

*n*-Pentane.—By hydrogenating the above pentene-2, using nickel catalyst and purifying the product with sulfuric acid, very pure pentane was obtained, boiling at 36.1–36.2° (corr.).

Isopentane was obtained in a manner similar to that used for *n*-pentane but using the amylene mixture from the dehydration of *tert.*-amyl alcohol. The final product boiled at 27.9–28.0° (corr.).

### Summary

1. The temperatures have been determined at which *n*-pentane, isopentane, pentene-1, pentene-2 and trimethylethylene begin to decompose when heated in a Pyrex glass vessel for one hour.

2. The determination of the rates at which these hydrocarbons decompose showed that for each hydrocarbon the increase in rate per ten degrees was constant within certain temperature limits and that this increase in rate was greater within the higher temperature limits. It appears probable that the limiting temperatures of the several ranges are the temperatures at which certain bonds undergo pyrolysis.

3. It was found that a trace of moisture had no effect on the cracking temperature of pentene-2, but that it doubled the rate at which the hydrocarbon decomposed.

4. When the olefins were heated at increasing temperatures, a temperature was found at which no expansion took place. The results indicate that at this point the expansion which results from pyrolysis is equal to the contraction resulting from polymerization.

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[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE JOHNS HOPKINS UNIVERSITY]

## THE STRUCTURE AND SOME DERIVATIVES OF PARA-DIMETHYLAMINO-PARA-CHLOROBENZOIN

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The object of this report is to present the experimental evidence in support of a structure which is assigned to *p*-dimethylamino-*p*-chlorobenzoin<sup>2</sup> and to describe the preparation and some properties of a number of related compounds.

The starting material was prepared by condensing *p*-dimethylaminobenzaldehyde and *p*-chlorobenzaldehyde by means of potassium cyanide. The substance thus prepared melts at 128° and is readily oxidized with Fehling's solution to the corresponding benzil, which fuses at 144.5°.

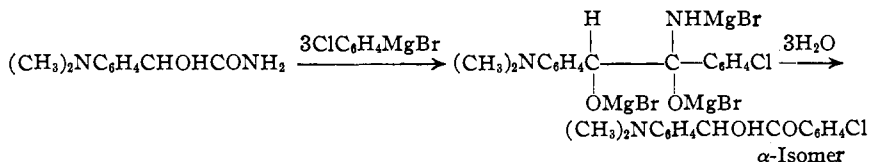
Several methods have been employed for determining the structures

<sup>1</sup> Grafflin Scholar, 1930–1931.

<sup>2</sup> Staudinger, *Ber.*, **46**, 3538 (1913).

of unsymmetrical benzoin. For example, Meisenheimer and Jochelson<sup>3</sup> obtained benzanisoin by the action of alkali on  $\alpha$ -bromobenzyl *p*-methoxyphenyl ketone. Tiffeneau and Levy<sup>4</sup> have determined the structures of a number of these substances by fission with potassium hydroxide and more recently the method of Werner and Detscheff,<sup>5</sup> which makes use of the Beckmann rearrangement of the benzoin oximes has been extended by Buck and Ide.<sup>6</sup> The foregoing, however, cannot be classed as direct methods since other standards are required for comparison. Other methods,<sup>7</sup> in which the Grignard reaction is used, are directly applicable to studies of this nature, since they lead to the synthesis of isomers of known structure and hence direct comparison with the original material is possible.

Thus, with the aid of the Grignard reaction<sup>7c</sup> both of the isomeric *p*-dimethylamino-*p'*-chlorobenzoin were prepared and studied. *p*-Dimethylaminomandelamide was condensed with *p*-chlorophenylmagnesium bromide and after hydrolysis of the resulting complex *p*-dimethylamino- $\alpha$ -hydroxybenzyl *p'*-chlorophenyl ketone was obtained and for convenience will be called  $\alpha$ -*p*-dimethylamino-*p'*-chlorobenzoin. This melted at 104.5° and on oxidation with Fehling's solution it produced the same benzil as did the "starting material" with like treatment; however, a mixed melting point determination with the latter substance showed a large depression. It is thus evident that  $\alpha$ -*p*-dimethylamino-*p'*-chlorobenzoin is different from but isomeric with the starting material. In order to obtain the other or  $\beta$ -isomer, *p*-chloromandelamide<sup>8</sup> was reacted with *p*-dimethylaminophenylmagnesium bromide and *p*-chloro- $\alpha$ -hydroxybenzyl *p'*-dimethylaminophenyl ketone was obtained. This substance melted at 128°, produced the above benzil on oxidation and when mixed with the starting material showed no depression of the melting point. Thus it is seen that *p*-dimethylamino-*p'*-chlorobenzoin as produced by condensing the respective aldehydes is the  $\beta$ -isomer and has the structure  $(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{COCHOHC}_6\text{H}_4\text{Cl}$ . The reactions are



<sup>3</sup> Meisenheimer and Jochelson, *Ann.*, **355**, 292 (1907).

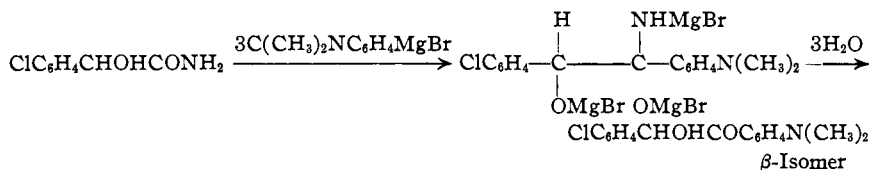
<sup>4</sup> Tiffeneau and Levy, *Compt. rend.*, **192**, 287 (1931).

<sup>5</sup> Werner and Detscheff, *Ber.*, **38**, 69 (1905).

<sup>6</sup> Buck and Ide, *THIS JOURNAL*, **53**, 1912 (1931).

<sup>7</sup> (a) Asahina and Terasaka, *J. Pharm. Soc. Japan*, **494**, 219 (1923); (b) McKenzie, Louis, Tiffeneau and Weill, *Bull. soc. chim.*, [4] **45**, 414 (1929); (c) Jenkins, Bigelow and Buck, *THIS JOURNAL*, **52**, 5198 (1930).

<sup>8</sup> Jenkins, *ibid.*, **53**, 2341 (1931).

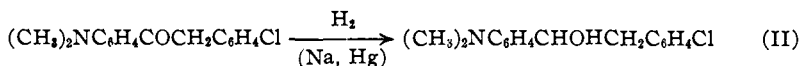
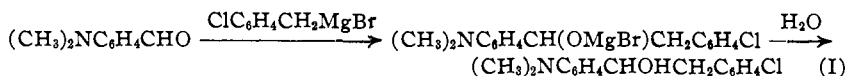


A very interesting observation was made during the study of the above isomers. It was found that the  $\alpha$ -derivative readily produced the  $\beta$ -compound when heated for a short time with alcoholic potassium cyanide. An investigation of this reaction is being made, the results of which will be published in the near future.

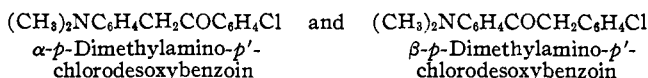
As was stated above *p*-dimethylamino-*p*'-chlorobenzil was obtained when either  $\alpha$ - or  $\beta$ -*p*-dimethylamino-*p*'-chlorobenzoin was oxidized with Fehling's solution. On reduction with tin and hydrochloric acid the  $\alpha$ -benzoin produced a compound melting at 140° which was found to be *p*-dimethylaminobenzyl *p*'-chlorophenyl ketone, and for convenience will be designated  $\alpha$ -*p*-dimethylamino-*p*'-chlorodesoxybenzoin. When the  $\beta$ -benzoin was reduced in a similar manner, not only the  $\alpha$ -desoxy derivative was isolated but another substance melting at 170° was obtained. The latter proved to be *p*-chlorobenzyl *p*'-dimethylaminophenyl ketone, which will be called  $\beta$ -*p*-dimethylamino-*p*'-chlorodesoxybenzoin. These compounds were readily separated since the  $\alpha$ -isomer was much more soluble in acid solution than the  $\beta$ -derivative. Furthermore, on reduction with sodium amalgam  $\beta$ -*p*-dimethylamino-*p*'-chlorobenzoin formed the corresponding hydrobenzoin, melting at 180°, which, when heated with a mixture of acetic and hydrochloric acids, readily lost water and formed  $\alpha$ -*p*-dimethylamino-*p*'-chlorodesoxybenzoin in good yield. No attempt was made to reduce the  $\alpha$ -benzoin under similar conditions since only a small quantity of this material was available.

The oximes were obtained by heating the desoxybenzoins in pyridine solution with an excess of hydroxylamine hydrochloride.  $\alpha$ -*p*-Dimethylamino-*p*'-chlorodesoxybenzoin oxime melted at 150.5° while  $\beta$ -*p*-dimethylamino-*p*'-chlorodesoxybenzoin oxime was found to melt at 152°. An interesting observation was made in preparing the  $\alpha$ -oxime—in the reaction mixture a substance was always found, in small percentage, which melted at 229° and proved to be *p*-dimethylamino-*p*'-chlorostilbene (described below). This unusual side reaction is being studied further.

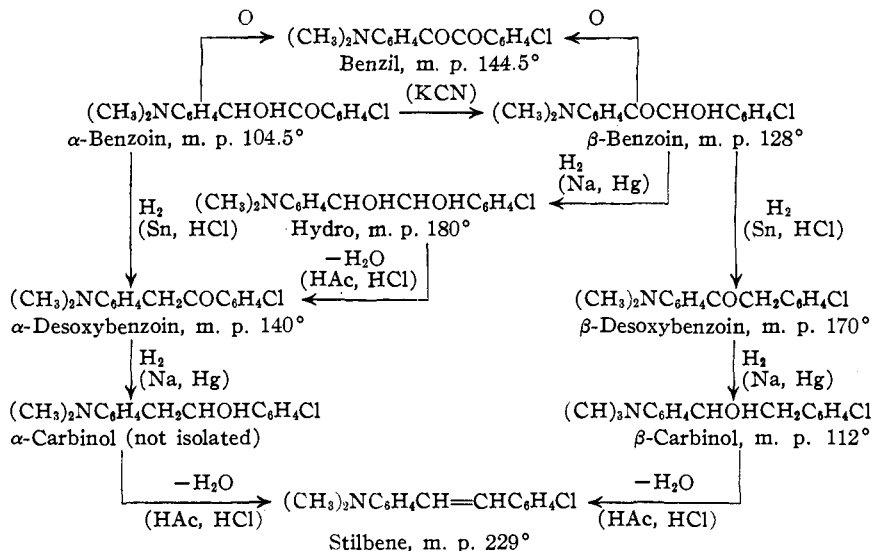
The structure of  $\beta$ -*p*-dimethylamino-*p*'-chlorodesoxybenzoin was deduced in the following manner. *p*-Dimethylaminobenzaldehyde was condensed with *p*-chlorobenzylmagnesium bromide and *p*-chlorobenzyl-*p*'-dimethylaminophenylcarbinol was obtained, which melted at 112°. When the  $\beta$ -desoxy derivative was reduced with sodium amalgam, this carbinol was also produced. The following reactions will make this clear.



When dehydrated by the acetic-hydrochloric acid mixture the above carbinol, produced by either method (I or II), lost water and formed *p*-dimethylamino-*p*'-chlorostilbene, melting at 229°. When  $\alpha$ -*p*-dimethylamino-*p*'-chlorodesoxybenzoin was likewise reduced with sodium amalgam, an isomeric carbinol was produced which also readily lost water in an analogous manner with the formation of this stilbene. Thus it is seen that the  $\alpha$ - and  $\beta$ -desoxy derivatives are isomeric and have the structures



The reactions which have been described above are represented in the following diagram



The writer wishes to thank Dr. E. Emmet Reid for his interest and help in this work.

### Experimental

$\alpha$ -*p*-Dimethylamino-*p*-chlorobenzoin.—A mixture of 15 g. of *p*-chlorobromobenzene, 2 g. of magnesium turnings and 50 cc. of anhydrous ether was refluxed for three hours. Then 3 g. of *p*-dimethylaminomandelamide,<sup>7c</sup> finely powdered, was added in small portions, over a period of thirty minutes. After heating over a twenty-five watt light for eight hours, the solution was poured into 200 g. of water and crushed ice containing 20 g. of concentrated sulfuric acid. The ether was removed and the

aqueous layer extracted with two 100-cc. portions of ether. The extracts were discarded. The acid solution was then neutralized with ammonia and the yellow precipitate which formed was separated, dissolved in 50 cc. of alcohol and filtered hot. On cooling a mass of yellow needles separated which weighed 2.0 g. and melted at 96–98°. After two crystallizations from ligroin (Skellysolve "C") the product melted at 104.5° (corr.).

*β-p*-Dimethylamino-*p*'-chlorobenzoin

**First Method.**—To a solution of 28.7 g. of *p*-dimethylaminobenzaldehyde and 28.1 g. of *p*-chlorobenzaldehyde dissolved in 140 cc. of 95% alcohol was added 5 g. of potassium cyanide dissolved in 60 cc. of water. The mixture was refluxed for one hour and allowed to cool. The crystalline material was separated, washed twice with water and once with a small portion of alcohol; yield, 21 g. To the filtrate was added 3 g. more of potassium cyanide. The solution was then heated for an hour, allowed to cool and the solid filtered. After washing with water and alcohol it weighed 6 g. The two crops of crystals were then combined and recrystallized twice from alcohol. The almost white needles melted at 128° (corr.).

**Second Method.**—A mixture of 20 g. of *p*-bromodimethylaniline, 3 g. of magnesium and 100 cc. of anhydrous ether was refluxed for forty-eight hours. After the heating, 1.3 g. of finely powdered *p*-chloromandelamide<sup>8</sup> was added in small portions over a period of thirty minutes. The solution was then refluxed for an additional eight hours and poured into 200 g. of crushed ice and water containing 20 g. of concentrated sulfuric acid. The mixture was stirred until all of the solid matter had dissolved. The clear solution was then extracted with six 50-cc. portions of ether. In this case the water layer was discarded. The ether solution was washed first with a 2% solution of sodium carbonate and then with water. The ether was distilled. The solid was then dissolved in a small amount of alcohol, filtered hot and allowed to crystallize; weight, 0.50 g. After two further crystallizations from alcohol, it melted at 128° (corr.).

**Third Method.**—Five-tenths of a gram of the  $\alpha$ -benzoin and 0.10 g. of potassium cyanide dissolved in 20 cc. of 70% alcohol were heated for thirty minutes and allowed to cool. The crystalline material was filtered off, washed with water and recrystallized from a small portion of alcohol. It weighed 0.40 g. and melted at 128° (corr.). When this material was mixed with that obtained by the other methods, there was no depression of the melting point; when a mixed melting point determination was made with the  $\alpha$ -benzoin, however, there was a depression of 10–15°.

***p*-Dimethylamino-*p*'-chlorobenzil.**—This material was obtained from  $\alpha$ - or  $\beta$ -*p*-dimethylamino-*p*'-chlorobenzoin in 90% yield when an alcoholic solution of either of these substances was heated for half an hour with an excess of concentrated Fehling's solution on the water-bath. The benzil crystallized from alcohol in thin orange-colored needles which melt at 144.5° (corr.).

***p*-Dimethylamino-*p*'-chlorohydrobenzoin.**—To 15 g. of *p*-dimethylamino-*p*'-chlorobenzoin was added 200 cc. of alcohol. The benzoin was dissolved by heating to 50–60° on the water-bath. To the solution was added 20 cc. of water and 150 g. of 4% sodium amalgam in portions, over a period of one hour. The mixture was kept at 50–60° for four hours while a steady stream of carbon dioxide was passed into it. Small portions of acetic acid were added at intervals but at no time was the mixture allowed to become acid. The mixture was occasionally shaken. After standing overnight at room temperature, 200 cc. of water was added and the suspended matter was decanted from the mercury onto a filter. After washing several times, the solid was recrystallized from alcohol; yield, 7.5 g.; m. p. 180° (corr.). There was another substance present which was quite soluble in the dilute alcohol, perhaps the isohydrobenzoin, but no attempt was made to purify it.

**$\alpha$ -*p*-Dimethylamino-*p*'-chlorodesoxybenzoin**

**First Method.**—To 1 g. of the hydrobenzoin (prepared in the above manner) were added 15 cc. of glacial acetic acid and 5 cc. of concentrated hydrochloric acid. The mixture was boiled for ten minutes, after which 50 cc. of water was added. The solution was then neutralized with 10% sodium carbonate solution allowed to stand for a few minutes and filtered. The solid was washed several times with water and crystallized from alcohol. The product was obtained in a yield of 0.80 g. and melted at 140° (corr.).

**Second Method.**—A mixture of 0.50 g. of  $\alpha$ -*p*-dimethylamino-*p*'-chlorobenzoin, 0.50 g. of mossy tin, 3 cc. of concentrated hydrochloric acid, a small crystal of copper sulfate and 10 cc. of alcohol was heated for five hours. The solution was filtered hot, 50 cc. of water added and neutralized with sodium carbonate. The mixture was allowed to stand for a while, after which the precipitate was filtered, washed several times with water, sucked dry and extracted twice with hot alcohol (25-cc. portions). The extract was evaporated to a volume of about 15 cc. and cooled; yield, 0.30 g. After two crystallizations from alcohol the substance melted at 140° (corr.).

**Third Method of Preparing  $\alpha$ -*p*-Dimethylamino-*p*'-chlorodesoxybenzoin.**—The filtrate from which the  $\beta$ -desoxy compound (described below) had been obtained was made slightly alkaline with sodium carbonate. After standing for a while the solid was separated, washed several times with water and extracted three times with 100-cc. portions of alcohol. The solution was then evaporated to a volume of about 50 cc. and allowed to cool. The solution deposited 3.50 g. of substance which after two crystallizations from alcohol formed almost white plates and melted at 140° (corr.). Mixed melting point determinations with the substance made by methods *one* and *two* showed no depression.

**$\beta$ -*p*-Dimethylamino-*p*'-chlorodesoxybenzoin.**—A mixture of 11 g. of  $\beta$ -*p*-dimethylamino-*p*'-chlorobenzoin, 10 g. of mossy tin, 0.50 g. of copper sulfate, 20 cc. of concentrated hydrochloric acid and 40 cc. of alcohol was refluxed for five hours. The hot solution was filtered and 10 cc. of water added. On cooling the solution deposited 5 g. of fine white prisms. To the filtrate 40 cc. of water was added and 0.50 g. more of the same material was obtained. After two recrystallizations from alcohol the substance melted at 170° (corr.).

***p*-Chlorobenzyl-*p*'-dimethylaminophenylcarbinol**

**First Method.** ( **$\beta$ -*p*-Dimethylamino-*p*'-chlorostilbene Hydrate.**)—A mixture of 20 g. of *p*-chlorobenzyl bromide, 2.50 g. of magnesium turnings and 100 cc. of ether was refluxed for three hours. A solution of 14 g. of *p*-dimethylaminobenzaldehyde in 100 cc. of ether was then added slowly over a period of forty-five minutes from a dropping funnel. The solution was heated for two hours and poured into 200 cc. of crushed ice and water containing 20 g. of concentrated sulfuric acid. The ether layer was separated and discarded. The water layer was made slightly alkaline with ammonium hydroxide and extracted twice with 100-cc. portions of ether. The ether solution was dried over anhydrous sodium sulfate and the ether distilled off. A yield of 20 g. of crude material was obtained. After two crystallizations from 80% alcohol the substance was obtained as white needles melting at 112° (corr.).

**Second Method.**—A mixture of 1.0 g. of  $\beta$ -*p*-dimethylamino-*p*'-chlorodesoxybenzoin, 50 cc. of alcohol (90%) and 25 g. of sodium amalgam was warmed to 50–60° while a slow stream of carbon dioxide was admitted. The reaction was allowed to proceed for four hours at this temperature. During this time the mixture was frequently shaken. The solution was then decanted from the mercury and diluted with 100 cc. of water. The solid was filtered off and washed with water, after which it was re-

crystallized from a small amount of 80% alcohol. A yield of 0.40 g. of product melting at 109–111° was obtained. When this was mixed with the carbinol obtained by the first method, there was no depression of the melting point.

*p*-Dimethylamino-*p*'-chlorostilbene.—The carbinol above as made by either method readily lost water when heated with a mixture consisting of 80% glacial acetic acid and 20% concentrated hydrochloric acid and produced *p*-dimethylamino-*p*'-chlorostilbene. A mixture of 10 g. of the carbinol, 40 cc. of glacial acetic acid and 10 cc. of concentrated hydrochloric acid was heated for ten minutes on the hot-plate. The solution was diluted with 200 cc. of water and neutralized with sodium carbonate. The solid matter was separated, washed with water and dried; weight 8.0 g. After two crystallizations from benzene it formed almost white scales and melted at 229° (corr.).

The above stilbene was also obtained by reducing  $\alpha$ -*p*-dimethylamino-*p*'-chlorodesoxybenzoin with sodium amalgam and dehydrating the resulting product with the acetic-hydrochloric acid mixture. A mixture of 1.0 g. of the  $\alpha$ -desoxy compound, 25 cc. of alcohol and 25 g. of sodium amalgam was warmed to 50–60° and occasionally shaken. At intervals small portions of 50% acetic acid were added, but not enough to make the solution acid. After four hours 100 cc. of water was added, the solid filtered, washed with water and recrystallized from dilute alcohol; yield, 0.50 g. This material was heated with 10 cc. of the acetic-hydrochloric acid mixture for a few minutes, the solution then diluted with 50 cc. of water and neutralized with sodium carbonate. The solid was filtered, washed with water and recrystallized twice from alcohol. It weighed 0.20 g. and melted at 229°. When mixed with *p*-dimethylamino-*p*'-chlorostilbene, the melting point was not depressed.

$\alpha$ -*p*-Dimethylamino-*p*'-chlorodesoxybenzoin Oxime.—A mixture of 1.0 g. of the  $\alpha$ -desoxy compound, 1.0 g. of hydroxylamine hydrochloride and 10 cc. of pyridine was heated on the water-bath for four hours. The solution was then diluted with 10 cc. of water, cooled in ice water for a while and the solid which formed separated. On recrystallization from alcohol it melted at 227–229° and proved to be *p*-dimethylamino-*p*'-chlorostilbene. It weighed 0.20 g.

The filtrate was diluted with 30 cc. of water and the precipitate which formed on standing was separated, washed with water and dried. It weighed 0.7 g. and after two crystallizations from ligroin (Skellysolve "C") it formed white prisms melting at 150.5° (corr.).

$\beta$ -*p*-Dimethylamino-*p*'-chlorodesoxybenzoin Oxime.—This oxime was obtained in a manner similar to the  $\alpha$ -compound. In this instance, however, not a trace of the

TABLE I  
COMPOUNDS, CONSTANTS AND ANALYTICAL DATA

Formula	Appearance	M. p., ° (corr.)	Analytical data			
			Nitrogen, % Found, (Kjel- dahl)		Chlorine, % Found, (Parr bomb)	
$(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{COCOC}_6\text{H}_4\text{Cl}$	Orange needles	144.5	4.87	4.78	12.32	12.35
$\alpha\text{-(CH}_3)_2\text{NC}_6\text{H}_4\text{CHOHCOC}_6\text{H}_4\text{Cl}$	Cream needles	104.5	4.83	4.75	12.24	12.10
$\beta\text{-(CH}_3)_2\text{NC}_6\text{H}_4\text{COCHOHC}_6\text{H}_4\text{Cl}$	Cream needles	128	4.83	4.90	12.24	12.20
$(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{CHOHCHOHC}_6\text{H}_4\text{Cl}$	White prisms	180	4.80	4.65	12.15	12.32
$\alpha\text{-(CH}_3)_2\text{NC}_6\text{H}_4\text{CH}_2\text{COC}_6\text{H}_4\text{Cl}$	White scales	140	5.12	4.95	12.95	13.11
$\beta\text{-(CH}_3)_2\text{NC}_6\text{H}_4\text{COCH}_2\text{C}_6\text{H}_4\text{Cl}$	White leaflets	170	5.12	5.09	12.95	13.17
$(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{CHOHCH}_2\text{C}_6\text{H}_4\text{Cl}$	Colorless needles	112	5.08	5.20	12.86	12.70
$(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{CH}=\text{CHC}_6\text{H}_4\text{Cl}$	White scales	229	5.44	5.35	13.76	13.75
$\alpha\text{-(CH}_3)_2\text{NC}_6\text{H}_4\text{CH}_2\text{C}=\text{NOHC}_6\text{H}_4\text{Cl}$	White prisms	150.5	9.70	9.63	12.28	12.40
$\beta\text{-(CH}_3)_2\text{NC}_6\text{H}_4\text{C}=\text{NOHCH}_2\text{C}_6\text{H}_4\text{Cl}$	Colorless needles	152	9.70	9.85	12.28	12.35

stilbene was obtained. This oxime could be crystallized from alcohol but ligroin proved to be a better solvent. A yield of 0.80 g. was obtained which crystallized in long colorless needles and melted at 152° (corr.). A mixed melting point determination showed that the oximes were unlike.

### Summary

1. By means of the Grignard reaction both isomeric ( $\alpha$  and  $\beta$ ) *p*-dimethylamino-*p'*-chlorobenzoin have been synthesized. The benzoin as prepared by condensing *p*-dimethylaminobenzaldehyde and *p*-chlorobenzaldehyde was found to be *p*-chloro- $\alpha$ -hydroxybenzyl *p'*-dimethylaminophenyl ketone ( $\beta$  isomer) and has the structure  $(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{-COCHOHC}_6\text{H}_4\text{Cl}$ .

2. Under the influence of potassium cyanide,  $\alpha$ -*p*-dimethylamino-*p'*-chlorobenzoin was found to rearrange and produce the  $\beta$ -isomer.

3. Upon reduction under suitable conditions, the  $\alpha$ -benzoin produced *p*-dimethylaminobenzyl *p'*-chlorophenyl ketone. Under similar treatment, however, the  $\beta$ -benzoin produced the above ketone and also its isomer, *p*-chlorobenzyl *p'*-dimethylaminophenyl ketone. Nine new substances were prepared and characterized in the course of this work.

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[CONTRIBUTION FROM THE CHEMICAL LABORATORIES OF COLUMBIA UNIVERSITY]

## THE AUTOXIDATION OF ALPHA NORMAL-AMYL CINNAMIC ALDEHYDE. CIS- AND TRANS-ALPHA-NORMAL-AMYL-CINNAMIC ACIDS

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### Introduction

In the course of our study of the rates of autoxidation of commercial aldehydes,<sup>2</sup> we were led to examine the products resulting from the action of air on  $\alpha$ -*n*-amylcinnamic aldehyde,  $\text{C}_8\text{H}_5\text{CH}=\text{C}(\text{Am})\text{CHO}$ . Since, in general, the action of air or oxygen on an aldehyde leads to the formation of the corresponding acid through the intermediate per-acid, thus<sup>3</sup>



and, since unsaturated aldehydes such as crotonaldehyde and cinnamic aldehyde behave in accordance with this formulation, we expected that this aldehyde would yield  $\alpha$ -amylcinnamic acid. Investigation revealed, however, that, when blown with air,  $\alpha$ -amylcinnamic aldehyde undergoes

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<sup>2</sup> Bogert and Davidson, *Am. Perfumer Ess. Oil Rev.*, **24**, 587, 667 (1929-1930).

<sup>3</sup> Baeyer and Villiger, *Ber.*, **33**, 1582 (1900); Jorissen and van der Beek, *Rec. trav. chim.*, **45**, 245 (1926); **47**, 286 (1928); H. L. J. Bäckström, *Medd. Vetenskapsakad. Nobelinst.*, **6**, No. 15 (1927).