4. **1,2,3,3a,8,8a-Hexahydrocyclopent[*a*]indene (29).** A.—**Cyclopentene-1-carboxylic acid (26)** was synthesized according to the procedure of Maitte.[29] 1,2-Dibromocyclopentane (**24**; 120 g., 0.53 mole) was dehydrobrominated at 120° (46 mm.) by a mixture of 170 g. of dry sand and 125 g. (2.2 moles) of powdered potassium hydroxide. 1-Bromocyclopentene (**25**) was obtained in 50% yield, b.p. 115–132°.

The Grignard reagent obtained when the bromide **25** (21 g., 0.15 mole) was treated with 4.8 g. (0.20 g.-atom) of magnesium in 50 ml. of purified tetrahydrofuran was carboxylated by Hussey's procedure.[30] The title compound (**26**) was obtained in 43% yield. After sublimation, it melted at 119–120° (lit.[29] m.p. 123–123.5°).

B.—**1,2,3,3a,8,8a-Hexahydrocyclopent[*a*]indene** (**29**) was synthesized after Baker and Jones.[31] The carboxylic acid **26** (8.8 g., 0.08 mole) was first converted with 10 ml. (0.14 mole) of thionyl chloride at 110° for 0.5 hr. to its acyl chloride (**27**, b.p. 72–73° at 19 mm.) with a yield of 85%. The acyl chloride (**27**; 8.7 g., 0.07 mole) was allowed to react with 75 ml. of benzene with constant stirring in the presence of 26.5 g. (0.20 mole) of aluminum chloride. The mixture was kept at 0° for 1 hr., then heated up to 110° for 2 hr., and allowed to stand overnight at room temperature. After decomposition of the mixture by dilute hydrochloric acid, the organic layer was washed and dried. An 86% yield of [2,3]cyclopenta-1-indanone (**28**) was obtained. On Clemmensen reduction,[32] **28** gave a 76% yield of the title compound (**29**) which was distilled at 120–122° (17 mm.), $n^{20}\mathrm{D}$ 1.5503 (lit.[31] $n^{17}\mathrm{D}$ 1.5511).

Anal. Calcd. for $C_{12}H_{14}$: C, 91.07; H, 8.91. Found: C, 90.75; H, 9.11.

5. *trans*- **and** *cis*-**1-Methyl-2-phenylcyclohexane (32, 33).**—The 1-phenyl-2-methylcyclohexanols (**30**), obtained from phenylmagnesium bromide and 2-methylcyclohexanone, b.p. 132–139° (15 mm.), were dehydrated to 1-phenyl-2-methylcyclohexenes (**31**) by refluxing with a small amount of potassium bisulfate. Hydrogenation of the olefins over platinum oxide yielded a mixture of the title hydrocarbons (**32**, **33**), $n^{27}\mathrm{D}$ 1.5258 (lit.[33] $n^{18.5}\mathrm{D}$ 1.5360). The ratio of *cis/trans* (**33**/**32**) isomers produced was 7.3.

Anal. Calcd. for $C_{13}H_{18}$: C, 89.60; H, 10.41. Found: C, 89.77; H, 10.29.

6. **8-Methyl-1,2,3,3a,8,8a-hexahydrocyclopent[*a*]indene (35).** A.—**1-Methyl[2,3]cyclopenta-1-indanol (34)** was prepared in 67% yield by treating 8.3 g. (0.059 mole) of methyl iodide, 1.4 g. (0.059 g.-atom) of magnesium, with 9.6 g. (0.056 mole) of [2,3]cyclopenta-1-indanone (**28**). Compound **34** recrystallized from *n*-hexane, m.p. 74–75°, and was characterized by an infrared absorption spectrum (KBr pellet)[34] having a band of methyl group at 7.29 μ and of hydroxyl group at 2.90 μ.

B. **8-Methyl-1,2,3,3a,8,8a-hexahydrocyclopent[*a*]indene** (**35**).—Dehydration of the alcohol **34** by means of a small amount of potassium bisulfate, followed by catalytic hydrogenation over platinum oxide, gave 1.5 g. of compound **35** in 37% yield: b.p. 120–124° (12 mm.), $n^{15}\mathrm{D}$ 1.5467.

Anal. Calcd. for $C_{13}H_{16}$: C, 90.64; H, 9.36. Found: C, 90.50; H, 9.41.

7. **1,2,3,4,4a,9a-Hexahydrofluorene (36).**—The hydrogenation of fluorene (47.5 g., 0.30 mole) was performed in a 450-ml. rotating autoclave in the presence of 4.0 g. of nickel–kieselguhr catalyst under an initial hydrogen pressure of 110 atm. at 150–170° for 4 hr. At the end of the reaction, the pressure had dropped to 56 atm. (at room temperature). Compound **36** was obtained in 64% yield: b.p. 124–125° (13 mm.), $n^{20}\mathrm{D}$ 1.5530 (lit.[35] b.p. 127° at 15 mm., $n^{10.9}\mathrm{D}$ 1.5572).

Anal. Calcd. for $C_{13}H_{16}$: C, 90.64; H, 9.36. Found: C, 91.00; H, 9.24.

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[29] P. Maitte, *Bull. soc. chim. France*, 502 (1959).

[30] A. S. Hussey, *J. Am. Chem. Soc.*, **73**, 1364 (1951).

[31] W. Baker and P. G. Jones, *J. Chem. Soc.*, 791 (1951).

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[33] N. G. Sidorova and I. P. Tsukervanik, *J. Gen. Chem. USSR*, **10**, 2073 (1941).

[34] K. Nakaniski, "Infrared Absorption Spectroscopy," Holden-Day, Inc., San Francisco, Calif., 1962, p. 20.

[35] J. W. Cook and C. L. Hewett, *J. Chem. Soc.*, 62 (1936).

The Alkali Metal Catalyzed Reactions of ω-Phenyl-1-alkenes. The Dimerization of 4-Phenyl-1-butene. The Cleavage and Cyclization Reaction of 5-Phenyl-1-pentene[1,2]

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Received February 5, 1965

The reaction of 4-phenyl-1-butene and of 5-phenyl-1-pentene in the presence of sodium, potassium, and cesium has been studied. 4-Phenyl-1-butene at 185° in the presence of potassium as a catalyst yielded 39.6% of 1-phenyl-2-ethyl-3-benzylcyclopentane, 23.7% of butylbenzene, and some naphthalene. 5-Phenyl-1-pentene at 135° in the presence of potassium or cesium as a catalyst gave C_3 and C_4 gases, toluene, ethylbenzene, 1,3-diphenylpropane, 1-methylnaphthalene, phenylcyclopentane, etc. The difference in catalytic behavior between potassium and cesium was characterized by (1) production of cyclopropane and allene only in the potassium-catalyzed reaction, and (2) the ratio of cleavage products to cyclization products being greater with potassium than with cesium. Sodium caused primarily the migration of double bonds.

The alkali metal catalyzed reactions[4,5] of 6-phenyl-1-hexene and 7-phenyl-1-heptene have been described in the preceding papers.[1,6] In the presence of potassium or cesium as catalyst, the predominant reaction was the cyclization resulting in the formation of 1-methyl-2-phenylcyclopentane from 6-phenyl-1-hexene and of 1-methyl-2-phenylcyclohexane from 7-phenyl-1-heptene. The size of the rings seemed to be determined by the stability of their carbanion intermediates (polar effect, *i.e.*, primary > secondary > tertiary). On the other hand, sodium as catalyst gave products[5] mainly resulting from double-bond migrations.

In order to shed more light on the possible merits and limitations of this type of reaction, these studies have been extended to 4-phenyl-1-butene and 5-phenyl-1-pentene. The primary purpose was to investigate whether these hydrocarbons might undergo cyclization reactions under the conditions described in the preceding papers.[1,6] The catalysts used in this study were sodium, potassium, and cesium.

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[1] Paper XXX of the series, "Base-Catalyzed Reactions". Paper XXIX: H. Pines, N. C. Sih, and E. Lewicki, *J. Org. Chem.*, **30**, 1457 (1965).

[2] This work was supported in part by a National Science Foundation Grant, NSF-G14503.

[3] Taken in part from the Ph.D. Thesis of N. C. Sih, Northwestern University, June 1964.

[4] The actual catalyst is alkali metal–organoalkali metal.

[5] For literature references, see H. Pines and L. A. Schaap, *Advan. Catalysis*, **12**, 116 (1960).

[6] E. Lewicki, H. Pines, and N. C. Sih, *Chem. Ind.* (London), 154 (1964).

TABLE I

EXPERIMENTAL CONDITIONS USED FOR THE REACTION OF 4-PHENYL-1-BUTENE AND 5-PHENYL-1-PENTENE

Catalyst (g.-atom)	Reactants, mole ω-Phenyl-1-alkene	Reactants, mole o-Chlorotoluene	Solvent (mole)	Reactn. time, hr.	Temp., °C.	% conversion
			4-Phenyl-1-butene[a]			
Na (3.75×10^{-2})	7.5×10^{-2}	7.5×10^{-3}	*sec*-Butylcyclohexane (7.5×10^{-2})	9	~185	80.8
K (7.5×10^{-2})	1.5×10^{-1}	1.5×10^{-2}	*n*-Butylcyclohexane (1.5×10^{-1})	4.5	~185	98.7
Cs (1.15×10^{-2})	2.3×10^{-2}	2.3×10^{-3}	*sec*-Butylcyclohexane (6.9×10^{-2})	5.75	~185	86.2
			5-Phenyl-1-pentene[b]			
Na (5.0×10^{-2})	1.0×10^{-1}	1.0×10^{-2}	Ethylcyclohexane (1.0×10^{-1})	7.25	~135	75.2
K (6.85×10^{-2})	1.37×10^{-1}	1.37×10^{-2}	Ethylcyclohexane (1.37×10^{-1})	18.5	~135	65.2
Cs (2.07×10^{-2})	4.13×10^{-2}	4.13×10^{-3}	Ethylcyclohexane (1.24×10^{-1})	8.5	~135	84.5

[a] Purity was 99.5% (v.p.c., column A). [b] Purity was >98% (v.p.c., column A).

Discussion of Results

The reactions were performed according to the procedure described for 6-phenyl-1-hexene and 7-phenyl-1-heptene,[1] except that a lower reaction temperature (about 135°) was used for the reaction with 5-phenyl-1-pentene (Table I).

A. 4-Phenyl-1-butene (Table II).—The formation of *n*-butylbenzene (**2**), 4-phenylbutenes (**3**–**5**), naphthalene (**6**), and 1-phenyl-2-ethyl-3-benzylcyclopentane (**7**) is illustrated schematically in Scheme I, p. 1464.

TABLE II

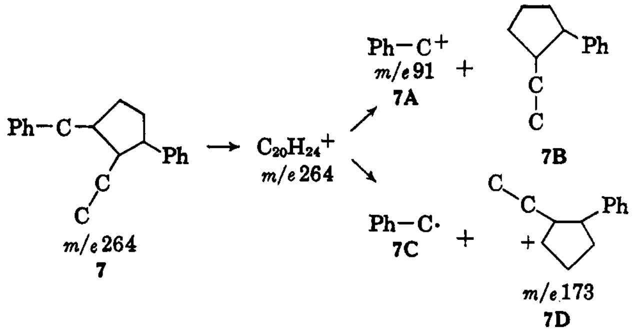
COMPOSITION OF PRODUCTS OBTAINED FROM 4-PHENYL-1-BUTENE

Products	Catalyst Na	Catalyst K	Catalyst Cs
	Composition, mole %		
Toluene[a]	Detected	Detected	Detected
n-Butylbenzene (**2**)	9.8	23.7	3.6
1-Phenyl-2-butene (**3**)	5.8	0	2.3
1-Phenyl-*cis*-1-butene (**4**)	15.4	0	4.2
1-Phenyl-*trans*-1-butene (**5**)	39.9	0	32.9
Naphthalene (**6**)	Trace	2.6	Trace
1-Phenyl-2-ethyl-3-benzylcyclopentane (**7**)	3.0	39.6	0
Others	26.1	34.1	56.2

[a] Originated from the initiator.

Compound **2** arises from hydride addition and compounds **3**–**5** are formed by double bond migrations. Unlike the reactions observed previously,[1,6] the benzylic carbanion **1b** failed to undergo an internal addition to the terminal double bond to form 1-methyl-2-phenylcyclopropane (**8**) and phenylcyclobutane (**9**). Furthermore, the possible products of ring openings of compounds **8** and **9**, namely 2-phenylbutenes, were also not observed. This absence of intramolecular cyclization could be attributed to the high strain existing in small-ring compounds. Instead, two other modes of cyclization occurred leading eventually to naphthalene (**6**) and 1-phenyl-2-ethyl-3-benzylcyclopentane (**7**). Compound **6** is a dehydrogenation product, either of tetralin (**6e**) or of benzocyclohexene (**6b**), resulting from the intramolecular nuclear attack of corresponding carbanion **3a** or **2a**. The dimeric compound **7** might be produced either in a concerted or in a stepwise manner involving an intermolecular attack of 1-phenyl-1-butene (**4, 5**) on the allylic carbanion **1a** to give the secondary cyclopentyl carbanion **7b**. Thus the difference in the ring strain between a four- and five-membered system outweighs the difference in the stability between a primary and secondary carbanion. If the polar effect were the only controlling factor, a primary cyclobutyl carbanion would be formed preferentially. Other dimers from the reaction, which could not be identified because of the small yields, might be the stereoisomers of compound **7**, open-chain dimers or hydro dimers similar to those obtained from β-methylstyrene.[7,8]

The structure of compound **7** was assigned on the basis of physical and chemical evidence. Its infrared spectrum showed the presence of a methyl group (part of an ethyl group, *cf.* n.m.r. data), a tertiary benzylic hydrogen (Ph-CHR'R''), and monosubstituted phenyl groups. The n.m.r. spectrum indicated that compound **7** possessed two phenyl groups of different environments, but only one ethyl group, characterized by a methyl H triplet which had an integrated area ratio, methyl/other H, of 3:21. The mass spectrum gave the following *m/e* values: 91, 173, and 264. The mass unit of 264, representing the parent ion ($C_{20}H_{24}^+$), implied that compound **7** contained an alicyclic ring besides two phenyl groups, since compound **7** was stable to potassium permanganate solution[9] and was resistant toward hydrogenation. Therefore compound **7** corresponds to 1-phenyl-2-ethyl-3-benzylcyclopentane, which underwent fragmentation into the benzylium ion (**7A**) and the 2-ethyl-3-phenylcyclopentyl radical (**7B**), or into the benzyl radical (**7C**) and the 2-ethyl-3-phenylcyclopentanium ion (**7D**) during the mass spectrometric analysis. Cations **7A** ($C_7H_{17}^+$) and **7D** ($C_{13}H_{17}^+$) have mass units of 91 and 173, respectively.



B. 5-Phenyl-1-pentene (Table III and IV).—The different catalytic behavior of the alkali metals was further illustrated with the reactions of 5-phenyl-1-pentene (**10**).

The gaseous products collected from the potassium- and cesium-catalyzed reactions are derived from the

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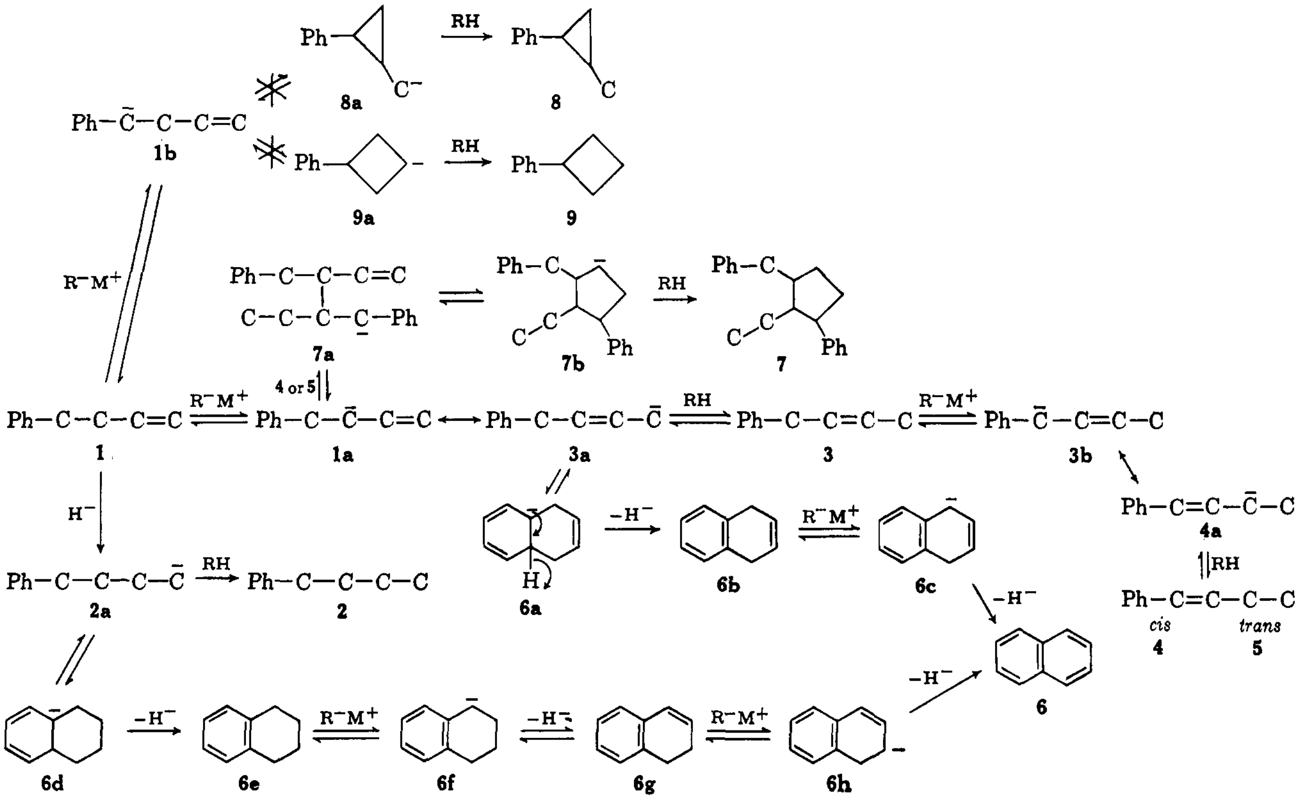
(7) J. Shabtai, E. M. Lewicki, and H. Pines, *J. Org. Chem.*, **27**, 2618 (1962).

(8) J. Shabtai and H. Pines, *ibid.*, **29**, 2408 (1964).

(9) V. N. Ipatieff, W. W. Thompson, and H. Pines, *J. Am. Chem. Soc.*, **70**, 1658 (1948).

SCHEME I

MECHANISM FOR THE FORMATION OF THE PRODUCTS FROM THE REACTION OF 4-PHENYL-1-BUTENE



M^+ = cation of alkali metal

R^- = $C_6H_5CH_2^-$ (from the initiator) or any anion produced in the reaction

cleavage of the allylic (**10a**) as well as the benzylic (**10b**) carbanions as outlined in Scheme II. The recovery of the C_3 gases was nearly quantitative, while that of the C_4 gases was less than 7%. The low yield of the C_4 gases may be attributed to the ease of polymerization of butadiene (**20**). Several features seem to be noteworthy. The unexpectedly high reactivity of the propenylpotassium was demonstrated by its ability to cyclize to cyclopropane, and to undergo hydride elimination to form allene. The cyclopropane precursor **15a** is not only a less stable species (secondary), but also consists of a ring of great angular strain. Thus, in spite of the high contribution of the two equivalent allylic resonance structures (sp^2), the propenyl carbanion **13a** must retain some tetrahedral character (sp^3) to permit cyclization to occur. The formation of the highly energetic allene provides another unusual characteristic of the propenylpotassium. *trans*- and *cis*-2-butene (**18**, **19**) were kinetic products[10] judging by the ratio of these gases produced from the reactions. Toluene (**21**), ethylbenzene (**22**), and styrene (**23**) are also cleavage products. Interaction between the tolyl carbanion (**21a**) and styrene (**23**) gives 1,3-diphenylpropane (**30**). The mechanism of the formation of *n*-pentylbenzene (**24**) and 5-phenylpentenes (**25**, etc.) is the same as that described for compounds **2** and **3**, etc. The relatively low yield of phenylcyclopentane (**26**) is likely owing to the fact that the secondary carbanion intermediate **26a** is comparatively a less stable one (*cf.* the relative yield of methylphenylcycloalkanes from the reactions of 6-phenyl-1-hexene and 7-phenyl-1-heptene in the preceding papers[1,6]). 1-Methyl-1,2,3,4-tetrahydronaphthalene (**28**) and 1-methylnaphthalene (**29**) probably originate from methylbenzocyclohexene (**27**) which, in turn, results from the intramolecular alkylation of the reactive carbanion **25b**.

TABLE III

COMPOSITION OF THE GASEOUS PRODUCTS OBTAINED FROM 5-PHENYL-1-PENTENE[a]

	Catalyst	
	K[b]	Cs[c]
Products	Composition, mole %	
Ethylene (**11**)	3.5[d]	Trace
Propane (**12**)	57[e]	4.6[f]
Propene (**13**)	11[e]	86[f]
Allene (**14**)	9.2[e]	0
Cyclopropane (**15**)	11[e]	0
n-Butane (**16**)	0	Trace
1-Butene (**17**)	1.7[g]	0.48[h]
trans-2-Butene (**18**)	2.4[g]	1.2[h]
cis-2-Butene (**19**)	2.4[g]	1.6[h]
Butadiene (**20**)	0	Trace

[a] Only a trace amount of gases was produced from the sodium-catalyzed reaction. [b] Total = 0.0143 mole at STP. [c] Total = 0.00799 mole at STP. [d] Based on total amount of the gases produced. [e] Based on the total moles of toluene and 1,3-diphenylpropane produced; total percentage of C_3 gases, 82.7%. [f] Same as *e*, except the total percentage of C_3 gases, 90.7%. [g] Based on the total moles of ethylbenzene, styrene, and 1,3-diphenylpropane produced; total percentage of C_4 gases, 6.5%. [h] Same as *g*, except the total percentage of C_4 gases, 3.2%.

The sodium-catalyzed reaction of 5-phenyl-1-pentene (**10**) gave only very little of the cleavage product but none of cyclization product.

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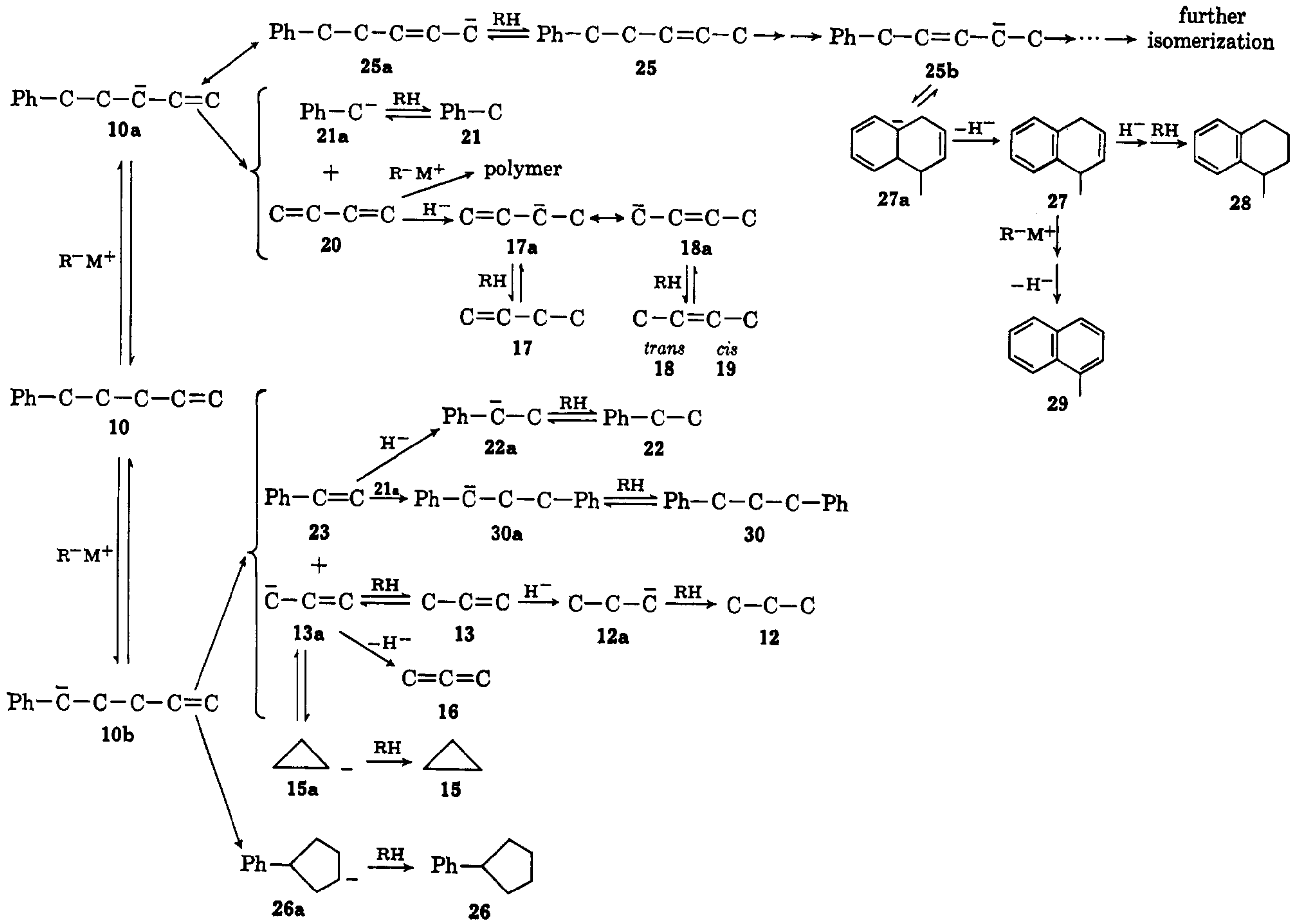
(10) The *cis/trans* equilibrium ratio of 2-butenes is 3.32 (25°) calculated from their free energies of formation given in "Selected Values of Physical and Thermodynamic Properties of Hydrocarbons and Related Compounds," Carnegie Press, Pittsburgh, Pa., 1953, p. 475.

SCHEME II

MECHANISM FOR THE FORMATION OF THE PRODUCTS FROM THE REACTION OF 5-PHENYL-1-PENTENE



M⁺ = cation of alkali metal

R⁻ = $C_6H_5CH_2^-$ (from the initiator) or any anion produced in the reaction

TABLE IV

COMPOSITION OF AROMATIC HYDROCARBONS OBTAINED FROM 5-PHENYL-1-PENTENE

Products	Catalyst		
	Na	K	Cs
	Composition, mole %		
Toluene (**21**)	Detected[a]	42.3[b,c]	17.1[b,d]
Ethylbenzene (**22**)	1.5	12.2[e]	6.3[f]
Styrene (**23**)	0	0	1.4[f]
n-Pentylbenzene (**24**)	10.2	10.7	15.0
5-Phenyl-*x*-pentenes (**25**, etc., *x* = 2, 3, 4)	56.1	15.1	13.5
Phenylcyclopentane (**26**)	0	8.8	8.9
1-Methyl-1,2,3,4-tetrahydronaphthalene (**28**)	0	0	15.4
1-Methylnaphthalene (**29**)	0	>0.9	6.3
1,3-Diphenylpropane (**30**)	0	6.2[c,e]	16.1[d,f]
Others	32.2	<3.8	~0

[a] Originated from the initiator. [b] The amount of toluene originated from the initiator was deducted. [c] The amount of cleavage, giving C_4 gases, was 48.5%. [d] The amount of cleavage, giving C_4 gases, was 33.2%. [e] The amount of cleavage, giving C_3 gases, was 18.4%. [f] The amount of cleavage, giving C_3 gases, was 23.8%.

C. Summary.—The catalytic behavior of sodium, potassium, and cesium toward 5-phenyl-1-pentene is noticeably different. Sodium causes mainly double-bond migrations, while potassium and cesium give rise to cleavage and cyclization reactions. Cesium is especially effective as a catalyst for those cyclizations involving nuclear alkylation resulting in the production of tetralin and naphthalene derivatives (**28**, **29**) from 5-phenyl-1-pentene, and tricyclic compounds from 6-phenyl-1-hexene and 7-phenyl-1-heptene.[1,6] Potassium differs from cesium in its capability to form cyclopropane and allene from a propenyl carbanion.

No significant differences were observed from the reactions of 4-phenyl-1-butene. Only potassium causes an appreciable amount of dimerization.

4-Phenyl-1-butene and 5-phenyl-1-pentene did not cyclize to give cyclopropyl and cyclobutyl compounds. Whenever a ring of low strain (five- or six-membered) can be produced,[1,6] the intramolecular cyclization will occur in the presence of potassium or cesium. Otherwise, other competing reactions, such as cleavage, hydride addition, dimerization, and isomerization, will prevail.

Experimental

The reactions were carried out according to the procedure described in the preceding paper.[1] Four additional v.p.c. columns were used: column F, 0.25 in. × 2.4 m., Davison's 950 silica gel; column G, 0.25 in. × 3.8 m., 33% dimethylsulfolane on 100–120-mesh firebrick; column H, 0.375 in. × 4.3 m., 10.75% silicone oil DC-550 on 45–60-mesh Chromosorb P; and column I, 0.25 in. × 4.0 m., 10% silicone oil DC-550 on 30–60-mesh Chromosorb P. The v.p.c. relative retention times for gases and liquids are given separately in Table V and VI.

TABLE V

RELATIVE RETENTION TIMES OF GASES OVER COLUMN G[a]

Compd.	Time
Ethylene (**11**)	0.274
Propane (**12**)	0.592
Propene (**13**)	1.00
Cyclopropane (**15**)	2.14
1-Butene (**17**)	2.68
Allene (**14**)	3.14
trans-2-Butene (**18**)	3.44
cis-2-Butene (**19**)	4.05

[a] Flow rate of helium, 40 ml./min.; temperature, 52°.

TABLE VI

RELATIVE RETENTION TIMES OF AROMATIC HYDROCARBONS INVOLVED IN THE REACTIONS

	Column						
	A			B			I
	Temp., °C.						
	100	150	200	150	200	225	125
	Flow rate of helium, ml./min.						
Compd.	82	75	75	75	75	75	75
Toluene (**21**)	0.109						0.271
Ethylbenzene (**22**)	0.172						0.474
Styrene (**23**)		0.494		0.293			
n-Butylbenzene (**2**)		0.645		0.602			
n-Pentylbenzene (**24**)	1.00	1.00		1.00			2.46
4-Phenyl-1-butene (**1**)		0.755					
5-Phenyl-1-pentene (**10**)		1.25					
Phenylcyclopentane (**26**)		1.96		1.63			
Naphthalene (**6**)		3.97	1.00		1.00	1.00	
1-Methyl-1,2,3,4-tetrahydronaphthalene (**28, 31**)			0.586		1.03		
1-Methylnaphthalene (**29**)			1.54			1.61	
1,3-Diphenylpropane (**30**)			3.99			4.80	
1-Phenyl-2-ethyl-3-benzylcyclopentane (**7**)			(9.72)			(14.7)	
1-Phenyl-2-butene (**3**)		0.948					
1-Phenyl-*cis*-1-butene (**4**)		0.858					
1-Phenyl-*trans*-1-butene (**5**)		1.31					

The following hydrocarbons were available either from commercial sources or from this laboratory: ethylene, propane, cyclopropane, allene, butane, 1-butene, *cis*- and *trans*-2-butene, butadiene, toluene, ethylbenzene, styrene, *n*-butylbenzene, *n*-pentylbenzene, phenylcyclopentane, naphthalene, 1-methylnaphthalene, 1,3-diphenylpropane, *cis*- and *trans*-1-phenyl-1-butene, and 4-phenyl-1-butene.

5-Phenyl-1-pentene (**10**) was synthesized by a condensation reaction between a Grignard reagent of β-phenylethyl bromide and allyl bromide in 67% yield. The product always contained some phenylethyl bromide as impurity, even after careful distillation using a spinning-band column. It was further purified by a Grignard reaction to eliminate phenylethyl bromide as ethylbenzene. Subsequent distillation (b.p. 84–85° at 17 mm., $n^{25}\mathrm{D}$ 1.5032; lit.[11] b.p. 77–78° at 10 mm., $n^{25}\mathrm{D}$ 1.5065) gave 5-phenyl-1-pentene (**10**) in 98% purity.

1-Methyl-1,2,3,4-tetrahydronaphthalene (**31**) was synthesized from α-tetralone and methylmagnesium iodide, followed by dehydration and catalytic hydrogenation. Pure compound **31** ($n^{26}\mathrm{D}$ 1.5340, lit.[12] n^{20}_{He} 1.5332), separated by v.p.c. over column D, had the same infrared spectrum as that published.[13]

Structure of 1-Phenyl-2-ethyl-3-benzylcyclopentane (**7**).—Pure compound **7** (b.p. 209–211° at 13 mm., m.p. 44.5–45.8°) was obtained from v.p.c. purification over column H. Among other bands, its infrared spectrum was characterized by a methyl group (7.26 μ, KBr),[14] monosubstituted phenyl groups (5.0–6.0-μ region, Nujol),[15] and a tertiary benzylic hydrogen (13.2 μ, KBr).[16] The n.m.r. spectrum[17] (Varian A-60, TMS as standard) exhibited a methyl triplet (δ center 0.81), and two phenyl groups (δ 7.00, 7.16), and the ratio of methyl/other H was 3.06:21.0, calculated from its integrated area. The mass spectrum gave *m/e* values of 91, 173, and 264. It resisted hydrogenation (over PtO_2) and was stable toward potassium permanganate solution.

Anal. Calcd. for $C_{20}H_{24}$: C, 90.84; H, 9.15. Found: C, 90.88; H, 9.00.

Acknowledgment.—The authors are indebted to Universal Oil Products Company for the mass spectrometric measurement, and to Mr. Edward Lewicki for technical assistance.

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