Ionic Liquids-Promoted Addition of Arylsulfinic Acids to *p*-Quinones: A Green Synthesis of Diaryl Sulfones

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Abstract : Arylsulfinic acids undergo smooth conjugate addition to *p*-quinones in air- and moisture-stable second generation room temperature ionic liquid [bmim]BF_4 under mild conditions to produce the corresponding arylsulfonylhydroquinones in excellent yields with high selectivity. In this reaction, ionic liquid plays the dual role as the solvent and the catalyst. The quinones show enhanced reactivity in ionic liquid thereby reducing the reaction times and improving the yields significantly. The presence of ionic liquids helps to avoid the use of either acid or base catalysts for this conversion. The recovered ionic liquid was reused for four to five times with consistent activity.

Keywords: ionic liquids, *p*-quinones, sulfinic acids, diaryl sulfones

The conjugate addition of arylsulfinic acids to *p*-quinones to form carbon–sulfur bonds constitutes a key reaction in biosynthetic processes as well as in organic synthesis.[1] The simple and direct method for the synthesis of 2,5-dihydroxydiaryl sulfones involves the nucleophilic addition of arylsulfinic acids to quinones and is generally catalyzed by an acid or a base catalyst.[2–4] Recently, the reaction has been studied using various solvents at different pH conditions.[5] However, many of these methods often involve the use of an acid or a base catalyst which always demand aqueous work-up for the catalyst separation, recycling and disposal. Since organosulfur compounds have become increasingly useful and important in drugs and pharmaceuticals, the development of simple, convenient, and environmentally benign approaches is desirable.

In recent years, second generation air and moisture stable room temperature ionic liquids (Figure 1) such as [bmim]BF_4 and [bmim]PF_6 are being used as green solvents for the immobilization of transition metal based catalysts, Lewis acids and enzymes.[6] They are referred to as 'designer solvents' as their properties such as hydrophilicity, hydrophobicity, Lewis acidity, viscosity and density can be altered by the fine-tuning of parameters such as the choice of organic cation, inorganic anion and the length of alkyl chain attached to an organic cation. These structural variations offer flexibility to the chemist to devise the most idealized solvent, catering to the needs of any particular process.[7]

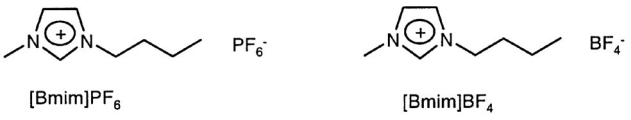

Figure 1 Chemical structure of representative ionic liquids

Their high polarity and ability to solubilize both organic and inorganic compounds can result in enhanced rates of chemical processes and can provide higher selectivities compared to conventional solvents. As a result of their green credentials and potential to enhance rates and selectivities, ionic liquids are finding increasing applications in organic synthesis.[8] Recent reports have shown that they can also promote and catalyze many transformations of commercial importance under mild conditions without the need of any additional acid catalyst.[9,10]

In this article, we describe a novel use of ionic liquids for the conjugate addition of arylsulfinic acids to *p*-quinones to produce diaryl sulfones in high yields under mild and neutral conditions (Scheme 1).

Scheme 1

Accordingly, treatment of *p*-benzoquinone with phenylsulfinic acid in [bmim]BF_4 afforded 2,5-dihydroxydiphenyl sulfone **3a** in 92% yield. The reaction went to completion in a short time (25 min). The product thus obtained was isolated by simple extraction with diethyl ether. The ionic liquid was further washed with diethyl ether and reused several times without further purification. Encouraged by the results obtained with *p*-benzoquinone, we turned our attention to various substituted quinones and sulfinic acids. Interestingly, numerous quinones such as 1,4-benzoquinone and its methoxy, ethoxy, methyl, dimethyl analogues and 1,4-naphthoquinone afforded quite effectively Michael adducts in high yields (Table 1, entries **a**–**n**). In case of mono-substituted quinones such as toluquinone (2-methyl-1,4-benzoquinone) and 2-methoxy-1,4-benzoquinone, the addition occurs at the 5- and 6-positions with the former predomi-

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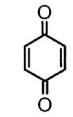
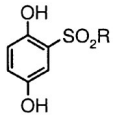
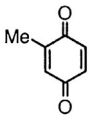
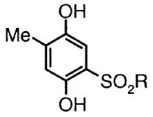
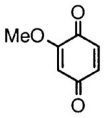
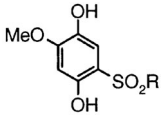
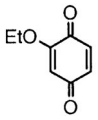
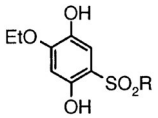
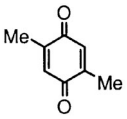
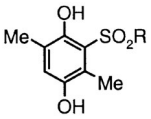
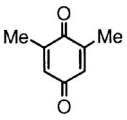
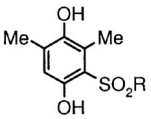
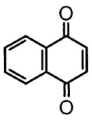
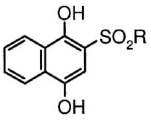
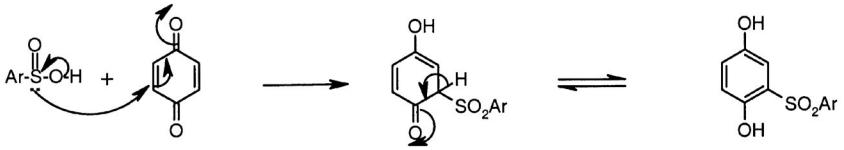
nating by 9:1 (Table 1, entries **c**–**h**). Like benzoquinone, 1,4-naphthoquinone also afforded 2-phenylsulfonylnaphthalene-1,4-diol under identical conditions (Table 1, entries **m**,**n**). In all cases, the reactions proceeded efficiently at room temperature in [bmim]BF_4 ionic liquid without the need of any additional acid catalyst. The probable mechanism seems to be addition of sulfinic acid to the *p*-quinone, which is activated by ionic liquid. The initially formed addition product may undergo tautomerization to give the desired hydroquinone as shown in Scheme 2.

The reaction was studied in both hydrophilic [bmim]BF_4 and hydrophobic [bmim]PF_6 ionic liquids and [bmim]BF_4 was found to give the best results. Comparatively low conversions (20–50%) were obtained when [bmim]PF_6 was used as a solvent. Furthermore, the use of organic solvents such as acetonitrile and tetrahydrofuran also gave poor yields (30–65%) even after long reaction times (approximately 8.0–12 h). The quinones exhibit enhanced reactivity in the ionic liquid thereby reducing the reaction times and improving the yields significantly. For instance,

Table 1 Addition of Arylsulfinic Acids to *p*-Quinones Using [bmim]BF_4 Ionic Liquid

Entry	Quinone **1**	Product[a] **3**	R	Reaction Time (min)	Yield (%)[b]
a			Ph	25	92
b			4-MeC_6H_4	30	95
c			Ph	35	85 (10)[c]
d			4-MeC_6H_4	30	87 (6)[c]
e			Ph	40	83 (8)[c]
f			4-MeC_6H_4	50	86 (7)[c]
g			Ph	35	85 (9)[c]
h			4-MeC_6H_4	40	89 (5)[c]
i			Ph	30	92
j			4-MeC_6H_4	25	90
k			Ph	35	93
l			4-MeC_6H_4	30	91
m			Ph	25	88
n			4-MeC_6H_4	20	92

[a] All products were characterized by ^{1}H NMR, IR and mass spectroscopy.
[b] Isolated and unoptimized yields.
[c] Yield indicated in parenthesis refers to the other regioisomeric product observed in the ^{1}H NMR spectrum.



Scheme 2

treatment of 2-methylbenzoquinone with phenylsulfinic acid in [bmim]BF_4 afforded the product as a mixture of 2-methyl-5-phenylsulfonylhydroquinone (**3c**) and 2-methyl-6-phenylsulfonylhydroquinone (**3c′**) in 85 and 10% yields respectively whereas the same reaction in acetonitrile gave the products **3c′** and **3c** in 65% overall yield in the ratio of 2:1. In addition, the ionic liquid was easily recovered after the reaction and reused several times without loss of activity; even after the fourth cycle the product **3a** was obtained with similar yield and purity to that obtained in the first cycle. The yields were generally high to quantitative and the reaction was complete in a few minutes. The scope and generality of this method is illustrated with respect to various substituted quinones and sulfinic acids and the results are presented in Table 1. The use of ionic liquids as the reaction media for this transformation helps to avoid the use of moisture-sensitive reagents or corrosive acids as promoters, thereby minimizing the production of acid waste during workup.

In summary, we have described a simple, convenient and efficient protocol for the conjugate addition of arylsulfinic acids to *p*-quinones to produce diaryl sulfones using ionic liquids as recyclable solvents. The ionic liquid plays a dual role as reaction solvent and promoter. The *p*-quinones exhibit enhanced reactivity in ionic liquids, thereby reducing the reaction times and improving the yield significantly. The simple experimental procedure, combined with ease of recovery and reuse of this novel reaction media, is expected to contribute to the development of a green strategy for the synthesis of 2,5-dihydroxydiaryl sulfones.

Melting points were recorded on Buchi R-535 apparatus and are uncorrected. IR spectra were recorded on a Perkin-Elmer FT-IR 240-c spectrophotometer using KBr optics. ^{1}H NMR spectra were recorded on Gemini-200 spectrometer in $CDCl_3$ using Me_4Si as internal standard. Mass spectra were recorded on a Finnigan MAT 1020 mass spectrometer operating at 70 eV. TLC was monitored on 0.25 mm precoated silica gel plates (60F-254). The commercially available ionic liquids were purchased from Fluka Chemical Company and used as received without further purification. The purity of [bmim]BF_4 ionic liquid is 97% (NMR). All the solvents were distilled, dried, and stored under N_2 prior to use.

Sulfonation of Quinones; General Procedure

A mixture of the *p*-quinone (1 mmol) and arylsulfinic acid (1 mmol) in 1-butyl-3-methylimidazolium tetrafluoroborate (3 mL) was stirred at r.t. for the appropriate time (Table 1). After completion of the reaction, as indicated by TLC, the reaction mixture was washed with Et_2O (3 × 10 mL). The combined Et_2O extracts were concentrated in vacuo and the resulting product was directly charged on small silica gel column and eluted with a mixture of EtOAc–*n*-hexane to afford the pure diaryl sulfone. The products thus obtained were characterized by comparison of their NMR, IR, Mass, TLC, mixed TLC analysis and physical data with authentic samples. The spectral data of all the products were identical with those of authentic samples.[5]

2-Phenylsulfonyl-1,4-hydroquinone (3a)

Solid;[3a] mp 193 °C.

IR (KBr): 3286, 2362, 1597, 1507, 1461, 1365, 1288, 1137, 1084, 818, 729 cm^{-1}.

^{1}H NMR (200 MHz, $CDCl_3$): δ = 6.70 (d, 1 H, *J* = 8.1 Hz), 6.85 (dd, 1 H, *J* = 1.9, 8.1 Hz), 7.19–7.20 (d, 1 H, *J* = 1.9 Hz), 7.40–7.55 (m, 3 H), 7.90 (d, 2 H, *J* = 8.1 Hz), 8.85 (br s, 1 H, OH), 9.0 (br s, 1 H, OH).

MS (EI): *m*/*z* (%) = 250 (M^+, 100), 185 (10), 160 (30), 151 (30), 108 (95), 77 (70), 51 (80).

2-(4-Methylphenylsulfonyl)-1,4-hydroquinone (3b)

Solid; mp 211–212 °C.

IR (KBr): 3286, 1596, 1507, 1465, 1367, 1283, 1137, 1087, 914, 878, 817, 712 cm^{-1}.

^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.45 (s, 3 H), 6.60 (d, 1 H, *J* = 1.7 Hz), 6.75 (d, *J* = 7.9 Hz, 1 H), 6.90 (dd, *J* = 1.7, 7.9 Hz, Hz, 1 H), 7.30 (d, *J* = 8.1 Hz, 2 H), 7.80 (d, *J* = 8.1 Hz, 2 H), 8.90–9.08 (br s, 2 H, OH).

^{13}C NMR (proton decoupled, DMSO-d_6): δ = 20.6, 113.7, 118.3, 122.4, 126.3, 127.3, 127.3, 128.8, 128.8, 138.6, 143.1, 147.8, 149.3.

MS (EI): *m*/*z* (%) = 264 (M^+, 40), 165 (10), 154 (100), 136 (90), 120 (20), 95 (30), 81 (40), 69 (70), 55 (80).

2-Methyl-5-phenylsulfonyl-1,4-hydroquinone (3c)

Solid;[5] mp 140 °C.

IR (KBr): 3463, 3384, 1615, 1472, 1418, 1298, 1223, 1192, 1147, 1081, 1013, 874, 725 cm^{-1}.

^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.15 (s, 3 H), 6.70 (s, 1 H), 7.20 (s, 1 H), 7.45–7.60 (m, 3 H), 7.95 (d, 2 H, *J* = 8.1 Hz), 8.80 (br s, 2 H, OH).

MS (EI): *m*/*z* (%) = 264 (M^+, 100), 122 (80), 111 (20), 94 (50), 77 (50), 66 (30), 51 (50).

2-Methyl-5-(4-methylphenylsulfonyl)-1,4-hydroquinone (3d)

Solid; mp 175–176 °C.

IR (KBr): 3424, 3366, 2927, 1595, 1411, 1275, 1136, 1081, 866, 811, 673 cm^{-1}.

^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.20 (s, 3 H), 2.40 (s, 3 H), 5.40 (br s, 1 H, OH), 6.75 (s, 1 H), 7.05 (s, 1 H), 7.29 (d, 2 H, *J* = 8.0 Hz), 7.80 (d, 2 H, *J* = 8.0 Hz), 8.60 (br s, 1 H, OH).

^{13}C NMR (proton decoupled, DMSO-d_6): δ = 16.1, 20.9, 112.9, 119.2, 123.4, 127.6, 127.6, 129.1, 129.1, 132.9, 138.9, 143.2, 147.5, 147.8.

MS (EI): *m*/*z* (%) = 278 (M^+, 100), 122 (85), 92 (30), 66 (30).

2-Methoxy-5-phenylsulfonyl-1,4-hydroquinone (3e)
Solid; mp 199–200 °C.

IR (KBr): 3348, 1738, 1619, 1502, 1477, 143, 1387, 1286, 1194, 153, 1050, 978, 860, 799, 754 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 3.80 (s, 3 H), 6.40 (s, 1 H), 7.23 (s, 1 H), 7.40–7.55 (m, 3 H), 7.80 (d, 2 H, *J* = 8.1 Hz), 8.05 (br s, 1 H, OH), 8.65 (br s, 1 H, OH).

^{13}C NMR (proton decoupled, DMSO-d_6): δ = 55.5, 100.6, 113.6, 126.2, 127.0, 127.0, 128.8, 128.8, 133.2, 138.9, 139.2, 149.4, 153.1.

MS (EI): *m*/*z* (%) = 280 (M^+, 10), 135 (15), 106 (20), 77 (100), 69 (60), 51 (15).

2-Methoxy-5-(4-methylphenylsulfonyl)-1,4-hydroquinone (3f)
Solid; mp 151 °C.

IR (KBr): 3293, 2364, 1699, 1620, 1514, 1440, 1351, 1282, 1240, 1139, 1086, 1047, 824, 666 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 2.38 (s, 3 H), 3.78 (s, 3 H), 6.38 (s, 1 H), 7.15 (s, 1 H), 7.20 (d, 2 H, *J* = 8.0 Hz), 7.70 (d, 2 H, *J* = 8.0 Hz), 8.45 (br s, 1 H, OH), 9.20 (br s, 1 H, OH).

^{13}C NMR (proton decoupled, DMSO-d_6): δ = 20.9, 55.4, 100.7, 113.5, 126.3, 126.9, 126.9, 128.7, 128.7, 138.9, 139.1, 142.8, 149.5, 153.2.

MS (EI): *m*/*z* (%) = 294 (M^+, 100), 187 (10), 156 (60), 138 (100), 123 (60), 111 (60), 91 (65), 65 (65), 53 (30).

2-Ethoxy-5-phenylsulfonyl-1,4-hydroquinone (3g)
Solid; mp 190 °C.

IR (KBr): 3341, 1713, 1599, 1461, 1282, 1138, 845 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 1.35 (t, 3 H, *J* = 7.0 Hz), 3.95 (q, 2 H, *J* = 7.0 Hz), 6.45 (s, 1 H), 6.85 (s, 1 H), 7.35–7.50 (m, 3 H), 7.81–7.91 (m, 2 H), 8.01–8.15 (br s, 1 H, OH), 8.82–8.93 (br s, 1 H, OH).

^{13}C NMR (proton decoupled, DMSO-d_6): δ = 14.2, 64.4, 104.0, 106.6, 126.2, 127.5, 127.5, 128.7, 128.7, 132.9, 138.1, 141.5, 148.5, 149.5.

MS (EI): *m*/*z* (%) = 294 (M^+, 80), 266 (30), 124 (100), 96 (35), 69 (40), 43 (40).

2-Ethoxy-5-(4-methylphenylsulfonyl)-1,4-hydroquinone (3h)
Solid; mp 156 °C.

IR (KBr): 3343, 1597, 1522, 1472, 1434, 1282, 1201, 1132, 1087, 843 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 1.45 (t, 3 H, *J* = 6.9 Hz), 2.41 (s, 3 H), 4.13 (q, 2 H, *J* = 6.9 Hz), 5.15 (br s, 1 H, OH), 6.39 (s, 1 H), 7.15 (s, 1 H), 7.30 (d, 2 H, *J* = 8.1 Hz), 7.75–7.81 (d, 2 H, *J* = 8.1 Hz), 9.03 (br s, 1 H, OH),

^{13}C NMR (proton decoupled, DMSO-d_6): δ = 14.4, 20.9, 63.9, 101.8, 113.9, 116.5, 127.4, 127.4, 129.1, 129.1, 138.9, 139.2, 143.0, 149.5, 152.6.

MS (EI): *m*/*z* (%) = 308 (M^+, 40), 280 (10), 150 (20), 124 (30), 98 (25), 69 (70), 57(100).

2,5-Dimethyl-3-phenylsulfonyl-1,4-hydroquinone (3i)
Solid;[3a] mp 155–156 °C.

IR (KBr): 3439, 3284, 1728, 1598, 1423, 1324, 1259, 1225, 1181, 1120, 1068, 1009, 855, 668, 594 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 2.15 (s, 3 H), 2.25 (s, 3 H), 4.35 (br s, 1 H, OH), 6.80 (s, 1 H), 7.45–7.65 (m, 3 H), 7.85 (d, 2 H, *J* = 8.1 Hz), 10.2 (br s, 1 H, OH).

MS (EI): *m*/*z* (%) = 278 (M^+, 50), 136 (100), 118 (90), 91 (90), 65 (30), 43 (20).

2,5-Dimethyl-3-(4-methylphenylsulfonyl)-1,4-hydroquinone (3j)
Solid; mp 149 °C.

IR (KBr): 3439, 3284, 1728, 1598, 1423, 1324, 1259, 1225, 1181, 1120, 1068, 1009, 855, 668, 594 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 2.23 (s, 3 H), 2.30 (s, 3 H), 2.50 (s, 3 H), 4.35 (br s, 1 H, OH), 6.97 (s, 1 H), 7.40 (d, 2 H, *J* = 8.0 Hz), 7.85 (d, 2 H, *J* = 8.0 Hz), 10.35 (br s, 1 H, OH).

^{13}C NMR (proton decoupled, DMSO-d_6): δ = 11.9, 15.7, 21.0, 119.9, 119.9, 123.9, 124.9, 125.9, 125.9, 129.3, 129.3, 138.6, 143.9, 147.5, 148.3.

MS (EI): *m*/*z* (%) = 292 (M^+, 100), 220 (15), 136 (95), 107 (50), 91 (80), 79 (70), 65 (75), 39 (75).

2,6-Dimethyl-3-phenylsulfonyl-1,4-hydroquinone (3k)
Solid;[3a] mp 146 °C.

IR (KBr): 3421, 3219, 2365, 1613, 1568, 1469, 1347, 1216, 1128, 1083, 854, 816, 726 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 2.08 (s, 3 H), 2.20 (s, 3 H), 4.75–4.90 (br s, 1 H, OH), 6.60 (s, 1 H), 7.40–7.60 (m, 3 H), 7.78 (d, 2 H, *J* = 8.0 Hz), 9.80 (br s, 1 H, OH).

MS (EI): *m*/*z* (%) = 278 (M^+, 100), 212 (30), 197 (30), 183 (20), 151 (20), 136 (30), 77 (30), 67 (30), 51 (20).

2,6-Dimethyl-3-(4-methylphenylsulfonyl)-1,4-hydroquinone (3l)
Solid; mp 161–162 °C.

IR (KBr): 3422, 3217, 1597, 1568, 1469, 1348, 1211, 1126, 1086, 1011, 858, 819, 684 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 2.18 (s, 3 H), 2.23 (s, 3 H), 2.40 (s, 3 H), 4.30 (br s, 1 H, OH), 6.70 (s, 1 H), 7.25 (d, 2 H, *J* = 8.0 Hz), 7.78 (d, 2 H, *J* = 8.0 Hz), 9.95 (br s, 1 H, OH).

^{13}C NMR (proton decoupled, DMSO-d_6): δ = 13.1, 17.2, 21.0, 119.9, 119.9, 125.2, 126.5, 126.5, 129.4, 129.4, 130.2, 139.7, 143.5, 146.1, 150.0.

MS (EI): *m*/*z* (%) = 292 (M^+, 100), 136 (90), 107 (20), 91 (20), 67 (20), 43 (20).

2-Phenylsulfonyl-1,4-dihydroxynaphthalene (3m)
Solid; mp 179 °C.

IR (KBr): 3416, 3338, 2364, 1577, 1451, 1275, 1131, 1069, 859, 723 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 6.90 (s, 1 H), 7.40–7.60 (m, 5 H), 7.90 (d, 2 H, *J* = 8.0 Hz), 8.05 (d, 1 H, *J* = 8.1 Hz), 8.23 (d, 1 H, *J* = 8.1 Hz), 9.48–9.59 (br s, 1 H, OH), 9.82 (s, 1 H, OH).

^{13}C NMR (proton decoupled, DMSO-d_6): δ = 103.6, 122.2, 123.0, 126.1, 126.5, 127.0, 127.0, 128.1, 128.3, 128.9, 128.9, 128.9, 133.0, 141.8, 145.5, 146.2.

MS (EI): *m*/*z* (%) = 300 (M^+, 100), 158 (25), 151 (20), 130 (25), 102 (30), 77 (25), 51 (20).

2-(4-Methylphenylsulfonyl)-1,4-dihydroxynaphthalene (3n)
Solid; mp 169 °C.

IR (KBr): 3429, 2366, 1528, 1459, 1317, 1273, 1128, 862, 763 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 2.40 (s, 3 H), 6.89 (s, 1 H), 7.30 (d, 2 H, *J* = 8.1 Hz), 7.45–7.60 (m, 2 H), 7.79 (d, 2 H, *J* = 8.1 Hz),

8.10 (d, 1 H, *J* = 7.9 Hz), 8.25 (d, 1 H, *J* = 7.9 Hz), 9.45 (br s, 1 H, OH), 9.65 (br s, 1 H, OH).

^{13}C NMR (proton decoupled, DMSO-d_6): δ = 20.9, 103.6, 120.4, 122.3, 123.1, 126.2, 126.6, 127.2, 127.2, 128.1, 128.3, 129.5, 129.5, 139.0, 143.7, 145.4, 146.3.

MS (EI): *m*/z (%) = 314 (M^+, 90), 158 (90), 151 (100), 130 (60), 102 (60), 91 (50), 77 (25), 65 (25), 51 (20).

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