HERZIG AND ZEISEL\textsuperscript{1} observed that under the normal conditions of O-alkylation of phenols, certain polyhydroxy phenyl derivatives exhibit a tendency to undergo substitution in the benzene nucleus itself. Later Perkin\textsuperscript{2} and almost simultaneously Gregor\textsuperscript{3} as well as Weschler\textsuperscript{4} independently observed that methylation of $\beta$-resorcylic acid with methyl iodide and potassium hydroxide gave 2-hydroxy-4-methoxy-3-methylbenzoic acid. Perkin\textsuperscript{2}, however, failed to similarly ethylate $\beta$-resorcylic acid, resacetophenone and $\beta$-resorcylaldehyde. Later Robinson and Shah\textsuperscript{5} basing their work on the observation of Crabtree and Robinson\textsuperscript{6} that C-alkylation precedes O-alkylation where excess of alkyl iodide and potassium hydroxide are used, were successful in nuclearly ethylating $\beta$-resorcylaldehyde, resacetophenone and methyl $\beta$-resorcylate. Shah and Shah\textsuperscript{7} later extended this work to methylation of various 4-acylresorcinols.

In continuation of this work, nuclear benzylation has been studied with various acylresorcinols, namely, resacetophenone, respropiophenone, resbutyrophenone and resbenzophenone.

Resacetophenone (I, $R = \text{Me}$, $R_1 = \text{H}$) on benzylation with excess of benzyl chloride and potassium hydroxide gave 2-hydroxy-3-benzyl-4-benzyloxyacetophenone (II, $R = \text{Me}$, $R_1 = \text{CH}_2\text{Ph}$) along with 4-benzyloxy-2-hydroxyacetophenone (I, $R = \text{Me}$, $R_1 = \text{CH}_3\text{Ph}$). The former (II, $R = \text{Me}$, $R_1 = \text{CH}_3\text{Ph}$) on debenzylation gave 2:4-dihydroxy-3-benzylacetophenone (II, $R = \text{Me}$, $R_1 = \text{H}$), the constitution of which was proved by its synthesis by Hoesch reaction on 2-benzylresorcinol (III) with acetonitrile.

\begin{align*}
\text{(I)} & \quad \text{(II)} & \quad \text{(III)} & \quad \text{(IV)} \\
\text{R}_1\text{O} & \text{R}_1\text{O} & \text{ HO} & \text{ PhCH}_2\text{O} \\
\text{COR} & \text{OH} & \text{CH}_2\text{Ph} & \text{OCH}_2\text{Ph} \\
\end{align*}

Respropiophenone (I, $R = \text{Et}$, $R_1 = \text{H}$) and resbutyrophenone (I, $R = \text{Pr}$, $R_1 = \text{H}$) on similar treatment gave 2-hydroxy-3-benzyl-4-benzylxy-
propiophenone (II, \( R = \text{Et} \), \( R_1 = \text{CH}_2\text{Ph} \)) and 2-hydroxy-3-benzyl-4-benzyl-oxbytrophonone (II, \( R = \text{Pr} \), \( R_1 = \text{CH}_3\text{Ph} \)) respectively along with the corresponding 4-O-benzyl-respropiophenone and -resbutyrophenone (I, \( R = \text{Et} \), \( R_1 = \text{CH}_2\text{Ph} \) and I, \( R = \text{Pr} \) and \( R_1 = \text{CH}_3\text{Ph} \) respectively). The former compounds (II, \( R = \text{Et} \), \( R_1 = \text{CH}_2\text{Ph} \) and \( R = \text{Pr} \), \( R_1 = \text{CH}_3\text{Ph} \)) on debenylation gave respectively 2:4-dihydroxy-3-benzylpropiophenone (II, \( R = \text{Et} \), \( R_1 = \text{H} \)) and 2:4-dihydroxy-3-benzylbutyrophenone (II, \( R = \text{Pr} \), \( R_1 = \text{H} \)). The constitutions were established by their synthesis by Hoesch reaction on 2-benzylresorcinol (III) with the corresponding nitriles.

Resphenzophenone (I, \( R = \text{Ph} \), \( R_1 = \text{H} \)) gave a mixture from which the components could not be separated. Hence, the product was directly debenzylated when 2:4-dihydroxy-3-benzylbenzophenone (II, \( R = \text{Ph} \), \( R_1 = \text{H} \)) and resbenzophenone were obtained. The constitution of the former (II, \( R = \text{Ph} \), \( R_1 = \text{H} \)) was established by its synthesis by Hoesch reaction on 2-benzylresorcinol (III) with benzonitrile. 2:4-Dihydroxy-3-benzylbenzophenone (II, \( R = \text{Ph} \), \( R_1 = \text{H} \)) on benzylolation with benzyl chloride and potassium carbonate gave the corresponding 4-O-benzyl derivative (II, \( R = \text{Ph} \), \( R_1 = \text{CH}_3\text{Ph} \)).

As the 2:4-dibenzyloxy derivatives of the above 4-acylresorcinols (IV) were expected to be formed during the course of this reaction they were also prepared by benzylolation of the corresponding 4-O-benzyl derivatives (I, \( R_1 = \text{Ph} \)). Attempts to nuclearily benzylate 4-O-benzylresacetophenone (I, \( R = \text{Me} \), \( R_1 = \text{CH}_3\text{Ph} \)) were unsuccessful, thus confirming the observation of Crabtree and Robinson.\(^8\)

It is interesting to note that though the benzyl group has greater bulk than methyl or ethyl group, nuclear substitution is more facile in the case of benzyl than in the case of ethyl halide. This may be due to the fact that the halogen has enhanced reactivity when attached to the structure C: C: C: C\(_1\) found in allyl and benzyl chlorides.\(^8\)

**EXPERIMENTAL**

**Nuclear Benzylation of Resacetophenone : 2-Hydroxy-3-benzyl-4-benzyloxyacetophenone (II, \( R = \text{Me} \), \( R_1 = \text{CH}_3\text{Ph} \))**

Resacetophenone\(^8\) (15.0 g.) was dissolved in methanolic potassium hydroxide (17.0 g., 3 mols. in 125 c.c.), benzyl chloride (38.0 g., 3 mols.) added and the mixture left overnight, when some solid separated. It was refluxed on a steam-bath for 5 hours, methanol was removed by distillation and excess of benzyl chloride by steam distillation. The residue, after
dilution with water, was extracted with ether, and the ether extract was washed several times with 10% alkali, then with water and dried. On removal of ether, an oily product was obtained which was extracted with hot alcohol. The alcohol extract on cooling deposited colourless needles of 2-hydroxy-3-benzyl-4-benzyloxyacetophenone (II, R = Me, R1 = CH2Ph), m.p. 120-21° (3.4 g.) (Found: C, 79.1; H, 6.4. C25H20O3 requires C, 79.5; H, 6.0%). It is insoluble in alkali and gives cherry-red coloration with alcoholic ferric chloride. It is soluble in acetic acid and chloroform and very soluble in benzene.

The oily residue that remained after treatment with hot alcohol, on distilling under reduced pressure, gave 2-hydroxy-4-benzyloxyacetophenone, m.p. and mixed m.p. 106-07°. Sheth, Gulati and Venkataraman⁹ give m.p. 111°.

Debenzylation of 2-Hydroxy-3-benzyl-4-benzyloxyacetophenone (II, R = Me, R1 = CH2Ph): 2:4-Dihydroxy-3-benzylacetophenone (II, R = Me, R1 = H)

A mixture of 2-hydroxy-3-benzyl-4-benzyloxyacetophenone (1.0 g.), glacial acetic acid (10 c.c.) and concentrated hydrochloric acid (5 c.c.) was refluxed for 2 hours. It was diluted and subjected to steam-distillation to remove benzyl alcohol and acetic acid. The residual liquid on cooling gave a solid which was crystallised from alcohol when 2:4-dihydroxy-3-benzylacetophenone (II, R = Me, R1 = H) separated in colourless plates, m.p. 195-97° (0.5 g.) (Found: C, 73.9; H, 5.7. C13H14O3 requires C, 74.4; H, 6.0%).

Hoesch Reaction on 2-Benzylresorcinol (III) with Acetonitrile: 2:4-Dihydroxy-3-benzylacetophenone (II, R = Me, R1 = H)

Dry hydrogen chloride was passed through a mixture of ethereal solution (10 c.c.) of 2-benzylresorcinol (III, 1.5 g.), acetonitrile (0.5 g.) and fused zinc chloride (0.2 g.) for 3 hours at 0°. Ether was removed and the residue was diluted with water and boiled for 10 minutes. The product obtained on cooling was crystallised from dilute alcohol (charcoal) when colourless plates of 2:4-dihydroxy-3-benzylacetophenone (II, R = Me, R1 = H) were obtained, m.p. and mixed m.p. 195-97° (0.2 g.).

Attempted Nuclear Benzylation of 2-Hydroxy-4-benzyloxyacetophenone (I, R = Me, R1 = CH2Ph)

2-Hydroxy-4-benzyloxyacetophenone (1.0 g.) was dissolved in a solution of potassium hydroxide (2.0 g.) in methyl alcohol (40 c.c.), benzyl chloride (4.0 g.) added, and kept overnight. The next day the mixture was
refluxed for 5 hours and methanol and benzyl chloride removed as in the previous case. On working up, 2-hydroxy-4-benzyloxyacetophenone m.p. and mixed m.p. 106-0°C was recovered unchanged. No nucleyly benzylated product could be isolated.

**Benzylation of 2-Hydroxy-4-benzyloxyacetophenone (I, R = Me, R₁ = CH₂Ph)**

2 : 4-Dibenzyloloxacetophenone (IV, R = Me)

A mixture of 2-hydroxy-4-benzyloxyacetophenone (4.0 g.), benzyl chloride (8.0 g.) and anhydrous potassium carbonate (5.0 g.) in dry acetone (40 c.c.) was refluxed for 10 hours. Acetone was distilled off, and excess of benzyl chloride was removed by steam distillation. The residual solid was collected and washed several times with hot dilute alkali. The product that remained was washed with water and crystallised from rectified spirit when 2 : 4-dibenzyloloxacetophenone (IV, R = Me) separated in colourless needles, m.p. 81-82°C (2.0 g.) (Found: C, 79.1; H, 5.5. C₁₆H₁₆O₃ requires C, 79.5; H, 6.0%).

**Nuclear Benzylation of Respropiophenone:** 2-Hydroxy-3-benzyl-4-benzyloxypropiophenone (II, R = Et, R₁ = CH₂Ph)

Respropiophenone¹⁰ (3.0 g.) was dissolved in methanolic potassium hydroxide (3.2 g. in 25 c.c.), benzyl chloride (7.5 g.) was added and the mixture left overnight. The next day it was refluxed on steam-bath for 5 hours and methanol and benzyl chloride removed as in the previous case. The product was washed with sodium hydroxide (10%) and the residue crystallised from rectified spirit. After several crystallisations 2-hydroxy-3-benzyl-4-benzyloxypropiophenone (II, R = Et, R₁ = CH₂Ph), m.p. 120°C (0.75 g.) was separated from the more soluble 2-hydroxy-4-benzyloxypropiophenone (I, R = Et, R₁ = CH₂Ph). 2-Hydroxy-3-benzyl-4-benzyloxy-propiophenone (II, R = Et, R₁ = CH₂Ph) is insoluble in alkali and gives cherry red coloration with alcoholic ferric chloride (Found: C, 80.3; H, 6.0. C₂₃H₂₈O₇ requires C, 79.8; H, 6.4%).

The mother liquor after the removal of the above gave 2-hydroxy-4-benzyloxypropiophenone, crystallised from rectified spirit, m.p. and mixed m.p. with an authentic specimen prepared as described below, 113-14°C (Found: C, 75.5; H, 6.0; C₁₆H₁₆O₃ requires C, 75.0; H, 6.2%).

**Debenzylation of 2-Hydroxy-3-benzyl-4-benzyloxypropiophenone (II, R = Et, R₁ = CH₂Ph): 2 : 4-Dihydroxy-3-benzylpropiophenone (II, R = Et, R₁ = H)**

A mixture of 2-hydroxy-3-benzyl-4-benzyloxypropiophenone (1 g.), glacial acetic acid (10 c.c.) and concentrated hydrochloric acid (5 c.c.) was
refluxed for 2 hours and worked up as in the previous corresponding experiment. The solid obtained was crystallised from rectified spirit when colourless plates of 2:4-dihydroxy-3-benzylpropiophenone (II, R = Et, R₁ = H) were obtained, m.p. 157°-58° (0.2 g.) (Found: C, 74.6; H, 5.8. C₁₆H₁₆O₃ requires C, 75.0; H, 6.2%).

**Hoesch Reaction on 2-Benzylresorcinol (III) with Propionitrile : 2:4-Dihydroxy-3-benzylpropiophenone (II, R = Et, R₁ = H)**

Dry hydrogen chloride was passed through a mixture of 2-benzylresorcinol (1.0 g.) and propionitrile (0.55 g.) dissolved in ether (10 c.c.) and fused zinc chloride (0.22 g.) for 3 hours. It was then worked up as in the previous corresponding case. The product obtained on crystallisation from rectified spirit (charcoal) gave colourless plates of 2:4-dihydroxy-3-benzylpropiophenone, m.p. and mixed m.p. with the above product 157°-58° (0.15 g.).

**Benzylation of Respropiophenone : 2-Hydroxy-4-benzyloxypropiophenone (I, R = Et, R₁ = CH₂Ph)**

A mixture of respropiophenone (2.5 g.), benzyl chloride (7.5 g.) and anhydrous potassium carbonate (2.1 g.) in dry acetone (15 c.c.) was refluxed for 8 hours. After removing acetone and benzyl chloride, the residue obtained was crystallised from rectified spirit which gave orange needles of 2-hydroxy-4-benzyloxypropiophenone, m.p. and mixed m.p. with the above product 113°-14° (1.8 g.).

**Benzylation of 2-Hydroxy-4-benzyloxypropiophenone (I, R = Et, R₁ = CH₂Ph) : 2:4-Dibenzyloxypropiophenone (IV, R = Et).**

A mixture of 2-hydroxy-4-benzyloxypropiophenone (2.56 g.), benzyl chloride (7.0 g.) and anhydrous potassium carbonate (4.12 g.) in dry acetone (15 c.c.) was refluxed for 20 hours. Acetone and benzyl chloride were removed, the product was dissolved in hot rectified spirit. The first fraction of crystals of the unreacted monobenzyl ether, obtained on cooling was collected. The mother-liquor was concentrated and 2:4-dibenzyloxypropiophenone (IV, R = Et) obtained was crystallised from the same solvent, m.p. 64°-65° (0.8 g.) (Found: C, 79.4; H, 5.7. C₂₀H₂₂O₃ requires C, 79.7; H, 6.1%).

**Nuclear Benzylation of Resbutyrophenone : 2-Hydroxy-3-benzyl-4-benzyloxy butyrophenone (II, R = Pr, R₁ = CH₂Ph)**

Resbutyrophenone¹¹ (9.0 g.) was dissolved in methanolic potassium hydroxide (8.9 g. in 100 c.c.), benzyl chloride (19.0 g.) was added and the mixture left overnight. The next day, it was refluxed for 5 hours, methanol
and benzyl chloride removed and the product washed with sodium hydroxide
solution and worked up as described in the previous corresponding experi-
ment. The residue obtained was fractionally crystallised from rectified
spirit when 2-hydroxy-3-benzyl-4-benzyloxybutyrophenone (II, \(R = Pr, \ R_1 = CH_2Ph\)) was obtained in colourless needles, m.p. 108-09° (1.25 g.) (Found: 
C, 80.4; H, 6.7. \(C_{24}H_{25}O_4\) requires C, 80.0; H, 6.7%). It was insoluble
in alkali and gave cherry red coloration with alcohol ferric chloride.

From the filtrate 2-hydroxy-4-benzyloxybutyrophenone (I, \(R = Pr,\
\ R_1 = CH_2Ph\)) m.p. and mixed m.p. with an authentic specimen prepared
as described below, 85°, was obtained (Found: C, 75.2; H, 6.4. \(C_{17}H_{18}O_3\
requires C, 75.5; H, 6.7%).

**Debenzylation of 2-Hydroxy-3-benzyl-4-benzyloxybutyrophenone (II, \(R = Pr,\
\ R_1 = CH_2Ph\))** : 2:4-Dihydroxy-3-benzylbutyrophenone (II, \(R = Pr,\n\ R_1 = H\))

A mixture of 2-hydroxy-3-benzyl-4-benzyloxybutyrophenone (1.0 g.)
glacial acetic acid (15 c.c.) and concentrated hydrochloric acid (7.5 c.c.)
was refluxed for 3 hours. After working up as before, the product obtained
was crystallised from rectified spirit, when colourless plates of 2:4-
dihydroxy-3-benzylbutyrophenone (II, \(R = Pr, \ R_1 = H\)), m.p. 140-42° were
obtained (Found: C, 76.0; H, 6.9. \(C_{17}H_{18}O_3\) requires C, 75.5; H, 6.7%).

**Hoesch Reaction on 2-Benzylresorcinol (III) with Butyronitrile** : 2:4-
Dihydroxy-3-benzylbutyrophenone (II, \(R = Pr, \ R_1 = H\))

Dry hydrogen chloride was bubbled through a mixture of an ethereal
solution (10 c.c.) of 2-benzylresorcinol (1.0 g.), butyronitrile (0.5 g.) and
fused zinc chloride (0.23 g.) for 3 hours. It was worked up as in the pre-
vious case and the dark blue pasty product obtained was crystallised several
times from rectified spirit (charcoal) when 2:4-dihydroxy-3-benzylbutyro-
phenone (II, \(R = Pr, \ R_1 = H\)) separated in colourless plates, m.p. and mixed
m.p. 140-42° (0.15 g.).

**Benzylation of Resbutyrophenone : 2-Hydroxy-4-benzyloxybutyrophenone (I,\n\(R = Pr, \ R_1 = CH_2Ph\))**

A mixture of resbutyrophenone (12.0 g.), benzyl chloride (33.4 g.)
and anhydrous potassium carbonate (18.4 g.) in dry acetone (50 c.c.) was
refluxed for 8 hours. Acetone and benzyl chloride were removed. The
product obtained was crystallised from rectified spirit, when 2-hydroxy-4-
benzyloxy-butyrophenone (I, \(R = Pr, \ R_1 = CH_2Ph\)) separated in needles,
m.p. 85° (9.0 g.).
Benzylation of 2-Hydroxy-4-benzyloxybutyrophenone (I, R = Pr, R₁ = CH₂Ph) : 2:4-Dibenzylbenzophenone (IV, R = Pr) 

A mixture of 2-hydroxy-4-benzyloxybutyrophenone (9·0 g.), benzyl chloride (25·0 g.) and anhydrous potassium carbonate (15·8 g.) in dry acetone (50 c.c.) was refluxed for 20 hours. Acetone and benzyl chloride were removed as usual. The product was dissolved in hot rectified spirit. The first fraction of crystals of the unreacted monobenzyl ether, obtained on cooling, was removed. The mother-liquor on concentration gave crystals of 2:4-dibenzylbenzophenone (IV, R = Pr), m.p. 61-62° (1·2 g.) (Found: C, 76·0; H, 7·0. \( \text{C}_{24}\text{H}_{32}\text{O}_3 \) requires C, 75·5; H, 6·7%).

Nuclear Benzylation of Res benzophenone : 2:4-Dihydroxy-3-benzylbenzophenone (II, R = Ph, R₁ = H) 

Res benzophenone (6·5 g.) was dissolved in methanolic potassium hydroxide (5·0 g. in 30 c.c.), benzyl chloride (11·4 g.) was added and the mixture kept overnight. The next day it was refluxed for 5 hours, methanol and benzyl chloride were removed and the product washed with sodium hydroxide solution. An oily product remained, which after several crystallisations gave a mixture, m.p. 69-94° (2·0 g.). As no pure product could be isolated from it, it (2·0 g.) was directly debenzylated by boiling with concentrated hydrochloric acid (20 c.c.) for 2 hours. On working up as usual, 2:4-dihydroxy-3-benzylbenzophenone (II, R = Ph, R₁ = H) was obtained, crystallised from rectified spirit in colourless plates, m.p. 159-60° (0·2 g.) (Found: C, 78·4; H, 5·3. \( \text{C}_{20}\text{H}_{16}\text{O}_3 \) requires C, 78·9; H, 5·3%).

On concentrating the filtrate after removal of the above product (II, R = Ph, R₁ = H), orange needles of res benzophenone separated, m.p. and mixed m.p. 142-144°.

Hoesch Reaction on 2-Benzylresorcinol (III) with Benzonitrile : 2:4-Dihydroxy-3-benzylbenzophenone (II, R = Ph, R₁ = H) 

Dry hydrogen chloride was passed through a mixture of 2-benzylresorcinol (1·0 g.), benzonitrile (0·5 g.) and fused zinc chloride (0·2 g.) in dry ether (10 c.c.) for 3 hours and subsequently worked up as usual. The dark blue solid residue was crystallised from rectified spirit (charcoal) when colourless needles of 2:4-dihydroxy-3-benzylbenzophenone, m.p. and mixed m.p. 159-60° (0·12 g.) separated.
Benzylation of 2:4-Dihydroxy-3-benzylbenzophenone \((II, R = Ph, R_1 = H)\):

2-Hydroxy-3-benzyl-4-benzyloxybenzophenone \((II, R = Ph, R_1 = CH_2Ph)\)

A mixture of 2:4-dihydroxy-3-benzylbenzophenone \((0.2 \text{ g.})\), benzyl chloride \((0.15 \text{ g.})\) and anhydrous potassium carbonate \((0.13 \text{ g.})\) in dry acetone \((10 \text{ c.c.})\) was refluxed for 8 hours. Benzyl chloride and acetone were removed. The residue after washing with sodium hydroxide was crystallised from rectified spirit when 2-hydroxy-3-benzyl-4-benzyloxybenzophenone \( (II, R = Ph, R_1 = CH_2Ph) \) separated in colourless needles, m.p. 92–93° \( (0.08 \text{ g.}) \) (Found: C, 81.8; H, 5.2. \( C_{27}H_{21}O_3 \) requires C, 82.2; H, 5.6%).

Benzylation of Resbenzophenone:

2-Hydroxy-4-benzyloxybenzophenone \((I, R = Ph, R_1 = CH_2Ph)\)

A mixture of resbenzophenone \((6.5 \text{ g.})\), anhydrous potassium carbonate \((8.28 \text{ g.})\) and benzyl chloride \((15.8 \text{ g.})\) in dry acetone \((30 \text{ c.c.})\) was refluxed for 8 hours. Acetone and benzyl chloride were removed. The product on crystallisation from rectified spirit gave orange needles of 2-hydroxy-4-benzyloxybenzophenone \( (I, R = Ph, R_1 = CH_2Ph) \), m.p. 120–21° \( (3.2 \text{ g.}) \) (Found: C, 79.2; H, 5.2. \( C_{20}H_{16}O_3 \) requires C, 78.9; H, 5.3%).

Benzylation of 2-Hydroxy-4-benzyloxybenzophenone \((I, R = Ph, R_1 = CH_2Ph)\):

2:4-Dibenzyloxybenzophenone \((IV, R = Ph)\)

A mixture of 2-hydroxy-4-benzyloxybenzophenone \((3.0 \text{ g.})\), benzyl chloride \((5.0 \text{ g.})\) and potassium carbonate \((2.7 \text{ g.})\) in dry acetone \((15 \text{ c.c.})\) was refluxed for 10 hours. After removing acetone and benzyl chloride, the residue was washed several times with hot dilute sodium hydroxide solution. The insoluble portion, after washing with water, was crystallised from rectified spirit, when 2:4-dibenzyloxy-benzophenone \( (IV, R = Ph) \) was obtained, colourless plates, m.p. 116–17° \( (1.3 \text{ g.}) \) (Found: C, 82.1; H, 5.8. \( C_{27}H_{21}O_3 \) requires C, 82.2; H, 5.6%).

**SUMMARY**

In continuation of the work of Shah and collaborators on nuclear alkylation of resorcinol derivatives, nuclear benzylation has been studied with various 4-acylresorcinols, namely, resacetophenone, respropiophenone, resbutyrophenone and resbenzophenone. In all the cases the benzyl group was found to enter the \( \gamma \)-position giving 4-O-benzyl-3-benzylresacylophenones. The constitutions have been established by comparison of the debenzylated products, that is, 3-benzyl-resacylophenones, with the product obtained by Hoesch reaction on 2-benzylresorcinol with the corresponding acylnitrile.
Nuclear alkylation is found to be more facile with benzyl than with ethyl halide.

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