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CHAPTER 7

Nucleophilic Aromatic Substitution

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General

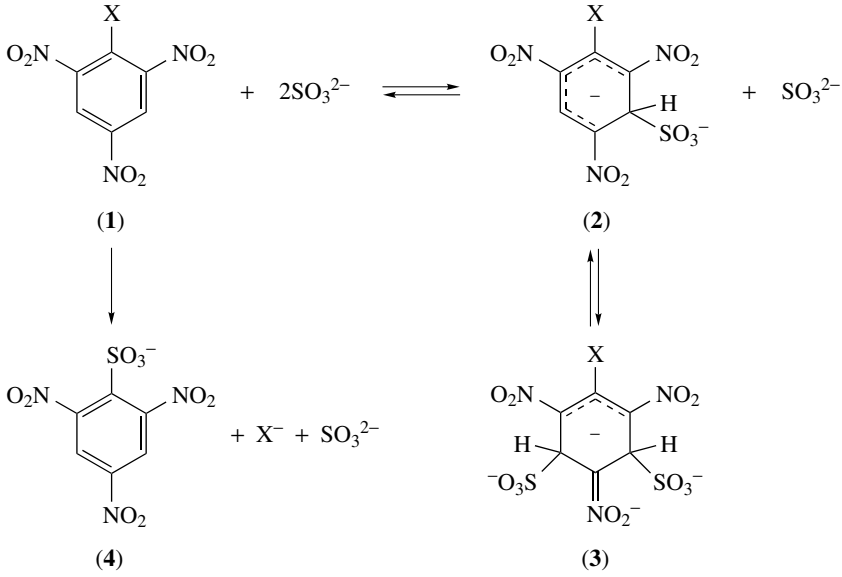
The dediazoniation of arenediazonium ions may occur by heterolytic or homolytic pathways. In a new study of the decomposition of 4-methylbenzenediazonium tetrafluoroborate in aqueous solution, the concentrations of the products, *p*-cresol with a small amount of 4-chlorotoluene, and also the unreacted diazonium salt were simultaneously monitored. The results are in accord with the heterolytic mechanism involving a highly reactive aryl cation which shows little discrimination for water or chloride ions.[1] The dediazoniation of 2,4,6-trimethylbenzenediazonium ions in concentrated aqueous solutions of acetamide, *N*-methylacetamide and *N,N*-dimethylacetamide also involves heterolysis; the aryl cation intermediate may be trapped by the oxygen or nitrogen atoms of the amides as well as by water. This approach provides a possible new method for obtaining information on the topologies and orientations of aggregate-bound polypeptides.[2]

The radical-chain $S_{RN}1$ reaction is a useful method for the formation of new carbon–carbon bonds; reactions of the enolate ions of 2-acetylthiophene and 2-acetylfuran with aryl halides have been investigated using photo-initiation and also iron(II) initiation.[3]

The S_NAr Mechanism

Sulfite is an extremely good nucleophile for activated aromatic systems and reaction with 1-substituted-2,4,6-trinitrobenzenes (**1**) may result in σ-adduct formation or in displacement of the 1-substituent as shown in Scheme 1. When X = OEt or SEt, adducts (**2**) and (**3**) formed by reaction at unsubstituted positions are long-lived.

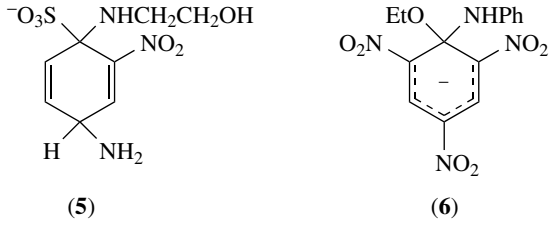
However when X = OPh or SPh, the substitution product (**4**) is eventually produced reflecting the better leaving-group ability of the phenyl compared to the alkyl derivatives.[4] The intermediate (**5**) has been postulated[5] in the catalysis by sulfite of the displacement by ammonia of the hydroxyethylamino group in 1-hydroxyethylamino-2-nitro-4-aminobenzene.



X = OEt, SEt, OPh, SPh

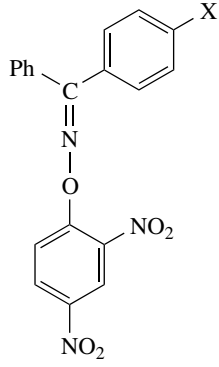
SCHEME 1

The reaction of ethyl 2,4,6-trinitrophenyl ether with aniline in dimethyl sulfoxide (DMSO) in the presence of Dabco occurs in two stages via the intermediate (**6**). Kinetic studies show that proton transfer is rate-limiting both in the formation of the intermediate and in the subsequent acid-catalysed decomposition to give 2,4,6-trinitrodiphenylamine. Phenoxide is a considerably better leaving group than ethoxide so that substitutions of phenyl 2,4,6-trinitrophenyl ethers and phenyl 2,4-dinitronaphthyl ether with aniline occur without the accumulation of intermediates. Both uncatalysed and base-catalysed pathways are involved.[6]



The $^{18}F/^{19}F$ kinetic isotope effect (KIE) has been measured in the reactions of 2,4-dinitrofluorobenzene with 2- and 4-methylaniline in DMSO. The large KIE for 2-methylaniline suggests rate-limiting expulsion of fluoride with this sterically hindered nucleophile whereas the low KIE for 4-methylaniline is consistent with rate-limiting nucleophilic attack.[7] The role of 2(1*H*)-pyridones as bifunctional catalysts has been investigated in the substitution reaction of 2-cyano-4-nitrofluorobenzene with piperidine in chloroform.[8] There has been a kinetic study of the reactions of 2-nitrofluorobenzene with aliphatic amines in DMSO and in toluene.[9]

In non-polar solvents many aminolysis reactions show a third-order dependence on the amine, B. This may be explained by catalysis of leaving-group departure by hydrogen-bonded homoconjugates, BH^+B. Evidence for this pathway has been adduced from studies of the reactions of some nitro-activated *O*-aryl oximes (**7**) with pyrrolidine in benzene, chlorobenzene, and dioxane,[10] and with piperidine and hexylamine in cyclohexane.[11] The third-order dependence on amine of the reaction of 2,6-dinitroanisole with butylamine in toluene and toluene–octanol mixtures has been interpreted in terms of a mechanism involving attack by dimers of the nucleophile.[12]



(**7**)

The reaction of aliphatic amines with *sym*-trichlorotrinitrobenzene results in substitution of chlorine and, in the case of ammonia and monoalkylamines, the formation of di- and tri-substituted products is facilitated by monosubstitution. This has been attributed to hydrogen bonding between an amino hydrogen and an *ortho*-nitro group which helps to relieve steric strain so that nitro groups may more nearly achieve coplanarity with the aromatic ring.[13] Nitranions, formed from aryl and heteroaryl amines and sodium hydride, may replace chlorine in 4-chloronitrobenzene to yield diarylamines; reactivity is higher in DMSO than in toluene owing to ion-pair formation in the latter solvent.[14] Microwave irradiation has been found to speed reaction, compared with conventional reflux, in the substitution of halogens by piperidine or potassium *t*-butoxide in DMSO or in dimethylformamide (DMF).[15]

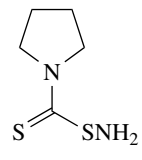
A spectrochemical study has been reported[16] of the reactions in dimethylacetamide of benzyl thiolate, RS^-, and disulfide, RS_2^-, ions with nitrobenzenes, $XC_6H_4NO_2$,

activated by a second electron-withdrawing group. Nitro group displacement is observed yielding unsymmetrical monosulfides, XC_6H_4SR, or (poly)sulfide anions, $XC_6H_4S_y^-$ ($y = 1, 2$). Reaction of 4-bromophenoxide ions with 3-substituted or 3,5-disubstituted nitrobenzenes in DMF may also result in displacement of the nitro group, and a correlation has been established between reactivity and σ_M substituent constants.[17] It is reported that reaction in DMSO or DMF of 1-nitro-4-chloroanthraquinone with thiophenolate ions results in nitro group displacement; however, with phenolate ions chlorine is preferentially substituted.[18] A kinetic study of the formation of 1,2,3,4-tetrachlorodibenzo-*p*-dioxin in DMSO indicates a rate-limiting step involving nucleophilic attack by a pyrocatechol anion on hexachlorobenzene.[19]

An unusually facile hydroxydemethoxylation reaction has been reported for 1-benzoyl-4-methoxynaphthalene. The use of ^{18}O-enriched hydroxide confirmed that reaction occurs by the S_NAr pathway rather than by cleavage of the oxygen to methyl bond. A related reaction is observed in 3-(4-methoxy-1-naphthoyl)indole derivatives. These results indicate the possibility of S_NAr activation by a 4-carbonyl group; the reaction is facilitated by use of a naphthyl ketone as substrate and DMSO as solvent.[20]

The vicarious nucleophilic substitution (VNS) of hydrogen is an important method of replacement of ring hydrogen atoms. It is termed vicarious since it involves departure of an anion from the incoming nucleophile rather than hydride ion from the aromatic ring. A short review[21] has summarized studies of the mechanism and the orientation of substitution in VNS reactions. The synthesis of a hexasubstituted benzene has been reported by VNS reaction of 2-chloro-3,5-dinitro-4-methylbenzene with the anion of chloromethyl *p*-tolyl sulfone.[22] VNS reaction of nitroarenes with the anion of 1-cyano-2,2-diethoxycarbonyl cyclopropane proceeds by σ-adduct formation and base-induced elimination which results in cleavage of the cyclopropane ring; reaction *ortho* to the nitro group may eventually yield substituted *N*-hydroxyindoles.[23]

Full reports have appeared, following preliminary communications, of both the amination and hydroxylation of nitroarenes. Amination may be achieved by use of sulfenamides as nucleophiles. The mechanism is analogous to VNS with thiolates as leaving groups. Use of the sulfenamide (**8**) results in amination *para* to the nitro group, while (**9**) and (**10**) allow reaction at the *ortho* position.[24] The amination of dinitroarenes by this procedure is not satisfactory and S_NAr substitution of nitro groups may occur. Hydroxylation of nitroarenes may be observed following reaction with the anions of *t*-butyl or cumyl hydroperoxides;[25] again VNS is involved with base-induced elimination of the alcohol occurring by an *E*2-type mechanism.



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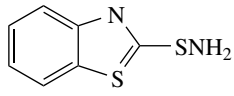

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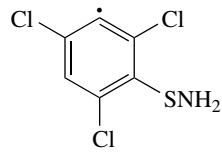
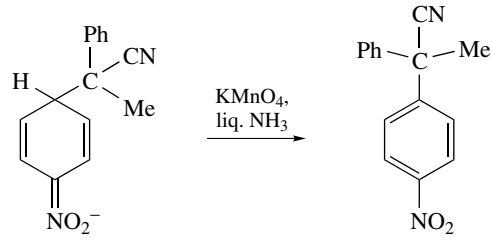
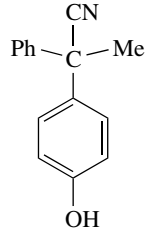


(**10**)

There have been several studies of the oxidative substitution of hydrogen by carbanions. Reaction of the carbanion of 2-phenylpropionitrile with nitroarenes in liquid ammonia yields σ-adducts (**11**), which may be oxidized with potassium permanganate to yield the neutral products (**12**). Addition of methyl iodide to a 1:1 carbanion–nitroarene mixture indicates the presence of little free carbanion, showing that formation of the initial σ-adduct goes almost to completion.[26] The value of 9.8 found for the isotope effect k_H/k_D measured by comparison of the rates of reaction of nitrobenzene and [4 − ^{2}H]nitrobenzene confirms that carbon–hydrogen bond breaking is rate limiting in the oxidation process.[27] Studies with related phenylacetonitrile derivatives PhCHRCN [R = Et, $n - C_5H_{11}$, Bn, MeO, PhO, NMe_2, Ph, CH(Me)Ph and $CHPh_2$] show that σ-adduct formation at the 4-position of nitroarenes is the norm; when suitable leaving groups, R = MeO, PhO, are present in the carbanion then VNS may compete with oxidation of the adducts.[28] Interestingly, in the reaction of 2-phenylpropionitrile with nitroarenes the use of dimethyldioxirane as the oxidant rather than permanganate results in nitro group displacement to yield substituted phenols (**13**); it is likely that a dienone intermediate is involved.[29] Kinetic studies of substituent effects on the reaction in alcohols as solvents of substituted nitrobenzenes with the anion of phenylacetonitrile show that initial complexation may be rate limiting; the eventual products are 2,1-benzisoxazoles.[30] Mass spectrometry has been used to identify 3-methylbenz-1,2-isoxazole as a decomposition product formed from substituted 1-phenylethanone oximes on electron ionization. A mechanism was proposed involving intramolecular attack of the oxime hydroxyl group followed by a 1,2-elimination of the *ortho*-substituent and the hydrogen of the hydroxyl group.[31]



(**11**) (**12**)



(**13**)

The reaction of the carbanion derived from diethyl methylphosphonate with perhalogenated aromatics may result in substitution of halide to yield perhaloaryl(hetaryl)methylphosphonates, which can be converted into tris- or bis-(perhaloaryl)methanes.[32] Displacement of fluoride ion has been reported in the reaction of dimethoxycarbene with 1-fluoro-2,4-dinitrobenzene and with hexafluorobenzene.[33] The hydrodehalogenation of halogenated aryl ketones may be facilitated using hydrogen over a Pt/C catalyst.[34]

There has been a useful review of phase-transfer catalysis in nucleophilic aromatic substitution.[35] A comparison has been reported of the reactions with nucleophiles of 1-chloro-2,4-dinitrobenzene (substitution) and 4-nitrophenyl diphenyl phosphate (dephosphorylation) in neutral micelles of dodecyl (10) and (23) polyoxyethylene glycol. In the substitution reaction considerable amounts of ether may be formed by reaction with alkoxide ions at the micellar surface. Differences in reactivity of the two substrates are probably due to differences in their location in the micellar structures.[36]

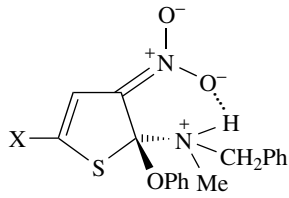
It has been shown that when nucleophilic aromatic photo-substitution reactions are carried out in non-deoxygenated solutions of aprotic solvents, such as DMSO and acetonitrile, destructive superoxide anions may be formed from aromatic radical anions. Such solvents are best avoided.[37] There has been a review of mechanistic aspects of photo-substitutions of the cyano group in aromatic compounds.[38]

A kinetic study has been reported[39] of the nucleophilic exchange reaction of the radionuclide ^{131}I with 15-(4-iodophenyl)pentadecanoic acid in the presence of Cu(I). The activation of arenes to nucleophilic attack by complexation with transition metal ligands is well known. It is reported that potassium hydride will react with $Cr(CO)_3$–aryltrimethylsilanes to generate aryl anions which can further react with electrophiles.[40] Several new rhodium(III) complexes have been synthesized and shown to be efficient catalysts for methoxydefluorination reactions of fluoroarenes.[41] The transition metal-catalysed reactions of nitrogen, oxygen, sulfur, and phosphorus nucleophiles with aromatic and with heteroaromatic compounds have been reviewed; palladium or nickel complexes containing phosphine ligands are often used and inter- and intra-molecular substitutions may occur.[42] It has been reported that arene–chromium complexes may be used effectively as supporting ligands in the palladium-catalysed amination reactions of both electron-rich and electron-deficient aryl bromides with secondary amines.[43] A new co-catalysed cyanation reaction involving Pd(0) catalysis has been reported, providing an efficient synthetic route to aryl nitriles. Thus the CuI–$Pd(PPh_3)_4$ system allows conversion of aryl halides and triflates into the corresponding nitrile derivatives using sodium or potassium cyanide.[44]

Heterocyclic Systems

A kinetic study has been reported of substituent effects on the reactions of 2-phenoxy- and 2-(4-nitrophenoxy)-3-nitro-5-X-thiophenes with benzylamine and with *N*-methylbenzylamine in benzene as solvent. The intramolecularly hydrogen-bonded intermediate (**14**) is postulated. Reactions of the 5-unsubstituted thiophenes (X = H) are not base-catalysed, indicating that nucleophilic attack is rate limiting, and the more basic secondary amine shows higher reactivity than the primary

amine. The introduction of electron-withdrawing groups at the 5-position results in a weakening of the hyper-*ortho* interaction between the reaction centre and the 3-nitro group, so that the strength of the intramolecular hydrogen bond is reduced. A consequence of this is that reversal of the intermediate to reactants is facilitated and with *N*-methylbenzylamine base catalysis, probably by the SB–GA mechanism, is observed.[45]



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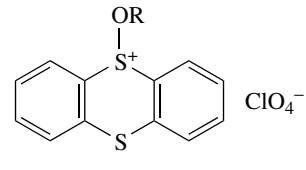
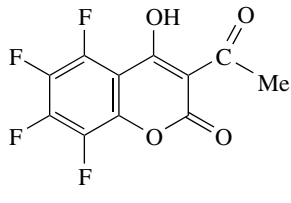


(**15**)

Some 5-(alkyloxy)thianthrenium perchlorates (**15**) have been prepared in which the alkyl group may be primary or secondary. Reaction with iodide ions may result in S_N2 reaction at the alkyl group or S_NAr reaction at the sulfonium sulfur atom leading to the formation of thianthrene.[46]

2,4,6-Tribromo-3,5-difluoropyridine may be prepared by reaction of pentafluoropyridine with a mixture of hydrogen bromide and aluminium tribromide. Surprisingly, reaction with hard nucleophiles, such as methoxide in methanol or aqueous ammonia, resulted in exclusive displacement of fluoride whereas reaction with softer nucleophiles, such as sodium thiophenolate, gave exclusive displacement of bromide.[47] Fluoride displacement at the 7-position is observed in the reaction of 3-acetyl-5,6,7,8-tetrafluoro-4-hydroxycoumarin (**16**) with ammonia or morpholine in DMSO; however, reaction with ammonia in water, alcohol, or glyme results in attack at the acetyl group to yield the corresponding 3-(1-iminoethyl) derivative.[48] It is reported[49] that the rate of nucleophilic displacement of halogens at the 7-position in halogenated derivatives of *N*-substituted 4-oxo-1,4-dihydroquinoline-3-carboxylic acid may be enhanced by the formation of boron chelates such as (**17**). Acceleration of the alkaline



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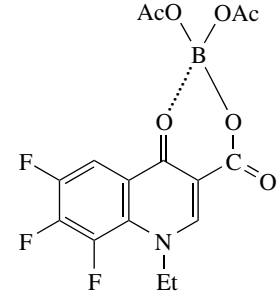
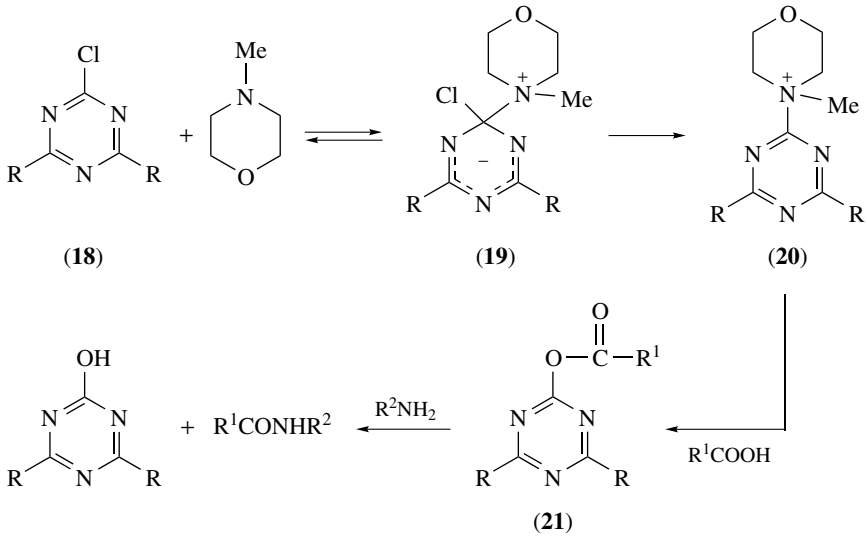


(**17**)

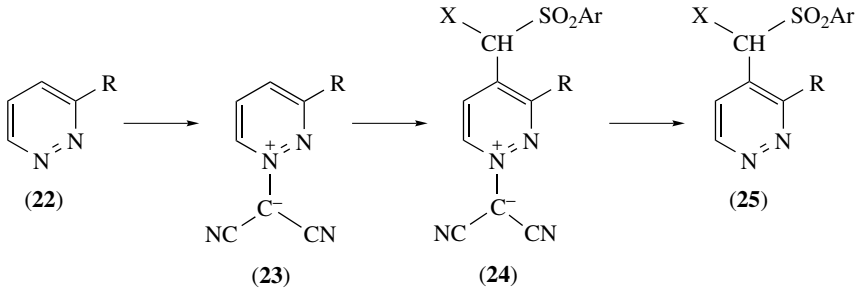
hydrolysis of 2-phenoxyquinoxaline has been found in micelles of cetyltrialkylammonium hydroxides and in related cationic micelles; increases in rate constants with increasing size of the head group were attributed to exclusion of water from the micellar surface.[50]

There has been a study of the mechanism of the activation of carboxylic acids to peptide formation by chloro-*s*-triazines in combination with tertiary amines. The first step, exemplified in Scheme 2 by the reaction of 2-chloro-4,6-disubstituted-1,3,5-triazines (**18**) with *N*-methylmorpholine, is formation of a quaternary triazinylammonium salt (**20**). Here there is ^{1}H NMR evidence for the formation at $-50\,^{\circ}$C of the intermediate (**19**), showing that the substitution involves the two-step S_NAr mechanism rather than a synchronous pathway. The subsequent reaction of (**20**) with a carboxylic acid yields the 2-acyloxy derivative (**21**), which carries an excellent leaving group for the amide-forming step.[51]

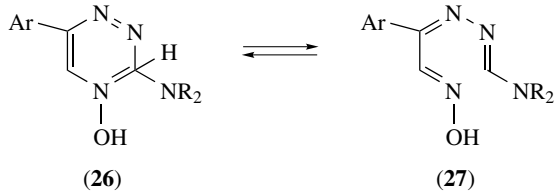


SCHEME 2

The nucleophilic substitution of hydrogen in pyridazines (**22**) may be achieved by vicarious substitution in the dicyanomethylide derivatives.[52] As outlined in Scheme 3, treatment with tetracyanoethylene oxide gives the *N*-dicyanomethylide (**23**) which will react under VNS conditions to give (**24**); the dicyanomethylene group may be eliminated by a radical pathway to yield the 4-substituted pyridazine (**25**). The replacement of ring hydrogen and of good leaving groups has been compared in a review of the reactions of 1,2,4-triazines with carbon nucleophiles including cyanide ions.[53] The reaction of 6-aryl-1,2,4-triazine-4-oxides with secondary amines in alcohol yields σ-adducts (**26**), which form isolable ring-opened intermediates (**27**); oxidative aromatisation with permanganate allows the isolation of 1,2,4-triazines substituted in the 3-position.[54] The reaction of 3-amino- or 3-methylthio-1,2,4-triazine with electron-rich



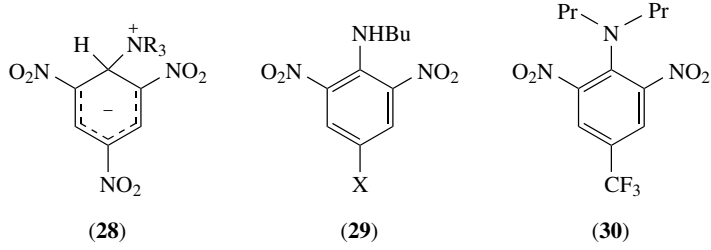
SCHEME 3



arenes, such as 2,6-dimethylphenol, resorcinol, or indole, may result in the formation of 4,5-dihydro derivatives.[55]

Meisenheimer and Related Adducts

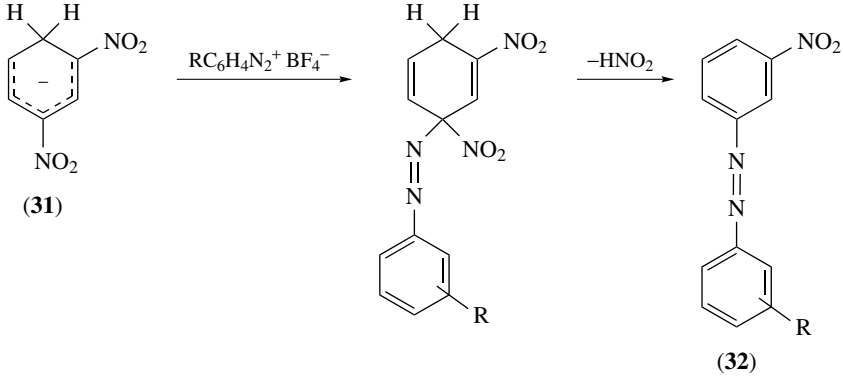
A kinetic study of the formation of zwitterionic adducts (**28**) from 1,3,5-trinitrobenzene and diazabicyclo derivatives indicates that reactions are surprisingly slow, with rate constants many orders of magnitude lower than those for related reactions with primary or secondary amines.[56] The use of rapid-scan spectrophotometry was necessary to study the kinetics of reaction of 4-substituted-2,6-dinitro-*N*-*n*-butylanilines (**29**) with *n*-butylamine in DMSO; the two processes observed were identified as rapid deprotonation to give the conjugate base and competitive σ-adduct formation at the 3-position.[57] The reactions of *N,N*-di-*n*-propyl-2,6-dinitro-4-trifluoromethylaniline (**30**), the herbicide trifluralin, and its *N*-ethyl-*N*-*n*-butyl analogue with deuteroxide ions and with sulfite ions in [$^2H_6$]DMSO–D_2O have been investigated by ^{1}H NMR spectroscopy. With deuteroxide σ-adduct formation at the 3-position is followed by



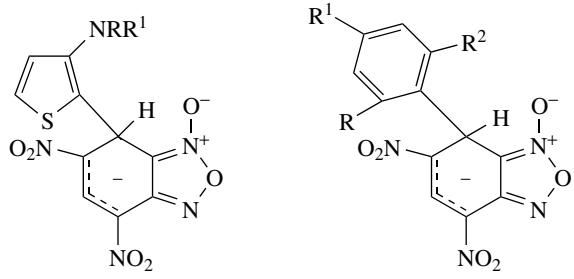
nucleophilic displacement of the *N,N*-dialkyl substituents, while in the case of sulfite attack at the 3-position is followed by the formation of isomeric *cis*- and *trans*-diadducts resulting from addition at the 3- and 1-positions. The reactions with deuteroxide are accompanied by slow aryl H–D exchange.[58] Note that σ-adducts have also been observed during the reactions with sulfite of 1-substituted-2,4,6-trinitrobenzenes.[4]

Hydride adducts (**31**) may be formed by the reaction of 1,3-dinitrobenzene with potassium borohydride; their treatment[59] with phenyldiazonium salts leads to nitro group displacement yielding azo-coupled products (**32**).

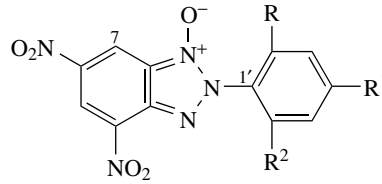
The super-electrophile 4,6-dinitrobenzofuroxan (DNBF) has been used to probe the reactivity of 3-aminothiophenes; their ready formation of adducts (**33**) by reaction at the 2-position indicates their strongly enaminic nature.[60] Mixing DNBF and hydroxy- and methoxy-substituted benzenes in DMSO yields adducts (**34**; R, R^1, R^2 = OH, OMe). In the reaction with 1,3,5-trimethoxybenzene an isotope effect, k_H/k_D, of 3.71 is observed, indicating that in the reaction, viewed as an S_EAr substitution, proton transfer of ring hydrogen is partially rate limiting.[61] The highly electrophilic benzotriazole derivative (**35**), in which R, R^1 and R^2 may include one, two, or three nitro groups, has been used to monitor reactivities of sterically hindered phenoxide ions. NMR studies show that for both 2,6-di-*t*-butylphenoxide, acting as a carbon nucleophile, and 3,5-di-*t*-butylphenoxide, acting as an oxygen nucleophile, the propensity



(**31**) (**32**)



(**33**) (**34**)



(**35**)

for reaction at the 1′-position of (**35**) relative to the 7-position increases with nitro substitution.[62]

Benzyne and Related Intermediates

It has been shown that, in the presence of lithium diethylamide at −70 °C, bromobenzoic acids form arynes which may react with arylacetonitriles to yield, predominantly, 2-cyanobenzoic acids.[63] The reaction of alkyl and aryl isocyanides with benzyne may yield benzamide derivatives, showing their ability to act as charge-reversed equivalents to isocyanates.[64] The generation and cyclization of a benzyne-tethered alkyllithium have been reported, and lead to a convenient synthetic route for 4-substituted indans.[65]

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