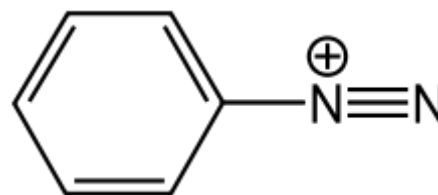


Diazonium compound

Diazonium compounds or **diazonium salts** are a group of organic compounds sharing a common functional group $\text{R-N}_2^+\text{X}^-$ where R can be any organic group, such as an alkyl or an aryl, and X is an inorganic or organic anion, such as a halogen.



Benzenediazonium cation

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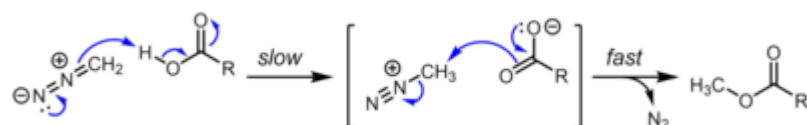
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General properties and reactivity

According to tabulated linear free energy relationship constants (e.g. Hammett σ_m and σ_p), the diazonium group (N_2^+) is among the most strongly electron-withdrawing substituents. Thus, the α position of alkyldiazonium species and acidic protons on diazonio-substituted phenols and benzoic acids have greatly reduced pK_a values compared to their unsubstituted counterparts. For example, the aqueous pK_a of methyl diazonium is estimated to be <10 ,^[1] while that of the phenolic proton of 4-hydroxybenzenediazonium was measured to be 3.4.^[2]

In terms of reactivity, the chemistry of diazonium salts is dominated by their propensity to dediazotize via the thermodynamically (enthalpically and entropically) favorable expulsion of dinitrogen gas. The reaction ($MeN_2^+ \rightarrow Me^+ + N_2$) has an enthalpic change of 43 kcal/mol, while ($EtN_2^+ \rightarrow Et^+ + N_2$) has an enthalpic change of 11 kcal/mol.^[3] For secondary and tertiary alkyldiazonium species, the enthalpic change is calculated to be close to zero or negative, with minimal activation barrier to expulsion of nitrogen. Hence, secondary and (especially) tertiary alkyldiazonium species are either unbound, nonexistent species or, at best, extremely fleeting intermediates.



Methyldiazonium carboxylate is believed to be a fleeting intermediate in the methylation of carboxylic acids by diazomethane, and alkyldiazonium species generated via diazotization of amines have been studied in physical organic chemistry studies.^{[4][5]} However, alkyldiazonium salts are otherwise synthetically unimportant due to their extreme and uncontrolled $S_N2/S_N1/E1$ reactivity.

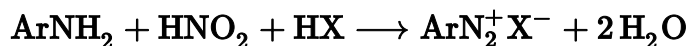
In contrast, aryldiazonium salts are more stable, though still dangerously explosive under certain conditions, because S_N2 is geometrically impossible and does not occur, while S_N1 occurs with much greater difficulty compared to alkyldiazonium species due to difficulty in forming the high energy aryl cation and stronger $C(sp^2)-N$ bond compared to the $C(sp^3)-N$ bond in alkyldiazonium compounds. Classically, the chloride salt is prepared at 5 °C from the aniline and $NaNO_2/aq. HCl$, and $ArN_2^+Cl^-$ salts tend to decompose (sometimes explosively) at higher temperatures. However, the use of redox inactive and voluminous HSO_4^- , BF_4^- , or TsO^- as counteranions has allowed aryldiazonium salts to be stored indefinitely at 0 °C and safely handled for short periods of time at temperatures up to 50 °C. Aryldiazonium salts undergo $S_N1(Ar)$ (dissociative unimolecular nucleophilic aromatic substitution) in a few cases, while the majority of their reactions take place by $S_{RN}1(Ar)$ (dissociative radical nucleophilic aromatic substitution) in which an initial electron transfer takes place, allowing for the formation of an aryl radical rather than cation.

Aryldiazonium salts are exceptionally versatile reagents for chemical synthesis. Because aryl radicals and cations are highly electrophilic and will react with virtually any nucleophile reagent, aryldiazonium salts form a "transit hub" for arene chemistry from which almost any other aromatic derivative can be prepared. Moreover, this utility is accentuated by their ready availability: aryldiazonium salts are easily accessible from diazotization of arylamines (anilines and heteroarylamines), which, in turn, are derived from the parent arene by electrophilic nitration followed by metal-mediated or -catalyzed reduction.^[6] After electrophilic aromatic substitution, diazonium chemistry is the most frequently applied strategy to prepare aromatic compounds.

Industrially, aryldiazonium salts are important intermediates in the organic synthesis of azo dyes.^[7]

Preparation

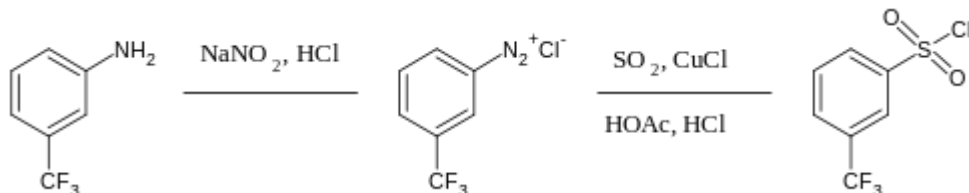
The process of forming diazonium compounds is called "diazotation", "diazonation", or "diazotization". The reaction was first reported by Peter Griess in 1858, who subsequently discovered several reactions of this new class of compounds. Most commonly, diazonium salts are prepared by treatment of aromatic amines with nitrous acid and additional acid. Usually the nitrous acid is generated *in situ* (in the same flask) from sodium nitrite and the excess mineral acid (usually aqueous HCl, H₂SO₄, *p*-H₃CC₆H₄SO₃H, or HBF₄):



Sample of phenyldiazonium tetrafluoroborate.

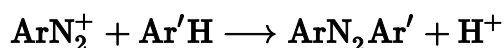
Aqueous solutions of diazonium chloride salts, traditionally prepared from the aniline, sodium nitrite, and hydrochloric acid, are unstable at room temperature and are classically prepared at 0 – 5 °C. However, one can isolate diazonium compounds as tetrafluoroborate or tosylate salts,^[8] which are stable solids at room temperature. It is often preferred that the diazonium salt remain in solution, but they do tend to supersaturate. Operators have been injured or even killed by an unexpected crystallization of the salt followed by its detonation.^[9]

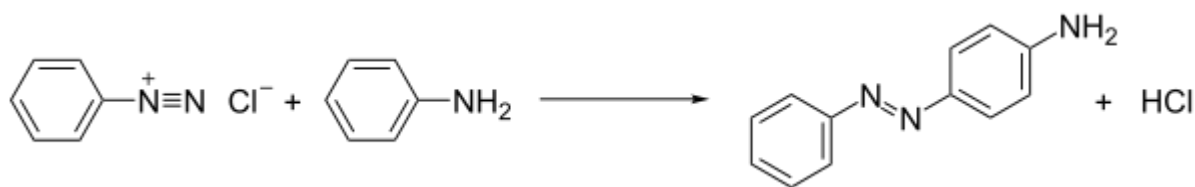
Due to these hazards, diazonium compounds are usually not isolated. Instead they are used *in situ*. This approach is illustrated in the preparation of an arylsulfonyl compound:^[10]



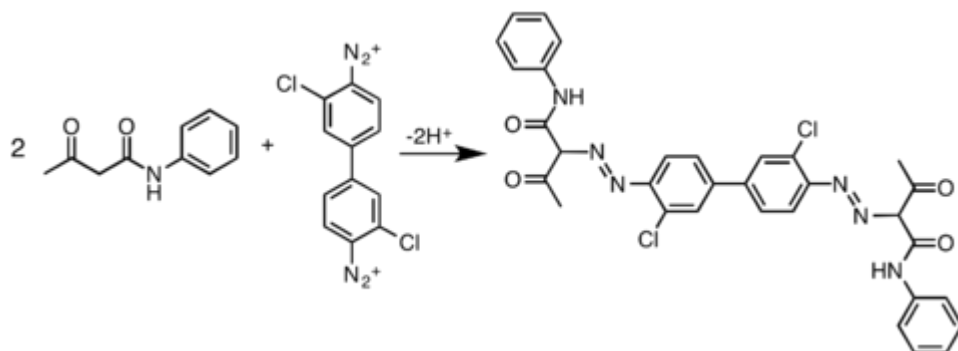
Diazo coupling reactions

The most widely practiced reaction of diazonium salts is azo coupling. In this process, the diazonium compound is attacked by, i.e., coupled to, electron-rich substrates. When the coupling partners are arenes such as anilines and phenols, the process is an example of electrophilic aromatic substitution:





Another commercially important class of coupling partners are acetoacetic amides, as illustrated by the preparation of Pigment Yellow 12, a diarylide pigment.^[11]



The resulting azo compounds are often useful dyes and in fact are called azo dyes.^[12] The deep colors of the dyes reflects their extended conjugation. For example, the dye called aniline yellow is produced by mixing aniline and cold solution of diazonium salt and then shaking it vigorously. Aniline yellow is obtained as a yellow solid.^[13] Similarly, a cold basic solution of Naphthalen-2-ol (beta-naphthol) give the intensely orange-red precipitate.^[13] Methyl orange is an example of an azo dye that is used in the laboratory as a pH indicator.

Displacement of the N_2 group

Arenediazonium cations undergo several reactions in which the N_2 group is replaced by another group or ion. Some of the major ones are the following.^{[14][15]}

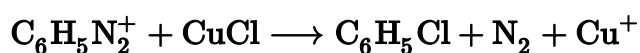
Biaryl coupling

A pair of diazonium cations can be coupled to give biaryls. This conversion is illustrated by the coupling of the diazonium salt derived from anthranilic acid to give diphenic acid ($(\text{C}_6\text{H}_4\text{CO}_2\text{H})_2$).^[16] In a related reaction, the same diazonium salt undergoes loss of N_2 and CO_2 to give benzynes.^[17]

Replacement by Halides

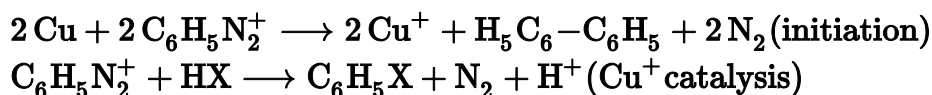
Sandmeyer reaction

Benzenediazonium chloride heated with cuprous chloride or cuprous bromide respectively dissolved in HCl or HBr yield chlorobenzene or bromobenzene, respectively.



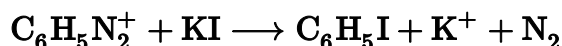
Gatterman reaction

In the Gatterman reaction, benzenediazonium chloride is warmed with copper powder and HCl or HBr to produce chlorobenzene and bromobenzene respectively. It is named after the German chemist Ludwig Gattermann.^[18]



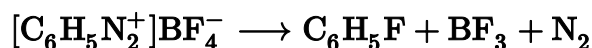
Replacement by iodide

Iodine is not easily introduced into the benzene ring directly. However it can be introduced by treating aryldiazonium cations with potassium iodide:



Replacement by fluoride

Fluorobenzene is produced by thermal decomposition of benzenediazonium fluoroborate. The conversion is called the Balz-Schiemann reaction.^[19]

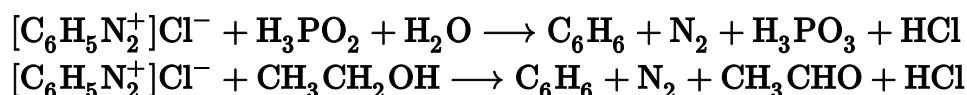


The traditional Balz–Schiemann reaction has been the subject of many motivations, e.g. using hexafluorophosphates (PF_6^-) and hexafluoroantimonate (SbF_6^-) in place of tetrafluoroborates. The diazotization can be effected with nitrosonium salts such as $[\text{NO}]\text{SbF}_6$.^[20]

Miscellaneous Replacements

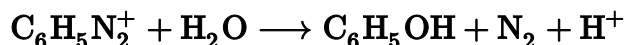
Replacement by hydrogen

Arenediazonium cations reduced by hypophosphorous acid, ethanol or sodium stannite gives benzene:



Replacement by a hydroxyl group

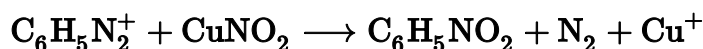
Phenols are produced by heating aqueous solutions of aryldiazonium salts:^{[21][22][23][24]}



This reaction goes by the German name *Phenolverkochung* ("cooking down to yield phenols"). The phenol formed may react with the diazonium salt and hence the reaction is carried in the presence of an acid which suppresses this further reaction.^[25] A Sandmeyer-type hydroxylation is also possible using Cu_2O and Cu^{2+} in water.

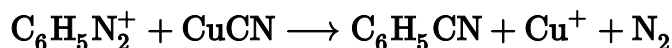
Replacement by a nitro group

Nitrobenzene can be obtained by treating benzenediazonium fluoroborate with sodium nitrite in presence of copper. Alternatively, the diazotisation of the aniline can be conducted in presence of cuprous oxide, which generates cuprous nitrite in situ:



Replacement by a cyano group

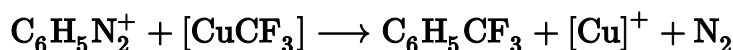
The cyano group usually cannot be introduced by nucleophilic substitution of haloarenes, but such compounds can be easily prepared from diazonium salts. Illustrative is the preparation of benzonitrile using the reagent cuprous cyanide:



This reaction is a special type of Sandmeyer reaction.

Replacement by a trifluoromethyl group

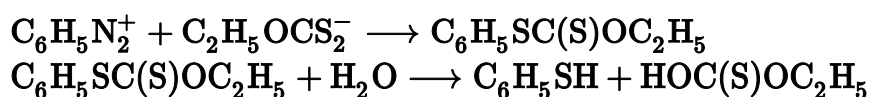
Two research groups reported trifluoromethylations of diazonium salts in 2013. Goossen reported the preparation of a CuCF_3 complex from CuSCN , TMSCF_3 , and Cs_2CO_3 . In contrast, Fu reported the trifluoromethylation using Umemoto's reagent (*S*-trifluoromethyldibenzothiophenium tetrafluoroborate) and Cu powder (Gattermann-type conditions). They can be described by the following equation:



The bracket indicates that other ligands on copper are likely present but are omitted.

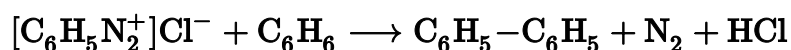
Replacement by a thiol group

Diazonium salts can be converted to thiols in a two-step procedure. Treatment of benzenediazonium chloride with potassium ethylxanthate followed by hydrolysis of the intermediate xanthate ester gives thiophenol:



Replacement by an aryl group

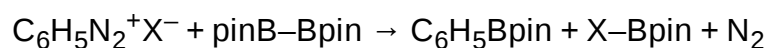
The aryl group can be coupled to another using aryldiazonium salts. For example, treatment of benzenediazonium chloride with benzene (an aromatic compound) in the presence of sodium hydroxide gives diphenyl:



This reaction is known as the Gomberg–Bachmann reaction. A similar conversion is also achieved by treating benzenediazonium chloride with ethanol and copper powder.

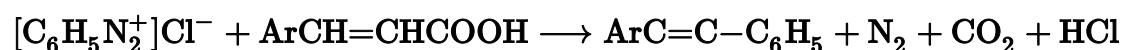
Replacement by boronate ester group

A Bpin (pinacolatoboron) group, of use in Suzuki-Miyaura cross coupling reactions, can be installed by reaction of a diazonium salt with bis(pinacolato)diboron in the presence of benzoyl peroxide (2 mol %) as an initiator:^[26]



Meerwein reaction

Benzenediazonium chloride reacts with compounds containing activated double bonds to produce phenylated products. The reaction is called the Meerwein arylation:



Metal complexes

In their reactions with metal complexes, diazonium cations behave similarly to NO^+ . For example, low-valent metal complexes add with diazonium salts. Illustrative complexes are $[\text{Fe}(\text{CO})_2(\text{PPh}_3)_2(\text{N}_2\text{Ph})]^+$ and the chiral-at-metal complex $\text{Fe}(\text{CO})(\text{NO})(\text{PPh}_3)(\text{N}_2\text{Ph})$.^[27]

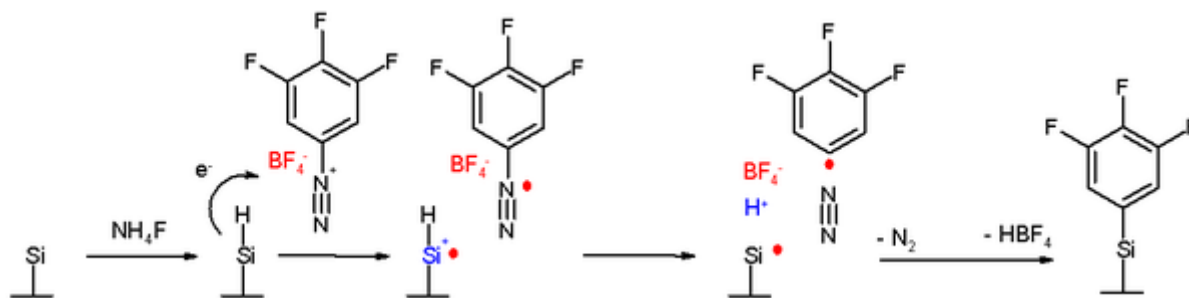
Other methods for dediazotization

- by organic reduction at an electrode
- by mild reducing agents such as ascorbic acid (vitamin C)^[28]
- by gamma radiation from solvated electrons generated in water
- photoinduced electron transfer
- reduction by metal cations, most commonly a cuprous salt.
- anion-induced dediazotiation: a counterion such as iodine gives electron transfer to the diazonium cation forming the aryl radical and an iodine radical
- solvent-induced dediazotiation with solvent serving as electron donor

Grafting reactions

In a potential application in nanotechnology, the diazonium salts 4-chlorobenzenediazonium tetrafluoroborate very efficiently functionalizes single wall nanotubes.^[29] In order to exfoliate the nanotubes, they are mixed with an ionic liquid in a mortar and pestle. The diazonium salt is added together with potassium carbonate, and after grinding the mixture at room temperature the surface of the nanotubes are covered with chlorophenyl groups with an efficiency of 1 in 44 carbon atoms. These added substituents prevent the tubes from forming intimate bundles due to large cohesive forces between them, which is a recurring problem in nanotube technology.

It is also possible to functionalize silicon wafers with diazonium salts forming an aryl monolayer. In one study, the silicon surface is washed with ammonium hydrogen fluoride leaving it covered with silicon-hydrogen bonds (hydride passivation).^[30] The reaction of the surface with a solution of diazonium salt in acetonitrile for 2 hours in the dark is a spontaneous process through a free radical mechanism.^[31]



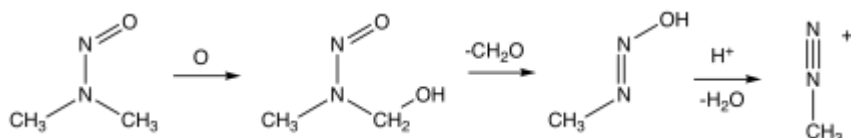
So far grafting of diazonium salts on metals has been accomplished on iron, cobalt, nickel, platinum, palladium, zinc, copper and gold surfaces.^[32] Also grafting to diamond surfaces has been reported.^[33] One interesting question raised is the actual positioning on the aryl group on the surface. An in silico study^[34] demonstrates that in the period 4 elements from titanium to copper the binding energy decreases from left to right because the number of d-electrons increases. The metals to the left of iron are positioned tilted towards or flat on the surface favoring metal to carbon pi bond formation and those on the right of iron are positioned in an upright position, favoring metal to carbon sigma bond formation. This also explains why diazonium salt grafting thus far has been possible with those metals to right of iron in the periodic table.

Reduction to a hydrazine group

Diazonium salts can be reduced with stannous chloride (SnCl_2) to the corresponding hydrazine derivatives. This reaction is particularly useful in the Fischer indole synthesis of triptan compounds and indometacin. The use of sodium dithionite is an improvement over stannous chloride since it is a cheaper reducing agent with fewer environmental problems.

Biochemistry

Alkyldiazonium ions, otherwise rarely encountered in organic chemistry, are implicated as the causative agents in the carcinogens. Specifically, nitrosamines are thought to undergo metabolic activation to produce alkyldiazonium species.



Metabolic activation of the nitrosamine NDMA, involving its conversion to an alkylating agent.^[35]

Applications

The first use of diazonium salts was to produce water-fast dyed fabrics by immersing the fabric in an aqueous solution of the diazonium compound, followed by immersion in a solution of the coupler (the electron-rich ring that undergoes electrophilic substitution). The major applications of diazonium compounds remains in the dye and pigment industry.^[12]

Other uses

Diazonium compounds are standard reagents used in synthesis of organic compounds, especially aryl derivatives.

Diazonium salts are light sensitive and break down under near UV or violet light. This property has led to their use in document reproduction. In this process, paper or film is coated with a diazonium salt. After contact exposure under light, the residual diazo is converted to a stable azo dye with an aqueous solution of coupler. A more common process uses a paper coated with diazo, coupler and an acid to inhibit coupling; after exposure the image is developed by a vapor mixture of ammonia and water which forces coupling.

Safety

Solid diazonium halides are often dangerously explosive, and fatalities and injuries have been reported.^[9]

The nature of the anions affects stability of the salt. Aryl diazonium perchlorates, such as nitrobenzenediazonium perchlorate, have been used to initiate explosives.

See also

- Diazo
- Diazo printing process
- Benzenediazonium chloride
- Triazene cleavage
- Dinitrogen complex

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31. Reaction sequence: silicon surface reaction with ammonium hydrogen fluoride creates hydride layer. An electron is transferred from the silicon surface to the diazonium salt in an open circuit potential reduction leaving a silicon radical cation and a diazonium radical. In the next step a proton and a nitrogen molecule are expelled and the two radical residues recombine creating a surface silicon to carbon bond.
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