Displacement of Alkali Metal by Mercury and the Dissociation of Ion Pairs

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I. Introduction

A standard method for preparing organoalkali metal compounds is by addition of an alkali metal to an organomercurial. Most textbooks in organic chemistry mention it. Schlenk and Holtz[1] used it in 1917 in their classical preparation of alkylsodium compounds. Generally stated it is a reaction in which a more positive metal of the electromotive force (EMF) series displaces a less positive one. Rarely is reversibility mentioned. But mercury does, indeed, displace an alkali metal from these ion pairs. An amalgam forms. That fact affects the interpretation of the chemistry of these half-organic–half-inorganic salts, for the cations of inorganic salts such as sodium chloride and hydroxide form no amalgam. The cation must first acquire an electron at the cathode of an electrolytic cell, as in well-known commercial processes. Only a metal amalgamates with mercury, and the cation cannot acquire the necessary electron from mercury or an anion such as chloride or hydroxide. So in this reversal that electron must come from the carbanion, for it is the only chemical agent that is different. In other words, the ion pair dissociates to a radical–metal pair. That change contradicts the popular notion of an ionic dissociation, from which have come the designations (anionic, carbanionic, nucleophilic reactions) that attribute to the carbanion a dominant or sole role, with little regard to the cation. But atomic metal, formed in this dissociation, is a very active agent. Both halves of the ion pair really function. Ion pair or radical–metal pair chemistry appears more appropriate as a name than carbanion chemistry.

This review describes these interesting formations of amalgams—this seeming reversal of a rule. Some other metals displace alkali metals too. No study of optimum conditions has been made, but enough has been done to ensure that amalgam formation is the rule. The few exceptions are easily explained. The subject is important. It affects ideas on mechanism about which much has been written in the anionic fashion.

A dissociation to radical–metal is reasonable, too. Physical properties (the low electron affinity of carbon, the thermal instability of these ion pairs, the nondissociating media) accord with, even favor, the idea.

A distinction exists between the EMF series for irreversible displacement and the present type of reversible ones. For good reason they seem different.

II. Displacement of Alkali Metals

A. Sodium by Mercury

The first of these direct formations of amalgam was observed by Wanklyn[2] in 1866. He had been preparing ethylsodium in the customary way from sodium and diethylzinc in accord with the "well-known electrochemical precipitation from ordinary metallic solutions". Very sensibly he tested the reverse order by sealing ethylsodium with mercury, always with another metal (copper, iron, or silver) as an amalgam, for he was seeking formation of any organometallic compound. Then he heated the tubes on a water bath. Sodium amalgam and diethylmercury were obtained in each case.

In a superb and thorough study of the electron affinity of triarylmethyls, Bent and coworkers[3] regulated the amounts of sodium cation and free radicals by dilute sodium amalgam as in eq 1. The equilibrium in ether was approached from both

$$R_3CNa + nHg \text{ (from } NaHg_n) \rightleftarrows NaHg_n + R_3C\cdot \quad (1)$$

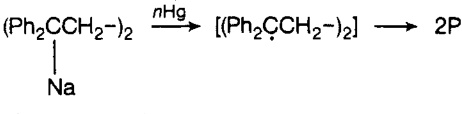
sides. Sodium amalgam with triphenylchloromethane gave the dark red sodium compound. The color partially disappeared when shaken with mercury and was restored by mercury amalgam. The electron affinity, to be mentioned again in section III, was 59 ± 5 kcal. For a series of seven triarylmethyls listed in Table I the value of 60 kcal can be taken as a first approximation. The larger free radicals have slightly greater affinities for sodium and therefore slightly greater electron affinities.

The conductance[4] of triphenylmethylsodium in ether, which has commonly been accepted as evidence for an ionic dissociation, was only 4.8×10^{-2}—so low as to indicate, according to Bent, an insignificant amount of dissociation. Later measurement,[5] particularly the work of Swift, confirmed its minor role and suggested that the electron affinity of triphenylmethyl should be lowered to 48 ± 5 kcal.

If the resonance imparted by three aryl groups in the very stable carbanion salts cannot maintain carbon in the ionic state, how can a carbanion without resonance hold an electron firmly and perform as a free anion in a reaction?

Schlenk and Bergmann[6] found that mercury and tetraphenylethanedisodium formed amalgam and tetraphenylethylene as in eq 2. Also mercury with 1,1,4,4-tetraphenylbutanedisodium-1,4 as in eq 3, gave amalgam and 1,1-diphenylethylene, a

$$\underset{\text{Na}}{Ph_2C}-\underset{\text{Na}}{CPh_2} + nHg \longrightarrow Na_2Hg_n + Ph_2C{=}CPh_2 \quad (2)$$



$$(\underset{\text{Na}}{Ph_2C}CH_2-)_2 \xrightarrow{nHg} [(Ph_2\underset{\cdot}{C}CH_2-)_2] \longrightarrow 2Ph_2C{=}CH_2 \quad (3)$$

cleavage to the original hydrocarbon, which with sodium had formed the tetraphenylbutanedisodium.

Ziegler and coworkers[7,8] questioned the direct formation of

TABLE I. Free Energies and Equilibrium Constants of Triarylmethyls

R in R_3C	F, kcal	Log K[a]
$3C_6H_5$	−17.9	13.1
$2C_6H_5$, $C_6H_5C_6H_4$	−18.1	14.0
$2C_6H_5$, α-$C_{10}H_7$	−18.6	13.6
$2C_6H_5C_6H_4$, β-$C_{10}H_7$	−18.6	13.9
C_6H_5, α-$C_{10}H_7$, $C_6H_5C_6H_4$	−19.5	14.3
$2C_6H_5C_6H_4$, α-$C_{10}H_7$	−20.2	14.8
$3C_6H_5C_6H_4$	−20.6	15.0

[a] Log K for the reaction $R + Na = R^- + Na^+$ from which the free energy is obtained by $\Delta F = -RT \ln K$.

tetraphenylethylene in eq 2, claiming that it might form through an intermediate mercurial, $Ph_2\underline{C\text{-}Hg\text{-}C}Ph_2$, which decomposed. For evidence they showed that benzyllithium with mercuric chloride yielded dibenzylmercury, eq 4, which was

$$2PhCH_2Li \xrightarrow{HgCl_2} (PhCH_2)_2Hg \xrightarrow{170°} PhCH_2CH_2Ph \qquad (4)$$

stable at room temperature but decomposed at 170°C to diphenylethane, analogous to tetraphenylethane. Also this method for preparing a mercury compound, when applied to diphenylmethylsodium (eq 5), yielded tetraphenylethane instead, so the assumed mercury intermediate was plausible.

$$2Ph_2CHNa + HgCl_2 \longrightarrow Ph_2CHCHPh_2 \qquad (5)$$

Both sides of this controversy have merit. The direct formation of tetraphenylethylene is reasonable in the light of Bent's work that demonstrated so clearly that a radical does form when mercury displaces sodium. Two radicals on adjacent carbons should join promptly to make a double bond. Union of a radical with metal is also reasonable in view of Paneth's work with gaseous radicals and metal films. But if mercury compounds decompose at the reaction temperature, perhaps they never form. There need not be just one road to the hydrocarbon. And regardless of such details sodium amalgam is produced by either course. An amalgam is proof that alkali metal forms. It is incredible that mercury, far down in the EMF series, can furnish the necessary electron to produce the alkali metal, far up in the series. No metal forms from mercury and sodium chloride or hydroxide. The reasonable source of that electron is the carbanion. The ion pair dissociates to a radical–metal pair.

Conflicting opinions arose also over the displacement reaction in the case of diphenylmethylsodium. Ziegler and Colonius[7] noted no reaction whereas Bergmann[9] obtained (eq 6)

$$2Ph_2CHNa \xrightarrow{nHg} Na_2Hg_n + Ph_2CHCHPh_2 + \text{an isomer} \qquad (6)$$

the amalgam, *tetraphenylethane, and an unidentified isomer.* A possible obstacle in that displacement will be explained later in connection with similar trouble with some lithium compounds.

B. Lithium by Mercury

In their notable paper on the preparation of butyllithium from butyl halides and lithium, Ziegler and Colonius[7] recorded that butyllithium in cyclohexane with mercury formed lithium amalgam and dibutylmercury. They recognized that the preparation of alkyllithium from dialkylmercury had to be reversible. Also benzyllithium[8] in ether similarly yielded an amalgam and dibenzylmercury, which, as noted before, decomposed to diphenylethane at 170°.

In 1939 Talalaeva and Kocheshkov[10] reported that phenyllithium with mercury formed diphenylmercury. They observed also the displacement of lithium by a number of other metals. The latter will be described in section II.C.

TABLE II. Products, besides Amalgam, from the Reaction of Organolithium Compounds with Mercury

R of RLi	Solvent	Yield of R_2Hg, %
n-Butyl	Benzene	64
n-Amyl		62
n-Hexyl		74
n-Dodecyl		80[a]
Benzyl	Ether	10[b]
2-Phenylethyl		[c]
sec-Butyl	Benzene	37
Cyclohexyl	Ether	[c]
tert-Butyl	Pentane	Traces
Triphenylmethyl	THF	
Phenyl	Ether	10
o-Tolyl		30
m-Tolyl		20
p-Tolyl		41
2-Naphthyl		20
p-Ethylphenyl		14[d]
Diphenylmethyl		
1-Naphthyl		
1-Phenylethyl		29[b]
Cumyl	THF	33

[a] The yield is a mixture of didodecylmercury and tetracosane. [b] The yield is based on the alkyl halide used in the preparation of RLi. [c] The organomercurial was so difficult to isolate that the crude product was converted to RHgX. [d] Yield of RR.

The work of Beinert and Parrod[11,12] embraces the largest number of organoalkali metal compounds so far studied in this field. They stirred mercury with dilithium compounds having the formula $Li(CH_2)_nLi$ where n is 4, 5, 7, 8, 9, 10. The products where n is 5, the preferred number, were amalgam, cyclopentamethylenemercury, cyclodipentamethylenemercury, and a gray powder which had the formula $[Hg(CH_2)_5]_n$.

They also agitated solutions of a number of monolithium compounds[12] with a large excess of mercury. Table II shows that about half of the yields were moderate to high. A few were very low or zero. Condensation products sometimes formed.

According to Beinert[12] the reaction depends on the polarity of the carbon–lithium bond. If the polarity is relatively weak, as in primary and secondary butyllithium, one gets the organomercurial. If moderate, as in phenyllithium, one finds an equilibrium. If strong, as in *tert*-butyllithium, there is no reaction.

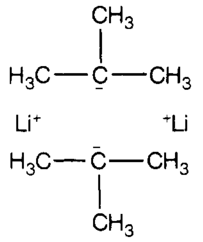
These conclusions are proper, if judgment is based on those yields. But there are questions. Consider, for example, the condensation products, RR, which form either before or after any mercurial. In either case the road to RR is irreversible. Therefore, even if the equilibrium were far to the left, the reaction would steadily be drawn to the right. The yield should reach 100%. The highest was only 33%.

Next, the displacement of mercury by alkali metal is one of the surest ways to get an organoalkali metal compound. It rarely fails. The reverse reaction is different. Sometimes it does not work. Possibly the weight of mercury prevents good contact despite vigorous mixing. One does not make mercury sand, chips, or wire as with alkali metals. Particles of lithium, sodium, or potassium settle more slowly than mercury and thus have longer contact with the solution. Different surface tensions about the particles might affect the reaction too.

Finally what do *tert*-butyllithium and triphenylmethyllithium have in common that causes both to resist displacement? They are very unlike otherwise. The former is a very active compound. The methyl groups release electrons and lower the acidity of a system. The carbon–metal bond should be weak. By contrast, the latter has a fairly stable carbanion.

The phenyl groups impart resonance and enhance acidity.

Usually when reactivity does not line up as expected, a steric factor is present. That possibility exists here because these salts tend to associate. The cation could be hidden, for the percentage of metal by weight is low and the volume percentage of cation is still lower. A possible dimeric form (there are others) is shown in I and II. Tri- and tetrameric forms would conceal the cation even more. The lithium cation would be shielded more than sodium, other factors being equal. Bent[3] had no trouble, but he worked with very dilute solutions of triarylmethylsodium. The same difficulty might have existed with diphenylmethylsodium mentioned in section II.A. Only in the work of Bent and coworkers was the equilibrium determined to a certainty. No similar steric difficulty is experienced when an alkali metal reacts with a mercury compound.



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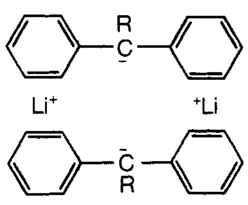


R = H or Ph

II

All of these factors indicate that formation of the organoalkali metal compound is very fast. The reverse reaction is extremely slow. Better conditions should be used. For one thing an amalgam, such as with copper, iron, or silver, as used by Wanklyn,[2] might provide a better surface for the reaction with mercury. Dilution should help too. Triethylamine or *N,N,N′,N′*-tetramethylethylenediamine should have a favorable effect. The former accelerated a polymerization of butadiene with amylsodium[13] and increased the activity of butyllithium so that it metalated *tert*-butylbenzene.[14] The latter promoted telomerization[15] of ethylene with aromatic hydrocarbons and also facilitated the metalation of cumene[16] by the same reagent, butyllithium. Amines coordinate with the cation and therefore increase the distance between the two ions. Thus they make dissociation of the ion pair easier. They do not coordinate with, and stabilize, the anion. It has the same low electron affinity. So dissociation of the ion pair to a radical–metal pair should be facilitated but not altered, that is, not made ionic.

Maercker and Roberts[17] added mercury to a preparation of γ,γ-diphenylallylcarbinyllithium with the intention of removing only excess sodium–potassium left over from a step in the preparation—clear evidence of a belief that a cation does not react with mercury. Nevertheless, an amalgam and the corresponding mercury compound, $[(C_6H_5)_2C{=}CHCH_2CH_2]_2Hg$, formed. They explained this unexpected result just as did Ziegler and Colonius[7]: the exothermic formation of an amalgam shifted the equilibrium to the organomercury side. Probably the heat of adsorption or collision has some effect, but a metal must form before amalgamation. Also displacement by some other metals occurs as will be described in the next section. No significant amount of heat seems to occur there. And even if that heat is important, mercury is not unique. Alkali metal halides and alkoxides form and produce heat in reactions with compounds that contain halogen and oxygen. In other words dissociation to radical–metal is a function of the ion pair, whatever the reactant.

They also obtained, by mercury displacement of alkali metal with high-speed stirring in an ice bath, diphenyl- and dibutylmercury, the latter in 91% yield, the highest ever. But potassium and lithium carbinyl salts, $Ph_2\bar{C}(M^+)\overline{CHCH_2C}H_2$, were reported as not reacting with mercury. That result accords with the steric difficulty mentioned previously. However, the potassium salt probably did react somewhat, for they could not explain the presence of 25% of the methane, $Ph_2CH\overline{CHCH_2C}H_2$, after a 1 *N* solution of that salt was stirred with mercury to remove sodium–potassium alloy. That concentration, at least 350 times more than used by Bent,[3] would conceal the alkali metal cation and make displacement by mercury difficult. In spite of that obstacle 25% displacement of potassium apparently took place, and the carbon radical probably removed hydrogen from the solvent in order to form the methane.

C. Sodium or Lithium by Other Metals

The experiments of Wanklyn[2] mentioned in section II.A included two where ethylsodium reacted with amalgams of magnesium and zinc. No solvent was mentioned. The reactants were heated together in a sealed tube on a water bath. The respective products were diethylmagnesium and -zinc. An analysis of the latter showed that "it did not contain as much as 0.5% of sodium". These two experiments do not prove that magnesium and zinc directly replaced sodium, although that is possible. But diethylmercury might have formed first and the mercury been displaced later by magnesium or zinc. However, the work next described leaves little doubt but that a direct displacement can occur.

Talalaeva and Kocheshkov[10] added a large excess of finely powdered metal to phenyllithium in ether or xylene and refluxed the mixture under nitrogen for 4–200 hr. The yields of phenylmetal compound (Ph_yM) where *y* is 2, 3, or 4) from magnesium, tin, lead, arsenic, and antimony were 13.8, 19.6, 27, 3.6, and 8.9%, respectively. The only product isolated from the same treatment with silicon or bismuth was biphenyl, but that hydrocarbon must come via an intermediate displacement of lithium and the formation of phenyl radical.

The reverse of the above reaction, that is, excess lithium with powdered Ph_yM, failed when M was tin, arsenic, and antimony. When it was lead or bismuth the yields of phenyllithium were 15.7 and 22.4%, respectively.

Incidentally phenylmagnesium bromide was similarly exposed to tin, antimony, lead, and arsenic. Only the last displaced magnesium to give 6% of triphenylarsine, Ph_3As. The conditions were not ideal but the result is suggestive.

Finally there is the displacement of lithium by potassium as reported by Gilman and Young[18] in the reaction of triphenylmethyllithium in ether with sodium–potassium alloy. Later Bryce-Smith and Turner[19] used the method for the preparation of a number of alkylpotassium compounds in hydrocarbon medium. This reaction, too, is a reversal of the EMF series in that lithium is a little above potassium.[20]

Possibly the EMF series, as commonly understood, applies only to the irreversible transfer of an electron from metal to cation as in zinc with copper sulfate. When an electron can shift from anion to cation the displacement should be reversible. That happens when the electron affinity and the thermal stability (to be discussed in the next section) are low.

III. Discussion

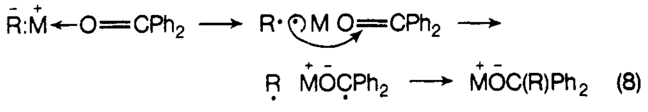
The foregoing facts indicate that the dissociation of organoalkali metal ion pairs is to radical–metal rather than to separate ions. That is, after all, a sensible conclusion because Pauling,[21] in his well-known text, describes two ways by which alkali metal halides dissociate: ionically to separate ions and thermally to the elements, halogen and metal. The latter occurs when the ions are completely separated by heat. Only three salts, cesium and rubidium fluorides and cesium chloride, fail to dissociate thermally to the elements; the reason is that the electron affinity of the halogen exceeds the ionization potential of the metal. The electron affinity of triarylmethyls, as Bent observed, is 60 (or 48) kcal, whereas 79.2 kcal (for iodine) is the lowest for the halogens. Methyl is still lower. Baughan, Evans, and Polanyi[22] put its value at 20. Such low values and the unsuitable environment for ionization, where butyllithium, for example, is associated[23] rather than dissociated, are highly favorable for a thermal type of dissociation.

Moreover, the organoalkali metal compounds are relatively unstable thermally. Butyllithium[24,25] decomposes around 140°C. Amylsodium[26] decomposes slowly at room temperature. Amylpotassium[27] appears to be even less stable. To explain the pyrolysis of butyllithium ionically, lithium hydride was postulated as evolving from the molecule with corresponding formation of butene; but that notion requires that two bonds of unequal strengths, C–Li and C–H, be broken ionically and simultaneously. It is far simpler for the weaker bond, C–Li, to break into two active units, butyl radical and atomic metal. Thereafter disproportionation yields butene and lithium hydride, as in eq 7, in a process typical for radicals, as these two units are.

$$C_4H_9Li \longrightarrow C_4H_9\cdot + Li \longrightarrow C_4H_8 + LiH \qquad (7)$$

Also mercury provides a critical test for the presence of radicals and metals. Its use is analogous to the lead film, which in Paneth's[28] work captured radicals shortly after they were formed. The mercury film is in contact with the radical and metal the instant they are formed. So it is an excellent trap for the slightest amount of the dissociation.[31]

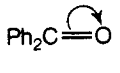
The term thermal dissociation, as used here, includes other forms of energy besides heat. Thus Maercker and Roberts[17] used high-speed stirring at ice-bath temperature even though butyllithium alone decomposes at 140°. Also solvation has an effect. Ether, for example, has long been regarded as coordinating with magnesium in the Grignard reagent and with the cation of organoalkali metal ion pairs. Its dipole moment is 1.15. Common reactants such as aldehydes, esters, nitriles, and halohydrocarbons have higher dipole moments (1.5 to 4.0) and should displace ether at the cation. Then an intermediate, for example, with benzophenone, would be $^{-}R{:}M^{+}\leftarrow O{=}CPh_2$. The old carbanion and the potential new one would contend for the cation. Thus the bond is weakened. Its breaking temperature is lowered. Thermal dissociation occurs. A single electron shifts[29] as in eq 8 from the strained bond, past



$$\overset{-}{R}{:}\overset{+}{M}\leftarrow O{=}CPh_2 \longrightarrow R\cdot \odot M\ O{=}CPh_2 \longrightarrow \underset{\bullet}{R}\ \overset{+}{M}\overset{-}{O}\underset{\bullet}{C}Ph_2 \longrightarrow \overset{+}{M}\overset{-}{O}C(R)Ph_2 \qquad (8)$$

the metal, which transiently is in the metallic state, and finally to the ketone. The radical, R·, is free to move and join with the nearby carbon radical of the reactant. These changes evolve about the cation. Just one electron shift triggers the reaction. Just one positive pole is present. The ion pair is bifunctional.

In this picture the carbonyl group cannot avoid being the nucleophilic center which donates electrons to the cation, rather than the electrophilic agent



$$Ph_2C{=}O$$

by virtue of a self-induced polarization as in the ionic view of these reactions. The cation, an ionic substituent in a hydrocarbon (for there is no real separation of the two ions), must be the true electrophilic reagent. This view, which is the reverse of the ionic concept of these processes, may disturb advocates of the old ionic interpretation, but it is logical, realistic, and in excellent accord with all facts: those about amalgams, low electron affinities, low thermal stabilities, thermal dissociation in general, and media unfavorable to ionization.

This paper is primarily about carbanion compounds, but the principles are not limited to them. Some amide salts probably behave similarly as the interesting paper by Ottolenghi, Bar-Eli, and Linschitz[30] suggests. They point out that "solutions of alkali metals in ammonia or amines decompose to form the corresponding amides" . . . and that "illumination of such faded solution regenerates the metal or solvated electron components of the original metal–solvent combination". So a reversible situation exists. For the potassium or rubidium ethylamide in ethylamine they wrote a charge transfer to solvated electrons according to eq 9. Reasonably we can assume a similar change to a greater or lesser degree in other amine solutions.

$$M^{+}(Et\ddot{N}H) \longrightarrow M + EtNH\cdot \qquad (9)$$

Somewhere between carbanion and halide salts is a changeover in the manner of dissociation. Mercury and probably some other metals, below the alkali metals in the EMF series, provide at least one decisive test, but they must be used properly.

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(31) One of the referees has suggested that a "polarized organometallic compound" reacts with mercury to form the mercurial and amalgam. Thus an anionic interpretation would be possible, but the halide ion in an alkali metal halide is polarized too [see ref 21 and A. E. Van Arkel, "Molecules and Crystals" (J. C. Swallow, Translator), Interscience, New York, N.Y., 1949, p 140]; yet it does not react with mercury. Therefore such a notion requires a special kind or degree of polarization. In other words, a third and unusual type of ion pair must be assumed to exist. Its carbanion combines with mercury in an ionic process even though mercury, as an element, functions as would a radical. There is no need for such imaginings. A long-standing rule, known as Occam's razor, says that "entities are not to be multiplied without necessity". No necessity exists here. Conventional concepts in chemistry as described by Pauling[21] and by Bent[3] provide adequate explanations.