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MACROCYCLES CONTAINING AZINE FRAGMENTS (REVIEW)

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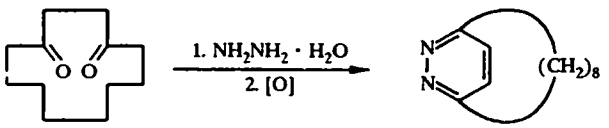
Data published over the last two decades on macrocycles containing azine fragments in the ring are reviewed and analyzed.

It is well known that macrocyclic compounds are of great significance in chemistry and biology. The inclusion of azine fragments in the ring of macrocycles can be expected to extend the range of the applications of these compounds, for example, as ligands and biologically active substances. This clearly explains the increased interest in macrocycles with azine fragments in the literature in recent years.

The present review covers all types of macrocycles: cyclophanes, crown ethers, and macrocycles with various types of bonds in the linking chains. Papers which were not covered in the fundamental review of 1997 [1] are examined, and individual aspects of the review and of the monographs [2-4] are incorporated. In each section the macrocycles are given in the order of seniority of the azine fragment contained in the ring of the macrocycle.

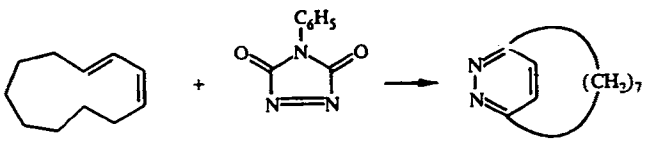
AZINOCYCLOPHANES

Azinocyclophanes can be regarded as the aza analogs of cyclophanes in which at least two CH groups of the benzene ring are replaced by nitrogen atoms. Only two piperazinophanes have been described. [8](3,6)Pyridazinophane (I) was obtained during the condensation of cyclododecane-1,4-dione with hydrazine followed by oxidation by atmospheric oxygen [5].



I, 50%

The synthesis of [7](3,6)pyridazinophane, obtained as a Diels–Alder adduct from 1,3-cycloundecadiene and 4-phenyl-1,2,4-triazoline-3,5-dione [5], is interesting.



II, 65%

Comparatively few papers have been devoted to cyclophanes containing only one pyrimidine fragment in the molecule. The oxidation of 4,6-bis(4-pentynyl)-2-methylpyrimidine (III) with copper acetate results in intramolecular cyclization with the formation of the pyrimidinophane (IV), containing triple bonds in the aliphatic fragment of the macrocycle. During catalytic hydrogenation the latter forms 13-methyl[10](4,6)pyrimidinophane (V) [6].

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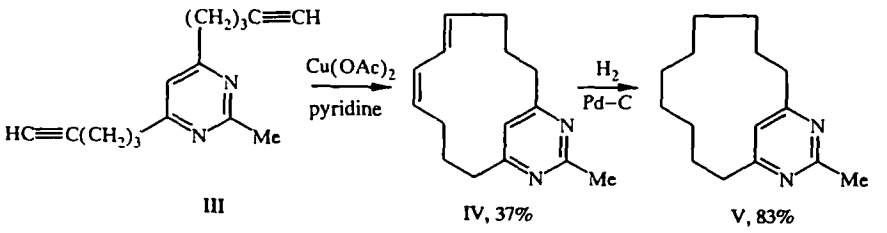
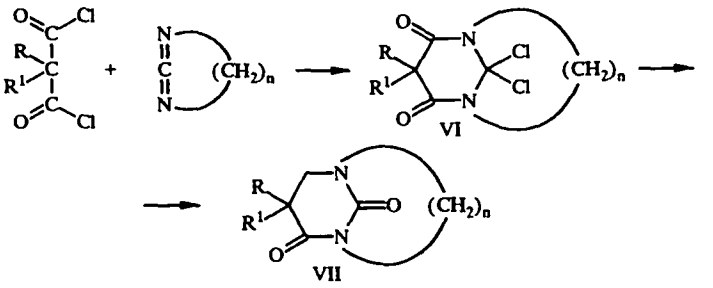
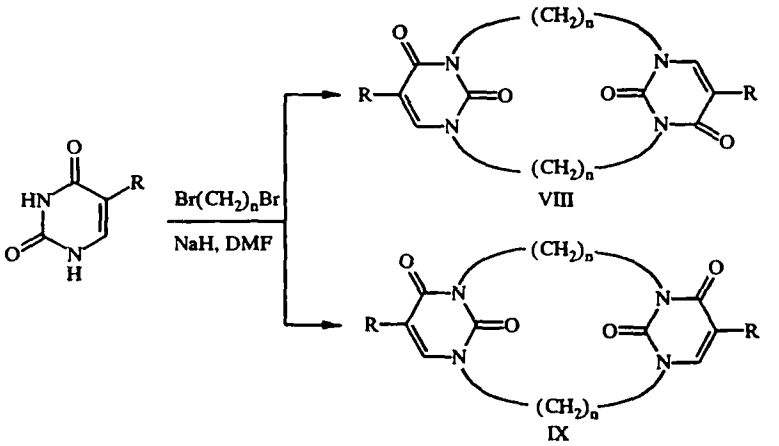


High yields of the cyclophanes (VI) were obtained from carbodiimides and disubstituted malonyl chlorides, and their structures were confirmed by the formation of the cyclophanes (VII) during hydrolysis. Compounds (VII) contain barbituric acid fragments and are therefore of interest as potential medicinal products [7].



R, R^1 = Cl, Me, Et; n = 6, 7, 11

A series of papers have been devoted to the synthesis of pyrimidinocyclophanes containing two pyrimidine rings in the macrocycle. The reaction of uracil, thymine, and 5-fluorouracil with dihalogenoalkanes in DMFA solution in the presence of sodium hydride leads to the formation of two isomeric pyrimidinocyclophanes (VIII) and (IX) [8].

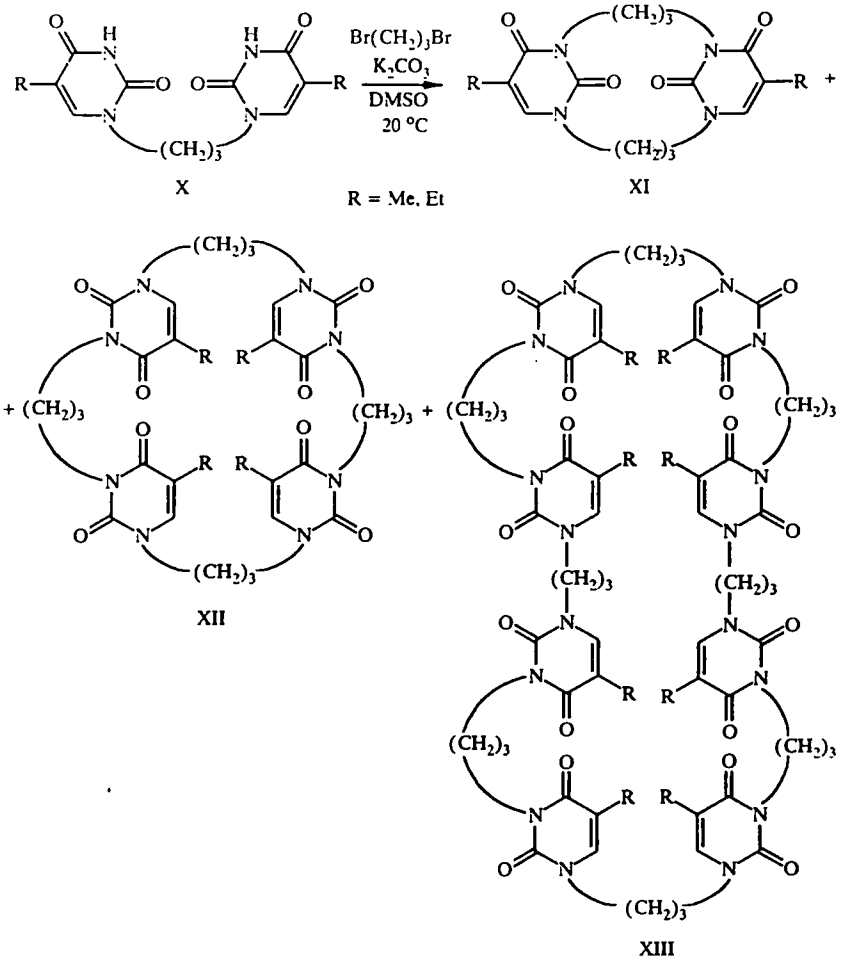


R = H, Me, F; n = 5–12

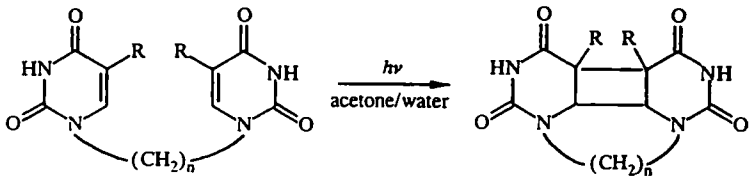
Cyclization with the formation of macrocycles takes place by a different mechanism during the alkylation of 1,1'-trimethylenebis(5-alkyl)uracils (X) in DMSO solution with potassium carbonate.

The macrocyclic tetramers (XII) and octamers (XIII) were also obtained in addition to the cyclophanes (XI).

Under conditions with high dilutions the macrocycles (XI) predominate. In more concentrated solutions the yields of the macrocycles (XII) and (XIII) are higher. The yields of the cyclophanes decrease with increase in the size of the macrocycle. Thus, in the case of the macrocycles (XI, XII, XIII) with thymine fragments (R = Me) under optimal conditions the yields amounted to 28, 20, and 5% respectively [9].

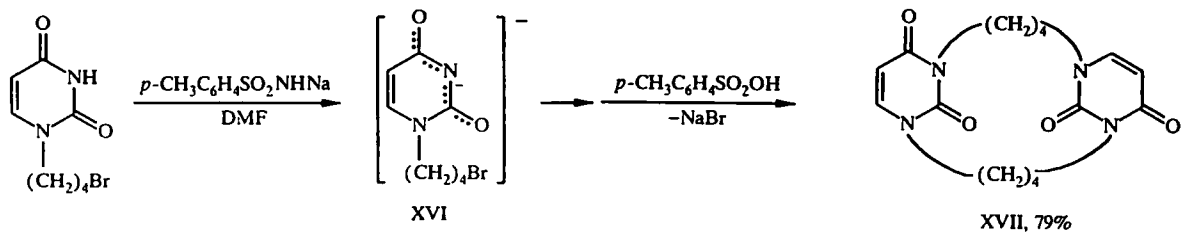


The photodimerization of polymethylenebisuracils (XIV) by UV irradiation with the formation of the macrocycles (XV), containing a cyclobutane fragment in addition to pyrimidine, has been described [10].



R = Me, Et ; n = 5, 6

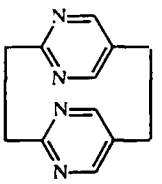
An original method for the synthesis of uracilocyclophanes in the reaction of 1-(1-bromobutyl)uracil with the sodium salt of *p*-toluenesulfonamide in DMFA solution was described in [11, 12].



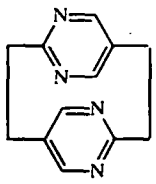
The authors explain the high yield of 1,1',3,3'-tetramethylenebisuracil (XVII) by the low solvation of the anion in the aprotic solvent, promoting intermolecular interaction of the anions (XVI). This is supported by the fact that in butanol solution

the yield of the macrocycle (XVII) amounts to only 10%. The reaction by the described mechanism is also promoted by the fact that the acidic characteristics of the uracil and the sulfonamide are close (pK_a values 9.5 and 9.0 respectively) and that there is an equilibrium between the anion (XVI) and the sulfonamide in the solution.

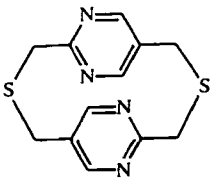
The isomeric [2,2](2,5)pyrimidinophanes (XVIII) and (XIX) were obtained during the photolytic desulfurization of 2,11-dithia[3,3](2,5)pyrimidinophane (XX) [13].



XVIII

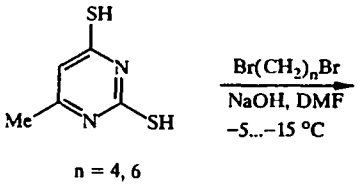


XIX

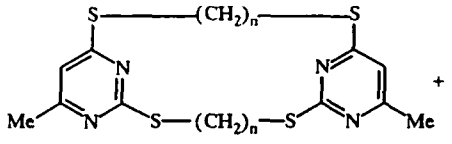


XX

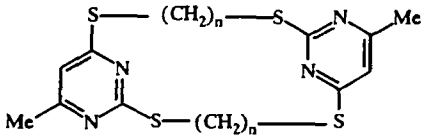
The reaction of the disodium salt of 2,4-dimercapto-6-methylpyrimidine with α,ω-dibromoalkanes leads to the formation of a mixture of two isomers of pyrimidinothiacyclophane (XXI, XXII) with yields of up to 46% [14].



n = 4, 6

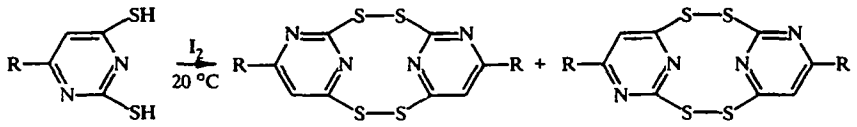


XXI



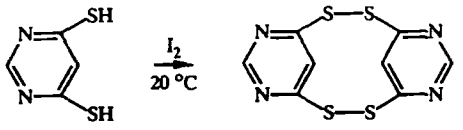
XXII

Pyrimidinothiacyclophanes in which the pyrimidine rings are linked by only sulfur atoms were described for the first time in [15, 16]. The macrocycles were produced with yields of 25-35% during the oxidation of 2,4- and 4,6-dimercaptopyrimidines in solution in a mixture of benzene and dioxane.



R = H, Me, Ph

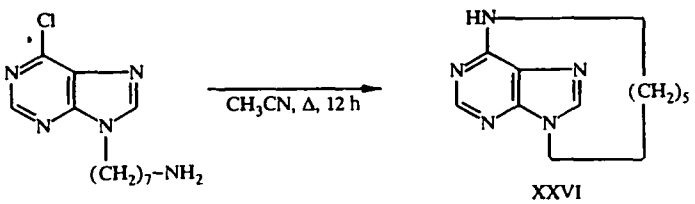
XXIII XXIV



XXV

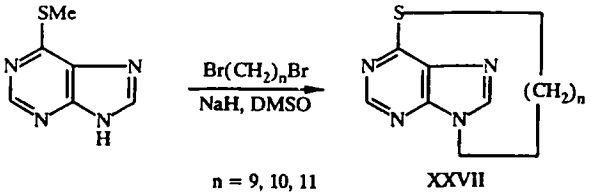
The cyclophanes (XXIII, XXIV) were isolated in the form of a mixture of two isomers. The structure of the macrocycle (XXV) corresponds to the only possible symmetrical structure.

Of the condensed derivatives of pyrimidine only cyclophanes with purine rings have been described. For spectroscopic investigations $N_{(6)}$,$N_{(9)}$-octamethylenepurinocyclophane (XXVI) was synthesized by intramolecular cyclization of 6-chloro-9-(7-aminoheptyl)purine [17].



XXVI

A thiacyclophane with an analogous structure (XXVII) was obtained from 6-methylthiopurine and dibromoalkanes with yields of 8-13%, depending on the number of methylene groups in the ring [18].

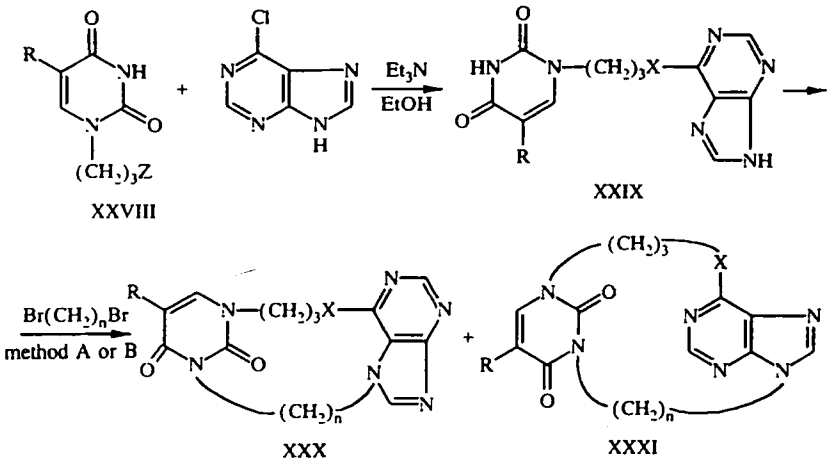


Attention is drawn to papers on the specific synthesis of pyrimidinopurinophanes and purinophanes each containing two heterocycles in the macrocycle. These cyclophanes are models for studying the reaction between the pyrimidine and purine bases in nucleic acids by electronic molecular spectroscopy.

In contrast to nucleic acids, in which the bases in the polynucleotide chains are linked by hydrogen bonds, the cyclophanes are rigid systems in which the distances between the bases in the various chains are fixed by valence bonds. Comparison of the interplanar interactions of the bases in the cyclophanes and DNA makes it possible to obtain further information on the geometry of the bonds in DNA. The mutual effect of the bases in the cyclophanes and the chains of DNA is characterized by a change in the intensity of the absorption in the UV spectra or by a hypsochromic effect [19].

A series of pyrimidinopurinoaza- and pyrimidinopurinothiacyclophanes were synthesized by the stepwise introduction of chains linking the pyrimidine and purine rings [20-23].

The reaction of the uracils (XXVIII) with 6-chloropurine gives high yields of the initial compounds (XXIX) for cyclization to pyrimidinopurinophanes (XXIX). The second chains were introduced during the cyclization of these compounds with α,ω-dibromoalkanes in the presence of potassium carbonate in DMSO solution (method A) and with sodium hydride in DMFA solution (method B). Depending on the method, the cyclization takes place selectively with the formation of two isomers: (1',3')pyrimidino(6,7)purinophanes (XXX) and (1',3')pyrimidino(6,9)purinophanes (XXXI).



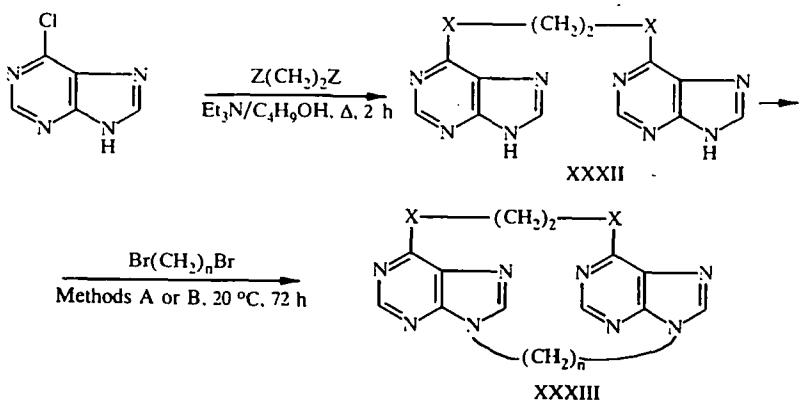
Z = NH_2, SH; X = NH, S; n = 3, 4; R = H, Me, OMe, NO_2

During cyclization by method A, except in one case (R = H, X = NH, n = 4), only the isomers (XXX) are obtained with yields of 15-40%. In method B both isomers (XXX) and (XXXI) are formed with yields of 14-28% and 6-21% respectively.

As indicated above, purinophanes are used as models for the investigation of the molecular interaction of the bases in the chains of nucleic acids. Syntheses of cyclophanes containing two purine rings in the macrocycle were described in a series of papers [24-28], and the first representatives of these compounds were described in [24]. Isomerization in cyclophanes depends on the positions at which the purine rings are linked in the macrocycle.

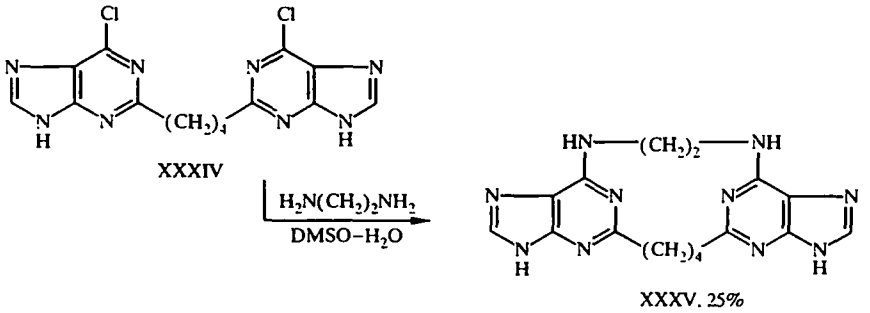
By analogy with the pyrimidinopurinophanes (XXX, XXI) the purinophanes (XXXIII) were obtained by the successive introduction of the linking polymethylene chains. In the reaction of 6-chloropurine with ethylenediamine or with ethanediol the

starting compounds (XXXII) for cyclization were obtained with yields of 49-67%. (6,6')(9,9')Purinophanes were synthesized with yields of 4-15% during the cyclization of dibromoalkane compounds (XXXII) by two methods: A) potassium carbonate in DMSO; B) sodium hydride in DMFA.



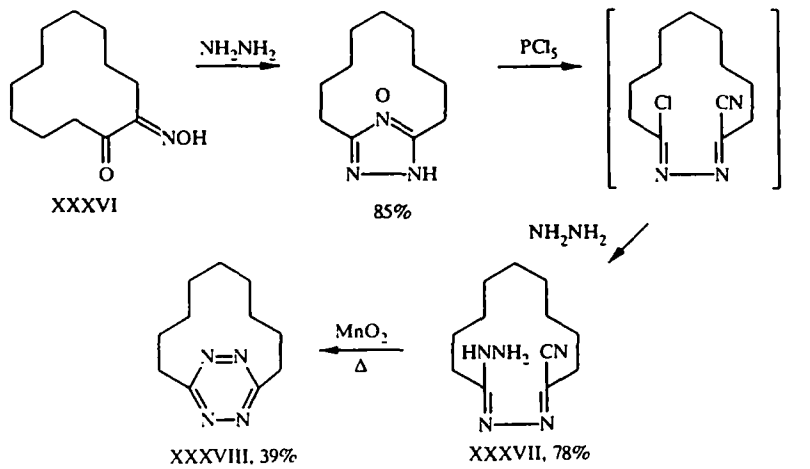
Z = NH_2, SH; X = NH, S; n = 3, 4

When the pyrimidine rings in 2,2'-tetramethylenebis(6-chloro)purine (XXXIV) are linked with ethylenediamine under conditions with high dilutions, the (2,2)(6',6')purinophane (XXXV) is formed.



In this cyclophane, unlike its isomers (XXXIII), the purine rings are only linked through pyrimidine fragments joined at the carbon atoms. They are of further interest as models of DNA for spectroscopic investigations.

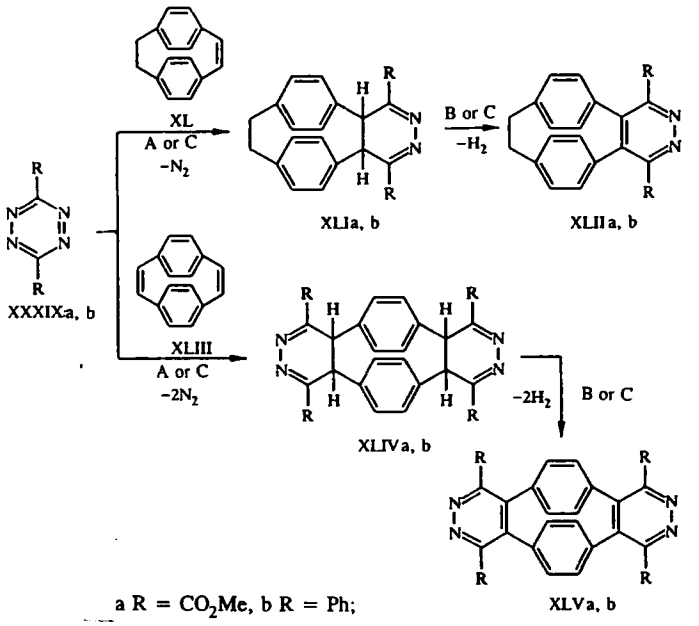
Only one representative of cyclophanes of the tetrazine series — [9][3,6]-*s*-tetrazinophane (XXXVIII) — has been described. It was obtained as a result of a multistage synthesis starting from 1,3-cyclododecanedione monoxime (XXXVI). In the last stage the cyclophane (XXXVIII) is formed with a 39% yield by heating cyanohydrazinodiazacyclophane (XXXVII) with a strong oxidizing agent (manganese dioxide) [29].



AZINE MACROCYCLES WITH AROMATIC STRUCTURES IN THE LINKING CHAINS

In this section macrocycles in which the azine fragments are linked by chains containing aromatic systems are examined.

It is known that many azadienes are synthons in the synthesis of six-membered nitrogen-containing heterocycles [30]. Heterodiene synthesis by the Diels–Alder reaction has been called the "reverse diene synthesis," since in most cases reaction occurs between π-deficient azadienes and π-excessive dienophiles.



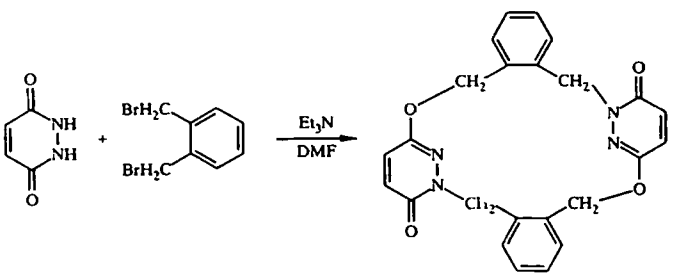
Methods: A, $CHCl_3$, 20°C, 12 h; B, $CHCl_3$, DDB, 20°C, 2 h; C, xylene 140°C, 48 h

3,6-Disubstituted 1,2,4,5-tetrazines (XXXIX), which contain the 2,3-diaza-1,3-butadiene fragment, react with [2,2]paracyclophan-1-ene (XL) and [2,2]paracyclophane-1,9-diene (XLIII) as dienophiles and form the pyridazine-annellated [2,2]paracyclophanes (XLII, XLV) through the initial diene-synthesis adducts (XLI) and (XLIV) [31].

The initial diene-synthesis adducts (XLIa) and (XLIVa) are easily formed with yields of 85 and 80% respectively during the reaction of dienophiles with the tetrazine (XXXIXa) (method A). The presence of an electron-withdrawing substituent increases the reactivity of the diene according the mechanism of the reverse Diels–Alder diene synthesis. The adducts are oxidized by dichlorodicyanobenzoquinone (DDB, method B) to the pyridazine-annellated [2,2]paracyclophanes (XLIIa) (91%) and (XLVa) (61%).

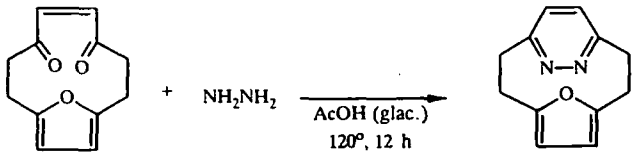
Only under more severe conditions (method C) does the tetrazine (XXXIXb) (R = Ph) in reaction with dienophiles form the pyridazine-annellated [2,2]cyclophanes (XLIIb, XLVb) directly with yields of 80 and 33% as a result of the oxidation of the intermediate initial adducts by atmospheric oxygen.

The polyfunctional macrocycle with pyridazine fragments (XLVI) is formed with a small yield from maleic hydrazide and α,α-dibromo-*o*-xylene in the presence of a base [32].



The macrocycle (XLVI) may have practical significance owing to the fact that its molecule contains the types of bonds and groups found in crown ethers, cryptands, and cyclophanes.

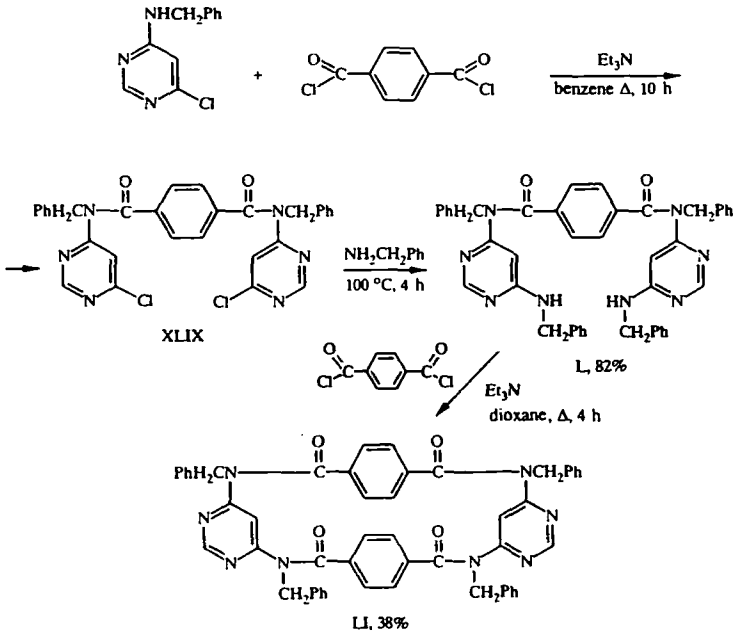
The synthesis of [2,2](2,5)furano(3,6)pyridazinophane (XLVIII) from dioxofuranophane (XLVII) and hydrazine is described in [33].



XLVII XLVIII, 20...40%

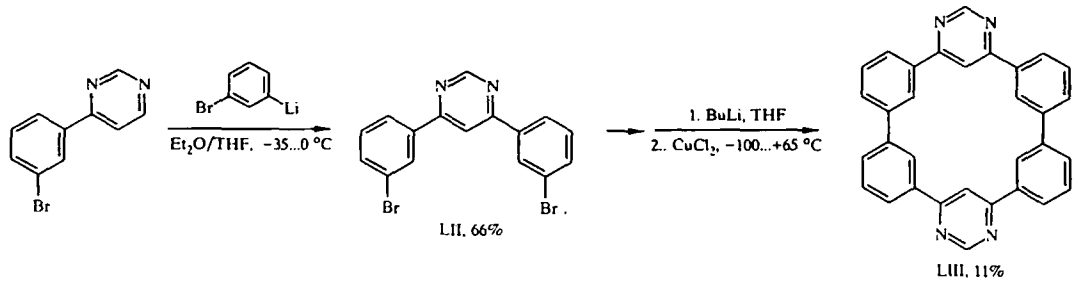
The macrocycle (XLVIII) is the first representative of [2,2]pyridazinophanes. The macrocycle contains π-excessive and π-deficient fragments, and this is of interest for studying the electronic interaction between the furan and pyridazine rings by spectroscopic methods.

The synthesis of the polyfunctional macrocycle (LI), in which the pyrimidine fragments are linked by aromatic and amide bonds, was described in [34]. According to data in [2], they have specific complexing characteristics. The synthesis was realized by a two-stage scheme starting from benzylaminopyrimidines and terephthaloyl dichloride, which eliminates the need to conduct the reactions under the conditions of high dilution. The intermediate N,N'-bis[(pyrimidinyl)(benzyl)]diamides of terephthalic acid (XLIX, L) were obtained with high yields.



XLIX L, 82% LI, 38%

The relative ease of formation of the final macrocycle tetraoxotetraaza[2,2,2,2](4,6)pyrimidinoparacyclophane (LI) (yield 38%) is clearly explained by the "rigid" conformation of the initial diamide (L).

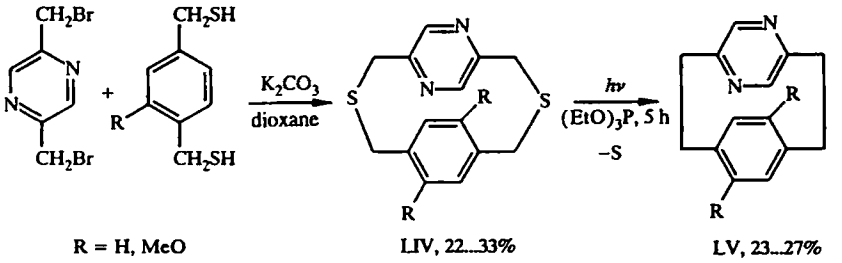


LII, 66% LIII, 11%

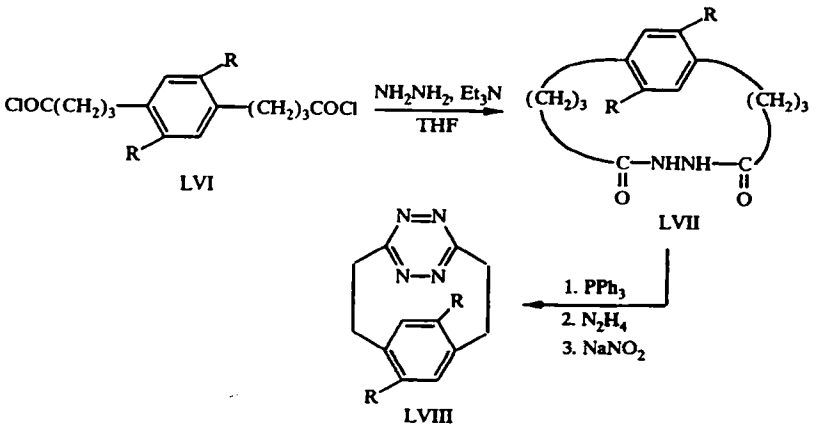
Only one example of macrocycles in which the pyrimidine fragments are linked by benzene rings has been described in the literature [35, 36]. The synthesis was based on direct lithiation of the pyrimidine ring at positions 4 and 6.

The obtained tetraazahexaphenylene (LIII) is of interest for investigations as ligand on account of the presence of donating and accepting rings in the macrocycle and also of cavities capable of enclosing whole neutral molecules.

In the opinion of the authors of [37] the presence of electron-donating and accepting fragments in the macrocycle is of interest for biochemical investigations. Pyrazinoparacyclophanes were synthesized for these purposes. The reaction of 2,5-bis(bromomethyl)pyrazine with 1,4-bis(methylthio)benzene gives 5,8-diaza-2,11-dithia[3,3]paracyclophane (LIV), which is converted by irradiation with a mercury lamp in triethyl phosphate solution with removal of the sulfur atoms into 4,7-diaza[2,2]paracyclophane (LV).



5,6,8,9-Tetraaza[3,3]paracyclophanes (LVIII) were synthesized as models for the investigation of transannular interaction in cyclic structures. *s*-Tetrazine as acceptor and benzene as donor of electrons are arranged symmetrically in the macrocycle.



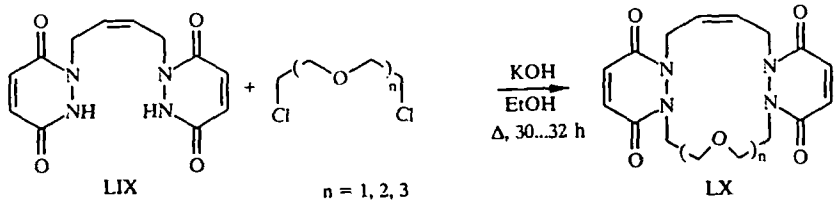
The initial acid dichlorides (LVI) were cyclized with hydrazine by the method of high dilutions in THF solution with the formation of 4,7-dioxo-5,6-diaza(10)paracyclophanes (LVII). As a result of complex transformations the latter form 5,6,8,9-tetraaza[3,3]paracyclophanes (LVIII) with small yields: R = H (0.6%), R = OCH_3 (5%) [38].

CROWN ETHERS WITH AZINE FRAGMENTS

The introduction of azine fragments into the macrocycle of crown ethers extends the possibilities of their use in science and technology.

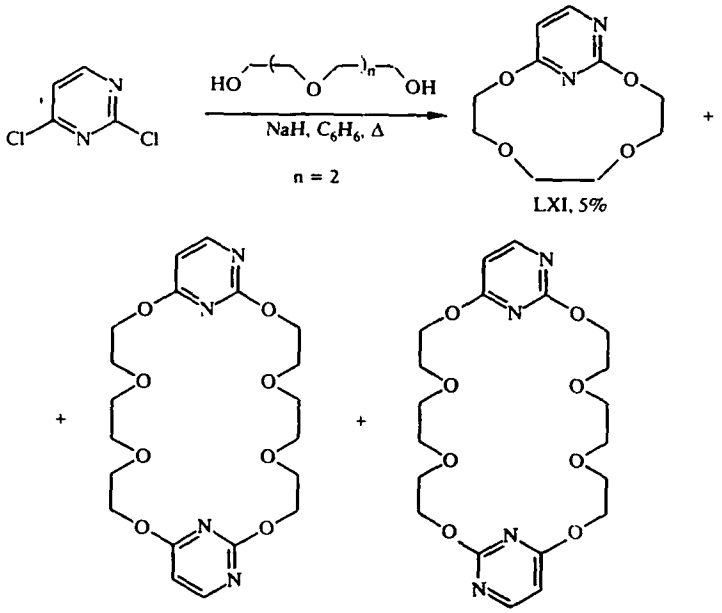
Azacrown compounds containing pyridazine fragments in the ring were synthesized for investigations into their biological activity [39]. The macrocycles (LX) were obtained with yields of 29-75% as a result of the cyclocondensation of 1,4-di(3,6-dioxo-1-pyridazinyl)-2-butene (LIX) with di-, tri-, and tetraethylene glycol dichlorides.

In addition to their complexing ability, the pyrimidinocrown ethers are of particular interest for biological tests as analogs of benzocrown ethers.



LIX LX

Crown ethers containing pyrimidine fragments added at positions 2 and 4 were described for the first time in [40, 41].

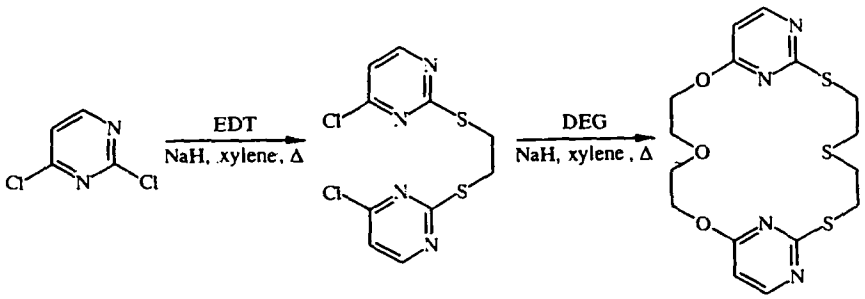


LXI, 5%

LXII, A 11% LXII, B 79%

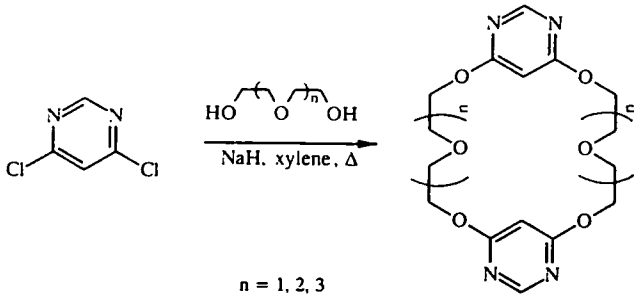
In reaction with the dianion generated from triethylene glycol with sodium hydride in benzene solution 2,4-dichloropyrimidine gives small yields of the pyrimidinocrown ether (LXI) (1:1) and the crown ethers (LXII), isolated in the form of two isomers A and B.

The 2,4-pyrimidinocrown ether (LXIV) with C–S and C–O bonds was also obtained. Heterocyclization takes place during the reaction of 2,4-dichloropyrimidine with the ethanedithiol (EDT) and diethylene glycol (DEG) anions through the intermediate compound (LXIII).



LXIII, 5% LXIV, 55%

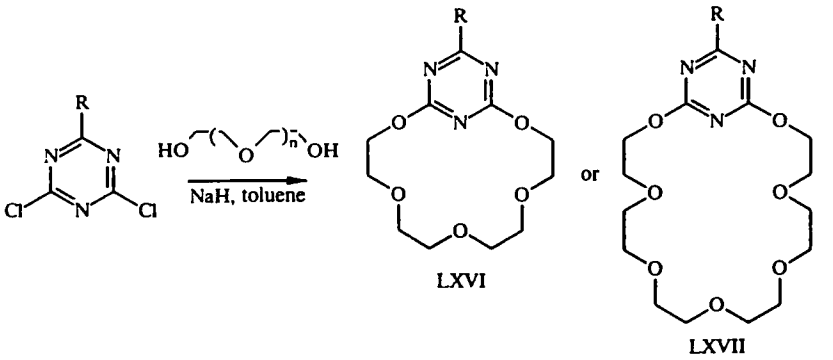
4,6-Pyrimidinocrown ethers (LXV) were obtained by a similar method in the reaction of 4,6-dichloropyrimidine with di-, tri-, and tetraethylene glycols in the presence of sodium hydride and gave small yields (3%), irrespective of the value of *n* or the size of the obtained macrocycles (LXV) [42].



n = 1, 2, 3

LXV

A series of macrocyclic crown ethers containing a 1,3,5-triazine fragment in the ring were synthesized for investigation as surface-active catalysts [43]. In the reaction of 2-alkyl- or 2-alkoxy-4,6-dichloro-1,3,5-triazines with tetra- or hexaethylene glycols $HO(CH_2CH_2O)_nH$ (n = 4, 6), which form anions with sodium hydride, the 4-substituted 1,3,5-triazinocrown ethers (LXVI, LXVII) were obtained.

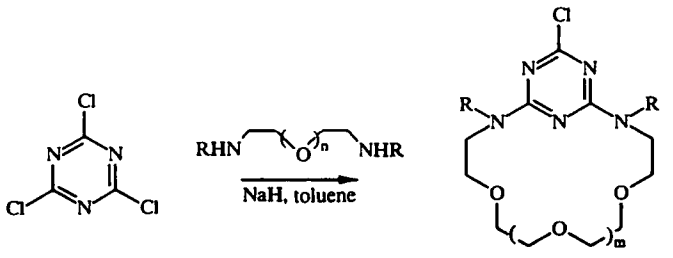


LXVI LXVII

n = 3, 5; R = MeO, $C_6H_{13}O$, C_8H_{17}, $C_{12}H_{25}$, $C_{16}H_{33}$

The nature of the substituents in the triazine rings of the macrocycles (LXVI) and (LXVII) ensures the presence of the lipophilic centers required for the activity of the surface-active catalysts.

6-Chloro-1,3,5-triazinocrown ethers (LXVIII) were synthesized as ligands for study of the selectivity during the aminolysis of cyanuric chloride with the diamines of oligoethylene glycols [44].



LXVIII

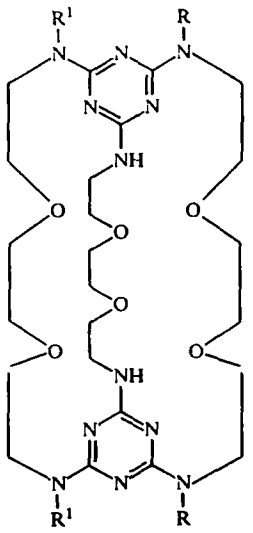
n = 2, 3, 4, 5; m = 0, R = H; m = 1...3, R = Me; m = 1, R = $PhCH_2$

Cryptands with 1,3,5-triazine fragments in the ring were described. (Cryptands are crown ethers containing tertiary nitrogen atoms in the linking chains.) The cryptands (LXIX) and (LXX) were obtained by aminolysis of 1,3,5-cyanuric chloride by α,ω-diaminooligoethylene glycols or diazacrown ethers respectively [45].

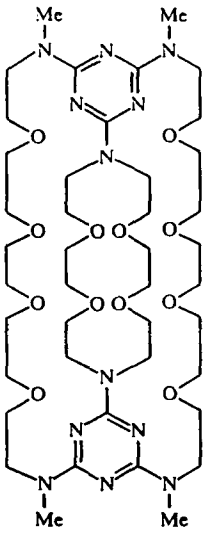
The metal complexes of cryptands are more stable than the crown ethers, and they are formed more selectively.

Thus, the data presented in the review indicate that among all the known types of macrocycles there are representatives containing azine fragments in the ring. A large number of papers have been published on pyrimidino- and purinomacrocycles.

This is explained by the special significance of the derivatives of these heterocycles in biology as bases forming part of the chains in nucleic acids.



LXIX



LXX

n = 1...4; R = H, Me, $(CH_2)OH$; R^1 = Me, $(CH_2)OH$

The presence of azine fragments in the types of macrocycles under discussion extends their application range both in theoretical and in practical respects.

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