$SnCl_4/SiO_2$: An Efficient Heterogeneous Alternative for One-pot Synthesis of β-Acetamidoketones

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Enolizable ketones have been reacted in a one-pot method with aromatic aldehydes, acetyl chloride and acetonitrile at room temperature in the presence of $SnCl_4/SiO_2$ to furnish the corresponding β-acetamidoketones in improved yields. Acetylation of an aromatic hydroxyl group was observed while using 4-hydroxybenzaldehyde or vanillin and the corresponding β-acetamidoketones were isolated in an excellent yield.

Keywords: Tin chloride ($SnCl_4$); β-Acetamidoketones; One-pot synthesis; $SnCl_4/SiO_2$.

INTRODUCTION

β-Acetamidoketones are valuable building blocks for the preparation of β-amino ketones or β-amino alcohols such as antibiotic nikkomycins[1,2] and neopolyoxines.[3] Recently, Iqbal et al. have developed a new route based on condensation of aromatic aldehydes, enolizable ketones and acetonitrile in the presence of acetyl chloride and a catalytic amount of an acid such as $CoCl_2$[4] or montmorillonite K-10 clay.[6] As a result, many catalysts such as $CeCl_3.7H_2O$,[7] silica sulfuric acid,[8] $H_6P_2W_{18}O_{62}$,[9] $K_5CoW_{12}O_{40}.3H_2O$,[10] ZnO,[11] sulfated zirconia,[12] $FeCl_3$,[13] some heteropoly acids,[14,15] silica supported $H_3PW_{12}O_{40}$,[16] nano ZnO,[17] sulfamic acid,[18] $Sc(OTf)_3$,[19] $SnCl_2.2H_2O$[20] and Nafion-H[21] have been applied in this one-pot reaction. Many authors have erroneously reported that β-acetamidoketones could be prepared by the Dakin-West reaction,[7-16] but, as we clearly know, in the Dakin-West reaction, α-acetamidoketones were prepared.[22]

Stannic chloride or tin chloride ($SnCl_4$), a thin, colorless and fuming liquid, is used in industry and in organic synthesis. Since stannic chloride is a liquid with a high specific gravity that fumes in air and reacts with the moisture in the air to form solid hydrates and HCl, the handling and the usability of $SnCl_4$ as a liquid form is laborious, and the supported form is indeed preferable. It has been claimed that the supported $SnCl_4$ is a solid superacid. If the complex $SnCl_4$ in MeOH is used to prepare a silica-supported $SnCl_4$ catalyst, Brönsted surface sites will be obtained and probably the silica supported $SnCl_4$ will have a surface species such as $Si\text{-}OSnCl_3$ or the ion pair $Si\text{-}OSnCl_4^-H^+$. Tin tetrachloride ($SnCl_4$) is used extensively in organic syntheses as a Lewis acid for enhancing a variety of organic reactions such as Friedel-Crafts alkylation,[23] the Mukaiyama-Michael reaction,[24] the Diels-Alder reaction,[25] the ene reaction,[26] the arotamization of enamines,[27] the rearrangement of 2,3-epoxy alcohol derivatives,[28] the dimerization of 1-naphthols to 2,2′-binaphthoquinones,[29] the allylation of α-benzyloxyaldehydes[30] and the formation of 1,2-dioxolanes,[31] among others. Because of the importance of solid acids in organic synthesis,[32-37] herein, we report an efficient and simple procedure for synthesis of β-acetamidoketones using $SnCl_4/SiO_2$ as catalyst.

RESULTS AND DISCUSSION

In order to utilize $SnCl_4$ as a catalyst in the synthesis of β-acetamidoketones, the reaction of 2-chlorobenzaldehyde and 4-chloroacetophenone was examined in the presence of several catalysts and under various conditions (Table 1). According to the obtained data, the 70% $SnCl_4/SiO_2$ (10 mol%) in the presence of acetylchloride and acetonitrile was the best system for β-acetamidoketone formation (Table 1, entry 9). Therefore, various aromatic aldehydes and ketones were transformed into the corresponding β-acetamidoketones in the presence of 70% $SnCl_4/SiO_2$ as a catalyst without the formation of any side products and with improved yields (Table 2).

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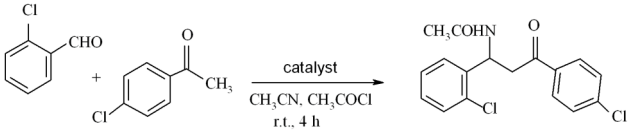
Table 1. Standardisation of reaction conditions for one-pot formation of β-acetamidoketone from 2-chlorobenzaldehyde (1 mmol) and 4-chloroacetophenone (1 mmol)



Entry	Acetyl chloride (mL)	Aceto-nitrile (mL)	Catalyst (mmol)	Yield (%)[a]
1	1	1	$FeCl_3$ (10 mol%)	46
2	1	1	$AlCl_3$ (10 mol%)	55
3	1	1	$ZnCl_2$ (10 mol%)	60
4	1	1	$ZrCl_4$ (10 mol%)	62
5	1	1	$SnCl_4$ (10 mol%)	80
6	1	1	$BF_3{\cdot}Et_2O$ (10 mol%)	63
7	0.3	0.3	70% $SnCl_4/SiO_2$ (10 mol%)	74
8	0.3	0.7	70% $SnCl_4/SiO_2$ (10 mol%)	81
9	0.3	1	70% $SnCl_4/SiO_2$ (10 mol%)	89
10	1	0.3	70% $SnCl_4/SiO_2$ (10 mol%)	68
11	0.3	1	70% $SnCl_4/Al_2O_3$ (10 mol%)	76
12	0.3	1	70% $SnCl_4/TiO_2$ (10 mol%)	72
13	0.3	1	70% $SnCl_4/ZrO_2$ (10 mol%)	45
14	0.3	1	60% $SnCl_4/SiO_2$ (10 mol%)	80
15	0.3	1	50% $SnCl_4/SiO_2$ (10 mol%)	75
16	0.3	1	80% $SnCl_4/SiO_2$ (10 mol%)	90
17	0.3	1	70% $SnCl_4/SiO_2$ (8 mol%)	82
18	0.3	1	70% $SnCl_4/SiO_2$ (5 mol%)	74
19	0.3	1	70% $SnCl_4/SiO_2$ (14 mol%)	91
20	0.3	1	70% $SnCl_4/SiO_2$ (18 mol%)	89

[a] Isolated yield

Consequently, acetylation of an aromatic hydroxyl group was observed while using 4-hydroxybenzaldehyde or vanillin, and the corresponding β-acetamidoketones were isolated in an excellent yield. 4-Dimethylamino benzaldehyde, however, was inert to the present reaction conditions. The preparative efficacy of this one-pot synthesis was further checked by scaling-up (5 fold) of the reaction of benzaldehyde with acetophenone and other ingredients which proceeded with an 80% yield. Previously, four types of mechanisms for the Iqbal procedure for β-acetamidoketone formation were proposed.[6,7,9,11,17,18] In some cases,[7,11,17] β-acetoxyketone was offered as an intermediate product that converted into β-acetamidoketone with acetonitrile. In our investigation, when the reaction was not subjected to acetonitrile, no β-acetoxyketone was formed and only a crossed aldol condensation reaction occurred. Meanwhile, in the preparation of β-acetamidoketones, no β-acetoxyketones were obtained as a by-product; also, a mixture of chalcone, acetyl chloride and acetonitrile in the presence of a catalyst failed to generate any β-acetamidoketones. In the absence of acetyl chloride or benzoyl chloride, the reaction failed to provide the desired product, obviously indicating that they play a necessary role in this reaction, although they are not involved in the final product. When benzylcyanide or phenylcyanide was used instead of acetonitrile, the corresponding β-amidoketones such as β-phenylacetamidoketone or β-benzamidoketone were obtained. Note that neither a mixture of 4-methylbenzaldehyde, 4-nitroacetophenone, aceticanhydride and acetonitrile in the presence of $SnCl_4/SiO_2$, nor a mixture of 4-methylbenzaldehyde acylal, 4-nitroacetophenone and $SnCl_4/SiO_2$ in acetonitrile could produce any of the corresponding β-acetamido ketones (Scheme I).

The proposed mechanism for the formation of $SnCl_4/SiO_2$ catalyzed β-acetamidoketone is illustrated in Scheme II. Condensation of acid chloride and aldehyde in the presence of the acidic catalyst (A^+) produces an intermediate (I) which then reacts with nitrile to produce (II) after the elimination of A^+ and Cl^-. Intermediate (II) rearranges into the cyclic intermediate (III). Next, the enol form of ketone attacks (III) to form (IV) and then (V) with an exchange of H^+. The nucleophilic attack of (V) accompanied by tautomerization gives the desired β-acetamidoketone.

CONCLUSION

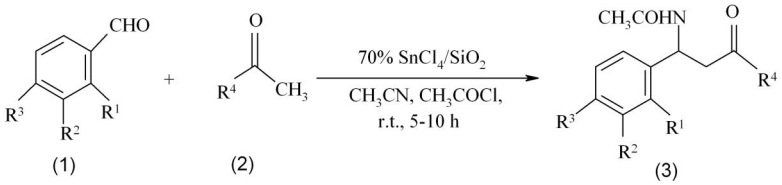
In conclusion, a significantly important and new catalytic activity of $SnCl_4/SiO_2$ has been studied for the synthesis of β-acetamido ketones in excellent yields. A mild reaction condition, a simple experimental procedure, an easy work-up and scale up and also improved yields of products are some strong features of this reported method.

EXPERIMENTAL

General

$SnCl_4$, aldehydes, ketones and other necessary chemical compounds were purchased from Fluka and Merck companies. Some products were known and were characterized by IR and ^{1}H-NMR and by comparing their physical properties with those reported in the literature. IR spectras were run on a Shimadzu IR-470 spectrometer. ^{1}H-NMR was obtained using a Bruker Avance 400 and 300 MHz spectrometer and mass spectras was obtained on a Fifons (TRIO 10000) spectrometer. Melting points were deter-

Table 2. One-pot condensation of aldehydes, ketones, acetyl chloride and acetonitrile to give the corresponding β-acetamidoketones catalyzed by 70% $SnCl_4/SiO_2$[a]



Entry	Product (3)	Time (h)	Reference	Yield[b] (%)	m.p (°C)
1	R_1,R_2,R_3=H, R_4=Ph	7.5	9	82	104-105
2	R_1,R_2,R_3=H, R_4=4-NO_2-C_6H_4	7	12	89	97-98
3	R_1=Cl, R_2,R_3=H, R_4=Ph	9	8	79	135-136
4	R_1=H, R_2=OCH_3, R_3=$OCOCH_3$, R_4=Ph	10	-	80	89-91
5	R_1,R_2=H, R_3=CH_3, R_4=4-NO_2-C_6H_4	5.5	12	80	84-85
6	R_2,R_3=H, R_1=OCH_3, R_4=4-NO_2-C_6H_4	6	-	78	145-146
7	R_1,R_3=H, R_2=NO_2, R_4=4-NO_2-C_6H_4	4	12	79	105-106
8	R_1,R_3=H, R_2=NO_2, R_4=4-Cl -C_6H_4	9	7	79	145-146
9	R_3,R_2=H, R_1=Cl, R_4=3-OCH_3-C_6H_4	9.5	-	84	101-103
10	R_2,R_1=H, R_3=Cl, R_4=Ph	8	9	85	148-149
11	R_2,R_3=H, R_1=Cl, R_4=4-Cl-C_6H_4	9	-	92	168-169
12	R_1,R_3=H, R_2=Cl, R_4=4-NO_2-C_6H_4	5	-	85	171-172
13	R_2,R_3=H, R_1=Cl, R_4=4-Br-C_6H_4	8	-	83	192-193
14	R_1,R_2=H, R_3=OCH_3, R_4=Ph	10	9	79	110-111

[a] Molar ratio of aldehyde (mmol):ketone (mmol):acetyl chloride (mL):acetonitrile (mL): 70% $SnCl_4/SiO_2$ (g) [mmol] equal to 1:1:0.3:1:0.04 [0.1]

[b] Isolated yield.

mined with a Barnstead Electrothermal melting point apparatus.

Preparation of silica-supported $SnCl_4$

5 mL of ethanol containig 1 mL of $SnCl_4$ and 1 g of preheated silica gel was stirred for 1 h at room temperature. The slurry was dried slowly on a rotary evaporator at 40 °C. The obtained solid (70% $SnCl_4/SiO_2$) was dried at an ambient temperature for 2 h and then stored in a dry container.

General procedure for one-pot synthesis of β-acetamidoketones using 70% $SnCl_4/SiO_2$

Enolizable ketone (1 mmol), aromatic aldehyde (1 mmol), acetyl chloride (0.3 mL), acetonitrile (1 mL) and 70% $SnCl_4/SiO_2$ (0.04 g, 0.1 mmol of $SnCl_4$) were placed in a round bottom flask. The materials were mixed at an ambient temperature. The progression of the reaction was followed by TLC (eluent:*n*-hexane:ethylacetate). After the completion of reaction, the mixture was poured into 30 mL ice water. The oily solid was isolated and washed with diethyl ether to remove any residual starting materials. The pure product was obtained from ethanol and water by crystallization or by preparative thin layer chromatography (eluent:*n*-hexane:ethylacetate).

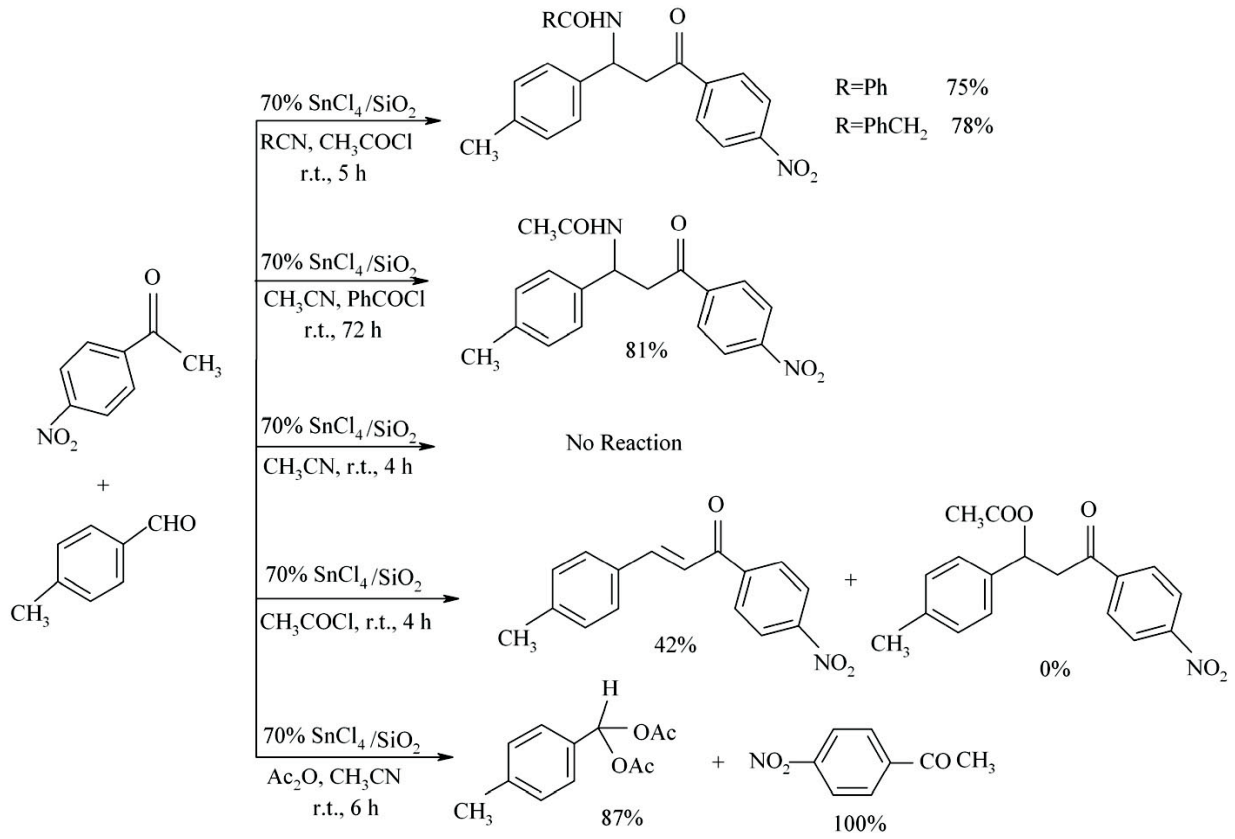
Data for β-acetamido-β-(3-methoxy, 4-acetoxy-phenyl) propiophenone (Table 2, entry 4)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.1 (s, 3H), 2.3 (s, 3H), 3.5 (dd, *J* = 6 and 16.8 Hz, 1H), 3.7 (dd, *J* = 5.6 and 16.8 Hz, 1H), 3.8 (s, 3H), 5.6 (dd, *J* = 6 and 5.6 Hz, 1H), 6.9 (d, *J* = 8 Hz, 1H), 7.0 (d, *J* = 8 Hz, 1H), 7.1 (s, 1H), 7.5 (t, *J* = 7.6 Hz, 2H), 7.6 (t, *J* = 7.6 Hz, 1H), 8 (d, *J* = 7.6 Hz, 2H), 8.6 (sbr, 1H). ^{13}C-NMR ($CDCl_3$, 100 MHz): δ 21.6, 24.38, 44.35, 51.09, 57.02, 112.6, 119.32, 123.95, 129.31, 129.91, 134.82, 137.57, 140.09, 141.09, 152.19, 170, 172, 200. IR (KBr, cm^{-1}) 3279, 3135, 2930, 1767, 1684, 1651, 1508, 1414, 1367, 1302, 1195, 1121, 1025.

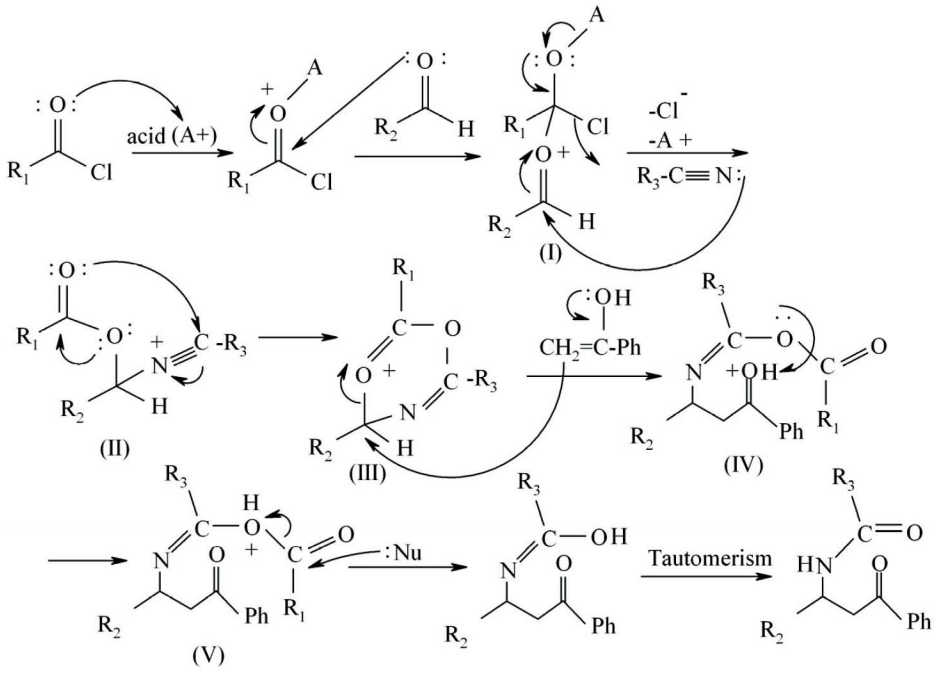
Data for β-acetamido-β-(2-methoxy)-4-nitro propiophenone (Table 2, entry 6)

^{1}H NMR ($CDCl_3$, 300 MHz): δ 2 (s, 3H), 3.55 (dd, *J* = 6.9 and 17.5 Hz, 1H), 3.6 (dd, *J* = 6.5 and 17.5 Hz, 1H), 3.9 (s, 3H), 5.7 (dd, *J* = 6.9 and 16 Hz, 1H), 6.7 (d, *J* = 6.9 Hz, 1H), 6.9 (m, 2H), 8.1 (m, 3H), 8.3 (m, 3H); IR (KBr, cm^{-1}) 3260, 1684, 1637, 1545, 1510, 1337, 1234, 837, 742; MS

Scheme I Various reactions as evidence for verification of the proposed mechanisms



Scheme II The proposed mechanism for the formation of $SnCl_4/SiO_2$ catalyzed β-acetamidoketone



(*m/z*, %) 344 ($M+2^+$, 1.87), 342 (M^+, 10.65), 299 (100), 150 (85), 107 (25), 77 (17.8), 43 (27.6, CH_3-C≡O^+).

Data for β-acetamido-β-(2-chloro)-3-methoxy propiophenone (Table 2, entry 9)

^{1}H NMR ($CDCl_3$, 300 MHz): δ 2 (s, 3H), 3.45 (dd, *J* = 17 and 5.7 Hz, 1H), 3.77 (dd, *J* = 17 and 6 Hz, 1H), 3.84 (s, 3H), 5.82 (dd, *J* = 15 and 6 Hz, 1H), 6.93 (dbr, *J* = 6.7 Hz, 1H), 6.9-7.5 (m, 8H). IR (KBr, cm^{-1}) 3265, 1681, 1643, 1547, 1284, 1002, 747; MS (*m/z*, %) 332 (M^+, 3.75), 296 (83), 135 (100), 107 (49), 77 (94), 43 (73, CH_3-C≡O^+).

Data for β-acetamido-β-(2-chloro)-4-chloro propiophenone (Table 2, entry 11)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.1 (s, 3H), 3.5 (dd, *J* = 16.8 and 5.6 Hz, 1H), 3.8 (dd, *J* = 16.8 and 6 Hz, 1H), 5.8 (sbr, 1H), 7.1 (sbr, 1H), 7.2 (m, 2H), 7.4 (d, *J* = 7.6, 1H), 7.6 (m, 3H), 7.9 (d, *J* = 8 Hz, 2H). ^{13}C-NMR ($CDCl_3$, 100 MHz): δ 24.27, 42.88, 49.17, 128.28, 128.73, 129.46, 130.0, 130.18, 131.09, 133.59, 135.86, 139.13, 141.27, 173, 196. IR (KBr, cm^{-1}) 3292, 1688, 1653, 1552, 1353, 1200, 814, 753. MS (*m/z*, %) 338 ($M+2^+$, 1.87), 336 (M^+, 2.5), 184 (3), 182 (10), 141 (24), 139 (100), 43 (25.6, CH_3-C≡O^+).

Data for β-acetamido-β-(3-chloro)-4-nitro propiophenone (Table 2, entry 12)

^{1}H NMR ($CDCl_3$, 300 MHz): δ 2.05 (s, 3H), 3.5 (dd, *J* = 17.1 and 6.6 Hz, 1H), 3.82 (dd, *J* = 17 and 5.1 Hz, 1H), 5.53 (m, 1H), 6.5 (dbr, *J* = 6.9 Hz, 1H), 7.24 (m, 3H), 7.34 (s, 1H), 8.07 (d, *J* = 8.7 Hz, 3H), 8.32 (d, *J* = 8.7 Hz, 2H). IR (KBr, cm^{-1}) 3235, 1687, 1641, 1546, 1530, 1316, 1156, 843, 688; MS (*m/z*, %) 346 (M^+, 0.91), 311 (59), 150 (100), 77 (26), 43 (53, CH_3-C≡O^+).

Data for β-acetamido-β-(2-chloro)-4-bromo propiophenone (Table 2, entry 13)

^{1}H NMR ($CDCl_3$, 300 MHz): δ 2.04 (s, 3H), 3.43 (dd, *J* = 18 and 6 Hz, 1H), 3.73 (dd, *J* = 18 and 5.7 Hz, 1H), 5.81 (m, 1H), 6.88 (d, *J* = 7.8 Hz, 1H), 7.1 (t, *J* = 5.7 Hz, 2H), 7.2 (d, *J* = 7.2 Hz, 1H), 7.36 (d, *J* = 7.2 Hz, 3H), 7.59 (d, *J* = 8.1 Hz, 2H), 7.76 (d, *J* = 8.1 Hz, 2H). IR (KBr, cm^{-1}) 3260, 1677, 1653, 1642, 1573, 1539, 1221, 801, 744; MS (*m/z*, %) 382 ($M+2^+$, 2.93), 380 (M^+, 2.18), 346 (64), 344 (58), 183 (73), 43 (31, CH_3-C≡O^+).

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