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PREPARATION OF $\text{Os}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$ ($\text{X} = \text{Me}, \text{Cl}$) AND PYROLYSIS TO GIVE THE DIMERIC DERIVATIVES, $[\text{Os}(\text{CO})_4(\text{SiXCl}_2)]_2$

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Summary

Ultraviolet irradiation of $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiXCl}_2)_3$ ($\text{X} = \text{Me}, \text{Cl}$) in hexane solution under carbon monoxide (2 atm) gives *cis*- $\text{Os}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$ as the major product. These compounds along with the corresponding $[\text{Os}(\text{CO})_4(\text{SiXCl}_2)]_2$ derivatives may also be isolated by the action of carbon monoxide, at elevated temperatures and pressures, on the appropriate $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiXCl}_2)_3$ species. The hydrides *cis*- $\text{Os}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$ react (at 75°C) with PPh_3 to give *mer*- $\text{Os}(\text{CO})_3(\text{PPh}_3)(\text{SiXCl}_2)(\text{H})$. Pyrolysis of $\text{Os}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$, in solution under vacuum, gives mainly $[\text{Os}(\text{CO})_4(\text{SiXCl}_2)]_2$. However, if the pyrolysis is carried out under carbon monoxide (90 atm) the dimerization is suppressed and $\text{Os}(\text{CO})_5$ may be detected in the reaction solution. The similarities between these results and the known thermal disproportionation of $\text{Os}(\text{CO})_4(\text{CH}_3)(\text{H})$ is discussed.

Introduction

Recently, the preparation of the cluster compounds $\text{M}_3\text{H}_3(\text{CO})_9(\text{SiXCl}_2)_3$ ($\text{M} = \text{Ru}, \text{Os}$; $\text{X} = \text{Me}, \text{Cl}$) has been reported from these laboratories [1]. One possible mechanism for the formation of these molecules, from $\text{M}_3(\text{CO})_{12}$ and Cl_2XSiH , appeared to be the initial formation of $\text{M}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$ and then trimerization to give the cluster compound in a manner analogous to that observed for $\text{M}(\text{CO})_5$ [2].

In this paper we report the preparation of $\text{Os}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$ and the pyrolysis of the compounds to give, not the trinuclear species, but dinuclear products. The results reported here are complementary to those reported by Norton and coworkers on the thermal disproportionation of $\text{Os}(\text{CO})_4(\text{CH}_3)(\text{H})$ [3].

Experimental

Unless otherwise stated, reactions, including the purification of solvents, were carried out under a nitrogen atmosphere with use of standard Schlenk techniques. Hydrocarbon solvents were refluxed over potassium, distilled and stored over type 4A molecular sieves before use. The cluster compounds $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiXCl}_2)_3$ ($\text{X} = \text{Me}, \text{Cl}$) were prepared as reported previously [1]. Carbon monoxide was an ultrapure grade from Matheson. High pressure reactions were carried out in a 200 ml general purpose bomb from Parr Instrument Company. It was fitted with a safety disc rated at 3000 psig (the working pressure of some of the reactions described here was ca. 2100 psig). The ultraviolet irradiations were carried out with use of a Hanovia 200-W lamp inside a water-cooled, quartz jacket; the reactants were contained in a cylindrical quartz tube (30 cm \times 3 cm diameter) fitted with a Teflon valve. The reactions carried out under vacuum were done so in round-bottom flasks (80 ml total volume) fitted via a 10 cm stem with a Teflon valve. Infrared spectra were obtained with a Perkin-Elmer 237 instrument fitted with an external recorder. The spectra were calibrated with carbon monoxide. Proton NMR spectra were recorded on a Varian XL-100 spectrometer, Me_4Si was used as an internal standard. Mass spectra were obtained on a Hewlett-Packard 5985 GC-MS system. Microanalyses were carried out by Mr. M.K. Yang of this department. Except for the $\text{Os}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$ compounds, melting points were determined under nitrogen in sealed capillaries with a Gallenkamp apparatus. (Because of the very small quantities of the $[\text{Os}(\text{CO})_4(\text{SiXCl}_2)]_2$ derivatives actually isolated pure, it was not possible to perform duplicate melting points on these compounds.) The melting points of the $\text{Os}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$ products were measured by placing the Schlenk flask containing the compound in question in an ice-water bath and then allowing the bath to warm slowly. The temperature of the water bath at which the compound melted was then recorded.

Analytical and spectroscopic data for the new compounds are given in Table 1.

Preparation of cis-Os(CO)₄(SiCl₃)(H)

A quartz tube containing $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiCl}_3)_3$ (0.125 g, 0.10 mmol) in hexane (15 ml) was cooled to -196°C and evacuated. The solution was degassed with one freeze-thaw cycle. The vessel was then pressurized with carbon monoxide (2 atm) and the stirred solution subjected to UV irradiation for 3.5 h. (There was approximately 5 cm between the UV source and the edge of the solution.) During the irradiation some decomposition in the form of a white precipitate occurred. After the irradiation the CO was released and the solution transferred to a Schlenk flask. An infrared spectrum of the solution at this stage indicated that the major product was $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ with no evidence for $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$. The hexane was removed at 0°C on the vacuum line and a second Schlenk flask fitted to the first via interconnecting joints and both vessels evacuated to $<5 \times 10^{-3}$ mmHg (the solid was kept at -196°C during this operation). The second Schlenk flask was then cooled to -196°C and the first allowed to room temperature whereupon, over the next 5 h, *cis*- $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ (0.043 g, 0.098 mmol, 33%) sublimed to the second flask. Several times during

TABLE I
ANALYTICAL AND SPECTROSCOPIC DATA

Compound	M.p. (°C)	Mol. wt.		Analysis (%)				$\nu(\text{CO})$ (cm ⁻¹) ^b	¹ H NMR (ppm)		
		calcd.		found		C				H	
		calcd.	found	calcd.	found	calcd.	found			calcd.	found
Os(CO) ₄ (SiCl ₃)(H)	32	437.7	304 ^c	10.98	—	0.23	—	2150w 2087m 2069s 2142w 2079m 2059s 2106w 2050m 2028s 2108w 2049m 2027s	-7.94 ^d		
Os(CO) ₄ (SiMeCl ₂)(H)	7	417.2	304 ^c	14.39	—	0.97	—	2098w 2041m 2015s 2093w 2053vs 2080w 2039s	1.23 -8.34 ^d		
Os(CO) ₃ (PPh ₃)(SiCl ₃)(H)	184	672.0	^e	37.54	38.04	2.40	2.47		7.51 ^f -7.54 ^{d, h}		
Os(CO) ₃ (PPh ₃)(SiMeCl ₂)(H)	148-149	651.6	^e	40.56	40.73	2.94	3.01		7.49 ^f 1.24 -7.84 ^{d, i}		
[Os(CO) ₄ (SiCl ₃)] ₂	148-151	873.4	874	11.00	11.28	0.0	0.0		—		
[Os(CO) ₄ (SiMeCl ₂)] ₂	142-144	832.5	884	14.43	14.50	0.73	0.69		1.45 ^j		

^a By mass spectroscopy. ^b In hexane solution. ^c [¹⁹²Os(CO)₄]⁺. ^d CD₂Cl₂ solution. ^e Insufficiently volatile for a mass spectrum. ^f CH₂Cl₂ solution. ^g Multiplet. ^h $J(\text{P-H}) = 15.8$ Hz. ⁱ $J(\text{P-H}) = 16.0$ Hz. ^j CDCl₃ solution.

this procedure the apparatus was quickly reevacuated so as to maintain the vacuum within the flasks.

The dichloromethylsilicon analogue, $\text{Os}(\text{CO})_4(\text{SiMeCl}_2)(\text{H})$ was prepared in a similar manner. Both hydrides decompose to a pale yellow oil after only a few minutes exposure to air.

Preparation of $\text{Os}(\text{CO})_3(\text{PPh}_3)(\text{SiMeCl}_2)(\text{H})$

A solution of $\text{Os}(\text{CO})_4(\text{SiMeCl}_2)(\text{H})$ (0.030 g, 0.072 mmol) and PPh_3 (0.020 g, 0.076 mmol) in hexane (10 ml) was heated at 75°C under vacuum for 32 h. After the 32 h period the solution was allowed to cool to approximately 50°C , filtered (from a small quantity of insoluble matter), and then placed in the refrigerator at -15°C overnight, whereupon $\text{Os}(\text{CO})_3(\text{PPh}_3)(\text{SiMeCl}_2)(\text{H})$ (0.022 g, 47%) was obtained as white needles. Recrystallization from benzene-hexane gave the analytical sample.

An infrared spectrum of reaction solution after 17 h revealed significant amounts of unreacted starting material. After 32 h only bands due to $\text{Os}(\text{CO})_3(\text{PPh}_3)(\text{SiMeCl}_2)(\text{H})$ were present; i.e., there was no evidence for any other metal carbonyl product.

The trichlorosilicon analogue, $\text{Os}(\text{CO})_3(\text{PPh}_3)(\text{SiCl}_3)(\text{H})$, was prepared in the same manner. It is only sparingly soluble in hexane. Both triphenylphosphine derivatives appeared air-stable.

Action of carbon monoxide on $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiCl}_3)_3$

A glass liner containing $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiCl}_3)_3$ (0.20 g; 0.16 mmol) in hexane (15 ml) was placed in a 200 ml general purpose bomb and sealed. The bomb was flushed with carbon monoxide and pressurized with the gas (90 atm). The bomb was then heated to 170°C for 24 h after which it was cooled and the gases vented. The hexane solution was transferred to a Schlenk flask. Most of the remaining solid (0.056 g) was insoluble in CH_2Cl_2 . (The CH_2Cl_2 -soluble fraction had bands in the infrared spectrum at 2121s, 2099m, and 2059s cm^{-1} . We have isolated this carbonyl, chloro compound of osmium by other routes, using non-silicon-containing reagents; it is currently being fully characterized [4].)

The hexane from the solution was removed at 0°C on the vacuum line. From the remaining solid, $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ (0.035 g, 0.08 mmol, 17%) was isolated as previously described. The solid (0.10 g) remaining after removal of the hydride was heated to 50°C at $<5 \times 10^{-3}$ mmHg in the presence of a water-cooled probe for 5 days. After each 24 h period the material which had sublimed to the probe was removed and checked by infrared spectroscopy. The spectrum revealed that the sublimate consisted mainly of $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$ plus a slightly more volatile component. After the fifth day, however, the infrared spectrum indicated that the material which had sublimed was essentially pure $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$. The temperature of the sublimation process was then increased to 65°C and continued for a further 24 h, whereupon $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$ (ca. 0.020 g, 0.023 mmol, 9.5%) was obtained as a very pale yellow, microcrystalline solid. The third, minor product ($\nu(\text{CO}) = 2108, 2089, \text{ and } 2060 \text{ cm}^{-1}$, hexane solution) which sublimed at 50°C and $<5 \times 10^{-3}$ mmHg could not be separated from $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$. (Previous experience has shown that chromatographic methods are not suitable for the separation of mixtures

of compounds which contain the reactive SiCl_3 ligand [5]).

Use of lower temperatures or shorter times for the reaction resulted in significant amounts of unreacted $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiCl}_3)_3$. An infrared spectrum of the reaction solutions (even after short reaction periods) always showed the most intense CO stretching absorptions of $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ and $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$ to be of approximate equal intensity.

The cleavage of $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiMeCl}_2)_3$ by carbon monoxide was essentially the same as described for the trichlorosilicon analogue except that the reaction proceeded at a convenient rate at 145°C . The dimeric complexes, $[\text{Os}(\text{CO})_4(\text{SiXCl}_2)]_2$, can be handled in air for short periods without apparent decomposition.

Pyrolysis of $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ under vacuum

A solution of $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ (0.040 g, 0.092 mmol) in hexane (15 ml) was heated at 140°C for 4.5 h. An infrared spectrum of the solution indicated that the major product was $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$ with traces of a second product ($\nu(\text{CO}) = 2080, 2073 \text{ cm}^{-1}$). An attempt to separate the products by sublimation was unsuccessful since both compounds sublimed at 60°C ($<5 \times 10^{-3}$ mmHg).

Pyrolysis of $\text{Os}(\text{CO})_4(\text{SiMeCl}_2)(\text{H})$ gave similar results.

Pyrolysis of $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ under carbon monoxide

A solution of $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ (0.030 g, 0.069 mmol) in hexane (15 ml) was heated at 170°C under carbon monoxide (90 atm) for 20 h. The autoclave was then cooled and the gases vented. An infrared spectrum (in the carbonyl region) of the reaction solution showed that the major component was unreacted starting material. Strong absorptions at 2035 and 1993 cm^{-1} were assigned to $\text{Os}(\text{CO})_5$ (Lit. [2] $\nu(\text{CO}) = 2034s, 1991vs \text{ cm}^{-1}$). Weak absorptions at 2081 cm^{-1} and 2052 cm^{-1} were attributed to *trans*- $\text{Os}(\text{CO})_4(\text{SiCl}_3)_2$ [1] and $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$, respectively.

A similar experiment with $\text{Os}(\text{CO})_4(\text{SiMeCl}_2)(\text{H})$ gave a solution with a more complicated infrared spectrum. This may have been due to scrambling of the methyl and chloro substituents on the silicon ligands of the various products [6]. In this way products such as $\text{Os}_2(\text{CO})_8(\text{SiMeCl}_2)(\text{SiR}_2\text{Cl})$ ($\text{R} = \text{Cl}, \text{Me}$) could result. * It did appear, however, that more of the dimeric product $[\text{Os}(\text{CO})_4(\text{SiMeCl}_2)]_2$ had formed than in the trichlorosilicon case. Bands due to $\text{Os}(\text{CO})_5$ were also clearly present.

The CO stretching absorptions assigned to $\text{Os}(\text{CO})_5$ were not due to $\text{Fe}(\text{CO})_5$. Thus, repeating the experiment described above (with the same CO source) but without $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ gave a hexane solution which had no significant absorptions in the $2200\text{--}1800 \text{ cm}^{-1}$ region of the infrared spectrum.

Results and discussion

By the UV irradiation of $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiXCl}_2)_3$ ($\text{X} = \text{Me}, \text{Cl}$) in hexane solution under carbon monoxide (2 atm) the hydrides *cis*- $\text{Os}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$

* A similar scrambling has been observed, in this laboratory, in the attempted synthesis of (arene) $\text{Os}(\text{CO})(\text{SiMeCl}_2)_2$ derivatives from $\text{Os}(\text{CO})_4(\text{SiMeCl}_2)_2$ and the arene, at elevated temperatures.

have been isolated. These compounds were characterized by infrared and ^1H NMR spectroscopy (Table 1). Thus, the compounds exhibited a characteristic high field resonance in the ^1H NMR for a terminal metal hydride linkage. The resonances were not as high field as those found [1] for the parent clusters which contain a bridging MHM unit. This is consistent with what has been observed previously [8]. Although four terminal CO stretching frequencies are expected in the infrared spectrum for molecules of the type *cis*- $\text{M}(\text{CO})_4(\text{X})(\text{Y})$, it is often found that the two modes of lowest energy are accidentally degenerate. The intensities and number of the CO stretching bands observed for the $\text{Os}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$ resemble those of *cis*- $\text{Os}(\text{CO})_4(\text{SiCl}_3)_2$ [9] and *cis*- $\text{Ru}(\text{CO})_4(\text{GeCl}_3)_2$ [10]. Stone and coworkers have reported the preparation of *cis*- $\text{Os}(\text{CO})_4(\text{SiR}_3)(\text{H})$ ($\text{R} = \text{Me}, \text{Et}$) [11]. These compounds, which were liquids at room temperature, exhibited four CO stretches.

The set of peaks of highest mass observed in the mass spectra of $\text{Os}(\text{CO})_4(\text{SiXCl}_2)(\text{H})$, corresponded to $[\text{Os}(\text{CO})_4]^+$ when either electron impact or chemical ionization was employed. Since ions due to $[\text{Os}(\text{CO})_4\text{H}]^+$ were not present to any significant intensity, elimination of HSiXCl_2 appears more probable than the separate loss of $\cdot\text{SiXCl}_2$ and $\cdot\text{H}$. If this is the case, it may indicate that reductive elimination of HSiXCl_2 is a more facile process in the positive ion than it is in the neutral molecule (vide infra). We could find no precedent for this behaviour in the literature, although in the mass spectrum of $(\text{C}_5\text{H}_5)\text{Mo}(\text{CO})_3(\text{SiH}_3)$ no ions containing the SiH_3 function were observed [12]. Apparently, the parent ion was observed in the mass spectrum of $\text{Os}(\text{CO})_4(\text{CH}_3)(\text{H})$ [13].

Because these hydrides were air-sensitive, low-melting solids, no attempt was made to characterize these compounds by elemental analysis. However, the triphenylphosphine derivatives, $\text{Os}(\text{CO})_3(\text{PPh}_3)(\text{SiXCl}_2)(\text{H})$, were easily prepared and were air-stable, crystalline solids which gave satisfactory analyses. The small value (~ 16 Hz) of the $^{31}\text{P}-^1\text{H}$ coupling shown by the metal-hydride signal, in the ^1H NMR spectrum of these derivatives, is indicative [14] of a *cis* arrangement of the PPh_3 and hydride ligands. The infrared spectrum (carbonyl region) is typical [9] of a *mer* arrangement of the non-carbonyl ligands about the osmium atom. This evidence indicates that substitution has occurred *trans* to the SiXCl_2 group, which is as was previously observed with *cis*- $\text{Ru}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ [15]. As has been previously found for the pair of molecules *cis*- $\text{M}(\text{CO})_4(\text{SiCl}_3)_2$ ($\text{M} = \text{Ru}, \text{Os}$) [9], substitution in *cis*- $\text{M}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ occurs much more readily for the second row transition metal complex. It is also interesting that substitution occurs more readily in $\text{M}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ than with the *cis*- $\text{M}(\text{CO})_4(\text{SiCl}_3)_2$ compounds [15].

Treatment of $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiCl}_3)_3$ in hexane at elevated temperatures with carbon monoxide (at 90 atm) also gave $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$. The other major product was $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$, although the yields were not good and there was infrared evidence for other products. Although it could not be completely ruled out for the SiMeCl_2 analogue, the hydride $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ does not give $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$ under these conditions (see below). An infrared spectrum taken in the initial stages of the reaction showed both the hydride and the dimeric derivative present which suggests they are formed concurrently in the reaction. A possible explanation for these observations is that once the first $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ molecule is cleaved from the $\text{Os}_3\text{H}_3(\text{CO})_9(\text{SiCl}_3)_3$ cluster the remaining

suggests that under these conditions reductive elimination of Cl_3SiH from $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ can occur. Note that $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$ is stable under the reaction conditions so that $\text{Os}(\text{CO})_5$ cannot arise from reaction of CO with this molecule. The formation of $\text{Os}(\text{CO})_5$ required higher temperatures (and longer times), which indicates that reductive elimination from $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ is a higher energy process than dimer formation.

The small amount of *trans*- $\text{Os}(\text{CO})_4(\text{SiCl}_3)_2$ detected in the reaction may be explained by the combination of $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ and the Cl_3SiH liberated in the reductive elimination. It has previously been found that *trans*- $\text{Os}(\text{CO})_4(\text{SiCl}_3)_2$ can be prepared from the action of excess Cl_3SiH on $\text{Os}_3(\text{CO})_{12}$ under carbon monoxide at 180°C [5].

It should not be forgotten that there were further minor products in the reactions described above. This suggests that $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ might decompose by still other routes. These products could, however, have resulted from the thermal decomposition of $[\text{Os}(\text{CO})_4(\text{SiCl}_3)]_2$. It is known that pyrolysis of $[\text{Fe}(\text{CO})_4(\text{SiCl}_3)]_2$ gives $[\text{Fe}(\text{CO})_4(\text{SiCl}_2)]_2$ [21]. With $\text{Os}(\text{CO})_4(\text{SiMeCl}_2)(\text{H})$ the results appeared further complicated by redistribution of the methyl and chloro groups in the products (see the Experimental section).

It should also be pointed out that Knox and Stone have reported the thermal decomposition of $\text{Os}(\text{CO})_4(\text{SiMe}_3)(\text{H})$ to give $[\text{Os}(\text{CO})_4(\text{SiMe}_3)]_2$ [11].

Dimer formation by $\text{Os}(\text{CO})_4(\text{SiCl}_3)(\text{H})$ or $\text{Os}(\text{CO})_4(\text{CH}_3)(\text{H})$ appears an energetically more favorable process than reductive elimination. However, elimination of Ph_3SiH from $(\text{C}_5\text{H}_5)\text{Mn}(\text{CO})_2(\text{SiPh}_3)(\text{H})$ is an extremely facile process although Cl_3SiH and Ph_3SiH , respectively, are eliminated more slowly from $(\text{C}_5\text{H}_5)\text{Mn}(\text{CO})_2(\text{SiCl}_3)(\text{H})$ and $(\text{C}_5\text{H}_5)\text{Re}(\text{CO})_2(\text{SiPh}_3)(\text{H})$ [22]. The factors which affect the reductive elimination of a silane from a metal complex obviously requires further investigation.

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