Journal of Organometallic Chemistry, 179 (1979) 123—132

INTERACTIONS OF ALKOXIDES

XI. * REACTIONS OF SUBSTITUTED *N*-LITHIUM AMIDES WITH HEAVIER ALKALI METAL ALKOXIDES. A NOVEL METHOD FOR THE PREPARATION OF *N*-SODIUM AND *N*-POTASSIUM DIALKYLAMIDES

L. LOCHMANN * and J. TREKOVAL

Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, 162 06 Prague 6 (Czechoslovakia)

(Received April 19th, 1979)

Summary

Reactions of substituted *N*-lithium amides (RR'NLi) with heavier alkali metal alkoxides give rise to the corresponding heavier alkali metal amides. In this way, some *N*-sodium and *N*-potassium dialkylamides may be advantageously prepared in the pure state. These amides are much more reactive than lithium amides and readily metalate compounds containing C—H bonds as weakly acidic as those in toluene. Metalations can also be carried out directly using a mixture of lithium amide and a heavier alkali metal alkoxide. The reactivity of *N*-sodium diisopropylamide was increased by the presence of sodium t-butoxide. A medium strength absorption was observed in the IR spectra of substituted alkali metal amides in the range 2800—2450 cm^{-1}; this absorption is due to the stretching vibrations of the C—H bond under the influence of "anionic hyperconjugation".

Introduction

Reactions of alkyllithium compounds with heavier alkali metal alkoxides proceed according to eq. 1 giving rise to organic compounds of a heavier alkali

$$RLi + R'OM \rightarrow RM + R'OLi \quad (M = Na, K, Cs), \tag{1}$$

metal [2,3,11,12]. The reaction is of some importance for preparative reactions. It has also been observed with other organolithium compounds, such as lithioesters [7] or lithioketones [10], for which the enolate form of these compounds

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* The papers mentioned in references 1—10 are regarded as parts I—X of this series.

with the O—Li bond is assumed to predominate [13,14], and the exchange reaction seems to be more generally applicable. In an attempt to establish the scope of the reaction, attention has now been concentrated on compounds in which lithium is bound to another element, e.g. nitrogen in *N*-lithium alkyl- and dialkyl-amides. Our interest in these systems was also stimulated by their use in anionic polymerization.

Results and discussion

It was found that when alkoxides of heavier alkali metals react with substituted *N*-lithium amides a metal—metal exchange takes place:

$$RR'NLi + R''OM \rightarrow RR'NM + R''OLi \qquad (2)$$

(M = Na, K; most favourable when $R'' = (CH_3)_3C$)

In this way, it is possible to prepare some dialkylamides of heavier alkali metals in benzene in a good yield and in satisfactory purity (Table 1). The amides formed are poorly soluble in benzene, unlike lithium t-butoxide, and may therefore be readily isolated. The presence of this alkoxide has been confirmed by IR spectroscopy in the mother liquors from some preparations. The exchange reaction itself proceeds satisfactorily in aromatic hydrocarbons (mainly in benzene), but not in aliphatic hydrocarbons (heptane, cyclohexane), or ethers (with which the amides may react). A 1—1.3 molar ratio of the initial alkoxide to lithium amide was used. The exchange reaction described offers a novel and simple method for the preparation of certain *N*-sodium and *N*-potassium dialkylamides.

Not all the lithium amides follow the typical course of the exchange reaction. Thus, e.g., on mixing of benzene solutions of *N*-lithium t-butylamide and potassium t-butoxide no precipitate appeared. On the other hand, the solution exhibited increased metalation properties, because even after addition of toluene the metalation product immediately separated from solution; its analysis shows that the exchange Li → K must have occurred at some stage of the reaction (Table 2). In the case of *N*-lithium amides which are poorly soluble in benzene, e.g. *N*-lithium diethyl- or di-n-butyl-amide, the exchange reaction with sodium or potassium t-butoxides proceeds to some extent, but the resulting isolated product contains a considerable amount of lithium (Table 1). The analyses and IR spectra of the products indicated that the exchange of metals was not complete in this case and extension of the reaction time to 70 h did not significantly change the result. The reaction between *N*-lithium diisopropylamide and cesium t-butoxide, which is poorly soluble in benzene, was also unsatisfactory.

Toluene contains somewhat more acidic hydrogen atoms than benzene and they approach aliphatic amines in acidity. Thus the exchange reaction in toluene may be complicated by side reactions to a much greater degree than in benzene. It was also observed that these side reactions are strongly dependent on the concentration of alkoxide of the heavier alkali metal in the initial mixture (Table 2). Thus, the product isolated in the reaction of potassium t-butoxide (t-BuOK) with an equimolecular amount of *N*-lithium diisopropylamide in toluene contained predominantly benzylpotassium. At higher concentrations of t-BuOK, the benzylpotassium formed bound one part of potassium alkoxide in the form of an adduct, the composition of which depended on the amount of the

TABLE 1

REACTION OF *N*-LITHIUM DIALKYL AMIDES WITH t-BUTOXIDES OF HEAVIER ALKALI METALS IN BENZENE

Amide	Initial conditions			Isolated precipitate					
	$(CH_3)_3COM$ M	$(CH_3)_3COM$/ amide	Reaction time (h)	Yield [a] (% mol)	Alkalinity [b] (%)	M content [c] (Found (calcd.) (%))	N content [d] (Found (calcd.) (%))	Li content [c] (%)	Amide content [e] (%)
$[(CH_3)_2CH]_2NLi$	Na	1	2	68	18.68	18.80 (18.66)	11.26 (11.37)	0.15	95
$(CH_3)_2CHN(Li)C_6H_{11}$	Na	1	2	78	14.04	14.78 (14.08)	8.21 (8.58)	0.07	
$(C_6H_{11})_2NLi$	Na	1.3	20	91	11.62	11.59 (11.31)	6.73 (6.89)	0.11	
$(C_2H_5)_2NLi$	Na	1.3	28	96	24.15	16.39 (24.17)	14.71 (14.73)	3.58	
$(C_4H_9)_2NLi$	Na	1.3	42	93	15.43	13.21 (15.20)	9.31 (9.26)	1.12	
$[(CH_3)_2CH]_2NLi$	K	1.3	2	92	28.07	28.40 (28.06)	9.50 (10.06)	0.14	88
$(CH_3)_2CHN(Li)C_6H_{11}$	K	1	2	59	21.70	22.26 (21.80)	7.76 (7.81)	0.25	
$(C_6H_{11})_2NLi$	K	1.3	20	74	17.66	17.01 (17.82)	6.21 (6.38)	0.17	
$\underline{CH_2CH_2CH_2CH_2CH_2N}Li$	K	1.3	30	73	31.81	30.90 (31.73)	9.08 (11.37)	0.36	
$(C_2H_5)_2NLi$	K	1.3	26	97	38.40	23.70 (35.15)	11.38 (12.59)	2.38	
$(C_4H_9)_2NLi$	K	1.3	42	88	23.65	20.44 (23.37)	8.17 (8.37)	1.41	
$[(CH_3)_2CH]_2NLi$	Cs	1	18	49	69.5	66.41 (57.02)	1.01 (6.01)	0.29	

[a] Number of moles of all the alkali metals in the precipitate relative to the amount of the initial lithium amide. [b] After hydrolysis and removal of amine by boiling, acidimetrically. [c] Determined by atomic absorption spectrometry. [d] According to Kjeldahl. [e] After the sample was hydrolyzed in cumene, amine was determined by GLC by use of a reference standard. The hydrolyzate also contained a little benzene, which means that this was metalated to a slight degree.

TABLE 2

REACTION OF SUBSTITUTED *N*-LITHIUM AMIDES WITH HEAVIER ALKALI METAL t-BUTOXIDES IN TOLUENE

Amide	Initial compounds		Isolated precipitate						
	$(CH_3)_3COM$ M	$(CH_3)_3COM$/ amide	Yield [a] (% mol)	Alkalinity [b] (%)	M content [c] (Found (calcd.) (%))	N content [d] (Found (calcd.) (%))	Li content [c] (%)	Amide content [e] (% mol)	Mol. ratio $(CH_3)_3COH$/ toluene [e]
$[(CH_3)_2CH]_2NLi$	Na	1	62		18.55 (18.66)	11.12 (11.37)	0.12	88 [f]	
$[(CH_3)_2CH]_2NLi$	Na	2	64		20.59 (21.32) [g]	 (0) [g]	0.13		0.53
$[(CH_3)_2CH]_2NLi$	Na	3	72	23.6	21.37 (22.33) [h]	00.11 (0) [h]	0.41		1.62
$[(CH_3)_2CH]_2NLi$	K	1	94		27.7 (30.03) [i]	0.96 (0) [i]	0.43	86 [i]	
$[(CH_3)_2CH]_2NLi$	K	2	128	33.1	29.95 (31.37) [j]	0.46 (0) [j]	0.26		0.46
$[(CH_3)_2CH]_2NLi$	K	3	158	31.55	30.52 —	0.74 —	0.24		[k]
$(CH_3)_3CNHLi$ [l]	K	1.3	59	30.07	30.9 (30.03) [i]	1.78 (0) [i]	0.41		

[a,b,c,d] Cf. Table 1. [e] Determined after hydrolysis by GLC. [f] Calculated for $[(CH_3)_2CH]_2NNa$. [g] Calculated for $[C_6H_5CH_2Na \cdot 0.53(CH_3)_3CONa]$. [h] Calculated for $[C_6H_5CH_2Na \cdot 1.62(CH_3)_3CONa]$. [i] Calculated for $C_6H_5CH_2K$. [j] Calculated for $[C_6H_5CH_2K \cdot 0.45(CH_3)_3COK]$. [k] All the bands of t-BuOK can also be seen in the IR spectrum of the product. [l] In a benzene-toluene mixture (3 + 1 vol.).

TABLE 3

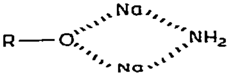
INTERACTION OF *N*-SODIUM DIISOPROPYLAMIDE WITH SODIUM t-BUTOXIDE IN TOLUENE [a]

Molar ratio alkoxide/amide	Insoluble fraction (% by wt.)	N content [b] (%)	
		After reaction	In the initial amide
0	95.6	10.62	11.10
0.1	91.8	9.80	11.10
1	90	6.73	11.10
2	92.7	2.16	11.10

[a] Shaken with three steel beads at 25°C for 4 h. [b] After Kjeldahl.

excess of the alkoxide. Sodium t-butoxide (t-BuONa) yielded a similar adduct of benzylsodium and t-BuONa, when alkoxide was present in excess. If, however, t-BuONa was mixed with *N*-lithium diisopropylamide in an equimolecular ratio, *N*-sodium diisopropylamide was isolated as the resulting product even in toluene. This means, that under the given conditions this sodium amide was unable to metalate toluene, unlike the potassium compound. In the presence of t-BuONa in excess, however, the reactivity of *N*-sodium diisopropylamide increased so much that toluene was metalated.

This promoting effect of t-BuONa on the reactivity of *N*-sodium diisopropylamide was confirmed in a number of experiments, in which the toluene suspension of *N*-sodium diisopropylamide was stirred in the presence of various amounts of t-BuONa. The results are summarized in Table 3; and show that under the conditions used the amide alone gave virtually no reaction with toluene. In the presence of t-BuONa the N content of the product decreased more rapidly the greater the concentration of alkoxide. The IR spectra of isolated products showed that in the presence of t-BuONa the initial amide reacted with toluene. This promotion of the reactivity of *N*-sodium diisopropylamide is obviously analogous to that described by Caubère [15—17] for unsubstituted sodium amide, which he attributed to the formation of an adduct such as



The reaction of *N*-lithium diisopropylamide proceeding in toluene in the presence of various amounts of t-BuONa may thus be described by the scheme:

$$(\text{i-}C_3H_7)_2NLi + m(CH_3)_3CONa \longrightarrow$$

$$\xrightarrow{m=1} [\text{i-}C_3H_7)_2NLi \cdot (CH_3)_3CONa \rightleftharpoons (\text{i-}C_3H_7)_2NNa + (CH_3)_3COLi] \qquad (3)$$

$$\Big\downarrow \text{toluene}$$

$$(\text{i-}C_3H_7)_2NNa + (CH_3)_3COLi$$

$$(\text{i-}C_3H_7)_2NLi + m(CH_3)_3CONa \longrightarrow$$

$$\xrightarrow{m>1} [(\text{i-}C_3H_7)_2NLi \cdot n(CH_3)_3CONa \rightleftharpoons (\text{i-}C_3H_7)_2Na \cdot n'(CH_3)_3CONa + (CH_3)_3COLi]$$

$$\Big\downarrow \text{toluene}$$

$$C_6H_5CH_2Na \cdot n''(CH_3)_3CONa + (\text{i-}C_3H_7)_2NH + (CH_3)_3COLi \qquad (4)$$

The interaction between lithium amides and sodium or potassium alkoxides evidently begins with mutual coordination. The increase in the reactivity of *N*-sodium diisopropylamide in the presence of t-BuONa may be caused by an increase in its basicity and/or solubility, and possibly formation of an adduct between benzylsodium and t-BuONa also helps.

Mono- and di-alkylamides of heavier alkali metals exhibit much higher reactivities than the corresponding lithium amides. Hence, by using the system *N*-lithium amide + alkoxide of a heavier alkali metal it is possible to carry out reactions, in which the analogous lithium amide alone fails. Recently, a similarly effective metalation agent, prepared from amine and a mixture of butyllithium with t-BuOK, was described [18]. In reaction mixtures in which a number of reactions occur simultaneously, it should be possible to change the relative rates of competitive reactions and so vary the ratio of products. In the light of the easy availability of substituted *N*-lithium amides (by direct reaction of lithium with amines in the presence of activator [19,20]), this would seem to be a potentially useful reagent system for preparative chemistry in which the reactivity of the metalation species may be selectively changed by varying the type and quantity of the alkoxide added.

IR spectra of alkali metal organic amides RR'N(M) (M = Li, Na, K)

The IR spectra of some substituted alkali metal amides are given in the Experimental section. A noteworthy feature is the relatively intense absorptions in the range 2800—2450 cm^{-1} observed with all amides having at least one hydrogen atom in the α-position with respect to nitrogen. Table 4 gives some examples, together with data for reference compounds.

It is known that the frequencies of symmetrical stretching vibrations of the C—H bonds in the vicinity of an atom with a free electron pair are situated at lower values [21]. This effect can be made even stronger by a partial negative charge on this atom, arising from a strong polar bond with another atom, e.g. an alkali metal. For alkali metal alkoxides, the interaction of a free electron pair of the oxygen atom bearing a partial negative charge with the C—H bond in the immediate vicinity has been described by Seubold and called "anionic hyperconjugation" [22]. It was reflected in the IR spectra of alkoxides by pronounced shifts in the frequencies of the C—H bonds and was confirmed by the spectra of specifically deuterated alkoxides (Table 4). Simonov [23] interpreted the spectra of alkali metal methoxides similarly.

Alkyl amides of alkali metals are formally analogous to alkali alkoxides, cf. $R—\underline{O}—M$ and $(RR')=\overline{N}—M$. This similarity is also reflected in their IR spectra, as shown in Table 4. One may therefore assume "anionic hyperconjugation" also for these compounds, and attribute the absorptions in the range 2800—2450 cm^{-1} to the stretching vibrations of the C—H bond in the α-position with respect to the heteroatom. In agreement with this, no such absorptions were observed with *N*-lithium or *N*-potassium t-butylamides, which do not possess any α-hydrogens, and which correspond to t-butoxides (Table 4). Since, in the case of amides, these medium intense bands are situated in a range which includes few other bands they are well suited for the investigation of these compounds.

In the long-wave spectral range (700—250 cm^{-1}), substituted *N*-lithium amides

TABLE 4

IR SPECTRA IN THE RANGE 3000–2400 cm^{-1}

Spectra recorded in benzene solution or suspension. Very weak bands are not given. s, strong; m, medium; w, weak; (sh), shoulder.

	M		
	Li	Na	K
$[(CH_3)_2CH]_2NM$	2958(sh), 2938s, 2910(sh), 2854s, 2796m, 2628w, 2587m	2958(sh), 2944s, 2915(sh), 2856s, 2760m, 2632w, 2586m	2954(sh), 2934s, 2910(sh), 2853s, 2718m, 2624w, 2525m
$C_6H_{11}NMCH(CH_3)_2$		^{a}2754m, 2617w, 2584m	^{a}2718m, 2626w, 2602w, 2507m
$[C_6H_{11}]NM$	2916s, 2880s, 2850s, 2831s, 2757m, 2620w, 2572w	2918s, 2880s, 2854s, 2831s, 2756m, 2614w, 2566w	2920s, 2874s, 2852s, 2827s, 2725m, 2600w, 2548w, 2500m, 2472m
$(CH_3)_3NHM$	2942s, 2870s		2942s, 2858s
$(CH_3)_2CHOM$	^{b}2792m, 2688w, 2608m	2950s, 2920(sh), 2860s, 2798m, 2660m, 2556s	
$(CH_3)_2CDOM$		^{c}2941, 2032, 1984, 1942	
CH_3OM	^{d}2924m, 2842s, 2792s, 2595m	^{d}2930m, 2922m, 2733s, 2590m	^{d}2930m, 2900s, 2708s, 2580m
$(CH_3)_3COM$	2954s, ~2925(sh), 2856s	2956s, 2900(sh), 2863s	2938s, 2900(sh), 2877(sh), 2846s

^{a} In Nujol. ^{b} In toluene. ^{c} After [22] (in hexachlorobutadiene). ^{d} After [23] (in Nujol or perfluorinated paraffin).

exhibit at least two intense bands (e.g., *N*-lithium diisopropylamide at 636 and 270 cm^{-1}, dicyclohexylamide at 635 and 270 cm^{-1} and t-butylamide at 636 and 310 cm^{-1}), which with similar amides of heavier alkali metals are found at lower frequencies. The IR spectra of alkali alkoxides exhibit similar shifts [24,25]. Thus, the observed bands of lithium amides are related to the stretching or deformation vibrations of the N—Li bond.

Experimental

Techniques

All the operations involving organic compounds of alkali metals were carried out under pure argon. The amide content in the sample was determined by means of GLC with a Perkin—Elmer 30 chromatograph after hydrolysis in cumene with a small excess of water. The alkali metal content was determined, after hydrolysis of the sample in an excess of water, with a Perkin—Elmer 303 atomic absorption spectrometer. The IR spectra were recorded with Perkin—Elmer 457 and 577 spectrometers using 5—10% solutions or suspensions. Benzene suspensions of the samples were prepared by shaking with steel beads for 24 h; the suspensions did not give any appreciable sediment during the measurement.

Chemicals

Amines were dried with calcium hydride and rectified. The solvents were dried with lithiumaluminium hydride and rectified. *N*-Lithium diisopropylamide was prepared according to [20], the other *N*-lithium amides were obtained by treating the respective amine with a 1 *M* benzene solution of butyllithium (0.95/1 molar). Those amides which were poorly soluble in benzene were isolated and recrystallized, if necessary (Table 5); the soluble amides were used as a reaction mixture. Sodium and potassium t-butoxide were prepared according to [26].

TABLE 5

PREPARATION OF *N*-LITHIUM ALKYL AND DIALYK AMIDES

Amide	Yield (%)	Alkalinity [a] (Found (calcd.) (% Li))	N content [b] (Found (calcd.) (%))
$(C_2H_5)_2NLi$	67 [c]	8.78 (8.78)	18.70 (17.71)
$(C_4H_9)_2NLi$	88	5.14 (5.13)	10.13 (10.36)
$C_6H_{13}NHLi$	77	6.48 (6.48)	12.23 (13.08)
$(C_6H_{11})_2NLi$	73	3.72 (3.71)	7.20 ((7.48)
$\underline{CH_2CH_2CH_2CH_2CH_2NLi}$	45 [c]	7.62 (7.62)	13.92 (15.38)

[a] After hydrolysis and removal of amine by boiling, acidimetrically. [b] After Kjeldahl. [c] Crystallized from diethyl ether in the presence of an amount of tetrahydrofuran equimolar to the amide at —78°C, crystals dried at 50°C in vacuo.

Reaction of substituted N-lithium amides with t-butoxides of heavier alkali metals (Tables 1 and 2)

A benzene solution of a *N*-lithium amide was mixed with a 0.2—0.4 molar solution of sodium or potassium t-butoxide in benzene (or toluene) (prepared while hot and cooled immediately before use), and the mixture was stirred at 20—25°C for the time given (in toluene, 3 h). The precipitate of the new amide separated within few minutes of mixing. With initial compounds which were poorly soluble in benzene, the suspension was shaken with a solution of the other component and three steel beads for the times shown. After the reaction had been completed, the insoluble fraction was isolated by filtration or centrifugation, washed, and dried in vacuo at room temperature for 8 h.

Interaction of N-sodium diisopropylamide with toluene in the presence of t-BuONa

The amide was shaken in toluene suspension with three steel beads in the presence of various amounts of t-BuONa at 25°C for 4 h. The insoluble fraction was isolated and analyzed; the results are listed in Table 3. The IR spectra of products obtained by the interaction with toluene without t-BuONa were quite different from those of products prepared by treating amide with toluene in the presence of t-BuONa.

IR spectra of N-lithium, sodium and potassium diisopropylamides in the range 4000—250 cm^{-1} (in benzene; s, strong; m, medium; w, weak, (sh), shoulder; very weak bands are omitted).

(a) *N*-Lithium diisopropylamide: 2958(sh), 2938s, 2910(sh), 2854s, 2796m, 2628w, 2587m, 1542m, 1456m, 1373s, 1356s, 1330m, 1302s, 1154s, 1125(sh), 1114m, 1093w, 1000s, 932w, 901m, 827w, 809m, 632(s), 270(s) cm^{-1}

(b) *N*-Sodium diisopropylamide: 2958(sh), 2944s, 2915(sh), 2856s, 2760m, 2632w, 2586m, 1520m, 1457m, 1449m, 1434m, 1415w, 1375m, 1356m, 1348m, 1304s, 1152s, 1144m, 1121m, 1087w, 1044w, 985s, 929w, 895m, 825, 796m, 550m, 498m, 498w, 422w, 238m.

(c) *N*-Potassium diisopropylamide: 2954(sh), 2934s, 2910(sh), 2853s, 2718m, 2624w, 2525s, 1548w, 1433w, 1408w, 1367m, 1344m, 1296s, 1163s, 1142m, 994m, 929w, 895m, 823w, 791m, 547m, 404w.

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