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The Debenzhydrylation and Transbenzhydrylation Reactions

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The review describes various reactions involving the cleavage of benzhydrylated compounds and their rearrangements and also the use of the benzhydryl group as a protective group. The lability of the bonds between the benzhydryl group and oxygen, sulphur, nitrogen, carbon, and other atoms is demonstrated.
The bibliography includes 484 references.

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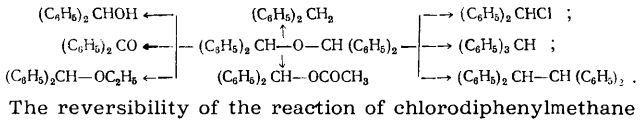
I. INTRODUCTION

There are reviews in the literature on the mobility of the benzyl (phenylmethyl) [1] and trityl (triphenylmethyl) [2] groups. The mobility of the benzhydryl (diphenylmethyl) group, which is of both theoretical and preparative interest, has also been known for a long time. However, until recently there have been no surveys in the literature of data on this problem. In the present review an attempt is made to fill this gap. A particularly large number of studies on the debenzhydrylation and transbenzhydrylation reactions have been published during the last two decades.

II. THE CLEAVAGE OF THE BONDS BETWEEN THE BENZHYDRYL GROUP AND OXYGEN, SULPHUR, AND NITROGEN ATOMS

1. The Bond between the Benzhydryl Group and an Oxygen Atom

As early as the end of the past century, Nef[3] showed that the reaction of dibenzhydryl ether with water gives rise to benzhydrol, the reaction with benzene produces triphenylmethane, the reaction with ethanol gives rise to benzhydryl ethyl ether, and the product of the reaction with acetic acid is benzhydryl acetate. When dibenzhydryl ether reacts with hydrogen chloride, it is converted into chlorodiphenylmethane[4], its reaction with nitrogen dioxide converts it into benzophenone[5], it can be hydrogenated to diphenylmethane[6], and on treatment with potassium butoxide it is converted into 1,1,2,2-tetraphenylethane[7]:



The reversibility of the reaction of chlorodiphenylmethane and its derivatives with ethanol has been demonstrated in a number of studies[4,8-11]. The decomposition of benzhydryl ethers with the following second groups has been described: ethyl[12-15], allyl[16], n-butyl[17], benzyl[18], phenyl[19-22], benzhydryl[7,12,23-26], trityl[18], and others[17,27-29]. The benzhydryl ethers of benzoin[30], hydroquinone[31], and cellulose[32], α-bromo-,[14] 4,4′-dinitro-,[7] and 4,4′-dimethoxy-benzhydryl ethers[15], dixanthydryl ether[33], *O*-benzhydryloximes[34], *O*-benzhydryl-α-aminoperoxides[35], as well as benzophenone ketals[12,36-46] have been decomposed.

Benzhydryl ethers are readily decomposed by liquid potassium–sodium alloy even at room temperature[12]. Shorygin showed[19] that, on treatment with sodium, benzhydryl phenyl ether decomposes with formation of diphenylmethane, *sym*-tetraphenylethane, phenol, and triphenylmethanol. In his survey of extensive researches into the decomposition of ethers by metallic sodium, Shorygin concludes that the cleavage of all the ethers proceeds via one pathway—the oxygen atom always remains bound to the phenyl or tolyl residue, since the benzhydryl, trityl, and naphthylmethyl groups are comparatively weakly bound to oxygen.

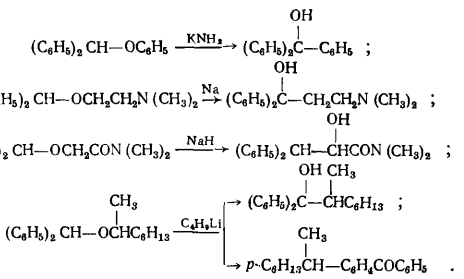
On reaction with hydrogen bromide in acetic acid, benzhydryl ethyl ether is converted to bromodiphenylmethane[5]. On direct chlorination of allyl benzhydryl ether, the addition of chlorine atoms to the double bond of the olefin is also accompanied by the dissociation of the bond between the benzhydryl group and the oxygen atom with formation of chlorodiphenylmethane[16]. Catalytic hydrogenation of benzhydryl benzyl ether in the presence of Raney nickel gives rise to diphenylmethane and benzyl alcohol, while hydrogenation of benzhydryl trityl ether produces triphenylmethane and diphenylmethanol, which is converted into diphenylmethane[18]. In the former case the cleavage takes place at the $O-CH(C_6H_5)_2$ bond, while in the latter case dissociation of the $O-C(C_6H_5)_3$ bond has been postulated. Certain benzhydryl ethers undergo thermal decomposition[15,22,32].

O-Benzhydrylhydroxylamine and its derivatives undergo interesting reactions[47-51]. Their oxidation by lead tetraacetate gives rise to a mixture of substances, including the products of the migration of the benzhydryl group from the oxygen to the nitrogen atom. Treatment of *O*-benzhydryl-*N*-tosylhydroxylamine with sodium hydride or butyl-lithium leads to the formation of the corresponding salts whose pyrolysis gives rise to benzophenone oxime in 100% yield as a result of the loss of the sulphinate group and the migration of the benzhydryl group from the oxygen to the nitrogen atom:

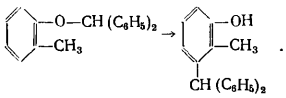
In the presence of a large excess of sodium hydride, diphenylmethanol is formed. Under the same conditions, *O*-benzhydryl-*N*-methyl-*N*-tosylhydroxylamine gives rise to diphenylmethanol, benzophenone, and *N*-methyltoluene-*p*-sulphonamide.

In 1924 Shorygin[52,53] discovered the carbinol rearrangement. He showed that, on heating with sodium wire in a sealed tube, benzyl phenyl ether produces diphenylmethanol.

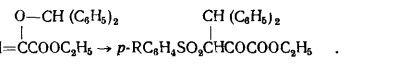
Subsequently Wittig and Lohmann[36,54] observed an analogous rearrangement of benzhydryl methyl ether, which isomerises to diphenylmethylmethanol on treatment with phenyl-lithium. A rearrangement occurring when the dimethylketal of benzophenone is treated with powdered sodium has been described by Schlenk and Bergmann[36] and was subsequently confirmed by Wittig ahd Happe[36] [Ref.20? (Ed.of Translation)]. An analogous rearrangement involving these and other benzhydryl ethers was achieved still later with the aid of metallic sodium[55], sodium hydride[56], potassium hydroxide[7,30], potassium amide[57], butyl-lithium[58], and lithium dimethylamide[59]. The reaction proceeds in accordance with the following scheme:



It has been found[60] that the benzhydryl ether of *o*-cresol undergoes a rearrangement with formation of 3-benzhydryl-2-methylphenol on heating for 5 h at 150 °C in the presence of zinc chloride:

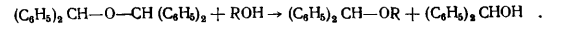


It has been observed quite recently[61] that the ethyl esters of β-arenesulphonyl-α-diphenylmethoxyacrylic acids rearrange quantitatively in the temperature range from 160° to 190 °C to the ethyl esters of β-arenesulphonyl-β-diphenylmethylpyruvic acids:



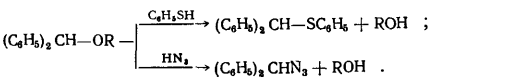
According to the authors, the *C*-alkylation products are apparently formed as a result of suprafacial [1,3]-sigmatropic rearrangement of the benzhydryl group.

The reactions of dibenzhydryl ether with butyl and benzyl alcohols proceed with displacement of one benzhydryl group[62]:



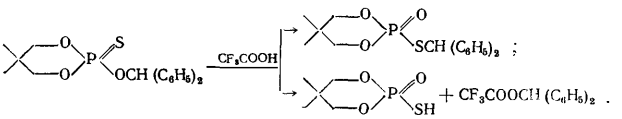
When butyl alcohol reacts with benzhydryl trityl ether, butyl trityl ether is formed together with benzhydryl butyl ether[62]. Yet another instance of the replacement of the benzhydryl group in ethers by the methyl group has been described recently[63]. The yields of the products of all these reactions are high.

It has been shown[28,64] that, on reaction with thiophenol and hydrazoic acid in the presence of trifluoroacetic acid, alkyl benzhydryl ethers are quantitatively converted into the corresponding sulphide and azide:



As early as 1865, Linnemann[65] pointed out the ready decomposition of the benzhydryl esters of acetic, benzoic, and succinic acids. According to Cheeseman's data[5], benzhydryl nitrate decomposes even at room temperature with formation of dibenzhydryl ether and benzophenone.

The hydrolysis of benzhydryl nitrate[5,66], benzhydryl formate[67,68], benzhydryl acetate[67-71], and benzhydryl benzoate[68,71], the ethanolysis of the last compound[72,73], the pyrolysis of the benzhydryl esters of nitric[5,74] and acetic[75,76] acids, the reduction of benzhydryl acetate[77] and benzhydryl benzoate[78], and the cleavage of other benzhydryl esters[79-85] have been described. Many studies have been devoted to the investigation of the kinetics and mechanisms of the above processes[66-73,81]. Thiocarbonates[86,87] and xanthates containing the benzhydryl group[88-90] have also been decomposed. The debenzhydrylation and transbenzhydrylation reactions of benzhydryl esters containing phosphorus[91-95], germanium[96], and silicon[96-100] atoms are of interest. For example[95]:



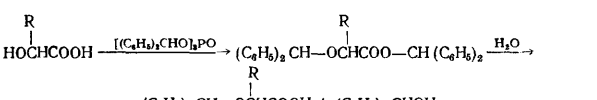
Chlorodiphenylmethane has been obtained in 90% yield by the reaction of benzhydryl acetate with acetyl chloride[77]. French workers[101,102] showed that, when benzhydryl acetate and benzoate are used in the Reformatskii reaction, the latter proceeds anomalously with formation of diphenylmethane and 1,1,2,2-tetraphenylethane.

Benzhydryl nitrate has been used successfully as the benzhydrylating agent for ammonia, hydrozine, amines, the amides of carboxylic and sulphonic acids, alcohols, mercaptans, urethanes, heterocyclic bases, and other compounds[5]. The kinetics of the benzhydrylation of aromatic hydrocarbons[103] and anisole[104,105] by benzhydryl acetate have been investigated:



Benzhydryl benzoate can be used for the benzhydrylation of thiophenol[64].

Methods for the synthesis of the benzhydryl esters of phosphoric acid and their employment for phosphorylation and the removal of protective groups have been investigated[106-109]. A new method of synthesis of benzhydryl esters based on the reactions of alcohols and carboxylic acids with tribenzhydryl phosphate was published in 1978.[110,111] In the reactions with hydroxyacids two benzhydryl groups are introduced one of which is removed more readily:



The benzhydryl protective group has found applications in the synthesis and the investigation of the properties and structures of nucleosides, nucleotides, and oligonucleotides[106,109,112,113]. In the study of the comparative stabilities under various conditions of the benzhydryl and trityl esters of uridine 3'-phosphate, it was shown that the trityl ester is more resistant to hydrolysis in 80% acetic acid and 50% formic acid than the benzhydryl ester[109].

The protection of the phenolic hydroxyl by the benzhydryl group has found applications in the synthesis of polyesters (including macrocyclic polyesters) containing two dinaphthyl residues[114,115]. The benzhydryl protective group has been used to obtain the esters of phenols and depsides[116-118]. Here one should mention also the protection of phenols and catechols by the diphenylmethylene group[118-126].

The benzhydryl group has found extensive applications for the protection of the carboxy-group in the synthesis and the investigation of the properties and structures of aliphatic

and aromatic carboxylic acids containing from several to several tens of carbon atoms[27,116-118,127-134]. Catalytic hydrogenation is most often employed for the removal of the protective group[27,116,118,127-131,133,134]. The catalyst employed is usually palladium. Furthermore, the benzhydryl protective group can be removed with the aid of trifluoroacetic[128,132] and 80% acetic[117] acids, a saturated solution of hydrogen chloride in acetic acid[132], hydrogen chloride or bromide in nitromethane[133], and basic reagents in ethanol and dioxane[132].

Khorlin and Privalova[135] were the first to describe the protection by the benzhydryl group of the carboxy-group in the most common representative of sialic acids—*N*-acetylneuraminic acid. This protective group and the methods for its removal have been investigated also for various reactions of other carbohydrate derivatives[80,128,131,136-138]. The selective hydrolysis of the protective group has been indicated[80].

The benzhydryl group has found extensive applications for the protection of the carboxy-group in the synthesis and investigation of β-lactam antibiotics, including nocardicin, penicillin, and particularly cephalosporin[134,139-216]. The benzhydryl protective group has been used also in the synthesis of other antibiotics[217] and analogues of the anti-inflammatory agent indomethacin[218] and derivatives of benzofuranone[219]. 4-Chloro-,[106] 2-nitro,[220] and 4-nitrobenzhydryl protective groups[221] have been described in the literature.

The instability of the benzhydryl esters of organic sulphur-containing acids has been pointed out in a number of studies[222-233]. Thus the benzhydryl ester of trichloromethanesulphenic acid rearranges to the corresponding sulphoxide on refluxing in hexane, while in polar solvents it undergoes thermal decomposition to chlorodiphenylmethane and dichlorosulphine[222]. The rearrangements of the benzhydryl esters of aromatic sulphenic[225] and sulphinic[226-228] acids to the corresponding sulphoxides and sulphones, which take place with high yields, have been described:

$$\mathrm{ArS{-}O{-}CH(C_6H_5)_2 \rightarrow ArS(O){-}CH(C_6H_5)_2}\ ;$$

$$\mathrm{ArS(O){-}O{-}CH(C_6H_5)_2 \rightarrow ArS(O)_2{-}CH(C_6H_5)_2}\ .$$

A sulphone has been obtained instead of the ester on treatment of the sodium salt of toluene-*p*-sulphinic acid with benzhydryl nitrate[5].

When toluene-*p*-sulphonyl chloride is treated with diphenylmethanol, the product is dibenzhydryl ether (yield 90 to 100%) and not the expected benzhydryl ester of the sulphonic acid[223,224]. Diphenylmethane, diphenylmethanol, and benzophenone are formed on thermal decomposition of benzhydryl toluene-*p*-sulphonate[229-231]. If the latter is refluxed with benzamide in glacial acetic acid for 30 min, transbenzhydrylation takes place with formation of *N*-benzhydrylbenzamide[228]:

$$p\text{-}CH_3C_6H_4SO_3{-}CH(C_3H_5)_2 + C_6H_5CONH_2 \rightarrow C_6H_5CONH{-}CH(C_6H_5)_2 + p\text{-}CH_3C_6H_4SO_3H\ .$$

The reaction proceeds analogously also with propionamide[228]. The benzhydryl ester of toluene-*p*-sulphonic acid has likewise been used for the benzhydrylation of phenylacetylene[231].

2. The Bond between the Benzhydryl Group and a Sulphur Atom

The decomposition of benzhydryl sulphides with following second organic groups has been described: *N*,*N*-dimethylaminoethyl[55], benzyl[234,235], phenyl[228,236-238], benzhydryl[24,239,240], and others[235,241,242]. Di(benzhydrylthio)acetylene[243] and dibenzhydryl sulphide[244,245] have been decomposed. The oxidation of benzhydrylmethanesulphonyl sulphide by chlorine in an aqueous medium takes place in accordance with the following mechanism[246]:

$$(C_6H_5)_2CH{-}SCH_2SO_2CH_3 \xrightarrow{Cl_2} (C_6H_5)_2CHCl + ClSO_2CCl_2SO_2CH_3\ .$$

When an attempt was made at the α-chlorination of benzhydryl benzyl sulphoxide by sulphuryl chloride, only the decomposition products were isolated: chlorodiphenylmethane and benzylsulphinic acid[247]. 1,1,2,2-Tetraphenylethane, dibenzhydryl ether, and debenzhydrylation products were detected among the products of the thermal decomposition of benzhydryl methyl and benzhydryl *p*-tolyl sulphoxides[233]. The following rearrangement has been described:

$$p\text{-}CH_3C_6H_4S(O){-}CH(C_6H_5)_2 \rightleftarrows p\text{-}CH_3C_6H_4S{-}O{-}CH(C_6H_5)_2\ .$$

Benzhydryl sulphides have been used to synthesise asymmetric disulphides. The *S*-benzhydryl protective group can then be removed with the aid of sulphenyl thiocyanates or thiocyanogen without affecting the disulphide bond[126,234,248,249]. Certain benzhydryl sulphides undergo very interesting reactions[126,234,248]. For example:

$$\begin{array}{c} (C_6H_5S{-}SCH_2CH_2CONHCH_2CH_2S{-})_2 \\ \uparrow S(CN)_2 \\ C_6H_5S{-}SCH_2CH_2CONHCH_2CH_2S{-}CH(C_6H_5)_2\ ; \\ \downarrow S(CN)_2 \\ C_6H_5S{-}CH(C_6H_5)_2 \end{array}$$

$$(C_6H_5)_2CH{-}SCHCH_2COOH \rightarrow C_6H_5S{-}SCHCH_2COOH\ .$$

The thermal decomposition of benzyl diphenylmethanethiosulphonate yields a mixture of products[250]:

$$\begin{array}{ccccc} (C_6H_5)_2CH{-}SCH_2C_6H_5 & & SO_2 & & (C_6H_5)_2CH{-}CH(C_6H_5)_2 \\ \uparrow & & \uparrow & & \uparrow \\ & & \multicolumn{3}{c}{\rule{4em}{0.4pt}} & \\ & & \downarrow & & \\ (C_6H_5)_2CH_2 & \leftarrow & (C_6H_5)_2CH{-}SO_2SCH_2C_6H_5 & \rightarrow & (C_6H_5)_2CS \end{array}\ .$$

This can apparently explain the failure of the attempt to oxidise the above thiocyanate to the corresponding disulphone[251].

When di-*p*-methoxybenzhydryl *p*-tolyl sulphone is heated with ethanol in the presence of potassium hydroxide, the alcohol undergoes di-*p*-methoxybenzhydrylation with formation of the corresponding ether[15].

Benzophenone thioketals have also been subjected to decomposition[36,38,44,45,252,253].

3. The Bond between the Benzhydryl Group and a Nitrogen Atom

The decomposition of dibenzhydrylamine in the presence of alkali metal amides in liquid ammonia[254,255] and by photolysis[24,256] has been described. One of the products of the reduction of benzhydryl(benzyl)methyl amine[257,258], benzhydryl(methyl)-*p*-phenylbenzylamine[259], and other *N*-benzhydrylated compounds[260] is diphenylmethane. *sym*-Tetraphenylethane has been obtained from *N*,*N*-dibenzhydrylhydrazine[5]. The methylation of *N*-benzhydryl-*N*′,*N*′-dimethylethylenediamine by formaldehyde takes place with the removal of the benzhydryl group and the formation of the tetramethyl derivative of ethylenediamine[261]. The cleavage of benzhydrylguanidines in 80% sulphuric acid has been indicated[262]. *N*-Benzhydryl derivatives of acetamide[263], hydroxylamine[34,43,264-267], nitrosohydroxylamine[268], benzylideneamine[269], oxides[270,271], urethanes[272], *N*-substituted ammonium salts[273,274], Schiff bases[268,275-277], azo-compounds[278-283], azides[268], and other nitrogen-containing compounds[284] have also been subjected to decomposition.

An instance of the simultaneous elimination of the *O*-benzyl and *N*-benzhydryl groups is known[260].

When chlorodiphenylmethane is heated with aminoalcohols (for example with *N*,*N*-di-isopropylethanolamine), the hydrochlorides of the corresponding aminoethers are formed[285]. A mechanism has been proposed for this reaction, according to which the N atom is initially alkylated with formation of an ammonium salt; the subsequent N–O transbenzhydrylation leads to the formation of sterically hindered benzhydryl aminoethers[285]. The possibility of this mechanism has been confirmed by other studies[286,287], which showed that at 175 °C benzhydryl(β-hydroxyethyl)dimethylammonium chloride undergoes a thermal rearrangement via a similar mechanism:

$$(CH_3)_2\overset{+}{N}(Cl^-)CH_2CH_2OH \rightarrow (CH_3)_2NHCH_2CH_2O{-}CH(C_6H_5)_2 \quad .$$
$$\qquad\qquad\qquad\qquad\quad |$$
$$\qquad\qquad\qquad\qquad\quad CH(C_6H_5)_2$$

The dimethylaminoalkyl ethers of diphenylmethanol are also formed on rearrangement under these conditions of other compounds of the type $[Ar_2CH\overset{+}{N}(CH_3)_2(CH_2)_nOH]Cl^-$, where $n = 2–7$. Diphenylmethanol, chlorodiphenylmethane, *sym*-tetraphenylethane, and dibenzhydryl ether have been detected as side products in this process[286,287].

There are indications in the literature of the possibility of the rearrangement of benzophenone *N*-benzyloxime to *N*-benzhydrylbenzaldoxime[34,288]:

$$(C_6H_5)_2C{=}\overset{+}{N}(O^-){-}CH_2C_6H_5 \rightarrow C_6H_5CH{=}\overset{+}{N}(O^-){-}CH(C_6H_5)_2 \quad .$$

When α,α-diaryl-*N*-benzhydrylnitrones, i.e. the *N*-benzhydryloximes of benzophenone and its derivatives, are heated to 200 °C, the corresponding isomeric *O*-benzhydryloximes are formed[34,50,264,270,271,288–298]:

$$(C_6H_5)_2C{=}\overset{+}{N}(O^-){-}CH(C_6H_5)_2 \rightarrow (C_6H_5)_2C{=}N{-}O{-}CH(C_6H_5)_2 \quad .$$

The kinetics and mechanism of this thermal rearrangement as well as the decomposition of *N*-benzhydryloximes have been widely investigated[34,264,270,271,288–297].

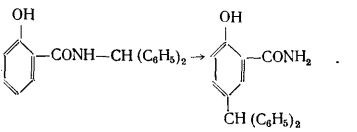
The decomposition of benzophenone Schiff bases[266,268,275,298–308], oximes[43,267,309–317], and hydrazones[139,279,318–327] has been described. The oximes have found applications in the synthesis of cycloserine (with removal of the *N*-protective group),[309–311] while the hydrazones have been used as benzhydrylating agents for carboxylic acids[319] and chlorobenzotriazole[326].

On reaction with benzylamine, the acyl derivatives of benzophenone imine give rise to the usual products of the addition of the azomethine group or are cleaved to the amides of the corresponding acids and benzophenone *N*-benzylimine[308]:

$$(C_6H_5)_2C{=}NCOR + C_6H_5CH_2NH_2 \xrightarrow{20°} [(C_6H_5)_2C(NHCH_2C_6H_5)NHCOR] \rightarrow$$
$$\rightarrow RCONH_2 + (C_6H_5)_2C{=}NCH_2C_6H_5 \quad .$$

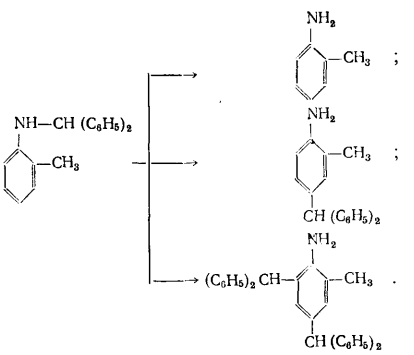
It is extremely likely that in the latter case the *N*-acylimines initially also give rise to addition products, but the latter are unstable and decompose rapidly. In one case the formation of the unstable addition products was demonstrated experimentally. In the reaction of benzylamine with benzophenone *N*-chloroacetylimine in the cold, it was possible to isolate an unstable addition product, which decomposed fully in the course of several days at a temperature as low as 20 °C.[308]

When *N*-benzhydrylsalicylamide is heated with toluene-*p*-sulphonic acid in acetic acid, the benzhydryl group migrates from the nitrogen atom to the carbon atom and 5-benzhydryl-2-hydroxybenzamide is formed[228]:



The same *N*-benzhydryl derivatives of salicylamide undergo debenzhydrylation and not hydrolysis on being refluxed for 3 h with concentrated hydrochloric acid in glacial acetic acid[328].

The lability of the bond between the benzhydryl group and the nitrogen atom in the *N*-benzhydryl derivatives of aromatic amines has been investigated[329–333]. *sym*-Tetraphenylethane was detected among the decomposition products obtained on distilling *N*-benzhydryl-*N'*,*N'*-dimethyl-*N*-phenylethylenediamine[330]. On heating to 150 °C, the hydrochloride of *N*-benzhydrylaniline gives rise to an isomerisation product (*p*-benzhydrylaniline) and also the *N*,*C*-dibenzhydryl derivative of aniline and tetraphenylethylene[331]. When *N*-benzhydryl-*o*-toluidine is heated in a mixture of acetic and hydrochloric acids, the debenzhydrylation and transbenzhydrylation reactions proceed in accordance with the following scheme[332]:

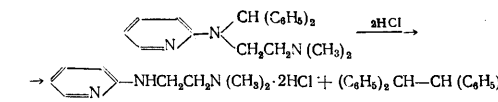


If the reaction is carried out in the presence of *o*-cresol, the *C*-benzhydrylation product of the latter is also formed[332]. It is striking that, on heating to 145 °C with hydrochloric acid, *o*-benzhydrylphenylaniline produces 1-phenylfluorene and aniline[334].

Since the alkylation of 6-azauracil takes place preferentially at the imide group, the latter must be protected in the synthesis of 6-aza-analogues of uridine and related compounds. The benzhydryl group has been used for this purpose. 1-β-D-Ribofuranosyl-6-azauracil[112], 6-azauridine 5'-phosphate[113], and their 5-methyl-derivatives have been synthesised in this way. The *N*-benzhydryl protective group has found applications in the synthesis and reactions of organophosphorus analogues of biologically active compounds[277], β-lactams[335], 1,2-dihydropyridine[336], and 9-amino-9*H*-purines[337].

The debenzhydrylation reaction takes place on hydrogenation of derivatives of *N*-benzhydrylazetidine[338–342]. Protection by the *N*-benzhydryl group has also been described for derivatives of 1,2-diazetidine and the possibilities for its removal have been investigated[343–345]. The possibility of the migration of the benzhydryl group in the benzhydryl derivatives of 2-aminopyridine has been assumed[346]. On distillation and on treatment with dry hydrogen chloride, *N*-benzhydryl-*N'*,*N'*-dimethyl-*N*-(2-pyridyl)ethylenediamine

decomposes[330]:



The same derivative of 2-aminopyridine could not be obtained by the reaction of 2-benzhydrylaminopyridine with β-chloroethyldimethylamine, since here too the debenzhydrylation reaction takes place[347]. Because of this, the attempts to synthesise 2-benzhydrylaminomethylimidazoline were also unsuccessful[261]. The hydrogenolysis of the 1-methyl- and 1-benzyl-derivatives of 4-benzhydrylpiperazine has been investigated[348]. When (pyrazinylmethyl)dimethylmethanol is refluxed in ethanol in the presence of potassium hydroxide, it undergoes reductive decomposition with formation of methylpyrazine and diphenylmethanol[349]. The hydrolysis of benzotriazolylchlorodiphenylmethane leads to the formation of benzotriazole and benzophenone, while treatment with methanol leads to benzotriazole and the dimethylacetal of benzophenone[326].

The *N*-diphenylacetyl protective group has been investigated[350–353].

III. THE BENZHYDRYL PROTECTIVE GROUP IN THE SYNTHESIS AND REACTIONS OF AMINOACIDS AND PEPTIDES

The benzhydryl and diphenylmethylene protective groups have found applications in the synthesis and study of the properties and structures of aminoacids and peptides[111,125,126,129,130,132,133,219,234,238,248,249,301,305,317,354–405]. Mention must be made of derivatives of proline[111,129,354,355], histidine[356], hydroxyaminoacids[219,357], tyrosine[357], α-vinyl-α-aminoacids[125], sulphur-containing[132,234,238,248,249,348–382] and selenium-containing[383–386] aminoacids and peptides, glucopeptides[387,388], and individual fragments of the insulin molecule[375,377–380,389].

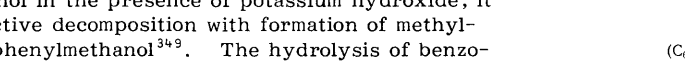
The benzhydryl group has been usually employed to protect the carboxy-group[111,126,129,130,132,133,219,383–388,390–396]. The *O*-benzhydryl[357], *N*-benzhydryl[301,356], *S*-benzhydryl[234,249,358,360,361,363–382], and *Se*-benzhydryl[384,386] protective groups have been described. We may also mention *O*-diphenylmethylene[125], *N*-benzhydryloxycarbonyl[132,359,361,362], *S*-benzhydryloxycarbonyl[132], *N*-4,4′-dimethoxybenzhydryl[364], *S*-4,4′-dimethoxybenzhydryl[364,368], *N*- and *S*-3,3′,4,4′-tetramethoxybenzhydryl[364], *N*-diphenylacetyl[353], *N*-benzhydrylacrylamide[397], *S*-α-methylbenzhydryl[369], *S*-α,4,4′-trimethylbenzhydryl[369], and other *S*-protective groups for cysteine[369]. The benzhydryl[376], 4,4′-dimethoxybenzhydryl[389,398–403], and xanthyl[404,405] groups have been used to protect the amide group in asparagine, glutamine, and in the amides of other α-aminoacids. The oxime of the *p*-polymer-substituted benzophenone has been proposed quite recently as a protective group in the synthesis of peptides[317].

One of the methods for the removal of the benzhydryl protective group is catalytic hydrogenolysis[129,130,354,355]. In the synthesis of carcinolytic peptides, the complete removal of the benzhydryl group is virtually achieved with the aid of 98% formic acid at room temperature (3 h).[392] Under mild conditions, the benzhydryl protective group can be eliminated with anhydrous hydrofluoric acid[126,376].

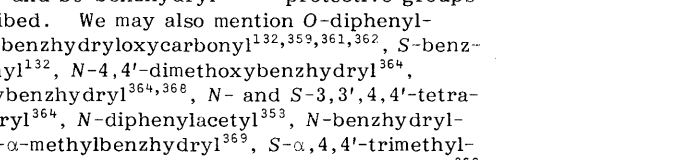
For the synthesis of asymmetric (relative to the cysteine residues) cysteine peptides, it is essential that the sulphhydryl groups should be substituted by different protective groups to permit their separate elimination[359,363]. *S*-Trityl-cysteine and *S*-benzhydrylcysteine have been proposed for this purpose. Conditions have been found for the cleavage of *S*-trityl sulphides with retention of the *S*-benzhydryl group[248,358,359,363,372–374]. The *S*-trityl protective group can be readily eliminated at room temperature by treatment with heavy metal salts. The *S*-benzhydryl group is not then affected and can be removed by treatment with trifluoroacetic acid. It has been reported[238] that *S*-trityl sulphides are cleaved by an alcoholic solution of iodine at room temperature, while their *S*-benzhydryl analogues are stable even on boiling.

IV. THE CLEAVAGE OF THE BOND BETWEEN THE BENZHYDRYL GROUP AND A CARBON ATOM

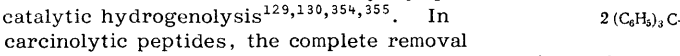
The thermal decomposition of benzopinacol was described as early as 1877:[406]



Phenyltolylpinacol behaves analogously, decomposing at the instant of fusion to phenyl tolyl ketone and phenyltolylmethanol[406]. The conversion of dinitrosotetraphenylethane into benzophenone oxime was later described[407]. Many studies devoted to the cleavage of benzopinacol[14,408–415], its derivatives and analogues[14,20,21,57,411,416,417], phenylbenzoin[39,418–421], α-*C*-substituted derivatives of diphenylmethanol[349,422–425], triphenylethanol[426–430], tetraphenylethanol[7,408,431,432], and benzhydryl phenyl ketone[429,430,432–434] were published subsequently. Less attention has been devoted to DDT,[435] 1,1-diphenylethanediol[436], benzoyldiphenylethanediol[437], benzoyltriphenylethanol[438], 1,1,2-triphenyl-1-propanol[410], 1,1,2-triphenyl-1-butanol[410], 1,1-diphenyl-4-pent-1-enol[439], 1,1,4,4-tetraphenylbuta-1,2-dien-4-ol [40] and its 3-substituted derivatives[441], hexaphenylallene glycol[440], benzhydryl methyl ketone[279], benzhydryl benzyl ketone[442], *p*-anisyl benzhydryl ketone[429], dibenzhydryl ketone[30], benzhydryl α-hydroxybenzhydryl ketone[30], benzoyldiphenylaminomethane[39], 1,2,2-triphenylaminomethane[428], triphenylaminoethanol[254], triphenylacetamide[443], 2-diphenylacetyl-1-methylbenzimidazole[444], and other *S*-benzhydrylated compounds[39,84,85,417,425,445–449]. The products of the cleavage of the above compounds are as a rule diphenylmethane, diphenylmethanol, and diphenyl ketone:



In an early investigation Chichibabin showed[450,451] that pentaphenylethane decomposes at an elevated temperature at the C–C bond of ethane, like hexaphenylethane. In this case the pentaphenylethane molecule decomposes into radicals, which then recombine differently:

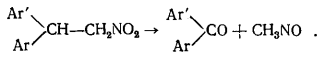


According to the observations of Cone and Robinson[452], treatment of this compound with phosphorus pentachloride yields chlorotriphenylmethane. The instability of the central C–C bond in pentaphenylethane was subsequently investigated by other workers[12,453–461]. The dissociation of the carbon–carbon bond in *sym*-tetraphenylethane[12,455,462–464] and *p*-benzhydrylphenyltriphenylmethane[36,465] was indicated.

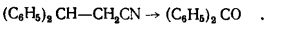
The photolysis of 1,1,2,2-tetraphenylethane, 1,1,3,3-tetraphenylpropane, 1,1,4,4-tetraphenylbutane, and 1,1,5,5-tetraphenylpentane has been described[24,466,467]. The oxidation of 1,1-diphenylethane, 1,1-diphenylpropane,

1,1-diphenylbutane, 1,1-diphenylpentane, and 1,1,diphenylhexane by oxygen has been investigated[468,469]. It has been shown that tertiary carbon atoms are acted upon by oxygen with formation of hydroperoxides, whose decomposition leads to the formation of benzophenone, its oxidation products, and tarry substances. The product of the electrochemical oxidation of diphenylacetic acid in water is diphenylmethanol, while the same reaction in acetonitrile gives rise to a mixture of diphenylmethane, tetraphenylethane, and the benzhydryl ether of the starting material[470,471].

Attention has been drawn[472] to the anomalous course of the Nef reaction with salts of 1,1-diaryl-2-nitroethanes, in which diaryl ketones are obtained instead of the corresponding diarylacetaldehydes:

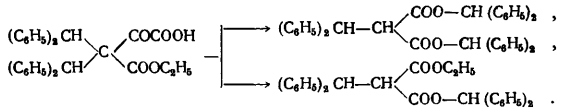


The oxidative hydrolysis of 3,3-diphenylpropionitrile, one of the products of which is diphenyl ketone (yield 22%), has been described quite recently[473]:



Diphenylmethane (yield 49%) and 1,1,2,2-tetraphenylmethane (yield 6%) have been detected and isolated from the products of the thermolysis (250 °C, NaH) of 1,1-diphenylpropanone azine $[(C_6H_5)_2CHC(CH_3)=N-]_2$.[474] The formation of the expected pyrrole derivatives is not observed under these conditions.

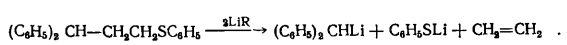
When β,β-dibenzhydryl-β-ethoxycarbonyl-α-oxopropionic acid is heated for 20 min at 190–200 °C, the following benzhydryl derivatives of malonic acid are formed[475]:



It has been found[476] that the reaction of trichloroethylene and 2-chloro-1,1-diphenylethylene with benzene in the presence of aluminium chloride results in the formation of polynuclear condensation products, which give rise to anthracene, triphenylmethane, and diphenylmethane on thermal decomposition. These experiments were subsequently repeated[463].

2-Hydroxy-1,4-naphthoquinone and benzophenone are formed in the oxidation and other reactions of 2-benzhydryl-1,4-naphthoquinone[477]. The interaction of 2,3-dibenzhydryl-1-phenylnaphthalene with benzene in the presence of aluminium chloride gives rise to triphenylmethane, 1-phenylnaphthalene, and its isomerisation product—2-phenylnaphthalene[478].

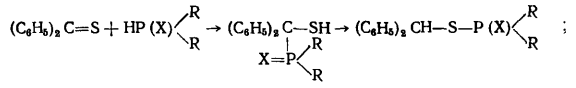
The reaction of 3,3-diphenylpropyl phenyl sulphide under the influence of an organolithium compound proceeds in accordance with the following mechanism[479]:



If the SC_6H_5 group is replaced by the $NHSO_2C_6H_5$ group, the oxidation of the resulting compound with potassium permanganate gives rise to benzophenone[480].

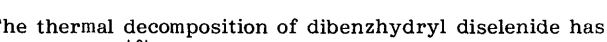
V. THE CLEAVAGE OF THE BONDS BETWEEN THE BENZHYDRYL GROUP AND OTHER ATOMS

It has been shown[481] that dibutylphosphinous and dibutylphosphonothious acids react with thiobenzophenone in the absence of catalysts at room temperature. The main products isolated in all cases were the isomeric *S*-benzhydryl phosphorothioates and their analogues instead of the expected α-mercapto-derivatives of phosphorus acids:

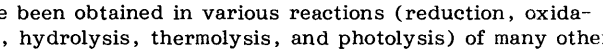


Such isomerisation of α-mercapto-derivatives of phosphorus acids, i.e. the C → S migration of the phosphoryl and thiophosphoryl groups, has been investigated also in relation to other examples[481]. α-Mercapto benzhydrylphosphonites rearrange on heating to benzhydrylphosphonothionates[482].

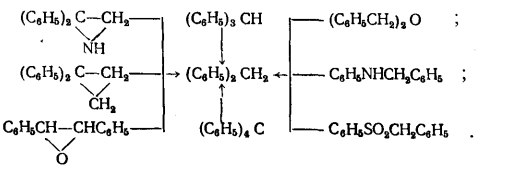
Benzhydryl acetate has also been obtained in accordance with the following mechanism[483]:



The thermal decomposition of dibenzhydryl diselenide has been described[484]:



Diphenylmethane, diphenylmethanol, and diphenyl ketone have been obtained in various reactions (reduction, oxidation, hydrolysis, thermolysis, and photolysis) of many other compounds containing phenyl groups (usually attached to a carbon atom), for example:



These instances of the formation of the benzhydryl group are not considered in this review, since they can be only formally included among debenzhydrylation reactions.

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