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AN EXAMINATION OF THE PRODUCT-CATALYZED REACTION OF TRIMETHYLGALLIUM WITH ARSINE

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Summary

The reaction of $(CH_3)_3Ga$ with AsH_3 at 203°C and 259°C has been examined over the product surfaces which were $(CH_3)_{3-x}GaAsH_{3-x}$ where the average values of x were 1.1 and 2.2 at 203°C and 259°C respectively. The surface reaction (catalyzed by the product surface) forming $(CH_3)_2GaAsH_2$ occurred on the surface between adsorbed molecules of $(CH_3)_3Ga$ and AsH_3. The surface coverages of the reactants (gas pressures between 18 and 36 mmHg) were clearly less than monomolecular and for AsH_3, possibly as low as 0.01. For AsH_3 at a surface coverage of 0.12, adsorption data were consistent with AsH_3 bound to the surface as a mobile film.

The formation of GaAs via CH_4 elimination from $(CH_3)_2GaAsH_2$ or CH_3GaAsH was hindered by deposition of films of $(CH_3)_{3-x}GaAsH_{3-x}$ even at 420°C. This was most significant for formation of GaAs (or even CH_3GaAsH) from $(CH_3)_2$-$GaAsH_2$ formed at 203°C and then heated at 420°C. The product surfaces also served as a catalyst for decomposition of AsH_3 to form H_2 and decomposition of $(CH_3)_3Ga$ to form CH_4.

Introduction

The reaction of $(CH_3)_3Ga$ with AsH_3 [1] is representative of a long series of reactions in which CH_4 is eliminated. Although these reactions have been utilized for their synthetic value; to our knowledge, there are no kinetic data on these reactions. These CH_4 elimination reactions have been utilized by two distinct groups. First, for the formation of organometallic compounds containing bonds between Main Group III and Group V elements such as $(Me_2AlPMe_2)_3$ [2], $(Me_2AlAsMe_2)_3$ [2], $(Me_2GaPMe_2)_3$ [2], $(Me_2InAsMe_2)_3$ [3], $(Me_2AlPPh_2)_2$ [3], $(Me_2GaNPh_2)_2$ [3] and $(MeBNMe)_3$ [4]. Second, for the preparation of semi-conductor materials such as SbB [5], InP [6] and InSb [1]. The latter group also contains the preparation of single crystal thin films of GaAs [7], GaP [7], $GaAs_{1-x}P_x$ [7], $GaAs_{1-x}Sb_x$, AlAs [8] and InAs [9].

We have examined the neat reaction of $(CH_3)_3Ga$ with AsH_3 as hopefully being somewhat representative of both classes. We found that the reaction was catalyzed by the product surface and that the homogeneous reaction was not competitive under our reaction conditions. The reaction of $(CH_3)_3Ga$ with AsH_3 was studied in an effort to determine the products as a function of reaction condition and an attempt was made to gain some understanding of the nature of the heterogeneous gas-solid reaction.

Results and discussion

We have examined the reaction of $Ga(CH_3)_3$ with AsH_3 in a static system between 203°C and 259°C. The pyrolyses commenced over a GaAs film deposited from the thermolysis of $(CH_3)_3Ga$ and AsH_3 at 259°C with a final 18-hour heating at 420°C. As will be seen later, the reaction surface during our experiments can be described as $(CH_3)_{3-x}GaAsH_{3-x}$ where x averaged 1.1 at 203°C and 2.2 at 259°C.

I. Control reactions

The only volatile products from the $(CH_3)_3Ga—AsH_3$ reaction were CH_4 and H_2. Methane was expected from reaction 1. We examined neat decompositions

$$(CH_3)_3Ga + AsH_3 \rightarrow GaAs + 3\ CH_4 \qquad (1)$$

of both AsH_3 and $(CH_3)_3Ga$ under our reaction conditions to determine whether CH_4 and H_2 could be obtained from their neat decompositions.

The pyrolysis of AsH_3 (1.5 mmol) at 275 mmHg was carried out for 2 h at 265°C over a silicon film, deposited from SiH_4 pyrolyses, without any observable decomposition. However, when this experiment was repeated in the same vessel over a GaAs film at 219 mmHg (1.2 mmol AsH_3) and 172°C, 0.09 mmol of H_2 was formed while 0.07 mmol of AsH_3 was consumed. These data demonstrate that our product surface catalyzed the decomposition of AsH_3 to form H_2.

The pyrolysis of Me_3Ga over our GaAs surface was examined. Typically, Me_3Ga (0.2 mmol) at 37 mmHg was heated for 75 min at 257°C, 0.0025 mmol of CH_4 was produced while 0.014 mmol of $(CH_3)_3Ga$ was consumed. Similarly, when $(CH_3)_3Ga$ at 36 mmHg was heated for 240 min at 204°C, 0.0072 mmol of CH_4 was produced while 0.019 mmol of $(CH_3)_3Ga$ was consumed. We have previously examined the pyrolysis of $(CH_3)_3Ga$ over germanium and arsenic films at 245°C and 300°C at pressures of 175 mmHg in similar vessels where CH_4 was not produced [10]. This result was consistent with those reported by Jacko and Price [11]. These data demonstrate that $(CH_3)_3Ga$ is catalytically decomposed by our product surface to form CH_4. In these experiments, the quantity of CH_4 formed was essentially constant under one set of conditions while the consumption of $(CH_3)_3Ga$ varied, presumably due to hydrolysis or oxidation reactions during manipulations.

II. Solid products and demonstration of heterogeneous character of reaction

The results of our reactions of $(CH_3)_3Ga$ with AsH_3 over our surfaces are list-

TABLE 1

REACTIONS OF $(CH_3)_3Ga$ WITH AsH_3

Initial pressure (mmHg)			Consumed (mmol)		Produced (mmol)			
Exp. [a]	$(CH_3)_3Ga$	AsH_3	$(CH_3)_3Ga$	AsH_3	B [b]	A [b]	B : A	B' : A [b]
1	35.9	35.9	0.0365	0.0303	0.0227	0.0250	0.91	1.14
2	35.9	35.9	0.0381	0.0245	0.0218	0.0200	1.09	1.38
3	35.4	35.4	0.0541	0.0278	0.0272	0.0237	1.15	1.39
4	35.5	18.1	0.0365	0.0168	0.0125	0.0131	0.95	1.17
5	36.0	18.1	0.0348	0.0147	0.0104	0.0119	0.88	1.09
6	36.0	18.0	0.0443	0.0168	0.0174	0.0135	1.29	1.56
7	27.1	27.1	0.0320	0.0193	0.0163	0.0148	1.10	1.35
8	18.0	36.2	0.0213	0.0180	0.0105	0.0123	0.85	1.05
9	18.2	36.4	0.0176	0.0213	0.0176	0.0168	1.05	1.27
10	18.0	36.0	0.0250	0.0234	0.0253	0.0189	1.34	1.53
11	18.2	36.4	0.0234	0.0234	0.0232	0.0197	1.18	1.37
12	18.0	36.0	0.0217	0.0213	0.0228	0.0172	1.33	1.54
13	18.0	36.0	0.0230	0.0193	0.0165	0.0152	1.09	1.33
14	18.0	35.8	0.0270	0.0217	0.0259	0.0172	1.51	1.72
15	18.1	36.3	0.0225	0.0221	0.0162	0.0180	0.90	1.11
16	35.9	23.5	0.0262	0.0262	0.0495	0.0193	2.56	2.90
17	36.2	23.8	0.0332	0.0299	0.0549	0.0221	2.48	2.72
18	35.7	23.3	0.0258	0.0246	0.0414	0.0172	2.41	2.85
19	36.4	25.6	0.0459	0.0348	0.0577	0.0262	2.20	2.94
20	18.4	26.2	0.0164	0.0205	0.0324	0.0143	2.27	2.70
21	18.0	26.1	0.0139	0.0250	0.0299	0.0151	1.98	2.39
22	27.1	24.8	0.0180	0.0279	0.0436	0.0189	2.31	2.70
23	27.1	25.4	0.0217	0.0275	0.0342	0.0205	1.67	2.00
24	27.5	36.0	0.0299	0.0311	0.0501	0.0205	2.44	2.84
25	26.9	36.4	0.0245	0.0336	0.0461	0.0246	1.87	2.14
26	26.9	17.1	0.0176	0.0168	0.0235	0.0115	2.04	2.47
27	26.4	17.3	0.0197	0.0164	0.0227	0.0111	2.05	2.59

[a] Experiments 1—15 carried out at 203°C for 240 min while experiments 16—27 were carried out at 259°C for 75 min. [b] See text.

ed in Table 1. The minimum set of equations required to explain our data are:

$$AsH_3 \rightarrow As + 3/2H_2 \tag{2}$$

$$2(CH_3)_3Ga \rightarrow \text{Product} + CH_4 \tag{3}$$

$$(CH_3)_3Ga + AsH_3 \rightarrow (CH_3)_2GaAsH_2 + CH_4 \tag{4}$$

$$(CH_3)_2GaAsH_2 \rightarrow H_3CGaAsH + CH_4 \tag{5}$$

$$H_3CGaAsH \rightarrow GaAs + CH_4 \tag{6}$$

The experiments listed in Table 1 were carried out at 203°C and 259°C. The volatile materials were distilled off and then the solid film was heated at 420°C for 18 h. Since some H_2 (about 10% of total) was produced at 420°C, we assume that the initial solid product contained an arsenic film and an AsH polymer which was presumably converted to arsenic at 420°C. The ratio of 2 : 1 for eq. 3 is assumed and based on a presumed intermolecular nature of reaction 3. The ratio, CH_4 produced/$(CH_3)_3Ga$ consumed, was close to 1 : 2 in most control reactions. Such a ratio suggests that the product in reaction 3 could be $(CH_3)_2GaCH_2Ga(CH_3)_2$. The important points are that the $(CH_3)_xGaAsH_x$ surface catalytically

The theoretical rate law using Langmuir adsorption isotherms (assumes all catalytic sited equivalent) for a two-site model yields eq. 12 [12], where k is the surface reaction rate constant, b_a and b_b are adsorption equilibrium constants * and P_a and P_b are reactant pressures. The form of eq. 12 assumes that CH_4 desorption has no effect on the rate. This assumption was verified by obtaining rate data in the presence of CH_4. When reactions were carried out under the conditions of runs 1 and 2 in Table 1 with CH_4 present at 26 mmHg, the value of A was 0.20 compared to an average of 0.225 for runs 1 and 2 without added CH_4. The maximum pressure of CH_4 at the end of any experiment listed in Table 1 was 3 mmHg. Thus, these control experiments clearly demonstrate that CH_4 desorption was not rate controlling. This conclusion was supported by examination of a CH_4 isotherm at —78°C on our reaction surface which suggested that the heat of adsorption of CH_4 was very small.

$$\text{rate} = \frac{kb_a b_b P_a P_b}{(1 + b_a P_a)(1 + b_b P_b)} \tag{12}$$

The decrease in reaction order from unity for a Langmuir—Hinshelwood mechanism can be due to either surface poisoning by the reagents, decreasing values of the adsorption constants b at higher pressure or due to both effects. The surface poisoning is related to the bP terms in the denominator of eq. 12 while the changes in b are in effect lower values of the heat of adsorption, Q, with an increase in surface coverage, θ, as the reactant pressures increase.

If $dQ/d\theta$ is zero, the data in Table 1 and eq. 12 allow for calculation of average values for the adsorption constants of AsH_3 and $(CH_3)_3Ga$ on our reaction surfaces during the kinetic experiments. These values ranged from 0.003 for AsH_3 at 259°C to 0.05 for $(CH_3)_3Ga$ at 203°C. Since the values of Q normally decrease with an increase in θ, the above are maximum values.

Adsorption equilibrium constants can also be obtained from static experiments. We examined the equilibrium adsorption of AsH_3 on a silica supported surface (280 m^2) essentially identical to that present in our kinetic experiments. The results from these static experiments are listed in Table 2. It is interesting to note the small change in θ with pressure due to changes in b with θ. The data in Table 2 demonstrate that $dQ/d\theta$ for AsH_3 is not zero. We have a value for $b(AsH_3)$ of 4×10^{-3} at 0°C at θ equal to 0.121. With Q equal to 6 kcal mol^{-1}, one obtains a value for $b(AsH_3)$ at 259°C equal to 2×10^{-5} ($\theta = 0.12$) which would be at 7×10^3 mmHg of AsH_3. Since our kinetic runs were carried out at 36 mmHg and lower, the values for $b(AsH_3)$ would most probably be greater than 2×10^{-5}. Thus the actual value of $b(AsH_3)$ during our kinetic experiments lies between 10^{-5} and 10^{-3} or probably about 10^{-4}. We were unable to study the $(CH_3)_3Ga$ adsorption due to catalytic decomposition on the surface.

The changes in b with p in Table 2 are sufficient, if the trend continues at lower pressure, to account for the non-first order kinetics observed from our kinetic runs. However, the non-first order kinetics are probably due both to lower values of b at higher pressure and more surface poisoning at higher pressure.

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* For reaction $A + S \rightleftharpoons AS$ where A is a gas phase molecule and S is a surface site and where $\theta = bp/(1 + bP)$ = surface coverage.

TABLE 2

AsH_3 ADSORPTION RESULTS ON A $(CH_3)_xGaAsH_x$ SURFACE

T 0°C			T 23°C		
θ	$(b \times 10^3)^a$	P(mmHg)	$(b \times 10^3)^a$	P(mmHg)	Q^b (kcal mol^{-1})
0.121	3.90	35.4	1.64	84.3	6.0
0.125	3.13	45.7	1.43	98.9	5.4
0.140	2.16	75.4	1.30	125.4	3.5
0.158	1.50	125.4	1.14	164.1	1.9

[a] The values of b were obtained from experimental values of θ and the relationship: $bp = \theta/(1-\theta)$. The units of b in this table are mmHg^{-1}. [b] Obtained from the b values at 23°C and 0°C and the relation, $\ln P_2/P_1 = Q/R\,(1/T_1 - 1/T_2)$.

IV. Nature of the arsine—surface complex

It is possible to elucidate the nature of the arsine—surface complex present during the adsorption experiments from isotherms at 23°C and 0°C. The experimental differential molar entropy of the adsorbed species is given by eq. 13 [13].

$$S_a(\theta) = S_g^0 - R\ln\left(\frac{P}{P^0}\right) + \frac{\Delta H(\theta)}{T} \tag{13}$$

where S_g^0 is the AsH_3 gas phase entropy, P is the equilibrium pressure and P^0 is the standard pressure (1 atm) while ΔH is the heat of adsorption at the appropriate fractional θ.

The total gas phase entropy can be calculated from standard statistical thermodynamic formulae [14] which yield $S_g^0 = 52.45$ e.u. at 1 atm pressure and 273 K. At a θ of 0.121, where $\Delta H = 6.05$ kcal mol^{-1} and $P_{273} = 35.4$ mmHg (see Table 2) we have eq. 14.

$$S_a\,(0.121) = 52.45 - R\ln\left(\frac{35.4}{760}\right) - \frac{6050}{273} = 36.4 \text{ e.u.} \tag{14}$$

It is possible to calculate upper and lower limits for the entropy of the surface complex from theoretical models and see which more closely approximates the experimental value.

If the molecule is totally adsorbed to one site without rotation or vibration, the only contribution to the entropy would be the configurational entropy given by eq. 15 [15].

$$S_a = -R\ln\frac{\theta}{1-\theta} = 3.9 \text{ e.u.} \tag{15}$$

However, if we have a totally mobile film with rotation and vibration as in the gas phase (with 2 degrees of translational freedom) the entropy is given by eq. 16 [15].

$$S_a = R\ln\left(\frac{MTA}{\theta}\right) + 63.8 + 13.9 = 35.2 \text{ e.u.} \tag{16}$$

where M is the molecular weight, T is temperature and A is the area of one molecule (29 Å^2 as determined from the −78°C isotherm).

Thus the experimental entropy is within experimental error of the calculated entropy for an adsorbed arsine mobile film. Thus it appears that at these high coverages ($\theta = 0.121$), the AsH_3 is adsorbed as a mobile film. However, during the kinetic runs (Table 1) the surface coverages may have been lower by a factor of ten (if b was 3×10^{-4}) with possible higher values of ΔH which would decrease the surface mobility. A similar study carried out for AsH_3 on GaP suggested that a mobile film had been present [16].

Experimental

All experiments were carried out in a standard high vacuum line. The $(CH_3)_3$-Ga was obtained from Ventron Corporation (Alfa products) while the AsH_3 was prepared by reduction of AsO_2^- with $NaBH_4$ in acid solution. The purity of $(CH_3)_3Ga$ and AsH_3 were demonstrated by infrared and mass spectral analyses which failed to detect the presence of any other compounds.

Kinetic runs

The runs listed in Table 1 were carried out in an 80 cm^3 Pyrex reaction vessel wrapped with a heating tape and heavily insulated to stabilize temperature. The temperature of the vessel was monitored by a chromel-alumel thermocouple inserted into a well which extended to the center of the vessel. A U trap with an attached 200 cm^3 expansion bulb was connected to this vessel. This assembly was connected to a standard high vacuum system. Pressures were measured with a mercury manometer read through a travelling telescope which gave an accuracy of 0.1 mmHg. The interior of the vessel was coated with the solid product from previous runs.

In a typical experiment $(CH_3)_3Ga$ (1.0 mmol) was expanded into the 200 cm^3 expansion bulb. The pressure was noted, and the Teflon valve to the bulb was closed and the $(CH_3)_3Ga$ removed from the rest of the system. Arsine (2.5 mmol) was expanded into the trap which gave a higher pressure of AsH_3 in the trap than of $(CH_3)_3Ga$ in the bulb. The Teflon valve connecting the trap to the bulb was then opened for 5 sec, and the AsH_3 was allowed to flow into the bulb. During this interval, the pressure in the two parts came to equilibrium, and the pressure was read to obtain the ratio of the two reactants. It was assumed that no $(CH_3)_3$-Ga was lost from the bulb by diffusion. Control experiments were carried out to show that this was the case. The AsH_3 left in the trap was removed and the two reactants allowed to stand for about 1 min to ensure that complete mixing had taken place. This mixture was then expanded into the reaction vessel and the temperature and pressure were noted. From these data, it was possible to calculate the number of mmol of each reactant in the vessel. Reactions were carried out at 259°C, 230°C, and 203°C for 75, 115 and 240 min respectively. The ratio of $(CH_3)_3Ga$ to AsH_3 was varied from 2.0 to 0.5. The pressures are given in Table 1.

After the reaction was complete, the products were toepler pumped into a known volume and a pressure reading taken. The products were then recirculated through a —78°C trap and again toepler pumped into the known volume. The pressure was read and the amount of $(CH_3)_3Ga$ removed determined by difference. It was determined by infrared spectral analysis that a —78°C trap would quantitatively separate $(CH_3)_3Ga$ from AsH_3 in this system. The remaining products were recirculated through a —196°C trap to remove the AsH_3 for quantitative determination. The noncondensible products were then toepler pumped into the same volume and measured. A sample of the noncondensible gases was taken, and a mass spectrum obtained on a Hitachi—Perkin—Elmer RMU-6E spectrometer. This spectrum was compared with that of a known standard to determine the relative quantities of hydrogen and methane. The solid product was then baked at 420°C for 18 h and any gases produced were toepler pumped into the known volume and a mass spectrum taken, if possible, to determine the composition of the fraction. In all cases the products from the baking procedure were hydrogen and methane.

Reaction in a packed reactor

The reaction vessel was packed with Pyrex Raschig rings which had a geometrical surface area of 230 cm^2. The vessel had an interior surface area of 130 cm^2. Thus the area was increased by a factor of 2.8 as the volume was decreased from 80 to 55 cm^3. A surface similar to those present in the kinetic runs was deposited by the reaction of $(CH_3)_3Ga$ and AsH_3. Arsine (0.0631 mmol) and $(CH_3)_3Ga$ (0.0574 mmol) were expanded into the vessel as in the previous experiments. The pressure in the vessel was 54.1 mmHg with the temperature 259°C. The reaction was continued for 20 min at which time 0.0234 mmol of AsH_3 had been consumed. This quantity was then corrected for the H_2 produced which resulted in a value of 0.0152 mmol of AsH_3 which reacted with $(CH_3)_3Ga$. This can be compared to 0.0246 mmol of AsH_3 which reacted with $(CH_3)_3Ga$ from an initial 0.0836 mmol of AsH_3 in 75 min at the same temperature and pressure in the unpacked vessel.

The change in rate constant due to the increase in surface area can be calculated as follows. If the assumption is made that $(CH_3)_3Ga$ is essentially constant for both reactions then the rate constant can be approximated by the expression:

$$k' = \frac{1}{t} \ln(A_0/A)$$

where A_0 is the initial concentration of AsH_3, A is the final concentration of AsH_3, t is the time and k' is $k[(CH_3)_3Ga]$.

The ratio of the two rate constants in the packed and unpacked is then:

$$\frac{k'_1}{k'_2} = \frac{t_2 \ln(A_{01}/A_1)}{t_1 \ln(A_{02}/A_2)}$$

where subscript 1 signifies the packed vessel and subscript 2 signifies the unpacked vessel. Thus for these experiments:

$$\frac{k'_1}{k'_2} = \frac{75 \ln(0.0631/0.0479)}{20 \ln(0.0836/0.0590)} = 2.96$$

where the value of A is A_0 less the amount reacted with $(CH_3)_3Ga$. This ratio will be a lower limit since the AsH_3 concentration was actually slightly lower due to production of H_2 and $(CH_3)_3Ga$ consumption was not strictly equal for the two reactions.

Adsorption experiments

These experiments were carried out in a 150 cm^3 reaction flask having a flat bottom to provide maximum accessibility of the reactants to the catalyst. A heating mantle was used to heat the vessel, and glass wool was wrapped around the assembly to prevent rapid temperature fluctuations. Silica gel (30/60 mesh) was used as the support. The silica gel was coated with GaAs by allowing an equimolar mixture of $(CH_3)_3Ga$ and AsH_3 to expand into the flask which was maintained at 260°C. The reaction was allowed to proceed for 30 min at which time the products were removed. The procedure was repeated several times until the silica gel was completely coated. The surface area (280 m^2) of the catalyst was measured utilizing the one point BET method [17] using nitrogen at −196°C as the adsorbate and assuming a cross-section for nitrogen of 16.5 Å^2/molecule. A full adsorption isotherm of nitrogen on GaAs at −196°C was carried out and it was found that the assumptions made in using the one point BET method were justified in this case.

The adsorption isotherm was measured by allowing a known quantity of AsH_3 to expand into the vessel which was immersed in a temperature controlling bath. Temperatures used were 23°C, 0°C and −78°C. The system was allowed to equilibrate for 12 h and the pressure recorded utilizing a travelling telescope to read a mercury monometer. A small known quantity of AsH_3 was then removed, and the system allowed to equilibrate for 30 min. The equilibrium pressures ranged from 35 to 215 mmHg. After the isotherm had been recorded, the system was baked under vacuum at 300°C for 18 h to outgas any AsH_3 on the surface before the next isotherm was taken. The heat of adsorption was then calculated from eq. 17 and 18.

$$\left[\frac{\partial(\ln P)}{\partial\left(\frac{1}{T}\right)}\right]_\theta = \frac{\Delta H}{R} \tag{17}$$

TABLE 3

ADSORPTION DATA OF AsH_3

T 23.4°C		T 0°C		T −78°C	
P (mmHg)	mmol ads.	P (mmHg)	mmol ads.	P (mmHg)	mmol ads.
215.3	0.297	162.3	0.311	102.2	2.17
188.0	0.261	142.7	0.265	94.0	2.11
164.1	0.250	125.4	0.259	87.7	2.02
143.2	0.244	110.4	0.250	81.5	1.95
125.4	0.230 [a]	97.4	0.238	75.5	1.89
110.5	0.208	85.6	0.235	72.1	1.79
96.5	0.204	75.4	0.230	65.5	1.77
84.3	0.199	66.8	0.217	60.9	1.71
74.2	0.191	58.8	0.215 [a]	56.6	1.66
64.7	0.187	51.9	0.208	52.7	1.62
56.6	0.183	45.7	0.205	49.4	1.56
		40.1	0.202	46.0	1.52
		35.4	0.199	43.1	1.48

[a] Average of two points.

$$\theta = bp/(1 + bP) \tag{18}$$

From a BET analysis of the —78°C adsorption data (Table 3) it was possible to obtain a value for V_m (volume of gas at monolayer coverage) which was 1.64 mmol. Thus the value of θ is equal to: mmol of AsH_3 adsorbed divided by 1.64.

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