ELECTROPHILIC HETEROCYCLIZATION OF UNSATURATED SULFUR AND PHOSPHORUS COMPOUNDS (REVIEW)

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UDC 547.269'298.4'367.398.4'341'732'738'77'789.04(047)

The literature on the electrophilic heterocyclization of unsaturated sulfur and phosphorus compounds has been reviewed. The factors influencing the reactivity of unsaturated compounds in such reactions are discussed, and data on the stereochemistry of the addition are presented.

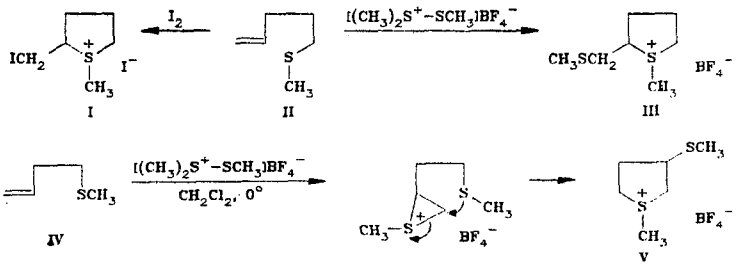
The electrophilic heterocyclization of unsaturated sulfur and phosphorus compounds as a general method of synthesis of heterocycles [1-3] has been used successfully in the synthesis of sulfur and phosphorus heterocycles. Some examples of the preparation of sulfur heterocycles have been given in a review [4]. The electrophilic heterocyclization of nonconjugated dienes has also been used to obtain these heterocycles [5].

We here discuss the electrophilic heterocyclization of unsaturated thiols, thiiranes, sulfides, thioamides, thiourethanes, allenephosphonates and -phosphinates, butadiene-1,3-diphosphonates, and buten-4-yl phosphates, by reaction with a variety of electrophiles, to give heterocycles with one or more heteroatoms. The factors influencing the reactivity of unsaturated compounds in this reaction are discussed, and data on the stereochemistry of the addition of electrophiles are presented.

ELECTROPHILIC HETEROCYCLIZATION OF UNSATURATED MERCAPTANS, SULFIDES, DISULFIDES, AND THIIRANES

The electrophilic heterocyclization of unsaturated compounds in which the nucleophilic group is a sulfide sulfur atom constitutes a convenient method for the preparation of cyclic sulfonium salts.

For example, iodination [4] of the unsaturated sulfide (II) gives the salt (I). The sulfonium salt (III) was also obtained from (II) by treatment with dimethyl(methylthio) sulfonium fluoroborate [6]. But-3-enyl methyl sulfide (IV), when treated similarly, gave the sulfonium salt (V).



Bromination of methyl allyl sulfide (VI) gave quantitative yields of the sulfide (X). Heating this sulfide (X) in $CDCl_3$ for 5 h afforded a mixture of sulfides (VIII) and (IX) in a ratio of 82:18. The formation of the sulfide (X) may be rationalized by assuming that the reaction proceeds via a thiiranium ion intermediate (IX), formed from the bromonium ion (VII) [7].

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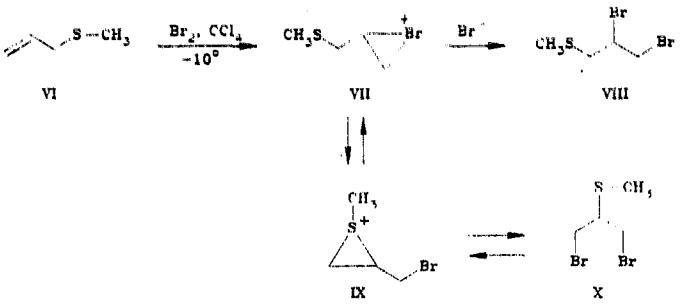
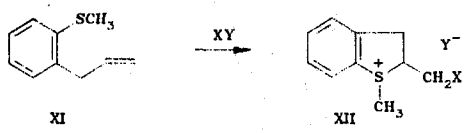
Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 3, pp. 291-302, March, 1986. Original article submitted October 2, 1984.

0009-3122/86/2203-0231$12.50

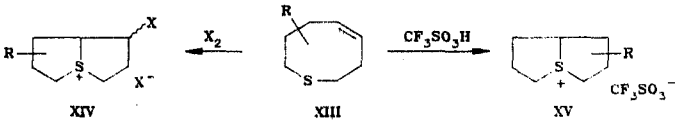


2-(Allylphenyl) methyl sulfide (XI) reacts with iodine, phenylsulfenyl chloride, and HBr [8] to give the salt (XII).



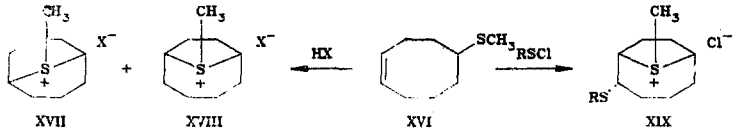
XY=I_2, PhSCl, HBr; X=I, PhS, H; Y=I, Cl, Br

The cyclic Z,E-thiacyclooct-4-enes (XIII) react with halogens and CF_3SO_3H [9] to give cis-1-thiabicyclo[3.3.0]octanes (XIV) and (XV). E-Thiacyclononene is converted on treatment with CF_3SO_3H into cis-thiabicyclo[4.3.0]nonane [10].



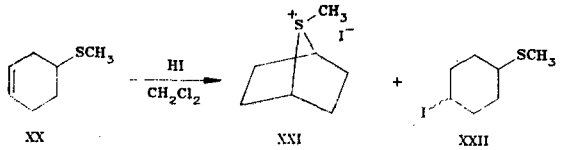
X=Cl, Br; R=H, CH_3

5-Methylthiocyclooctene (XVI) with RSCl gives the sulfonium salt (XIX) [11, 12], and with HX it affords a mixture of the sulfonium salts (XVII) and (XVII).

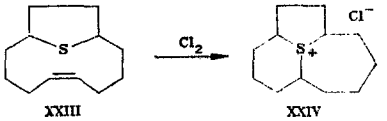


X=Cl, I; R=Ph, Me

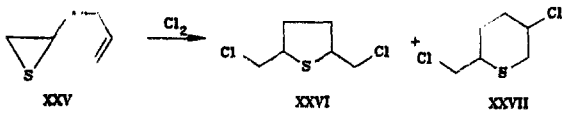
4-Methylthiocyclohexene (XX) reacts with HI to give 7-methyl-7-thiabicyclo[2.2.1]heptane-sulfonium iodide (XXI) [11] together with addition products of HI to the double bond (XXII).



13-Thiabicyclo[8.2.1]tridec-5-ene (XXIII) on chlorination affords 1-thioniatricyclo [8.2.1.$0^{1,7}$]tridecane chloride (XXIV) [12]. Similarly, the sulfoxide obtained from the sulfide (XXIII) on treatment with bromine is converted into 1-thioniaoxide-6-bromocyclo[8.2.1.$0^{1,7}$]tridecane bromide [13].

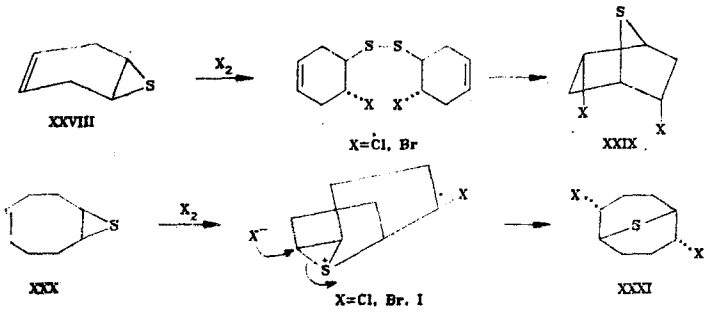


On chlorination, the thiirane (XXV) gives five- and six-membered heterocycles (XXVI) and (XXVII) [14].



XXV XXVI XXVII

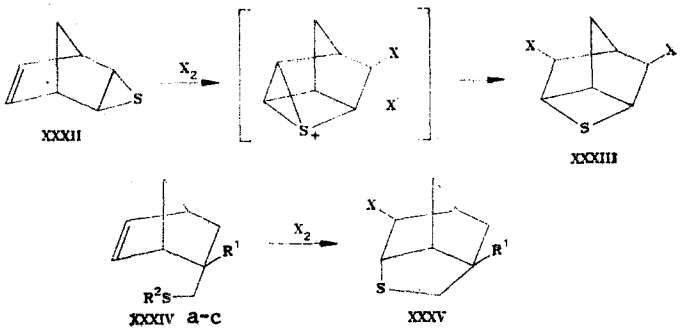
Similarly, halogenation of the cyclic thiiranes (XXVIII) and (XXX) affords the bicyclic systems (XXIX) and (XXXI) [14-16].



XXVIII X=Cl, Br XXIX

XXX X=Cl, Br, I XXXI

The thiaskeletal tricyclic systems (XXXIII) and (XXXV) were obtained by the heterocyclization of the thiirane (XXXII) [16] and norbornenethiols (XXXIV) [17, 18].

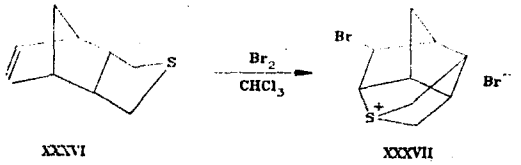


XXXII XXXIII

XXXIV a-c XXXV

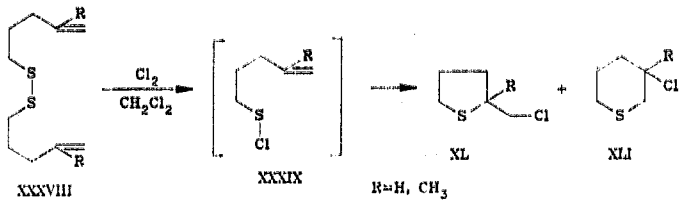
X_2=Br_2, PhSeCl; XXXIV a,c R^1=H, b R^1=CN; a,b R^2=H, c R^2=Ac

It interesting to note that bromination of the sulfide (XXXVI), in contrast to (XXXII), affords the stable 2-methyl-6-thiatricyclo[3.2.1.$1^{3,8}$]nonasulfonium bromide (XXXVII) [19].



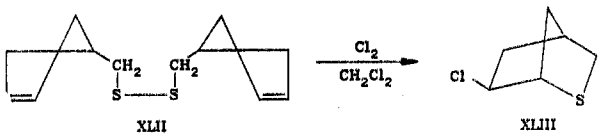
XXXVI XXXVII

On chlorination, the unsaturated disulfides (XXXVIII) give the sulfenyl chlorides (XXXIX), which then give rise to tetrahydrothiophens (XL) and tetrahydrothiopyrans (XLI). The tetrahydrothiophens (XL) are the kinetically controlled reaction products. When the reaction temperature is raised, they are converted into the thermodynamically-controlled products (XLI) [20].



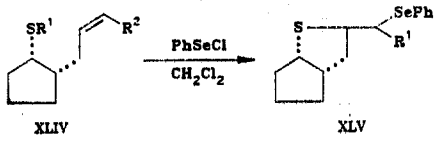
XXXVIII XXXIX XL XLI R=H, CH_3

Chlorination of the disulfide (XLII) gives the thianorbornane (XLIII) [21].



XLII XLIII

One method for the synthesis of thiaprostacyclins is heterocyclization of unsaturated sulfur compounds. Reaction of the thiols and thioacetates (XLIV) with PhSeCl and iodine [18, 22], or with phenylselenosuccin(or -phthal)imides [23, 24] affords the bicyclic systems (XLV), for example:

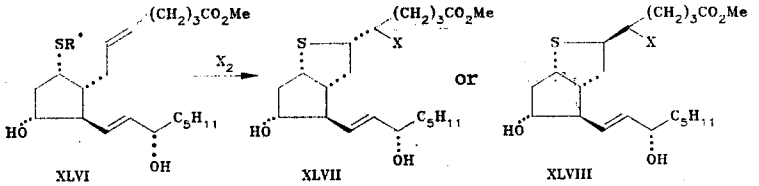


XLIV XLV

XLIV a,c R^1 = H, b,d R^1 = Ac; a,b R^2 = Me, c,d R^2 = $-(CH_2)_3COOMe$

Pentene-4-thiol [25] and 2-allylpentane (and hexane-)thiols [26, 27] on treatment with 75% sulfuric acid give 2-methyltetrahydrothiophen, 1-thiabicyclo[3.3.0]octane, and 2-methyl-1-thiahydrindane respectively.

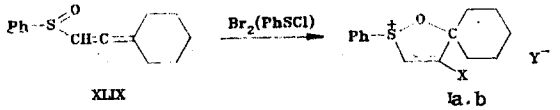
The 5Z- and 5E-thioacetates (XLVI), on iodination [28], phenylselenylchlorination [29], and bromination with bromine or N-bromosuccinimide [30-31] are converted into the corresponding thiaprostacyclins (XLVII) and XLVIII).



XLVI XLVII XLVIII

R=H, Br, I, Ac; X=Br, I, SePh

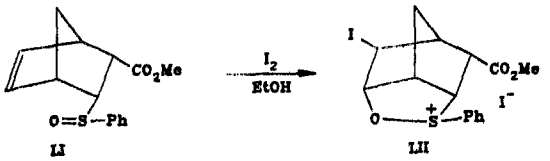
The sulfinylallene (XLIX) reacts with bromine and PhSCl [32] to give the heterocycles (La, b:, which contain two heteroatoms.



XLIX Ia.b

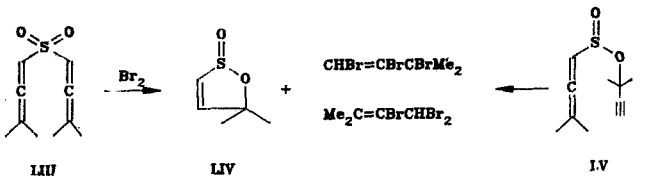
I a X=Y=Br; b X=PhS, Y=Cl

Similarly, iodination of sulfoxide (LI) affords the tricyclic heterocycle (LII) [33].



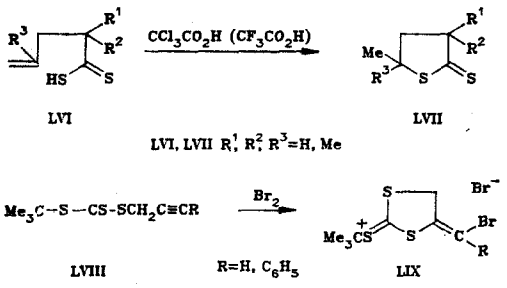
LI LII

Electrophilic fragmentation-cyclization of the diallenyl sulfones (LIII) and the propargyl allensulfinates (LV) by treatment with bromine affords the α,β-unsaturated γ-sultones (LIV) [34]:



LIII LIV LV

On reaction with trichloro (or -fluoro)acetic acid, the thiaocids (LVI) give the γ-dithiolactones (LVII) [35], and bromination of the propargyl trithiocarbonates (LVIII) affords the 1,3-dithioles (LIX) [36].



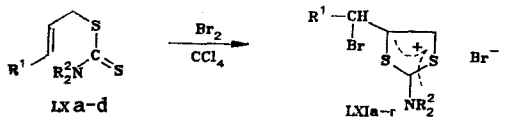
LVI LVII

LVI, LVII R^1, R^2 R^3=H, Me

LVIII R=H, C_6H_5 LIX

Iodination and bromination of substituted allylxanthate and allyltrithiocarbonate esters affords the 4,5-disubstituted 1,3-dithiol-2-ones and 1,3-dithiolan-2-thiones respectively [37].

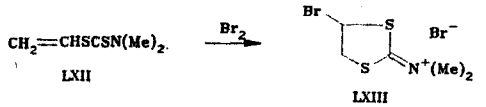
The β, γ-unsaturated dithiocarbamates (LX) on bromination give quantitative yields of the 2-dialkylamino-4-(2-bromoalkyl)-1,3-dithiolanium bromides (LXI) [38-40].



LX a-d LXIa-r

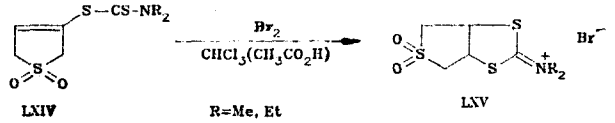
LX, LXI a,b R^1=H, c R^1=Me, d R^1=Ph; a,c,d R^2=Me, b R^2=Et

The vinyl NN-dimethyldithiocarbamates (LXII), on bromination, are converted into the 1,3-dithiolane-2-iminium salts (LXIII) [41, 42].



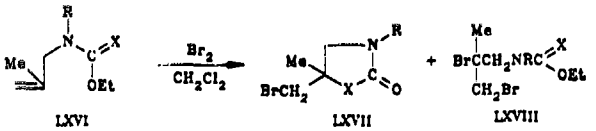
LXII LXIII

Similarly, bromocyclization of 1,1-dioxo-3-thiolen-3-yl NN-dialkyldithiocarbamates (LXIV) gives rise to the 1,3-dithiolane-2-iminium salts (LXVa, b) [43].



LXIV R=Me, Et LXV

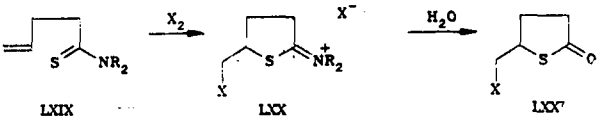
On bromination in CH_2Cl_2, the N-substituted methallylurethanes (LXVI) (X = O) afford the 5-bromomethyl-5-methyloxazolidin-2-ones (LXVII) (X = O), together with products of addition of bromine to the double bond (LXVII) [44]. Similar products were obtained when the methallylurethanes (LXVI) were reacted with sulfenyl chlorides [45]. The thiourethanes (LXVI) (X = S) are converted by bromination [46-48] or treatment with chlorine [49] into 2-bromomethyl- (LXVII) (X = S) and 2-chloromethylthiazolidin-2-ones respectively.



LXVI LXVII LXVIII

R=H, Me, Et, *i*-Pr, Bu, Ph, C_5H_{11}, C_6H_{13}; X=O,S

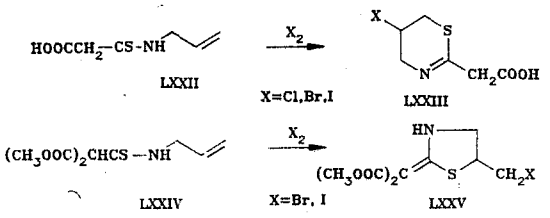
Allylthioacetamides (LXIX), on bromination or iodination followed by hydrolysis of the cyclization products (LXX), give substituted γ-halothiolactones (LXXI) [50]. The rate of iodination of the thioamide (LXIX) is nearly twice as great as that of iodocyclization of the appropriate amide.



LXIX LXX LXX'

X=Br, I

The allylthioamides (LXXII) and (LXXIV) on halogenation are converted, depending on their structure, either into thiazines (LXXIII) [51] or thiazolidines (LXXV) [52].



LXXII LXXIII

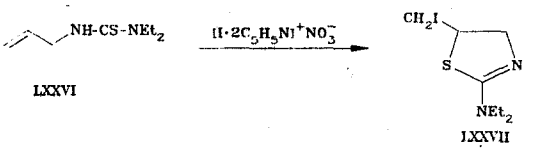
X=Cl,Br,I

LXXIV LXXV

X=Br, I

Allylthioureas [4, 53, 54], N-(cyclopenten-3-yl)- and N-(cyclohexen-3-yl)thioureas [55-57] on bromination give the corresponding 2-amino-5-bromomethyl-2-thiazolines, 2-aminosubstituted 6-bromocyclopenta[2,3-d]thiazolines, and 7-bromohexahydrobenzothiazoles. Allylthioureas also cyclize readily on treatment with acids [58, 59].

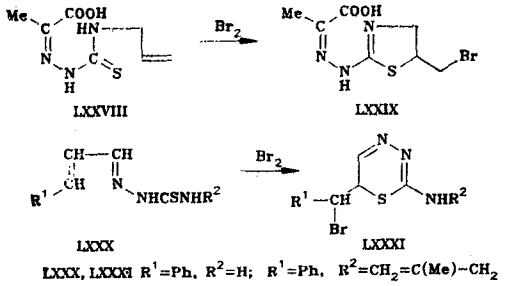
2-Diethylamino-5-iodomethyl-2-thiazoline (LXXVII) has been obtained by iodinating the thiourea (LXXVI) with an iodine–pyridine complex [60].



LXXVI LXXVII

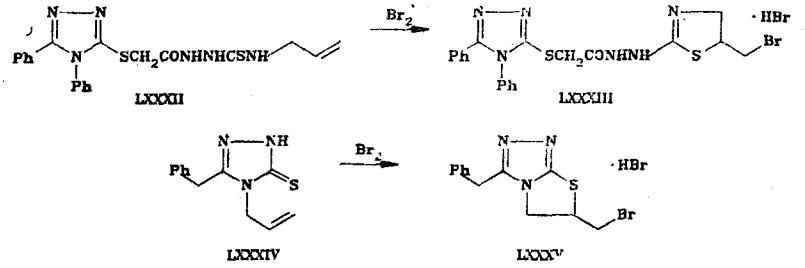
The iodocyclization of allyl- and propargylthioureas proceeds at approximately the same rate [61].

4-Allylthiosemicarbazone (LXXVIII) on bromination is converted into the (5-bromomethyl-2-thiazolin-2-yl)hydrazone (LXXIX) [62], and the thiosemicarbazide (LXXX) gives the 2,6-disubstituted 1,3,4-thiadiazines (LXXXI) [63].

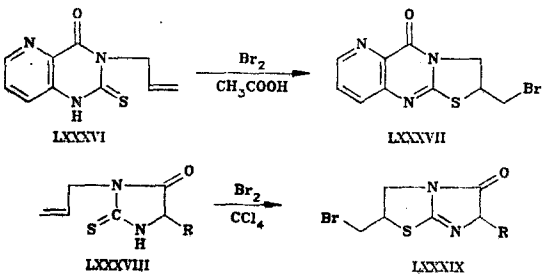


LXXX, LXXXI R^1=Ph, R^2=H; R^1=Ph, R^2=CH_2=C(Me)–CH_2

The N-allyl-N-acylthiosemicarbazide (LXXXII), on treatment with bromine, gives 2-[(4,5-diphenyl-1,2,4-triazol-3-yl)mercaptoacetylhydrazino]-5-bromomethyl-1,3-thiazole (LXXXIII) [64], and 4-allyl-5-benzyl-1,2,4-triazoline-3-thione (LXXXIV) affords 2-bromomethyl-5-benzyl-2,3-dihydro-1,3,4-thiazolo[2,1-b]triazolidine (LXXXV) [65].

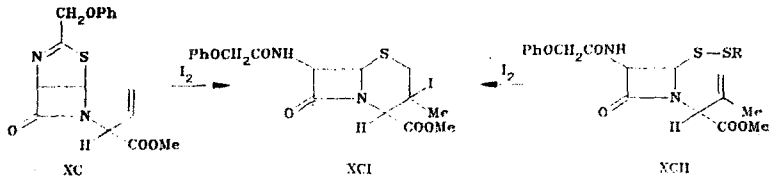


3-Allyl-3,4-dihydro-2-mercaptopyrido[3,2-d]pyrimidin-4-one (LXXXVI) is converted on bromination into 2-bromomethyl-2,3-dihydro-5H-pyrido[3,2-d]thiazolo[3,2-a]pyrimidin-5-one (LXXXVI) [66], and 3-allylthiohydantoins (LXXXVIII) give 2,3,5,6-tetrahydro-2-bromomethyl-5-oxoimidazo[2,1-b]thiazoles (LXXXIX) [67].



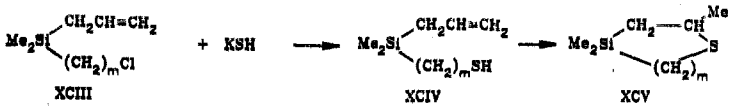
LXXXVIII, LXXXIX R=H, Me, Pr, *i*-Bu, CH_2COOH

The thioazetidinones (XC) and (XCII) on iodination give the 3-iodo-3-methylcepham (XCI) [68].



R=2-benzothiazolyl

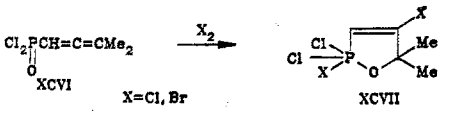
A novel method for the synthesis of five- and six-membered thiacycloalkanes is the reaction of dimethyl(ω-chloroalkyl)alkenylsilanes (XCIII) with KSH in alcoholic solution. The intermediate unstable thiols (XCIV) undergo intramolecular cyclization to give the heterocycles (XCV) [69].



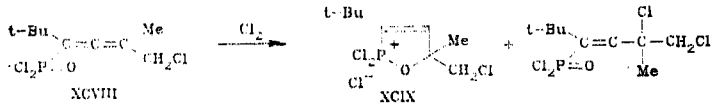
XCIII-XCVa m = 1; m = 3

SYNTHESIS OF PHOSPHOROUS HETEROCYCLES BY THE ELECTROPHILIC HETEROCYCLIZATION OF UNSATURATED ORGANOPHOSPHORUS COMPOUNDS

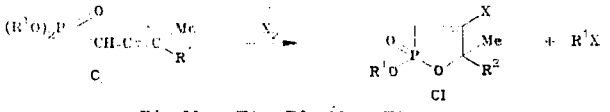
In 1975 A. A. Petrov et al. [70], in applying the electrophilic heterocylization reaction to unsaturated organophosphorus compounds, discovered a new route to phosphorus heterocycles. The reaction of 3-methyl-1,2-butadienephosphonyl chloride (XCVI) with chlorine or bromine in an inert solvent afforded quantitative yields of 2,2,2,4-tetrachloro- (XCVII, X = Cl) or 2,2-dichloro-2,4-dibromo-5,5-dimethyl-1,2-oxaphosphol-3-ene (XCVII, X = Br) [70].



In the chlorination of the acid chloride (XCVIII) it was found that the formation of 2,2,2,4-tetrahalo-1,2-oxaphospholenes (XCVII) occurred via the intermediate phosphonium salts (XCIX) [71].



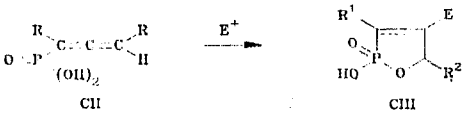
Halocyclization of the dialkyl 1,2-alkadienephosphonates (C) affords 1,2-oxaphosphol-3-enes (CI) [72-74].



R^1=Me, Et; R^2=Me, Et, *i*-Pr

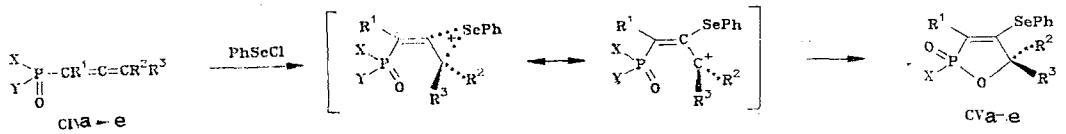
The relative rates of reaction of iodine and interhalides with dialkyl 1,2-alkadienephosphonates decreases in the sequence ClI > BrI > I_2 [75]. The presence of hydroquinone has no effect on the rate of the reaction, and it was therefore concluded that the reaction was an electrophilic one.

The allenephosphonic acids (CII) also react with Br_2 and $Hg(OAc)_2$ to give 1,2-oxaphosphole-3-enes (CIII) [76].



R=*t*-Bu; E=Br; HgOAc

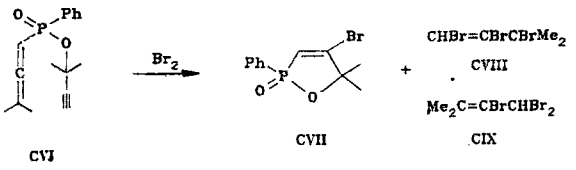
Reaction of the allenephosphonates (CIVa-c) and the allenephosphinates (CIVd, e) with RSCl and PhSeCl [77-80] has given the 4-alkyl (or -aryl) thio (or -seleno)-1,2-oxaphosphol-3-enes (CV).



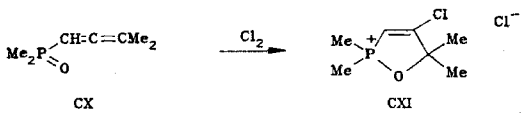
CIV, CV a R^1=*t*-Bu, b–e R^1=H; a R^2=*t*-Bu, a R^3=H, b,d,e R^2=R^3=Me, c R^2=R^3=$(CH_2)_5$; a X=MeO, b,c X=EtO, d,e X=Ph; a–e Y=MeO, b,c Y=EtO, d Y=OH

1,2-Oxphosphol-3-enes are also obtained by hydrochlorinating dialkyl 1,2-alkadienephosphonates with gaseous HCl in inert solvents [81-83], and by reacting substituted propargyl alcohols with phosphorus trihalides [84-86].

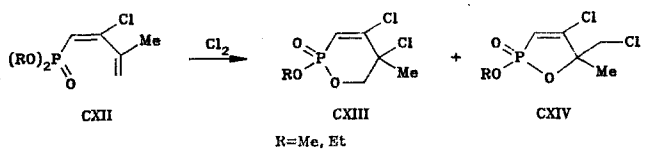
The bromination of α,α-dimethylpropargyl phenyl-γ,γ-dimethylallenephosphinate (CVI) gives 4-bromo-5,5-dimethyl-2-phenyl-1,2-oxaphosphol-3-ene (CVII) in addition to fragmentation products (CVIII) and (CIX) [34].



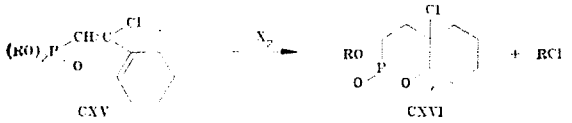
Chlorination of dimethyl(3-methyl-1,2-butadienyl)phosphine oxide (CX) [87], or hydrochlorination [82], gives 4-chloro-2,2,5,5-tetramethyl-1,2-oxaphosphol-3-ene bromide (CXI).



On chlorination of dialkyl 2-chloro-3-methyl-1,3-butadienephosphonates (CXII), as in the case of allenephosphonates, the principal pathway of the reaction is halocyclization, which here leads to the formation of mixtures of the isomeric 2-phosphacyclohex-3-enes (CXIII) and 1,2-oxaphosphol-3-enes (CXIV) [88-90]. In the absence of substituents in positions 1, 2, or 3 of the alkadiene system, bromination affords only the products of addition of bromine to the double bond [91].

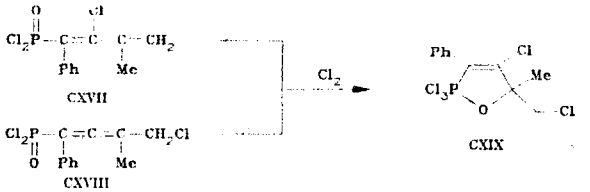


Halogenation of dialkyl 2-chloro-2-(1-cyclohexenyl)ethenephosphonates (CXV) gives the six-membered, 1,2-oxaphosphorines (CXVI) only [92, 93].

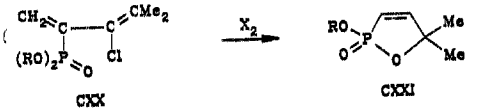


R=Me, Et, *i*-Pr, Bu; X=Cl, Br

The chlorination of 1-phenyl-2-chloro-3-methyl-1,3-butadienephosphonyl dichloride (CXVII) and 1-phenyl-4-chloro-3-methyl-1,2-butadienephosphonyl dichloride (CXVIII) results in the formation of 2,2,2,4-tetrachloro-5-methyl-5-chloromethyl-1,2-oxaphosphol-3-ene (CXIX) [94].



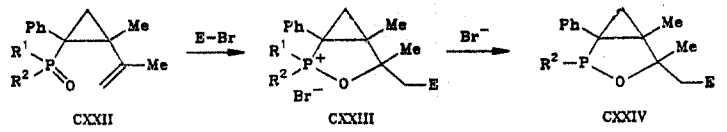
Halogenation of dialkyl 3-chloro-4-methyl-1,3-pentadiene-2-phosphonate (CXX) with a tertiary carbon atom in the 4-position of the 1,3-alkadiene chain gives the 1,2-oxaphospholen-3-enes (CXXI) [95].



CXX CXXI

R = Me—Bu; X = Cl, Br

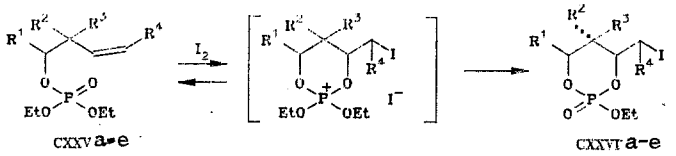
cis-2-Isopropenylcyclopropylphosphonates (CXXIa-c) react with Br_2 and HBr to give the 3,2λ⁵-oxaphosphabicyclo[3.1.0]hexan-2-ones (CXXIVa-c) [96]. The initially formed phosphonium salts (CXXIIIa-e) are converted by the Arbuzov reaction into the final cyclization products. In the case of the salt (CXXIIId), in which this reaction is not possible, the phosphonium salt is stable and could be isolated. The trans-isomers of (CXXII) react with HBr to give the products of hydrolysis of the ester bond, but the stereoisomeric 2-vinylcyclopropylphosphonates add Br_2 and HBr at the double bond.



CXXII CXXIII CXXIV

CXXII—CXXIV a,b R^1 = OMe, c,d R^1 = Ph; a,c R^2 = OMe, b,d R^2 = Ph; E = Br, H

Iodination of the buten-4-yl phosphates (CXXV) in acetonitrile affords the cyclic phosphates (CXXVI) [97]. Allyl phosphates fail to react with iodine under similar conditions.



CXXV a-e CXXVI a-e

CXXV, CXXVI a,c,d R^1 = Me, b R^1 = Pr, e R^1 = Et; a,b,d,e R^2 = H, c R^2 = Me; a–c,e R^3 = H, d R^3 = Me; a–d R^4 = H, e R^4 = Me

OO-Dialkyl-S-allyl and OO-dialkyl-O-allyl thiophosphates [98] and substituted OO-dialkyl 2-propenylphosphonates [99] react with Cl_2 and Br_2 to give the normal products of addition of the halogen at the double bond.

The electrophilic heterocyclization of unsaturated sulfur and phosphorus compounds thus makes it possible to obtain the corresponding heterocycles under mild conditions and in high yields.

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