Efficient three-component Strecker reaction of acetals and aromatic amines catalysed by hafnium tetrachloride at room temperature

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A straightforward, mild, efficient, one-pot method has been found for the synthesis of α-aminonitriles via three-component Strecker reaction using acetals or cyclic acetals, curious aromatic amines and trimethylsilyl cyanide (TMSCN) catalysed by hafnium tetrachloride at room temperature. It is with good to excellent yields under mild conditions. This developed approach has been successfully applied for the synthesis of a wild rang of α-aminonitriles with a variety of functional groups.

Keywords: acetals, Strecker reaction, α-aminonitriles, trimethylsilyl cyanide

Acetals play a key role in natural products,[1] and in synthetic and medicinalchemistry,[2] and the medicinal chemistry. In particular, they are extensively used as protecting groups in synthetic chemistry.[3] They undergo a large number of reactions such as the Mukaiyama aldol reaction of acetals with enol silyl ethers which was reported in 1974.[4] Since then, there are many examples reported of the displacement of only one alkoxy group, as well as transprotection reactions.[5]

The Strecker reaction has been one of the most important multicomponent reactions in organic chemistry since it was first reported 1850.[6,7] It is used in C–C bond formation to make α-aminonitriles.[8] α-Aminonitriles are very useful, compounds which possess important biological activities.[9] α-Aminonitriles are generally prepared by the nucleophilic addition of the cyanide anion to imines using a variety of cyanating agents[6,7] under Strecker-type reaction conditions.[10] Me_3SiCN is an effective and safe source of cyanide anions and is commonly employed as cyanide source in the presence of various Lewis acids, metal complexes, solid supported acids and organic catalysts.[11–16]

However, there are many short-comings in the reported methods, such as long reaction time, the formation of large amounts of toxic by-products, the need for large amount of catalysts and harsh reaction conditions.[17] Several alternatives have been developed to overcome these drawbacks as well as devising novel processes to efficiently catalyse the Strecker reaction.[18] The reaction has been mostly reported to take place efficiently using aldehydes. Three-component Strecker reactions using acetals as substrates have rarely been reported.

We now report an efficient method for the synthesis of α-aminonitriles involving the reaction of the corresponding acetals, aromatic amines, and TMSCN using $HfCl_4$ as the catalyst under mild conditions (Scheme 1). To the best of our knowledge, this is the first report of the synthesis of α-aminonitriles employing acetals.

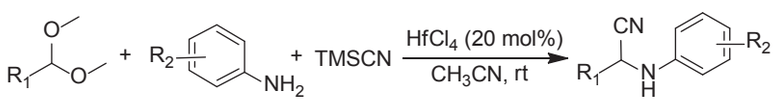
Initially, in order to obtain the best reaction conditions, various Lewis acids were screened in the three-component Strecker reaction using benzaldehyde dimethyl acetal (1.2 equiv.), *p*-toluidine (1.0 equiv.), and TMSCN (1.3 equiv.) in acetonitrile under an argon atmosphere at room temperature. The results are summarised in Table 1. The three-component Strecker reaction proceeded smoothly and generated the desired product in 91% yield, representing one of the best results when 20 mol% of $HfCl_4$ was used as the catalyst without any cocatalyst or activator at very short time (0.5 h) (Table 1, entry 1). When using $Hf(OTf)_4$, $ZrCl_4$ and $InCl_3$, although the reaction time was short, the yields were lower on contrary with $HfCl_4$ (Table 1, entries 2–4). Compared with $HfCl_4$, the reaction time of $FeCl_3$, I_2, $AlCl_3$, $BiCl_3$ and $Bi(OTf)_3$ was longer, and the yields were much lower (Table 1, entries 5–9). $SnCl_2$ and Cu salts (such as CuCl, CuBr, CuI, CuOTf) were inferior and generated the desired product in 88, 42, 76, 87 and 31 yields, respectively, and the reaction time was much longer than $HfCl_4$ (Table 1, entries 10–14). $HfCl_4$ was therefore chosen as the catalyst for other substrates.

Table 1 Three-component Strecker reaction of benzaldehyde dimethyl acetal, *p*-toluidine and trimethylsilyl cyanide (TMSCN) with different catalysts[a]

Entry	Catalyst	Time/h	Yield/%[b]
1	$HfCl_4$	0.5	91
2	$Hf(OTf)_4$	1	76
3	$ZrCl_4$	1	75
4	$InCl_3$	2	88
5	$FeCl_3$	6	57
6	I_2	7	82
7	$AlCl_3$	8	53
8	$BiCl_3$	8	81
9	$Bi(OTf)_3$	12	84
10	$SnCl_2$	23	88
11	CuCl	24	42
12	CuBr	24	76
13	CuI	24	87
14	CuOTf	23	31

[a]Reaction conditions: benzaldehyde dimethyl acetal (1.2 mmol), *p*-toluidine (1.0 mmol), TMSCN (1.3 mmol), catalyst (0.20 mmol), acetonitrile (4.0 mL), r.t.
[b]Isolated yields after flash chromatography.

We next screened the effect of solvent on three-component Strecker reaction of the model substrates by using 20 mol% of $HfCl_4$ as the catalyst under an argon atmosphere at room temperature (Table 2). Among the solvents tested, it was observed that a much better yield was obtained when the reaction was carried out in acetonitrile at room temperature compared to other solvents. Acetonitrile was the most suitable reaction medium for the three-component Strecker reaction (Table 2, entry 1). Among the other solvents screened, toluene,



Scheme 1 Strecker reaction of acetals, aromatic amines, and TMSCN in CH_3CN.

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Table 2 Screening the effect of various solvents for the three-component Strecker reaction of benzaldehyde dimethyl acetal, *p*-toluidine and trimethylsilyl cyanide (TMSCN)[a]

Entry	Solvent	Time/h	Yield/%[b]
1	Acetonitrile	0.5	91
2	Toluene	1	80
3	Ethanol	1	28
4	THF	1	79
5	1,4-dioxane	1.5	31
6	Dichloromethane	1.5	82
7	DCE	2	78
8	Nitromethane	2	87

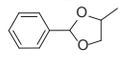
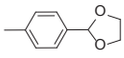
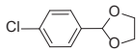
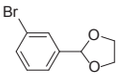
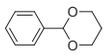
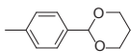
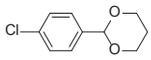
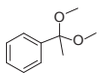
[a]Reaction conditions: benzaldehyde dimethyl acetal (1.2 mmol), *p*-toluidine (1.0 mmol), TMSCN (1.3 mmol), $HfCl_4$ (0.064 g, 0.2 mmol), solvent (4.0 mL), r.t.
[b]Isolated yields after flash chromatography.

ethanol, and THF were inferior and generated the corresponding products in 80%, 28%, and 79% yields, respectively after a prolonged reaction time (Table 2, entries 2–4). Note that when 1,4-dioxane and dichloromethane were used as the solvents, the yields were very different although the reaction time was the same (Table 2, entries 5 and 6). When DCE was used as the solvent, the yield was close to that of nitromethane at the same reaction time (Table 2, entries 7 and 8). Thus, all the reactions were performed in acetonitrile under an argon atmosphere with 20 mol% of $HfCl_4$ at room temperature without any cocatalyst or activator.

Subsequently, a variety of α-aminonitriles were prepared from various acetals, and aromatic amines using the previously optimised reaction conditions. The results are summarised in Table 3. At the beginning of the investigation into the acetal substrate scope, aniline and TMSCN were used as model substrates and a variety of acetals and cyclic acetals were examined in the three-component Strecker reactions (Table 3, entries 1–14). As can be seen from Table 3, acyclic acetals were often much more reactive than cyclic acetals. Acyclic acetals, with both electron-donating and electron-withdrawing groups attached to the benzene ring, underwent the three-component Strecker reaction and smoothly, generated the corresponding products in good to excellent yields (Table 3, entries 1–7). Then, the three-component Strecker reaction was examined using cyclic acetals (Table 3, entries 8–14). Aromatic cyclic acetal possessing no substituent group afforded good yields (Table 3, entry 8). Five-membered cyclic acetals with electron-donating groups attached to the benzene rings did not decrease the reactivity, but electron-withdrawing groups did (Table 3, entries 9–11). Interestingly, six-membered cyclic acetals with electron-withdrawing groups attached to the benzene rings gave the desired product more smoothly than those with electron-donating groups (Table 3, entries 12–14). Unfortunately, when (1,1-dimethoxy-ethyl) benzene was used as the substrate, the yield was much lower than acetals (Table 3, entry 15).

In order to expand the scope of the amine substrates, several aromatic amines were examined for the synthesis of α-aminonitriles using benzaldehyde dimethyl acetal and TMSCN as the model substrates. Good to excellent yields of the desired products were obtained in each case (Table 3. The reactions were completed within 0.5 h affording 81–95% yields. The results indicated that aromatic amines with both electron-donating and electron-withdrawing groups attached to the benzene rings underwent the reaction to furnish the desired products in good yields (Table 3, entries 16–21). With aliphatic alkynes such as octyne and decyne only a trace amount of the product was obtained. However, when secondary amines, such as Bn_2NH were used as an amine substrate, a good yield of

Table 3 Three-component Strecker reactions of acetals, aromatic amines and TMSCN using $HfCl_4$ catalyst[a]

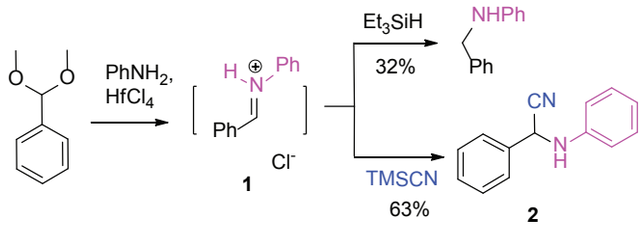
Entry	Acetal (R_1)	Amine (R^2)	Time/h	Yield/%[b]
1	C_6H_5	*p*-H	0.5	91
2	*p*-$CH_3C_6H_4$	*p*-H	0.5	86
3	*p*-$CH_3OC_6H_4$	*p*-H	0.5	88
4	*p*-FC_6H_4	*p*-H	0.5	96
5	*p*-ClC_6H_4	*p*-H	0.5	91
6	*p*-BrC_6H_4	*p*-H	0.5	92
7	*m*-$NO_2C_6H_4$	*p*-H	0.5	93
8		*p*-H	0.5	76
9		*p*-H	0.5	76
10		*p*-H	0.5	70
11		*p*-H	0.5	67
12		*p*-H	0.5	75
13		*p*-H	0.5	74
14		*p*-H	0.5	81
15		*p*-H	0.5	43
16	C_6H_5	*p*-CH_3	0.5	91
17	C_6H_5	*m*-CH_3	0.5	81
18	C_6H_5	*p*-CH_3O	0.5	87
19	C_6H_5	*p*-Cl	0.5	94
20	C_6H_5	*p*-NO_2	0.5	95
21	C_6H_5	*p*-COOEt	0.5	95
22	C_6H_5	Bn_2NH	5	74

[a]Reaction conditions: acetal (1.2 mmol), aromatic amine (1.0 mmol), TMSCN (1.3 mmol), $HfCl_4$ (0.064 g, 0.20 mmol), acetonitrile (4.0 mL), room temperature.
[b]Isolated yields after flash chromatography.

product was obtained under the standard reaction conditions, but with longer reaction time (Table 3, entry 22).

To elucidate the reaction pathway, we hoped to intercept the reaction intermediate. Yang and coworkers[19] demonstrated that [In–H] generated in the $InCl_3/Et_3SiH$/MeOH system is an active agent for the reductive amination of aldehydes with various amines. Accordingly, Et_3SiH-$InCl_3$ is able to trap the iminium ion if it occurs in the reaction solution. Under our standard reaction conditions, Et_3SiH (2.0 equiv.) was added to the reaction solution of benzaldehyde dimethyl acetal, aniline, and TMSCN. Both *N*-benzylaniline (the reductive amination product) and the corresponding α-aminonitrile **2** were obtained of 32% and 63% isolated yields, respectively (Scheme 2). The *N*-benzylaniline was formed from the reduction of benzyl iminium ion **1**, involving substitution of both the methoxy groups of benzaldehyde dimethyl acetal with aniline. At the same time, a α-aminonitrile product **2** was formed by the attack of TMSCN on the intermediate **1**. These results show that this protocol proceeds *via* an iminium ion intermediate **1**.

In conclusion, we have developed a simple and efficient method for the three-component Strecker reaction of acetals, aromatic amines and TMSCN in acetonitrile through C–C bond activation at room temperature to yield α-aminonitriles with



Scheme 2 Intercepting the reaction intermediate with iminium ion.

good to excellent yields using $HfCl_4$ as the catalyst. The process was simple. Aromatic acetals are particularly effective in the reaction, which provides an extension to the $HfCl_4$ catalysed three-component Strecker reaction. The reaction is efficient and has a high atom-economy in this mild three-component Strecker reaction. The scope, mechanism, stereoselectivity, and synthetic applications of this reaction are under further investigation.

Experimental

Reactions were performed under an argon atmosphere at room temperature. The materials were used as purchased. Unless otherwise stated, all solvents and reagents were commercially available and used as purchased without further purification. Reactions were monitored by thin-layer chromatography using gel F 254 plates. The silica gel (300–400 mesh) was used for column chromatography, and the distillation range of petroleum ether was 60–90 °C. NMR spectra was recorded in $CDCl_3$ on either a Varian 400 MHz or Bruker 400 MHz Fourier-transform spectrometer. Chemical shifts were reported in ppm referenced to TMS or the $CHCl_3$ solvent residual peak at 7.26 ppm for ^1H and 77.23 ppm for ^{13}C.

Acetal (1.2 mmol), aromatic amine (1.0 mmol), TMSCN (0.165 mL, 1.3 mmol), and $HfCl_4$ (0.064 g, 0.2 mol) were added to a flask (25 mL), followed by addition of acetonitrile (4.0 mL) under argon. The mixture was stirred at room temperature and monitored by TLC. The solution was then diluted with dichloromethane (5.0 mL), washed with brine. The aqueous layer was extracted with CH_2Cl_2 (3 × 10 mL), the combined organic layer was dried over $MgSO_4$, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel (petroleum ether) to afford the desired product.

2-Phenyl-2-(phenylamino)acetonitrile: White solid, m.p. 76–77 °C (lit.[20] 76–78 °C); yield 91% (Table 3, entry 1), 76% (Table 3, entry 8) and 75% (Table 3, entry 12); ^1H NMR (400 MHz, $CDCl_3$) δ 7.53 (dd, *J*=7.5, 1.9 Hz, 2H), 7.44–7.36 (m, 3H), 7.24–7.18 (m, 2H), 6.83 (t, *J*=7.4 Hz, 1H), 6.71 (d, *J*=8.2 Hz, 2H), 5.36 (s, 1H), 3.96 (s, 1H).

2-(Phenylamino)-2-(p-tolyl)acetonitrile: White solid, m.p. 77–78 °C (lit.[20] 69–71 °C); yield 86% (Table 3, entry 2), 76% (Table 3, entry 9) and 74% (Table 3, entry 13); ^1H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J*=8.0 Hz, 2H), 7.26 (t, *J*=8.2 Hz, 4H), 6.89 (t, *J*=7.8 Hz, 1H), 6.76 (d, *J*=8.5 Hz, 2H), 5.37 (d, *J*=7.9 Hz, 1H), 3.99 (d, *J*=7.7 Hz, 1H), 2.38 (s, 3H).

2-(4-Methoxyphenyl)-2-(phenylamino)acetonitrile: White solid, m.p. 95–96 °C (lit.[20] 93–94 °C); yield 88% (Table 3, entry 3); ^1H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J*=8.7 Hz, 2H), 7.23–7.18 (m, 2H), 6.90 (d, *J*=8.7 Hz, 2H), 6.83 (t, *J*=7.4 Hz, 1H), 6.70 (d, *J*=8.2 Hz, 2H), 5.29 (s, 1H), 3.90 (s, 1H), 3.77 (s, 3H).

2-(4-Fluorophenyl)-2-(phenylamino)acetonitrile: White solid, m.p. 98–99 °C (lit.[21] 98–100 °C); yield 96% (Table 3, entry 4); ^1H NMR (400 MHz, $CDCl_3$) δ 7.65–7.56 (m, 2H), 7.31–7.25 (m, 2H), 7.19–7.12 (m, 2H), 6.92 (t, *J*=7.4 Hz, 1H), 6.78 (d, *J*=7.7 Hz, 2H), 5.42 (s, 1H), 4.02 (s, 1H).

2-(4-Chlorophenyl)-2-(phenylamino)acetonitrile: White solid, m.p. 110–112 °C (lit.[20] 114–116 °C); yield 91% (Table 3, entry 5), 70% (Table 3, entry 10) and 81% (Table 3, entry 14); ^1H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, *J*=8.5 Hz, 2H), 7.37 (d, *J*=8.6 Hz, 2H), 7.25–7.18 (m, 2H), 6.85 (t, *J*=7.4 Hz, 1H), 6.70 (d, *J*=8.5 Hz, 2H), 5.35 (s, 1H), 3.97 (s, 1H).

2-(4-Bromophenyl)-2-(phenylamino)acetonitrile: White solid, m.p. 87–88 °C (lit.[20] 87–88 °C); yield 92% (Table 3, entry 6); ^1H NMR (400 MHz, $CDCl_3$) δ 7.67–7.55 (m, 2H), 7.48 (d, *J*=8.5 Hz, 2H), 7.33–7.25 (m, 2H), 6.92 (t, *J*=7.4 Hz, 1H), 6.76 (d, *J*=7.9 Hz, 2H), 5.40 (s, 1H), 4.06 (s, 1H).

2-(3-Nitrophenyl)-2-(phenylamino)acetonitrile: Light yellow solid, m.p. 89–90 °C (lit.[20] 86–88 °C); yield 93% (Table 3, entry 7); ^1H NMR (400 MHz, $CDCl_3$) δ 8.42 (s, 1H), 8.22 (d, *J*=8.2 Hz, 1H), 7.90 (d, *J*=7.7 Hz, 1H), 7.59 (t, *J*=8.0 Hz, 1H), 7.19 (dd, *J*=14.6, 6.9 Hz, 2H), 6.86 (t, *J*=7.4 Hz, 1H), 6.69 (d, *J*=8.0 Hz, 2H), 5.49 (s, 1H), 4.09 (s, 1H).

2-(3-Bromophenyl)-2-(phenylamino)acetonitrile[22]: White solid, m.p. 73–74 °C; yield 67% (Table 3, entry 11); ^1H NMR (400 MHz, $CDCl_3$) δ 7.77 (s, 1H), 7.57 (t, *J*=8.6 Hz, 2H), 7.37–7.25 (m, 3H), 6.93 (t, *J*=7.3 Hz, 1H), 6.77 (d, *J*=8.3 Hz, 2H), 5.42 (s, 1H), 4.08 (s, 1H).

2-Phenyl-2-(phenylamino)propanenitrile: White solid, m.p. 140–141 °C (lit.[20] 140–142 °C); yield 43% (Table 3, entry 15); ^1H NMR (400 MHz, $CDCl_3$) δ 7.57 (dd, *J*=5.7, 3.8 Hz, 2H), 7.33 (dd, *J*=10.5, 4.5 Hz, 2H), 7.20 (s, 1H), 7.09–7.02 (m, 2H), 6.74 (t, *J*=7.3 Hz, 1H), 6.48 (d, *J*=8.1 Hz, 2H), 4.24 (s, 1H), 1.89 (s, 3H).

2-Phenyl-2-(p-tolylamino)acetonitrile: White solid, m.p. 102–103 °C (lit.[20] 104–106 °C); yield 91% (Table 3, entry 16); ^1H NMR (400 MHz, $CDCl_3$) δ 7.61 (dd, *J*=7.6, 1.9 Hz, 2H), 7.47 (m, 3H), 7.09 (d, *J*=8.2 Hz, 2H), 6.71 (d, *J*=8.5 Hz, 2H), 5.41 (s, 1H), 3.93 (s, 1H), 2.29 (s, 3H).

2-Phenyl-2-(m-tolylamino)acetonitrile: White solid, m.p. 94–95 °C (lit.[23] 94–96 °C); yield 81% (Table 3, entry 17); ^1H NMR (400 MHz, $CDCl_3$) δ 7.59 (dd, *J*=7.6, 1.9 Hz, 2H), 7.45 (m, 3H), 7.16 (t, *J*=7.7 Hz, 1H), 6.72 (d, *J*=7.4 Hz, 1H), 6.59 (d, *J*=8.3 Hz, 2H), 5.41 (s, 1H), 3.97 (s, 1H), 2.32 (s, 3H).

2-((4-Methoxyphenyl)amino)-2-phenylacetonitrile: White solid, m.p. 94–95 °C (lit.[24] 75–77 °C); yield 87% (Table 3, entry 18); ^1H NMR (400 MHz, $CDCl_3$) δ 7.51–7.42 (m, 1H), 7.27 (m, 4H), 7.01–6.88 (m, 1H), 6.70 (d, *J*=8.9 Hz, 1H), 6.66–6.55 (m, 2H), 5.23–5.03 (m, 1H), 3.77–3.51 (m, 4H).

2-((4-Chlorophenyl)amino)-2-phenylacetonitrile: White solid, m.p. 107–108 °C (lit.[23] 91–92 °C); yield 94% (Table 3, entry 19); ^1H NMR (400 MHz, $CDCl_3$) δ 7.54–7.41 (m, 2H), 7.31 (d, *J*=5.5 Hz, 3H), 7.16–7.04 (m, 2H), 6.55 (d, *J*=8.8 Hz, 2H), 5.24 (s, 1H), 3.91 (s, 1H).

2-((4-Nitrophenyl)amino)-2-phenylacetonitrile: White solid, m.p. 128–129 °C (lit.[25] 128–129 °C); yield 95% (Table 3, entry 20); ^1H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J*=9.1 Hz, 2H), 7.51 (dd, *J*=6.4, 3.0 Hz, 2H), 7.42 (d, *J*=4.6 Hz, 3H), 6.69 (d, *J*=9.2 Hz, 2H), 5.45 (d, *J*=7.4 Hz, 1H), 4.78 (d, *J*=7.1 Hz, 1H).

Ethyl-4-((cyano(phenyl)methyl)amino)benzoate: White solid, m.p. 109–110 °C; yield 95% (Table 3, entry 21); ^1H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J*=8.7 Hz, 2H), 7.69–7.58 (m, 2H), 7.49 (d, *J*=5.3 Hz, 3H), 6.77 (d, *J*=8.7 Hz, 2H), 5.51 (s, 1H), 4.52 (s, 1H), 4.35 (q, *J*=7.1 Hz, 2H), 1.39 (t, *J*=7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.47, 148.33, 133.25, 131.70, 129.92, 129.59, 127.34, 121.99, 117.63, 112.98, 60.66, 49.56, 14.49. HRMS (ESI) calcd for $C_{17}H_{17}N_2O_2$ [M+H$^+$]: 281.12845, found 281.12845.

2-(Dibenzylamino)-2-phenylacetonitrile: White solid, m.p. 102–103 °C (lit.[26] 96.4 °C); yield 74% (Table 3, entry 22); ^1H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J*=7.4 Hz, 2H), 7.28 (m, 11H), 7.17 (dd, *J*=11.9, 4.9 Hz, 2H), 4.82 (s, 1H), 3.80 (d, *J*=13.4 Hz, 2H), 3.33 (d, *J*=13.4 Hz, 2H).

N-Benzylaniline[27]: Light yellow liquid; yield 32% (Scheme 2); ^1H NMR (400 MHz, $CDCl_3$) δ 7.45–7.35 (m, 4H), 7.32 (d, *J*=6.9 Hz, 1H), 7.26–7.17 (m, 2H), 6.76 (t, *J*=7.3 Hz, 1H), 6.67 (d, *J*=8.2 Hz, 2H), 4.36 (s, 2H), 4.14 (br s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.21, 139.53, 129.42, 128.79, 127.68, 127.39, 117.78, 113.06, 48.51.

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