



## Review

## Pentafluorobenzaldehyde and its utilizing in organic synthesis

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 Cyclization reactions

## ABSTRACT

Over the past 50 years, pentafluorobenzaldehyde has received much attention due to its unique chemical and physical properties as well as its real or potential applications in chemistry of porphyrines, additives, drug delivery and in analytical chemistry. Pentafluorobenzaldehyde is multifunctional aromatic compound containing five atoms of fluorine and one aldehydic function. That is why it can provide nucleophilic substitutions of all or some atoms of fluorine, nucleophilic additions of aldehydic function, "haloform reaction" and some cyclization reactions. This year, it is being the 50th year, since the first synthesis of this compound was published.

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## 1. Introduction

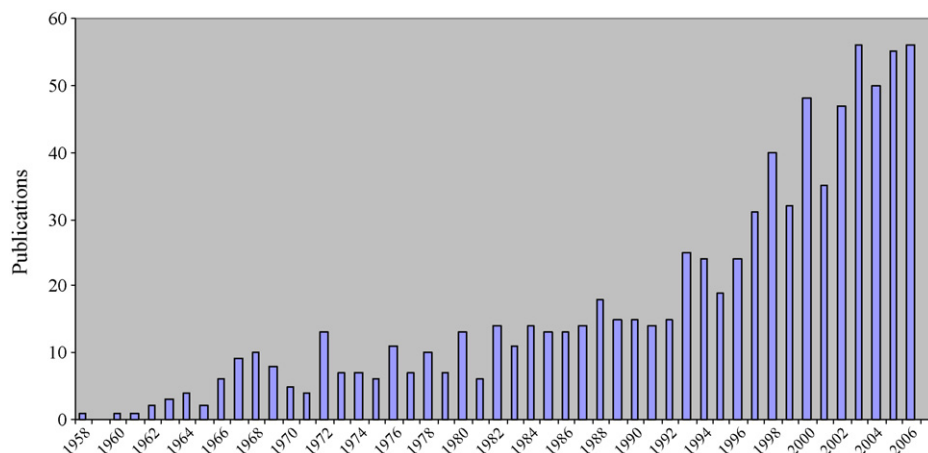
Pentafluorobenzaldehyde (PFBA, **1**) [C<sub>7</sub>H<sub>2</sub>F<sub>5</sub>O] has been known in Chemical Abstracts like "2,3,4,5,6-pentafluoro-benzaldehyde".

The literature search since 1958 revealed numerous pieces of information about this compound. There have been 841 references, containing 104 patents, however, 636 references have been actually accessible and synthetically interesting.

Pentafluorobenzaldehyde can be prepared from octafluorocyclohexane 1H:4H/2H:5H-, 1H:5H/2H:4H-, 1H:2H/4H:5H-octafluorocyclohexane. However, this is industrial mean of preparation. Pentafluorobenzaldehyde can be prepared directly from Grignard

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**Fig. 1.** The number of scientific publications containing PFBA as a function of publication year for the period 1958–2007, searched by Crossfire, Chemical Abstracts, ISI Web of Knowledge and Scopus.

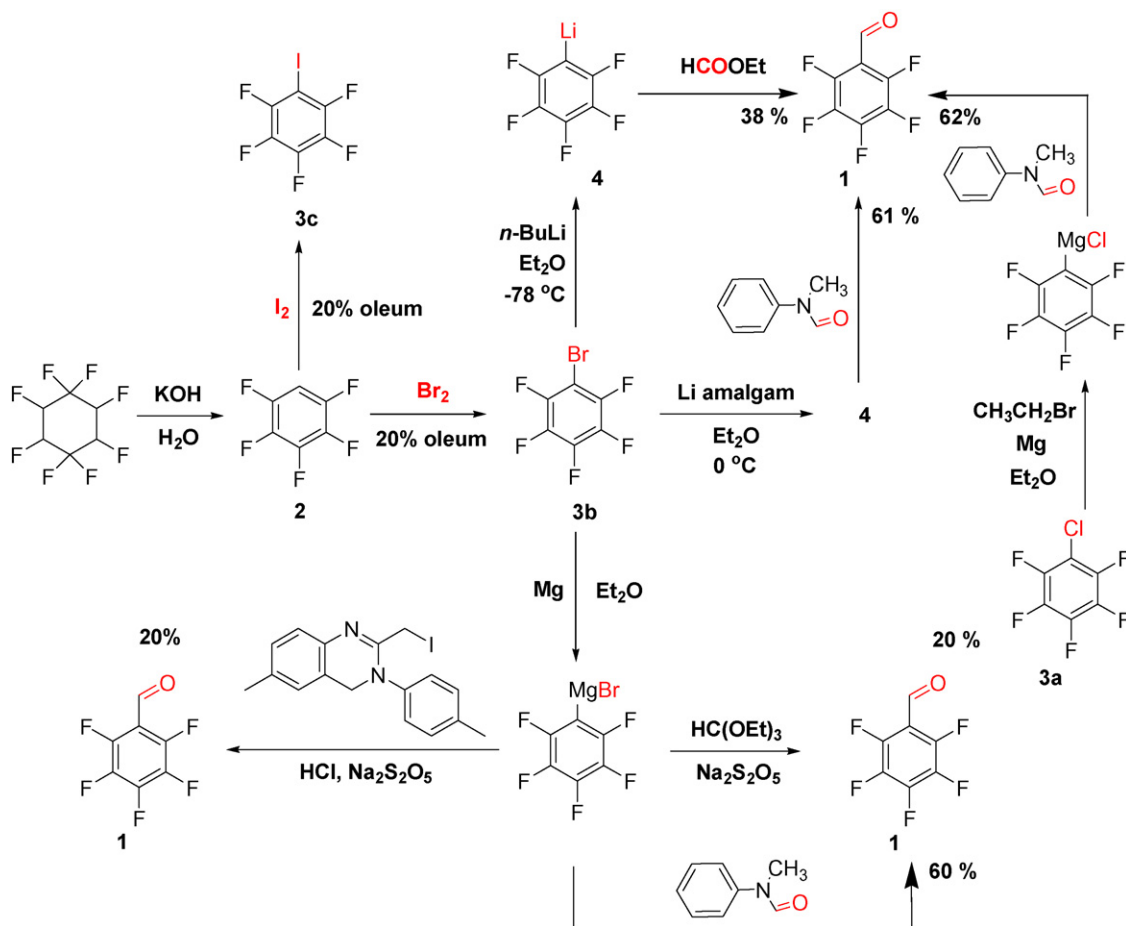
or lithiated reagents, from pentafluorobenzoic acid or other oxygen or pentafluorobenzene analogues, e.g. hexafluorobenzene.

The pentafluorobenzaldehyde is multifunctional aromatic compound containing five atoms of fluorine and one aldehydic function. Due to its physical and chemical properties and real or potential applications in various fields ranging from drug-delivery to analytical chemistry, interest in pentafluorobenzaldehyde has been growing rapidly, as demonstrated by the increasing number of publications within last 50 years (Fig. 1).

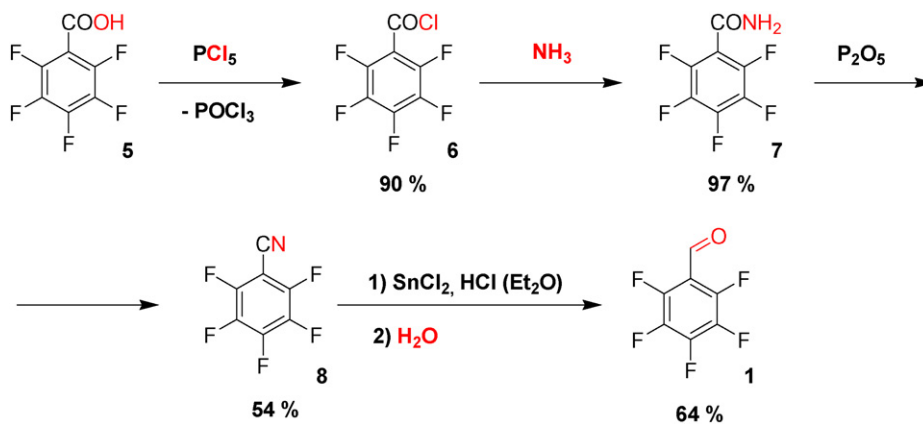
## 2. History and synthesis of pentafluorobenzaldehyde

As shown in Fig. 1, there has been steady increase of interest in chemistry of pentafluorobenzaldehyde. Only a few papers have been appeared the first 10 years since 1958, however, since 1994 a strong growth of interest is observed. In the year 2004, the rate of publication reached the limit 56 publications per year.

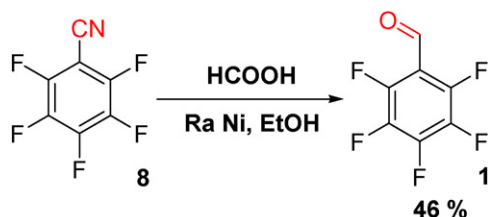
Pentafluorobenzaldehyde was first prepared in 1959 [1]. Nield et al. described in this paper the preparation of penta-



**Scheme 1.**



Scheme 2.



Scheme 3.

fluorobenzene (2), which was realized by dehydrofluorination of the industrial product, 1H:4H/2H:5H-, 1H:5H/2H:4H-, 1H:2H/4H:5H-octafluorocyclohexane. Bromopentafluorobenzene (3b) and iodopentafluorobenzene (3c) were prepared by subsequent bromination or iodination of the pentafluorobenzene (Scheme 1).

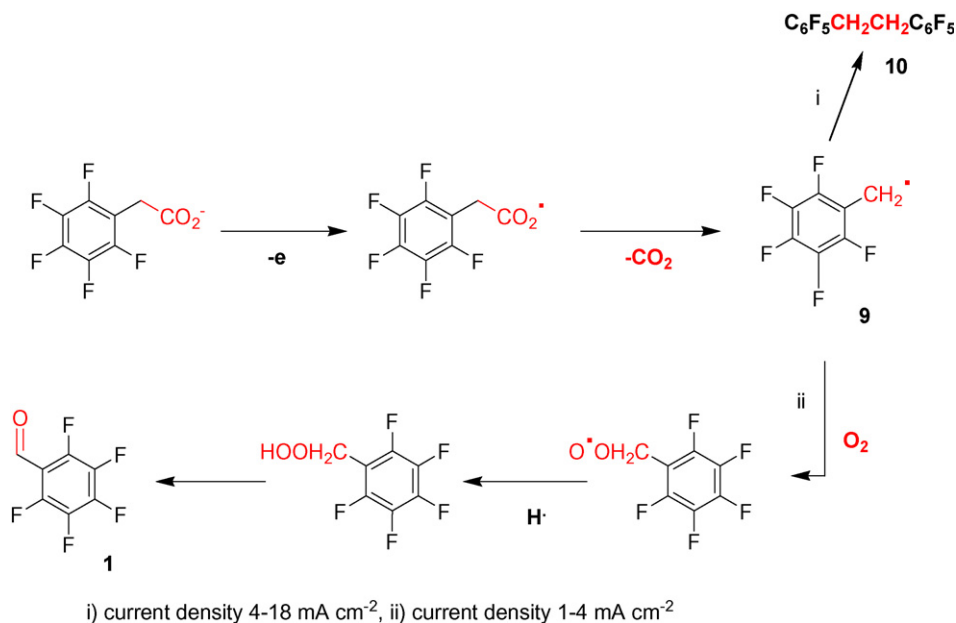
Compounds 3b and 3c react relatively easily with magnesium in dry ether and afford Grignard reagents. Pentafluorobenzaldehyde was prepared the first time by the reaction of these reagents with triethyl orthoformate or with 3,4-dihydro-2-iodomethyl-6-methyl-3-*p*-tolylquinazoline, however, the reaction was analogous in both cases. The drawback in both cases were rather low yields (20%).

In year 1964, Buxton and Tatlow published preparation of pentafluorobenzaldehyde by utilizing of the *N*-methylformanilide [2] and Grignard reagent (Scheme 1).

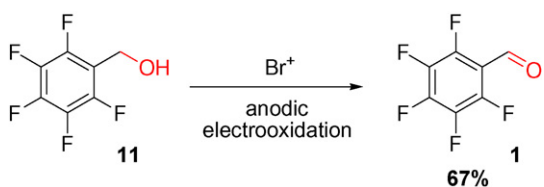
Another synthesis of pentafluorobenzaldehyde was based on preparation of organolithium compound 4 from bromopentafluorobenzene [3]. Preparation of 4 was realized by two ways: the first involving substitution of bromine by lithium with *n*-BuLi and the second being the direct metallation with lithium amalgam—a mixture of lithium and mercury. Pentafluorophenyllithium (4) from the first approach was allowed to react with ethyl formate to give pentafluorobenzaldehyde in the 38% yield. In the second approach, bromopentafluorobenzene reacted with lithium amalgam and 4 was allowed to react with *N*-methylformanilide to give pentafluorobenzaldehyde (1) in the 61% yield (Scheme 1). The preparation of 4 as a starting material for preparation of pentafluorobenzaldehyde (1) was described in other papers [4–7].

The first synthesis of pentafluorobenzaldehyde from chloropentafluorobenzene (3a) was published in 1964 [8]. Vorozhtsov et al. prepared the Grignard reagent from 3a and quenched it with *N*-methylformanilide to give 1 in 62% yield (Scheme 1).

For the preparation of pentafluorobenzaldehyde, Chapman et al. [9] in 1967 used as a starting material 2,3,4,5,6-pentafluorobenzoic acid as shown in Scheme 2. The first step in this synthesis



Scheme 4.



Scheme 5.

was preparation of 2,3,4,5,6-pentafluorobenzoyl chloride (**6**) by the reaction **5** with  $\text{PCl}_5$  in 90% yield. The conversion of **6** to amide by ammonia **7** gave 97% yield. Dehydration of **7** by phosphorus pentoxide  $\text{P}_2\text{O}_5$  gave pentafluorobenzonitrile (**8**) in 54% yield. The last step involved a reduction of **8** to **1** by stannic chloride in 62% yield. Authors described important pentafluorophenyl-substituted derivatives **6**, **7** and **8**, which were used later in many syntheses of other pentafluoro-substituted derivatives.

The reduction of **8** to **1** [10] was realized by Raney nickel in concentrated formic acid in 46% yield (Scheme 3).

Coleman et al. [11] found the predominance of pentafluorobenzaldehyde formation at low current density, when they used pentafluorophenylacetic acid for anodic oxidation on platinum (Scheme 4). It was attributed to the reaction between benzylic radical **9** and oxygen. By increasing the current density to  $15 \text{ mA cm}^{-2}$ , the yield of **1** plummeted (from 80% to 14%) and the dimer **10** was the major product, formed in 74% yield.

Takiguchi and Nonaka [12] published in 1987 electrooxidation of pentafluorobenzylalcohol **11** which gave pentafluorobenzaldehyde in 67% yield (Scheme 5).

Iwasaki et al. [13] claimed pentafluorobenzaldehyde formation as a by-product at electroreduction of pentafluorobenzoic acid on zinc amalgam cathode in 20% sulfuric acid in 31% yield.

Kunugi et al. [14] in year 1991 monitored pentafluorobenzoic acid electroreduction on Pb, Ni and Ni/PTE cathodes (Scheme 6). The major product of electroreduction was pentafluorobenzylalcohol, however, on Pb cathode 1% of **1** was identified. The electroreduction achieved reduction of *p*-fluorine except the reduction aldehydic function.

Vlasov et al. [15] in 1973 published the synthesis of pentafluorobenzaldehyde starting from 1,1-diethoxymethyl-2,3,4,5,6-pentafluorobenzene **13** in 75% yield (Scheme 7).

The preparation of **1** by reaction of Schiff base with trifluoroperoxyacetic acid, as shown in Scheme 8, proceeded with 50% yield [16,17]. The process involves is based on protection of the aldehydic function of pentafluorobenzaldehyde with aniline.

Petrenko et al. [16] and Furin et al. [17] planned to reach the nitron **15** formation with a Schiff base oxidation with trifluoroperoxyacetic acid, but they obtained **1** and the oxidation product—pentafluorobenzoic acid *N*-phenylamide **14** in 1:1 ratio.

In 2003 An et al. [18] published analogous epoxidation under the conditions of *m*-CPBA and  $\text{BF}_3 \cdot \text{OEt}_2$ . The preparation of **15** and its derivatives was described [16,19].

Activation of aromatic compounds towards oxygen by reductive photosensitization [20] is one of the possible methods of pentafluorobenzaldehyde preparation, provided two conditions are fulfilled:

- a leaving group (in our case bromine) must be present on benzylic carbon atom, and
- the photosensitizer in excited state must be an electron donor compound which is able to reduce an aromatic substrate (e.g. *N,N,N',N'*-tetramethyl-*p*-phenylenediamine).

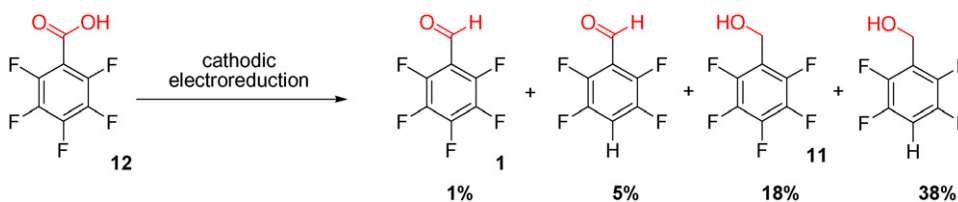
Butin et al. [21] used for oxidation of benzylbromide (**16**) to pentafluorobenzaldehyde in DMF a catalytic amount of  $\text{Et}_4\text{NBr}$ . The reaction involves the following sequence: an electron exchange between the photosensitizer and electron attracting fluorine—a radical anion formation, withdrawal of a leaving group leading to benzylic radical formation, which subsequently reacts with oxygen forming pentafluorobenzaldehyde **1** and pentafluorobenzylalcohol (Scheme 9).

Solčán in his Diploma Thesis [22] carried out a set of experiments in order to achieve electrophilic substitution of pentafluorobenzene (**2**) to pentafluorobenzaldehyde (**1**) with no luck. He wanted to use the Vilsmeier–Haack formylation with utilizing DMF and  $\text{POCl}_3$  and Reiche formylation in the presence of Lewis acid.

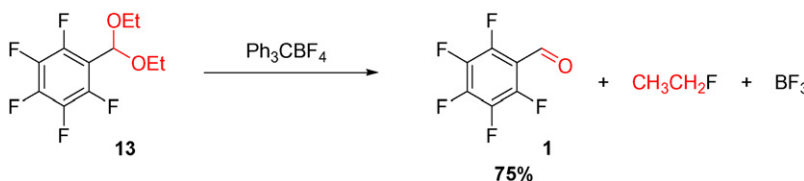
Végh et al. [23] published in 2007 a new method of pentafluorobenzaldehyde preparation, based on the synthesis 1,2,3,4,5-pentafluoro-6-nitromethylbenzene (**18**) from the easily available and cheap industrial product, hexafluorobenzene (**17**) (Scheme 10). From nitroderivative **18**, pentafluorobenzaldehyde, and a condensation by-product (**20**), was prepared by the action of  $\text{KOH}$  and  $\text{KMnO}_4$  in the presence of  $\text{MgSO}_4$ .

### 2.1. Physical properties of pentafluorobenzaldehyde and practically interesting materials obtained from it

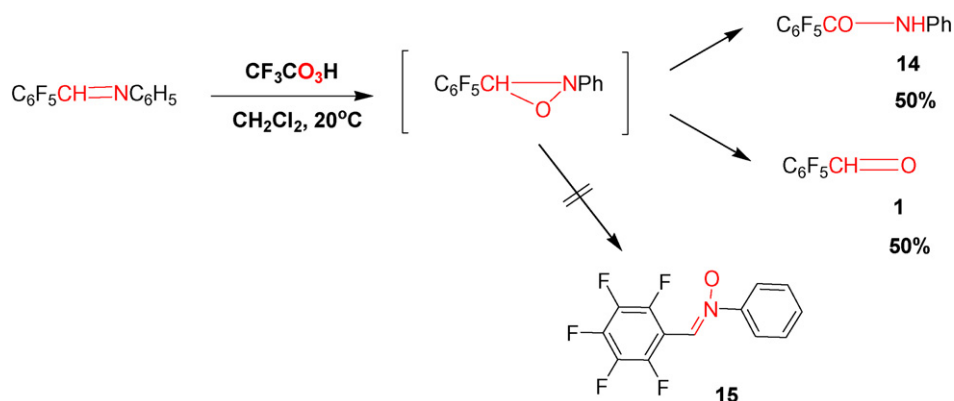
Pentafluorobenzaldehyde (Fig. 2) is colorless, lusciously smelling liquid, with b.p.  $168\text{--}170^\circ\text{C}$  and m.p.  $20^\circ\text{C}$  [24,25], density of



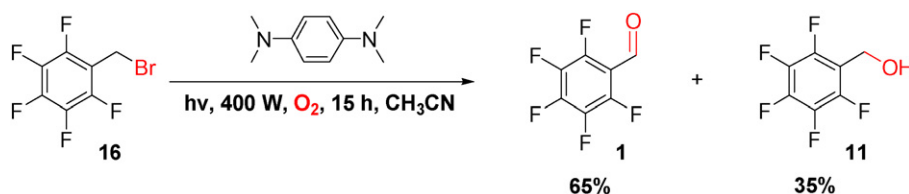
Scheme 6.



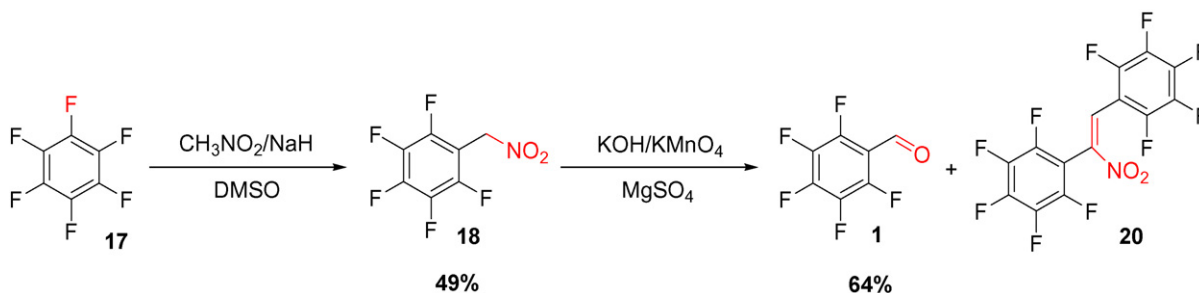
Scheme 7.



Scheme 8.



Scheme 9.



Scheme 10.

1.751 kg dm<sup>-3</sup> and refractive index  $n_D^{25} = 1.4488$  at  $\lambda = 589$  nm [26]. The dipole moment is  $\mu = 1.97$  D (Debye). <sup>1</sup>H NMR spectrum in CDCl<sub>3</sub>: hydrogen from aldehydic function resonates at 10.02 ppm (TMS). <sup>13</sup>C NMR (TMS): carbon nuclei C<sup>1</sup>–C<sup>5</sup> resonate at 181.14, 111.50, 147.81, 138.04, and 145.38 (the values in ppm, the chemical shift C<sup>6</sup> is identical to C<sup>4</sup>, C<sup>7</sup> to C<sup>3</sup> [27,28]).

<sup>19</sup>F NMR spectra were reported [29–32]: fluorine nuclei F<sup>3</sup>–F<sup>7</sup> resonate at –145.2, –161.5, –145.2, –161.5, and –145.2 (the values in ppm). UV spectrum (*n*-hexane):  $\lambda_{\max} = 235$  nm ( $\epsilon = 17,330$ ), 243 ( $\epsilon = 14,780$ ). IR spectrum in CCl<sub>4</sub> shows bands at 1630, 1715, 1653 cm<sup>-1</sup>, using other solvents did not give different results [33,34]. Mass spectrum MS anion radical from pentafluorobenzaldehyde:  $m/z = 196$  [35,36].

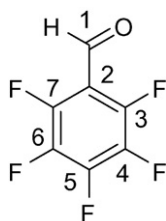


Fig. 2.

Many publications and patents have reported preparations of magnetic and conductive polymers and functionalized copolymers [37–49], dendrimers [50,51], dyes [52,53] and chemiluminescent materials too [54]. Other interesting data such as molecular polarizability, energies, electrochemical properties, half-time of fluorescence or phosphorescence, etc., have been reported too [55–78].

## 2.2. Biological properties of pentafluorobenzaldehyde and its derivatives

Some publications have reported about interesting data of biological activity [79] and toxicity of pentafluorobenzaldehyde [80–99], preparations of biologically active compounds (antibio-

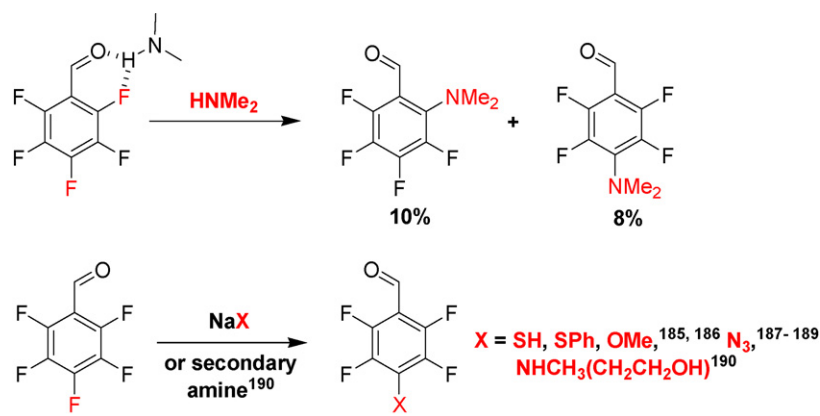
**Table 1**  
Toxicity of pentafluorobenzaldehyde.

Class	–log(LC <sub>50</sub> )	Duration	–log(LC <sub>50</sub> )
Harmful, irritating	3 <sup>a</sup>	96 h <sup>b</sup>	5.25 <sup>c</sup>

<sup>a</sup> Admans et al. [80], class 3: very toxic compound.

<sup>b</sup> Cash and Clements [82], Protić and Sabljčić al. [90], Ren et al. [91] (acute toxicity, LC<sub>50</sub> in mmol/l).

<sup>c</sup> Hall et al. [84], Gao et al. [85] (research on fish samples, LC<sub>50</sub> in mol/l).



Scheme 11.

tics [100–103], antiviral agents [104–111], dihydropyridines [112–117], aminoguanidines [118] and the other compounds [119–184]).

Pentafluorobenzaldehyde is harmful in contact with skin (S-26), it is harmful when inhaled or and if swallowed (R 20/22), irritating to eyes, respiratory system and skin (R 36/37/38).

Table 1 summarizes some biological and toxic properties of pentafluorobenzaldehyde.

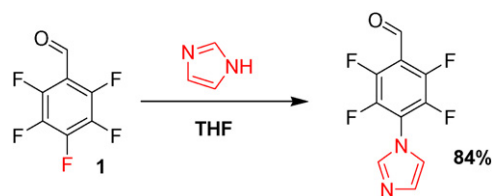
### 3. Chemical reactions of pentafluorobenzaldehyde

Pentafluorobenzaldehyde is a multifunctional aromatic compound, which contains in its molecule five atoms of fluorine and one aldehydic function. Possible reaction centres are shown in Fig. 3.

Concluding from <sup>19</sup>F NMR spectra of pentafluorobenzaldehyde, the atoms of fluorine are not equivalent [29–32]. In S<sub>N</sub>Ar types of reactions, there are activated *ortho*- and *para*-position, while *meta*-position are more resistant towards nucleophiles.

#### 3.1. S<sub>N</sub>Ar reactions of pentafluorobenzaldehyde

The first publications dealing with issue of nucleophilic substitution of fluorine originated in the year 1968 [185], and focused on nucleophilic substitution of *o*-fluorine and *p*-fluorine by dimethylamine (Scheme 11). Other works like that [186–190] were published later and dealt with nucleophilic substitution of *p*-fluorine by sulphur, oxygen and nitrogen



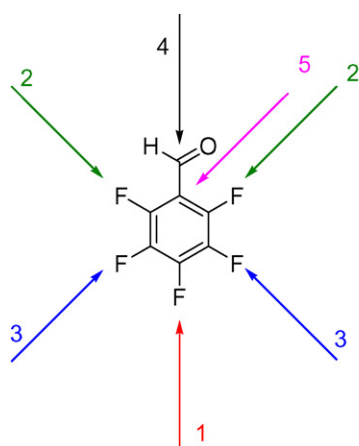
Scheme 12.

nucleophiles (Scheme 11). The most studied area has been the substitution with azide anion [187–189]. In the later patented work, *N*-(2-hydroxyethyl)-*N*-methylamine was utilized for nucleophilic substitution (Scheme 11).

The substitution of *o*-fluorine by dimethylamine has been explained by assuming a hydrogen bond between the aldehydic hydrogen and amine or by association between the nucleophile and the substrate, involving the positively polarized aldehydic carbon and lone-pair electrons of nitrogen in amino group (Scheme 11).

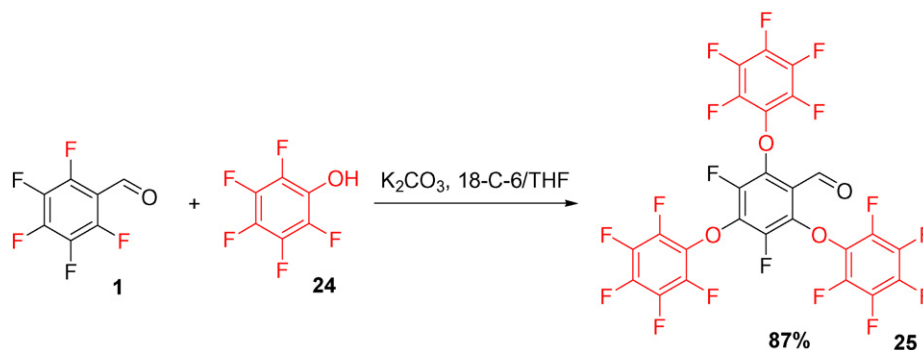
Imidazole is known to be a weak base, with lower reactivity towards arylhalides than other amines, however, in the imidazole substitution of pentafluorobenzaldehyde, the replacement of the fluorine in *p*-position proceeds in yield 84% (Scheme 12 [191]).

In a 2004 patent Roux et al. presented the synthesis of tri-substituted derivative of pentafluorobenzaldehyde **25** starting from pentafluorophenol **24** (Scheme 13). The substitution occurred both in *p*- and *o*-positions [192].



1. S<sub>N</sub>Ar – nucleophilic substitution oriented to *para*
2. S<sub>N</sub>Ar – nucleophilic substitution oriented to *ortho*
3. S<sub>N</sub>Ar – nucleophilic substitution oriented to *meta*
4. Ad<sub>Nu</sub> – nucleophilic addition
5. Haloform reactions

Fig. 3. Reactivity of functional groups of pentafluorobenzaldehyde. (1) S<sub>N</sub>Ar: nucleophilic substitution oriented to *para*. (2) S<sub>N</sub>Ar: nucleophilic substitution oriented to *ortho*. (3) S<sub>N</sub>Ar: nucleophilic substitution oriented to *meta*. (4) Ad<sub>Nu</sub>: nucleophilic addition. (5) Haloform reactions.



Scheme 13.

There are many known cases, when the substitution of all fluorine atoms occurs by utilizing 5 equivalents of alkaline salts of sulphur nucleophiles. In the first case, the potassium salt of benzenethiol in dimethylformamide (DMF) is utilized [185] and in the second, the sodium salt of benzenethiol 1,3-dimethyl-2-imidazolidinone (DMI) is utilized [193–195]. When the temperature was raised to 60 °C, the benzene derivative **26** resulted (Scheme 14 [185]).

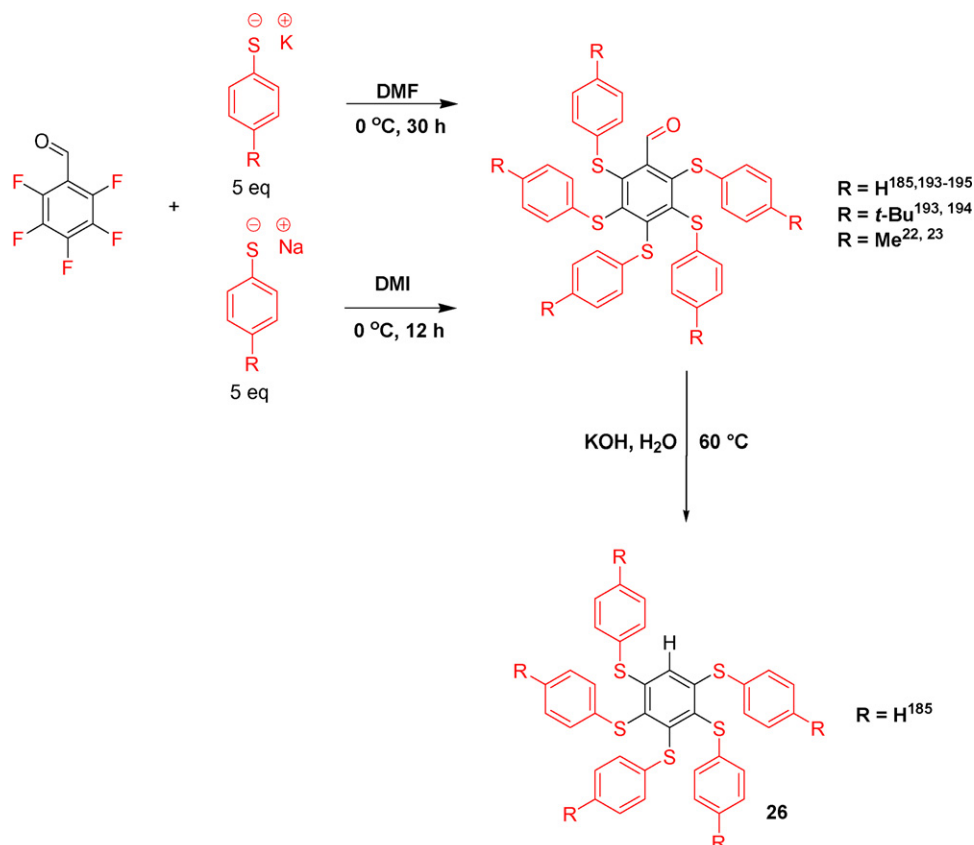
The *p*-fluorine substitution [196] has been involved, when the replacement of fluorine atom in *p*-position with hydrazine (**26**), morpholine (**27**) and *tert*-butoxy group occurs after protecting of aldehydic function into diethylacetal. In the last case, acidification with HCl yielded the phenol derivative (**28**). If the diethylacetal is being transformed by  $LiAlH_4$ , the substitution of fluorine with hydrogen occurs, and the reaction with BuLi and  $CO_2$  yields the carboxylic acid (**29**). If the hexa(ethylene glycol) **30** in THF with

BuLi in hexane is utilized, the reaction leads to disubstituted derivative of hexa(ethylene glycol) **31** (Scheme 15 [197]).

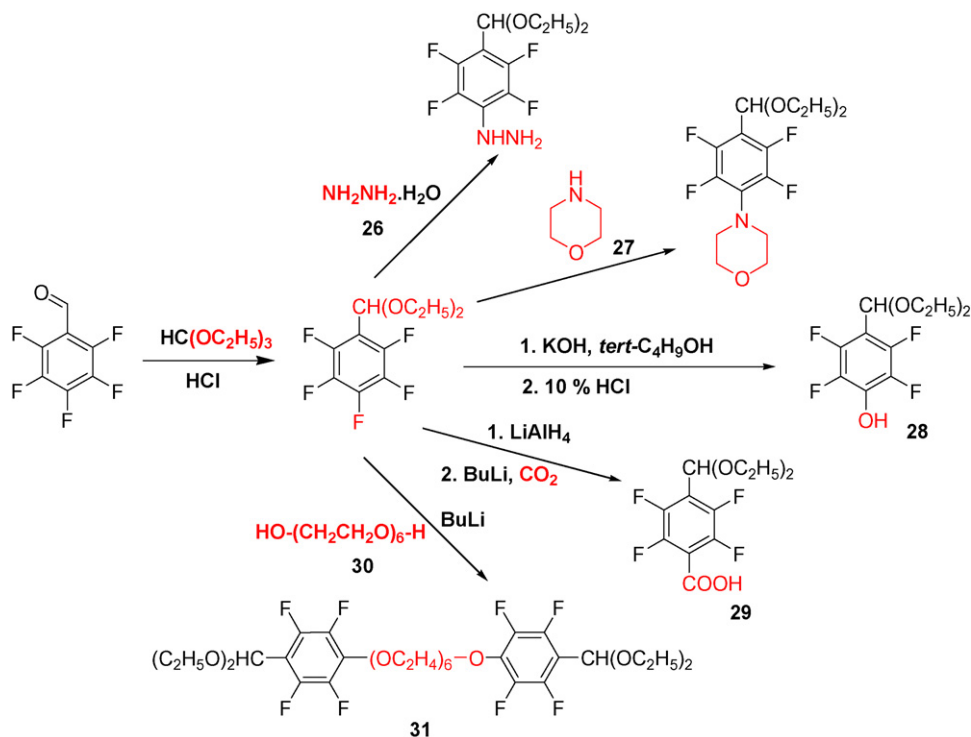
### 3.2. Chemical reactions of aldehydic group of pentafluorobenzaldehyde

Pentafluorobenzaldehyde is a highly reactive aromatic aldehyde which affords a large variety of products typical for aromatic aldehydes. The atoms of fluorine with strong electronegative character notably strengthen the electrophilic character of the carbonyl group.

Davies and Symes [198] presented the sequence for carbonyl compounds acting as nucleophile (without concerning the aldehydic oxygen):  $Cl_3CHO > Br_3CCHO > CF_2ClCOFCl_2 > (Cl_3C)_2CO > C_6F_5CHO$  (**1**)  $> CH_3CHO$ , which is manifested mainly in Grignard-like reactions [198].



Scheme 14.

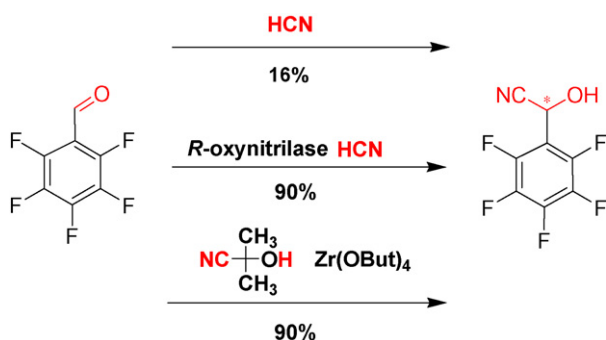


Scheme 15.

The literary data show that the pentafluorobenzaldehyde gives easily products with carbon (Section 3.2.1) and nitrogen nucleophiles (Section 3.2.2), with oxygen and sulphur nucleophiles (Section 3.2.3) and with other nucleophiles (Section 3.2.4). However, no examples have been found for benzoin condensation (although the utilization of pentafluorobenzaldehyde at the cross-benzoin condensation has been already published [199]), or for the Cannizzaro reaction and Darzens reaction.

### 3.2.1. Chemical reactions of pentafluorobenzaldehyde with carbon nucleophiles

Pentafluorobenzaldehyde affords by reaction with hydrogen cyanide in the solvent mixture of water–alcohol addition product cyanhydrine, nitrile of 2-hydroxy-2-(pentafluorophenyl)acetonitrile in yield 16% (Scheme 16). Analogous reaction was the first synthesis of (*R*)-fluorinated mandelonitriles, by hydrocyanation under micro-aqueous conditions with almond meal containing enzyme (*R*)-oxynitrilase as catalyst (Scheme 16 [200]). By utilizing sodium cyanide, acetyl chloride and zinc bromide at room temperature for analogous reaction, after 40 h 92% *O*-acetyl derivative of 2-hydroxy-2-(pentafluorophenyl)acetonitrile was obtained [201]. Analogous yield (92%) was found when utilizing



Scheme 16.

trimethylsilylated derivative of pentafluoromandelic nitrile in the reaction of pentafluorobenzaldehyde with trimethylsilyl cyanide and zinc bromide in acetonitrile. If NaCN is combined with protecting functional group (TBS, Ac), introduction of a cyano group makes further synthetic exploitation possible [202–204].

Meerwein–Pondorf–Verley cyanation is based on acetone cyanhydrine utilization [205–210] as relatively low-toxic chemical equivalent of hydrogen cyanide, catalysed with  $Zr(Ot-Bu)_4$ . After 10 min, at room temperature, 90% of racemic nitrile of pentafluoromandelic acid was formed (Scheme 16). Some new methods of acetone cyanhydrine formation have utilized hydroxynitrile lyase isolated from the seed of Japanese apricot [211].

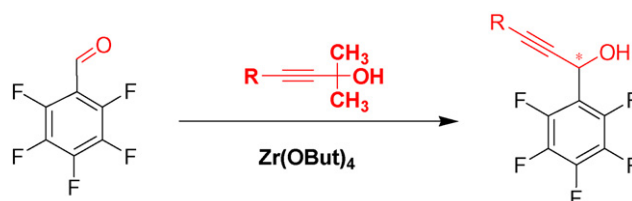
Under similar conditions, Meerwein–Pondorf–Verley alkynylation overshoots (Scheme 17 [205,207,208,212]).

Introducing of triple bond onto carbonyl group in other conditions has been known too [213].

Introducing of double bond onto carbonyl group was realized in the conditions of Baylis–Hillman reaction (Scheme 18 [214–217]) or by chiral catalyst with high yields (ene reactions) and often with high enantioselectivity (*ee* = 99%, Scheme 19 [218–221]).

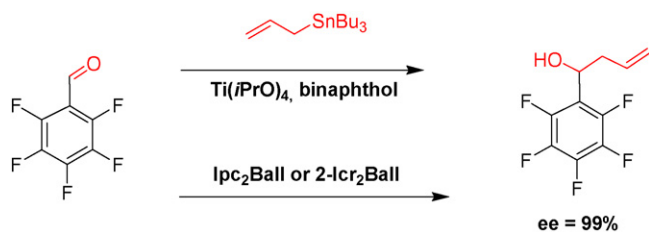
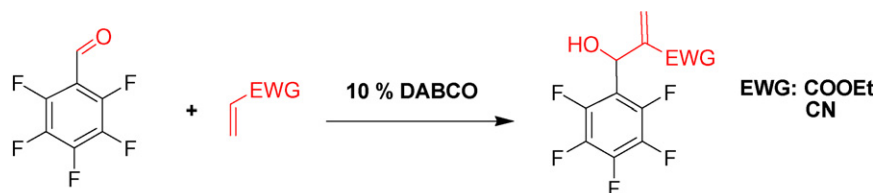
Many enantioselective alkylations of pentafluorobenzaldehyde by dialkyl zinc were published, where enantiomeric excess is ranged from 42% to 97%, depending on the utilized catalyst and alkyl group (Scheme 20 [222,223]).

In the year 1972 a “Grignard-like” reaction of aldehydic function of pentafluorobenzaldehyde in the presence of mercury salts was published, using the  $S_E2$  conditions. The reaction of



Scheme 17.





bis(acetonyl)mercury (**32**) with pentafluorobenzaldehyde in dimethoxyethane at room temperature gave the appropriate product **33** (Scheme 21, product was isolated as its 2,4-dinitrophenylhydrazone [224]).

However, as early as in year 1968, Leusink et al. dealt with reactions of some strong electrophilic carbonyl compounds with

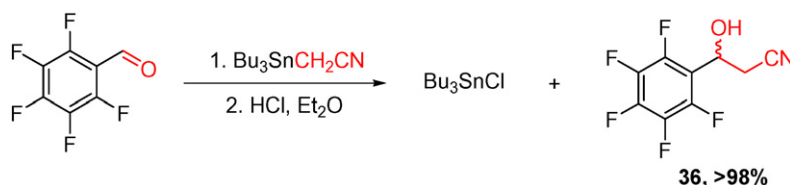
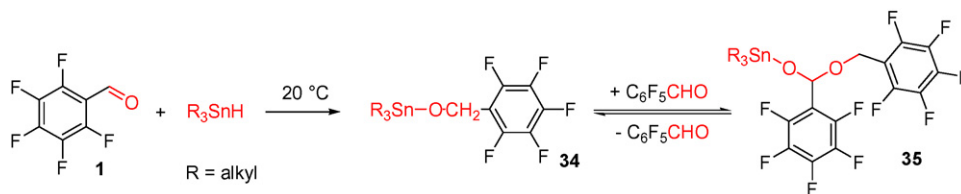
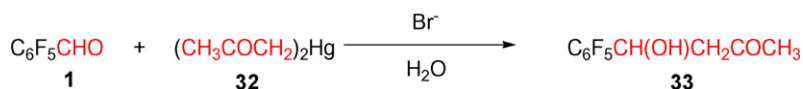
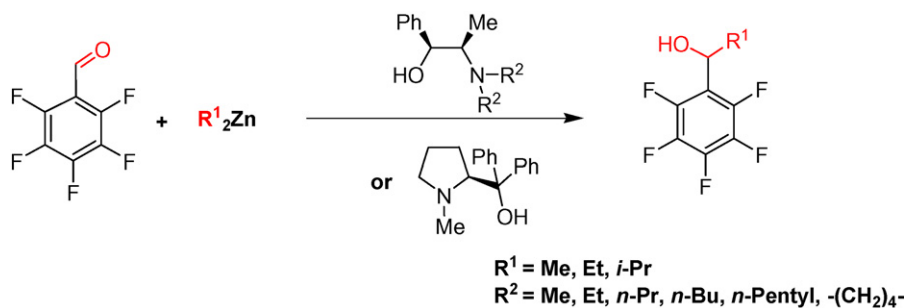
tin compounds. They gained some knowledge about hydrostannation of pentafluorobenzaldehyde with the organotin compounds **34** and **35** formation (Scheme 22 [225]).

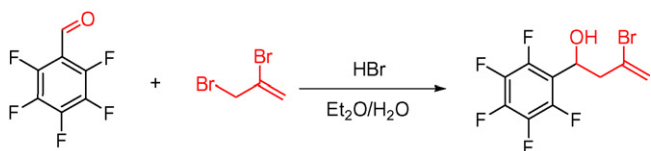
Utilizing of tin compounds allowed lengthening of the carbon skeleton (Scheme 23 [226,227]).

The reaction of pentafluorobenzaldehyde with 2,3-dibromopropene onto appropriate alcohol proceeded with the 80% yield (Scheme 24 [228]).

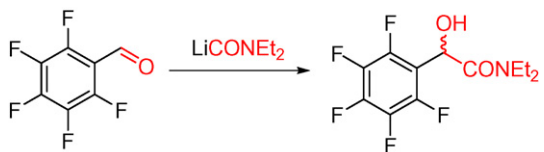
Other syntheses involving a reductive transformation of aldehydic function to hydroxy function (Scheme 25 [229]) were presented in earlier works [230–233] and later too [234–266].

The pentafluorobenzaldehyde in acetic anhydride, when treated with potassium acetate yielded 44% (*E*)-3-pentafluorophenyl-prop-2-ene acid (Scheme 26 [267]). In the conditions of Doebner condensation [267] with malonic acid in dry pyridine at temperature 100 °C during 1.5 h, it yielded 60% (*E*)-3-pentafluorophenyl-prop-2-ene acid. According to Barbour et al., if the basic

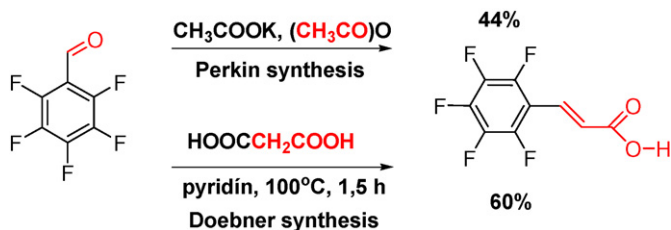




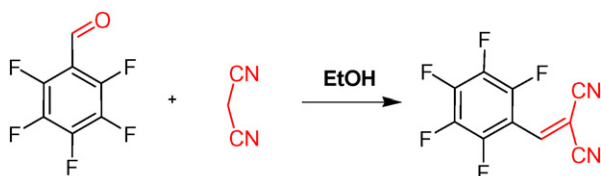
Scheme 24.



Scheme 25.



Scheme 26.



Scheme 27.

catalyst piperidine typical for the Doebner condensation was utilized, the yield was lower (Scheme 26).

The condensation of pentafluorobenzaldehyde with malonic dinitrile in ethanol at temperatures 20–25 °C yields 71% of pentafluorobenzylidene malonic dinitrile (Scheme 27 [268]).

For Knoevenagel condensations of pentafluorobenzaldehyde, authors commend low reaction temperatures 15–25 °C without basic catalysis. The strong and selective UV absorptions and the interaction constant values of vinyl protons (14–18 Hz) indicate *E*-isomer formation. The products of the Knoevenagel condensation afford saturated analogues after Ra-nickel hydrogenation.

The PFBA reacts very easily with nitromethane and with nitroethane in condensation reactions [23,269]. After 5 h heating in the presence of ammonium acetate, nitrostyrenes (**37b**) are formed in high yields (Scheme 28). The reaction of oxime with

PFBA (Scheme 28) is analogous, with oxazacyclic derivative forming (**37a** [270]).

The condensation reactions of pentafluorobenzaldehyde with methyl or with methylene function of some heterocycles or with methyl function of the carbonyl compounds proceed very easily [271–297]. Some of these condensation reactions are utilized in the synthesis of compounds with high biological activity [289,298]. Some examples of the condensation reactions and their products (**38–45**) are shown in Scheme 29, infrequent cases have been described too [299–302].

The reaction of the PFBA with 3-methylindole (**46**) in the presence of acid (methanol and concentrated hydrochloric acid) does not afford the condensation product with the methyl function, but addition product (**47**) with two equivalents of 3-methylindole in position 2 of the indole core. The reaction proceeds at room temperature and takes 18 h with 15% product **47** (Scheme 30 [303]).

The PFBA with the diketene (**48**) gives pentafluorobenzylidene acetone (**49**) in good yield. It is multi-step synthesis, where ketoforming cleavage is realized in final step and the pentafluorobenzylidene acetone forms (Scheme 31 [304]).

The PFBA does not afford the Cannizzaro reaction, but so-called “haloform reaction” [185], which is typical of fluorocarbonyl compounds with pentafluorobenzene and formic acid forming, proceeds in the presence of water solution NaOH and KOH (Scheme 32).

The Wittig reactions of pentafluorobenzaldehyde were researched widely [274,305–319]. The PFBA reacts easily with fluoromethylene triphenylphosphorane (**50**) in toluene and this reaction gives (*E*)-, (*Z*)-isomer fluorovinyl benzene (**51a**, **51b**, Scheme 33 [305]). These compounds are very important monomers for producing perfluorinated polymers and copolymers.

In the reaction of the PFBA with 2,3,4-methylene-oxadiazolyl-triphenylphosphorane (**52**) is formed pentafluorostyrene (**53**), which is substituted with heterocycle (Scheme 34 [306]).

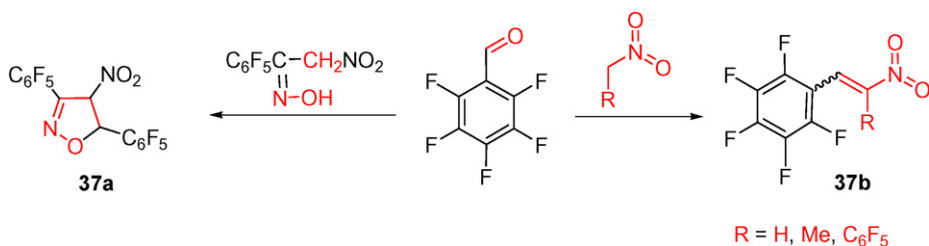
Danilenko et al. [307] realized the synthesis of decafluorostilbene (**54**) and pentafluorostilbene (**55**) by Wittig reaction. There were formed derivatives like pure (*E*)-isomers (Scheme 35).

If utilizing the difluorobromotriphenylphosphonium bromide and metal cadmium for ylide formation, according to Burton [274] the difluorovinylpentafluorobenzene is formed in Wittig reaction (**56**, Scheme 36).

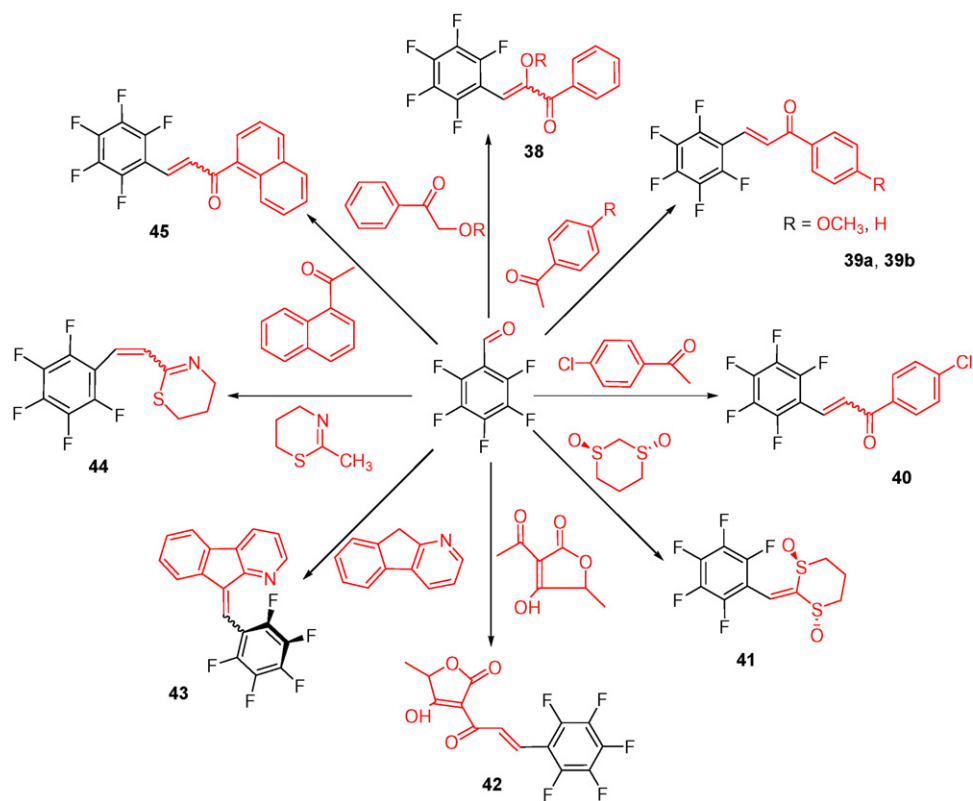
The identical derivative **56** is formed, when phosphonium ylide is generated “*in situ*” from dibromodifluoromethane and triphenylphosphine (Scheme 36).

The pentafluorobenzaldehyde with tetrabromomethane affords in Wittig conditions dibromovinylpentafluorobenzene, which is transformed to acetylene derivative by utilizing BuLi with yield 85% (Scheme 37 [320,321]).

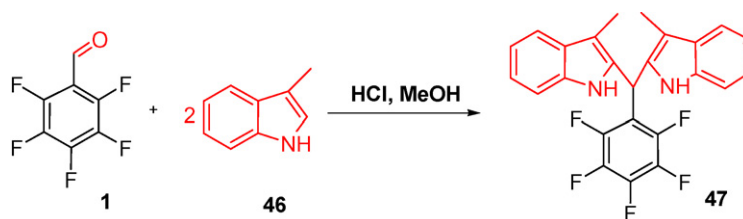
The pentafluorobenzaldehyde has been applied in research of new materials in porphyrin chemistry [111,184,322–441], chemistry of corroles (**58** [442–455]), porphocyanines (**60**) and their precursors (**59** [456–461]). The reaction of pentafluorobenzaldehyde with non-substituted and substituted pyrroles affords the set



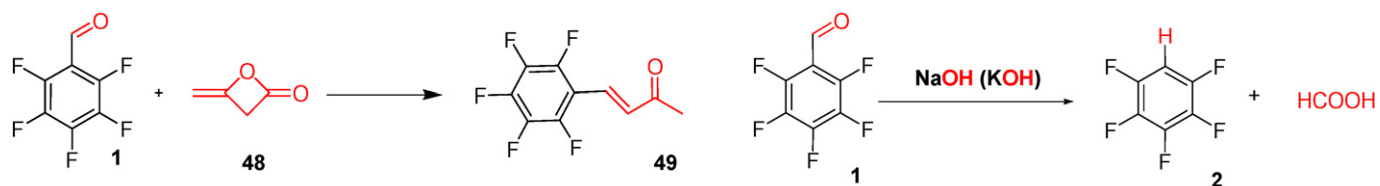
Scheme 28.



Scheme 29.

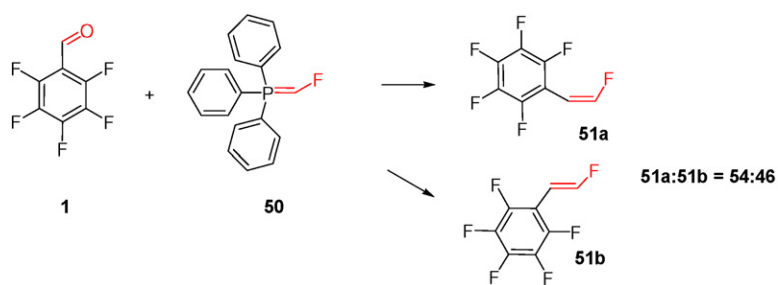


Scheme 30.

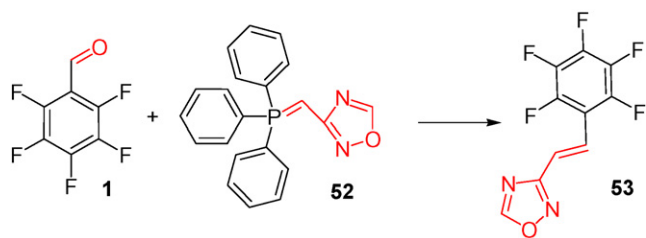


Scheme 31.

Scheme 32.



Scheme 33.

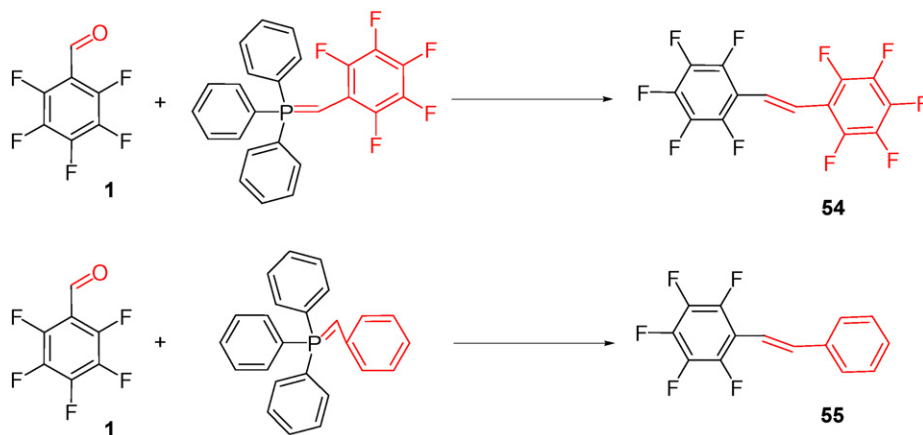


Scheme 34.

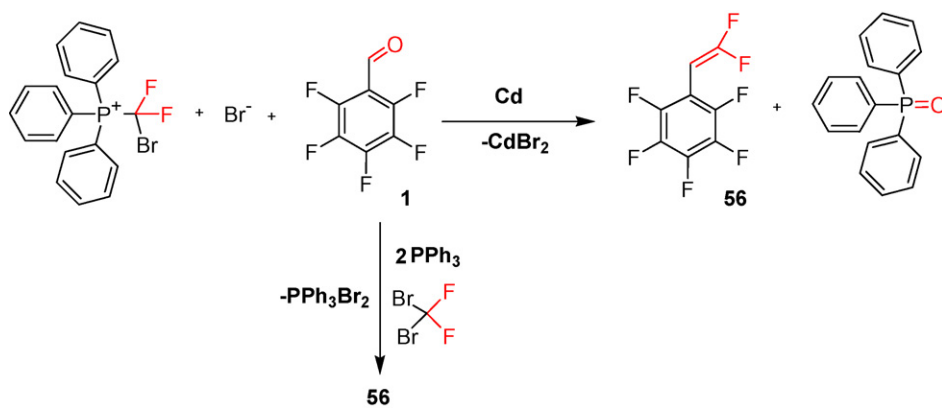
of porphyrins (**61**), which are utilizable in many fields of chemistry (Scheme 38 [348,462–467]).

Along this line, the synthesis of expanded porphyrin cycles—calixphyrins is interesting too (**62**, Scheme 39 [468–470]).

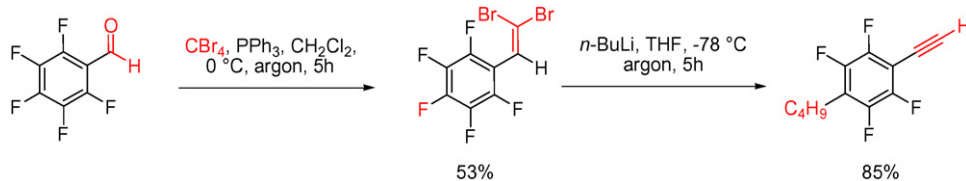
The reaction of pentafluorobenzaldehyde with carbon nucleophiles possessing reactive groups on the methylene group often gives miscellaneous heterocycles such as oxazoles (Scheme 40 [471–473]), oxazolones (Scheme 41 [474,475]) and terpyridines (Scheme 42 [476]).



Scheme 35.



Scheme 36.



Scheme 37.

Soloshonok et al. [471–473] has presented new knowledge about gold(I)-catalyzed asymmetric reaction of pentafluorobenzaldehyde with methyl isocyanoacetate in several publications (Scheme 40). The gained diastereomeric oxazoles can be hydrolyzed to β-hydroxyaminoacids (**63**, **64**). In the literature the preparations of isoxazole derivatives from pentafluorobenzaldehyde oximes [477–479] through the pentafluorobenzaldehyde are known.

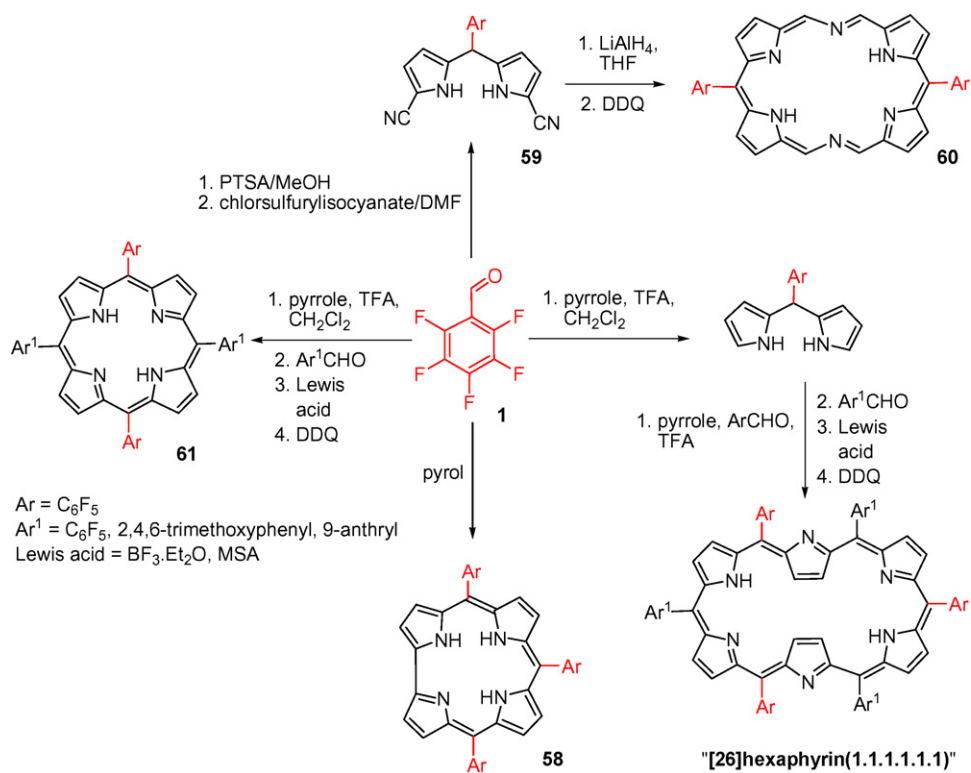
In earlier works [474,475], syntheses of oxazolone derivatives **65** and **66** (Scheme 41) have been presented.

In 2003, a preparation [476] of pentafluorophenyl substituted terpyridine **67** was published (Scheme 42).

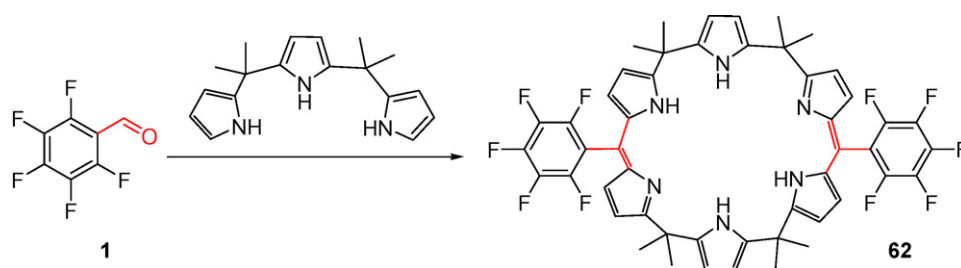
### 3.2.2. Chemical reactions of pentafluorobenzaldehyde with nitrogen nucleophiles

The pentafluorobenzaldehyde reacts with primary aliphatic amines with appropriate imines forming (**68**, Scheme 43 [480,481]). The PFBA reacts with aromatic amines in alcohol to afford high yields of Schiff bases (Scheme 43 [16,482–499]).

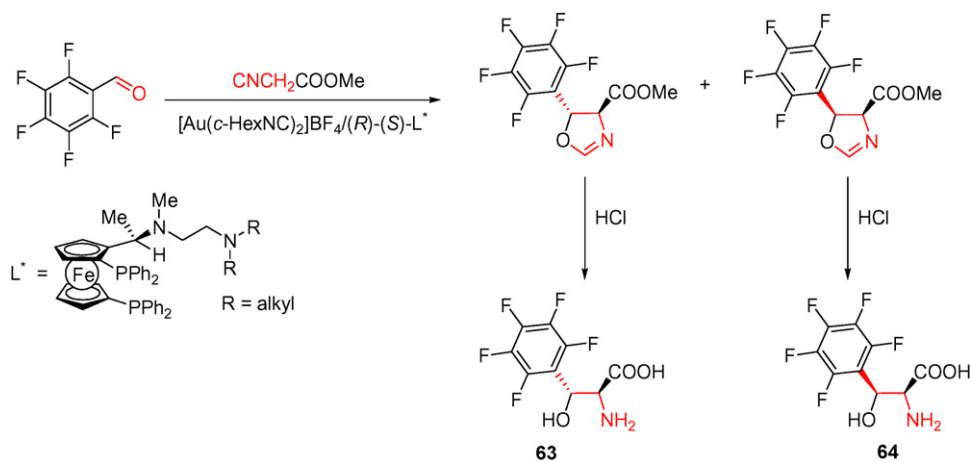
The pentafluorobenzaldehyde is utilizable in asymmetric synthesis too. Brethon et al. presented it in the synthesis of



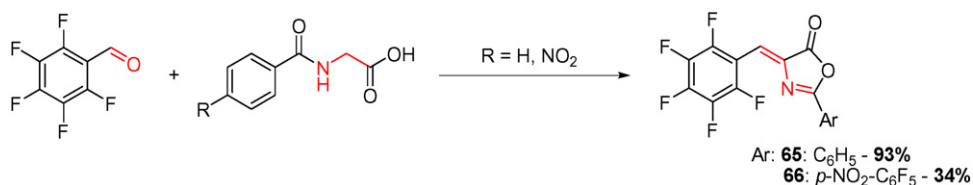
Scheme 38.



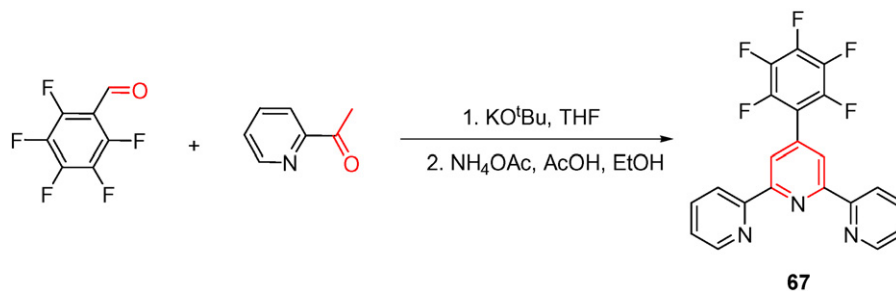
Scheme 39.



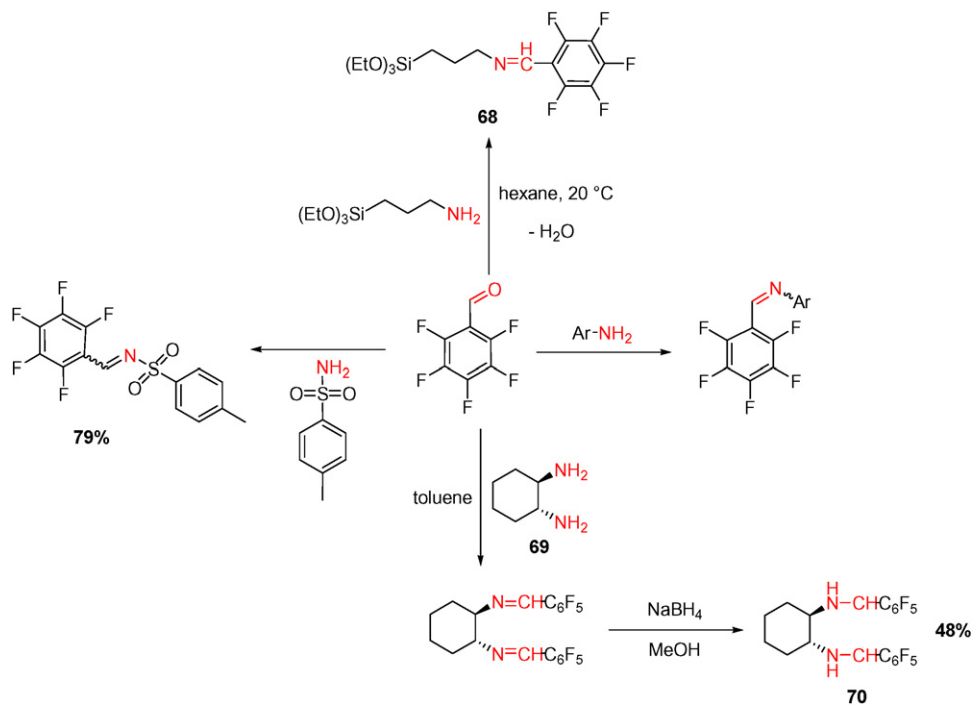
Scheme 40.



Scheme 41.



Scheme 42.



Scheme 43.

ligands from derivative of *trans*-(1*R*,2*R*)-diaminocyclohexane (**69**). The reaction of this compound with pentafluorobenzaldehyde in dry toluene followed by a reduction in methanol gives an appropriate product of reductive amination **70** in yield 48% (Scheme 43 [500]).

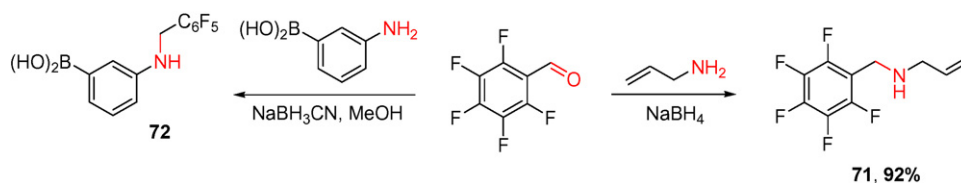
The pentafluorobenzaldehyde when treated the toluenesulfonamide yields 79% *N*-tosyl-pentafluorobenzylidene amine (Scheme 43 [501–503]).

In reducing conditions pentafluorobenzaldehyde with primary amines affords products of reductive amination, e.g. the secondary amine **71** (Scheme 44 [504]).

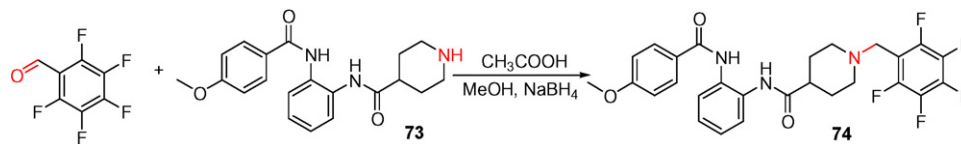
In 2004 the synthesis of 3-(pentafluorobenzylamine)phenylboronic acid (**72**) was published. This acid is among the other boronic acid inhibitor tyrosine kinase. Asano et al. [505] gained this product of reductive amination **72** in a one-step synthesis (Scheme 44).

The pentafluorobenzaldehyde reacts with secondary amines (**73**) to form an appropriate product of reductive amination **74**, which is biologically active compound (Scheme 45 [506]).

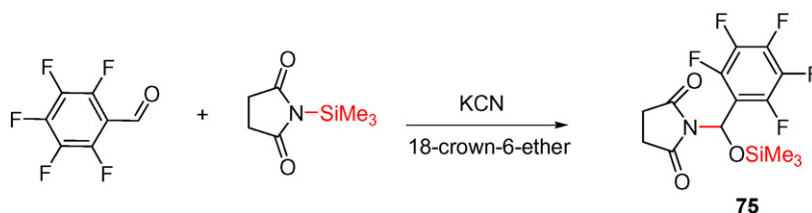
The reaction of the PFBA with *N*-trimethylsilylsuccinic amide involves addition of tetramethyl silane on aldehydic function, forming the appropriate product **75** (Scheme 46 [507]).



Scheme 44.



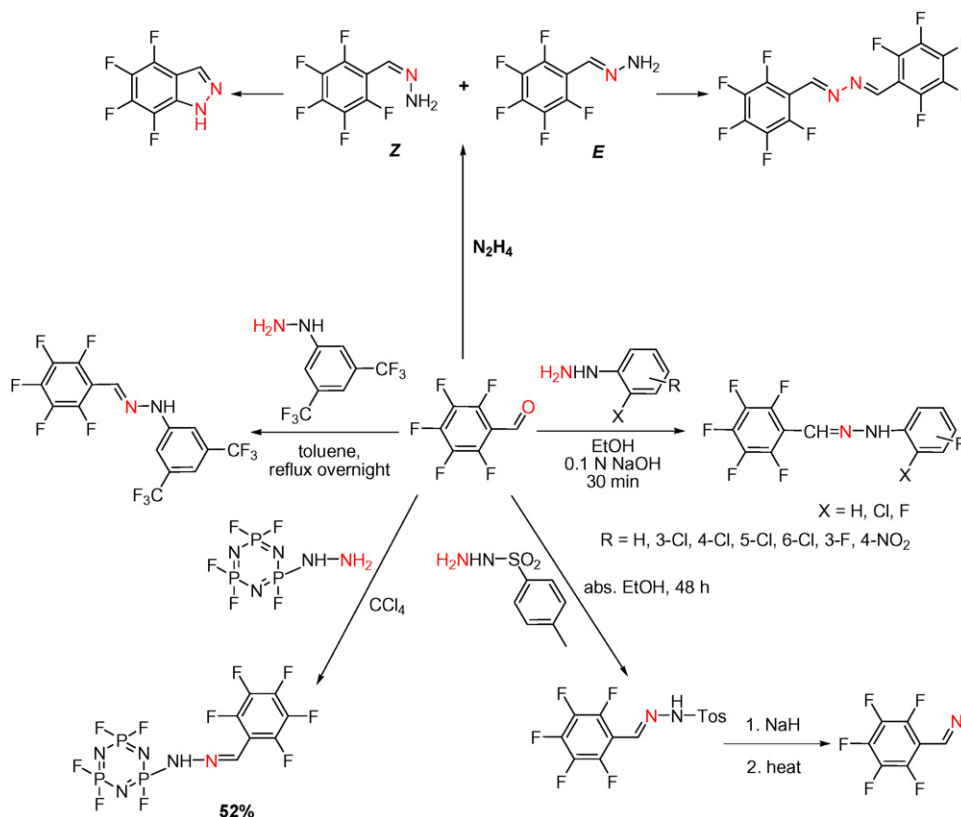
Scheme 45.



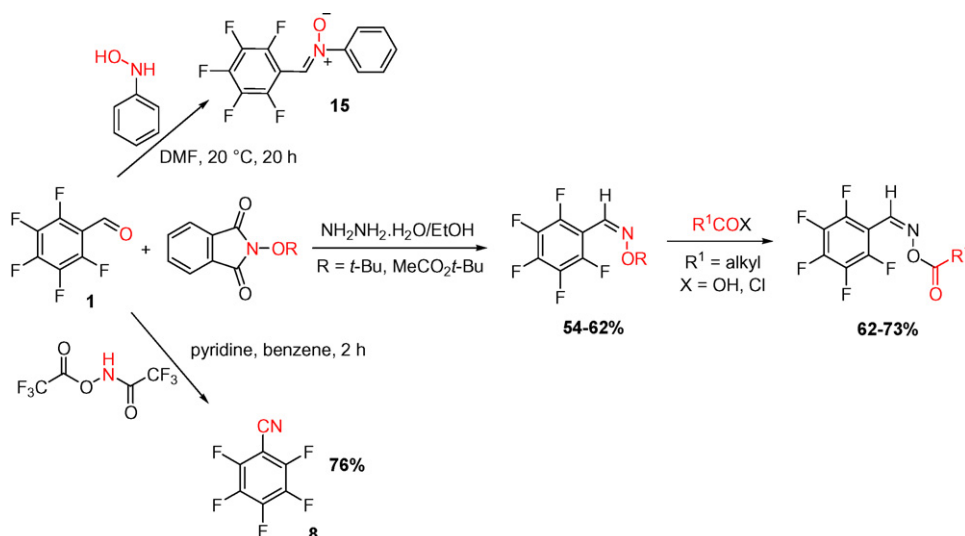
Scheme 46.

In the condensation of the PFBA with the hydrazine, the (*E*)- and (*Z*)-hydrazone form. While (*E*)-isomer undergoes next a condensation with an appropriate azine formation [508–510], the (*Z*)-isomer reacts in different manner, its cyclization reaction gives 4,5,6,7-tetrafluoro-1*H*-indazole (Scheme 47 [508]).

In 1997 a three-step synthesis of the 2,3,4,5,6-pentafluorophenyldiazomethane was published, in which the starting material was the PFBA and *p*-toluenesulfonyl hydrazide with a hydrazone forming in the first step, followed reduction of the tosyl group with NaH and heating to afford the target diazomethane (Scheme 47 [511,512]).



Scheme 47.



Scheme 48.

The reaction listed above is a modification of the Bamford–Stevens reaction [513,514].

Similarly to the hydrazine, the phenylhydrazine derivatives treated with the PFBA in ethanolic solution and 0.1N NaOH form the substituted phenylhydrazones (Scheme 47 [515–519]).

Frasca in 2004 published the synthesis of the hydrazone in 78% yield and starting from PFBA and 3,5-bistrifluoromethyl-phenylhydrazine (Scheme 47 [520]).

The PFBA reacts with fluorinated phosphazenes containing a hydrazine group, forming the pentafluorobenzylidene hydrazones (Scheme 47 [521]).

The PFBA oxime derivatives were prepared by the reaction with substituted phthalimide (Scheme 48 [522,523]). The PFBA with *O,N*-bis(trifluoroacetyl)hydroxylamine in benzene yields 76% pentafluorobenzonitrile (Scheme 48 [524]). The nitrone **15** was prepared from phenylhydroxylamine in DMF (Scheme 48 [16]). The similar decafluorine derivative of the nitrone **15** has not been prepared yet, which has been explained by strong deactivation by fluorine groups of the reaction of the PFBA with the pentafluorophenylhydroxylamine [19].

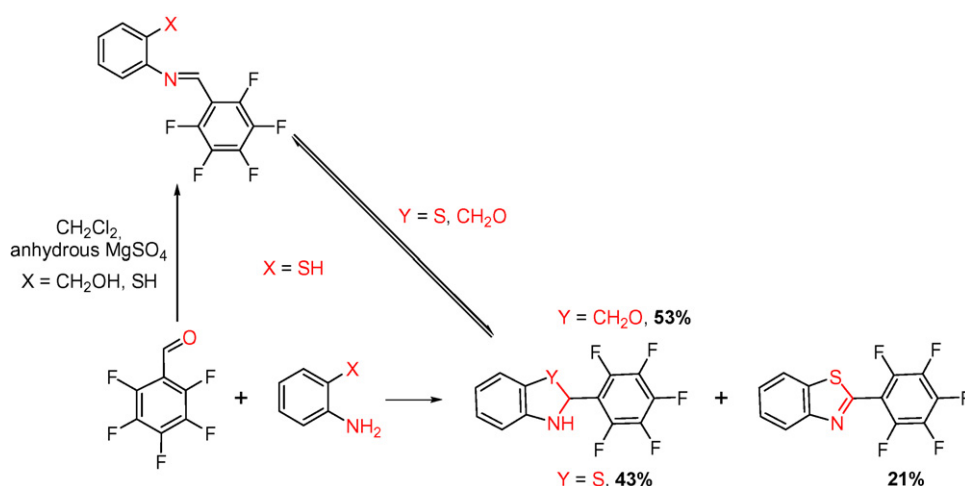
The Schiff bases and derivatized amines, oximes and azines or hydrazines synthesized from the PFBA has been utilized in analytical chemistry [525–535], in the analysis of the polymer

surface [536–558], in the analysis of the water due to the contain of the primary amines [559,560] and the galactose [561]. As well, the Schiff bases are utilized in the synthesis *N*-protected compounds too [562,563].

Utilizing the bifunctional *N*-nucleophiles, additional heterocycle forms on the aldehydic carbon of the pentafluorophenyl group. Konstantinova and Gerasimova [564,565] studied the reaction of the PFBA with *o*-aminobenzenethiole. After the nucleophilic addition of the amino group, a temporary cyclization of the thiole group on the aldehydic group occurs, yielding 43% of 2-dihydrobenzthiazolyl-pentafluorobenzene and 21% of 2-benzthiazolyl-pentafluorobenzene (Scheme 49). The similar synthesis of 2-(2,3,4,5,6-pentafluorophenyl)-1,4-dihydro-2*H*-benzo[*d*][1,3]oxazine and 2-(2,3,4,5,6-pentafluorophenyl)-1,4-dihydro-2*H*-benzo[*d*][1,3]thiazolidine from the 2-aminobenzylalcohol, *o*-aminobenzenethiole and the PFBA is shown below (Scheme 49 [566,567]).

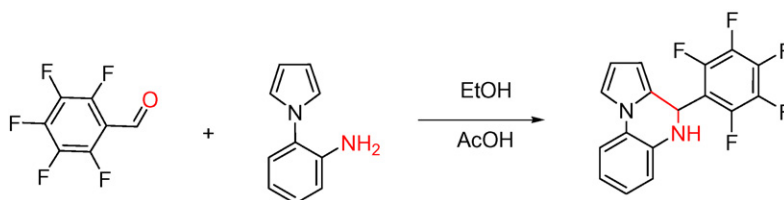
Analogously, the preparation of some 4,5-dihydropyrrolyl[1,2-*a*]quinoxalines, among the other the 4,5-dihydro-4-(pentafluorophenyl)pyrrolyl[1,2-*a*]quinoxaline, has been achieved starting from the PFBA and 1-(2-aminophenyl)-pyrrole (Scheme 50 [568]).

Bedford et al. [569] in 2005 published the reaction of the PFBA with disubstituted hydrazines. An analogous reaction was published by Nyce et al. in 2004 (Scheme 51 [570]).

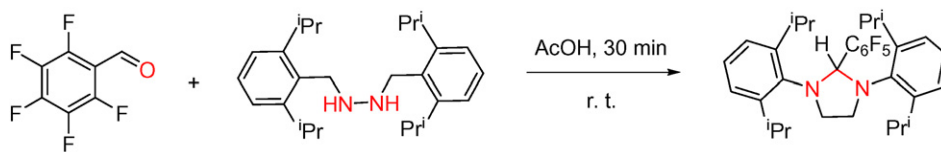


Scheme 49.





Scheme 50.



Scheme 51.

The preparation of isoxazole derivatives **76** from the PFBA in the form of oxime was published (Scheme 52 [571,572]).

The preparation of benzimidazolylidioxides from the PFBA and *O*-benzoquinone dioxime proceeds according to Scheme 53 [573].

Jiang et al. [574] present the utilization the PFBA in 1,3-dipolar cycloaddition, preparing thus 1,2,4-oxadiazole derivatives in yields 42–58% (Scheme 54).

The cycloaddition of the PFBA with a phosphonium ylide in Staudinger reaction conditions affords, after cycloreversion, the Schiff base (Scheme 55 [575]).

The salicylamidophosphites, when treated with the PFBA afford the product with retention of the phosphorinane cycle with high selectivity. Konovalova et al. studied *N,O*-rearrangement during the reaction. The structure of the main diastereoisomer was

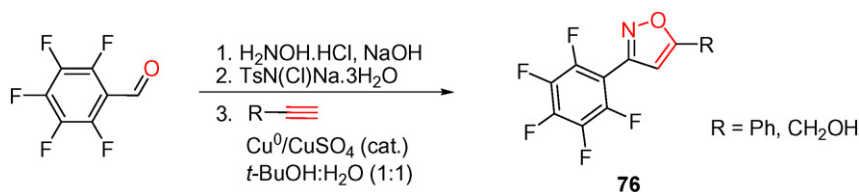
determined by X-ray diffraction. The product hydrolyses very easily to 1-hydroxypentafluorobenzylphosphonic acid (Scheme 56 [576]).

### 3.2.3. Chemical reactions of pentafluorobenzaldehyde with oxygen and sulphur nucleophiles

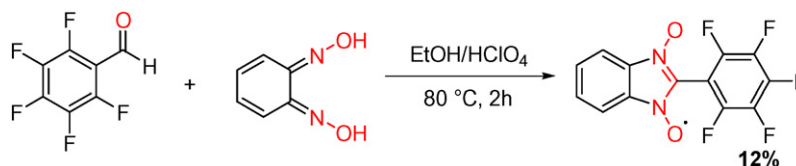
Vysochin et al. heated the pentafluorobenzaldehyde with the triethyl orthoformate in anhydrous ethanol to obtain 87% yield of diethylacetal (Scheme 19 [196]).

The reaction of the PFBA and (*R,R*)-1,2-diphenylethan-1,2-diol affords 1,3-dioxolane and its synthetic utilization is depicted in Scheme 57 [577].

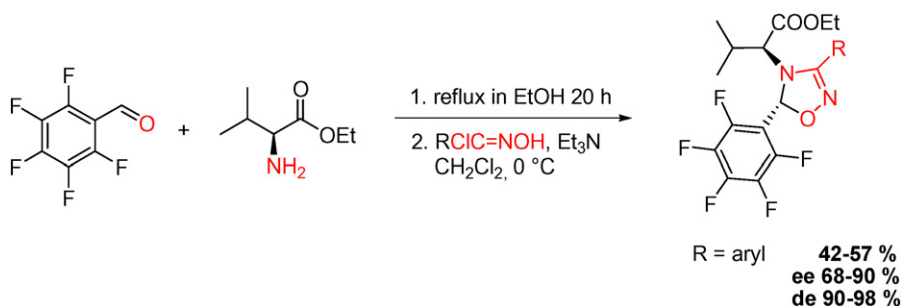
The reaction of the PFBA with bistrifluoromethylnitroxide gives pentafluorobenzoic acid derivative (Scheme 58 [578]).



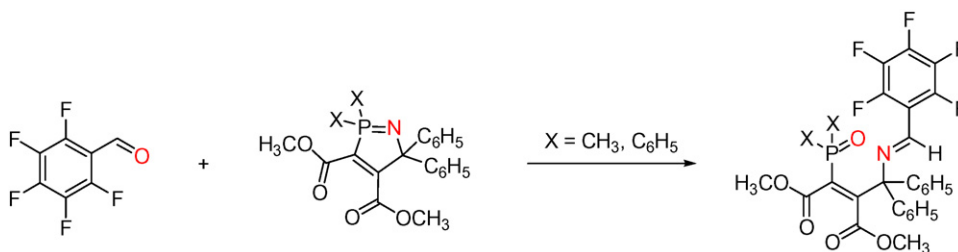
Scheme 52.



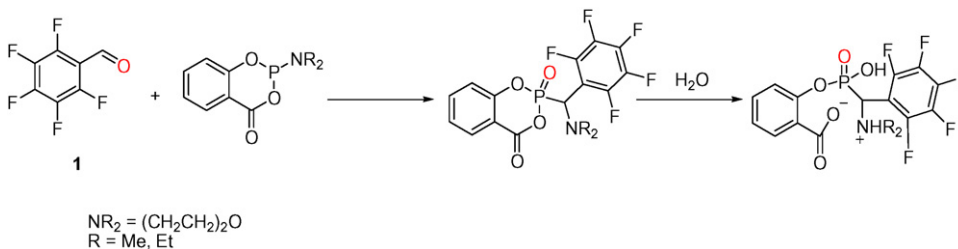
Scheme 53.



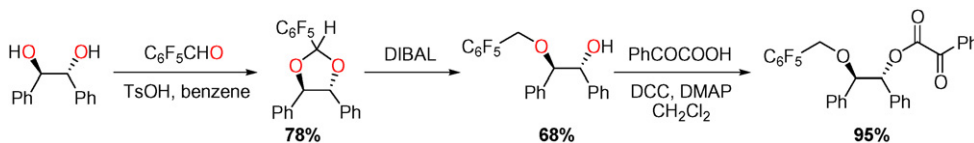
Scheme 54.



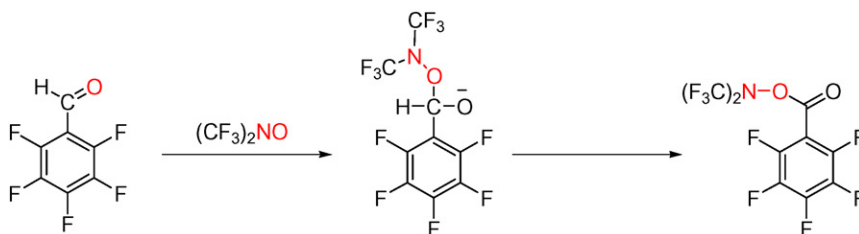
Scheme 55.



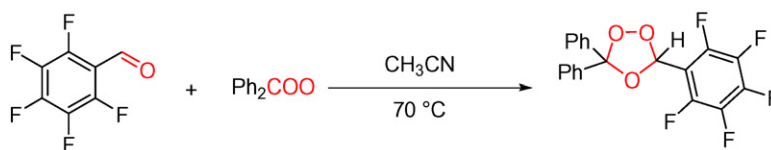
Scheme 56.



Scheme 57.



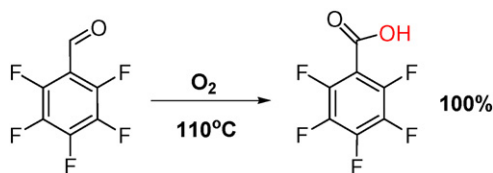
Scheme 58.



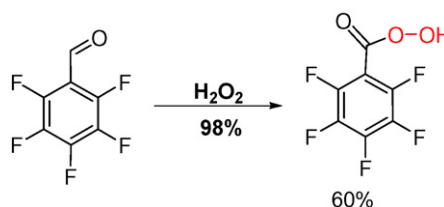
Scheme 59.

The reaction of pentafluorobenzaldehyde with diphenylcarbonyl peroxide is interesting from a preparative point of view (Scheme 59), resulting in five-membered cycle formation containing three atoms of oxygen [579,580].

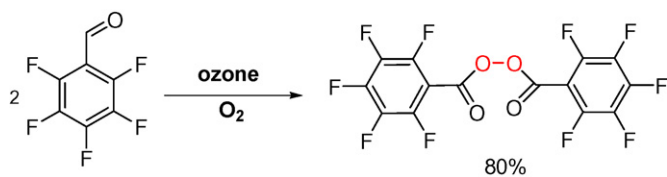
The pentafluorobenzaldehyde is low melting material (m.p. = 20 °C) and it is stable towards to the atmospheric oxygen. It does not form hydrates in water solution. Upon being exposed to pure oxygen at temperature of 110 °C it affords



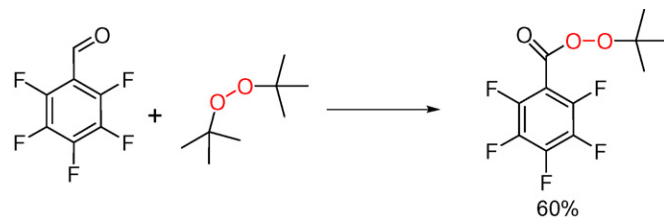
Scheme 60.



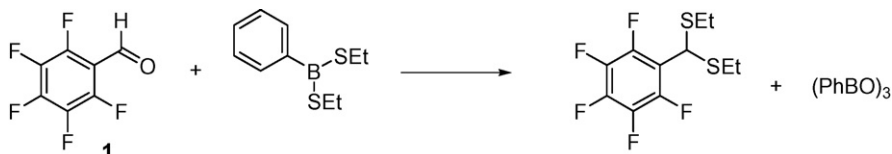
Scheme 61.



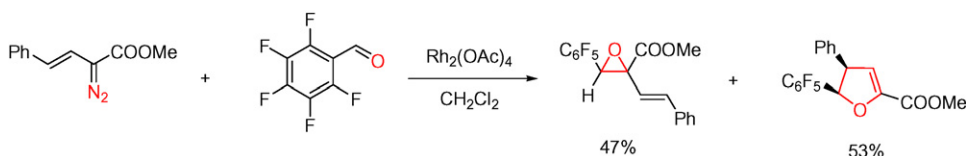
Scheme 62.



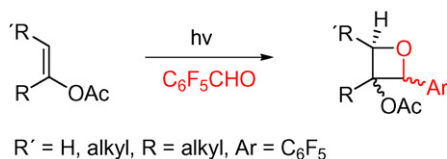
Scheme 63.



Scheme 64.



Scheme 65.



Scheme 66.

pentafluorobenzoic acid in quantitative yield (Scheme 60 [1,581,582]).

Rakhimov et al. [583] and Dzhemilev et al. [584] studied the oxidation of the PFBA with 98% hydrogen peroxide and they gained 60% of pentafluoroperoxybenzoic acid (Scheme 61).

Likewise, Dzhemilev et al. [584] studied the oxidation of the PFBA with ozone in oxygen atmosphere and they observed formation of 80% dipentafluorobenzoyl peroxide (Scheme 62).

Chapurkin [585] studied the reaction of the PFBA with *tert*-butylperoxide in CCl<sub>4</sub>. They isolated *tert*-butyl pentafluoroperoxybenzoate in the yield of 60% (Scheme 63).

The preparation of thioacetals from boric compounds such as PhB(SEt)<sub>2</sub> has been published (Scheme 64 [586]).

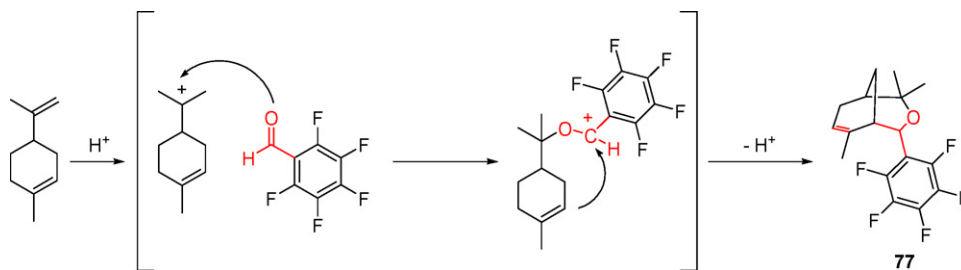
The cyclization reactions with utilizing the PFBA have been published too, the authors introduced the sulphur and phosphorus into the cycle, the reaction with fullerenes has been published [587–590].

### 3.2.4. Other chemical reactions of aldehydic function of pentafluorobenzaldehyde

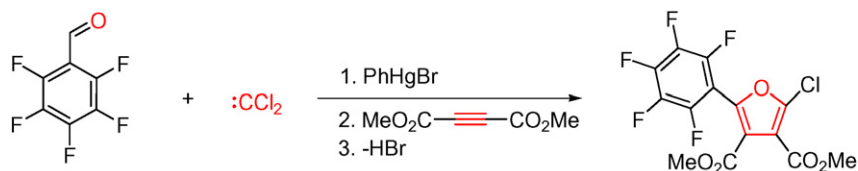
Doyle et al. [591] studied the reaction of the PFBA with methyl styryldiazoacetate catalysed with Rh<sub>2</sub>(OAc)<sub>4</sub> in CH<sub>2</sub>Cl<sub>2</sub>, yielded 47% oxirane and 53% 4,5-dihydrofuran derivative (Scheme 65). Analogous oxirane cycles were described by Filler and Rao [592].

The Paterno–Büchi reaction of enolacetate derivatives with PFBA gives cyclic oxetanes (Scheme 66 [593,594]).

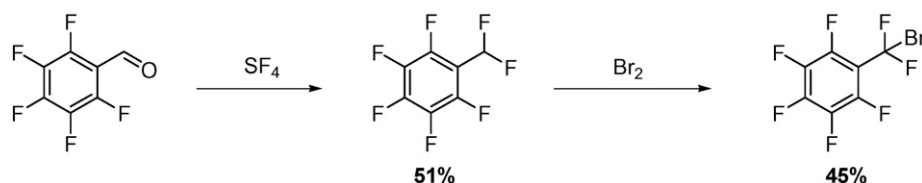
The cyclization of the diene, 1-methyl-4-isopropyl-cyclohexene with the PFBA affords bicyclic ether (77, Scheme 67 [595]). In the first step, addition of a proton leads to the formation of a carbocation which is attacked by the oxygen atom of the



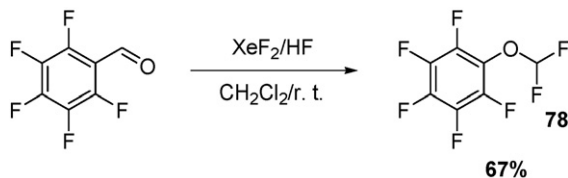
Scheme 67.



Scheme 68.



Scheme 69.



Scheme 70.

aldehydic group, the last step being the rearrangement of the simple bond to form a bicyclic ether [596].

The multi-component reaction of the PFBA with dihalogen carbene and dimethylacetylendicarboxylate affords 2-chloro-3,4-dicarbomethoxy-5-pentafluorophenyl-furan (Scheme 68 [597,598]).

The reaction of PFBA's aldehydic group with sulphur tetrafluoride affords difluoromethylpentafluorobenzene, which is utilized in the synthesis of the heptafluorobenzyl bromide (Scheme 69 [599,600]).

The introducing of the fluorine atom onto aldehydic function was less successful by using of cesium fluoroxysulphate in acetonitrile (yield 13% [601]). Fluorination of the aldehydic carbon yielded the difluoromethoxy derivative **78** (Scheme 70 [602]).

However, the reaction with cobalt trifluoride effects the introduction of fluorine atom into the pentafluorophenyl core to form the simple bonds [603].

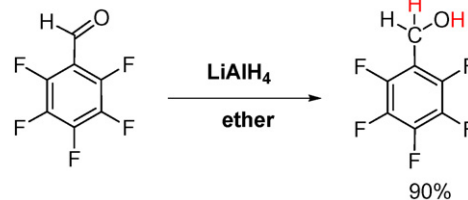
The PFBA can be chlorinated in Friedel–Crafts acylation to the dichloromethane derivative **79** and the chloride of appropriate carboxylic acid **80** (Scheme 71 [604,605]).

The reduction of the pentafluorobenzaldehyde with  $\text{LiAlH}_4$  in dry ether [267] gave pentafluorobenzylalcohol (Scheme 72, yield 90%).

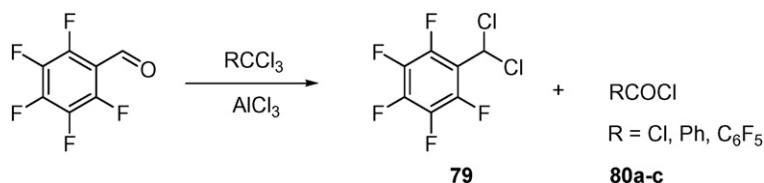
### 3.3. Cyclization reactions of pentafluorobenzaldehyde utilizing the aldehydic group and the atoms of fluorine

The reactivity of five atoms of fluorine in pentafluorobenzaldehyde was studied in the cyclization reaction, in which the aldehydic group is at first mostly condensed with another atom followed by the substitution of the adjacent fluorine. Afterwards, the cyclization can occur. In some cases, the cycle is enlarged [606].

The preparation of the 4,5,6,7-tetrafluorobenzo[*b*]thiophene-2-carboxylic acid [607] from the condensation of the PFBA with rodanine **81** is based on 2,3,4,5,6-pentafluorobenzylidene rodanine forming which affords the cyclization product **82** by utilization of NaOH in water (Scheme 73).



Scheme 72.



Scheme 71.

Petrova et al. [474] studied the cyclization of the 2-benzamido-3-pentafluorophenylacrylic acid derivatives. Upon heating of free acid in DMSO with KF, authors gained 3-benzamido-6,7,8,9-tetrafluorocoumarin (yield 60%). In analogous conditions, the methyl ester derived from that acid afforded 2-phenyl-4-carbomethoxy-6,7,8,9-tetrafluorobenz[*f*]oxazepine (yield 88%, Scheme 74).

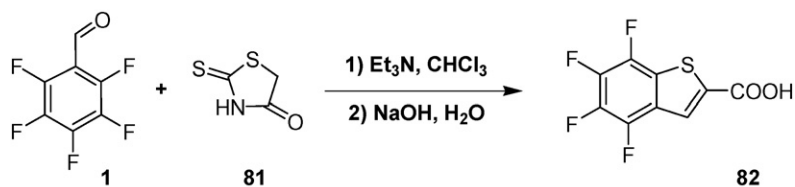
The typical example of cyclization reactions is the preparation of 5,6,7,8-tetrafluoroquinoline derivatives [271], where the first step is the condensation of the PFBA with ketones. The products will be the so-called 2,3,4,5,6-pentafluoro-chalcones, which undergo the cyclization by the action of ammonium acetate and acetic acid. Among introduced quinolines, the by-products, such as 4-pentafluorophenyl-2,6-diphenylpyridine derivatives form too. This is explained by retro–aldol reaction of the chalcones, and the formed ketones with chalcones affording Tschitchibabin reaction to form the pyridine derivatives (Scheme 75).

There were more other preparations of the quinoline derivatives from the PFBA published [134,608–610].

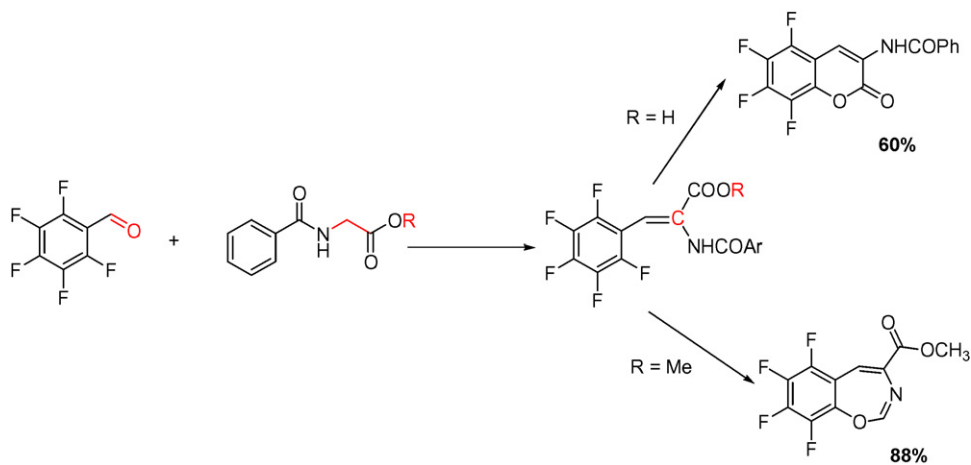
The reaction of the PFBA and two or three equivalents of aniline in 1,2-dichlorobenzene affords the mixture of products: 1,2,3,4-tetrafluoroacridine and its 3-anilinderivative or 7-substituted analogues, which are prepared by the condensation of the PFBA with *para*-substituted aniline derivatives. This condensation products undergo  $S_NAr$  reactions of *ortho*- and *para*-fluorine in the presence of excess arylamine (Scheme 76 [611–617]). The ratio of two forming product (**85**, **86a–g**) changes depended on the substituents and on amount aniline utilized. Here the 1,2,3,4-tetrafluoroacridine can form as a sole product or as a mixture of the products up to 1:1.

However, if the reaction conditions were changed and aniliniummagnesium bromide instead of aniline were to be utilized, tetrafluoroacridine **85** will result again [618].

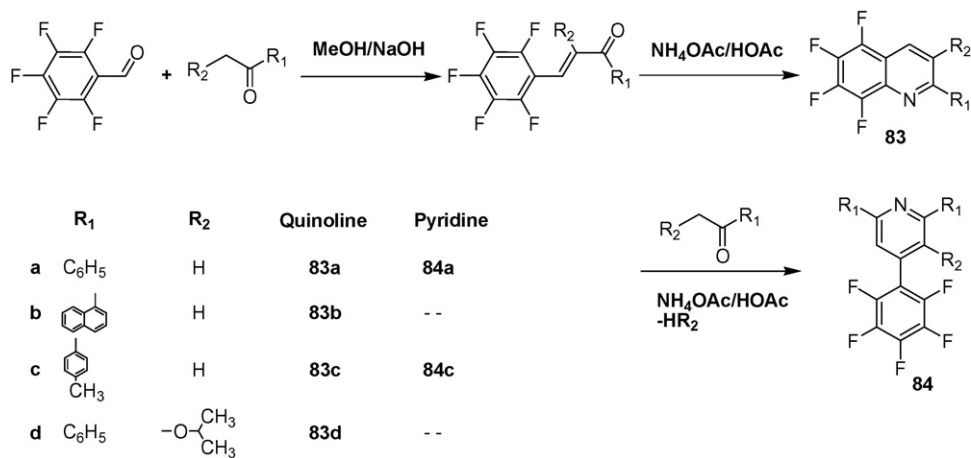
5,6,7,8-Tetrafluoro-1,3-substituted-pyrazole-, isoxazole- and isothiazole-quinoline derivatives [619] are being prepared in the reaction, where the condensation of the aldehydic function and



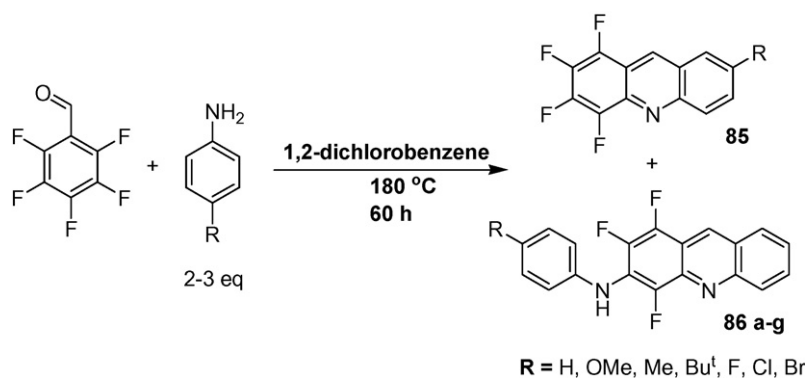
Scheme 73.



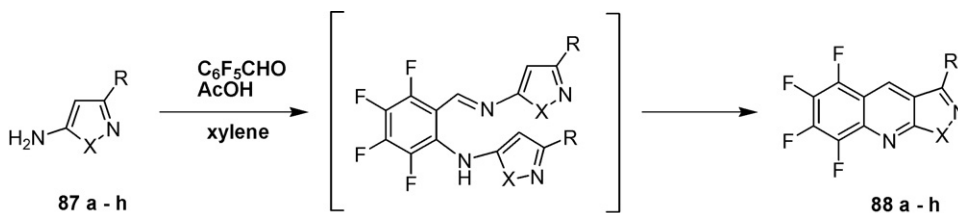
Scheme 74.



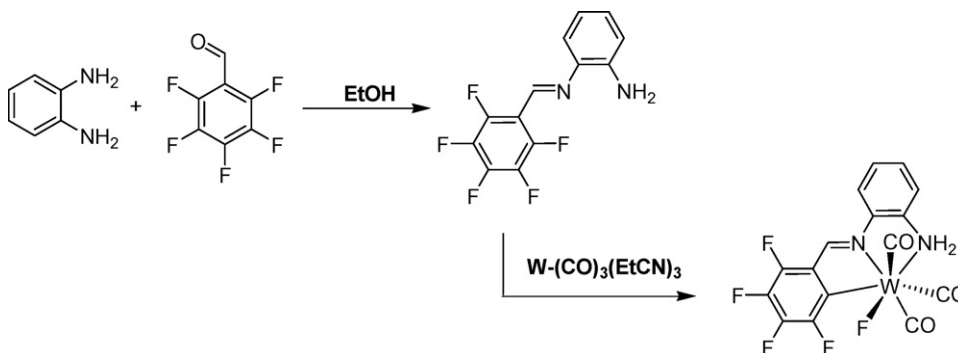
Scheme 75.



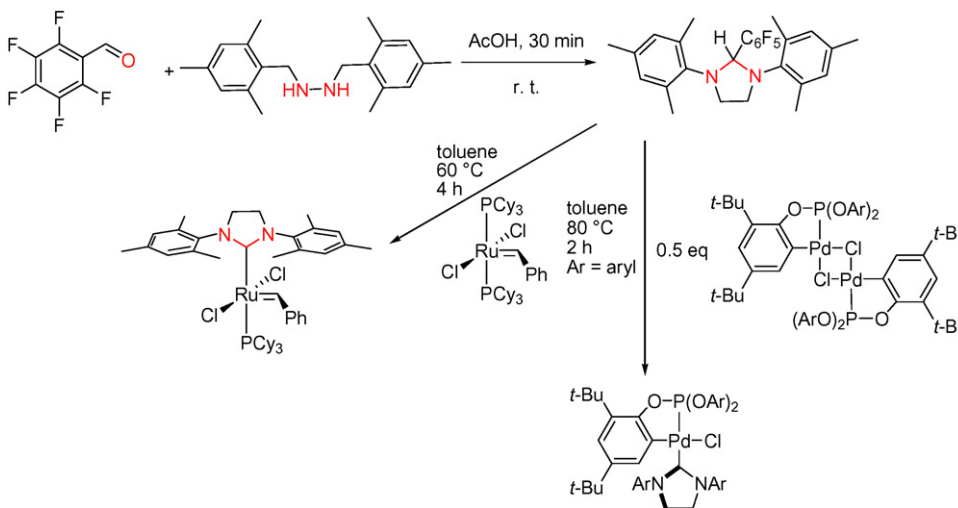
Scheme 76.



Scheme 77.



Scheme 78.



Scheme 79.

the substitution of the adjacent fluorine occur simultaneously. This leads to the cyclization products which are shown in Scheme 77. The yields of the products are shown in Table 2.

The reaction of the PFBA with one equivalent of 1,2-diaminobenzene affords the corresponding Schiff base which

forms the cyclization product in the oxidative addition when treated with  $W(CO)_3(EtCN)_3$  (Scheme 78 [620]).

Other syntheses of the complex compounds utilizing the PFBA were also published (Scheme 79 [569,621–636]). Some of these compounds have catalytic action [632–635].

Table 2

5-Amino-1,2-azole	X	R	Product	Yield
87a	NMe	Me	88a	52%
87b	NMe	<i>t</i> -Bu	88b	74%
87c	NMe	Ph	88c	75%
87d	NPh	Me	88d	80%
87e	NPh	Ph	88e	87%
87f	O	Me	88f	53%
87g	O	Ph	88g	61%
87h	S	Me	88h	58%

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