by a solution containing 10 ml of conecntrated sulfuric acid, 6.00 g (60 mmoles) of chromic oxide, and 20 ml of carbon dioxide free water; meanwhile a carbon dioxide absorber was connected to the system. The mixture was heated up in a oil bath slowly (maximum temperature 112°) under constant stirring for 1 hr. The resultant carbon dioxide carried by nitrogen was absorbed by 50 ml of 0.5 N sodium hydroxide and precipitated as barium carbonate (4.435 μcuries/mmole) after the addition of 55 ml of 0.5 M ammonium chloride and 62.5 ml of 0.15 M barium chloride, followed by washing with carbon dioxide free water, and drying at 110° overnight; yield of barium carbonate was 24% (238 mg).

B. Isolation of Acetic Acid as Barium Acetate.—The above reaction mixture was steam distilled and the distillate (150 ml) was neutralized by 0.1 N barium hydroxide. The water was evaporated off under reduced pressure. The barium acetate was dried overnight at 160°. Over-all yield according to the barium acetate obtained was 18% (220 mg), mainly owing to mechanical loss.

C. Degradation of Barium Acetate to Methylamine and Carbon Dioxide.—Under nitrogen atmosphere 220 mg (0.86 mmole) of barium acetate was dissolved in 2.4 ml of concentrated sulfuric acid, then 3.6 ml of chloroform was introduced, followed by slow addition of 2.6 mg of sodium azide. The carbon dioxide absorber was connected and the stirred reaction mixture was heated to 64° in a period of about 3 hr under a constant flow of nitrogen. Then 18 ml of carbon dioxide free water was added, after the mixture had been cooled down. It was heated up again for 20 min at 70–75°. The activity of barium carbonate (210 mg, 1.06 mmole, 61% yield) was 0.011 μcurie/mmole.

D. Isolation of Methylamine as N-Methyl-N′-phenylthiourea.—The above reaction mixture from the degradation of barium acetate was filtered, made alkaline with 15 ml of 10 N sodium hydroxide, and was steam distilled using 36 ml of 1 N hydrochloric acid as a trapping medium for methylamine. Methylamine hydrogen chloride (63 mg, 0.93 mmole) was obtained after evaporation of water under reduced pressure and dried in a desiccator, yield 54%.

The methylamine hydrochloride was placed in a small vial containing 1 ml of methanol. The solution was made distinctively basic by 3 N sodium hydroxide. Immediately 99 mg (0.73 mmole) of phenyl isothiocyanate was added and the vial was rubber capped. The mixture was shaken for 5 min and warmed in an oil bath for 15 min at about 100°. Then 2 ml of water was added to the vial by a syringe. A flake-like crystal precipitated out instantly. The precipitate was recrystallized from a mixed solvent of cyclohexane and benzene (1.5:1.0 by volume). N-Methyl-N′-phenylthiourea (74 mg, 0.45 mmole, 62% yield) had an activity of 0.101 μcurie/mmole, mp 116–117° (lit.[2] 115.5–116°).

Analysis. A. Vapor Phase Chromatography.—The gases were determined over a 0.25 in. × 2 m 33% dimethylsulfolane on 100–120 mesh firebrick column; the aldehyde, ether, pyridine, alcohol, and hexenes over a 0.25 in. × 4 m 15% Ucon 75H 90,000 on 60–80 WAB column.

B. Radiochemical Assay.—The radiochemical assay of the barium carbonate and thiourea was performed by using a Dynacon model 6000 (Nuclear-Chicago) and rate-of-charge method.[10]

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[10] H. Pines and G. Benoy, *J. Am. Chem. Soc.*, **82**, 2483 (1960).

Alumina: Catalyst and Support. XXXV.[1] 1,2- and 1,4-Phenyl Migrations in 1,1-Diphenylcyclohexane. Dehydroisomerization over "Nonacidic" Chromia–Alumina[2,3]

HERMAN PINES, W. F. FRY, NORMAN C. SIH, AND C. T. GOETSCHEL

The Ipatieff High Pressure and Catalytic Laboratory, Department of Chemistry, Northwestern University, Evanston, Illinois

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The dehydroisomerization of 1,1-diphenylcyclohexane over "nonacidic" chromia–alumina-B was studied using a pulse microreactor. *o*- and *p*-terphenyl were formed by a 1,2- and 1,4-phenyl migration, respectively, in addition to fission products. Free-radical species are proposed as intermediates.

Catalytic dehydrogenation and aromatization have been known and used for many years. Chromia–alumina is one of the catalysts that has been studied quite extensively, and lately it has been realized that the nature of the alumina used as support has an effect upon the behavior of the catalyst.[4]

Alumina has intrinsic acidic properties and its method of preparation has been shown to determine the strength of the acidic sites.[5] For example, alumina obtained by the hydrolysis of aluminum isopropoxide (alumina-A) catalyzed the isomerization of cyclohexene to methylcyclopentenes, which requires a relatively strong acidic catalyst, whereas alumina obtained from potassium aluminate (alumina-B) is less acidic and did not effect this isomerization.

The different natures of the chromia–alumina catalysts prepared from these aluminas were demonstrated by their behavior toward 1,1-dimethylcyclohexane.[6] Dehydrogenation over chromia–alumina-A (catalyst A) gave ethylbenzene and xylenes in addition to toluene and methane, whereas over chromia–alumina-B (catalyst B) only toluene and methane were formed. The more acidic catalyst A was able to promote a cationic-type isomerization of 1,1-dimethylcyclohexane *via* methide migration, but the low acidic character of catalyst B was not. It was suggested recently[2] that radical species are associated with the dehydrogenation reaction over catalyst B and the lack of methyl migration is in accord with this.[7]

Phenyl migrations have been observed in noncatalytic free-radical reactions. Among these are Grignard reactions in the presence of certain metal ions,[8] the thermal decomposition of azo compounds,[9] the oxidation of certain diarylethanes,[10] the pyrolysis of alkylbenzenes,[11] the peroxide-induced decomposition of

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[1] For paper XXXIII, see N. C. Sih and H. Pines, *J. Org. Chem.*, **31**, 4089 (1966).

[2] Paper XIX in the series Aromatization of Hydrocarbons. For paper XVIII see H. Pines and C. T. Goetschel, *J. Am. Chem. Soc.*, **87**, 4207 (1965).

[3] This research was supported by the Atomic Energy Contract AT-(11-1)-1196.

[4] C. T. Chen, W. O. Haag, and H. Pines, *Chem. Ind.* (London), 1379 (1959).

[5] H. Pines and W. O. Haag, *J. Am. Chem. Soc.*, **82**, 2471 (1960).

[6] H. Pines and C. T. Chen, *ibid.*, **82**, 3562 (1960).

[7] (a) L. H. Slaugh, R. D. Mullineaux, and J. H. Raley, *ibid.*, **85**, 3180 (1963); (b) F. H. Seubold, *ibid.*, **75**, 2532 (1953).

[8] (a) W. H. Urry and H. S. Kharasch, *ibid.*, **66**, 1438 (1944); (b) W. H. Urry and N. Nicolaides, *ibid.*, **74**, 5163 (1952).

[9] C. G. Overberger and H. Gainer, *ibid.*, **80**, 4561 (1958).

[10] H. H. Szmant and J. F. Deffner, *ibid.*, **81**, 958 (1959).

[11] V. N. Ipatieff, B. Kvetinskas, E. E. Meisinger, and H. Pines, *ibid.*, **75**, 3323 (1953).

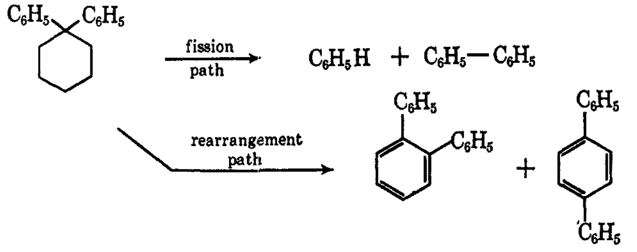
aldehydes,[7b,12] and the decomposition of peroxides or other radical sources in alkylbenzenes, *e.g.*, *t*-butylbenzene yielded isobutylbenzene.[13] The rearrangement of *t*-butylbenzene is of interest since it also occurred[2] when this hydrocarbon was passed over catalyst B, and is indicative of the free-radical nature of this catalyst; an acidic catalyst would yield benzene and isobutylene. In the above free-radical reactions, 1,2- and one example[12d] of 1,4-phenyl migrations were reported.

In view of the indication of the free-radical nature of chromia–alumina-B, the present study was aimed to investigate the possibility of phenyl migrations in 1,1-diphenylcyclohexane over this catalyst.

Discussion of Results

1,1-Diphenylcyclohexane yielded both fission and rearranged products during dehydrogenation over chromia–alumina-B at elevated temperatures. The product composition was investigated in relation to the reaction temperature, the space velocity, and the age of the catalyst used; no reaction occurred over glass beads.

The reaction of 1,1-diphenylcyclohexane over catalyst B at temperatures between 390 and 497° gave benzene, biphenyl, and *o*- and *p*-terphenyl, with traces of *m*-terphenyl.



The results (Tables I–III) are only semiquantitative since the low volatility of *p*-terphenyl makes it difficult to completely elute it from the pulse reactor into the vpc. Thus at a furnace temperature of 380° the loss of *p*-terphenyl is about 20%; at temperatures greater than 420° there is no loss. Further, at higher temperatures much of the *o*-terphenyl formed is lost: about 60% at 450° and 80% at 500°. It may form carbonaceous material or cyclize to triphenylene. Such a cyclization over chromia–alumina has been observed previously.[14] Therefore the percentage conversions are also inaccurate at high temperatures.

However, the formation under all conditions of only small amounts of *m*-terphenyl compared to *o*- and *p*-terphenyl indicates that the rearranged products are not the result of a stepwise phenyl migration since this should yield *o*-, *m*-, and *p*-terphenyls in decreasing order of abundance. The trace of *m*-terphenyl might arise from isomerization of *o*- and *p*-terphenyl.

TABLE I

EFFECT OF TEMPERATURE ON THE DEHYDROISOMERIZATION OF 1,1-DIPHENYLCYCLOHEXANE OVER CHROMIA–ALUMINA-B[a]

Expt	10	11	9	8
Temp, °C	391	420	450	497
Conversion, %	62	70	80	75
Product compn, moles %				
Benzene		Not estimated		
Biphenyl	89	80	74	64
o-Terphenyl	9	13	14	16
m-Terphenyl	Trace	<0.5	<1	<1
p-Terphenyl	2	6	8	12
Unknown	Trace	~3	~4	~8
Ratio of *o*-/*p*-terphenyl	4.5	2.2	1.8	1.3
Corrected ratio of *o*-/*p*-terphenyl[b]	*c*	2.2	4.4	6.5

[a] These runs were carried out at a reaction tube gas flow of 21.4 cc/min. This was chosen as the standard gas flow, and corresponds to that for a relative contact time of 4 (see Table III). [b] Corrected by using the approximate figures for the large losses of *o*-terphenyl at 450° and above, *i.e.*, about 60% at 450° and 80% at 500°. [c] Owing to the low temperature of the catalyst, and its consequent retention of the products no estimate of the corrected ratio was made.

TABLE II

THE EFFECT OF THE AGE OF CHROMIA–ALUMINA-B CATALYST ON THE DEHYDROISOMERIZATION OF 1,1-DIPHENYLCYCLOHEXANE[a]

Expt[b]	2	17
Temp, °C	421	426
Conversion, %	87	50
Product compn, moles %		
Benzene	Not estimated	
Biphenyl	91	53
o-Terphenyl	4	27
m-Terphenyl	<0.5	<0.5
p-Terphenyl	2	14
Unknown	~3	~6
Ratio of *o*-/*p*-terphenyl	2.0	1.9

[a] See footnote a, Table I. [b] Runs 2 and 17 are functions of the age of the catalyst. Fourteen runs were performed without regeneration between these two catalysts.

TABLE III

THE EFFECT OF CONTACT TIME ON THE DEHYDROISOMERIZATION OF 1,1-DIPHENYLCYCLOHEXANE OVER CHROMIA–ALUMINA-B AT 420°

Expt	13	12	17	11
Relative contact time[a]	1	2	4	4
Conversion, %	36	54	50	70
Product compn, moles %				
Benzene		Not estimated		
Biphenyl	68	72	53	80
o-Terphenyl	16	15	27	13
m-Terphenyl	Trace	<0.5	<0.5	<0.5
p-Terphenyl	9	7	14	6
Unknown	~8	~5	~6	~2
Ratio of *o*-/*p*-terphenyl	1.8	2.1	1.9	2.2

[a] See Table I, ref *a*.

Supplementary experiments were performed to establish that the rearranged products over catalyst B were predominantly primary ones resulting from intramolecular reaction: (1) *o*-, *m*-, and *p*-terphenyls underwent less than 3% interconversion under the reaction conditions. (2) Precursors of the rearranged products, namely *o*-, *m*-, and *p*-diphenylcyclohexanes did not rearrange under the reaction conditions and

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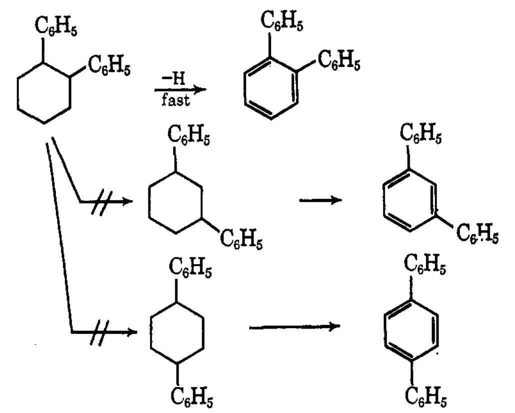
(12) (a) W. H. Urry, D. J. Trecker, and H. D. Hartzler, *J. Org. Chem.*, **29**, 1663 (1964); (b) S. Winstein and F. H. Seubold, *J. Am. Chem. Soc.*, **69**, 2916 (1947); (c) D. Y. Curtin and M. J. Hurwitz, *ibid.*, **74**, 5381 (1952); (d) S. Winstein, R. Heck, S. Lapporte, and R. Baird, *Experientia*, **12**, 138 (1956); (e) L. H. Slaugh, *J. Am. Chem. Soc.*, **81**, 2262 (1959); (f) D. Y. Curtin and J. C. Kauer, *J. Org. Chem.*, **25**, 880 (1960); (g) J. W. Wilt and H. Philip, *ibid.*, **24**, 441 (1959); **25**, 891 (1960); (h) J. W. Wilt and C. A. Schneider, *ibid.*, **26**, 4196 (1961); (i) C. Ruchardt, *Ber.*, **94**, 2599 (1961).

(13) (a) H. Pines and C. N. Pillai, *J. Am. Chem. Soc.*, **82**, 2921 (1960); (b) L. H. Slaugh and J. H. Raley, *ibid.*, **82**, 1259 (1960); **84**, 2640 (1962).

(14) C. Hansch and C. F. Geiger, *J. Org. Chem.*, **23**, 477 (1958).

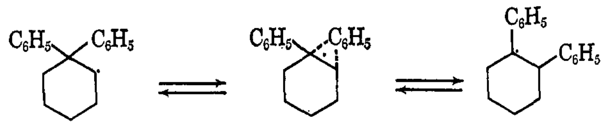
yielded only the products of dehydrogenation, *i.e.*, rate of dehydrogenation is very much greater than rate of phenyl migration, *e.g.*



(3) Passing a solution of biphenyl in benzene over chromia–alumina-B gave an almost quantitative recovery of biphenyl. Phenylation to yield terphenyls did not occur.

Evidence for the possible radical nature of chromia–alumina-B has been described and the phenyl migrations occurring in 1,1-diphenylcyclohexane can be understood in terms of radical intermediates.

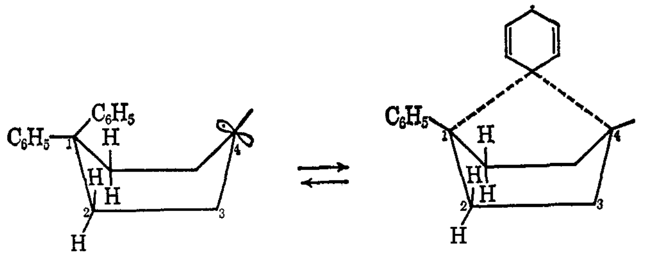
o-Terphenyl can be formed *via* a species similar to that already suggested[2,7b,8-12] in 1,2-phenyl migration.



If allowance is made for the approximate losses of *o*-terphenyl at 450° and above, it seems (Table I) that the 1,2-phenyl migration may be temperature dependent possibly reflecting a congested transition state with a high energy of activation.

The phenyl migrations are apparently independent of both the age of the catalyst and the contact time (Tables II and III) as the ratio *o*-terphenyl/*p*-terphenyl is approximately constant.

For the formation of *p*-terphenyl by a 1,4-phenyl migration, the transition state must involve the boat form of cyclohexane. If a radical is formed on C_4 the p orbital containing the spare electron will be suitably oriented toward C_1 to initiate bonding with the phenyl group because of sp^2 hybridization of C_4.



A similar mechanism has been proposed previously by Abdun-Nur and Bordwell,[15] who observed 1,4-phenyl migration in the base-catalyzed elimination–rearrangement reaction of 4,4-diphenylcyclohexyl *p*-toluenesulfonate with sodium *t*-butoxide. They proposed a concerted mechanism and pointed out the favorable geometry. The other phenyl group on C_1 is well staggered with respect to the hydrogens on the neighboring carbon atoms so that steric interaction is not critical and, therefore, the 1,4-phenyl migration is less temperature dependent than the 1,2 migration.

Conclusion

During the dehydrogenation of 1,1-diphenylcyclohexane over chromia–alumina-B 1,2- and 1,4-phenyl migrations were observed, both occurring by an intramolecular reaction. The virtual absence of *m*-terphenyl and the stability of possible intermediates to isomerization show that the 1,4 migration is a one-step process. The 1,2 migration may be more temperature dependent than the 1,4 migration, possibly owing to steric factors.

Both can be understood in terms of radical intermediates, thus supporting previous conclusions as to the nature of this catalyst.

Experimental Part

I. **Apparatus and Procedure.**—All the experiments were carried out in a pulse microreactor[16] linked directly to a gas chromatograph.

About 0.2 g of chromia–alumina-B was used and the charge of feedstock was about 2 μl of a solution of 1,1-diphenylcyclohexane (0.51027 g) and naphthalene (0.07688 g) in benzene (1 ml). The naphthalene was the internal standard and was stable under all the reaction conditions used.

Owing to their low solubility the terphenyls were injected as solid samples using a syringe (Model SS-60) made by Hamilton Co., Inc., for the injection of solids. Internal standards cannot be used and the sample size is not reproducible, but only qualitative information about possible isomerization was required.

The nongeminal cyclohexanes were used as 25% solutions in benzene with 2-μl samples.

The contact time was varied by varying the gas flow through the reactor and for slower flow rates the flow through the analytical column was kept constant by adding a second flow between the reactor and vpc unit.

The products were identified on two columns, namely, (a) 0.25 in. × 2 ft 12% Versamid 900 on 60–80 mesh Chromosorb W, and (b) 0.25 in. × 5 ft 15% GE-SE-52 on 60–80 mesh WAB. Analyses of reaction products were performed on column b at a gas flow of 85.5 cc/min (10 cc/7 sec) with the temperature programmed from 120 to 220° at 18°/min beginning the programming 6.5 min after the injection of the reaction mixture into the reactor. Table IV shows the relative retention times under these conditions and the molar area responses.

TABLE IV
RELATIVE RETENTION TIMES AND MOLAR AREA RESPONSES OF THE PRODUCTS FROM 1,1-DIPHENYLCYCLOHEXANE

Compd	Relative retention time	Molar area response
Naphthalene[a]	0.47	1.000
Biphenyl	0.63	0.803
o-Terphenyl	0.93	0.621
1,1-Diphenylcyclohexane	1.00	0.635
Unknown	1.05	...
m-Terphenyl[b]	1.28	...
p-Terphenyl	1.38	0.623

[a] Naphthalene was used as an internal standard. [b] The molar area response of *m*-terphenyl was not determined since it is formed in only trace amounts.

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(15) A. R. Abdun-Nur and F. G. Bordwell, *J. Am. Chem. Soc.*, **86**, 5695 (1964).

(16) P. Steingaszner and H. Pines, *J. Catalysis*, **5**, 356 (1966).

The alumina-B was prepared as described previously,[6] and graded (20–40 mesh) before being impregnated with chromia.[6]

II. **Chemicals.**—Naphthalene, biphenyl, and the terphenyls were provided by commercial sources, and were checked for purity before use.

1,1-Diphenylcyclohexane.—Following reported procedures[17,18] 4,4-diphenylcyclohexanone was prepared from benzoin in an over-all yield of 30%. The product was recrystallized from *n*-heptane and melted at 142–144° (lit.[17] mp 143–144°).

The 4,4-diphenylcyclohexanone (4.55 g, 0.019 mole) was heated under reflux with 5.6 ml of 85% hydrazine hydrate solution and 37 ml of triethylene glycol. After 20 min solid sodium hydroxide (3.7 g, 0.093 mole) was added. The reflux condenser was removed and the mixture was heated to 220° to allow the water to evaporate. The mixture was then kept at 180° for 3 hr, poured onto ice, and extracted with *n*-hexane (three 50-ml portions). The organic solution was washed with water until neutral and then dried over anhydrous magnesium sulfate. Removal of the solvent gave a crude product, which was purified by using a 20-ml alumina column with *n*-pentane as eluent. Recrystallization of the solid from 95% ethanol gave white, needlelike crystals of 1,1-diphenylcyclohexane (3.52 g, 0.016 mole, 82% yield), mp 45–46.5° (lit.[19] mp 47°).

1,2-Diphenylcyclohexane was prepared from a Grignard reaction (56% yield) of 2-phenylcyclohexanone and phenylmagnesium bromide, followed by dehydration (29% yield) over 85% phosphoric acid,[20] and subsequent hydrogenation over 5% palladium–carbon. During the dehydration, a substantial amount (47%) of rearranged products was also obtained, having mp 65–66° after hydrogenation, which was not reported by Parmerter.[20] The 1,2-diphenylcyclohexane, *cis/trans* = 3.3 (n^{20}_{D} 1.5695), was isolated by preparative vpc using a 5 ft × $^3/_8$ in. 15% GE-SE-52 on 60–80 WAB column, and gave *o*-terphenyl when aromatized over chromia–alumina-B[6] at 377 ± 2°. Unreacted 1,2-diphenylcyclohexane was partly epimerized with *cis/trans* = 0.87.

1,3-Diphenylcyclohexane was prepared according to the procedure described by Abdun-Nur.[21] Interaction of phenylmagnesium bromide (0.08 mole) and 3-phenylcyclohex-2-enone[22] (0.027 mole) gave 1,3-diphenylcyclohexadiene-1,3 (63%), mp 89–100° (lit.[22] mp 98–99°). This diene was hydrogenated in acetic acid using 10% palladium–carbon under 40 psi. The product was distilled [bp 140–145° (0.2 mm)], and then a small sample was further purified by preparative vpc on a 2-m column, packed with 15% Carbowax 20 M on 40–60 Chromosorb P at 200° and a helium flow of 75 ml/min. The compound had a retention time of 14 min.

1,4-Diphenylcyclohexane was prepared by the same procedure as described for 1,2-diphenylcyclohexane, but using 4-phenylcyclohexanone in the Grignard reaction. Here, no rearranged product was observed. The final product was recrystallized from benzene (70%), mp 159–173°, and was a mixture of *cis* and *trans* isomers. It yielded *p*-terphenyl on aromatization over chromia–alumina-B.[6]

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[17] F. G. Bordwell and K. M. Wellman, *J. Org. Chem.*, **28**, 2544 (1963).

[18] H. E. Zimmerman and D. I. Schuster, *J. Am. Chem. Soc.*, **84**, 4527 (1962).

[19] F. G. Bordwell, private communication.

[20] S. M. Parmerter, *J. Am. Chem. Soc.*, **71**, 1127 (1949).

[21] A. R. Abdun-Nur, Ph.D. Thesis, Northwestern University, 1965.

[22] G. F. Woods and I. W. Tucker, *J. Am. Chem. Soc.*, **70**, 2174 (1948).

Phenylthiomethyllithium and Bis(phenylthio)methyllithium

E. J. Corey and D. Seebach

Department of Chemistry, Harvard University, Cambridge, Massachusetts 02138

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Efficient procedures are described for the generation of the functionally substituted organolithium derivatives cited in the title. Several characteristic reactions which indicate the synthetic applicability of these reagents are presented.

Gilman and Webb have reported that thioanisole is metalated by *n*-butyllithium at the methyl group to form phenylthiomethyllithium (I), in contrast to anisole, which is converted under the same conditions to *o*-methoxyphenyllithium.[1,2] This discovery, while of definite theoretical significance, has not led to practical application in synthesis because of the fact that the maximum yield of I which was obtained in many experiments was only *ca.* 35%.[1,2] After numerous unsuccessful attempts to develop a satisfactory method for the generation of this potentially useful reagent, an excellent procedure was finally found which depends on a catalysis of metalation with *n*-butyllithium, which is produced from its interaction with 1,4-diazabicyclo[2.2.2]octane (Dabco).[3-5] Reaction of equimolar amounts of *n*-butyllithium, Dabco, and thioanisole in tetrahydrofuran at 0° for 45 min produces I in *ca.* 97% yield, as determined by subsequent quenching with deuterium oxide and quantitative nmr analysis. Reaction of the lithio derivative I, so produced with an equivalent amount of benzophenone, affords the β-hydroxysulfide II, R = C_6H_5, in 93% yield, confirming the nmr analytical result. Further, addition of I to acetone produces the carbinol II, R = CH_3 (75%), and alkylation of I with isopropyl iodide leads to phenyl isobutyryl sulfide III in 55% yield.

$C_6H_5SCH_2Li$

I

$$C_6H_5SCH_2\underset{\displaystyle OH}{\underset{|}{C}}R_2$$

II

$C_6H_5SCH_2CH(CH_3)_2$

III

It has also been found that I can be generated from thioanisole efficiently, using phenyllithium without the assistance of Dabco with tetrahydrofuran as solvent at 25°; this process is slow, however, and a 15-hr reaction period was required to give good conversion to I (shown by formation of II, R = C_6H_5, in 90% yield).

The benzophenone adduct II, R = C_6H_5, could be dehydrated to the vinyl sulfide IV, X = H, which underwent facile metalation using *n*-butyllithium in tetrahydrofuran at −30° without assistance from Dabco to form IV, X = Li.[6] Vinyl lithio derivatives such as IV, X = Li, are of interest as "masked"

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[1] H. Gilman and F. J. Webb, *J. Am. Chem. Soc.*, **62**, 987 (1940).

[2] H. Gilman and F. J. Webb, *ibid.*, **71**, 4062 (1949).

[3] W. A. Butte, *J. Org. Chem.*, **29**, 2928 (1964).

[4] C. G. Screttas and J. F. Eastham, *J. Am. Chem. Soc.*, **87**, 3276 (1965).

[5] "Dabco" has been used to precipitate a variety of organolithium compounds from solutions, in form of crystalline complexes containing 1 mole of diamine and 4 moles of the organometallic compound.[4]

[6] Organolithium derivatives of this type have been described previously; see, for instance, J. F. Arens, *et al.*, *Rec. Trav. Chim.*, **85**, 31, 560 (1966).