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Silylations of α,β-unsaturated and aromatic carbonyl compounds with cobalt carbonyls

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Dedicated to: Professor László Markó on the occasion of his 70th birthday in recognition of his important contribution in homogeneous catalysis.

Abstract

[η^3-η^5-(Silyloxy–alkenyl)]-cobalt carbonyls and various silyl ethers were formed in the cobalt carbonyl mediated silylations of α,β-unsaturated and aromatic carbonyl compounds. A silyloxonium tetracarbonylcobaltate tight ion pair was suggested as a common intermediate, which provides the products through a radical pair in the cases of aromatic aldehydes and ketones. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Cobalt; Silylation; Aromatic carbonyl compounds; Silicon; Ketones; Aldehydes

1. Introduction

α-Hydroxyalkylcobalt tetracarbonyls were proposed several times in the last three decades as intermediates in the hydrogenation of carbon monoxide and carbonyl compounds in the presence of cobalt carbonyls [1–3]. We reported the formation of such complexes in reactions of aldehydes and $HCo(CO)_4$ [4,5]. These species were found to be thermally very unstable, however, they could be stabilized by silylating with bis(trimethylsilyl)-trifluoroacetamide, (BSTFA) at low temperatures leading to [α-(trialkylsilyloxy–alkyl)]-cobalt tetracarbonyls [4,5]. The complex formed from $HCo(CO)_4$, crotonaldehyde and BSTFA was found to lose CO easily giving [η^3-(1-silyloxy-2-butenyl)]-cobalt tricarbonyl (**1**). We obtained the same species by allowing $Me_3SiCo(CO)_4$ to react with crotonaldehyde under argon exactly as described by Murai and coworkers [6].

Now we report the silylation of several α,β-unsaturated and aromatic carbonyl compounds and the preparation of some new η^3-, and η^5-coordinated cobalt carbonyl derivatives. The reactions also resulted in some very interesting organic products.

2. Results and discussion

The silylations were carried out using three different methods. In method A, $HCo(CO)_4$ was treated with an excess of carbonyl compound at low temperature and then a stoichiometric amount of BSTFA was added. In method B, $R_3SiCo(CO)_4$ was prepared in situ from $Co_2(CO)_8$ and the hydrosilane and a carbonyl compound was added at 0–25°C. Catalytic quantities of pyridine accelerated the reactions [7]. Method C was used to synthesize Co complexes of tetraphenylcyclopentadienone. The result of the experiments are summarized in Table 1, which along with Scheme 1 also gives the structures of the numbered complexes.

If an organometallic complex were present, it was an [η^3-(1-silyloxy–alkenyl)]-cobalt tricarbonyl type product, corresponding to the given α,β-unsaturated carbonyl compound. With sterically very hindered ketones (e.g. (+)-pulegone) no silylation took place.

Methyl vinyl ketone was allowed to react with $HCo(CO)_4$ at −40°C (method A). A sample of the

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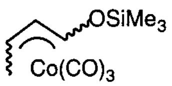
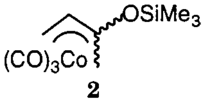
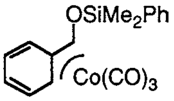
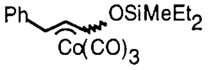
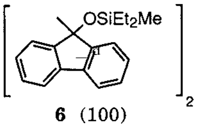
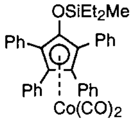
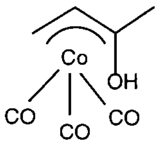
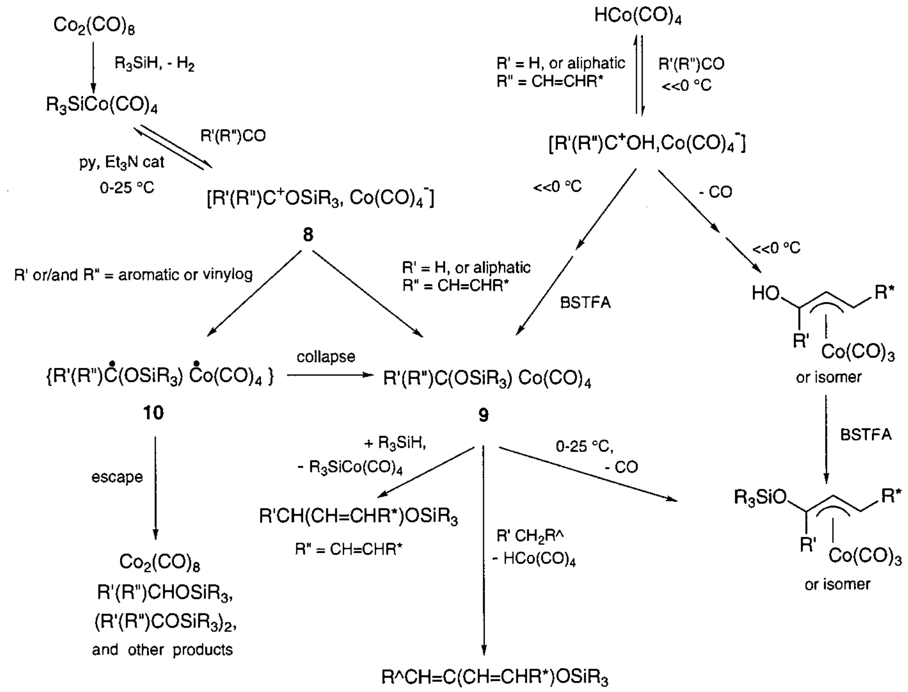
Table 1
Silylation of α,β-unsaturated carbonyl compounds

Carbonyl compound	Method [a]	Complex formed	Organic product formed (yield, wt.%) [b]
Crotonaldehyde [c]	A,B	 **1**	n.d.
Methyl vinyl ketone	A	 **2**	$CH_2{=}CHC(OSiMe_3){=}CH_2$ (9), E,Z-$CH_3CH{=}C(OSiMe_3)CH_3$ (37), $CH_3CH_2C(OSiMe_3){=}CH_2$ (34)
2-Cyclohexen-1-one	B	– [d]	$CH_2CH_2CH_2CH_2CH_2C{=}O$ (11) $CH_2CH_2CH_2CH_2CH{=}COSiEt_2Me$ (31), $CH{=}CHCH_2CH_2CH{=}COSiEt_2Me$ (3), condensation product (23)
Benzaldehyde	B [e]	 **3**	$PhCH_2OSiMe_2Ph$ (85), $(PhCHOSiMe_2Ph)_2$ (4), $PhC(OSiMe_2Ph){=}CHOSiMe_2Ph$ (4)
Cinnamaldehyde	B [e]	 **4**	E,Z-$PhCH_2CH{=}CHOSiMeEt_2$ (52), E-$PhCH{=}CHCH_2OSiMeEt_2$ (22), $(PhCH{=}CH_2CHOSiMeEt_2)_2$ (15)
Fluorenone	B	 **6** (100)	
Dibenzosuberenone	B	– [d]	$C_{15}H_{11}OSiMeEt_2$ **7** (100)
Tetraphenyl-cyclopentadienone	C [b]	 **5**	n.d.

[a] Methods used in complex preparation: see text.
[b] Based on GLC-analysis, see Section 3.
[c] See Ref. [5].
[d] Carbonyl compound containing Co complex has not been found as a characterizable product.
[e] The complex could not be isolated in pure form, see text.

reaction mixture was monitored in the temperature region of −30 to +25°C by IR spectroscopy. At −30°C the ν(C≡O) bands of $HCo(CO)_4$ disappeared completely. Beside bands belonging presumably to alkyl and acyl complexes and $Co_2(CO)_8$, strong bands at 2052 and 1968 cm^{-1} were detected. The intensity of these two bands decreased with rising temperature while those of $Co_2(CO)_8$ increased, but they were detectable even at room temperature (r.t.). For the unstable complex we suggest the structure:



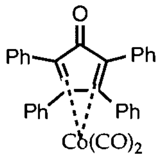


Scheme 1.

and/or isomer, but an oxapropenyl structure proposed by Orchin et al. [8] cannot be totally ruled out. Adding BSTFA to the mixture at −30°C led to [2,3,4-η-{2-methyl-2-(trimethyl–silyloxy)-butenyl}]-cobalt tricarbonyl (**2**) as the main product. No organic materials corresponding to a radical reaction pathway were found (see Ref. [9], vide infra).

Benzaldehyde gave a η^3-benzylic complex (**3**) which, however, could not be isolated, but its structure is inferred by comparing its IR spectrum with those of related compounds[1] (see Ref. [10]). The IR and ^{1}H-NMR spectra of the organometallic compound formed in situ in the silylation of cinnamaldehyde were in accord with an η^3-allyl structure (**4**).

In the case of tetraphenylcyclopentadienone, a cyclic dienone, [η^5-(1-silyloxy-2,3,4,5-tetraphenylcyclopentadienyl)]-cobalt dicarbonyl (**5**) was prepared and characterized. Another, less soluble species could be isolated as well. The latter IR spectrum showed not only two terminal, but also an organic ν(C≡O) band. It proved to be paramagnetic by NMR and magnetic measurements, however, no signal in its ESR spectrum could be found in solution. On the basis of IR, MS and elementary analytical data we propose the structure:



however, further investigations are needed for the full characterization of the complex.

Interestingly, silylation of fluorenone and dibenzosuberenone did not result in any π-complex; only $R_3SiCo(CO)_4$, and $Co_2(CO)_8$ were found.

Most of the silylations were performed in the presence of an excess (5- to 20-fold) of carbonyl compound, i.e. the reactions became quasicatalytic, and organic silylation products were found. It can be seen from Table 1 that the organic products from the aliphatic (or cycloaliphatic) and those from the aromatic (or vinylog) carbonyl compounds were different. Starting from the members of the first group the corresponding silyl enol ethers and silyl dienol ethers were formed. (see Table 1). 2-Cyclohexen-1-one also gave some condensation products.

On the other hand, the silylation of aromatic carbonyl compounds led to, among others, disilyl pinacol ethers. The highest selectivity for this compound was generated from fluorenone, where {[9,9′-bi-9*H*-fluorene]-9,9′-diyl-bis(oxy)}-bis(diethylmethyl-silane) (**6**) (see Ref. [11]), was the only characterizable product. In contrast, dibenzosuberenone provided only monosilyl ether **7** as product. Both types of compounds were found in the case of cinnamaldehyde, but from benzaldehyde several byproducts were formed. One of them, 1,2-bis(diethyl-methyl–silyloxy)-2-phenyl-ethene might come from

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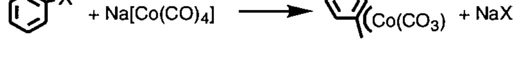
[1] The IR spectrum could be assigned by analogy to known η^3-benzylcobalt complexes, which were prepared according to the equation:



carbonylation side reactions because its analog was found in the hydrosilylation of benzaldehyde under CO by Murai and coworkers [12].

In our earlier study on the cobalt carbonyl mediated silylation of aldehydes [5] and oxiranes [13] we suggested that hydrido- or silylcobalt tetracarbonyl form an ionic intermediate with the substrate. Analogous suggestions were made by Gladysz and coworkers for the reaction of $Me_3SiMn(CO)_5$ with aliphatic and aromatic aldehydes and ketones [14]. These authors found a similarly different nature of the organic silylation products of aliphatic and aromatic compounds. From benzaldehyde, e.g. unstable $Me_3SiOCH(C_6H_5)Mn(CO)_5$ was formed which decomposed to the corresponding bis-(trimethylsilyl) pinacol ether. Aliphatic aldehydes or ketones gave, however, only $HMn(CO)_5$ and silyl enol ethers. It has been shown that both types of organic products could be produced catalytically by cobalt carbonyls even if the selectivity of silyl pinacol ethers varied strongly with the structure of aromatic carbonyl compounds (see Refs. [5,12] and this work).

Based on our former and current results as well as literature analogies, we propose a mechanism of the cobalt carbonyl mediated silylation of α,β-unsaturated carbonyl compounds as shown in Scheme 1. The crucial point of the mechanism is the transformation of the, presumably tight, silyloxonium tetracarbonylcobaltate ion pair **8** either to an [η^1-(1-silyloxy–alkenyl)]-cobalt tetracarbonyl intermediate (**9**), or to a radical pair **10** (see Ref. [12]). At low CO concentrations, **9** may lose CO and an η^3- (or η^5-) silyloxy–alkenyl complex will be formed. The collapse of **10** results again in the formation of **9**, the escape, however, provides solvent separated radicals. $Co(CO)_4$ will recombine to give $Co_2(CO)_8$ making a catalytic cycle possible. The substituted silyloxymethyl radical can react with the hydrosilane to a saturated silyl ether, or recombine to disilyl pinacol ether. $HCo(CO)_4$ formed by β-elimination decomposes under the reaction conditions (see Ref. [15]).

Aliphatic and cycloaliphatic α,β-unsaturated carbonyl compounds can be transformed to [η^3-(1-silyloxy–alkenyl)]-cobalt tricarbonyl derivatives (**1**, **2**) through a stoichiometric reaction with $HCo(CO)_4$ and BSTFA (see Ref. [5]). as was demonstrated in the case of methyl vinyl ketone (vide supra). The probability of the two ways leading to the product (Scheme 1) may depend on CO concentration.

We expected that in the case of aromatic aldehydes and ketones a CIDNP effect could be detected following their silylation by ^{1}H-NMR spectroscopy (see Ref. [14]). In the course of in situ silylation of benzaldehyde and cinnamaldehyde in an NMR tube however, no CIDNP effect has been observed.

3. Experimental

3.1. General

All manipulations involving air-sensitive compounds were carried out by the usual Schlenk technique using deoxygenated, dry solvents and gases, and reaction vessels with magnetic stirring. IR spectra were recorded by using a 0.06 mm CaF_2 cuvette on a Specord IR 75 (Carl Zeiss, Jena) spectrometer, which was calibrated with benzene (1959.6 cm^{-1}) and polystyrene (1601.4 cm^{-1}). ^{1}H- and ^{13}C-NMR spectra were obtained on a Varian Unity 300 spectrometer, using hexamethyldisiloxane internal reference. Gas chromatograms were recorded on a Hewlett–Packard model 5830 A chromatograph, GLC-MS analyses were performed on a JMS 01-SG-2 Jeol spectrometer and a Hewlett–Packard 5890 Series II GC-MSD equipment.

Solutions of $HCo(CO)_4$ in pentane or octane were prepared from $Co_2(CO)_8$, DMF, and concentrated HCl [16], and contained 1–3 mol% $Co_2(CO)_8$, according to IR and Co analyses. Me_3SiH [17] and $R_3SiCo(CO)_4$ complexes ($R_3 = Me_3$, Et_2Me, Et_3, Me_2Ph) [18] were prepared by literature methods. Other starting materials were commercial products and were purified by crystallization or distillation.

Most of the organic products (see Table 1) are known silyl ethers or their analogs (e.g. diethyl-methyl–silyl derivatives instead of trimethylsilyl ones). They were identified on the basis of known MS spectra. In the absence of such data authentic samples were prepared by described methods [11,12,14,19,20,22] and the GLC-MS data were compared wit those of the original samples.

3.2. Silylation procedures

3.2.1. Method A

To a cold (-40°C) solution of 190 μl (2.28 mmol) of methyl vinyl ketone in 5 ml of dichloromethane 1.5 ml of a 0.7 M stock solution of $HCo(CO)_4$ (1.26 mmol in octane) was added under carbon monoxide. The reaction was followed by IR spectroscopy (see text). The solution was allowed to warm up to -30°C and 175 μl of BSTFA (0.66 mmol) was added. After freezing out $Co_2(CO)_8$, [2,3,4-η-{2-methyl-2-(trimethylsilyloxy)-butenyl}]-cobalt tricarbonyl (**2**) as the main product was detected by IR spectroscopy (ν(C≡O): 2041 (vs), 1969 (vs), see Ref. [6]).

MS-data (m/z) of the identified organic products (see Refs. [19,20]).

2-Trimethylsilyloxy-1,3-butadiene: 142 (M^+), 127, 85, 75, 73, 45.

2-Trimethylsilyloxy-2-butene, (*E*,*Z*): 144 (M^+), 129, 75, 73, 45.

2-Trimethylsilyloxy-1-butene: 144 (M^+), 129, 75, 73, 45.

3.2.2. Method B

Up to 1.0 mmol of $Co_2(CO)_8$ was dissolved in 5–10 ml of dichloromethane or toluene (in the case of benzaldehyde—pentane) under carbon monoxide. Carbonyl compound (10–30 mmol) and 7–20 mmol of hydrosilane and—occasionally—up to 1 mmol of base (pyridine or triethyl amine) was added at 0°C. The reaction mixture was stirred for 0.5–2 h at 0–25°C (before the end of stirring at least 10 min at r.t.). Probe(s) for IR analysis were taken from the reaction mixture. At the end of the stirring $Co_2(CO)_8$ and $R_3SiCo(CO)_4$ were removed adding excess of pyridine and the organic products were analyzed by GLC-MS. Partial results are shown in Table 1, others are listed here:

3.2.2.1. 2-Cyclohexene-1-one. IR: $[Py^+SiEt_2Me.Co(CO)_4^-]$ tight ion pair [21] could be detected. MS data (m/z) of the organic products (see Refs. [9,20]). (Diethylmethyl–silyloxy)-cyclohexene, (E,Z): 198 (M^+), 169, 156, 142, 89, 61. (Diethylmethyl–silyloxy)-1,3-cyclohexadiene: 196 (M^+), 165, 137, 73, 61.

Another species with $M^+ = 294$ was found, which may be an aldol-condensations product.

3.2.2.2. Benzaldehyde. IR: an η^3-type complex was found as the only organometallic species, but it could not be isolated in pure form: $\nu(C{\equiv}O)$ (pentane): 2048 (vs), 1988 (vs), 1967 (vs). We assign the spectrum tentatively to [η^3-{phenyl-(dimethylphenyl–silyloxy)-methyl}]-cobalt tricarbonyl (**3**).

MS data (m/z) of the organic products (see Refs. [12,14]). (Dimethylphenyl–silyl) benzyl ether: 242 (M^+), 227, 164, 149, 91. 1,2-Bis(dimethylphenyl–silyloxy)-1,2-diphenyl-ethane, (erythro–threo): 271, 241, 167, 135, 91. 1,2-Bis(dimethylphenyl–silyloxy)-1-phenyl-ethene: 404 (M^+), 330, 209, 179, 135.

3.2.2.3. Cinnamaldehyde. IR: the expected η^3-allyl-type complex was found in the reaction mixture but it could not be isolated: $\nu(C{\equiv}O)$ (toluene): 2047 (vs), 1984 (vs), 1973 (vs). We assigned the spectrum tentatively to [1,2,3-η-{1-phenyl-3-(diethylmethyl–silyloxy)propenyl}]-cobalt tricarbonyl (**4**) (more isomers are possible).

^{1}H-NMR: the following procedure was used to test for CIDNP effect as well as to characterize the in situ formed products.

A total of 17 mg (0.05 mmol) of $Co_2(CO)_8$ and 38 µl (0.25 mmol) of Et_3SiH were reacted at 0°C under Ar in a Schlenk-tube. As H_2 evolution ceased, the reaction mixture was transferred in an NMR tube with a syringe, the tube was chilled at -80°C. Then, 0.45 ml of CD_2Cl_2 and 16 µl (0.13 mmol) of freshly distilled *trans*-cinnamaldehyde were added. ^{1}H-NMR spectra were recorded at 25°C. No really well-resolved spectra were obtained, presumably due to transient radical species (see text). No CIDNP effect could be observed, the following new signals were found (δ, ppm (rel. int.)): 1.82 (<1), 2.66 (≈ 1), 3.25 (≈ 1), 3.45 (≈ 2), 4.35 (≈ 1), 4.66 (≈ 1), 5.20 (<1), 5.57 (≈ 1), 6.35 (≈ 2), 6.62 (<1), 7.3 (broad, >10).

Six of these signals corresponds to the reported spectrum of 1-(trimethyl–silyloxy)-3-phenyl-1-propene (E:$Z \approx 2$:1) [22]. The signals at 1.82, 2.66 and 5.57 ppm may be assigned to **4** (see Refs. [6,10]) and those at 4.35, 6.35 (partly) and 6.62 to the third silyl ether isomer, respectively. The broad phenyl signal at 7.3 ppm belongs to all four components. GLC analysis of the reaction mixture showed an actual isomer ratio of [E-$PhCH_2CH=CHOSiEt_3$]:[Z-$PhCH_2CH=CHOSiEt_3$]:[$PhCH{=}CH{-}CH_2OSiEt_3$] $\approx$ 8:4:7. Disilyl pinacol ether was found only in traces under the above conditions.

MS data (m/z) of the organic products of the catalytic process (see Ref. [22]): 1-(diethylmethyl–silyloxy)-3-phenyl-2-propene, (E,Z): 234 (M^+), 205, 149, 121, 117, 91, 73. 1-(Diethylmethyl–silyloxy)-3-phenyl-1-propene, (E): 234 (M^+), 205, 149, 117, 115, 91, 61. 1,2-Bis(diethylmethyl-silyloxy)-1,2-bis(1-phenyl-ethenyl)-ethane (tentative assignment): 233, 175, 101, 73, 45.

3.2.2.4. Fluorenone. IR: only $Et_2MeSiCo(CO)_4$ and $Co_2(CO)_8$ could be detected as cobalt containing products.

MS of {[9,9′-bi-9H-fluorene]-9,9′-diylbis(oxy)}-bis(diethylmethyl–silane) (**6**) (see Ref. [12]), (m/z): 281, 251, 223, 165, 101, 73, 45.

3.2.2.5. Dibenzosuberenone. IR: see Section 3.2.2.4.

MS of 5-(diethylmethyl–silyloxy)-5H-dibenzo[a,d]-cycloheptene (**7**) (m/z): 308 (M^+), 274, 206, 190, 188, 161, 105, 89, 73.

3.2.3. Method C

From the reaction mixture of 0.58 g (1.54 mmol) of $Co_2(CO)_8$ and 0.5 ml (3.45 mmol) of Et_2MeSiH the excess of silane was removed in vacuo. To the resulting $Et_2MeSiCo(CO)_4$, 20 ml of toluene, 1.3 g (2.60 mmol) of tetraphenyl–cyclopentadienone and 80 µl (1.0 mmol) of pyridine were added. The reaction mixture was warmed to 50°C. The gas evolution ceased within 1.5 h. The brown–violet mixture was then cooled successively to -78°C. A brown–violet species could be frozen out which needs further characterization. The wine-red mother liquor was evaporated to dryness in vacuo. The residue was recrystallized twice from hexane which resulted in bright-red microcrystals which proved to be **5**.

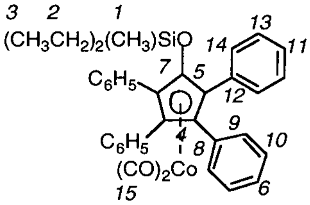
*3.2.3.1. [η^5-{(1-Diethylmethyl–silyloxy)-(2,3,4,5-tetraphenyl)-cyclopentadienyl}]-cobalt dicarbonyl (**5**).* IR: $\nu(C{\equiv}O)$ (hexane): 2013 (vs), 1953 (vs).

MS (m/z): 600 (M^+), 572 (M^+-CO), 544 (M^+-2CO), 515 ($[C_5(C_6H_5)_4Co]O^+Si(C_2H_5)_2CH_3$), 488

($[C_5(C_6H_5)_4Co]OSi(CH_3)$), or ($C_5(C_6H_5)_4HOSi(C_2H_5)_2CH_3$), 101 ($Si^+(C_2H_5)_2CH_3$), 73 (base peak, $HSi^+(C_2H_5)CH_3$), 59 (Co).

^{1}H-NMR (δ, ppm, $CDCl_3$): 0.07 (s, $SiC\underline{H}_3$, 3H), 0.23 (q, $C\underline{H}_2$, J 4.6 Hz, 4H), 0.68 (t, $C\underline{H}_3$, J 4.6 Hz, 6H), 6.95–7.46 (m, $C_6\underline{H}_5$, 20H).

^{13}C-NMR (δ, ppm, $CDCl_3$): −4.26, 1.01, 6.29, 96.75, 98.53, 127.14 (2C: *6*, *7*), 127.38 (3C: *8*, *9*), 127.70, 131.60, 131.98, 132.21, 132.48, 205.55.



Compound **5** gave correct elemental analysis.

Acknowledgements

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