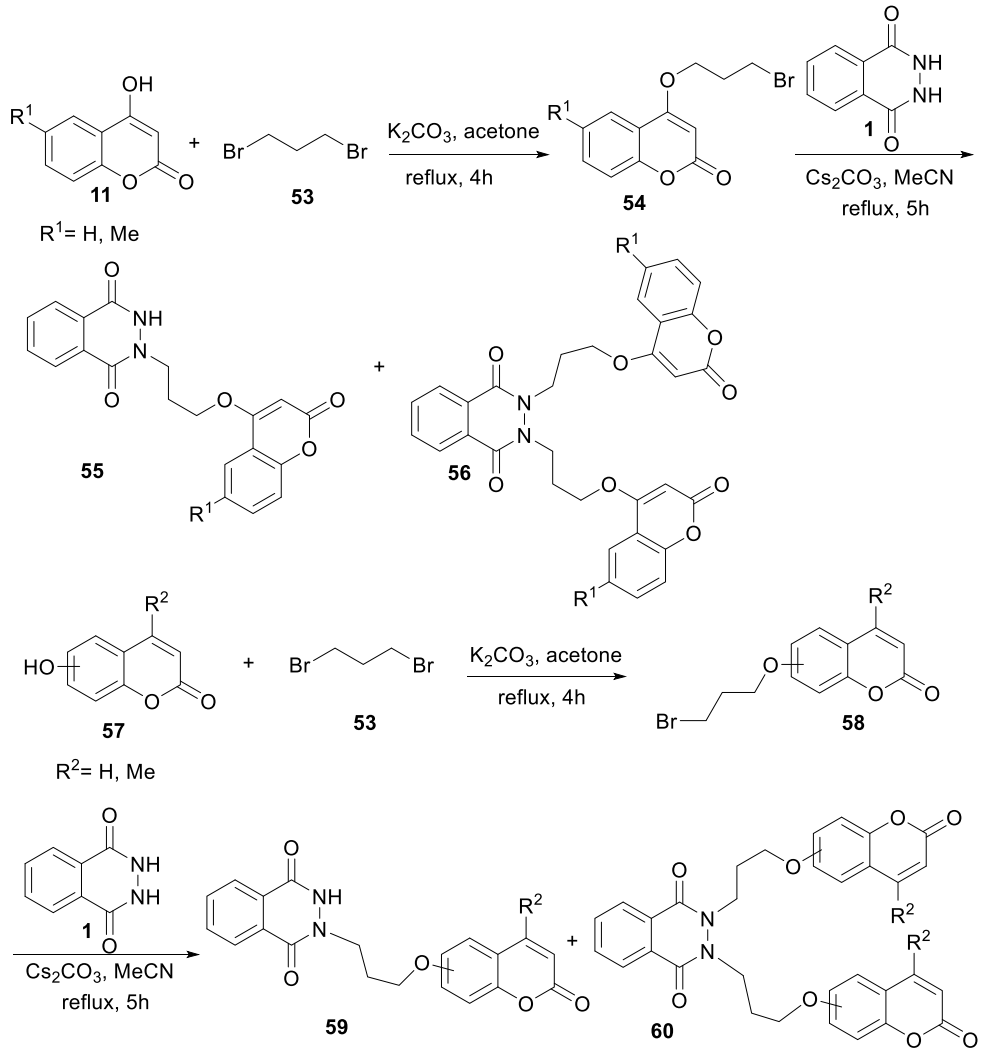


Scheme 26. Plausible mechanism for the synthesis of dialkyl 2-(dialkoxyphosphoryl)-3-(1,4-dioxo-3,4-dihydrophthalazin-2(1*H*)-yl)succinate derivatives **52**

5. MISCELLANEOUS REACTIONS

The reaction of phthalhydrazide **1** with bromopropoxycoumarin derivatives **54** in the presence of Cs_2CO_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_{2A}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).[186]

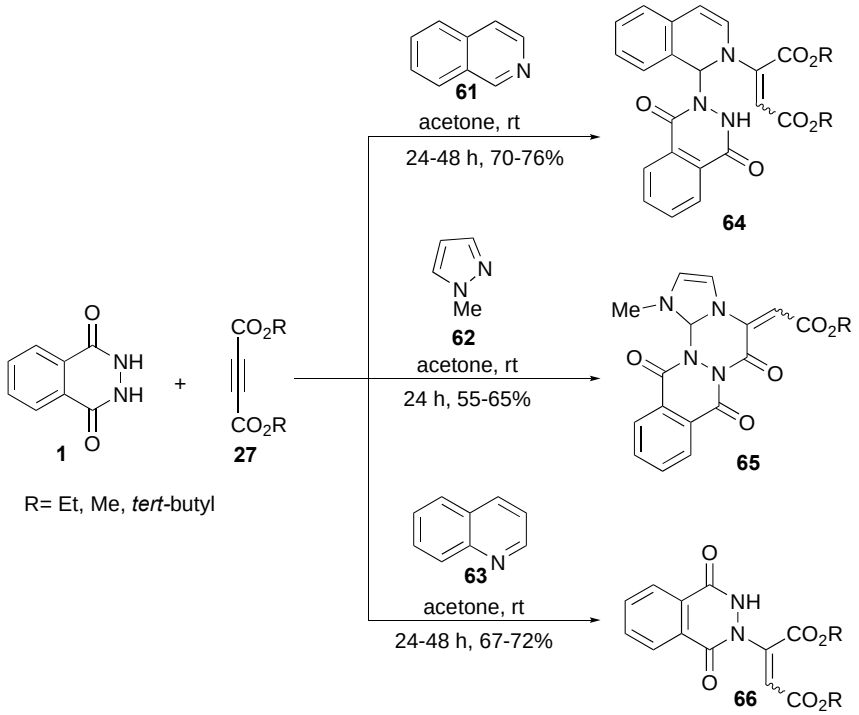


Scheme 27. The synthesis of phthalhydrazide-coumarin derivatives **55**, **56**, **59**, **60**

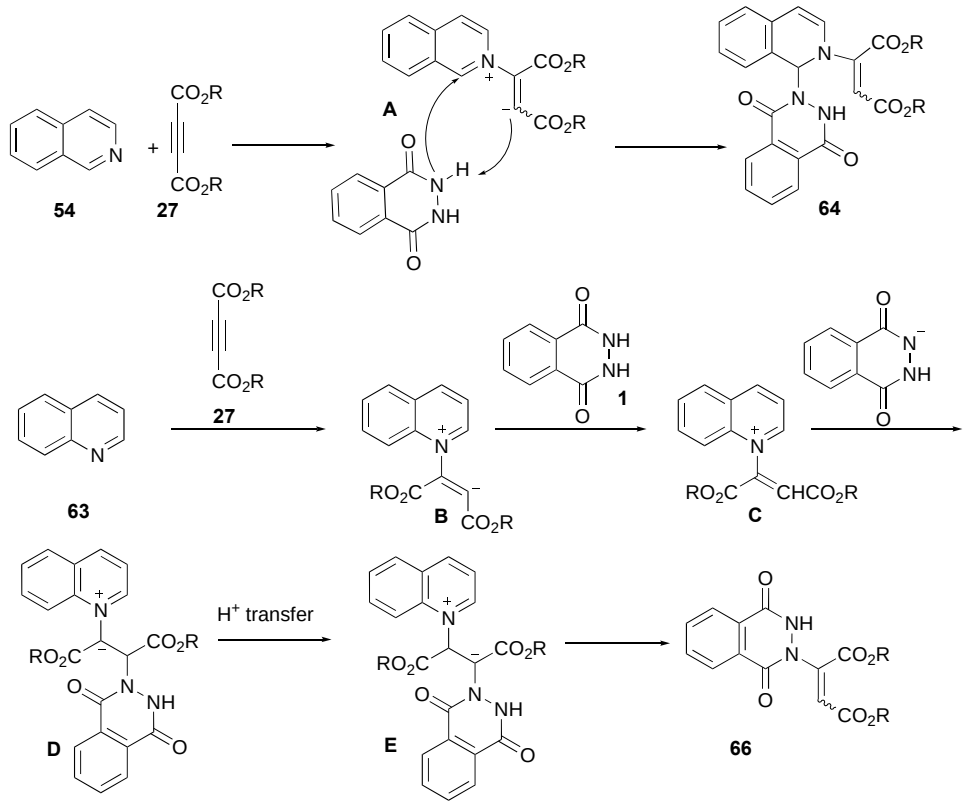
Ghahremanzadeh *et al*. prepared some new phthalazine derivatives through the reaction of phthalhydrazide **1** and dialkyl acetylenedicarboxylates **27** in the presence of isoquinoline **61**, *N*-methylimidazole **62**, and quinoline **63** as *N*-heterocycles compounds (Scheme 28).[187]

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).

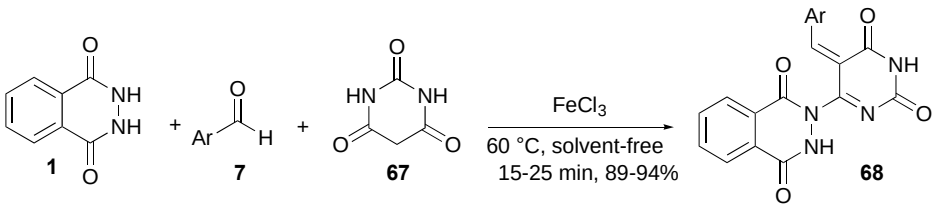


Scheme 28. Synthesis of some new phthalazine derivatives **64**, **65**, **66**



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% $FeCl_3$ under solvent-free conditions to provide (tetrahydro-2,6-dioxopyrimidin-4-yl)-2,3-dihydrophthalazine-1,4-dione derivatives **68** (Scheme 30).[188]



Ar= C_6H_5, 4-$EtOC_6H_4$, 4-$MeOC_6H_4$, 4-BrC_6H_4, 4-ClC_6H_4, 4-$O_2NC_6H_4$, 4-MeC_6H_4, 4-FC_6H_4, 4-HOC_6H_4, 3-BrC_6H_4, 3-MeC_6H_4 3-FC_6H_4, 3-$O_2NC_6H_4$, 2-FC_6H_4, 2-BrC_6H_4, 2-MeC_6H_4, 3,4,5-$(MeO)_3C_6H_2$, 2-thienyl, 4-*i*-PrC_6H_4, 1,3-Benzodioxole-5-carbaldehyde

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

This reaction in the presence of 1-ethyl-3-methylimidazolium tetrafluoroborate ([Emim][BF_4]) IL as a solvent and as well as an efficient catalyst was also reported (Table 5).[189] 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	—	$FeCl_3$	60 °C	15-25	89-94	[188]
2	—	[Emim][BF_4]	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.

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ABBREVIATIONS

[Bmim]OH	1-butyl-3-methylimidazolium hydroxide
BAILs	brønsted acid ionic liquid
[BMIm]Cl	1-butyl-3-methylimidazolium chloride
$[BSO_3HmIm]HSO_4$	4-(3-methyl-1-imidazolio)-1-butanesulfonic acid hydrogen sulfate
CPTES	3-chloropropyltriethoxysilane
CAN	ceric ammonium nitrate
ChCl	choline chloride
$[C_4(mim)_2](FeCl_4)_2$	1,4'-(butane-1,4-diyl)*bis*(3-methylimidazolium)*bis*-[tetrachloroferrate(III)]
DABCO	1,4-diazabicyclo[2.2.2]octane
Dsim	1,3-disulfonic acid imidazolium
DEA	diethanolamine
EN	ethylenediamine
FMSM	functionalized mesoporous silica materials
GOQDs	graphene oxide quantum dots
GO	graphene oxide
[HDEA][ClAc]	diethanolammonium chloroacetate
[Hmim]TFA	1-methyl-1*H*-imidazolium trifluoroacetate
HFIP	1,1,1,3,3,3-hexafluoro-2-propanol
MTSA	melaminetrisulfonic acid
$[MSPP]HSO_4$	4-methyl-1-(3-sulfopropyl)pyridinium hydrogen sulfate
NZF@HAP-Cs	caesiumcarbonate supported on hydroxyapatite-coated nickel zinc ferrite
PPA	polyphosphoric acid
PVP	poly(4-vinylpyrrolidonium)
PEG-MDIL	poly(ethylene glycol)bis(methylimidazolium dichloride)
PTA	phosphotungstic acid
PMA	phosphomolybdic acid
[PhBS]	phenanthrolinumbutane solfonate
PBBS	poly(*N*-bromo-*N*-ethylbenzene-1,3-disulfonamide)
RHA	rice husk ash silica
SiPMIM	(3-propyltrimethoxysilane)imidazolium
SBNPSA	silica bonded *N*-propylsulfamic acid
SuSA-H	succinimidinium *N*-sulfonic acid

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

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Ghodsi Mohammadi Ziarani was born in Iran, in 1964. She received her B.Sc. degree in Chemistry from Teacher Training University, Tehran, Iran, in 1987, her M.Sc. degree in Organic Chemistry from the Teacher Training University, Tehran, Iran, under the supervision of Professor Jafar Asgarin and Professor Mohammad Ali Bigdeli in 1991 and her Ph.D. degree in asymmetric synthesis (Biotransformation) from Laval University, Quebec, Canada under the supervision of Professor Chenevert, in 2000. She is Full Professor of Organic Chemistry in the chemistry department of Alzahra University. Her research interests include organic synthesis, heterocyclic synthesis, asymmetric synthesis, natural products synthesis, synthetic methodology and applications of nano-heterogeneous catalysts in multicomponent reactions.

Negar Jamasbi was born in Guilan, Iran. She received her B.Sc. and M.Sc. degrees in Applied and Organic Chemistry from Guilan University (2016 and 2018, respectively). She is Ph.D student under the supervision of Prof. Ghodsi Mohammadi Ziarani in Alzahra University in Tehran, Iran.

Fatemeh Mohajer was born in Tehran, Iran and she received her B.Sc. in Applied Chemistry from Bu-Ali Sina University and M.Sc degree in Organic Chemistry from Azad University in Karaj. She is Ph.D student under the supervision of Prof. Ghodsi Mohammadi Ziarani in Alzahra University in Tehran, Iran.