ABSTRACTS

Effects of leaching conditions on the extraction of chromium were studied. 1. Size of ore, 2. Leaching time, 3. Amount of \((\text{NH}_4)_2\text{Cr}_2\text{O}_7\) added, 4. Leaching temperature, and 5. Amount of charge.

Summary of Result: Results obtained are summarized in Table 1, which shows the optimum conditions for extraction of chromium.

If the acid concentration was increased up to 60%, insoluble brownish-yellow precipitate was formed in the presence of \((\text{HN})_2\text{Cr}_2\text{O}_7\) in both cases of leaching.

Reaction of \(n\)-Hexyl Bromide with Potassium Fluoride in Several Glycols

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Reactions of \(n\)-hexyl bromide with potassium fluoride in isomeric propanediols and butanediols are studied. \(n\)-Hexylfluoride is obtained in high yields by the use of such glycols that have two adjacent hydroxy groups in the molecule. Two geometrical isomers of 1,2-cyclohexanediols are used as the solvent. The yield of \(n\)-hexyl fluoride is ca. 30% in the *trans* isomer, whereas it's only 2% in the *cis* isomer.

Nitration of Desoxybenzoin

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The reaction routes of desoxybenzoin with nitric acid (\(d=1.30\)) were studied by using \(C^{14}\)-tracer technique. By the nitration of desoxybenzoin-carbonyl-\(C^{14}\) (I) (12.95\(\mu\)c/m.mole) there were obtained \(p\)-nitrobenzil-carbonyl-\(C^{14}\) (II) (12.92\(\mu\)c/m.mole), benzil-carbonyl-\(C^{14}\) (III) (12.96\(\mu\)c/m.mole), inactive \(p\)-nitrobenzoic acid (IV) and benzoic acid-carboxyl-\(C^{14}\) (V) (11.29\(\mu\)c/m.mole). When treated with alcoholic potassium hydroxide, II decomposed into inactive \(p,p'\)-azoxydibenzoic acid, inactive \(p\)-nitrobenzoic acid and benzoic acid-carboxyl-\(C^{14}\) (12.96\(\mu\)c/m.mole). These facts indicate that the nitration occurs at the *para* position to the methylene group of I.

The non-labeled specimens of \(p\)-nitrodesoxybenzoin, \(p\)-nitrobenzil, and benzil were treated with the nitric acid, respectively. \(p\)-Nitrodesoxybenzoin gave benzoic acid, \(p\)-nitrobenzoic acid, and \(p\)-nitrobenzil. Benzil and \(p\)-nitrobenzil, however, were resistant to nitration and oxidation, and were recovered without loss. The specific activity of V is comparatively lower than the theoretical value. These results would lead to the following conclusions. In the action of the nitric acid on desoxybenzoin-carbonyl-\(C^{14}\), the \(p\)-nitrodesoxybenzoin-carbonyl-\(C^{14}\) is formed as an intermediate, and its methylene group is oxidized to \(p\)-nitrobenzil-carbonyl-\(C^{14}\). While oxidative disruption of the same intermediate gives benzoic acid-carboxyl-\(C^{14}\).
and inactive \( p \)-nitrobenzoic acid. A similar oxidation and oxidative disruption prior to the nitration of desoxybenzoin-carbonyl-C\( ^{14} \) also take place and give benzil-carbonyl-C\( ^{14} \), benzoic acid-carboxyl-C\( ^{14} \) and inactive benzoic acid. The essential reaction routes are shown as follows.

\[
\begin{align*}
\text{NO}_2 \quad &\text{COC}^\text{O} \quad \text{CH}_2 \text{C}^\text{O} \quad \text{NO} \quad \text{CH}_2 \text{COOH} \quad \text{C}^\text{OOH}
\end{align*}
\]

Studies on Condensation Products of Chlorinated Phenols with Chloral. (I)

The Structure of Condensation Product of 2,4-Dichlorophenol or 2,4-Dichloroanisole with Chloral

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In recent paper, Riemschneider (*J. Am. Chem Soc.*, 76, 4853 (1952)) obtained a compound II, mp. 154\( ^\circ \), by methylating condensation product (I) of 2,4-dichlorophenol with chloral, and another compound V, mp. 160\( ^\circ \), by condensing 2,4-dichloroanisole with chloral. He proposed (1) these two compounds were stereoisomers of 1,1-bis-(2'-methoxy-3',5'-dichlorophenyl)-2,2,2-trichloroethane resulting from the restricted rotation of benzene rings, and (2) the dichloroethylene compounds III, mp. 166\( ^\circ \), and VI, mp. 157\( ^\circ \), obtained by dehydrochlorination of compounds II and V, respectively were also the same type stereoisomers.

This study was undertaken to ascertain the above results and obtained the following conclusion. To a mixture of above 1,1-bis-(x'-methoxy-y', z'-dichlorophenyl)-2,2,2-trichloroethane (compound II or V) and diethylene glycol was added a concentrated potassium hydroxide solution, and then the mixture was refluxed at 160-70\( ^\circ \) for 5 hours. Bis-(x-hydroxy-y, z-dichlorophenyl)-methane (IV), mp. 167.5 -68.5\( ^\circ \), was obtained from compound II through hydrolysis, decarboxylation and following demethylation. Compound IV was not identical with the corresponding compound VII, mp. 152.5-53.5\( ^\circ \), obtained from compound V. Compound IV was also prepared by condensing 3,4-dichlorophenol with paraformaldehyde. Compound IV was then dechlorinated to bis-(2-hydroxyphenyl)-methane, mp. 117-18\( ^\circ \), with sodium in liquid ammonia. From the above, it was confirmed that the structure of compound II is 1,1-bis-(2'-methoxy-3', 5'-dichlorophenyl)-2, 2, 2-trichloroethane which was identical with the structure proposed by Riemschneider.

On the other hand, a compound, mp. 96-97\( ^\circ \), which was obtained by the same procedure from compound VII, was identical with bis-(3-hydroxyphenyl)-methane.

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