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Acylative cleavage of aziridines with acid anhydrides catalyzed by Scandium triflate

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Abstract

Aziridines smoothly react with acid anhydrides in the presence of a catalytic amount of scandium triflate under mild reaction conditions to afford the corresponding β-aminoacetates, benzoates and propionates in high yields with high regioselectivity.

Keywords: Scandium reagents; Aziridines; Acid anhydrides; β-Aminoesters

Aziridines are well known carbon electrophiles, capable of reacting with various nucleophiles and their ability to undergo regioselective ring opening reactions contributes largely to their synthetic value [1]. They are very useful intermediates for the synthesis of many biologically interesting molecules such as amino acids [2], heterocycles [3] and alkaloids [4]. In consequence, several procedures have been reported for the regioselective ring opening of aziridines with various nucleophiles such as organometallic reagents [5], silyl nucleophiles [6], Wittig reagents [7], amines [8], halides [9], hydroxyl compounds [10] and alkenes [11] to produce ring-opened products. However, there are no reports on the regioselective ring opening of aziridines with acid anhydrides. To the best of our knowledge, this is the first report on the regioselective ring opening of aziridines with acid anhydrides. Lanthanide triflates are unique Lewis acids that are currently of great research interest [12]. Particularly, scandium salts are attractive because they are quite stable to water and reusable, and in addition, they are highly effective for the activation of nitrogen containing compounds. Therefore, scandium salts are efficient catalysts compared to traditional Lewis acids in several carbon–carbon bond-forming reactions and have found a widespread applications in organic synthesis [13].

In this report, we wish to describe our results on the regioselective ring opening of aziridines with acid anhydrides using a catalytic amount of scandium triflate. The treatment of styrene-*N*-tosyl aziridine with acetic anhydride in the presence of 5 mol% $Sc(OTf)_3$ at ambient temperature resulted in the formation of β-amino acetate derivative **2** in 85% yield (Scheme 1).

In a similar fashion, aryl-*N*-tosyl aziridines reacted smoothly with acid anhydrides to afford the corresponding β-amino acetates, benzoates and propionates in high yields. Aryl-*N*-tosyl aziridines underwent cleavage by an acid anhydride with preferential attack at benzylic position resulted in the formation of product **2** with a trace amount of **3** (entries g–m). However, the treatment of alkyl-*N*-tosyl aziridines with acid anhydrides gave predominantly the ring-opened product **3** with a minor amount of **2** (entries n–p). The ratios of products **2** and **3** were determined from the ^{1}H-NMR spectrum of the crude product. Alkyl-*N*-tosyl aziridines gave the ring-opened products resulting from terminal attack as well as internal attack of anhydrides as has been observed by others in most of the aziridine ring opening reactions [6]. A variety of aziridines reacted well with anhydrides to give the respective β-amino acetates, benzoates and propionates. In all cases, the reactions proceeded efficiently in high yields at ambient temperature. Furthermore, the treatment of cycloalkyl-*N*-tosyl aziridines with acid anhydrides afforded the corresponding β-amino esters in high yields (Scheme 2).

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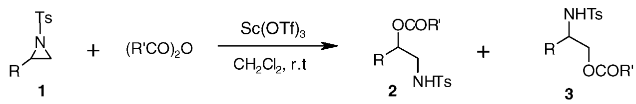
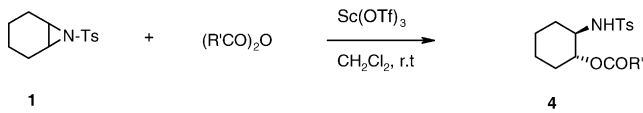
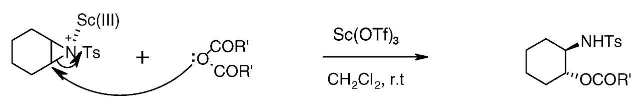


Scheme 1.



Scheme 2.



Scheme 3.

In the case of cycloalkyl aziridines, the stereochemistry of the ring product **4e** was found to be *trans* from the coupling constants of the ring protons at $\delta = 3.40$ ppm (ddd, $J = 5.0$, 9.0 and 9.0 Hz, 1H) for (NCH) in ^{1}H-NMR spectrum likewise the peak at $\delta = 4.80$ ppm for (CHOCOR) showed the similar kind of splitting pattern (ddd, $J = 5.0$, 9.0 and 10.0 Hz, 1H). The method is clean and highly regioselective, affording β-amino esters in excellent yields. Similar results were also obtained with 5 mol% $Bi(OTf)_3$. All the products are fully characterized by ^{1}H-NMR, IR, ^{13}C-NMR and mass spectroscopic data. However, in the absence of catalyst, the reaction did not yield any product even at long reaction time. The reaction seems to proceed through the activation of aziridine by a scandium triflate followed by the attack of acetate group resulting in the formation of β-amino-acetate (Scheme 3).

Finally, the catalyst was recovered on work-up from aqueous layer and recycled in subsequent reactions with gradual decrease in activity; for example, styrene-*N*-tosyl aziridine and acetic anhydride gave 85%, 80% and 78% yields over three cycles. The scope and generality of this process is illustrated with respect to various aziridines and anhydrides and the results are presented in Table 1.

In conclusion, we have described a novel and efficient method for the preparation of β-amino esters from aziridines and anhydrides using a catalytic amount of scandium triflate. The notable features of this method are high conversions, short reaction times, mild reaction conditions, greater regioselectivity, cleaner reaction profiles, simplicity in operation and reusability of the catalyst, which makes it a useful and attractive process for the synthesis of β-amino acetates, benzoates and propionates.

1. Experimental

IR spectra were recorded on a Perkin-Elmer FT-IR 240-c spectrophotometer using KBr optics. ^{1}H- and ^{13}C-NMR spectra were recorded on Gemini-200 spectrometer in $CDCl_3$ using TMS as internal standard. Mass spectra were recorded on a Finning MAT 1020 mass spectrometer operating at 70 eV.

1.1. Experimental procedure

A mixture of *N*-tosyl aziridine (5 mmol), acid anhydride (10 mmol) and scandium triflate or bismuth triflate (5 mol%) in dichloromethane (10 mL) was stirred at ambient temperature for an appropriate time (Table 1). After completion of the reaction, as indicated by TLC, the reaction mixture was diluted with water and extracted with dichloromethane (2×10 mL). The combined organic layers were dried over anhydrous Na_2SO_4, concentrated in vacuo and the resulting product was purified by column chromatography on silica gel (Merck, 100–200 mesh, ethyl acetate-hexane, 1:9) to afford pure β-amino ester. Spectral data for products:

1.2. ***4a****: 2-(4-methylphenylsulfonamido)cyclohexyl acetate*

IR (KBr): υ 3255, 2943, 2865, 1714, 1598, 1494, 1452, 1374, 1335, 1266, 1160, 1091, 1040, 970, 896, 851, 814, 665 cm^{-1}. ^{1}H-NMR($CDCl_3$, 200 MHz): δ 1.20–1.40 (m, 4H), 1.60–1.78 (m, 2H), 1.82 (s, 3H), 1.90–2.10 (m, 2H), 2.42 (s, 3H), 3.12–3.22 (m, 1H), 4.50–4.62 (m, 1H), 5.08 (d, 1H, $J = 6.9$ Hz), 7.25 (d, 2H, $J = 8.0$ Hz), 7.78 (d, 2H, $J = 8.0$ Hz). EIMS: *m/z* (%): 311 (M^+, 10), 252 (12), 188 (16), 156 (23), 96 (29), 91 (50), 40 (100).

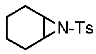
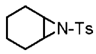
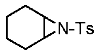
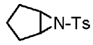
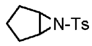
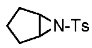
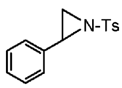
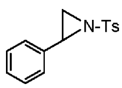
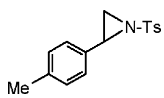
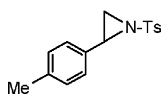
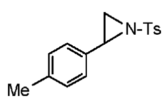
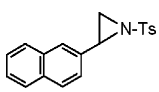
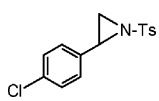
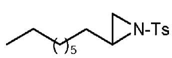
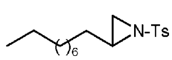
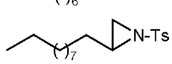
1.3. ***4b****: 2-(4-methylphenylsulfonamido)-1-phenylcarbonyloxycyclohexane*

IR (KBr): υ 3328, 2925, 2861, 1706, 1597, 1450, 1371, 1323, 1266, 1156, 1111, 1081, 1015, 914, 816, 714 cm^{-1}.^{1}H-NMR ($CDCl_3$, 200 MHz): δ 1.30–1.50 (m, 5H), 1.70–1.80 (m, 2H), 2.00–2.10 (m, 1H), 2.22 (s, 3H), 3.20–3.40 (m, 1H), 4.78–4.84 (m, 1H), 5.10 (d, 1H, $J = 6.8$ Hz), 6.90 (d, 2H, $J = 8.1$ Hz), 7.30–7.40 (m, 2H), 7.48–7.61 (m, 3H), 7.80 (d, 2H, $J = 8.1$ Hz). EIMS: *m/z* (%): 373 (M^+, 23), 281 (20), 267 (15), 252 (56), 229 (11), 221 (100), 207 (50), 191 (18), 165 (16), 159 (26).

1.4. ***4c****: 2-(4-methylphenylsulfonamido)cyclohexyl propionate*

IR (KBr): υ 3251, 2964, 2849, 1711, 1592, 1498, 1447, 1365, 1322, 1261, 1167, 1089, 1045, 967, 890, 843, 708, 664 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 200 MHz): δ 1.05 (t, 3H, $J = 6.5$ Hz), 1.48–1.68 (m, 2H), 1.72–1.82 (m, 4H), 1.94–2.24 (m, 4H), 2.40 (s, 3H), 3.10–3.30 (m, 1H), 4.50–4.70 (m, 1H), 5.24 (brs, NH, 1H), 7.15 (d, 2H, $J = 8.0$ Hz), 7.70 (d, 2H, $J = 8.0$ Hz). EIMS: *m/z* (%): 325 (M^+, 21), 252 (16), 170 (100), 155 (68), 97 (34), 82 (41), 55 (31).

Table 1
$Sc(OTf)_3$-catalyzed cleavage of activated aziridines with acid anhydrides

Entry	Aziridine	Anhydride	Reaction time (h)	Yield (%)[a]	Ratio[b] 2:3
a		Ac_2O	4.5	85	–
b		Bz_2O	7.0	80	–
c		$(CH_3CH_2CO)_2O$	6.0	81	–
d		$(CH_3CH_2CO)_2O$	6.5	83	–
e		Ac_2O	5.5	87	–
f		Bz_2O	6.0	78	–
g		Ac_2O	3.0	87[c]	95:5
h		Bz_2O	4.5	81	90:10
I		Ac_2O	3.0	85[c]	94:6
j		Bz_2O	5.5	80	85:15
k		$(CH_3CH_2CO)_2O$	3.5	83	92:8
l		Ac_2O	6.0	78[c]	93:7
m		Bz_2O	4.0	85[c]	95:5
n		$(CH_3CH_2CO)_2O$	6.5	80	10:90
o		Ac_2O	6.0	75[c]	12:88
p		$(CH_3CH_2CO)_2O$	6.5	82	14:86

[a] Isolated and unoptimized yield.
[b] Ratio of products from internal attack vs. terminal attack.
[c] 7–10% Diamide derivative was also obtained.

1.5. **4d**: 2-(4-methylphenylsulfonamido)cyclopentyl propionate

IR (KBr): υ 3439, 3408, 2973, 2898, 1718, 1604, 1547, 1483, 1436, 1381, 1323, 1271, 1168, 1089, 1042, 967, 890, 843, 759 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 200 MHz): δ 0.90 (t, 3H, $J = 6.6$ Hz), 1.18–1.41 (m, 4H), 1.84–1.95 (m, 4H), 2.40 (s, 3H), 3.40–3.50 (m, 1H), 4.80–4.90 (m, 1H), 5.66 (d, 1H, $J = 6.8$ Hz), 7.24 (d, 2H, $J = 8.1$ Hz), 7.70 (d, 2H, $J = 8.1$ Hz). EIMS: m/z (%): 311 (M^+, 16), 238 (10), 156 (100), 141 (31), 83 (62), 68 (74), 42 (39).

1.6. **4e**: 2-(4-methylphenylsulfonamido)cyclopentyl acetate

IR (KBr): υ 3434, 3412, 2969, 2928, 1727, 1607, 1499, 1462, 1379, 1337, 1256, 1142, 1108, 1045, 1012, 951, 907, 842, 780, 741, 702 cm^{-1}. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 1.40–1.65 (m, 4H), 1.70–1.88 (m, 2H), 1.95 (s, 3H), 2.40 (s, 3H), 3.40 (ddd, 1H, $J = 5.0$, 9.5, 9.5 Hz), 4.80 (ddd, 1H, $J = 5.0$, 9.5, 10.0 Hz), 5.60 (brs, 1H, NH), 7.25 (d, 2H, $J = 8.0$ Hz), 7.78 (d, 2H, $J = 8.0$ Hz). EIMS: m/z (%): 297 (M^+, 11), 238 (10), 142 (10), 127 (10), 83 (100), 47 (62).

1.7. **4*f***: *2-(4-methylphenylsulfonamido)cyclopentyl benzoate*

IR (KBr): υ 3326, 2947, 2859, 1704, 1593, 1456, 1376, 1319, 1261, 1153, 1109, 1085, 926, 835, 756 cm^{-1}. ^{1}H-NMR ($CDCl_3$, 200 MHz): δ 1.70–1.80 (m, 4H), 2.10–2.20 (m, 2H), 2.25 (s, 3H), 3.50–3.60 (m, 1H), 5.00–5.15 (m, 1H), 5.35 (d, 1H, $J = 6.7$ Hz), 7.10 (d, 2H, $J = 8.0$ Hz), 7.38–7.58 (m, 3H), 7.60–7.70 (m, 2H), 7.85 (d, 2H, $J = 8.0$ Hz). EIMS: *m/z* (%): 359 (M^+, 10), 238 (18), 147 (11), 137 (13), 123 (16), 109 (20), 105 (15), 99 (12), 95 (38), 83 (53), 69 (75), 55 (100), 43 (79).

1.8. **2*g***: *2-(4-methylphenylsulfonamido)-1-phenylethyl acetate*

IR (KBr): υ 3543, 3286, 3032, 2928, 1744, 1599, 1495, 1430, 1373, 1328, 1227, 1158, 1094, 1048, 950, 904, 816, 768, 701, 665 cm^{-1}. ^{1}H-NMR (200 MHz, $CDCl_3$): δ 2.0 (s, 3H), 2.43 (s, 3H), 3.25 (dd, 2H, $J = 7.0$, 12.5 Hz), 5.55 (brs, 1H, NH), 5.65 (t, 1H, $J = 7.0$ Hz), 7.20–7.38 (m, 7H), 7.68 (d, 2H, $J = 8.0$ Hz). EIMS: *m/z* (%): 333 (M^+, 11), 184 (16), 178 (10), 155 (19), 141 (12), 121 (34), 119 (90), 117 (100), 106 (11), 91 (20), 84 (51), 47 (33).

1.9. **2*h***: *4-methyl-1-(2-phenyl-2-phenylcarbonyloxyethylsulfamoyl)benzene*

IR (KBr): υ 3334, 2941, 1712, 1599, 1457, 1324, 1272, 1154, 1116, 1094, 1023, 922, 813, 761, 711, 663 cm^{-1}. ^{1}H-NMR (200 MHz, $CDCl_3$): δ 2.30 (s, 3H), 2.40 (s, 3H), 3.40–3.50 (m, 2H), 4.80–4.90 (brs, 1H, NH), 5.95 (t, 1H, $J = 6.8$ Hz), 7.20 (d, 2H, $J = 8.0$ Hz), 7.30–7.40 (m, 8H), 7.70 (d, 2H, $J = 8.0$ Hz), 8.00 (d, 2H, $J = 8.0$ Hz). EIMS: *m/z* (%): 395 (M^+, 12), 391 (10), 307 (13), 289 (10), 274 (100), 184 (14), 169 (11), 155 (25), 137 (60), 119 (30).

1.10. **2*i***: *1-(4-methylphenyl)-2-(4-methylphenylsulfonamido)ethyl acetate*

IR (KBr): υ 3349, 3052, 2940, 1719, 1605, 1463, 1321, 1267, 1149, 1086, 1016, 931, 816, 759, 718, 658 cm^{-1}. ^{1}H-NMR (200 MHz, $CDCl_3$): δ 2.00 (s, 3H), 2.35 (s, 3H), 2.43 (s, 3H), 3.25 (dd, 2H, $J = 6.9$, 12.3 Hz), 5.25 (brs, 1H, NH), 5.62 (t, 1H, $J = 6.9$ Hz), 7.08 (s, 4H), 7.28 (d, 2H, $J = 8.0$ Hz), 7.70 (d, 2H, $J = 8.0$ Hz). EIMS: *m/z* (%): 347 (M^+, 18), 288 (10), 192 (36), 177 (41), 155 (30), 133 (100), 118 (27), 91 (15), 76 (29), 51 (48).

1.11. **2*j***: *4-methyl-1-[2-(4-methylphenylsulfonamido)-1-phenylcarbonyloxyethyl]benzene*

IR (KBr): υ 3340, 3069, 3037, 2920, 1715, 1600, 1435, 1325, 1277, 1154, 1118, 1084, 1020, 920, 812, 712, 660 cm^{-1}. ^{1}H-NMR (200 MHz, $CDCl_3$): δ 2.38 (s, 3H), 2.42 (s, 3H), 3.40–3.50 (m, 2H), 5.38 (brs, 1H, NH), 5.90 (t, 1H, $J = 6.8$ Hz), 7.10 (d, 2H, $J = 8.2$ Hz), 7.15–7.22 (m, 3H), 7.38–7.55 (m, 4H), 7.70 (d, 2H, $J = 8.2$ Hz), 7.98 (d, 2H, $J = 8.2$ Hz). EIMS: *m/z* (%): 409 (M^+, 10), 397 (12), 341 (10), 325 (11), 304 (10), 288 (100), 281 (15), 253 (10), 207 (20), 149 (31), 132 (21), 116 (27), 89 (42), 51 (61).

1.12. **2*k***: *1–(4-methylphenyl)-2-(4-methylphenylsulfonamido)ethyl propionate*

IR (KBr): υ 3341, 3062, 2948, 2869, 1721, 1598, 1512, 1469, 1328, 1263, 1153, 1083, 1082, 1024, 935, 821, 762, 715, 651 cm^{-1}. ^{1}H-NMR (200 MHz, $CDCl_3$): δ 1.10 (t, 3H, $J = 6.7$ Hz), 2.24 (q, 2H, $J = 6.0$ Hz), 2.35 (s, 3H), 2.40 (s, 3H), 3.30 (dd, 2H, $J = 6.9$, 12.2 Hz), 4.75 (t, 1H, $J = 6.9$ Hz), 5.65 (t, 1H, $J = 6.0$ Hz), 7.10 (s, 4H), 7.25 (d, 2H, $J = 8.0$ Hz), 7.70 (d, 2H, $J = 8.0$ Hz). EIMS: *m/z* (%): 361 (M^+, 18), 318 (10), 288 (15), 192 (21), 177 (14), 133 (100), 118 (62), 91 (22), 76 (48), 51 (67).

1.13. **2*l***: *2-(4-methylphenylsulfonamido)-1-(2-naphthyl)ethyl acetate*

IR (KBr): υ 3341, 3074, 2936, 1721, 1611, 1536, 1462, 1323, 1261, 1152, 1085, 1018, 934, 823, 765, 721 cm^{-1}. ^{1}H-NMR (200 MHz, $CDCl_3$): δ 2.00 (s, 3H), 2.38 (s, 3H), 3.20–3.40 (m, 2H), 5.70 (brs, 1H, NH), 5.80–5.90 (m, 1H), 7.18 (d, 2H, $J = 8.0$ Hz), 7.25 (d, 1H, $J = 8.0$ Hz), 7.39–7.43 (m, 2H), 7.60–7.80 (m, 6H). EIMS: *m/z* (%): 383 (M^+, 16), 339 (10), 324 (26), 281 (10), 263 (15), 251 (10), 239 (12), 229 (10), 215 (16), 191 (20), 179 (18), 169 (32), 155 (30), 147 (21), 133 (40), 119 (56), 109 (100).

1.14. **2*m***: *1-(4-chlorophenyl)-2-(4-methylphenylsulfonamido)ethyl acetate*

IR (KBr): υ 3275, 3027, 2921, 2871, 1741, 1657, 1598, 1493, 1436, 1376, 1323, 1238, 1152, 1092, 1053, 1012, 951, 887, 818, 712, 665 cm^{-1}. ^{1}H-NMR (200 MHz, $CDCl_3$): δ 2.00 (s, 3H), 2.45 (s, 3H), 3.25 (dd, 2H, $J = 6.7$, 12.4 Hz), 5.70 (t, 1H, $J = 6.7$ Hz), 5.90 (brs, 1H, NH), 7.15–7.20 (m, 4H), 7.30 (d, 2H, $J = 8.1$ Hz), 7.70 (d, 2H, $J = 8.1$ Hz). EIMS: *m/z* (%): 369 (M^{+2}, 12), 367 (10), 308 (100), 184 (15), 155 (54), 121 (14), 109 (20), 91 (42), 69 (57), 55 (88).

1.15. **3*n***: *1-(4-methylphenylsulfonamidomethyl)nonyl propionate*

IR (KBr): υ 3521, 3281, 3025, 1743, 1598, 1493, 1332, 1232, 1160, 1093, 817, 763, 665 cm^{-1}. ^{1}H-NMR (200 MHz, $CDCl_3$): δ 0.93 (t, 3H, $J = 6.5$ Hz), 1.20–1.40 (m, 13H), 1.60–1.75 (m, 2H), 1.90–2.10 (m, 4H), 2.40 (s, 3H), 3.10–3.25 (m, 2H), 4.50–4.65 (m, 1H), 5.10 (d, 1H, $J = 6.9$ Hz), 7.30 (d, 2H, $J = 8.0$ Hz), 7.78 (d, 2H, $J = 8.0$ Hz). EIMS: *m/z* (%): 383 (M^+, 12), 310 (10), 252 (12), 187 (28), 170 (30), 157 (28), 141 (10), 114 (35), 96 (60), 91 (73), 57 (100), 43 (69).

1.16. **3*o***: *1-(4-methylphenylsulfonamidomethyl)decyl acetate*

IR (KBr): υ 3556, 3278, 3030, 2947, 2861, 1741, 1599, 1495, 1428, 1332, 1240, 1161, 1095, 1043, 963, 815, 758, 666 cm^{-1}. ^{1}H-NMR (200 MHz, $CDCl_3$): δ 0.96 (t, 3H, $J = 6.7$ Hz), 1.10–1.30 (m, 14H), 1.40–1.50 (m, 2H), 2.00 (s, 3H), 2.40 (s, 3H), 2.90–3.10 (m, 2H), 4.80 (brs, 1H, NH), 5.60–5.75 (m, 1H), 7.30 (d, 2H, $J = 8.0$ Hz), 7.75 (d, 2H, $J = 8.0$ Hz). EIMS: m/z (%): 383 (M^+, 18), 282 (12), 269 (22), 211 (12), 184 (52), 156 (81), 142 (15), 126 (18), 110 (10), 91 (80), 65 (20), 57 (10), 43 (100).

1.17. **3*p***: *1-(4-methylphenylsulfonamidomethyl)undecyl propionate*

IR (KBr): υ 3293, 2925, 2837, 1738, 1641, 1600, 1456, 1378, 1330, 1160, 1095, 971, 815, 665 cm^{-1}. ^{1}H-NMR (200 MHz, $CDCl_3$): δ 0.95 (t, 3H, $J = 6.5$ Hz), 1.15–1.36 (m, 17H), 1.65–1.75 (m, 2H), 1.90–2.05 (m, 4H), 2.40 (s, 3H), 3.50 (t, 2H, $J = 6.9$ Hz), 4.55 (brs, 1H, NH), 5.20–5.30 (m, 1H), 7.24 (d, 2H, $J = 8.0$ Hz), 7.70 (d, 2H, $J = 8.0$ Hz). EIMS: m/z (%): 411 (M^+, 10), 338 (12), 256 (18), 241 (10), 225 (12), 184 (65), 156 (79), 96 (15), 91 (100), 56 (34), 43 (30).

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