Ethyl azodicarboxylate has been shown to form adducts with natural rubber[9] and with 2-methyl-2-butene.[11] With disazodicarboxylates[12] natural and synthetic rubbers reacted rapidly at relatively low temperatures to produce good vulcanizates without the aid of other ingredients.

The previous discussion concerning natural rubber can also be applied with adaptations to the cross-linking of GR-S taking into account additional factors associated with the polymer structure such as units derived from 1,2-polymerization and the labile *t*-hydrogens in the styrene units.

Relationship between Dimaleimide Structure and Vulcanizate Properties.—Both aliphatic and aromatic dimaleimides gave similar vulcanizates with natural rubber in relation to the properties of resilience and compression set, whereas the aliphatic ones, on a molar basis, produced slightly lower modulus values. The most striking difference is that those dimaleimides derived from diamines possessing any degree of aliphatic hydrocarbon character yielded vulcanizates with particularly poor tensile strength. The underlying cause may reside in differences in flexibility of the crosslink structure, or in chemical participation of the methylene groups during vulcanization so as to affect tensile strength adversely.

Experimental[13]

Materials.—The dimaleimides, dimaleamic acids and N-phenylmaleimide were prepared as previously described.[2,4] Other materials used were the highest purity commercial grades available.

Testing Procedure.—The procedure followed is reported elsewhere.[2]

Acknowledgment.—We wish to acknowledge the support of this work by the U. S. Military Medical Supply Agency.

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(11) N. Rabjohn, THIS JOURNAL, **70**, 1181 (1948).

(12) P. J. Flory, N. Rabjohn and M. C. Shaffer, *J. Polymer Sci.*, **4**, 225 (1949); **4**, 435 (1949).

(13) The compounding, curing and testing were done by Mr. Ralph Hodous.

CLEVELAND 6, OHIO

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, POLYTECHNIC INSTITUTE OF BROOKLYN]

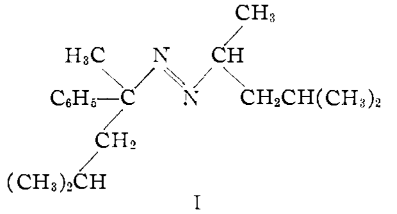
Azo Compounds. XXVIII.[1] A Decomposition Study of α,α-Methylisobutylbenzylazo-2-(4-methylpentane)

BY C. G. OVERBERGER AND A. V. DIGIULIO[2]

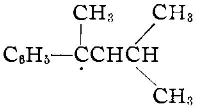
RECEIVED SEPTEMBER 19, 1958

The decomposition of I in xylene gave a mixture of products resulting from combination and disproportionation reactions of α,α-methylisobutylbenzyl radical (II) and the methylisobutylcarbinyl radical (III), as well as products derived from solvent interaction with these radicals. The lack of formation of any coupled product from the radical $(C_6H_5)C(CH_3)(CH_2CH)(CH_3)_2)$ and the low yield of unsymmetrical coupled product may be due to an unfavorable steric factor.

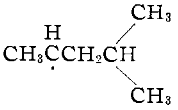
I. Products.—A 30% and a 12% solution of the azo compound I was decomposed in xylene at 110° yielding a mixture of the same products, with slightly varying yields of the individual products. The mixture was shown to consist of product principally derived from the α,α-methylisobutylbenzyl (II) and the methylisobutylcarbinyl (III) radicals as well as product which also incorporated solvent molecules.



I



II



III

The products were initially separated into four main fractions consisting of mixtures as shown in Table I (see Experimental section) by two fractionations. The initial separation into main fractions, Table I, indicated a total yield of products of 105 and 109%, respectively, which indicated that solvent radicals were being generated and were interacting with the radicals generated by the decomposition of the azo compound or possibly with other solvent radicals. The initial separation in both decompositions yielded approximately 72% of product, respectively, derived solely from radicals derived from the azo compound. The yield of product derived from the solvent and the azo radicals totalled approximately 32 and 35%, respectively.

The initial fraction consisted chiefly of 2-methylpentane and the two possible methylpentenes, derived from radical III. The infrared spectrum indicated the presence of olefin. Hydrogenation of this fraction indicated the presence of ~4.3% of the olefins in the decomposition of a 12% solution and ~15% olefin in the decomposition of a 30% solution. The refractive index and infrared spectrum of the hydrogenated product were identical with those of a pure commercial sample of 2-methylpentane, indicating that the above-mentioned isomers were the only three possible constituents of this fraction. The low olefin content of

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(1) This is the 28th in a series of papers concerned with the preparation and decomposition of azo compounds. For the previous paper in this series, see C. G. Overberger and A. V. DiGiulio, THIS JOURNAL, **80**, 6562 (1958).

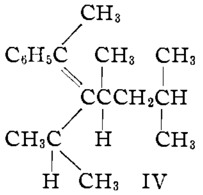
(2) This paper comprises a portion of a thesis presented by A. V. DiGiulio in partial fulfillment of the requirements for the degree of Doctor of Philosophy in the Graduate School of the Polytechnic Institute of Brooklyn.

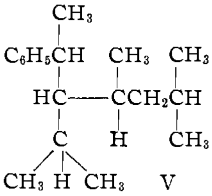
the 12% decomposition fraction may be due to a statistical factor, since the large concentration of the xylene molecules would facilitate the hydrogen abstraction by the methylisobutylcarbinyl radicals which escape the cage, thus decreasing the disproportionation effect on the methylisobutylcarbinyl radicals.

The second fraction consisted of 2-methyl-4-phenylpentane and possibly both styrenes, α-methyl-β-isopropylstyrene and α-isobutylstyrene derived from radical II. The presence of olefins was indicated by the decoloration of a bromine solution, as well as by olefin absorptions in the infrared spectrum and by the similarity of the ultraviolet spectrum to that of α-methyl-β-isopropylstyrene. The amounts of olefin present were, respectively, ~37 and ~45%. The boiling point, refractive index and infrared spectrum of the hydrogenated material were identical with those of 2-methyl-4-phenylpentane obtained by another route, and the ultraviolet spectra were nearly identical.

The third fraction consisted mostly of the unsymmetrical coupled product 2,4,5,7 tetramethyl-4-phenyloctane along with products which can arise by the attack of the methylisobutylcarbinyl radical III on the double bond of α-methyl-β-isopropylstyrene. Two other products which may also be present are corresponding isomers derived in a like manner by attack of the alkyl radical III on the disproportionated product α-isobutylstyrene.

The ultraviolet spectrum indicated the presence of a possible styrene type moiety for this fraction. Unsaturation was also indicated by the decoloration of a bromine–chloroform solution. However, the infrared spectrum was devoid of any characteristic unsaturation bands at 6.13, 10.05, 11.18 and 12.85 μ characteristic of α-methyl-β-isopropylstyrene. The absence of such bands may be indicative of the unsaturated moiety being of the type IV, $R_1R_2C{=}CR_3R_4$, or the corresponding trisubstituted ethylene isomer of IV of the type $R_1R_2C{=}CHR_3$.[3a] Two reasonable structures for these hydrocarbon products are represented in formulas IV and V. The elemental analyses corresponded to an empirical formula $C_{18}H_{30}$ and hydrogenation indicated the presence of ~18% olefin (calculated on basis of $C_{18}H_{28}$). The elemental analyses of the hydrogenated product corresponded to the empirical formula $C_{18}H_{30}$ and the ultraviolet spectrum was now characteristic of a monosubstituted benzene. Removal of the olefin by treatment with bromine in chloroform and chromatography gave a product, the ultraviolet spectrum of which gave a typical monosubstituted benzene spectrum, which differed only slightly in the values of the maximum when compared to the hydrogenated product. Also the elemental analyses and molecular weight of this product were consistent for the formula $C_{18}H_{30}$. The identity of this fraction was further established by comparison with a similar fraction obtained from the reaction of the Grignard reagent of 2-bromo-4-methylpentane with 2-bromo-2-phenyl-4-methylpentane. From this reaction was obtained a product whose boiling point and refractive index was the same as the above fraction, and whose ultraviolet spectrum indicated the presence of a styrene-type moiety. Treatment with bromine in chloroform and chromatography gave a product whose refractive index was the same as the product derived from the decomposition of the azo compound. The ultraviolet spectrum was also typical of a monosubstituted benzene and varied only slightly in the values of the extinction coefficient when compared with the product derived from the azo compound. The infrared spectrum on the other hand was identical with the spectrum of the product obtained from the decomposition of the azo compound. It follows then that the azo decomposition product, fraction III, must be essentially composed of an α,α-methylisobutylbenzyl fragment (II) and a methylisobutylcarbinyl fragment (III). Although the infrared spectrum of the purified product from the Grignard reaction and the decomposition product are the same, a comparison of the ultraviolet data, although qualitatively in agreement, shows small differences and suggests that perhaps more than one saturated substituted benzene may be present, the differing concentrations of which may be the cause in the difference of absorption in the three corresponding maxima of the compounds derived from the two sources.


IV


V

The fourth fraction appears to be products derived from the solvent although the structures were not proved rigorously. Evidence for this suggestion is presented in the Experimental section. Tentative structures for these products are available from the second author.

The higher boiling fractions were not characterized, but are believed to be products formed from solvent interactions with radicals derived from the azo compound. The presence of the radicals derived from the azo compound is indicated by the presence of the *gem*-dimethyl band at 7.22 and 7.33 μ.[3b]

In any event it is clear from the foregoing discussion that the decomposition of α,α-methylisobutylbenzylazo-2-(4-methylpentane) yields for the most part products consistent with our knowledge of radical reactions in solution.

The high yield of the first 2-methylpentane fraction (31 and 46%), the second 2-methyl-4-phenylpentane fraction (51 and 38%) and the moderate yield of the third 2,4,5,7-tetramethyl-4-phenyloctane fraction (16.6% and 18%) indicate that a considerable part of the radicals escape the cage. The moderate yield of the unsymmetrical coupled product may be due to an unfavorable steric factor due to the presence of the bulky groups present in both radicals.

Benzyl-type radicals such as cumyl are unreactive and usually couple easily. However, the

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(3) (a) L. Bellamy, "The Infrared Spectra of Complex Molecules," John Wiley and Sons, Inc., New York, N. Y., 1954, p. 32; (b) p. 24.

TABLE I

PRODUCTS OF DECOMPOSITION IN XYLENE OF α,α'-METHYLISOBUTYLBENZYLAZO-2-(4-METHYLPENTANE)

	Fractionation of 30% solution					Fractionation of 12% solution				
Fraction[a]	I Yield, %	II Yield, %	n^t_D	t, °C.	Olefin, %	I Yield, %	II Yield, %	n^t_D	t, °C.	Olefin, %
I	10.9[a]	10.9	1.3741	26	15	16.2	16.2	1.3710	25	4.3
II	36.5	33 4.7[b]	1.4933	24.5	37	34.1	24.9 10.2[b]	1.4956	24	45[c]
III	23.9	16.6 5.6	1.4915	26	17.5	20.0 6.2[b]	17.9	1.4922	25	18[c]
IV	13.0	5.7 1.8[b]	1.5322	25.5		10.7 3.5[b]	9.8 0.8[b]	1.5342	24.3	
Unidentified	20.4					18.1				
Total	104.7					108.8				

[a] This yield is probably low due to high volatility and improper precautions in isolating this fraction. [b] Intermediate fractions. [c] Estimated on basis of refractive index.

benzyl-type radical II in this case apparently does not undergo the low energy combination reaction with itself, or if it does, only to a small undetectable extent. A reasonable explanation would be the enhanced steric effcet which may increase the activation energy of the radical combination reaction. Another factor responsible for the apparent lack of formation of the symmetrical coupled product derived from radical II would be the possible interaction of the radical II with the less sterically hindered but quite stable benzyl-type radical derived from the solvent to yield products such as were obtained in fraction IV.

Experimental[4]

The product analyses are reported on two separate decompositions. A 12 and a 30% solution of the azo compound in xylene solvent gave the same product fractions in slightly differing yields (see Table I). The experimental procedure describes the 12% decomposition results, but some of the work on the 30% decomposition is incorprated to give a more complete picture, since all the experiments were not repeated in both cases when the fractions in both cases were obviously the same.

Decomposition of a 12% Solution of α,α-Methylisobutylbenzylazo-2-(4-methyl)-pentane at 110°.—Decomposition of 30.4 g. (0.111 mole) of I in 250 ml. of xylene at 110° evolved 96% gas. In a trap cooled by Dry Ice, interspersed between the reaction flask and the gas receiver was collected 0.82 g., $n^{21.8}_D$ 1.3678, of volatile water-white liquid. The initial distillation of this decomposition was carried out through a 10-inch spiral column, precaution being taken to cool the receiver and the distillation head with Dry Ice to minimize the loss of the low boiling component. In this fashion an additional 3.6 g., n^{25}_D 1.3710, of low boiling component was obtained, the temperature not registering over 45° due to the non-equilibrium conditions although the bath temperature had attained 120°. The total yield of low boiling component was 4.42 g. (16.2%). The infrared spectrum showed olefin absorption at 5.49, 6.90, 10.08 and 10.95 μ.

Hydrogenation of 0.77 g., of the trapped low boiling material in a 5-ml. flask in the presence of 10% palladium-on-charcoal with cooling indicated the presence of only 4.3% of the methylpentenes. (The 30% decomposition gave a yield of 15% olefin.) By connecting the original hydrogenation flask by means of an adapter to a small trap immersed in a Dry Ice mixture, the hydrogenated material was volatized into the trap by warming with hot water; 0.6 g., n^{24}_D 1.3689. This refractive index was identical with that of a commercial pure grade of 2-methylpentane, and the infrared spectrum of the hydrogenated decomposition product was identical in every detail with the spectrum of the known compound.

After the careful removal of the solvent through a spiral column, 6 fractions were obtained using an 8-inch center tube column; 29.7 g. (109%).

Redistillation of the first fraction, 9.3 g. (34.1%), from the above fractionation yielded 6.79 g. (24.9%), b.p. 197–214°, n^{24}_D 1.4956, a mixture of 2-methyl-4-phenylpentane, α-methyl-β-isopropylstyrene and α-isobutylstyrene; a second fraction, 1.84 g., (6.74%), b.p. 214–248°, n^{25}_D 1.4978; and a residue of 0.5 g., n^{25}_D 1.4895, which was added to fraction 2. A similar unsaturated fraction was obtained in the 30% decomposition.

This fraction indicated unsaturation by decoloration with bromine. The ultraviolet spectrum in absolute methanol was similar to that of α-methyl-β-isopropylstyrene showing a λ_{max} at 241 mμ, and a minimum at 223.5 mμ. The infrared spectrum indicated the presence of olefin absorption at 6.14, 11.17 and 12.86 μ and was similar to that of α-methyl-β-isopropylstyrene.

Hydrogenation of 0.976 g. of the unsaturated fraction in 20 ml. of ethyl acetate in the presence of 0.1 g. of 30% palladium-on-charcoal required 0.00227 mole of hydrogen, indicating that 37% of the sample consisted of the unsaturated moiety. After the removal of the ethyl acetate, 0.68 g., b.p. 200–201°, n^{25}_D 1.4828, of the hydrocarbon 2-methyl-4-phenylpentane and 0.1 g., of residual material, n^{25}_D 1.4849, was recovered. The infrared spectrum of the hydrogenated material was identical with the spectrum of 2-methyl-4-phenylpentane prepared by another route (see later Experimental section). The ultraviolet spectrum in absolute methanol showed only minor differences and had the following maxima: λ 248 mμ, ϵ 219; 252.5 mμ, ϵ 241; 258 mμ, ϵ 247; 261 mμ, ϵ 238; 264 mμ, ϵ 184; 267.5 mμ, ϵ 166.

Redistillation of the second fraction, from the original six fractions plus 0.5 g. residue from preceding fractionation, 5.95 g., at 0.7 mm. gave 4.89 g. (17.9%), b.p. 96–98°, n^{25}_D 1.4922. The ultraviolet spectrum of this fraction in absolute methanol was indicative of a styrene-type moiety with λ_{max} 246 mμ and an ϵ value of 1100 calculated on the basis of the saturated monosubstituted benzene, 2,4,5,7 tetramethyl-4-phenyloctane ($C_{18}H_{30}$). Unsaturation was also indicated by the decoloration of a bromine solution.

Anal. Calcd. for $C_{18}H_{30}$: C, 87.73; H, 12.27. Found: C, 87.69; H, 12.03.

Hydrogenation of 0.850 g. in 15 ml. of ethyl acetate in the presence of 0.1 g. of 30% palladium-on-charcoal required 0.000608 mole of hydrogen, indicating the presence of 17.5% of the possible unsaturated moiety ($C_{18}H_{28}$). After the removal of the solvent, the product was fractionated to give 0.67 g., b.p. 86–89° (0.2 mm.), n^{25}_D 1.4880. The ultraviolet spectrum in absolute methanol was characteristic of a monosubstituted benzene: λ 253.5 mμ, ϵ 211; 259 mμ, ϵ 250; 264.5 mμ, ϵ 221.

Anal. Calcd. for $C_{18}H_{30}$: C, 87.73; H, 12.27. Found: C, 87.94; H, 12.31.

In order to remove the olefin, the following procedure was followed. To a solution of 1.06 g. of material, n^{26}_D 1.4915, in 20 ml. of chloroform at 5° was added 9 ml. of 5% bromine–chloroform solution over a period of 20 minutes until the color was yellow-orange. After standing for 30 minutes with occasional shaking, the solvent and excess bromine was removed by bubbling nitrogen through the solution. The residue, 1.5 ml., was decanted off from approximately 2 drops of a heavy yellow oil and dissolved in 250 ml. of petroleum ether (b.p. 38–47°) and passed through a 1.5 inch diameter column packed with 100 g. of neutral alu-

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[4] Analyses and Molecular Weights by Dr. Schwarzkopf, New York, N. Y.

mina. The column eluate was collected and evaporated on the steam-bath, giving a total of 0.95 g. of colorless liquid, n^{26}D 1.4892. The ultraviolet spectrum in absolute methanol showed the absorptions: λ 253.5 mμ, ϵ 202; 259 mμ, ϵ 229; 265 mμ, ϵ 190.

Anal. Calcd. for $C_{18}H_{30}$: C, 87.73; H, 12.27; mol. wt., 246. Found: C, 88.13; H, 12.08; mol. wt. (Rast), 264.

Chromatography of 2.40 g. of a fraction, $n^{25.2}$D 1.4917, from the decomposition of a 12% solution gave a similar product. The infrared spectrum was identical with the spectrum of the product obtained in the 30% decomposition. Again the elemental analyses indicated that the compound was probably 2,4,5,7-tetramethyl-4-phenyloctane.

Anal. Calcd. for $C_{18}H_{30}$: C, 87.73; H, 12.27. Found: C, 87.93; H, 12.29.

The total yield of unseparated products derived only from the radicals produced from the decomposed azo compound was 19.17 g. (70.2%). The identified fractions totaled 59%, the remaining 11% being the intermediate mixtures of the known fractions.

Redistillation of the fourth fraction from the original six fractions, 2.91 g. (10.7%), gave 2.68 g. (9.8%) of light yellow product, b.p. 135–137° (0.4 mm.), $n^{24.3}$D 1.5342. The corresponding fraction in the 30% decomposition indicated the presence of unsaturation by the uptake of bromine, and also the ultraviolet spectrum showed a λ_{max} 246 mμ, indicating the presence of a styrene-type moiety, the ϵ-value being too high for a pure bibenzyl-type compound. The presence of an isopropyl group was indicated by the split adsorption at 7.22 and 7.32 μ in the infrared.

Treatment of 1.70 g. of this redistilled fraction with bromine in chloroform followed by chromatography as described previously, gave the fractions: (1) 0.03 g., $n^{24.5}$D 1.4872; (2) 0.45 g., $n^{24.3}$D 1.5378; (3-5) 0.63 g., n^{24}D 1.5400; (6-8) 0.15 g., $n^{23.4}$D 1.5389. Fraction 3-5 gave an ultraviolet spectrum with the following maxima in absolute methanol: 259.5 mμ, ϵ 472, 524; 265 mμ, ϵ 484, 536; 273 mμ, ϵ 300, 332. These data and analyses strongly indicate bibenzyl structures derived from the coupling of the radical II with the solvent xylene ($C_{20}H_{26}$) or the coupling of the radical II with a bibenzyl compound formed from the solvent ($C_{22}H_{30}$).

Anal. Calcd. for $C_{20}H_{26}$: C, 90.16; H, 9.84; mol. wt., 266.41. Calcd. for $C_{22}H_{30}$: C, 89.73; H, 10.27; mol. wt., 294.46. Found: C, 90.14; H, 10.16; mol. wt. (Rast), 267.

Preparation of 2-Methyl-4-phenylpentane.—Methylisobutylphenylcarbinol was prepared according to the procedure of Bodroux and Taboury[5] from methyl isobutyl ketone and phenylmagnesium bromide, b.p. 76° (2 mm.), n^{24}D 1.5057. The infrared spectrum showed a strong OH band at 2.90 μ and weak olefin bands at 6.13 and 12.85 μ (b.p. 125–128° (18 mm.), n^{16}D 1.5157, contaminated with α-methyl-β-iso-propylstyrene),[5] b.p. 112–114° (12 mm.), n^{20}D 1.5062[6]).

Treatment of 29 g. (0.17 mole) of the above carbinol with dry hydrogen chloride gas at −8 to 0° according to the method of Klages[7a] gave the crude 2-chloro-2-phenyl-4-methylpentane which after several washings with ice-water, was dissolved in 200 ml. of ether. The ether solution was washed thoroughly with water and sodium bicarbonate solution and dried over sodium sulfate and stored in the freezer. An attempted preparation of 2,4,5,7-tetramethyl-4,5-diphenyloctane from the reaction of the chloro compound with lithium in ether was unsuccessful. After the removal of the solvent, distillation of the residue, which was accompanied by considerable evolution of hydrogen chloride gas, gave 14 g. (54%) of α-methyl-β-isopropylstyrene, b.p. 210–212°, $n^{20.2}$D 1.5167 (b.p. 210–211° (755 mm.), n^{20}D 1.5162 by reduction of trimethylphenylallene with sodium and alcohol[7b]).

The ultraviolet spectrum showed a λ_{max} 242 mμ, ϵ 10,700 and a minimum at 223.5 mμ. The infrared spectrum indicated olefinic absorption at 6.13, 10.05, 11.18 and 12.85 μ.

Hydrogenation of the above styrene in ethyl acetate in the presence of 10% palladium-on-charcoal gave the water-white 2-methyl-4-phenylpentane, b.p. 198–200°, n^{18}D 1.4858 (b.p. 197°, n^{15}D 1.4876 by sodium–alcohol reduction of α-methyl-β-isopropylstyrene[7b]; 203.0–204.8° (756 mm.), n^{20}D 1.4849[6]). The ultraviolet spectrum in absolute methanol was characteristic of a monosubstituted benzene having the following maxima: 248 mμ, ϵ 179; 253 mμ, ϵ 225; 258.5 mμ, ϵ 249; 261 mμ, ϵ 244; 264 mμ, ϵ 187 and 267.5 mμ, ϵ 178.

Preparation of the Unsymmetrical Hydrocarbon 2,4,5,7-Tetramethyl-4-phenyloctane.—The compound 2-bromo-4-methylpentane was prepared from methylisobutylcarbinol and 48% hydrobromic acid in 38% yield, b.p. 46–47° (35 mm.), n^{24}D 1.4406, according to a procedure described[8] for the preparation of some secondary alkyl bromides (55%, b.p. 40–50° (25 mm.), methylisobutylcarbinol and phosphorus tribromide[9]).

Crude 2-bromo-2-phenyl-4-methylpentane was prepared by passing dry hydrogen bromide gas into 100 g. of crude methylisobutylphenylcarbinol at −5 to −20° in the same manner as used for the preparation of the corresponding chloro compound. The compound appears to be unstable and must be stored in the freezer, since at room temperature hydrogen bromide gas was slowly evolved.

The Grignard reagent from 2-bromo-4-methylpentane was readily prepared from 4.9 g. (0.2 mole) of sublimed magnesium shavings in 70 ml. of anhydrous ether and 33.1 g. (0.2 mole) of 2-bromo-4-methylpentane in 80 ml. of anhydrous ether. To the filtered Grignard solution was added dried ethereal solution of crude 2-bromo-2-phenyl-4-methylpentane containing approximately 29 g. (0.12 mole) of the bromo compound in 30 minutes without cooling (gentle refluxing). After stirring for an additional 1.5 hr., the mixture was decomposed with the careful addition of 23 ml. of saturated ammonium chloride solution. The ether solution was separated from the magnesium salts by filtration and the salts extracted with two 30-ml. portions of anhydrous ether. Removal of the solvent left 29.5 g. of a light yellow liquid residue. Distillation gave 1.5 g. of a fraction boiling at 89–92° (0.2 mm.), n^{24}D 1.4920. This material decolorized a bromine–chloroform solution, and the ultraviolet spectrum in absolute methanol was indicative of the presence of a styrene-type moiety, λ_{max} 248 mμ, ϵ 1760, a value which was too high for the saturated monosubstituted benzene, $C_{18}H_{30}$.

This material, 1.0 g., was treated with bromine in chloroform as described above and chromatography of the resultant residue gave 0.5 g. of product, n^{27}D 1.4893. The infrared spectrum of the chromatographed material was identical with that obtained for the analogously treated material obtained in the 12 and 30% decomposition of the azo compound. The ultraviolet spectrum in absolute methanol showed a principal λ_{max} 259 mμ, ϵ 251, a minor maximum at 253.5 mμ, ϵ 240, and a small peak at 265 mμ, ϵ 198.

Acknowledgment.—We wish to gratefully acknowledge the support of this work by the National Science Foundation, Grant NSF-1453. We also wish to thank Dr. R. G. Hiskey for his advice and help.

Brooklyn 1, N. Y.

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