DL-*β-Benzylaminoisobutyramide.* *N*-Benzyl-DL-*β*-aminoisobutyric acid (4 g.), which had been dried before in a vacuum desiccator over phosphorus pentoxide, was suspended in 100 ml. dry dioxane in a 3-necked flask equipped with a gas leading tube, reflux condenser connected to a calcium chloride tube, and a mechanical stirrer. Phosgene was bubbled in with stirring for 60 min. and the temperature maintained at 60°. Excess phosgene and solvent were removed *in vacuo* at 40°. The residue was dissolved in 50 ml. dry dioxane and dry ammonia gas passed in with stirring and cooling for 20 min. After leaving overnight, the solution was filtered from ammonium chloride and evaporated to dryness *in vacuo*. The substance refused to crystallize from ethyl acetate–petroleum ether. Cooling in a Dry Ice–acetone bath caused complete crystallization but on heating to room temperature the substance became sticky. The *N*-benzyl-DL-*β*-aminoisobutyramide was obtained on vacuum distillation at 165°/0.3 mm. On leaving overnight in a vacuum dessicator over phosphorus pentoxide the substance crystallized, m.p. 58°; yield 2.8 g. (70%). It is soluble in ethanol, acetone, chloroform, less in water (basic reaction) and ether.

Anal. Calcd. for $C_{11}H_{16}N_2O$: N, 14.6. Found: N, 14.3.

Reaction of alcoholic picric acid with the amide gave the picrate derivative, m.p. 168° on recrystallization from ethanol.

Anal. Calcd. for $C_{17}H_{19}N_5O_8$: N, 17.0. Found: N, 17.2.

JERUSALEM, ISRAEL

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, TEXAS TECHNOLOGICAL COLLEGE]

The "Thermal" Rearrangement of Hydrazo Compounds. IV. The Intramolecularity of the Rearrangement[1]

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The products of the rearrangement of 2,2′-hydrazonaphthalene, 1,2′-hydrazonaphthalene and 1-phenyl-2-*β*-naphthylhydrazine have been isolated. The 2,2′-hydrazonaphthalene was caused to rearrange in the absence of solvent at 90° and 150°, and in cyclohexane at 95°. The only products isolable and in excellent recovery were 2,2′-diamino-1,1′-binaphthyl and 3,4:5,6-dibenzocarbazole. The 1,2′-hydrazonaphthalene was caused to rearrange in cyclohexane at 95°. The only products isolable and in very poor recovery were 1,2′-diamino-1′,2-dinaphthyl and 1,2:5,6-dibenzocarbazole. The 1-phenyl-2-*β*-naphthylhydrazine was caused to rearrange in 95% ethanol at 90°. The only products isolable and in fairly good recovery were 1-(2-aminophenyl)-2-naphthylamine and 7-benzo[*c*]carbazole. For the last two hydrazo compounds authentic samples of products of intramolecular rearrangement were prepared by acid-catalyzed rearrangement. The results show that the thermal rearrangement is intramolecular.

In recent years reports have appeared[4–8] on the benzidine rearrangement that occurs when certain hydrazo compounds are heated. Only one reference has been made to the molecularity of this rearrangement. Vecera, Gasparic, and Petranek[6] found no evidence of intermolecular rearrangement in the case of 4-methylhydrazobenzene, and concluded, therefore, that the thermal rearrangement, like the well known acid catalyzed rearrangement, is intramolecular. We have also been occupied with this question. However, since hydrazobenzenes undergo intermolecular dismutation almost completely when heated, we have turned our attention to hydrazo compounds in which this does not occur. It is perhaps, a reasonable assumption that the rearrangement is intramolecular, if a hydrazo compound undergoes thermal rearrangement without evidence of dismutation. This is the case with 2,2′-hydrazonaphthalene, a detailed analysis of which has been given[8] and on which further information is now supplied.

Rearrangement of 2,2′-hydrazonaphthalene has been carried out in the absence of a solvent at 90°, which is below the melting point of the hydrazo compound; at 150°, which is above the melting point of the hydrazo compound; and in cyclohexane solution at 95°. The only products of these rearrangements were 2,2′-diamino-1,1′-binaphthyl and 3,4:5,6-dibenzocarbazole. The rather insoluble and easily isolable 2,2′-azonaphthalene was found in only one of these cases and in very small quantity, 0.9%, resulting, we believe, from air oxidation of the unrearranged hydrazonaphthalene. These results are similar to those obtained earlier with polar solvents.[8]

Vecera's work[6] concerned the rearrangement of 1,1′-hydrazonaphthalene and some hydrazobenzenes in the absence of solvent. In that research the products were not isolated but were identified semiquantitatively by dye-developing a paper chromatogram. The present and earlier results from 2,2′-hydrazonaphthalene show quite clearly that dismutation to azonaphthalene and naphthylamine does not occur. This indicates, therefore, that the rearrangement may be intramolecular, since, during

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[1] Taken in part from the Ph.D. degree thesis of R. L. Snell, Texas Technological College, 1959.

[2] Robert A. Welch Foundation Fellow, 1958–60.

[3] Robert A. Welch Foundation Fellow, 1955–58.

[4] L. G. Krolik and V. O. Lukashevich, *Dok. Akad. Nauk SSSR*, **65**, 37 (1949).

[5] H. J. Shine, *J. Am. Chem. Soc.*, **78**, 4807 (1956).

[6] M. Vecera, T. Gasparic, and J. Petranek, *Chem. & Ind.*, 299 (1957).

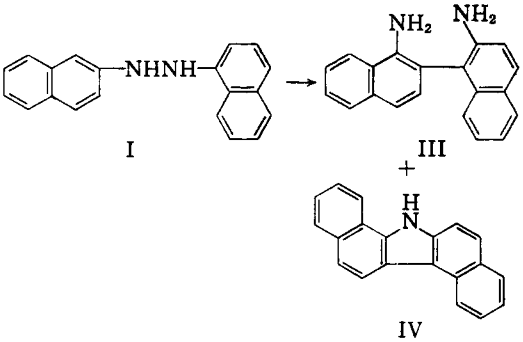
[7] H. J. Shine and R. L. Snell, *Chem. & Ind.*, 706 (1957).

[8] H. J. Shine and J. C. Trisler, *J. Am. Chem. Soc.*, **82**, 4054 (1960).

the rearrangement, if separation into two unassociated halves of the molecule occurred, one might expect that dismutation products would be formed too.

Our results with unsymmetrical hydrazo compounds are in agreement with the above indication. Our initial plans to use mixtures of 2,2′- and 1,1′-hydrazonaphthalene were abandoned when it was found impossible to ascertain, because of titration difficulties with the 1,1′-isomer, if the rates of rearrangement of the two isomers are approximately the same. Therefore, the two systems1,2 ′-hydrazonaphthalene (I) in cyclohexane and 1-phenyl-2-β-naphthylhydrazine (II) in ethanol were chosen. The products of the intramolecular rearrangement of each compound were first prepared by acid-catalyzed rearrangement.

Acid-catalyzed rearrangement of 1,2′-hydrazonaphthalene gave the expected 1,2′-diamino-1′,2-binaphthyl (III) and some 1,2:5,6-dibenzocarbazole (IV). The finding of IV as a product is slightly different from the report of acid-catalyzed rearrangement by Ward and Pearson,[9] which appeared subsequent to the completion of this work and in which it is noted that no product other than the diamine (III) was found.

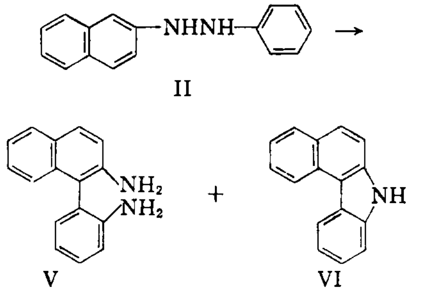


I III IV

The thermal rearrangement of I in cyclohexane solution appeared to go cleanly. The working up of the solution for product isolation, however, proved to be most contrary. Indefinable black solids were obtained from solutions that were at first only slightly colored. This behavior was reminiscent of 1,1′-hydrazonaphthalene.[10] It appears to be associated with a hydrazo group in the *alpha*-position of naphthalene and suggests again[8] that detailed analysis of the rearrangement of the 1,1′-isomer may provide greater insight to the intermediate steps in these rearrangements.

Notwithstanding the difficulties in work up, the only products isolable were the diamine (III) and the carbazole (IV). None of the stable, easily isolable 2,2′-diamino-1,1′-binaphthyl, nor any of the 1,1′-diamino-2,2′-binaphthyl was found.

Recovery of products from rearrangement of 1-phenyl-2-β-naphthylhydrazine (II) was more successful. Acid-catalyzed rearrangement of II gave 1-(2-aminophenyl)-2-naphthylamine (V). This rearrangement and the structure of the diamine have been established before.[11–13] Rearrangement in 95% ethanol at 90° gave only the diamine (V) and a very small amount of 7-benzo[*c*]carbazole (VI).



II V VI

It is interesting to note that II undergoes *ortho*-rearrangement. This type of rearrangement is found mostly in hydrazonaphthalenes and has rarely been observed in hydrazobenzenes. Yet, here, in II, both by acid-catalyzed and thermal rearrangement, the only diamine obtained is of the *ortho*-type. It is remarkable enough that the *ortho* rearrangement should be found mostly in the naphthalene compounds. But, II attracts even greater attention in that even the phenyl portion participates in an *ortho* rearrangement. It is apparent that the naphthalene nucleus controls the geometry of the rearrangement. It is significant also that wherever a hydrazo compound undergoes thermal rearrangement the products obtained are the same as those of acid-catalyzed rearrangement. This would indicate that the two processes involve either similar or identical steps, a conclusion that was recently stated by Shine and Trisler.[8]

EXPERIMENTAL

4-Amino-1,2′-azonaphthalene was prepared by a modification of the procedure of Nietzke and Göttig.[14] To a solution of 36 g. of 2-naphthylamine hydrochloride in 800 ml. of cold water was added slowly 72 ml. of concd. hydrochloric acid. Sodium nitrite, 14 g., was then added with stirring as a saturated solution. The diazonium salt solution was kept cold by adding ice. A solution of 36 g. of 1-naphthylamine hydrochloride in 1.5 l. of water containing 10 ml. of concd. hydrochloric acid was made. Ice was added to this solution and to it was added slowly the iced solution of 2-naphthylamine diazonium chloride. The dark violet-brown mixture was allowed to stand overnight and filtered. The product was dissolved in ethyl alcohol and re-precipitated by adding ammonium hydroxide and water. From two such experiments 125 g. of crude product was obtained. This was crystallized from benzene after treatment with Nuchar (West Virginia Pulp and Paper Co.), giving 85 g. (71.5%), m.p. 140–144° (lit., m.p. 152°,[14] 135–136°[15]).

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[9] E. R. Ward and B. D. Pearson, *J. Chem. Soc.*, 3378 (1959).

[10] Unpublished work of R. L. Snell.

[11] M. P. Cava and J. F. Stucker, *J. Am. Chem. Soc.*, **79**, 1706 (1957).

[12] W. Fuchs and F. Niszel, *Ber.*, **60**, 209 (1927).

[13] H. T. Bucherer and F. Seyde, *J. prakt. Chem.*, [2] **77**, 403 (1908).

[14] R. Nietzke and J. Göttig, *Ber.*, **20**, 612 (1887).

[15] J. W. Cook, C. L. Hewett, E. L. Kennaway, and N. M. Kennaway, *Am. J. Cancer*, **40**, 62 (1940).

1,2'-Azonaphthalene was prepared by a combination of the methods of Nietzke and Göttig[14] and of Cook.[15] To a stirred, boiling solution of 60 g. of 4-amino-1,2'-azonaphthalene in 3 l. of absolute ethyl alcohol was added slowly 180 g. of concd. sulfuric acid. Heating was continued while sufficient saturated sodium nitrite solution was added to dispel the violet color of the solution. A red-brown suspension formed. This was allowed to cool and was then diluted to 8 l. with water. The precipitate was allowed to coagulate and filtered, giving 54 g. of brown solid. Attempts to purify this by crystallization from benzene and from acetic acid were not successful. The solid was distilled at 0.01 mm. giving 28 g. of red solid. This was dissolved in benzene and passed through a column of activated alumina. The benzene solution was finally washed with dilute hydrochloric acid and water, dried, and concentrated to give a red solid. Crystallization from acetic acid gave 15 g. (26%) of 1,2'-azonaphthalene, m.p. 144–145° (lit., m.p. 136°,[14] 144–145°[15]).

1,2'-Hydrazonaphthalene was prepared by reduction with zinc and ammonium chloride solution as described for the 2,2'-isomer.[8] The pale yellow product had m.p. 149–153° dec., using a heating rate of 8°/min.

Acid-catalyzed rearrangement of 1,2'-hydrazonaphthalene. A solution of 1.0753 g. of 1,2'-hydrazonaphthalene in 150 ml. of ethyl alcohol was acidified with 15 ml. of 3*N* hydrochloric acid. After 20 min. the solution was poured into dilute ammonium hydroxide and filtered. The filtrate was extracted with benzene. The filtered solid was dissolved in the benzene solution which was then extracted several times with dilute hydrochloric acid. Neutralization with ammonia gave a solid which was dissolved in ethyl alcohol and again precipitated with dilute ammonium hydroxide solution, giving 0.8627 g. (80.2%), m.p. 146–149°. Sublimation under reduced pressure gave a colorless product, m.p. 146–148°. (lit.,[15] m.p. 150–151°). The benzene solution after acid extraction was washed, dried, and evaporated to give a brown solid. This was purified by sublimation and crystallized from methyl alcohol to give 89.8 mg. (8.9%) of colorless needles, m.p. 235–236°. The melting point of 1,2:5,6-dibenzocarbazole is given as 231°.[16]

Rearrangement of 1,2'-hydrazonaphthalene in cyclohexane solution. Considerable difficulty was encountered in isolating the products of this rearrangement. Although the rearrangement solution itself was only slightly colored (orange) after prolonged heating, the formation of black tars plagued most of the subsequent operations. The following results were obtained after three other attempts had failed. The hydrazonaphthalene, 0.9168 g., was sealed under vacuum in a flask with 150 ml. of cyclohexane after four degassing cycles. The cyclohexane was distilled and passed through activated alumina prior to use. The hydrazo solution was kept in a bath at 95° for 10 days and then cooled. A cream colored solid precipitated. The flask was opened and the solid was filtered. From 0.6259 g. of solid (68% recovery) successive crystallizations from benzene and then petroleum ether (b.p. 40–65°) gave 43.7 mg. (4.8%) of 1,2'-diamino-2,1'-binaphthyl, m.p. 144–148°, and 0.1488 g. (17.3%) of 1,2:5,6-dibenzocarbazole, m.p. 235°. Evaporation of the cyclohexane filtrate gave 0.1928 g. (21% recovery) of solid. Successive crystallizations from benzene-petroleum ether mixtures gave 37.4 mg. (4.1%) of the 1,2'-diamine. No other products could be isolated.

Rearrangement of solid 2,2'-hydrazonaphthalene at 90°. The sample of the hydrazonaphthalene, 0.6024 g., prepared as described earlier,[8] was weighed in a glass boat and sealed in a glass flask after degassing on the diffusion pump line. The flask was immersed in a bath at 95° for 72 hr. The originally almost colorless hydrazonaphthalene was then pale yellow. Some small yellow droplets were visible on the wall of the flask, and from them were growing small colorless needles. The flask was constructed so as to allow purging with nitrogen when opened. The nitrogen purge was bubbled through an aliquot of standardized hydrochloric acid in order to determine the ammonia formed during rearrangement to the dibenzocarbazole. Back titration of the acid showed that 8.7 mg. of ammonia (equivalent to conversion of 24.1% of the hydrazo to carbazole) had been collected. The flask contents were taken up in benzene and made up to 100 ml. One ml. of this solution was analyzed for hydrazo content using titanium trichloride, standardized with potassium dichromate, and Bindschedler's Green. The total unrearranged hydrazonaphthalene was 64 mg. The remaining benzene solution was extracted with dilute hydrochloric acid. Precipitation of the acid solution with ammonium hydroxide gave 0.4636 g. (77%) of 2,2'-diamino-1,1'-binaphthyl, m.p. 188–190°. The benzene solution was dried over magnesium sulfate and evaporated, giving 0.1337 g. (22%) of brown solid. Crystallization from a small volume of ethanol gave 5.6 mg. (0.9%) of crude 2,2'-azonaphthalene, m.p. 204–206°, as bright orange plates. Evaporation of the ethanol solution to dryness and crystallization from ligroin gave 3,4:5,6-dibenzocarbazole, colorless needles, m.p. 155–156°.

Rearrangement of 2,2'-hydrazonaphthalene at 150°. A sample, 0.6862 g., of the hydrazo compound was sealed as described for the case at 90°. The vessel was kept at 150° for 2.5 hr. At that time the wall of the vessel had become covered with small droplets of yellow oil. The characteristic red color of the azonaphthalene was not evident. Ammonia was not determined in this experiment. The flask contents were dissolved in benzene and treated as described before. No hydrazonaphthalene remained. From the acid solution was obtained 0.4886 g. (71.5%) of 2,2'-diamino-1,1'-binaphthyl, m.p. 191–192°. From the benzene solution was obtained 0.1722 g. (25.1%) of a yellow oily residue. Crystallization from ligroin gave the low-melting form[6] of 3,4:5,6-dibenzocarbazole, m.p. 120–123°.

Rearrangement of 2,2'-hydrazonaphthalene in cyclohexane at 95°. The cyclohexane used was Matheson, Coleman, and Bell technical grade. It was fractionally distilled and passed through activated alumina before use. The hydrazonaphthalene, 0.754 g., and cyclohexane, 175 ml., were placed in a nitrogen purged flask, degassed three times, and sealed. The sealed flask was kept in a bath at 95° for 69 hr., after which time the solution was pale yellow. The flask was opened and purged with nitrogen for ammonia determination, giving ammonia equivalent to 3.4% of the hydrazonaphthalene going to carbazole. The cyclohexane solution was cooled and the white solid (2,2'-diamino-1,1'-binaphthyl), 0.6016 g. (79.8%), which precipitated had m.p. 190–190.5°. The cyclohexane filtrate was stirred with aqueous potassium ferricyanide solution after first ascertaining that neither 2,2'-diamino-1,1'-binaphthyl nor 3,4:5,6-dibenzocarbazole were affected by this treatment. The washed cyclohexane solution was extracted with dilute hydrochloric acid which yielded a further 63.3 mg. (8.4%) of the 2,2'-diamine, m.p. 189.5–191.5°. The cyclohexane layer was washed, dried, and evaporated. The residue was dissolved in ethanol and treated with charcoal; evaporation of the ethanol gave 52.8 mg. (7.4%) of crude 3,4:5,6-dibenzocarbazole. Recrystallization from ligroin gave m.p. 153–156°.

2-Benzeneazonaphthalene was prepared by the method of Fierz-David.[17] Contrary to the description given by these authors, the crude product obtained by reaction of phenylhydroxylamine and thionyl-6-naphthylamine was obtained repeatedly as a viscous oil. Trituration with ethanol gave a suspension of an amber solid. Dilution of the suspension with water caused the separation of an oil. The amber solid was taken up in a minimum amount of 95% ethanol and decanted from the oil. Concentration of the ethanol solution

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(16) F. R. Japp and W. Maitland, *J. Chem. Soc.*, **83**, 267 (1903).

(17) H. E. Fierz-David, L. Blangley, and E. Merian, *Helv. Chim. Acta*, **34**, 846 (1951).

gave crude 2-benzeneazonaphthalene, m.p. 77–80°. Crystallization from 70% ethanol gave a 19% yield of product with m.p. 82–83° (lit.,[17] m.p. 84°). The phenylhydroxylamine was prepared from nitrobenzene.[18]

1-Phenyl-2-β-naphthylhydrazine was prepared by the reduction of 2-benzeneazonaphthalene in alcohol solution with aqueous ammonium chloride and zinc. The hydrazo compound was isolated as described earlier for 2,2′-hydrazonaphthalene.[8] The slightly yellow product had m.p. 104°. This method of reducing 2-benzeneazonaphthalene did not produce the hydrazo compound initially as an oil[11]; instead, a flocculent white solid was formed which became slightly yellow on filtration and washing.

Rearrangement of the hydrazo compound in aqueous ethanol solution with hydrochloric acid gave an authentic specimen of 1-(2-aminophenyl)-2-naphthylamine, m.p. 152–153° after recrystallization from aqueous ethanol (lit.,[11] m.p. 154–155°).

An authentic specimen of 7-benzo[*c*]carbazole was prepared by heating a sample of the above diamine with concd. hydrochloric acid in a sealed tube at 145–150° for 5 hr. The solids in the tube were then washed well with dilute hydrochloric acid and recrystallized from ethanol after treatment with charcoal. The product had m.p. 133–134° (lit.,[11] m.p. 135°).

Thermal rearrangement of 1-phenyl-2-β-naphthylhydrazine. The following procedure and results are representative of five separate experiments. A flask containing 0.677 g. of the hydrazo compound and 50 ml. of 95% ethanol under nitrogen was degassed on a diffusion pump line by two cycles of the customary freezing-thawing technique. The flask was sealed while under vacuum and placed in an oil bath at 90° for 7 days. It was then cooled and opened. The solution was transferred to a volumetric flask and made up to 100 ml. with ethanol. Analysis by the titanium trichloride–Bindschedler's Green method showed that 0.264 g. of hydrazo compound remained. The alcohol solution was made alkaline with solid sodium hydroxide, cooled in ice, and a gentle stream of oxygen was passed through it for 2 hr. The solution was concentrated by distillation and poured into a large volume of water. The precipitated solid was filtered, dried, and dissolved in benzene. Extraction of the benzene solution with dilute hydrochloric acid and neutralization of the acid solution gave 0.314 g. of product, m.p. 148–150°. Three crops from crystallization of this product gave a total of 0.274 g. of 1-(2-aminophenyl)-2-naphthylamine, m.p. 152–153°. No 2,2′-diamino-1,1′-binaphthyl, m.p. 190–191°, was found. The filtrate remaining from crystallizations of the crude diamine had an ultraviolet spectrum similar to that of a mixture of 1-(2-aminophenyl)-2-naphthylamine and 7-benzo[*c*]carbazole. Spectroscopic analysis on this basis gave 5 mg. of diamine and 0.7 mg. of carbazole in the filtrate. Thus, approximately 34 mg. was lost in crystallizations. There was no evidence in the ultraviolet spectrum in the region of 283 mμ to suggest the presence of benzidine, m.p. 127°. In this region benzidine has a broad pronounced absorption, and the spectrum recorded had the characteristic shape of that of 1-(2-aminophenyl)-2-naphthylamine. It is to be noted that the ultraviolet spectrum of 2,2′-diamino-1,1′-binaphthyl is almost identical with that of 1-(2-aminophenyl)-2-naphthylamine. The absence of the former in the rearrangement products is concluded on the basis of not finding the higher melting less soluble diamine by crystallization.

The benzene solution remaining after acid extraction of diamine was washed, dried and distilled to small volume. Evaporation to dryness under a stream of nitrogen gave 0.272 g. of solid. Successive crystallizations from 95% ethanol gave five crops of 2-benzeneazonaphthalene, each with m.p. within the range 80–82°, weighing a total of 0.211 g. The residual alcohol solution appeared, from its ultraviolet spectrum, to contain a mixture of 2-benzeneazonaphthalene and 7-benzo[*c*]carbazole. Spectroscopic analysis on this basis gave 28.2 mg. of 2-benzeneazonaphthalene and 8.2 mg. of 7-benzo[*c*]carbazole in the residual ethanol solution. Thus, approximately 25 mg. was lost in crystallizations.

In summary, the acid-soluble and acid-insoluble solids accounted for 87% of the original 1-phenyl-2-β-naphthylhydrazine, and appeared to be composed solely of the products of intramolecular rearrangement. The yields of quantitatively identified products, based on the amount of hydrazo compound which rearranged, were 1-(2-aminophenyl)-2-naphthylamine 77.2% and 7-benzo[*c*]carbazole 2.5%.

In the course of the spectroscopic work the following maxima were recorded. For 1-(2-aminophenyl)-2-naphthylamine: λ 239.5 mμ (log ε 4.82), 283.2 (3.98), and 342.5 (3.52). For 7-benzo[*c*]carbazole: λ 263.5 mμ (log ε 4.76), 284.5 (4.08), 325 (4.19), and 344.5 (3.75). For 2-benzeneazonaphthalene: λ 265 mμ (log ε 4.06), 276.5 (4.12), 287.5 (4.12), and 328 (4.32). The diamine had a pronounced shoulder at 294 mμ, while the carbazole had very weak shoulders at 272 and 312 mμ.

Acknowledgment. This work was generously supported by the Robert A. Welch Foundation, Houston, Tex.

LUBBOCK, TEX.

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[18] A. I. Vogel, *A Textbook of Practical Organic Chemistry*, 2nd ed., Longmans, Green and Co., New York, N. Y., 1954, p. 602.

[CONTRIBUTION FROM THE RESEARCH DIVISION, ABBOTT LABORATORIES]

The Preparation of 1-Halobenzoyl-2-isopropylhydrazines. Selective Hydrogenation of N=CH Function in the Presence of Aromatic Halogen

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Selective reduction of N=C function in the presence of aromatically bound halogen was carried out with platinum on carbon catalyst. Successful reductions of ten compounds are reported, several of which contain the more labile halogens bromine and iodine.

There are many descriptions in the literature of hydrogenation of reducible groups in the presence of aromatically bound halogen. In most of the reported work little emphasis is placed on selectivity. In many instances the halogen present is chlorine and the catalyst and reaction conditions do not favor dehalogenation.

The effect of strong bases in catalytic dehalogena-