Journal of Organometallic Chemistry
Elsevier Sequoia S.A., Lausanne
Printed in The Netherlands

REACTIONS AND THERMAL DECOMPOSITION OF ORGANOLITHIUM COMPOUNDS DERIVED FROM 4,4'-DIBROMOOCTAFLUOROBIPHENYL

R. A. CONTIGIANI*, HECTOR E. BERTORELLO AND M. MARTINEZ DE BERTORELLO
Instituto de Ciencias Químicas, Dpto de Química Orgánica, Universidad Nacional de Córdoba, Córdoba (República Argentina)

(Received October 21st, 1970; in revised form April 12th, 1971)

SUMMARY

The lithiation of 4,4'-dibromooctafluorobiphenyl with n-butyllithium to yield 4,4'-dilithiooctafluorobiphenyl and 4-lithio-4'-bromooctafluorobiphenyl as a by-product under various conditions is described. It is proposed that thermal decomposition of the lithiated products formed at low temperatures (−80° to −90°) leads to products originating principally by nucleophilic displacement reactions, while rapid thermal decomposition, after metal–halogen interconversion at temperatures of 0°, leads principally to products originating by elimination reactions. The intermediacy of benzyne species is proposed on the basis of observed addition reactions with furan to give the adduct 1,1',3,3',4,4'-hexafluoro-5,8,5',8'-tetrahydro-2,2'-binaphthyl 5,8,5',8'-diepoxide.

INTRODUCTION

Polyfluoroaryllithium reagents have been conveniently prepared through lithium–halogen[1] and, later, via lithium–hydrogen[2] interconversion reactions, both of which take place quite rapidly. The pentafluorophenyl anion from pentafluorophenyllithium has been used[3] as a nucleophile and as a source of tetrafluorobenzyne. The polyphenyls obtained in these reactions are formed by nucleophilic attacks[3] and are substituted in the 4-position.

Thermal decomposition of polyfluoroaryllithium reagents has been studied[4] widely. Thus, when 2,2'-dilithiooctafluorobiphenyl[5] decomposes at room temperature in the presence of furan, two isomeric difuran benzyne adducts are obtained; in absence of furan an intramolecular addition product, the 1-lithioheptafluorobiphenylene is isolated.

In the thermal decomposition of 3,3'-dilithio-4,4'-difluorobiphenyl[6] the intermediacy of dehydroaromatic compounds was proposed, based on the formation of the dehydroaromatic–furan adduct and on autocondensation reactions leading to

---

* Fellowship of the "Consejo Nacional de Investigaciones Científicas y Técnicas de la República Argentina". This work is taken from the Thesis by R. A. Contigiani to be presented at the Universidad Nacional de Córdoba. Córdoba. República Argentina.

macromolecules. Important questions about the process of polymerization remain unsolved, including the relative contributions of addition and nucleophilic displacement reactions.

RESULTS AND DISCUSSION

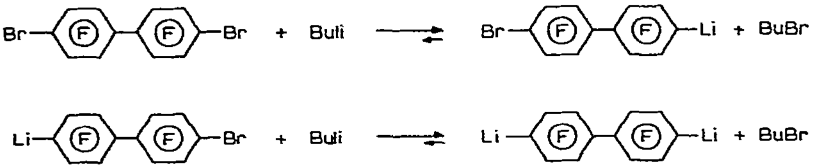
The present work describes a study of the role of perfluoroaryllithium compounds in processes of polymerization via dehydroaromatic species. The first step was to examine the capability of these organolithium compounds to intervene in elimination and/or in nucleophilic displacement reactions, and to determine the best conditions for each. 4,4′-Dilithiooctafluorobiphenyl was selected for study because of its ready synthesis by metal–halogen interconversion from 4,4′-dibromooctafluorobiphenyl and n-butyllithium.

The results of this interconversion for various reaction times and temperatures were determined by characterization of the organometallic products obtained, by carbonation of the reaction mixture and methylation to the respective esters, or by hydrolyses to the respective hydroderivatives and analysis by GLC.

Reactions carried out at −90° with an aryl halide/n-butyllithium ratio of 1/2.2 led after 30 min to 4,4′-dicarboxyoctafluorobiphenyl dimethyl ester (80 %) and to 4-carboxy-4′-bromooctafluorobiphenyl methyl ester (10 %) with (6 %) of unreacted 4,4′-dibromooctafluorobiphenyl, identified by mass spectra, ^{19}F NMR, IR and elemental analyses. At −90° and a reactant ratio of 1/5 yields of 99.4 % of 4,4′-dicarboxyoctafluorobiphenyl dimethyl ester were obtained. At −5°, a reactant ratio of 1/2.2, after 5 min of reaction gave 4,4′-dilithiooctafluorobiphenyl in 99 % yield, as evaluated by hydrolysis to 4,4′-dihydrooctafluorobiphenyl (Table 1).

The intermediacy of 4,4′-dilithiooctafluorobiphenyl has been previously demonstrated by Tamborski and Soloski[7] in the reaction of 4,4′-dihydrooctafluorobiphenyl with n-butyllithium through carbonation to the respective octafluorobiphenyl-4,4′-dicarboxylic acid.

In order to determine the extent of this equilibrium, some experiments were carried out with excess n-butyl bromide in the metalated mixture maintained at temperatures of −70° to −90°.



Only little effect was observed on the product composition, thus indicating that the equilibrium is well to the right.

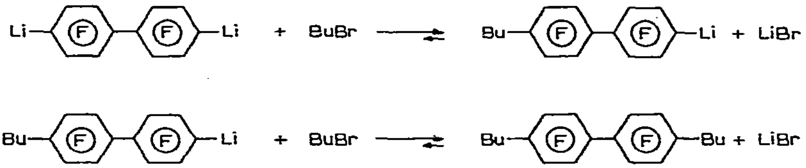
When the reaction mixture (aryl halide to n-butyllithium 1/2.2) at −80°, was allowed to reach room temperature stepwise (−60°/60 min, −40°/30 min, −20°/30 min, 0°/30 min, 20°/30 min) a mixture of more than 10 products was obtained. This was analyzed by GLC, fractional sublimation and by mass spectrometry. Among the products found were 4,4′-dibutyloctafluorobiphenyl (30 %) and 4-bromo-4′-butyloctafluorobiphenyl (19 %). The presence of both in the reaction mixture could be

TABLE I

CHARACTERIZATION OF THE ORGANOMETALLIC PRODUCTS

Ratio Arylhalide/BuLi	Temp. (°C)	By carbonation		By hydrolysis
		4,4'-dicarboxyoctafluorobiphenyl (%)	4-carboxy-4'-bromooctafluorobiphenyl (%)	4,4'-dihydrooctafluorobiphenyl (%)
1/2.2	−90°	80	10	
1/5.0	−90°	99.4		
1/2.2	−5°			99

explained by nucleophilic displacement reactions such as the following:



The 4,4'-dibutyloctafluorobiphenyl and the 4-bromo-4'-butyloctafluorobiphenyl were isolated by a combination of preparative GLC and fractional sublimation. They were identified by mass spectrometry, ^{19}F NMR, elemental analysis and by oxidation reactions leading to 4,4'-dicarboxyoctafluorobiphenyl and 4-carboxy-4'-bromooctafluorobiphenyl identical to that obtained by carbonation reactions.

Other products also present in the reaction mixture were similarly identified as 4,4'-dibromooctafluorobiphenyl (6%), 4-bromo-4'-hydrooctafluorobiphenyl (1.5%) and 4,4'dihydrooctafluorobiphenyl (0.2%). The presence of these substances, although in such small amounts, were indicative of the relatively high stability of the organolithium compounds obtained, commonly considered to be very unstable substances. Thus, careful experiments evaluating the lithio derivatives and the lithium fluoride formed showed elimination reactions at temperatures of 0° to 20° becoming most significant in reaction times longer than 60 min.

When the reaction mixture, in the same ratio (1/2.2) at −90° was allowed to reach room temperature stepwise (−60°/60 min, −40°/30 min, −20°/30 min, 0°/30 min, 20°/30 min) in the presence of furan and then boiled for some minutes, a similar mixture of products was obtained, without indication of any reaction with the added furan. Column chromatography of the crude product enabled us to separate the products described above and some polymeric products having the characteristics of polyphenyls[3] and assumed to be formed by nucleophilic displacement reactions. These polyphenyls were also present (35%) in the reactions carried out in the absence of furan.

A large five-fold excess of n-butyllithium over 4,4'-dibromooctafluorobiphenyl at −90°, followed by stepwise warming to room temperature gave a polymer, in yields of approximately 100%. The crude polymer extracted with petroleum ether, benzene, chloroform, ethanol, methanol and, finally, water in a Soxhlet extractor with little loss in weight, was a brown powder insoluble in all common solvents. One explanation for formation of this polymer and its structure could be that after effectively complete dimetalation, combined reactions of nucleophilic displacement and elimination-addition, favored by the butyl substituents, led to the formation of polyphenylic-polybiphenylic macromolecules. These were found to be infusible even at temperatures up to 400°. IR, elemental analysis and oxidation reactions indicate the presence of various butyl residues in phenyl rings. Further studies on the structure and properties of these polymers are under way.

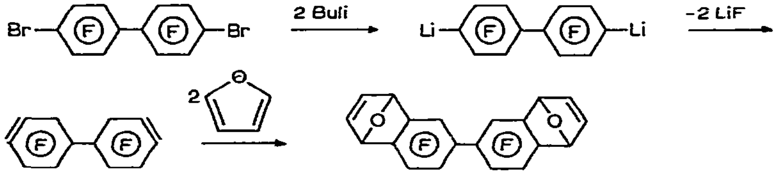
When the reaction 4,4'-dibromooctafluorobiphenyl with n-butyllithium (1/2.2) was carried out at temperatures of −5° to 0° and then the reaction mixture in its diethyl ether solvent was refluxed for more than 60 min, a yellow polymer was obtained in about 100% yield. Multiple extractions of the type indicated above gave a yellow powder, insoluble in common solvents and with a decomposition temperature (Td) (temperature for a rate of 10% weight loss/h) of 360°. IR indicated absence of butyl residues in the molecule.

Further studies of properties and structure of these polymers, formed under conditions of an elimination-addition sequence are in progress. Products such as 4,4'-dibutyloctafluorobiphenyl and others indicative of nucleophilic displacement reactions were absent in the crude product.

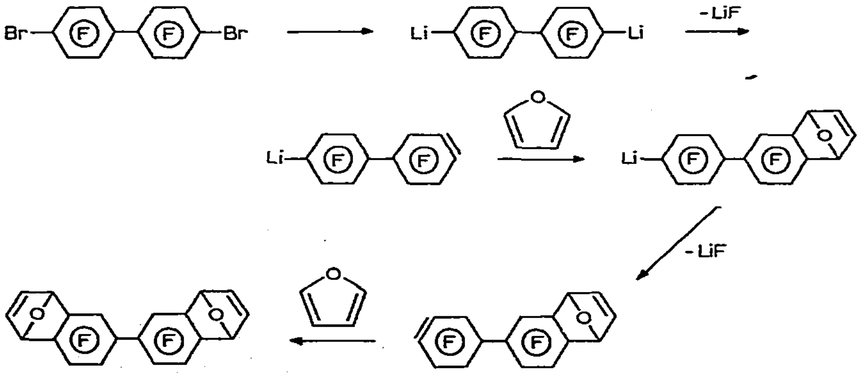
When the above reaction (1/2.2) was carried out at −5° to 0° in the presence of furan, no polymer was obtained. Instead a product was isolated in 70% yield, having m.p. 195–205° (dec.). Crystallization from carbon tetrachloride and petroleum ether yielded a yellowish-white product with m.p. 210° (dec.).

Mass spectra gave masses at 394.0421 ($C_{20}H_8F_6O_2$), 378.0491 ($C_{20}H_8F_6O$), 362.0530 ($C_{20}H_8F_6$), 366.0500 ($C_{19}H_8F_6O$), 338.0523 ($C_{18}H_8F_6$).

The elemental analysis is in accord with a furan–dehydroaromatic-adduct. A tentative mechanism for the reactions involved could be based on the formation of dehydroaromatic species:



or according to a stepwise mechanism:



The structure of the furan-didehydrohexafluorobiphenyl adduct, the 1,1',3,3',4,4'-hexafluoro-5,8,5',8'-tetrahydro-2,2'-dinaphthyl 5,8,5', 8'-diepoxide, was also confirmed by hydrolyzing it to the respective isomeric phenols. Phenols were separated by TLC and identified by IR, mass spectrometry and by coupling with diazotized benzidine.

EXPERIMENTAL

4,4'-Dibromooctafluorobiphenyl available from Pierce Chemical Co. (U.S.A.)

All organometallic reactions were carried out in an atmosphere of dry, oxygen-free nitrogen. Solvents were purified and dried by conventional methods and distilled prior to use. All melting points are uncorrected. GLC analyses were made on an F & M 776 gas chromatograph having a 4 ft column, packed with 20% UC-W 98 60–80P 776. Mass spectrometric analyses were performed on an AEI MS-902 spectrometer at the Ohio State University (U.S.A.) and at the Florida State University (U.S.A.). ^{19}F NMR spectra were run on a Brucker Scientific, Inc. B-90 Instrument.

Metal–halogen interconversion from 4,4'-dibromooctafluorobiphenyl at −90°. *Ratio 4,4'-dibromooctafluorobiphenyl to n-butyllithium* 1/2.2

A solution of 4,4'-dibromooctafluorobiphenyl (0.456 g, 0.001 mol) in 10 ml of diethyl ether/THF (0.4/1) was added dropwise, to a stirred, precooled (−90°) solution of 0.0022 mol of n-butyllithium in 12.5 ml of diethyl ether/THF (1/0.7), in 5 min. The temperature was maintained below −90° for 30 min. After this time, metal–halogen interconversion of the product was reached.

Reaction with carbon dioxide. The previous organometallic mixture was carbonated by bubbling carbon dioxide through the reaction. After 5 min the cooling bath was removed and the reaction was slowly warmed to room temperature with continued carbonation. The reaction mixture was then hydrolyzed with 10 ml of water and phase separated. The water layer was repeatedly washed with benzene and the expected acids were precipitated from the water solution by the addition of 4 ml of concentrated HCl, yielding 387 mg of the acid mixture. This mixture, treated with diazomethane, and after fractional sublimation and crystallization from ethanol, led to 4,4'-dicarboxyoctafluorobiphenyl dimethyl ester (80%), m.p. 90.5–91.2° (Found: C, 46.47; H, 1.41; F, 36.32; mol.wt. (mass spectrometric), 414, $C_{16}H_6F_8O_4$ calcd.: C, 46.39; H, 1.46; F, 36.69%; mol.wt. 414.) ^{19}F NMR (CH_2Cl_2 solvent, $CFCl_3$ standard): 134.5 (m, 1); 135.3 ppm (m, 1), and to 4-bromo-4'-carboxyoctafluorobiphenyl methyl ester (10%), m.p. 80.0–82.0° (Found: C, 38.22; H, 0.86; mol.wt. (mass spectrometric), 435. $C_{14}H_3BrF_8O_2$ calcd.: C, 38.65; H, 0.70; Br 18.37; F, 34.94; O, 7.36%; mol.wt. 435.) ^{19}F NMR (CH_2Cl_2 solvent, $CFCl_3$ standard): 132.7 (m, 1); 137.7 (m, 2); 139.6 ppm (m, 1). Both products were also separated by preparative GLC and purified by succesive recrystallizations from ethanol.

Reaction with water. The previous organometallic mixture was hydrolyzed with 10 ml of water and the phases separated. The organic layer was dried with magnesium sulfate. Aspiration of the solvent yielded 450 mg of a solid. The composition of the product was resolved by GLC into 4,4'-dihydrooctafluorobiphenyl (83%) and 4-bromo-4'-hydrooctafluorobiphenyl (11%).

The 4,4'-dihydrooctafluorobiphenyl was also separated from the mixture by sublimation and successive crystallizations from ethanol gave a white product with a m.p. 81.5–83° (lit.[8] 82–83.5°). Mol.wt. (mass spectrometric), 298. $C_{12}H_2F_8$ calcd.: 298. ^{19}F NMR (CH_2Cl_2 solvent, $CFCl_3$ standard): 137.9 (m, 1), 139.0 ppm (m, 1).

The 4-bromo-4′-hydrooctafluorobiphenyl was separated from the mixture by preparative thin layer chromatography and successive crystallizations from ethanol gave a white product with a m.p. 106–109°, and a mass spectrometric analysis with important peaks at 377.9132–375.9104 ($C_{12}HBrF_8$), 297.9946 ($C_{12}HF_8$), 295.9862 ($C_{12}F_8$), 277.9973 ($C_{12}HF_7$), 246.9966 ($C_{11}HF_7$). ^{19}F NMR (CH_2Cl_2 solvent, $CFCl_3$ standard): 132.3 (m, 1); 137.7 (m, 2); 138.7 ppm (m, 1).

Decomposition of the organometallic mixture. The previous organometallic mixture, was gradually allowed to reach room temperature (−60°/60 min, −40°/30 min, −20°/30 min, 0°/30 min, 20°/30 min) and finally boiled for one hour. The mixture was hydrolyzed by adding 10 ml of water and the organic layer was removed. The residue that remained after evaporation of the organic solvent was washed repeatedly with water and dried in vacuum in a desiccator over magnesium sulfate, yielding 374 mg of an oily product. From sublimation of this residue, 112 mg (30%) of a white product was obtained, which, by repeated crystallization from ethanol, yielded 80 mg of a white product with a m.p. 41.5–42.4°, identified by elemental analyses and by oxidation as 4,4′-dibutyloctafluorobiphenyl (Found: C 58.39; H, 4.43; F, 37.23; mol.wt. (mass spectrometric), 410. $C_{20}H_{18}F_8$ calcd.: C, 58.53; H, 4.42; F, 37.04%; mol.wt. 410.) ^{19}F NMR (CH_2Cl_2 solvent, $CFCl_3$ standard): 136.2 (m, 1) 138.2 ppm (m, 1).

Oxidation of 4,4′-dibutyloctafluorobiphenyl by potassium permanganate in an acid solution, gave 4,4′-dicarboxyoctafluorobiphenyl which, treated with diazomethane, led to 4,4′-dicarboxyoctafluorobiphenyl dimethyl ester, having a retention time identical to that of a sample obtained according to the previously described carbonation.

4-Bromo-4′-butyloctafluorobiphenyl was identified by mass spectrometry and by oxidation. Mol.wt. (mass spectrometric), 433. $C_{16}H_9BrF_8$ calcd.: 433. Oxidation of 4-bromo-4′-butyloctafluorobiphenyl by potassium permanganate in an acid solution, gave 4-carboxy-4′-bromooctafluorobiphenyl, having a retention time identical to that of a sample obtained according to the previously described carbonation. From GLC and reference to retention times determined for pure samples, the following substances were identified and evaluated: 4,4′dibromooctafluorobiphenyl (6%), 4-bromo-4′-hydrooctafluorobiphenyl (1.5%) and 4,4′-dihydrooctafluorobiphenyl (0.2%).

Decomposition of the organometallic mixture in the presence of furan. To the previous organometallic mixture 10 ml of anhydrous freshly distilled furan was added and then allowed to take room temperature in the way described above. The obtained residue, studied by fractional sublimation, GLC, and mass spectrometric analyses was resolved as a mixture of substances such as that observed in the previous described reaction of decomposition. By column chromatography the previously mentioned products were separated, in the proportion indicated above. Also isolated was an infusible, non-sublimate product in 35% yield, whose IR spectrum and physical properties, such as color, solubility, decomposition temperature and non-volatility (no mass spectrometric analyses could be run), are coincident with that described[3] to which polyphenyl structures were proposed.

Metal–halogen interconversion from 4,4′-dibromooctafluorobiphenyl at −90°. Ratio of 4,4′-dibromooctafluorobiphenyl to n-butyllithium 1/5.

A solution of 4,4′-dibromooctafluorobiphenyl (0.456 g, 0.001 mol) in 10 ml of diethyl ether/THF (0.4/1), was added dropwise to a stirred precooled (−90°) solution of 0.005 mol of n-butyllithium in 15 ml of diethyl ether/THF (1/0.5) over a 5 min period. The temperature was maintained at −90° and 30 min after the addition was complete, the metal–halogen interconversion was reached.

Reaction with carbon dioxide. The previous organometallic mixture was carbonated by bubbling carbon dioxide into the reaction. After 5 min the cooling bath was removed and the reaction was slowly warmed to room temperature, with continued carbonation. The reaction mixture was then hydrolyzed with 10 ml of water and the phases separated. By proceeding in the same manner as in the above described carbonation reaction, 4,4′-dicarboxyoctafluorobiphenyl dimethyl ester was identified and evaluated in 99.4% yield. No 4-bromo-4′-carboxyoctafluorobiphenyl methyl ester was found.

Decomposition of the organometallic mixture. The previous organometallic mixture was allowed to warm up to room temperature gradually (−60°/60 min, −40°/30 min, −20°/30 min, 0°/30 min, 20°/30 min) and finally boiled for an hour. The mixture was hydrolyzed by adding 10 ml of water and the organic layer removed by aspiration. The residue was washed repeatedly with water and dried yielding 506 mg of a brown-yellow product with a decomposition temperature (Td) of 360°.

91 mg of this product was extracted in a Soxhlet extractor with 20 ml fractions of petroleum ether, carbon tetrachloride, chloroform, benzene, diethyl ether, acetone, ethanol, methanol and finally with water. The yield after drying was 67 mg of a brown-yellow powder with a decomposition temperature (Td) of 360°. Its IR spectrum (KBr pellet) showed bands at: 2.95; 3.4; 6.8; 7.95; 10.2; 12.6; 13.95 and 14.15 μ (Found: C, 59.02; H, 3.55; Br, 8.49; F, 28.64. $C_{42}H_{27}F_{13}Br$ calcd.: C, 58.75; H, 3.17; Br, 9.38; F, 28.77.)

Metal–halogen interconversion from 4,4′-dibromooctafluorobiphenyl at −5° to 0°. Ratio 4,4′-dibromooctafluorobiphenyl to n-butyllithium 1/2.2

To a precooled (−5° to 0°) stirred solution of 2.2 mmole of n-butyllithium in 12 ml of dried diethyl ether, a solution of 0.456 g (1 mmole) of 4,4′-dibromooctafluorobiphenyl in 15 ml of dried diethyl ether was added dropwise in 4 min. The temperature was maintained for 3 min more and the metal–halogen interconversion reaction was complete.

Reaction with water. The previous organometallic mixture was hydrolyzed by adding 10 ml of water and phase separated. From the organic layer, after being dried, over magnesium sulfate and aspiration of the solvent, 341 mg of a white product was obtained. The composition of the product was resolved by GLC to be: 4,4′-dihydrooctafluorobiphenyl (about 100% of the product) and only traces of 4-bromo-4′-hydrooctafluorobiphenyl and 4,4′-dibromooctafluorobiphenyl.

Decomposition of the organometallic mixture. The previous organometallic mixture was allowed to reach room temperature and then boiled for 60 min. Forty minutes after boiling of the mixture, more than 80% lithium fluoride formation was found, as determined by titration of the lithium hydroxide liberated by hydrolysis of aliquots taken from the reaction mixture at various times. Then the mixture was cooled and hydrolyzed with 10 ml of water. After aspiration of the organic solvent, acidification with concentrated HCl and washing with water yielded a yellow insoluble powder, which, after drying, weighed 400 mg. It showed a decomposition temperature (Td) of 370°. This product was extracted in a Soxhlet extractor with fractions of petroleum ether, carbon tetrachloride, benzene, chloroform, diethyl ether, acetone, ethanol, methanol and finally water. Its IR spectrum (KBr pellet) had bands at: 2.95; 3.40; 6.80; 7.95; 10.20; 13.95 and 14.15 μ.

Decomposition of the metalated mixture in the presence of furan. The previous organometallic mixture in the presence of 10 ml anhydrous fresh distilled furan, was allowed to reach room temperature and then boiled for 3 h. The mixture was cooled and hydrolyzed with 10 ml of water and the phases separated. The organic layer was repeatedly washed with a solution of sodium carbonate (5%), then with water, and finally, dried over magnesium sulfate. Aspiration of the solvent yielded 381 mg of a yellow product. This product was resolved by preparative thin layer chromatography using NM Silica Gel N (eluent: cyclohexane/acetone 23/12) into two fractions. The fraction with R_f 0.53, yielded a yellowish-white product (63%) with a m.p. 210° (dec.) and a mass spectrometric analysis with important peaks at 394.0421 ($C_{20}H_8F_6O_2$), 378.0491 ($C_{20}H_8F_6O$), 362.0530 ($C_{20}H_8F_6$), 366.0500 ($C_{19}H_8F_6O$) 338.0523 ($C_{18}H_8F_6$).

This product was identified as 1,1′, 3,3′, 4,4′-hexafluoro-5, 8, 5′, 8′-tetrahydro-2,2′-dinaphthyl 5, 8, 5′, 8′-diepoxide. (Found: C, 60.78; H 1.81; F, 29.1. $C_{20}H_8F_6O_2$ calcd.: C, 60.92; H, 2.04; F, 28.91%.) ^{19}F NMR (CH_2Cl_2 solvent, $CFCl_3$ standard): 123.7 (m, 1), 134.9 (m, 1), 147.4 ppm (m, 1). Its IR spectrum (KBr pellet) had bands at 9.10; 9.90; 11.10; 11.58; 12.10 and 15.05 μ.

The crude product (381 mg) purified by solution in carbon tetrachloride followed by fractional precipitation with petroleum ether, gave a yellowish-white product with a m.p. 210° (dec.) in yield of 70%.

The furan–dehydroaromatic adduct was also identified through isomerization to the respective naphthols according to a general method[9], by treating the adduct with concentrated HCl/ethanol (50/50), and refluxing the mixture for 3 h. The naphthols were then dissolved by addition of a solution of sodium hydroxide in water and then precipitated by acidification with concentrated HCl. The pale yellow mixture of naphthols was resolved by preparative thin layer chromatography, using NM Silica Gel N, and identified by coupling with diazotized benzidine. The fraction with R_f 0.35 (eluent: cyclohexane/acetone 23/12) yielded a pale yellow product (72%) m.p. 205–209°. Mol.wt. (mass spectrometric), 394. $C_{20}H_8F_6O_2$ calcd.: 394. Its IR spectrum (KBr pellet) had bands at 9.95; 10.10; 12.55 and 13.42 μ.

ACKNOWLEDGEMENTS

This research was sponsored by the "Consejo Nacional de Investigaciones Científicas y Técnicas de la República Argentina" and by the "Air Force Office of Scientific Research", Grant No 68–1425. We are indebted to Dr. R. C. Dougherty and to Mr. D. C. Weissemberger for the mass spectrometric analyses, and to Dr. J. Dalton and Mr. R. Rosanske for the F NMR, carried out at the Florida State University and The Ohio State University (USA).

REFERENCES

1 P. L. Coe, R. Stephens and J. C. Tatlow, *J. Chem. Soc.*, (1962) 3227.
2 R. J. Harper Jr., E. J. Soloski and C. Tamborski, *J. Org. Chem.*, 29 (1964) 2385.
3 D. D. Callander, P. L. Coe and J. C. Tatlow, *Tetrahedron*, 22 (1966) 419.
4 D. E. Fenton, A. J. Park, D. Shaw and A. G. Massey, *J. Organometal. Chem.*, 2 (1964) 437.
D. E. Fenton and A. G. Massey, *Tetrahedron*, 21 (1965) 3009.
D. C. Cohen, D. E. Fenton, D. Shaw and A. G. Massey, *J. Organometal. Chem.*, 8 (1967) 321.
S. C. Cohen, A. J. Tomlinson, M. R. Wiles and A. G. Massey, *Chem. Ind. (London)*, (1967) 877.
S. C. Cohen, A. J. Tomlinson, M. R. Wiles and A. G. Massey, *J. Organometal. Chem.*, 11 (1968) 385.
5 S. C. Cohen, D. Moore, R. Price and A. G. Massey, *J. Organometal. Chem.*, 12 (1968) P36.
6 M. Martinez de Bertorello and Héctor E. Bertorello, *J. Organometal. Chem.*, 23 (1970) 285.
7 C. Tamborski and E. J. Soloski, *J. Org. Chem.*, 31 (1966) 746.
8 G. G. Yakobson, V. D. Shteingarts, A. I. Miroshnikov and N. N. Vorozhtsov Jr., *Dokl. Akad. Nauk SSSR*, 159 (5) (1964) 1109.
9 G. Wittig and L. Pohmer, *Chem. Ber.*, 89 (1956) 1334.