

Chapter 2

An Overview of Synthetic Methods for Preparation of Nitrosoaromatic Compounds

Outline of the basic preparative methods that include three possible ways of the synthesis of aromatic C-nitroso compounds are represented. Besides the classical methods of reduction of nitro- or oxidation of amino precursors, the approaches based on the direct nitrosation, solid-state syntheses as well as enzyme-catalyzed reactions are commented in more details. Since the nitroso compounds easily undergo formation of dimers and azoxy products, discussion about the preparation of these derivatives is added.

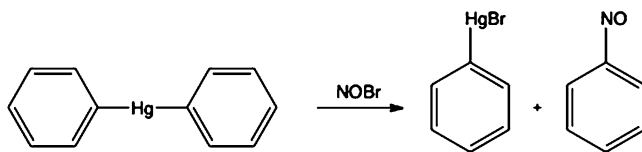
Nitrosobenzene as the parent aromatic nitroso compound has been for the first time prepared by Adolf Baeyer [1] who applied the reaction of diphenylmercury with NOBr. By the same type of reaction Baeyer also synthesized nitrosonaphthalene (Scheme 2.1).

As we have mentioned in the Introduction, since nitroso compounds easily dimerize to azodioxides, some of the synthetic methods, which were designed to prepare azodioxides also serve as methods for preparation of nitroso compounds. Because nitroso compounds appear as intermediates on the redox scale between limiting amino and nitro sides, both the synthetic options for their preparation are open, oxidation of amino-, or reduction of nitro-derivatives. Aliphatic derivatives can also be obtained by using other type of reactions, such as additions of nitrosyl-chloride on the carbon-carbon double bond, or different photoreactions. Additional complication with aliphatic nitroso compounds is their tendency to rearrange to oximes by [1,3] hydrogen shift from the alpha carbon atom (Scheme 2.2).

On the other hand, in the aromatic nitroso compounds, equilibrium between the nitroso and oxime form has been observed only in the *o*-hydroxy-substituted nitrosoaromatic molecules such as 1-nitroso-2-naphthol [2]. However, this rearrangement does not interfere with the preparation of the compound (Scheme 2.3).

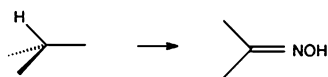
Heteroaromatic nitroso compounds (Scheme 2.4) can also exist in such tautomeric forms as it is exemplified by 4-nitroso-5-pyrazolone [3–5].

Application of redox reactions has two main difficulties. First is in finding proper conditions under which the reaction will not continue to the final oxidation or

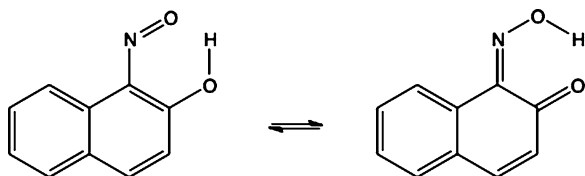


Scheme 2.1

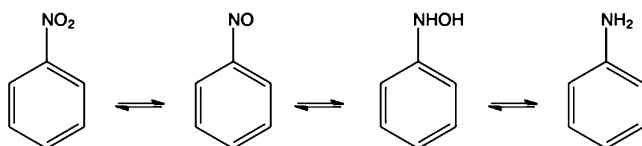
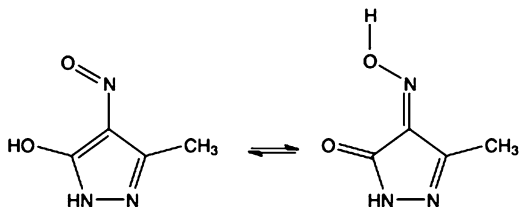
Scheme 2.2



Scheme 2.3



Scheme 2.4



Scheme 2.5

reduction product, but instead, will stop when the nitroso intermediate is formed (Scheme 2.5). The second problem has its origin in the tendency of intermediates to form azoxides as byproducts. To avoid these difficulties, besides the optimized classical synthetic pathways, a series of specific reagents as well as methods were developed. Most recently, the solvent-free solid-state preparative methods combined with purification by sublimation were used successfully. The method will be discussed later in this Chapter.

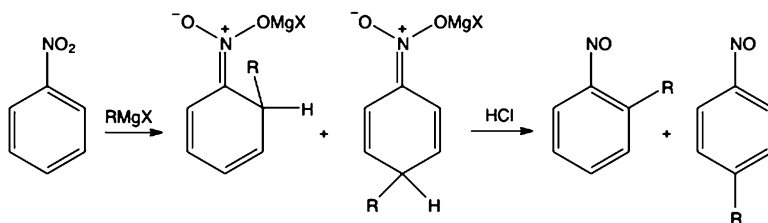
2.1 Reductive Methods

Starting material for preparation of nitroso molecules by reductive reactions are nitro-derivatives, i.e. in the case of aromatic compounds they are substituted nitrobenzenes. Historically, there is more than a 100 years long tradition in developing the reductive methods to prepare nitroso- from nitrobenzene derivatives, especially for possible applications in industrial synthesis [6]. As reducing reagents and/or catalysts, metals, metal amalgams, and even metal oxides were used [7–9]. It has been shown from results of calculations on DFT levels of theory, there are specific mechanisms in which metal or metal oxide reductions include single electron transfer [10]. Some of manganese oxide catalysts became already classics in the large-scale preparations. While strong conventional reductive reagents, such as Sn, $\text{SnCl}_2 + \text{HCl}$, $\text{Fe} + \text{HCl}$, or H_2 , Pt, transform nitro- to amino group, weaker reductive reagents, for instance Zn powder, can under controlled pH conditions yield hydroxylamine [11, 12], which in the next step can be re-oxidized to the nitrosobenzene by using FeCl_3 or $\text{Na}_2\text{Cr}_2\text{O}_7$ in sulfuric acid, as it is represented in the following equation. The method can widely be used in laboratory synthesis of nitrosoaromates. However, the yields obtained in these preparations are not always satisfactory because of the formation of byproducts, mainly azoxides and nitroaromates.

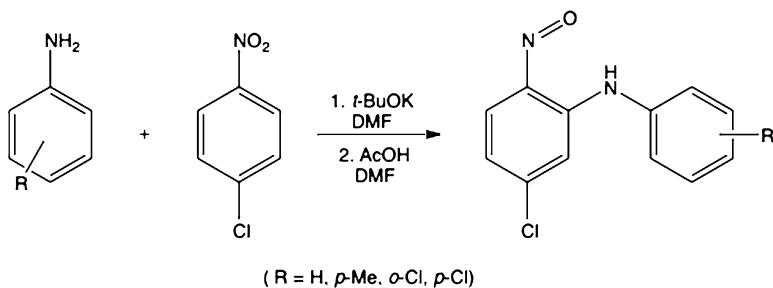


In the second step, the oxidation of hydroxylamine, a variety of other mild oxidants such as periodic acid [13], periodates [14, 15] or silver carbonate [16] could be useful. Main disadvantage in using such an oxidative method is again the appearance of products of a side reaction. Namely, nitrosobenzene forms azoxybenzene byproduct in the condensation reaction with hydroxylamine intermediate [17].

A number of polycyclic aromatic nitro compounds, especially heterocyclic systems can be reduced to nitroso analogs also by addition of Grignard reagents followed by reaction with strong acid [18, 19]. Unfortunately such a method frequently yields a mixture of *ortho* and of *meta* substituted derivatives, as it is shown on the Scheme 2.6.

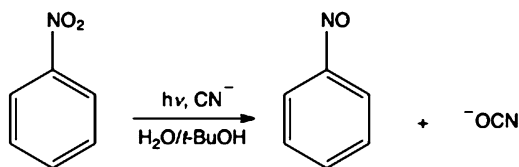


Scheme 2.6



Scheme 2.7

Scheme 2.8



Substituted nitrosobenzenes can also be prepared by direct reaction of aniline with corresponding nitrobenzene. The 2-nitroso-*N*-arylanilines (Scheme 2.7) were synthesized in a one-pot reaction from anilines and 1-chloro-4-nitrobenzene [20, 21].

Beside such “classical” methods, a series of the electrochemical and photoreductions of nitroaromatic precursors were developed. In principle, electrochemical reductions follow the same reaction pathway through formation of hydroxylamine intermediate [22, 23]. These methods include either a standard electrochemical synthesis or specially constructed electrodes [24–27]. Most of differently substituted nitrobenzenes with substituents in all positions, *ortho*, *meta* or *para*, were reduced to nitroso derivatives with high yield. By using a gold electrode, the aromatic nitro compounds can be reduced to nitroso derivatives by electrochemical reaction on the electrode surface.

Photoreduction of substituted nitrobenzene in most cases does not end with formation of corresponding nitroso compounds. Rearranged products with substituted *ortho* positions are preferred. Of the synthetic value could be photoreaction of nitrobenzene in the presence of potassium cyanide (Scheme 2.8). Oxygen atom is from nitro group removed as a cyanate anion [28]:

This reaction is of limited use because the nitroso products can be obtained only in the case of *para* substituted halogen derivatives. Nitronaphthalene is in similar reaction reduced exclusively to naphthalene.

Although metal- or metal-oxide catalysed photoreductions are less effective than photooxidations [29], careful selection of the catalyst could be useful not only for the preparation of nitro-, but also for nitrosoaromatic compounds. One of the promising catalysts is titanium dioxide, which promotes photoreduction of nitrobenzene [30, 31].

Some amount of nitrosobenzene was also isolated as one of products of enzymatic reduction of nitro compounds. It was already known from the industrial experience that some flavoenzymes (called “Old Yellow Enzyme”, OYE) are effective in biodegradation of nitro-explosives [32]. Recently, a wide variety of stereoselective reductions supported by enzymes such as OPR1 and OPR3 from *Lycopersicon esculentum* [33], OYE from *Saccharomyces carsbergensis* and OYE2-3 from *Saccharomyces cerevisiae* [34], NCR from *Zymomonas mobilis* [34], YqjM from *Bacillus subtilis* [33], PETN reductase from *Enterobacter cloacae* [35] were tested. Nitroreductase from *Salmonella typhimurium* [36] transforms nitrobenzene in the mixture consisting from nitrosobenzene, phenylhydrazine, as well as diphenylazodioxide.

2.2 Oxidative Methods

Preparation of aromatic nitroso compounds from amino precursors is more practical because of numerous available oxidative methods. A series of peroxoacids may be applied for oxidation of aromatic amines, peroxomonosulfuric acid (Caro's acid) [37, 38], peroxyacetic acid [39], 3-chloroperoxybenzoic acid [40], or peroxyformic acid [41, 42]. Hydrogen peroxide in combination with organometallic catalysts (peroxotungstophosphate) [43], $[\text{Mo}(\text{O})(\text{O}_2)_2(\text{H}_2\text{O})(\text{hmpa})]$ [44], *cis*- $\text{Mo}(\text{O})_2(\text{acac})_2(\text{acacH}=\text{MeC}(\text{O})\text{Me})$ [45], methylrhenum trioxide [46], oxoperoxo(pyridine-2,6-dicarboxylato)(hmpa)molybdenum(VI) [47], as well as with inorganic salts such as sodium tungstate or sodium-EDTA [48] is known to give nitrosoaromatics in good yields. The requirements for environmentally adequate methods, the green-chemistry preparations, prompted scientists to find new catalysts such as heteropolyacids (HPAs) [49]. In combinations with hydrogen peroxide HPAs can effectively oxidize aniline to nitrosobenzene [50]. Oxidation can be performed in triphase system comprising an aqueous solution of hydrogen peroxide, an organic solvent, and Aliquat 336 surfactant. It must be pointed out, that most of the investigated HPAs afford almost 100 % conversion with high selectivity in favor of nitrosobenzene over nitrobenzene [50].

Classical oxidants such as potassium permanganate in mixture of formaldehyde and sulfuric acid [51] could also be used. Perhaps the best results were obtained by using mixture of sodium peroxosulfate with sodium sulfate and sodium hydrogen-sulfate, the reagent known under popular name *Oxone* [52, 53].

2.3 Solid-State Syntheses

The last mentioned reagent (Oxone) is also very convenient for the recently investigated solvent-free reactions. Grinding of powdered samples of substituted anilines with oxone yields nitroso compounds, which can easily be sublimed out of the

Table 2.1 Yields of the solid-state preparation of differently substituted nitrosobenzenes (Data from Ref. [53])

Substituent	<i>p</i> -Cl	<i>p</i> -Br	<i>p</i> -I	<i>p</i> -NO ₂	<i>p</i> -CH ₃
Yield %	70	80	85	26	52
Purity %	80	95	92	90	69
Milling time/min	20	20	20	30	20
NaHCO ₃ addition	Yes	Yes	Yes	No	Yes

reaction mixture [54]. The reaction is more efficient if sodium hydrogencarbonate is added as a component for neutralization. The results of oxidation of differently substituted nitrosobenzenes are represented in the Table 2.1.

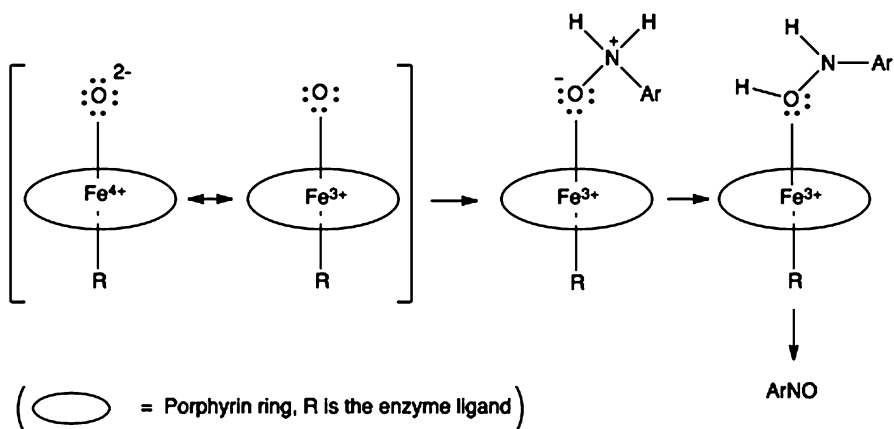
The only impurity found after sublimation was corresponding nitrobenzene.

Reactions in porous medium represent a perspective methodology in organic functional group transformations. Pillared interlayered clays (PILCs) are class of such a porous material with cavities large enough to serve for depositing metal catalysts [55]. Titania pillared montmorillonite clays with TiO₂ nanosized particles as a catalytic complex afford very high selectivity in production of nitrosobenzene and/or azoxybenzene by oxidation of aniline with H₂O₂. While low concentration of the catalyst results with 80 % yield of nitrosobenzene, high concentration of catalyst promotes high yield, almost 99 %, of azoxybenzene [56].

2.4 Enzyme Catalyzed Oxidations

Oxidation of arylamines into their corresponding nitroso derivatives is also possible by enzyme catalysis. Recent use of crude chloroperoxidase isolated from *M. paradiaciaca* for the *N*-oxidation of aromatic amines has demonstrated the efficiency of enzyme-assisted syntheses [57]. Since the enzyme active center is iron porphyrin, the proposed mechanism [57, 58] includes *N*-activation of amine by the trivalent iron cation radical complexed with oxygen. The mechanism is represented in Scheme 2.9.

In previous example, as well as in most aromatic nitrosations described to date, the nitroso derivative appears exclusively as an intermediate that is further oxidized to the corresponding nitro derivative. A series of cofactors are already known to be included in the enzymatic aminoxidations [59]. However, recent discovery of the natural enzymatic system opens a new perspective in the application of enzymes for the preparations of aromatic nitroso compounds. Noguchi et al. [60] have discovered the enzyme assigned as NspF from *Streptomyces murayamaensis* that catalyses biosynthesis of the nitroso natural product 4,3-HNBAm starting with the corresponding amine derivative. The active site of the enzyme includes two copper atoms which are able to bind oxygen atom and form the peroxo-dicopper(II) complex. Amino substrate reacts with oxygen from the complex and after elimination of water transforms directly to the nitroso derivative (Fig. 2.1).



Scheme 2.9

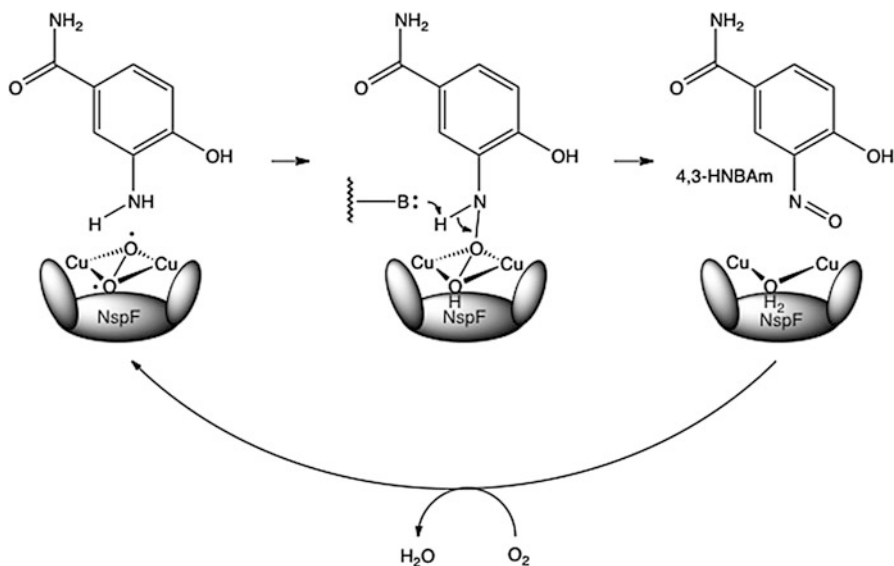


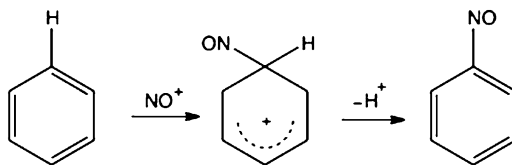
Fig. 2.1 Mechanism of the oxidation of the substituted aniline derivative by the enzyme NspF from *Streptomyces murayamaensis*

2.5 Direct Nitrosation

Relative stability of nitrosonium cation, NO^+ , affords good opportunity for direct nitrosation of arenes by electrophilic substitution reactions (Scheme 2.10).

The available methods differ in source of nitrosonium ion. In most of methods nitrosonium ion is prepared *in situ*, starting with gaseous nitric oxide in combination

Scheme 2.10



Scheme 2.11

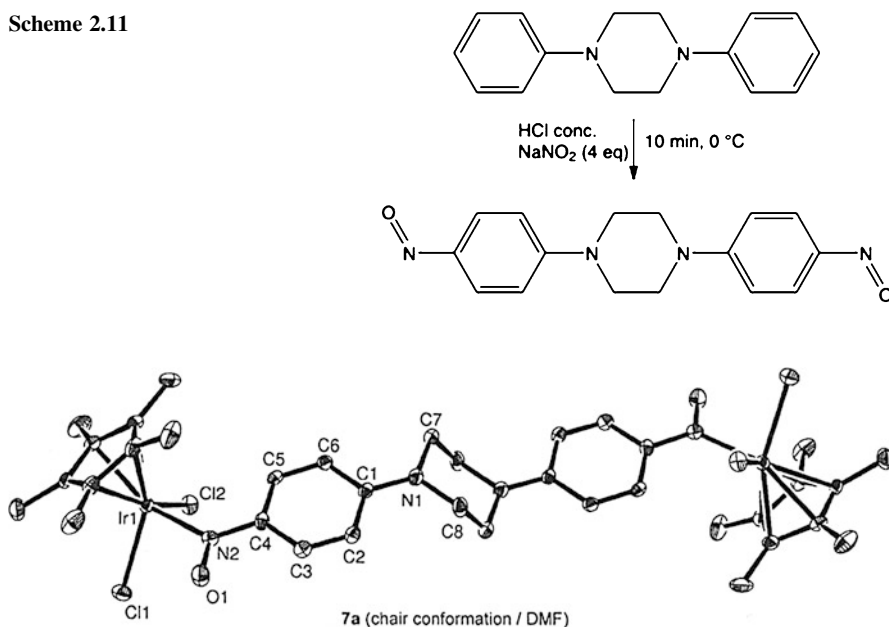
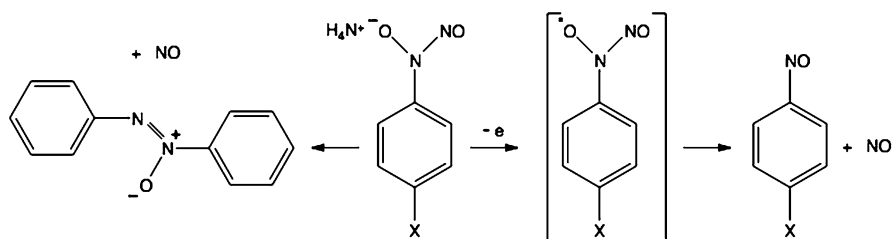


Fig. 2.2 Molecular structure of the iridium(III) metallocene complex of 1,4-bis(4-nitrosophenyl)piperazine (Reproduced by permission from the Ref. [63])

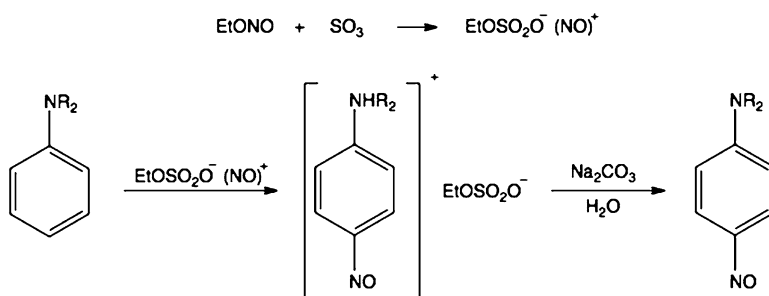
with strong (for instance trifluoroacetic) acid [61], or by mixing sodium nitrite with acid (acetic, hydrochloric) [62]. An interesting dinitroso derivative, 1,4-bis(4-nitrosophenyl)piperazine, has been prepared by this way (see Scheme 2.11) [63, 64]. The compound readily forms complexes with the iridium(III) metallocene, as in the example represented in the Fig. 2.2.

The method has also been successfully used in preparations of nitrogen containing heteroaromatic compounds. For *para*-nitrosation of *N*- or *N,N*-substituted anilines these methods are only partially useful, because in the case of *N*-monosubstituted anilines, the *N*-nitroso compounds as intermediate products were obtained.

Transformations of *N*-nitroso to *C*-nitrosoaromatic derivatives are known. An interesting example is thermal single electron oxidation or photoreaction of differently substituted *N*-nitroso-*N*-phenylhydroxylamine ammonium salt that yields azodioxides and/or corresponding nitrosobenzenes [65]. The reaction is also applicable for generation of NO (Scheme 2.12).



Scheme 2.12



Scheme 2.13

Since nitrosonium ion can be persistent as a salt of strong Lewis acids, suitable reagent for nitrosation of aromatic compounds is nitrosonium tetrafluoroborate that is especially efficient for direct nitrosation of alkyl- and polyalkyl-substituted benzenes [66].

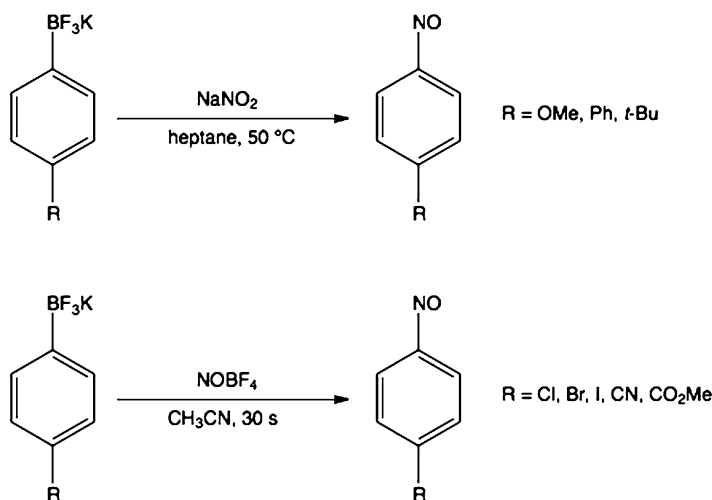
For nitrosation of arenes and arylmetal compounds the electrophilic nitrosonium agents have been systematically investigated [66–70]. However, the wider use of this type of reactions in the synthesis was limited because of the low yield, side products, and high toxicity of some organometal compounds. Efficient reagent for the preparation of *p*-dialkylamino nitrosobenzenes is nitrosonium ethylsulfate [67]. The reagent can be prepared *in situ* from EtONO and SO₃ (Scheme 2.13).

Treatment of the stable intermediate salt with sodium carbonate solution yields corresponding nitrosobenzene derivative.

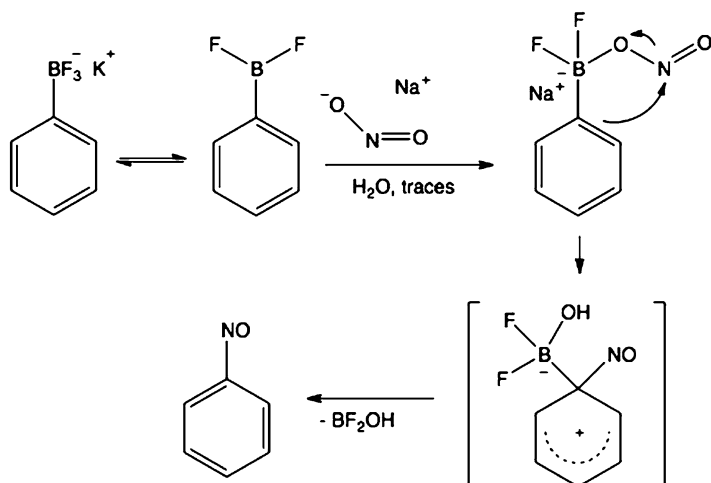
Recently, Molander and Cavalcanti [71] have developed very efficient method for nitrosation of the aromatic compounds (Scheme 2.14) by the reaction of aryl-trifluoroborate salt with NaNO₂ or NOBF₄.

In the proposed mechanism (Scheme 2.15), the first step is attack of the nitrite ion on the boron atom, followed by the rearrangement in which the NO⁺ group behaves as an electrophile.

The reaction is also applied for the nitrosation of heteroaromatics, and for *in situ* preparation of less stable nitrosoaromatic reagents, which can be used in syntheses



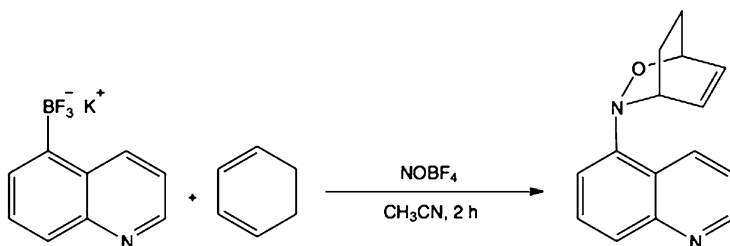
Scheme 2.14



Scheme 2.15

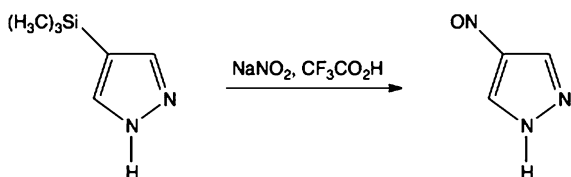
of polycyclic structures. For instance, the one pot reaction of trifluoro(isoquinolin-5-yl)borate with NOBF_4 and the corresponding diene yields the Diels-Alder adduct (Scheme 2.16).

Direct nitrosation of the heteroaromatic derivative is possible if the starting compound possesses trimethylsilyl substituent, as in the case of 4-trimethylsilylpyrazol (Scheme 2.17). Although not in high yield, the reaction of the trimethylsilylpyrazol with sodium nitrite and trifluoroacetic acid gives corresponding nitroso derivative [72].

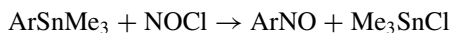


Scheme 2.16

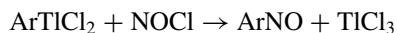
Scheme 2.17



Similarly the aromatic trimethylstanyl compounds can be transformed to the corresponding nitroso derivatives by the reaction with NOCl [73]:



Methylated nitrosobenzene derivatives can also be prepared by 50–90 % yield by reaction of aromatic organothallium compounds with nitrosyl chloride generated *in situ* [74].

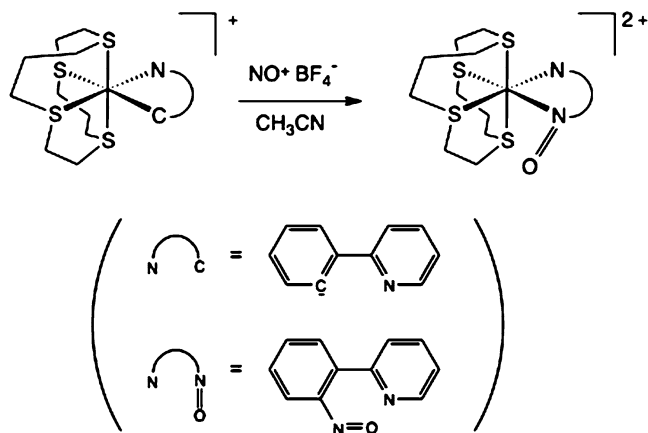


Direct nitrosation can be achieved by using the metal complexes as mediators. Insertion of NO^+ into the ruthenium-aryl bond of cyclometalated ruthenium(II) complex led to the formation of nitrosoaromatic ligands [75], as it is described in the Scheme 2.18.

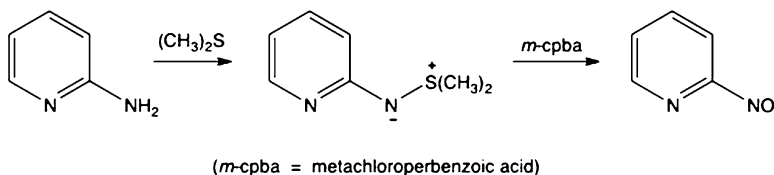
The NO distances measured by X-ray diffraction analysis fall in the range 1.235–1.244 Å, what is the expected value for the N-adduct of the nitrosoaryl ligands (see Chap. 3).

2.6 Heteroaromatic Compounds

Heteroaromatic nitroso compounds are relatively rare, and their systematic study is available only in the recent literature. Taylor et al. [40, 76] prepared parent heterocyclic nitroso compound, 2-nitrosopyridine, and their derivatives, 3-methyl-2-nitrosopyridine and 4-methyl-2-nitrosopyridine by reaction of corresponding



Scheme 2.18



Scheme 2.19

aminopyridines with dimethylsulfide and *N*-chlorosuccinimide (Scheme 2.19). The resulted sulfonium salts were deprotonated to the dimethylsulfilimides and oxidized by *m*-chloroperbenzoic acid.

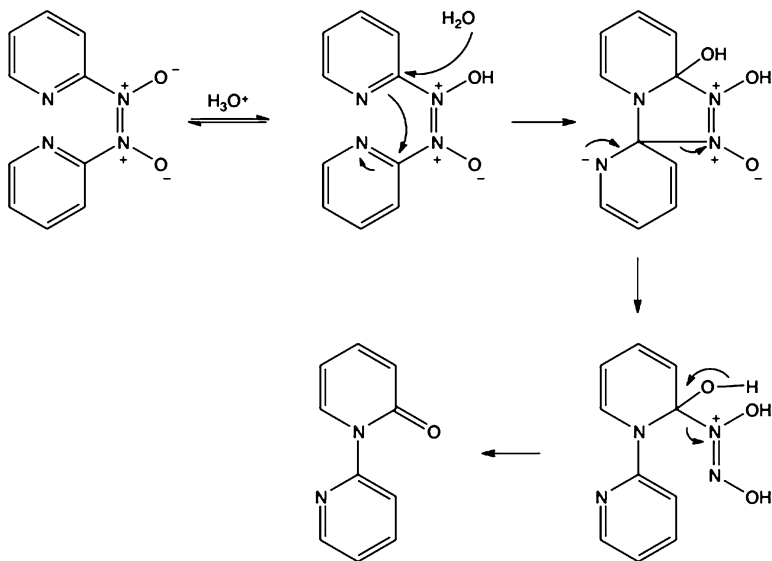
The obtained nitrosopyridine in its dimeric form can undergo hydrolysis (Scheme 2.20).

Nitrosopyrimidines can be easily synthesized by using the newly developed method by Marchal et al. [77]. A series of the nitrosoindolizine derivatives were prepared recently by Ghiviriga et al. [78].

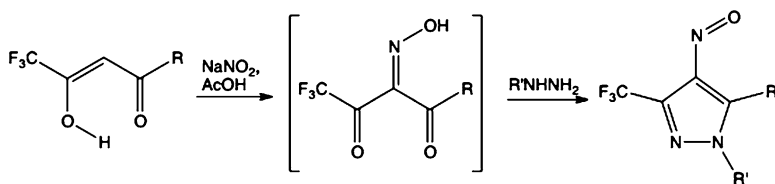
Substituted nitroso-pyrazolines (Scheme 2.21) can be prepared upon the treatment of 1,3-diketones with sodium nitrite in acetic acid and hydrazines *via* formation of the oxime as an intermediate [79].

2.7 Nitrosoaromatic Compounds with More Nitroso Groups

Of three parent compounds, *o*-, *m*-, and *p*-dinitrosobenzene, the first is unstable because of its rearrangement to benzofuroxane, and the others readily polymerize with the mechanism that will be commented in subsequent chapters.



Scheme 2.20



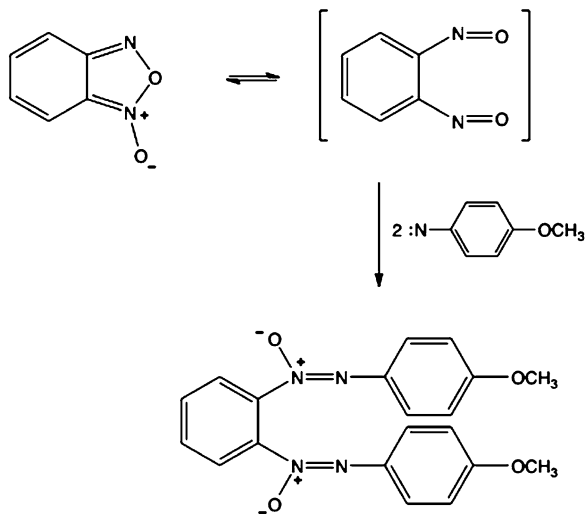
Scheme 2.21

Appearance of *o*-dinitrosobenzene by thermal rearrangement of benzofuroxan has in most cases been confirmed indirectly by trapping experiments [80–82]. Heating of benzofuroxan with *p*-anisyl azide yields diazoxy product, which is a result of the reaction of dinitrosobenzene with the *in situ* formed *p*-anisyl nitrene (Scheme 2.22) [82].

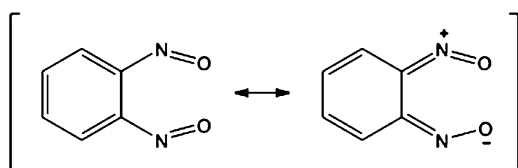
Hacker [83] succeeded to isolate 1,2-dinitrosobenzene in the argon matrix at 14 K and characterized it by IR and electron spectroscopy. Its $\text{N}=\text{O}$ stretching band appears as an intensive absorption at $1,516\text{ cm}^{-1}$. Of other characteristic signals the absorbances at 765, 790, 804, and $1,108\text{ cm}^{-1}$ were assigned to the $\text{C}-\text{N}=\text{O}$ vibrations. In the UV spectrum the maximum has been found at 266 nm. Its blue shift relatively to the spectrum of parent nitrosobenzene (281 nm) could be explained by the contribution of the quinonoid resonance structure (Scheme 2.23).

The approaches to the synthesis of tetranitrosobenzene (benzodifuroxan) [84] and hexanitrosobenzene (benzotrifuroxan) [85] are known in the literature [86]. The hexanitrosobenzene – benzotrifuroxan rearrangement represents also an interesting model for studying aromaticity phenomena (Scheme 2.24).

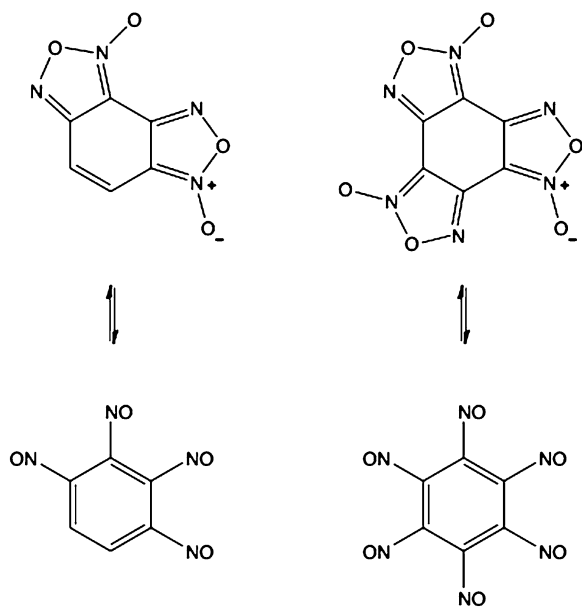
Scheme 2.22



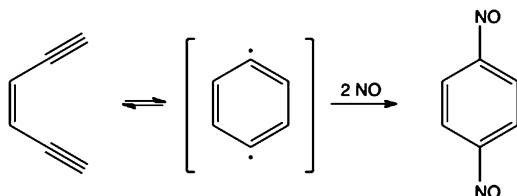
Scheme 2.23



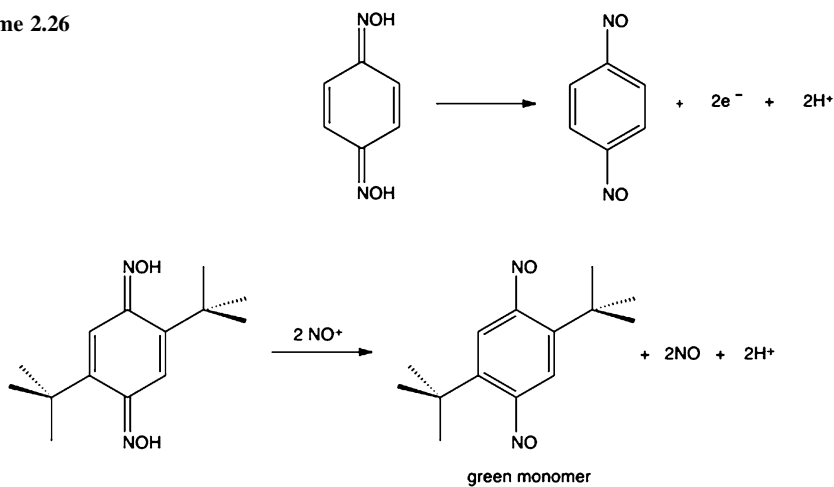
Scheme 2.24



Scheme 2.25



Scheme 2.26



Scheme 2.27

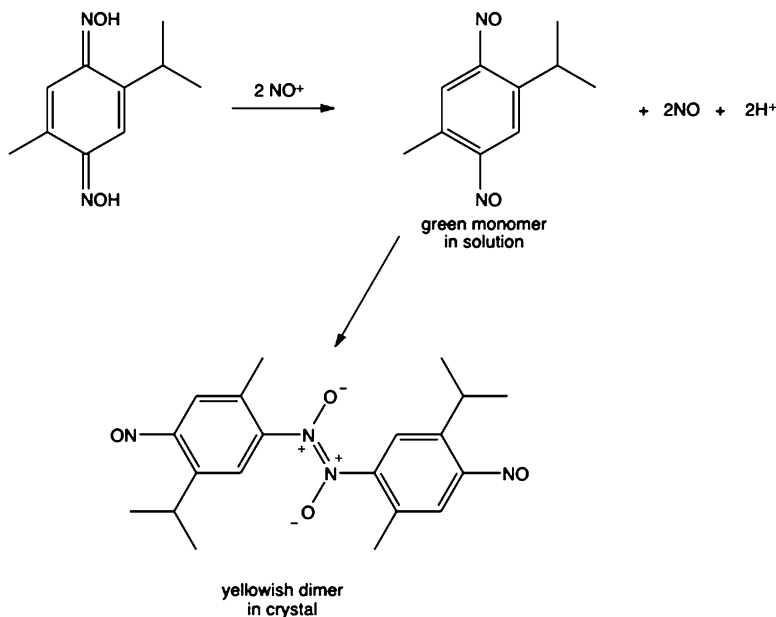
For the preparation of *m*- and *p*-dinitrosobenzenes, the classical dihydroxyamine preparative route from the corresponding dinitro precursor can be used, analogously to the preparation of mononitrosobenzenes [87]. An alternative preparation of *p*-dinitrosobenzene includes the pyrolytic reaction of *cis*-hex-3-en-1,5-diyne with nitric oxide, *via* the formation of *p*-benzine (Scheme 2.25) [88].

However, Kochi et al. [89] have developed the most practical method, which is based on the autoxidation of benzoquinone dioxime (Scheme 2.26).

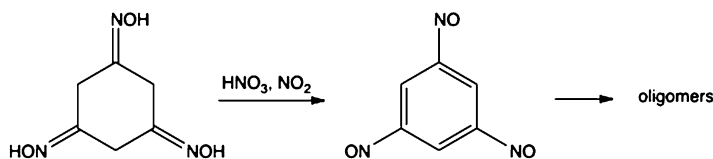
The preparation can be performed by the reaction with the nitrosonium ion, the origin of which could be either from nitrosonium tetrafluoroborate, or from disproportionation of NO₂ (Scheme 2.27). While the sterically hindered derivative yields green crystals with the monomeric structure, the substrate with the less voluminous *ortho* substituents leads to the formation of yellowish dimer, characterized with the IR absorption at 1,265 cm⁻¹ (Scheme 2.28).

Instead of the nitrosonium cation, the chlorine has been used as an oxidant for the preparation of *p*-dinitrosobenzene polymers [90].

The analogous method has been successfully used for the preparation of 1,3,5-trinitrosobenzene (Scheme 2.29) [91]. The compound has been characterized on the basis of the intensive IR band at 1,272 cm⁻¹, assigned to the (O)N=N(O) stretching vibration of the dimeric (or oligomeric) product.



Scheme 2.28

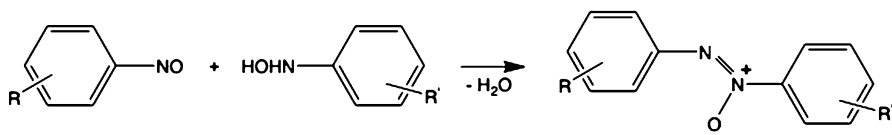


Scheme 2.29

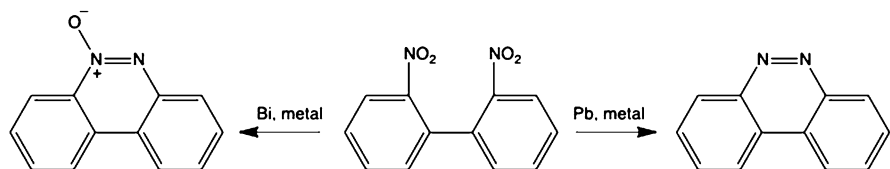
2.8 Synthesis of Azoxides and Azodioxides

Azoxides belong to the most frequent side products on the synthetic red-ox route between limiting nitro- and aminoarene functionalities. Intermediate in this spectrum of structures, hydroxylamine, easily undergoes condensation with the nitrosobenzene to form azoxybenzene derivatives (Scheme 2.30).

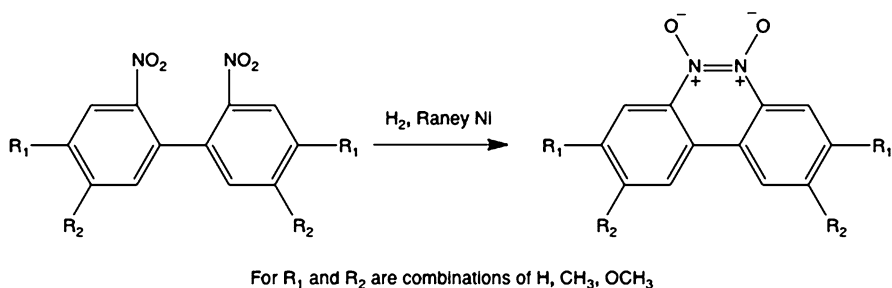
Although appearance of azoxides constrains the efficiency of the preparative methods for the nitroso compounds, their potential applications based on the property to form liquid crystals could be a good reason to review this chemistry in more details. The earliest knowledge about the formation, as well as decomposition of azoxides could be dated to the beginning of the past century. Bamberger [92] and Knipscheer [93] discovered azoxybenzene derivatives as products of heating or pyrolyzing corresponding nitrosobenzenes. The mechanism of such thermal reactions was later explained by formation of the phenyl, and the NO^\bullet radical in the first step [94].



Scheme 2.30



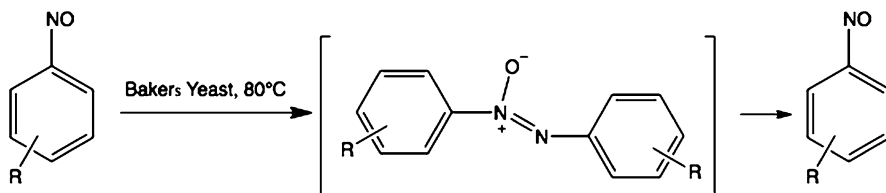
Scheme 2.31



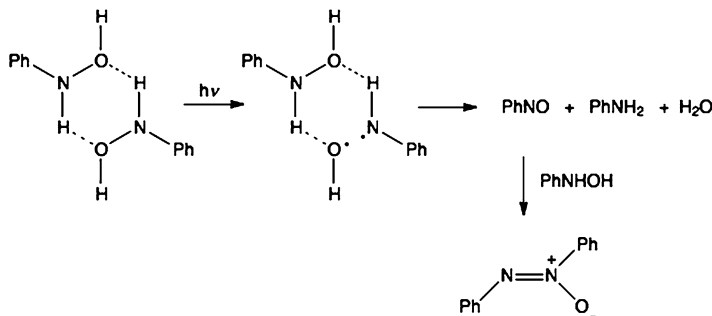
Scheme 2.32

Nitroarenes can be converted to azoxyarenes by using a series of methods based on heating of the nitro derivative with alkaline solutions such as alcoholic KOH [95], sodium alkoxide [96], phosphine [97], glucose [98], or zinc/NaOH [99]. Alternatively, such a transformation of nitro- to azoxyarenes could be obtained by standard methods of reductions with LiAlH₄, NaBH₄, or sodium arsenite [100–103]. It was found that especially efficient are reactions with metals, either as reductive reagents or as catalysts [104–106]. Solvent-free solid-state reactions of *p*-substituted nitrobenzenes with bismuth metal, yields substituted azoxybenzenes [107]. If 2,2'-dinitrobiphenyl is used as a starting compound, the product is cyclic azoxybiphenyl. However, replacing bismuth with lead affords formation of azo-derivative (Scheme 2.31).

Similar cyclizations were observed during reductions of differently substituted 2,2'-dinitrobiphenyls with hydrogen on Raney nickel [108] and with sodium bis(trimethylsilyl)amide [109]. Careful use of W-6 or W-7 Raney nickel as catalysts for hydrogenation, (Scheme 2.32) opened the opportunity to prepare also azodioxides [110].



Scheme 2.33



Scheme 2.34

Primary *p*-substituted aromatic amines can be converted to azoxy and azo derivatives by oxidation with KMnO_4 in dimethylformamide solution under ultrasound or microwave irradiation [111]. The ratio of azo versus azoxy products depends on the combination of irradiations. Similarly, ultrasound/microwave radiation could yield azoxides in chemoselective reductions of nitrobenzene derivatives [112].

Azoxybenzenes appear as intermediates in reduction of substituted nitrosobenzene by the use of Baker's Yeast [113]. A series of *p*-substituted nitrosobenzenes (Scheme 2.33) were reduced to the corresponding anilines through formation of azoxybenzene [114].

Azoxybenzenes were found as major products after photolysis of *N*-phenylhydroxylamine in acetonitrile [115]. Since the infrared spectra of the solution of *N*-phenylhydroxylamine showed bands assigned to the intermolecular hydrogen bonds between OH and NH groups [116], the reaction could begin from the hydrogen bonded dimer. The proposed mechanism (Scheme 2.34) that includes N–O cleavage is supported by findings that photolysis in crystalline state generates nitrosobenzene and aniline in almost equal amounts.

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