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Methods for the synthesis of polycyclic nitramines

S V Sysolyatin, G V Sakovich, V N Surmachev

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Abstract. **Published data on the methods of synthesis of polycyclic nitramines are analysed. The advantages, drawbacks and prospects of two main approaches to the formation of the molecular cage, namely, amine or amide condensation with carbonyl compounds and consecutive formation of the polycyclic cage with introduction of reactive terminal substituents, are discussed. The bibliography includes 64 references.**

I. Introduction

Currently, polycyclic nitramines (PCNA) are becoming significant as promising high-energy compounds, which are superior to other explosives in some properties. They have high density, which improves the explosive characteristics without increasing the sensitivity to mechanical action. High content of hydrogen in PCNA (unlike aromatic heterocyclic compounds) results in an increase in the heat of explosive transformation and a decrease in the average molecular mass of the detonation products.

Polycyclic nitramines meet the requirements imposed on promising components of high-energy compositions: they have the optimum oxygen balance, moderate enthalpy of formation and high density.

There are rather few studies devoted to the synthesis of PCNA, most being concerned with the methods of synthesis of 2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazatetracyclo-[5.5.-0.$0^{3,11}$.$0^{5,9}$]dodecane (hexanitrohexaazaisowurtzitane, CL-20, HNIW).[1] The present review describes systematically the known methods for the synthesis of PCNA, including approaches to the formation of the polycyclic cage and ways of substitution of nitro groups for functional groups at the nitrogen atoms.

The cages of most polycyclic nitramines are synthesised in one step. This way is most convenient for industrial implementation and, as a rule, represents the condensation of amines or amides with carbonyl compounds.

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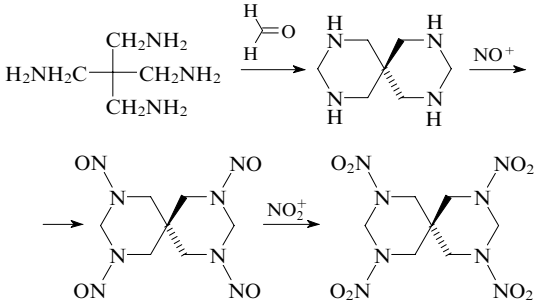
The second stage is introduction of nitro groups. This is accomplished by substituting them for *N*-alkyl and functional (*N*-acyl, *N*-sulfonyl) groups.[2–6] Nitrolysis of protected secondary amines for the synthesis of cyclic nitramines is fairly well studied.[7] Of the alkyl substituents, the *tert*-butyl group is replaced most easily. In the series of acyl substituents (CHO, Ac, EtCO, Me_2NCO, ROCO, ArCO), acetyl group is replaced most easily by the nitro group, while the replacement of formyl and propionyl groups requires more drastic conditions. The sulfo group, which undergoes nitrolysis under very mild conditions, is the substituent of choice for the introduction of the nitro group.

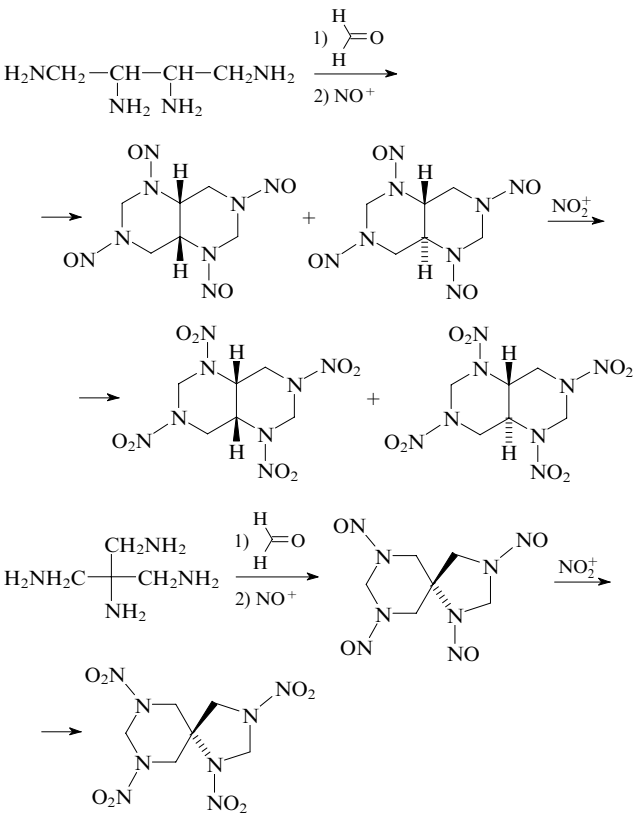
Thus, the main problem in PCNA synthesis is construction of a polycyclic cage with *N*-substituents appropriate for substitution of nitro groups. The simplest method for the construction of the hexaazaisowurtzitane cage is condensation of glyoxal with benzylamine or its derivatives. Direct nitrolysis of hexabenzylhexaazaisowurtzitane does not result in HNIW; the major part of investigations on this subject are devoted to the transformation of benzyl groups into substituents suitable for the replacement by the nitro groups.[1]

II. Synthesis of polycyclic nitramines by condensation of amines and their derivatives with formaldehyde

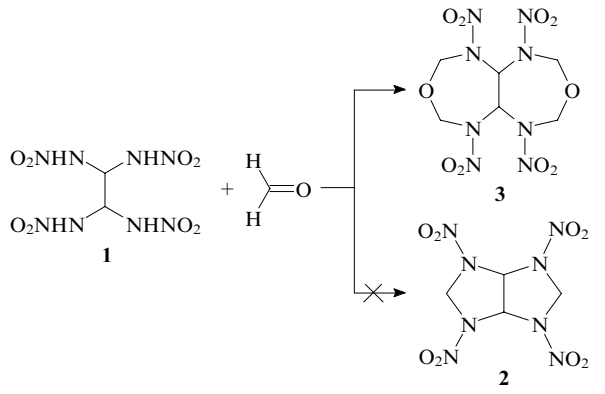
The polycyclic cage of nitramines can be formed by condensation of tetramines with formaldehyde. The condensation products are labile compounds, and they are usually converted into the corresponding nitroso derivatives, which are readily nitrated to nitramines.

This method was used to synthesise[8–10] a number of bicyclic nitramines.

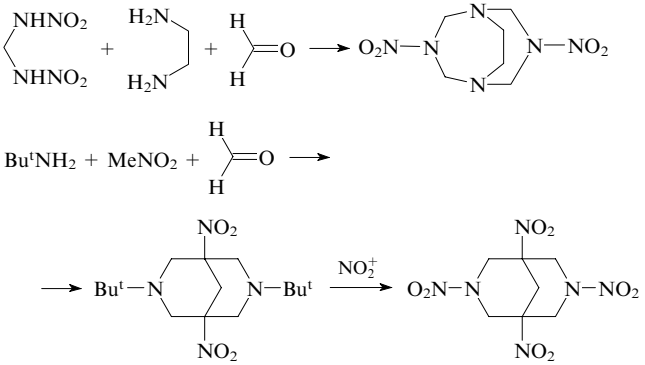




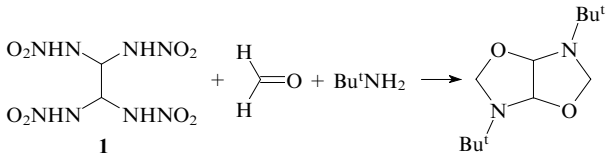
The reaction of 1,1,2,2-tetrakis(nitramino)ethane (**1**) with formalin gives, instead of the expected 2,4,6,8-tetranitro-2,4,6,8-tetraazabicyclo[3.3.0]octane (**2**), bicyclic compound **3** containing nitrogen and oxygen atoms in the seven-membered heterocycles.[11]



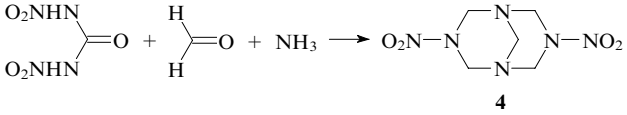
The cage of bicyclic nitramines can be prepared by aminomethylation of compounds with an active hydrogen atom (the Mannich condensation):[2, 12]



The reaction of nitraminoethane **1** with formalin and *tert*-butylamine results unexpectedly in elimination of nitramine groups.[13]



The reaction of *N*,*N*′-dinitrourea with ammonia and formaldehyde gives dinitropentamethylenetetramine **4**, which is the starting compound for the synthesis of octogen.[14] It is assumed that *N*,*N*′-dinitrourea is hydrolysed to nitramide during the synthesis.

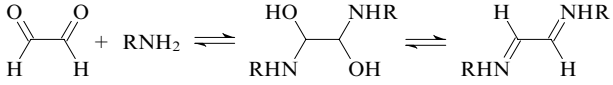


Despite the simplicity and, hence, attractiveness of the PCNA synthesis by condensation of amine derivatives with formaldehyde, it should be noted that compounds with high explosion characteristics cannot be obtained in this way.

III. Synthesis of polycyclic nitramines by condensation of amines and their derivatives with glyoxal

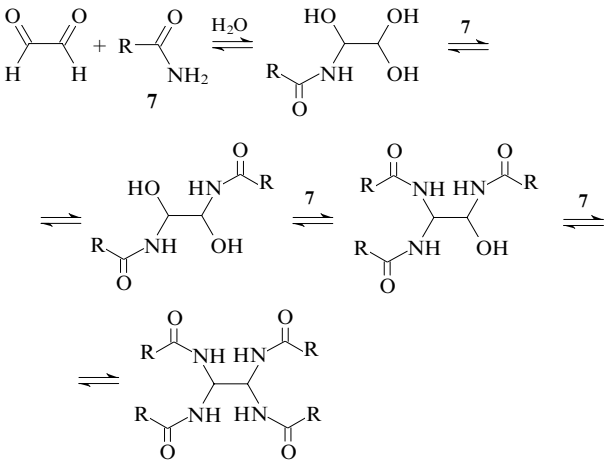
Glyoxal is widely used to prepare nitrogen-containing heterocyclic compounds.[15, 16] In an aqueous solution of glyoxal, glyoxal hydrate exists in equilibrium with its hydrated oligomeric derivatives.[17]

The condensation of glyoxal with primary amines affords mainly 1,2-diaminoethylene glycols **5**, which readily eliminate water being converted into conjugated diimines **6**.[18, 19]



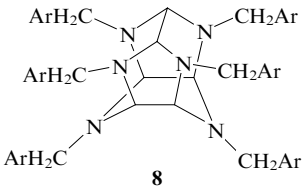
R = Bu^n, Bu^i, Bu^t, cyclo-C$_6$H$_{11}$, 4-MeC$_6$H$_4$, 4-MeOC$_6$H$_4$, 4-HOC$_6$H$_4$.

The reactions of glyoxal with primary amides **7** afford products with different degrees of substitution depending on the reactant ratio and reaction conditions.[20–25]



R = Me, Et, OMe, OEt, OPr^i, All, Bn, Ph.

Compounds **5** and **6** where R is the benzyl or substituted benzyl group (R = $ArCH_2$) usually serve as precursors of hexaazaisowurtzitane derivatives **8**.[1]

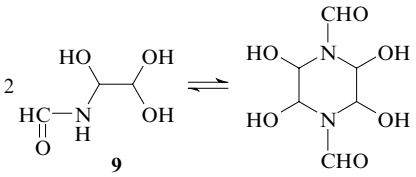


8

Ar = Ph, XC_6H_4 (X = 4-Me, 2-Me, 4-MeO, 2-MeO, 4-Pr^i, 4-Cl, 2-Cl, 2-F, 2-Br), 3,4-$(MeO)_2C_6H_3$.

Recently, similar hexaazaisowurtzitanes were synthesised[26] using other amines (R is furfuryl, allyl, propargyl, 3-pyridylmethyl, cinnamyl, 2-thienylmethyl, 1-naphthylmethyl).

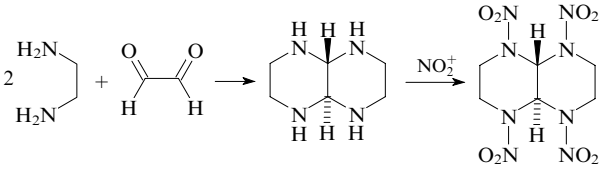
Dimerisation of compound **9** gives rise to piperazine derivative **10**.[27]



9 **10**

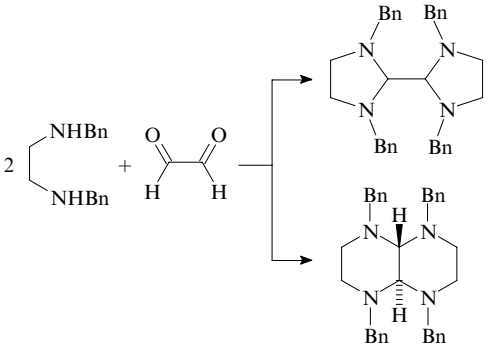
No polycyclic structures are formed in this case.

The reaction of ethylenediamine with glyoxal follows different routes depending on the reactant ratio and reaction conditions. When the molar ratio is 2 : 1, the reaction gives *trans*-fused tetraazadecalin; its nitration results in 2,5,7,10-tetranitro-2,5,7,10-tetraazabicyclo[4.4.0]decane (**11**).[28]

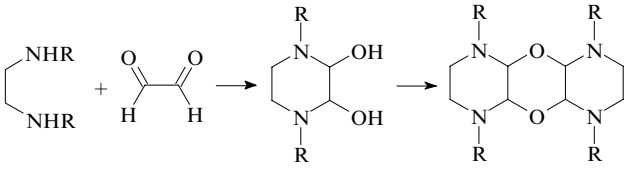


11

The reaction of *N*,*N*′-dibenzylethylenediamine with glyoxal in 2 : 1 ratio yields cyclic products containing fused and non-fused heterocycles.[29]

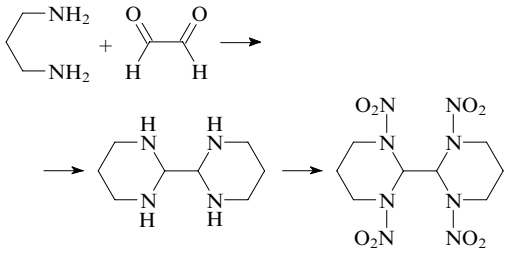


In the case of 1 : 1 molar ratio of the reactants, a tricyclic compound is formed in a relatively low yield (< 10%).[30, 31]

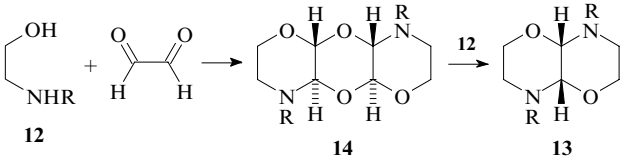


R = Me, Pr^i, Bn.

The use of a longer hydrocarbon chain between the amino groups in the diamine results in compounds with non-fused 1,3-diazacycles.[32]



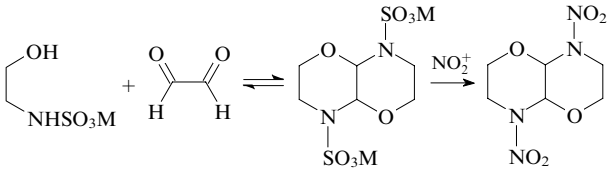
Glyoxal reacts with aminoethanols **12** to give oxaazabi- (**13**) and -tricyclic (**14**) compounds.[33]



12 **14** **13**

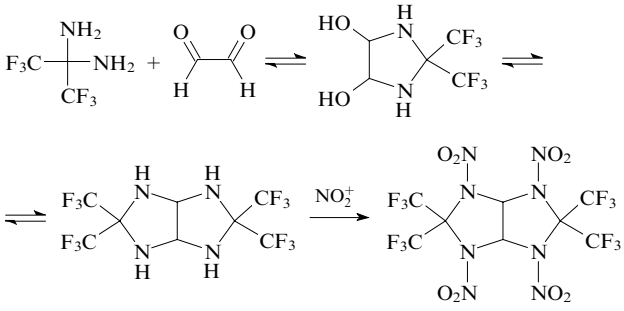
R = Me, Et, Bu^n, Ph.

The use of *N*-(2-hydroxyethyl)sulfamate in the condensation allows one to obtain the bicyclic cage and easily replace the sulfo group by the nitro group.[34]

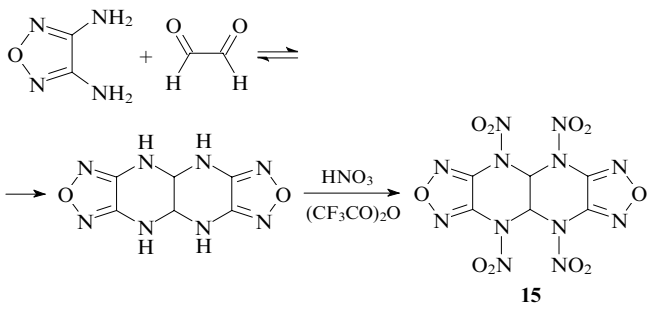


M = Na, K.

2,2-Diaminohexafluoropropane is a stable compound (unlike diaminomethane); it reacts with glyoxal to afford a bicyclic product, which is nitrated to give bicyclic nitramines.[35]

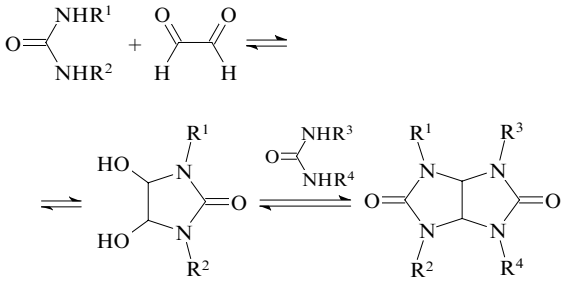


Weakly basic diaminofurazan is also readily coupled with glyoxal.[36, 37] Nitration of the coupling product results in a series of nitro derivatives, including the fully nitrated compound **15**.



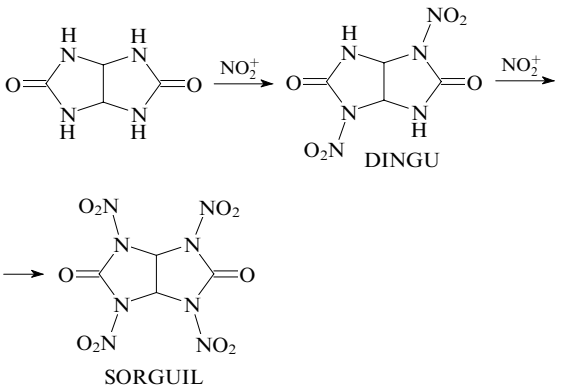
15

The reactions of glyoxal with urea derivatives proceed very easily to afford glycoluril.[38–41]



R^1, R^2, R^3, R^4 = H, Me, Et, Ph, CH_2OH.

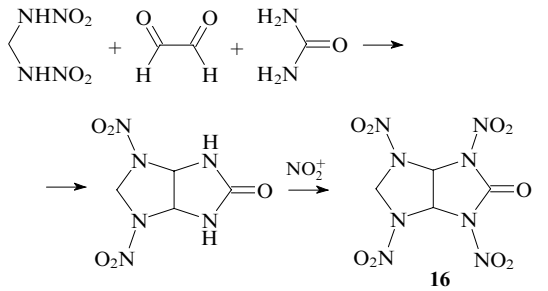
Nitration of glycoluril gave a series of its nitro derivatives, among which DINGU and SORGUIL (TENGU) are distinguished as regards explosion characteristics.[42,43]



DINGU

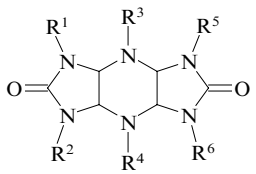
SORGUIL

In order to prepare 2,4,6,8-tetranitro-2,4,6,8-tetraazabicyclo[3.3.0]octane, attempts at multicomponent condensation of amines with amides and glyoxal or formaldehyde have been undertaken.[38,44] Thus condensation of *N*,*N*′-dinitromethylenediamine with urea and glyoxal afforded[45] 2,4,6,8-tetranitro-2,4,6,8-tetraazabicyclo[3.3.0]octan-3-one (**16**).



16

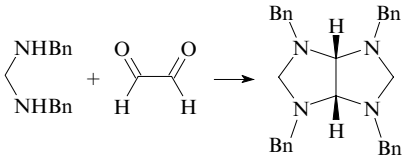
A series of tricyclic derivatives of urea **17a–e** were synthesised in a similar way.[46]



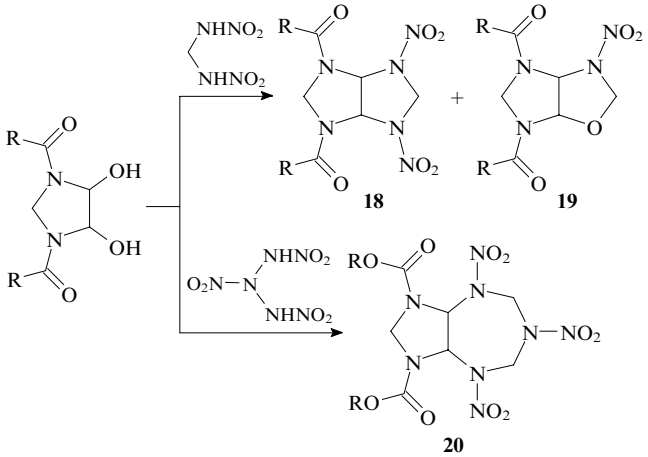
17a–e

Compound **17**	R^1	R^2	R^3	R^4	R^5	R^6
a	H	H	H	H	H	H
b	NO_2	H	NO_2	NO_2	NO_2	H
c	NO_2	H	NO_2	NO_2	H	NO_2
d	NO_2	NO_2	NO_2	NO_2	H	NO_2
e	NO_2	NO_2	NO_2	NO_2	NO_2	NO_2

In the search for synthetic routes to bicyclic nitramines, coupling of methylenediamine derivatives with glyoxal was studied. The condensation of *N*,*N*′-dibenzyldiaminomethane with glyoxal resulted[47] in a *cis*-fused bicyclic system.



The reaction of *N*,*N*′-diacyl-4,5-dihydroxyimidazolidine (obtained by condensation of methylenediamide with glyoxal) with dinitramines resulted[48–51] in a number of bicyclic structures **18–20**. However, attempts to replace the acyl substituents at the nitrogen atoms by the nitro groups failed.



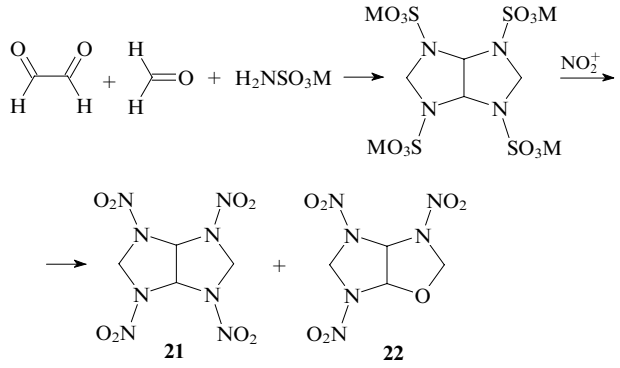
18 **19**

20

R = Me, Et.

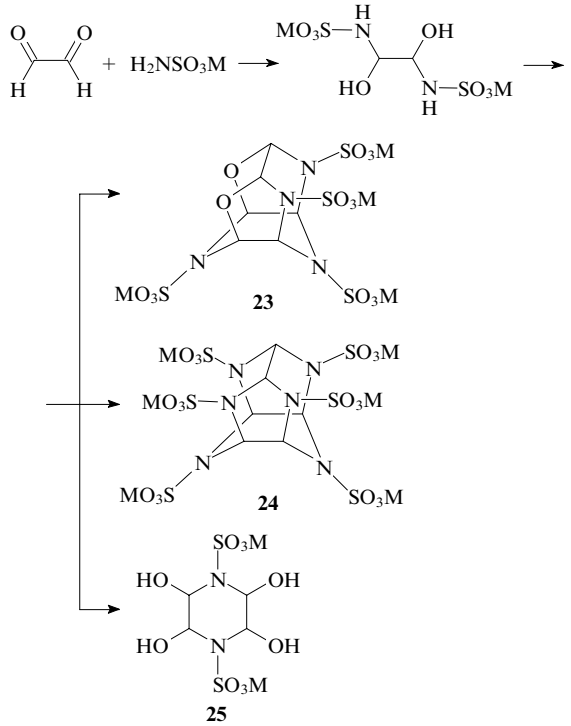
The coupling of glyoxal with metal sulfamates deserves special attention. The sulfo group is easily replaced by the nitro group, which makes it the protecting group of choice among those used for the synthesis of polycyclic nitramines. The coupling sulfamates with carbonyl compounds forms not only mono- and bi-, but also tetracyclic products.

The multicomponent condensation of glyoxal with formaldehyde and sulfamates followed by nitration of the bicyclic product with concentrated nitric acid at −30 °C affords 2,4,6,8-tetranitro-2,4,6,8-tetraazabicyclo[3.3.0]octane (**21**) (or, at 0 °C, its oxo analogue **22**).[52]



M = Na, K.

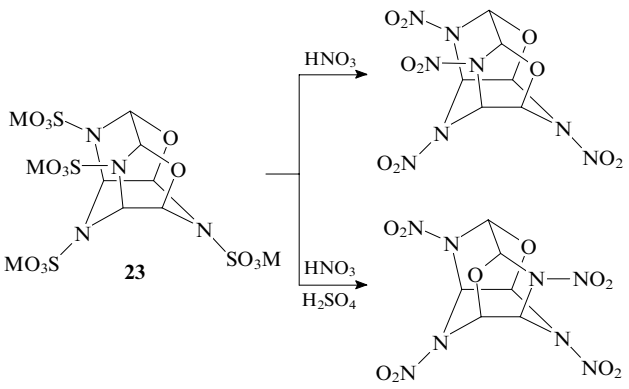
The reaction of glyoxal with sulfamates proceeds in different ways,[52, 53] depending on the reactant ratio and reaction conditions.

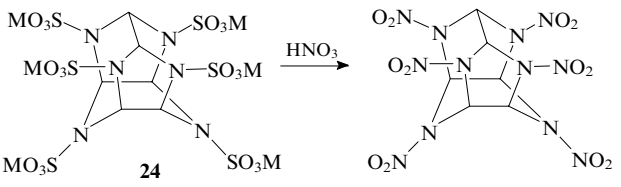


M = Na, K.

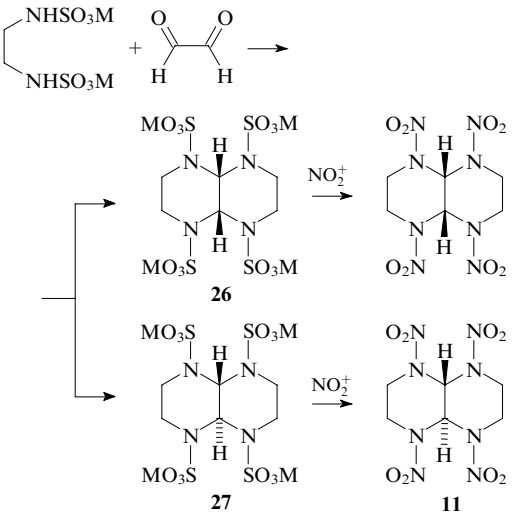
Compound **23** and **24** with the isowurtzitane structure are formed in an acid medium (pH 2.8–4.0) at 50–65 °C. At lower temperature and acidity, salts of 2,3,5,6-tetrahydroxypiperazine-1,4-disulfonic acid (**25**) are mainly formed.

Nitrolysis of sulfonates **23** and **24** with nitric acid–based mixtures proceeds under relatively mild conditions and results in nitramines with the isowurtzitane structure.[52–54]





The reaction of ethylenediaminosulfamate with glyoxal at various temperatures affords *cis*- (**26**) and *trans*-fused (**27**) tetraazadecalin derivatives, which were nitrated to give bicyclic nitramines.[55]

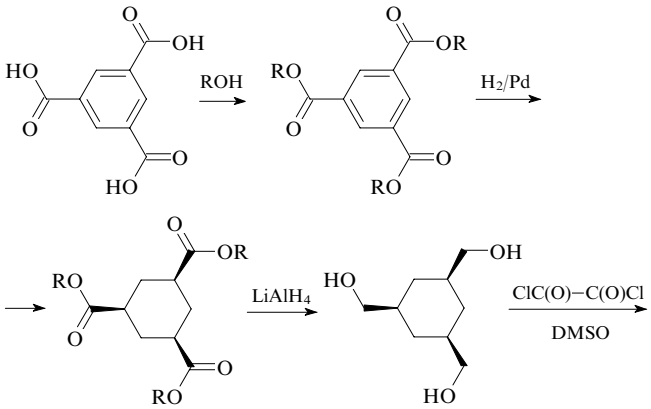


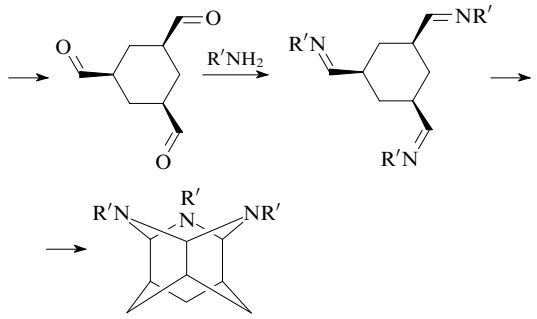
M = Na, K.

Thus, the reactions of amines and their derivatives with glyoxal provide a broad range of PCNA with different structures. Some of them present practical interest and are produced in industry. The main trend of future works would involve the search for economical two-step methods for the synthesis of hexanitrohexaazaisowurtzitane, the most powerful explosive among the PCNA synthesised and the study of application of PCNA and their precursors as biologically active compounds.

IV. Miscellaneous methods for the synthesis of polycyclic nitramines

The following method for the construction of the 3,5,12-triazawurtzitane cage **28** has been proposed:[56, 57]



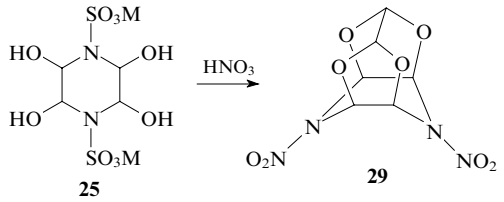


28

R = Me, Et, Pr^n; R′ = Me, Et, Bn, 4-$MeOC_6H_4CH_2$, 4-$Me_2NC_6H_4CH_2$.

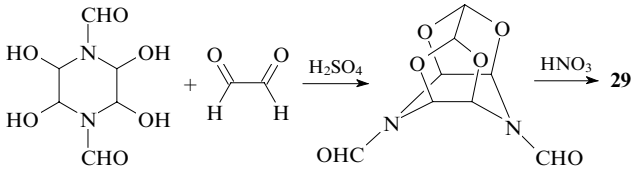
However, the attempts to carry out substitution at the nitrogen atoms in compound **28** failed.

Nitration of the condensation product of the sulfamate salts with glyoxal (**25**) gave 4,10-dinitro-2,6,8,12-tetraoxa-4,10-diazatetracyclo[5.5.0.$0^{3,11}$.$0^{5,9}$]dodecane (**29**).[53,54]

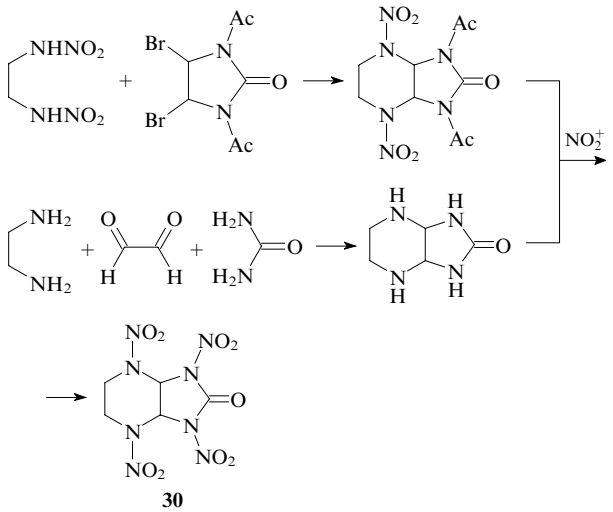


25 **29**

This compound can also be prepared in a different way.[58]



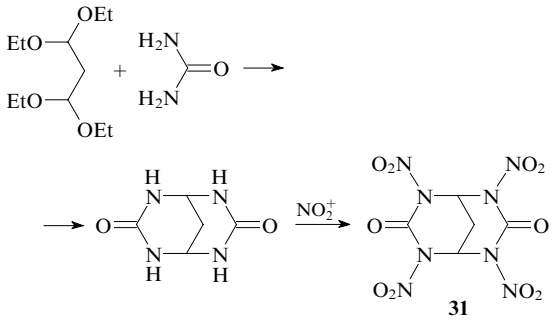
Alkylation of ethylenedinitramine with 1,3-diacetyl-4,5-dibromoimidazolidin-2-one with subsequent nitrolysis results in bicyclic urea derivative **30**. This compound was also prepared by nitration of the condensation product of ethylenediamine with urea and glyoxal.[59,60]



30

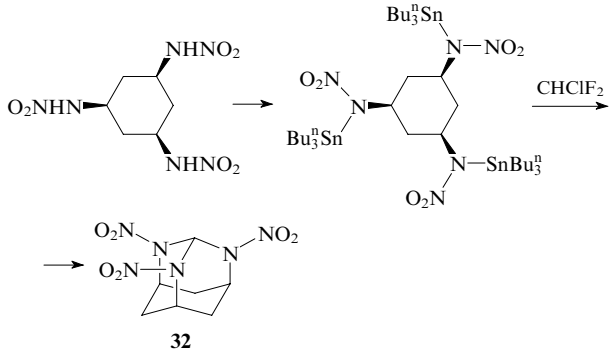
Polycyclic structures called cucurbiturils were prepared by condensation of glycoluril with formaldehyde,[61] and hexacyclic systems were formed upon the reaction of glycoluril, formaldehyde, and diamines.[62] The attempts to introduce nitro groups in these compounds were unsuccessful.

The reaction of urea with 1,1,3,3-tetraethoxypropane and subsequent nitration yielded 2,4,6,8-tetranitro-2,4,6,8-tetraazabicyclo[3.3.1]nonane-3,7-dione (**31**).[60]



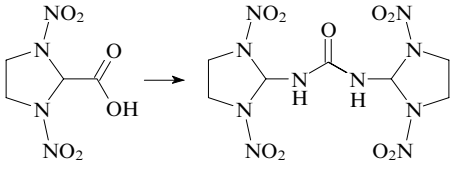
31

Alkylation of a stannyl derivative of tris(nitramino)cyclohexane was used to synthesise 2,4,10-trinitro-2,4,10-triazaadamantane (**32**).[56]

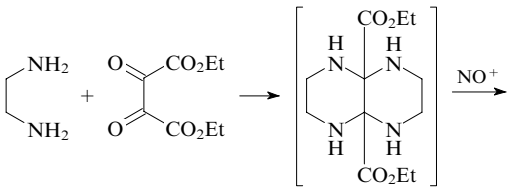


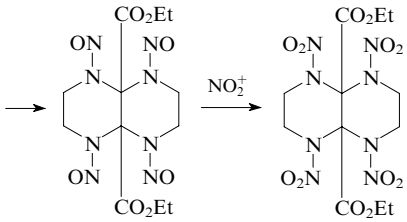
32

The possibility of attachment of an amino group at the carbon atom already containing two nitramino groups would give a broad range of PCNA with a tris(nitramino)methane fragment in the molecule, for example, hexanitrohexaazaadamantane. According to calculations, the introduction of terminal amino groups into the PCNA structure would sharply increase the density and explosion characteristic of the compound. Due to the ability of the carboxy group to be replaced by the amino group, the former is a preferable terminal group for the PCNA synthesis.[63]



The reaction of ethylenediamine with dioxosuccinate, the fixation of the reaction product as the nitrosamine and subsequent nitration with dinitrogen pentoxide in nitric acid gives rise to a bicyclic nitramine containing carboxy groups.[63]





The methods of synthesis of polycyclic structures by consecutive formation of the molecular skeleton have not been adequately developed as yet, but they are quite promising for the design of diverse PCNA.

* * *

Analysis of puibished data allows one to distinguish two main methods for the synthesis of PCNA: one-step construction of the polycyclic core of the molecule by condensation of amines and their derivatives with carbonyl compounds and the consecutive formation of the polycyclic cage. Most of PCNA described in the literature were prepared by the former method. The second method is not adequately developed as yet but holds much promise and may yield PCNA of different structures.

The chemistry of high-energy materials, in particular, methods for their synthesis is a topical field of research being actively developed, as indicated by regular publication of new studies (see, for example, the monograph [64]).

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