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Fused Heterocyclic Nitrogen Systems Containing Phosphorus Atom

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ABSTRACT

Synthesis of fused heterocyclic nitrogen systems containing phosphorus atom *via* [1+4] Cyclocondensation, [3+2] cycloaddition, and 1,5-electrocyclization reactions are reviewed. Also, the modified synthesis methodology applicable to the fused phospha-five and six-membered rings.

Key words: Synthetic, Fused, Phosphaheteocyclization, phosphorus.

INTRODUCTION

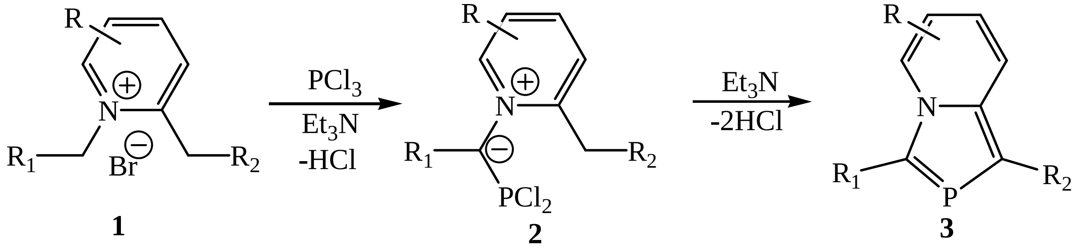
Organophosphorus compounds Research has steadily flourished[1] because these compounds have been reported to possess anti-TMV activity[2], herbicides[3,4], insecticides[5,6], molluscidal[7], and some of here useful as strong basic proazaphosphatrans[8], complexes[9,10] also as superior catalyst of the protective silyation of wide variety of sterically hindered and deactivated alcohols[11].

Fused and isolated heterocyclic nitrogen systems have been testified as anticancer[12,13] and plant protection[14] activities. In addition, phosphorus containing heterocyclic compounds exposed interesting biologically active[15, 16]. Thus, in the present review an attempt has been made to highlight the synthesis and properties of fused heterocyclic systems containing phosphorus atom.

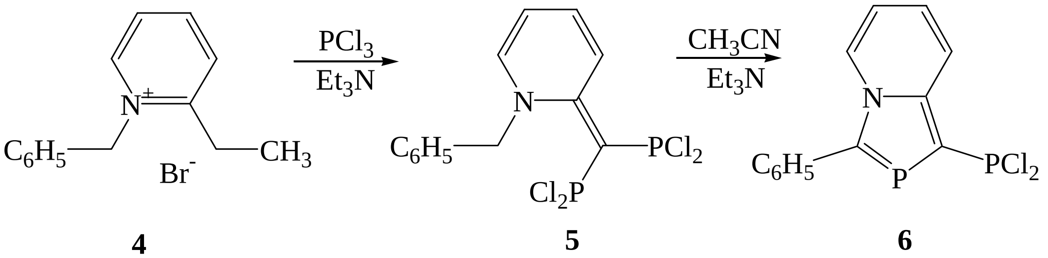
Distribution

The Synthetic strategy for new organophosphorus heterocyclic systems was analogous to the Krohnke's synthesis[17] *via* [4+1] cyclo condensation, [3+2] cycloaddition[18] and 1,5-electrocyclization[19]. Bansal *et al*[20,21] and abdel-Rahman[22] reported a brief reviews about the synthesis of indolizines and phosphaindolizines.

Synthesis *via* [4+1] cyclocondensation reactions are most important. Thus, synthesis of 2-phosphoindolizines (**3**) was deduced from condensation of 1,2-dialkylpyridinium bromide (**1**) with PCl_3 in presence of Et_3N *via* intermediate **2** [Scheme 1][23, 24].



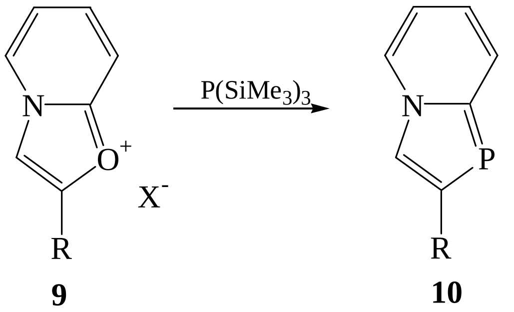
Scheme 1: Synthesis of 2-phosphoindolizines



Scheme 2: Synthesis of 1-dichlorophosphino-2-phosphoindolizine

If R^2 is methyl group, that reaction proceeds through the intermediate **5** to yield 1-dichlorophosphino-2-phosphoindolizine (**6**) [Scheme 2][25].

Similarly, 1,3-azaphospholo[5,1-b]thiazolines (**7**) and 1,3-azaphos-pholo[5,1-b]benzothiazoles (**8**) were synthetic[26].

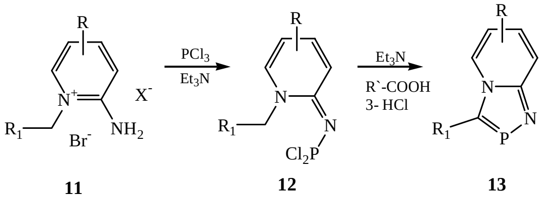


A large number of 3-aza-2-phosphaindolizines (**15**) were prepared[29] from condensation of 2-alkyl-1-aminopyridinium iodides (**14**) with PCl_3 in presence of Et_3N.

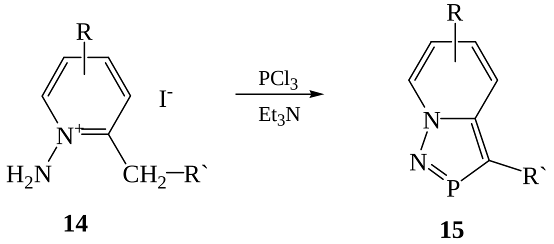
Also, 3-aza-phosphaindolizines (**17**) have been synthetic *via* O/P exchange of 1,3,4-oxadiazolo [3,2-a]pyridinium salts (**16**) with $P(SiMe_3)_3$[30].

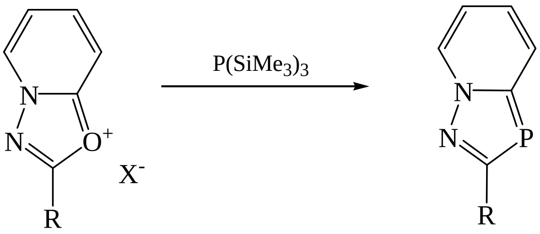
On the other hand, 1-phosphaindolizines (**10**) was obtained from condensation of 1,3-oxazolo[3,2-a]pyridinium salt (**9**) with $P(SiMe_3)_3$ *via* exchange oxygen by phosphorus[27].

A related condensation of 1-alkyl-2-aminopyridinium salt (**11**) with PCl_3 in presence of Et_3N led to the formation of 1-aza-2-phosphindolizines (**13**) *via* the intermediate **12.** [Scheme 3][28].



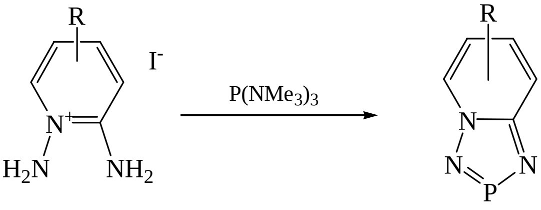
Scheme 3:





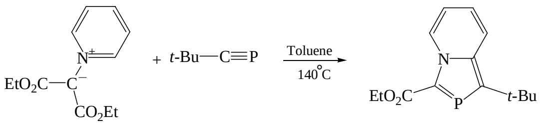
16 **17**

It is interesting that phospha-fused heterobicyclic as 1,3-diaza-2-phosphaindolizines (**19**) were obtained from cyclocondensation of 1,2-diaminopyridinium iodides (**18**) with P(NMe$_3$)$_3$ in boiling dry benzene[31].



18 **19**

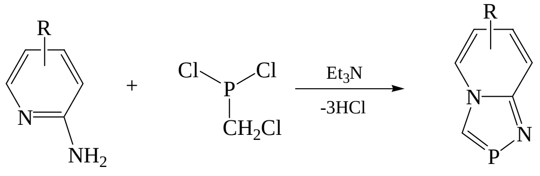
On the other hand, [3+2] cycloaddition reactions were used *tert*-butylphospha acetylene 1,3-dipolarophile to condense with pyridinium (**22**) to give 2-phosphaindolizine (**23**) by boiling in dry toluene[18].



22 **23**

Karaghiosoff *et al*[32] reported the using of chloromethyl dichloro-phosphine (ClCH$_2$PCl$_2$) as equivalent reagent yielding two-heterocyclic fused *via* C – P moiety by cyclocondensation with 2-substituted azoles and azines through [3+2]cyclocondensation reactions[33].

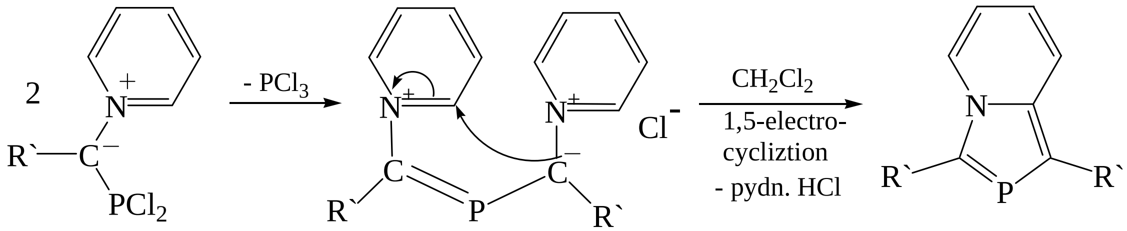
Thus, 2-aminopyridine (**20**) condenses with (ClCH$_2$PCl$_2$) in presence of Et$_3$N to produce 1,4,2-diazaphospholo[4,5-a]pyridine (**21**)[34].



20 **21**

Azomethine ylides and azomethine imines incorporate that structure of 1,3-dipolar moiety bonded directly to an olefinic or acetylenic bond possesses 1,5-dipolar structure and have been utilized for the synthesis of five-membered heterocycles[19, 35]

Thus, disproportionation of *N*-pyridinium dichlorophosphonium-methylides (**24**) would generate the bis-(*N*-pyridiniumylidyl) phosphonium chloride (**25**) which on disproportionation *via* 1,5-electrocyclization by loss the pyridinium hydrochloride to yield 2-phosphaindolizine (**26**) [Scheme 4][36].



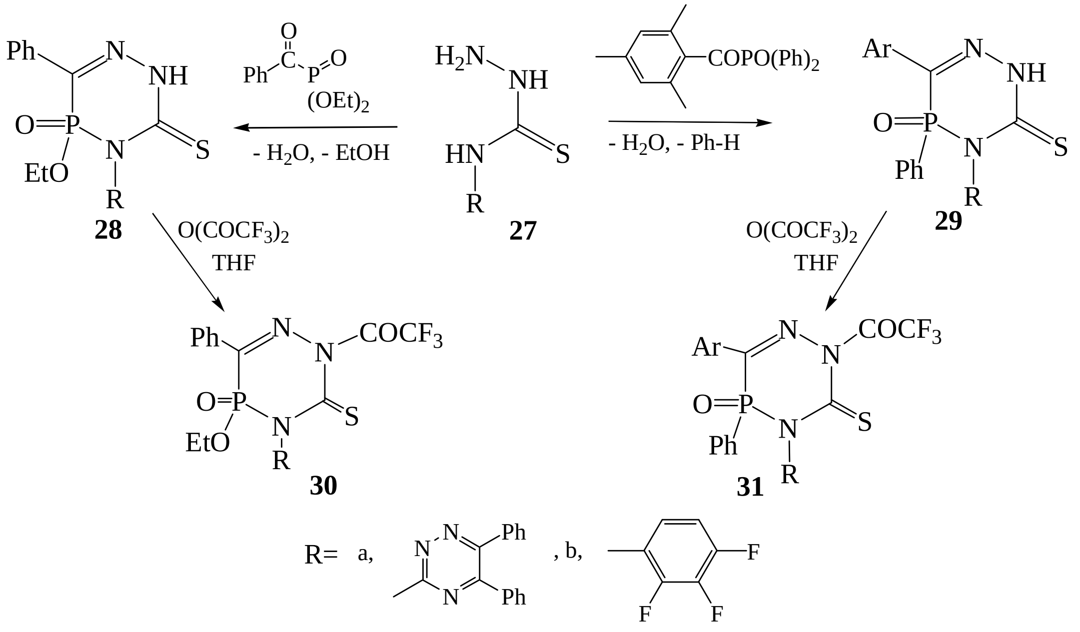
24 **25** **26**

Scheme 4:Synthesis of 2-phosphaindolizine

Abdel-Rahman[22], reported the synthesis of new phosphaheterobi-cyclic system containing 1,2,4-triazine moiety *via* heterocyclizationa, this in cloud a novel synthesis of fluorine bearing 5-phospha-1,2,4-triazin-3-thiones and 5-phospha-1,2,4-triazepin-3-thione.

Thus, a facile synthesis of fused

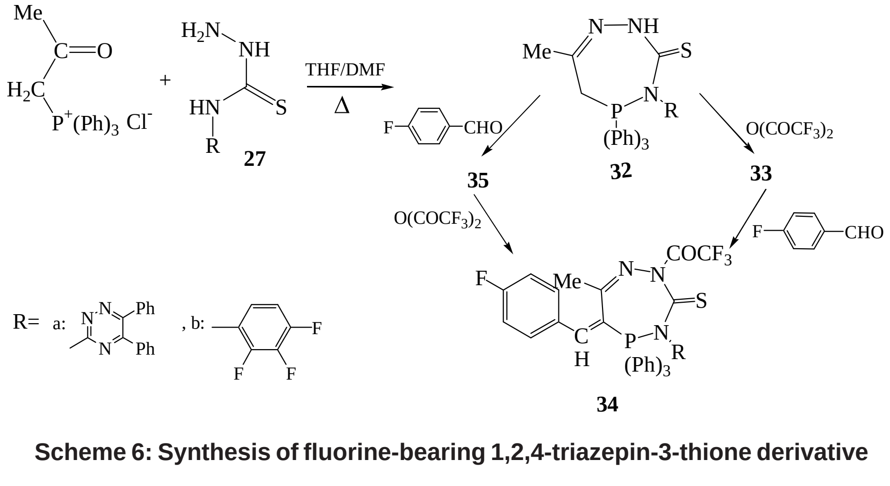
phosphaheterobi-cyclic nitrogen system containing 1,2,4-triazine moiety was describe earlier. The treatment *N*-substituted thiosemicarbazides[37] (**27**) with diethyl benzoyl phosphate and/or diphenyl(2,4,6-trimethyl benzoyl) phosphine oxide in boiling dry toluene or THF[38] afforded 4,5,6-trisubstitued-5-phospha-1,2,4-triazin-3(2H)thiones (**28** & **29**) respectively. Refluxing both the compounds **28** and **29** with trifluoroacetic anhydride yielded[39] the full fluorinated phospha-1,2,4-triazinethiones **30** and **31** respectively [Scheme 5][22].



Scheme 5: Synthesis of fused phosphaheterobi-cyclic nitrogen system containing 1,2,4-triazine moiety

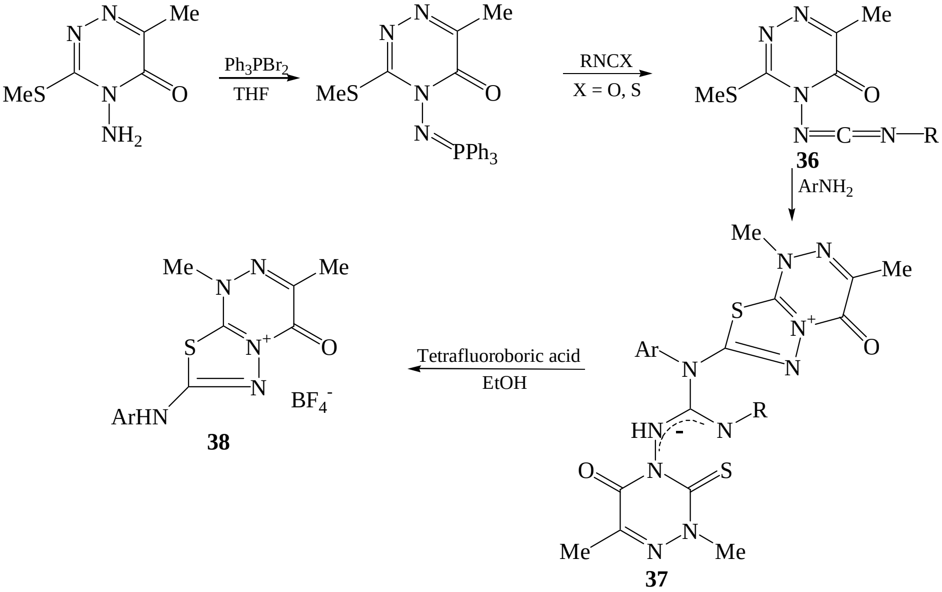
correspondingly, cyclocondensation of *N* 4-substituted thiosemicarbazide (**27**) with acetonyltriphenylphosphonium chloride in boiling THF-DMF[40] led to the direct formation of hexahydro-4,7-disubstituted-5-triphenyl-5-phospha-1,2,4-triazepin-3-(2H)thione (**32**)[22].

Presence of both NH and CH_2 groups in the compound **32** was deduced from acylation and or condensation with trifluorobenzaldehyde[40] to give the final products fluorine-bearing 1,2,4-triazepin-3-thione derivative (**34**) [Scheme 6][22].



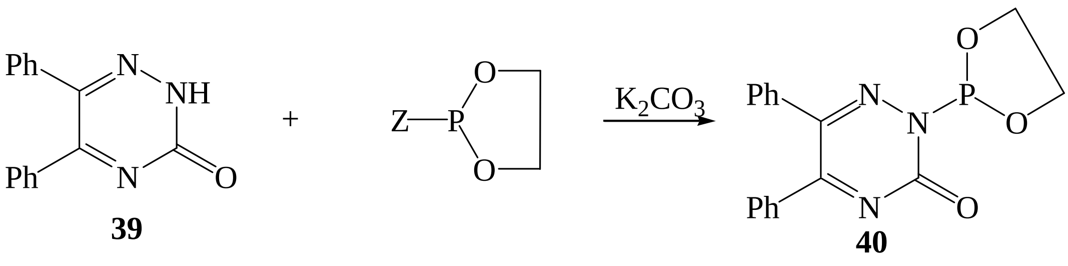
Scheme 6: Synthesis of fluorine-bearing 1,2,4-triazepin-3-thione derivative

Regioselective iminophosphoran-mediated annelation of a 1,3,4-thiadiazole ring into a 1,2,4-triazine ring was studied by Molina *et al*[42]. These studies based on aza-Wittig type reaction of iminophosphoane (**37**) followed by addition isothiocyanate-formed mesomeric or Zwitter ionic character **35** and **36**. Treatment of compound **36** with primary aromatic amines afforded the Betaine **37** which under treatment with ethanol in the presence of tetrafluoro boric acid by stirring for 7 hours, resulted the 1,3-thiadiazetidines **38** *via* carbodiimide under goes [2+2] cycloaddition reaction [Scheme 7][42].

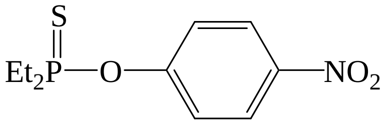


Scheme 7: Synthesis of 1,3-thiadiazetidines

From survey and various publications, it showed that a lethal work on the area of phospha-1,2,4-triazines[41-43]. The first study between phosphorus and 5,6-diphenyl-1,2,4-triazin-3(2H) one (**39**) and some phosphorus reagents such as TPP, $P(OR)_3$ and $(CH_2)_2O_2PZ$ (Z = Cl, OMe, $N(Me)_2$) were isolated phospha-heterocyclic system **40** obtained[43].



The pesticides are a group of chemicals intended for perveting/destroying production, processing storage transportation and distribution of food. Thus, thiophosphate pesticides are the most effective insecticides and are used to control a wide variety of insect's pest as Parathion[6] (**41**). Also the insecticide as Dimethaoate[5] (**42**).



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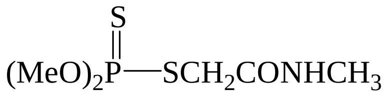
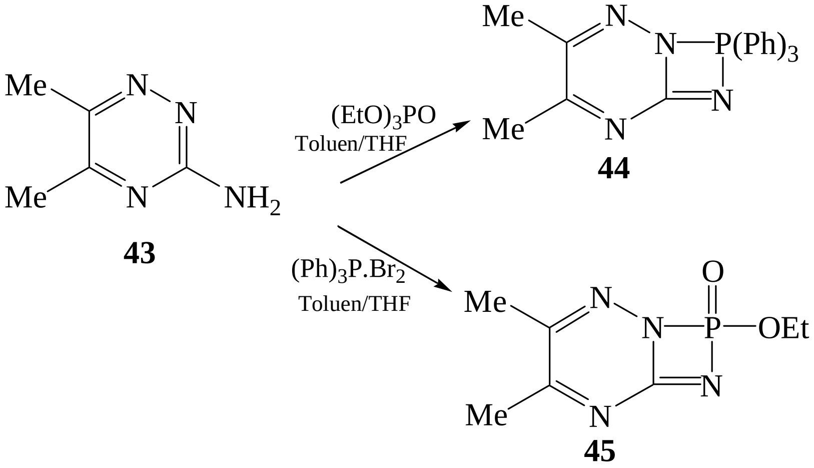


$(MeO)_2P(=S)—SCH_2CONHCH_3$

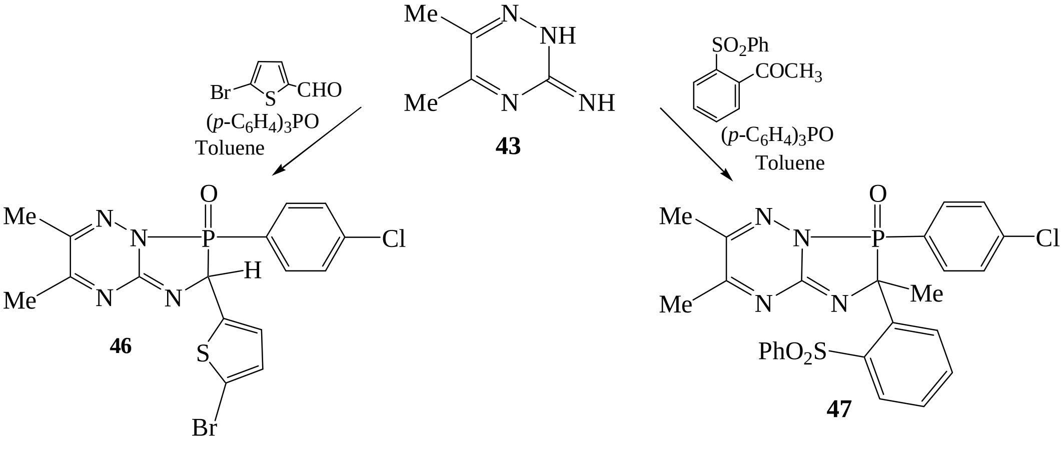
42

Several studies [12,13,44] found that fused heterobicyclic nitrogen systems have a wide spectrum of medicinal and biological activities. Thus, treatment[22] of 3-amino-5,6-dimethyl-1,2,4-triazine (**43**) with triphenylphosphine dibromide in toluene[38] afforded compound **44**, while refluxing of **43** with $(EtO)_3P=O$ in dry toluene[45] yielded compound **45** [Scheme 8][22].

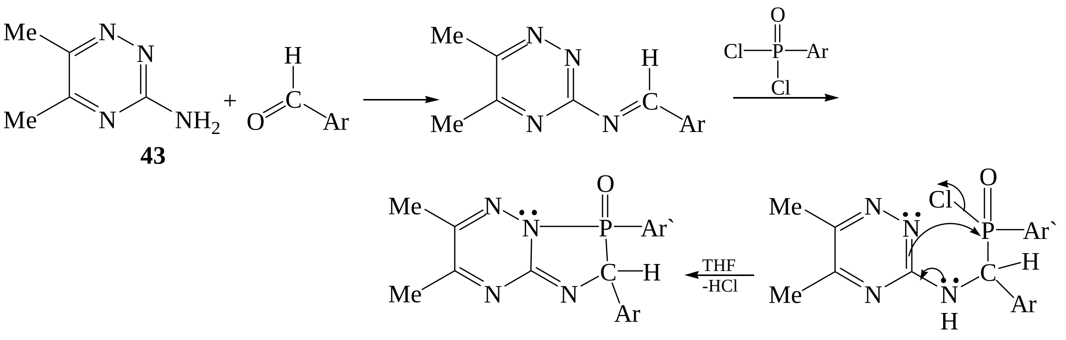


Scheme 8: Synthesis of fused heterobicyclic nitrogen systems

In addition, condensation of 3-amino-triazine **43** with aromatic aldehyde and (p-$ClC_6H_4)_3P$ and/or acetophenone/$ArPCl_2$ in presence of dry toluene[40,46]-TEA led to the direct formation of phospha heterocyclic systems **46** and **47** respectively [Scheme 9][23]. Formation of compound **46** may be takes place as shown in Scheme 10[47].

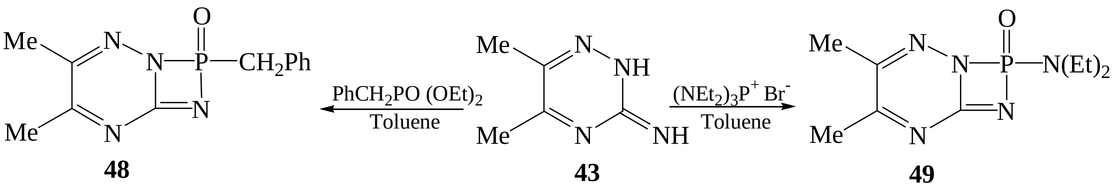


Scheme 9: Condensation of 3-amino-triazine 43 with aromatic aldehyde



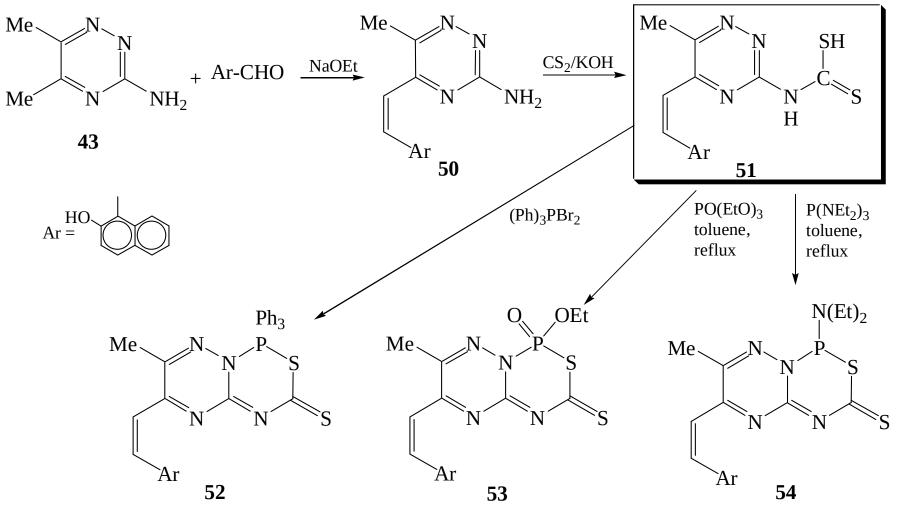
Scheme 10: Formation of compound 46

In addition, the reaction of 3-amino-5,6-dimethyl-1,2,4-triazine (**43**) with diethyl benzyl phosphonate and/or $(NEt_2)_3P^+$ Br^- in dry toluene produced the phospha heterocyclic systems **48** and **49** respectively [Scheme 11][23].



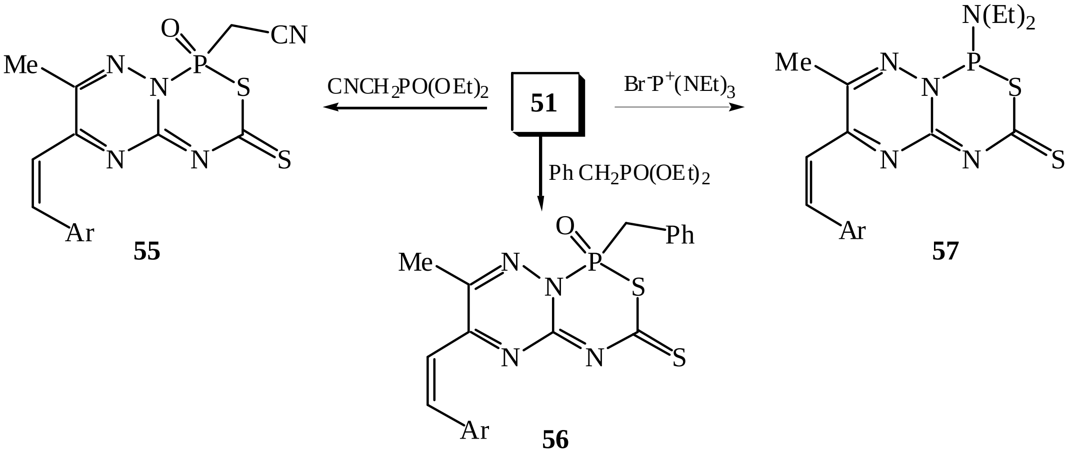
Scheme 11: Phospha heterocyclic systems

On the other hand, 3-amino-6-methyl-5-styryl-1,2,4-triazine (**50**) react with CS_2 in alcoholic KOH to give (6-methyl-5-styryl-1,2,4-triazin-3-yl)dithiocarbamic acid (**51**), which work as a building block for fused phospha-heterobicyclic systems **52-57** as bi-six-membered rings[22]. Thus, compound **51** on treatment with phosphorus reagents such as $(Ph)_3PBr_2$, $P(NR_2)_3$ and $(MeO)_3P{=}O$ in refluxing toluene-TEA furnished 1,2,4-triazino[2,3-c][1,2,5,2]thiadiapho-sphorin-6-thiones (**52**-**54**) respectively [Scheme 12][22].



Scheme 12: Synthesis of 1,2,4-triazino[2,3-c][1,2,5,2]thiadiapho-sphorin-6-thiones

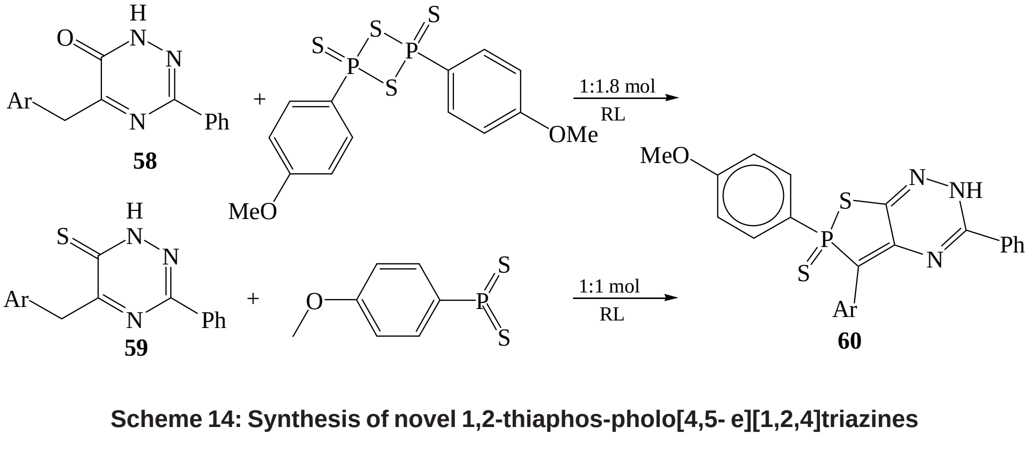
The behavior of marcapto group in compound **51** towards halogenated phosphorus reagents to produce compounds **55**-**57** is similar to its reaction with various alkylating agents and or ketonic agents. It is worthy of mention that nucleophilic attack on SH is more than NH group by chlorinated phosphorus reagents[48]. Thus, refluxing compound **51** with $CNCH_2P(O)(OEt)_2$, $PhCH_2P(O)(OEt)_2$ and $Br^- P^+(NEt_2)_3$ in THF-DMF furnished the target compounds **55**-**57** [Scheme 13][22].



Scheme 13: Synthesis of some marcapto group compound

The tautomeric structures and proton mobility in position 3 and 2 of 1,2,4-triazines considered to be important in heterocyclization process with halogenated phosphorus reagents. [22] Also, the reactivity structure relationship showed that phospha-1,2,4-triazine similar as Atranes which have unexpected properties[49].

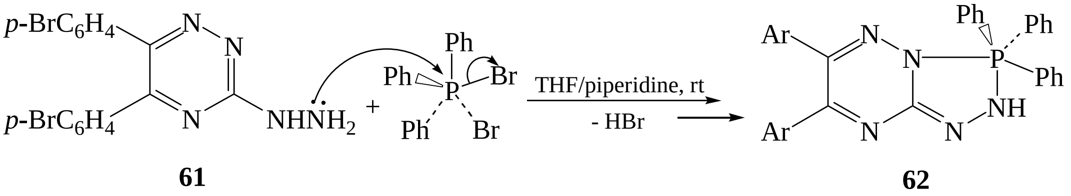
Ibrahim *et al*[50], reported the synthesis of novel 1,2-thiaphos-pholo[4,5- e][1,2,4]triazines (**60**) by treatment of 1,2,4-triazin-6-one (**58**) and or 1,2,4-triazin-6-thione (**59**) with different percentage of Lawesson's reagent (LR)[51] [Scheme 14] .



Scheme 14: Synthesis of novel 1,2-thiaphos-pholo[4,5- e][1,2,4]triazines

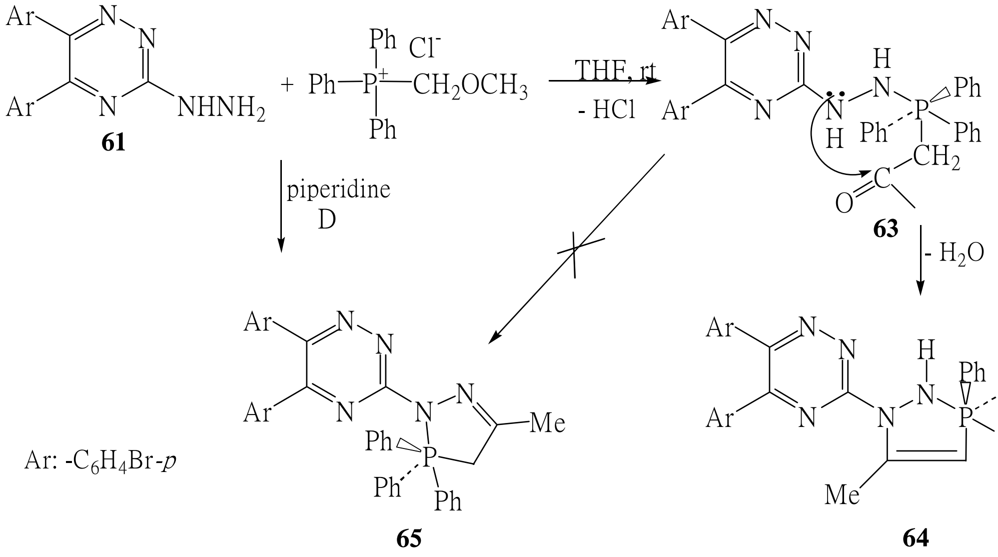
The molecular modifications of 1,2,4-triazine rings by introducing organophosphorus functionalities might be expected to exhibit the potential activities depending on the position of the phosphoryl group to 1,2,4-triazine ring, Abdel-Rahman *et al*[6], studied synthesis of phosphorus containing fused and isolated heterobicyclic nitrogen systems *via* reaction of 5,6-bis(4-

bromophenyl)-3-hydrazino-1,2,4-triazine (**61**) with some phosphorus reagents in non-polar solvent under various temperatures, the 6,7-diaryl-2,3-dihydro-3,3,3-triphenyl-1,2,4,-triazaphospholino[4,5-b][1,2,4]triazine (**62**) was synthesized by stirring compound **61** with dibromotriphenylphosphorane in THF at room temperature for 24 hours [Scheme 15][6]. Formation of **62** may be occurred *via* heterocyclization of the intermediate *N*[1],*N*[2]-disubstituted hydrazine. The ^{13}CNMR spectrum of compound **62** showed signal at ´ 29.61 ppm[52].



Scheme 15: Synthesis of 6,7-diaryl-2,3-dihydro-3,3,3-triphenyl -1,2,4,-triazaphospholino[4,5-b][1,2,4]triazine

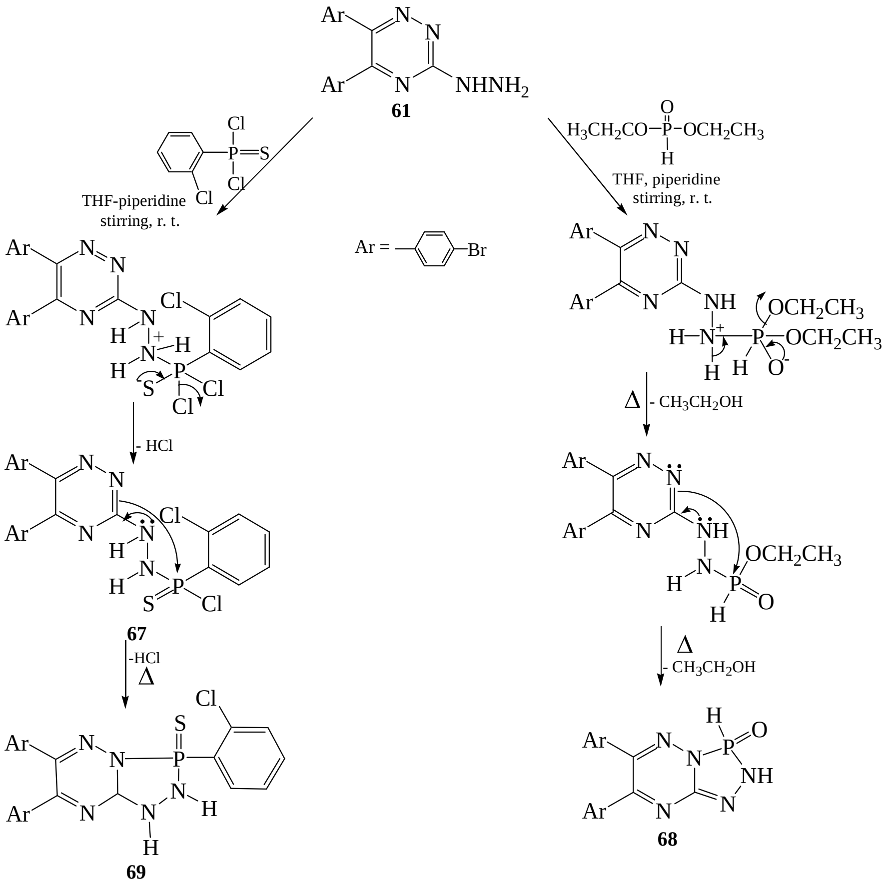
Phosphorylation of 3-hydrazino-1,2,4-triazine **61** *via* treatment with acetonyl triphenylphosphonium chloride under stirring in THF-piperidine for 24h, at room temperature furnished *N*[1],*N*[2]-disubstituted hydrazine **63**. Ring closure of **63** by heating above its melting point produced the heterobicyclic system **64** [Scheme 16][6]. On the other hand, that reaction when carried out under refluxing, the isomeric structure 5,6-bis-aryl-3-{(3,3,3-triphenyl)-5-methyl-3,4-dihydro-2H-1,2,3-*N*[5]-diazaphosphol-2-yl}-1,2,4-triazine (**65**) [Scheme 16][6].



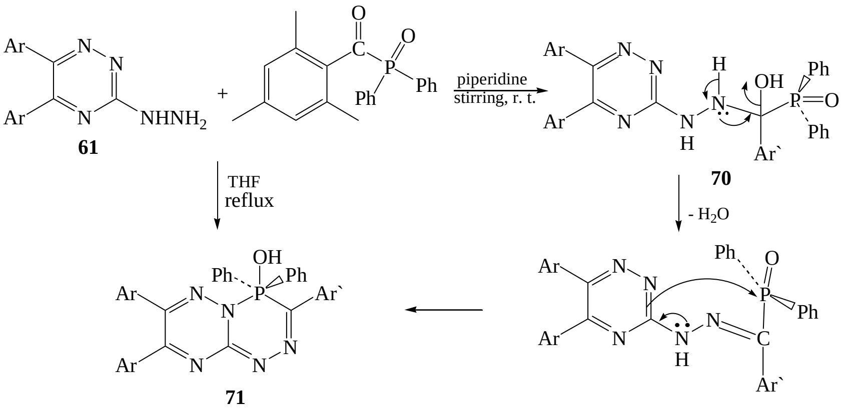
Scheme 16: Synthesis of some heterocyclic system

The treatment of 3-hydrazinotriazine **61** with diethylphosphite and/or 2-chlorophenyl dichlorothiophosphate in THF/piperidine at room temperature produced the phosphonohydrazide **66** and phosphono-hydrzidothionic acid **67** respectively [Scheme 17], while the reactions under reflux afforded directly the 1,2,4,3-triazaphospholo[4,5-b][1,2,4]triazines **68** and **69** respectively [Scheme 17]. ^{1}HNMR spectra of **68** and **69** showed signals of NH protons at ´10.92 and 10.34 ppm, respectively with respect to similar reported data[53-55].

Similarly, compound **70** isolated from treatment of **61** with diphenyl(2,4,6-trimethylbenzoyl)phosphorus oxide by stirring with THF at room temperature while 7,8-bisaryl-4,4-diphenyl-3-(2‘,4‘,6‘-trimethyl-phenyl)-4H-$4^5$1,2,4-triazino[3,2-c][1,2,4,5]triaza- phosphinin-4-ol (**71**) was isolated under refluxing [Scheme 18][6].Due to driving force of P=O bond is strong and phenyl groups are bad leaving groups, the nucleophilic attack of hydrazine moiety may be carried out at carbonyl group then P=O group[6].



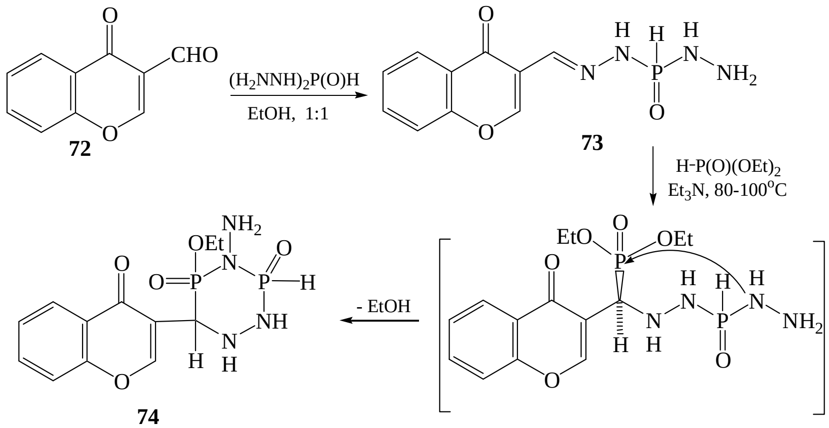
Scheme 17: Synthesis of 1,2,4,3-triazaphospholo[4,5-b][1,2,4]triazines 68 and 69



Scheme 18: Synthesis of compound 71

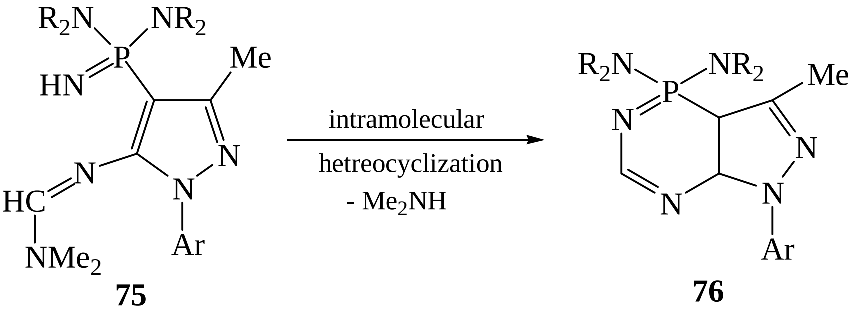
Phosphorus compounds with a ±-nitrogen or ±-oxygen more often exhibited interesting biological properties[56]. Thus, introducing of P-C-O or P-C-N pattern into heterocyclic systems may be enhancing their biocidal effects. Thus, compounds **62**-**71** showed strong effects on the tested snails, which due to facile donation and back donation in d-orbital of phosphorus atom[6].

Condensation of 4-oxo-4H-chromene-3-carboxaldehyde (**72**) with phosphonic dihydrazide (1:1 by moles) when heated in boiling ethanol yielded the mono-hydrazone **73**. Addition of diethyl phosphite to the azomethine bond of monohydrazone **73** by heating at 80-100ºC in TEA led to the direct formation of 3-(4-amino-5-ethoxy-3,5-dioxido-1,2,4,3,5-triazadiphosphinan-6-yl)4H-chromen-4one(**74**) [Scheme 19][57, 58].

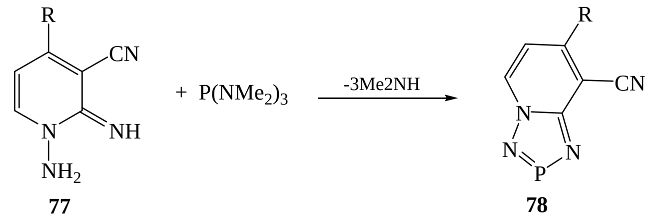


Scheme 19: Synthesis of 74

Pnickuk *et al*[59], reported that the intramolecular heterocyclization of polysubstituted pyrazole **75** afforded the fused phospha-heterobicyclic systems **76** *via* loss of dialkylamine.

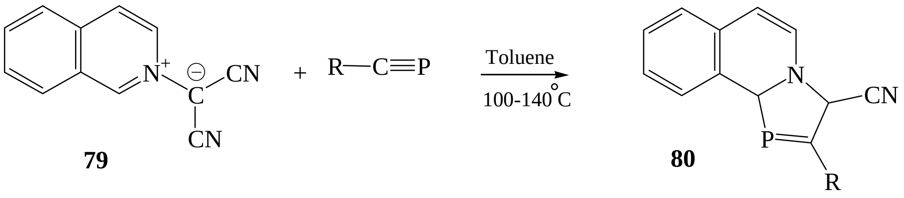


Functionally 1,2,4,3-triazaphospholopyridines (**78**) were obtained from [1+4] cycloaddition of 1-amino-2-imino-2H-pyridine-3-carbonitrile (**77**) with P(NMe_2)$_3$ in refluxing benzene[60].



In ^{13}CNMR spectra these compounds absorb further downfield in the range δ 265-218.

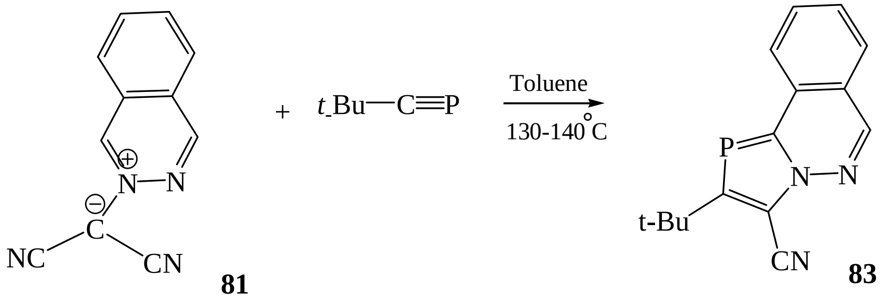
Also, *N*-isoquinolinium **79** reacts with R-CºP in boiling toluene (glass pressure tube)[61] to form the 1,3-azaphospholoquinoline derivatives **80**



79

80

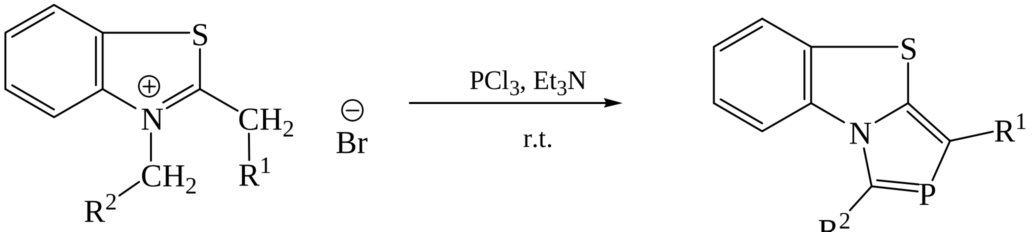
Under the same conditions, [3+2] cycloaddition of *N*-cycloimmoni-um ylide **81** with phosphaalkyne (**82**) has been successfully employed to synthetic of 1,3,4-azaphospholo[1,2-a]Phthalazine (**83**)[61].



81

83

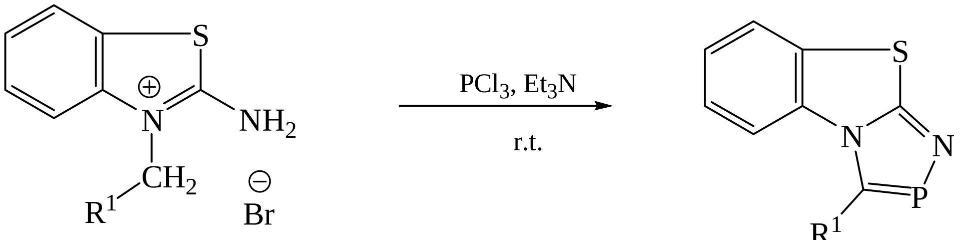
On the other hand, 2,3-dialkylbenzothiazolium bromides (**84**) when condense with PCl_3/ Et_3N led to the direct formation of compound **85**[62] .



84

85

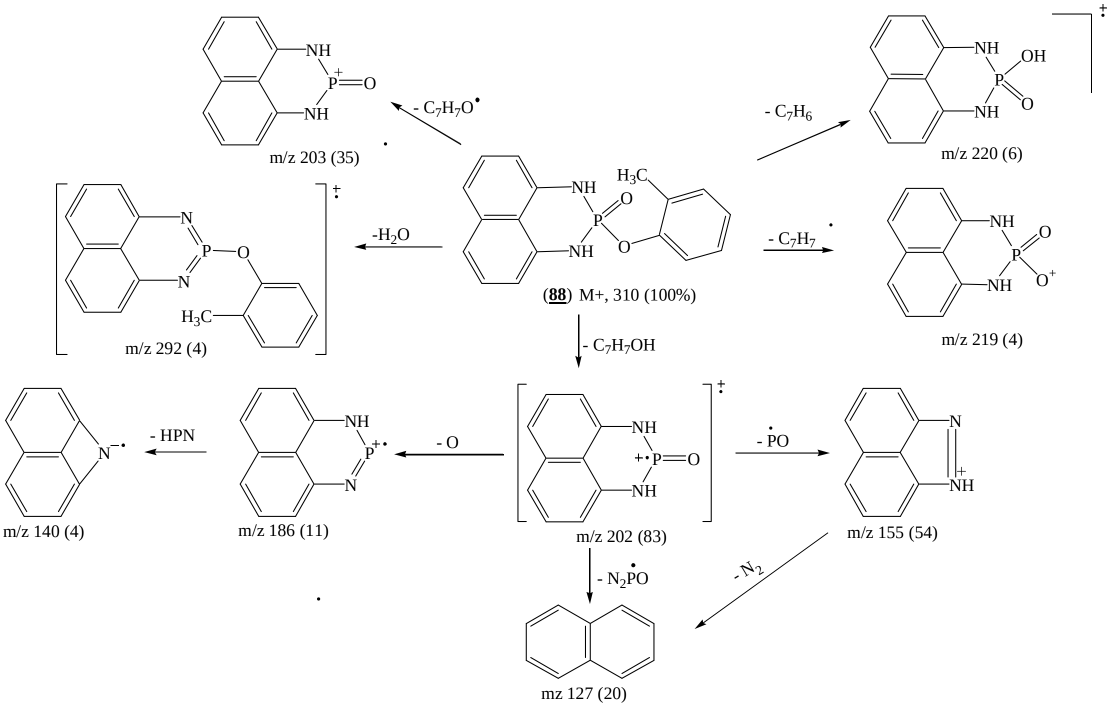
In addition, 3-alkyl-2-aminobenzthiazolium (**86**) when react with PCl_3/ Et_3N yielded the 1,2,4-phosphazabenzthiazole **87**[63,64].



86

87

Most spectral study of organophosphorus heterocyclic compounds has been undertaken because of their wide applications in agriculture as pesticide and industry, The mass spectra of a series of 2-substituted-2,3-dihydro-1H-naphtho[1,8-de](1,3,2)-diazaphosphorine-2-oxides (88) were studied to establish their fragmentation processes. The major fragmentation patterns are the loss of aryloxy substituent and RPO_2H moieties from the molecular ion. The fragmentation pattern is supported by meta stables, high resolution and collision activation dissociation data [Scheme 20A][61a]. Raju *et al*[61 b], reported the synthesis, electron impact and high resolution mass spectral of 2-substituted-3-(4-methylphenyl)-naphth[1,2-e](1,3,2)oxazaphosphorine-2-oxides/sulfide.



Scheme 20: Mass fragmentation of compound 88

Spectrophotometric Determinations of Phosphorus

Numbers of spectrophotometric methods for determination of phosphorus, based on the formation of heteropoly acid with molybdate have been described. The molar absorptivity of these heteropoly acids in the aqueous solution or in organic solvents after solvent extraction, are generally low[61]. Several cationic dyes, for example; methylene blue, ethyl violet, crystal violet, auramine, malachite green have been used for the determination of phosphorus[62]. The spectrophotometric determination of phosphates using above dyes processes have limited use due to several disadvantages.[63]

Recently, A novel curing agent of epoxy resins (EPO), bis(3-amino-2-thienyl) phenylphosphine oxide (ABTPPO), was synthesized and characterized. ABTPPO was used as a flame retardant curing agent, and used to prepare a novel halogen-free flame retardant EPO composite.[64] In addition to a reported of a miniature flow-through detector methods useful for bimodal, photometric and fluorimetric, determination of phosphates [65] and A simple manifold flow injection analysis (FIA) for determining phosphorus in the presence of arsenate in water[66]

CONCLUSION

This review cover published literature concerning the synthetic of fused biheterocyclic nitrogen containing phosphorus atom over the period 1999-2014, updating our earlier review[6] and on the area of bioactive 1,2,4-triazine derivatives[11,12,40,67,68, 69].

The developments in this area will help to persuade the important strategy to synthesis of phospha-heterocyclic nitrogen-five and six-membered rings.

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