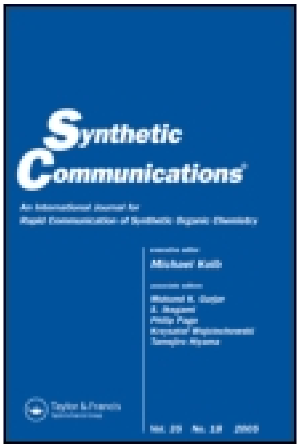

Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

An Alternative Method for the Highly Selective Iodination of Alcohols Using a CsI/$BF_3 \cdot Et_2O$ System

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Version of record first published: 20 Aug 2006.

To cite this article: Safdar Hayat, Atta-ur-Rahman, Khalid Mohammed Khan, M. Iqbal Choudhary, Ghulam Murtaza Maharvi, Zia-Ullah & Ernst Bayer (2003): An Alternative Method for the Highly Selective Iodination of Alcohols Using a CsI/$BF_3 \cdot Et_2O$ System, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 33:14, 2531-2540

To link to this article: http://dx.doi.org/10.1081/SCC-120021844

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SYNTHETIC COMMUNICATIONS®
Vol. 33, No. 14, pp. 2531–2540, 2003

An Alternative Method for the Highly Selective Iodination of Alcohols Using a CsI/$BF_3 \cdot Et_2O$ System

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M. Iqbal Choudhary, Ghulam Murtaza Maharvi,
Zia-Ullah, and Ernst Bayer#

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ABSTRACT

A variety of allylic and benzylic alcohols have been converted into the corresponding iodides using cesium iodide (CsI) in the presence of boron trifluoride etherate ($BF_3 \cdot Et_2O$) in acetonitrile under mild conditions.

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#This Communication is the result of joint research program with Late Prof. Dr. Ernst Bayer, Institut für Organische Chemie der Universität Tübingen, Auf der Morgenstelle 18, D-72076, Tübingen, Germany.

DOI: 10.1081/SCC-120021844
0039-7911 (Print); 1532-2432 (Online)
www.dekker.com

Alkyl halids, especially iodides and bromides are generally used as intermediates in substitution reactions, elimination reactions, and rearrangements. Consequently, the conversion of alcohols to halides is a useful transformation in synthetic organic chemistry. A variety of methods have been appeared in the literature[1–4] for transformation of alcohols into halides, however some of these involve expensive reagents, tedious reaction work-up and long reaction times. Most of the methodologies include: iodo[5]- and bromotrimethylsilanes,[6] chlorotrimethylsilane-sodium iodide[7] and hexamethyldisilazane/iodide ion.[8] Moreover, some reports[9–14] have been published regarding the selective conversion of allylic and benzylic, primary, secondary, and tertiary alcohols into halides. Very recently methanesulphonic acid/sodium iodide (CH_3SO_3H/ NaI) is reported for the conversion of alkyl, allyl, and benzyl alcohols into iodes.[15]

In this communication we describe the conversion of allylic and benzylic alcohols into its corresponding iodides by using cesium iodides (CsI) in the presence of $BF_3{\cdot}Et_2O$ under mild conditions.

The present methodology deals with the conversion of alcohols into iodines, using different allyl and benzyl alcohols as substrates. To establish the general effectiveness of our newly developed method, some selected iodination reaction was carried out. This reagent appears to be the most reactive and versatile. The reactions of allyl and benzyl alcohols with a molar equivalent of cesium iodide in the presence of $BF_3{\cdot}Et_2O$ in acetonitrile under mild conditions cleanly gives the corresponding iodides (Sch. 1).

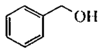
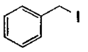
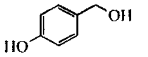
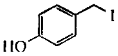
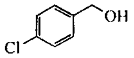
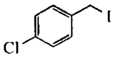
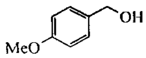
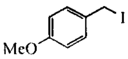
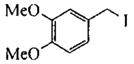
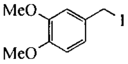
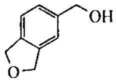
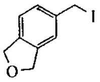
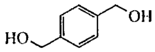
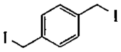
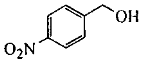
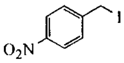
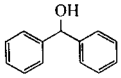
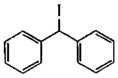
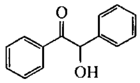
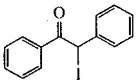
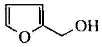
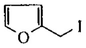
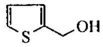
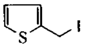
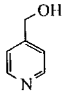
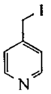
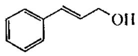
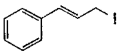
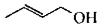
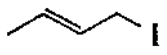
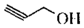
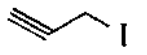
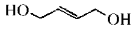
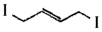
The reaction was found to proceed smoothly at room temperature within 5–30 min. The products were isolated by aqueous work-up followed by solvent extraction and were purified by column chromatography if necessary to afford pure alkyl iodide. The result and spectroscopic data of the following compounds were collected in Tables 1 and 2.

The reaction was found to be highly selective for conversion of allylic and benzylic alcohols into iodides in high yields (82–96%), whereas saturated aliphatic alcohols did not yield iodides even after prolonged stirring in acetonitrile (Entries 18–21). Iodination of benzyl alcohol and substituted benzyl alcohols with electron-donating groups, e.g., -OH,

$$\underset{1}{\mathrm{R{-}OH}} \xrightarrow[\mathrm{CH_3CN,\ 25^\circ C}]{\mathrm{CsI/BF_3{\cdot}Et_2O}} \underset{2}{\mathrm{R{-}I}}$$

Scheme 1.

Table 1. Conversion of alcohols to iodides using a CsI/BF$_3$·Et$_2$O system at 25°C.

Entry	Substrate		Product		Time (min)	Yield[a] (%)
1		(**1a**)		(**2a**)	5	96
2		(**1b**)		(**2b**)	15	95
3		(**1c**)		(**2c**)	15	93
4		(**1d**)		(**2d**)	20	90
5		(**1e**)		(**2e**)	20	92
6		(**1f**)		(**2f**)	25	88
7		(**1g**)		(**2g**)	25	95
8		(**1h**)		(**2h**)	30	85
9		(**1i**)		(**2i**)	20	84
10		(**1j**)		(**2j**)	20	82
11		(**1k**)		(**2k**)	10	91
12		(**1l**)		(**2l**)	10	88
13		(**1m**)		(**2m**)	15	85
14		(**1n**)		(**2n**)	20	85
15		(**1o**)		(**2o**)	15	83
16		(**1p**)		(**2p**)	15	82
17		(**1q**)		(**2q**)	20	80
18	*n*-Octanol	(**1r**)	No reaction	—	60	—
19	Isoamyl alcohol	(**1s**)	No reaction	—	60	—
20	Cyclohexanol	(**1t**)	No reaction	—	60	—
21	*n*-Butyl alcohol	(**1u**)	No reaction	—	60	—

[a]Yields of isolated products.

Table 2. Spectral data of compound **2a–2q**.

Compound	^{1}H NMR^{a,b,c} δ, J (Hz)	^{13}C NMR^{a,d,e} δ	IR ν (cm^{-1})
2a	7.55–7.33 (m, 5H, Ar-H), 4.57 (s, 2H, ArCH$_2$)	138.0, 129.3, 129.3, 128.9, 128.9, 128.5, 5.6	(KBr) 2916, 1588, 1148, 1048 831, 638
2b	7.05 (d, 2H, $J = 8.2$, Ar-H), 6.64 (d, 2H, $J = 8.2$, Ar-H), 4.22, (s, 2H, ArCH$_2$)	158.3, 130.7, 130.7, 130.0, 116.1, 116.1, 5.39	(KBr) 3448, 2914, 1476, 1084, 844, 642
2c	7.25 (d, 2H, $J = 8.1$, Ar-H), 7.18 (d, 2H, $J = 8.1$, Ar-H) 4.34 (s, 2H, ArCH$_2$)	137.9, 133.6, 130.1, 130.1, 129.0, 129.0, 129.0, 6.6	(KBr) 3010, 2912, 1508, 1016, 831, 714, 646
2d	7.05 (d, 2H, $J = 7.9$, Ar-H), 6.67 (d, 2H, $J = 7.9$, Ar-H), 4.31 (s, 2H, ArCH$_2$), 3.58 (s, 3H, OCH$_3$)	159.0, 133.3, 128.6, 128.6, 113.8, 113.8, 55.3, 6.7	(KBr) 2911, 1604, 1473, 1388, 1256, 1092, 811, 691
2e	6.70 (dd, 1H, $J = 8.1$, 1.6, Ar-H), 6.65 (d, 1H, $J = 1.6$, Ar-H), 6.62 (d, 1H, $J = 8.1$, Ar-H), 4.36 (s, 2H, ArCH$_2$) 3.65 (s, 3H, OCH$_3$), 3.64 (s, 3H, OCH$_3$)	148.9, 148.3, 133.8, 119.3, 111.1, 110.5, 55.8, 55.7, 4.6	(KBr) 3011, 2906, 1511, 1384, 1208, 832, 748, 688
2f	6.79 (dd, 1H, $J = 7.8$, $J = 1.7$, Ar-H), 6.77 (d, 1H, $J = 7.8$, Ar-H), 6.62 (d, 1H, $J = 1.7$, Ar-H), 5.86 (s, 2H, O-CH$_2$-O), 4.35 (s, 2H, ArCH$_2$)	147.7, 147.3, 132.9, 122.1, 109.2, 108.3, 101.2, 6.6	(KBr) 3018, 2898, 1618, 1482, 1254, 1034, 838, 710, 632
2g	7.20 (s, 4H, Ar-H), 4.42 (s, 4H, ArCH$_2$)	140.8, 140.8, 126.3, 126.3, 126.3, 5.2, 5.2	(KBr) 2924, 1503, 1478, 1172, 1084, 748, 632
2h	7.98 (d, 2H, $J = 8.3$, Ar-H), 7.35 (d, 2H, $J = 8.3$, Ar-H), 4.32 (s, 2H, ArCH$_2$)	146.6, 134.1, 129.1, 129.1, 122.2, 122.2, 7.0	(KBr) 3026, 2852, 1538, 1486, 1016, 843, 642
2i	7.31–7.12 (m, 10H, Ar-H), 5.32 (s, 1H, Ar-CH)	142.3, 142.3, 128.4, 128.4, 128.4, 128.4, 127.4, 127.4, 127.3, 127.3, 127.3, 127.3, 127.3, −4.1	(KBr) 3026, 2905, 1658, 1316, 1184, 1026, 736, 642

2j	7.84–7.30 (m, 5H, Ar-H), 7.29–7.17 (m, 5H, Ar-H), 5.23 (s, 1H, Ar-CH)	199.0, 139.1, 133.9, 133.5, 129.2, 129.2, 129.1, 128.7, 128.7, 128.6, 128.6, 127.8, 127.8, 6.5	(KBr) 3018, 2824, 1715, 1508, 1054, 934, 816, 638
2k	7.19 (d, 1H, $J = 1.7$, Ar-H), 6.16 (dd, 1H, $J = 3.8$, 1.7, Ar-H), 6.08 (d, 1H, $J = 3.8$, Ar-H), 4.33 (s, 2H, CH_2I)	154.3, 142.3, 110.3, 107.6, −3.3	(Neat) 2964, 2852, 1462, 1268, 1024, 804, 694
2l	7.08 (d, 1H, $J = 4.6$, Ar-H), 6.86 (dd, 1H, $J = 4.6$, 3.5, Ar-h), 6.79 (d, 1H, $J = 3.5$, Ar-H), 4.49 (s, 2H, CH_2I)	144.3, 127.0, 125.6, 125.5, −5.8	(Neat) 3012, 2862, 1462, 1256, 1026, 811, 752, 648
2m	8.47(d, 2H, $J = 5.7$, Ar-H), 7.31 (d, 2H, $J = 5.7$, Ar-H), 4.57 (s, 2H, CH_2)	151.7, 149.2, 149.2, 121.1, 121.1, 4.2	(KBr) 2916, 2852, 1643, 1584, 1262, 1022, 754, 646
2n	7.27–7.05 (m, 5H, Ar-H), 6.48 (d, 1H, $J = 14.5$, Ar-CH), 6.23 (m, 1H, =CH), 4.15 (d, 2H, $J = 6.4$, CH_2I)	136.8, 130.9, 128.7, 128.7, 128.6, 127.6, 126.5, 126.5, 5.4	(KBr) 3028, 2916, 1652, 1492, 1128, 1026, 764, 684
2o	5.91 (m, IH, $=\underline{CH}\text{-}CH_2\text{-}$), 5.64 (m, 1H, $CH_3\text{-}\underline{CH}=$), 3.83 (d, 2H, $J = 5.4$, CH_2I), 1.71 (d, 3H, $J = 5.4$, CH_3)	129.3, 128.4, 17.3, 6.1	(Neat) 2916, 2852, 1426, 1374, 890, 724, 648
2p	3.67 (d, 2H, $J = 2.9$, CH_2I), 2.45 (t, 1H, $J = 2.9$, $CH{\equiv}C\text{-}$)	82.7, 67.3, 5.1	(Neat) 2916, 2850, 2132, 1418, 724, 651
2q	5.61 (t, 2H, $J = 5.6$, $-CH{=}CH$), 3.42 (d, 4H, $J = 5.6$, CH_2I)	126.7, 126.7, 5.7, 5.7	(Neat) 2908, 2854, 1367, 892, 728, 652

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[a] Chemical shift δ, ppm, coupling constant J, Hz.

[b] **2a–2f, 2h–2l, 2n–2q**: 250 MHz, $CDCl_3$.

[c] **2g, 2 m**: 250 MHz, DMSO-d_6.

[d] **2a–2f, 2h–2l, 2n–2q**: 63 MHz, $CDCl_3$.

[e] **2g, 2m**: 63 MHz, DMSO-d_6.

-OCH_3, -Cl, -O-CH_2-O- proceeds very well and in high yields with $CsI/BF_3 \cdot Et_2O$ in acetonitrile (Entries 1–7). The electron-withdrawing group, e.g., *p*-nitro benzyl alcohol (**1h**) also give very good yield 85% at 30 min (Entry 8). The remarkable selectivity of this reaction allowed only benzylic hydroxyl group to be iodinated without effecting the phenolic OH group present in the same molecule (Entry 2). Similarly, secondary alcohols, such as diphenylcarbinoI (**1i**) and benzoin (**1j**) could easily be substituted by an iodide group in high yields using this reagent (Entries 9, 10).

Furthermore, the displacement of hydroxy groups with iodine from furfuryl alcohol (**1k**), 2-hydroxymethyl-thiophene (**1l**), and 4-hydroxymethyl-pyridine (**1m**) are performed well by this reagent and produces iodinated products in very good yields (Entries 11–13). The reaction of allyl alcohols e.g., cinnamyl alcohol (**1n**), 2-buten-1-ol (**1o**), 2-propy-1-ol (**1p**), and 1,4-dihydroxy-*cis*-2-butene (**1q**) with $CsI/BF_3 \cdot Et_2O$ also gave rise to the desired corresponding iodides in excellent yields (Entries 14–17).

In conclusion, this method is an excellent for the rapid, simple, mild, direct, highly regio-selective, and high yield conversion of allylic and benzylic alcohols to their corresponding iodides on reaction with $CsI/BF_3 \cdot Et_2O$ in acetonitrile. The present method will be a valuable addition into the useful synthetic methodology.

EXPERIMENTAL

General

Melting points were determined with a Büchi SMP-20 apparatus and are uncorrected. The ultraviolet spectra were measured in chloroform on a Lambda 5 UV/VIS spectrometer (Perkin-Elmer). Infrared spectra (KBr discs) were recorded on a Bruker FT-IR IFS 48 spectrometer. EI mass spectral data were recorded with Varian MAT 711 (70 eV) spectrometer and data are tabulated as *m/z*. 3-Nitrobenzyl alcohol was used in the matrix of mass spectra. ^1H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ and DMSO-d_6 using Brucker AC 250 (250 MHz and 62.9 MHz) spectrometer, respectively. Splitting patterns were as follows: *s*, singlet; *d*, doublet; *dd*, double doublet; *t*, triplet; *m*, multiplet; *br*, broad. Chemical shifts are reported in δ (ppm) and coupling constants are given in Hz. The progress of all reactions was monitored by TLC, which was performed on 2.0 × 5.0 cm aluminum sheets precoated with silica gel 60F$_{254}$ to a thickness of 0.25 mm (Merck). The chromatograms were visualized under ultraviolet light (254–366 nm) or iodine vapors.

Materials

All allylic and benzylic alcohols, furfuryl alcohol, 2-hydroxymethyl-thiophene and 4-hydroxymethyl-pyridine together with reagents CsI and BF$_3$·Et$_2$O are commercially available (Fluka, Aldrich). The anhydrous acetonitrile was purchased from Merck and used without purification.

General Procedure for Conversion of Benzylic and Allylic Alcohols into Iodides

To a stirred solution of the alcohol (3.0 mmol) and cesium iodide (3.0 mmol) in 10 mL dry acetonitrile was added a solution of BF$_3$·Et$_2$O (3.0 mmol) in 2 mL of acetonitrile during 15 min. The reaction mixture was stirred at room temperature for a period as indicated in Table l. The reaction mixture was poured into cooled water (20 mL), treated with aqueous solution (15%) of Na$_2$S$_2$O$_3$ and then extracted with ether (3 × 20 mL). The combined organic phases were washed with water (20 mL), brine (20 mL) and then dried over anhydrous Na$_2$SO$_4$. Evaporation of the solvent gave residue, which was purified by column chromatography over silica gel using *n*-hexane:ethyl acetate (9:1) as an eluent to afford pure alkyl iodides.

Benzyl iodide (2a). Yellow crystalline solid; yield: 0.63 g (96%); m.p.: 21–23°C (lit.[16] m.p.: 24–25°C); $R_f = 0.84$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 218 [M$^+$] (59), 141 (32), 127 (24), 91 (100), 77 (15), 65 (11), 53 (6), 43 (5). Anal. calcd. for C_7H_7I: C, 38.56; H, 3.24. Found: C, 38.47; H, 3.32.

4-Hydroxybenzyl iodide (2b). Colorless crystalline solid; yield: 0.67 g (95%); m.p.: 108–110°C; $R_f = 0.52$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 234 [M$^+$] (46), 219 (18), 127 (42), 117 (51), 107 (100), 91 (31), 77 (16), 59 (8), 43 (72). Anal. calcd. for C_7H_7IO: C, 35.93; H, 3.01. Found: C, 35.87; H, 2.99.

4-Chlorobenzyl iodide (2c). Colorless crystalline solid; yield: 0.70 g (93%); m.p.: 58–60°C (lit.[16] m.p.: 60–61°C); $R_f = 0.84$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 252 [M$^+$] (57), 127 (72), 125 (100), 101 (6), 89 (34), 76 (7), 63 (11). Anal. calcd. for C_7H_7ClI: C, 33.30; H, 2.40. Found: C, 33.41; H, 2.36.

4-Methoxybenzyl iodide (2d). Pale yellow solid; yield: 0.67 g (90%); m.p.: 25–27°C (lit.[17]m.p.: 27°C); $R_f = 0.82$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 248 [M$^+$] (65), 217 (16), 142 (100), 127 (81), 121 (84), 106 (7), 91 (11), 77 (15), 63 (8), 43 (6). Anal. calcd. for C_8H_9IO: C, 38.74; H, 3,66. Found: C, 38.82; H, 3.59.

3,4-Dimethoxybenzyl iodide (2e). Yellow crystalline solid; yield: 0.77 g (92%); m.p.: 40–42°C (1it.[17] m.p.: 43°C); $R_f = 0.48$ (hexane:ethyl acetate, 5:2); EI MS: m/z (%) = 278 [M$^+$] (35), 263 (41), 248 (32), 232 (19), 216 (23), 151 (100), 137 (14), 127 (17), 107 (8), 91 (6), 75 (8), 57 (11), 43 (7). Anal. calcd. for $C_9H_{11}IO_2$: C, 38.87; H, 3.99. Found: C, 38.78; H, 3.92.

3,4-Methylenedioxybenzyl iodide (2f). Colorless crystalline solid; yield: 0.69 g (88%); m.p.: 76–78°C; $R_f = 0.62$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 262 [M$^+$] (23), 214 (6), 135 (100), 127 (11), 105 (15), 83 (7), 79 (4), 77 (21), 67 (6), 51 (21). Anal. calcd. for $C_8H_7IO_2$: C, 36.67; H, 2.69. Found: C, 36.72; H, 2.78.

4-(Iodomethyl)-benzyl iodide (2g). Colorless crystalline solid; yield: 1.02 g (95%); m.p.: 174–175°C (lit.[18] m.p.: 176–177°C); $R_f = 0.82$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 358 [M$^+$] (62), 282 (8), 231 (24), 217 (31), 141 (43), 104 (100), 90 (19), 76 (11), 65 (14), 56 (7), 45 (16). Anal. calcd. for $C_8H_8I_2$: C, 26.84; H, 2.25. Found: C, 26.77; H, 2.18.

4-Nitrobenzyl iodide (2h). Yellow needle; yield: 0.67 g (85%); m.p.: 122–124°C (lit.[19] m.p.: 125–127°C); $R_f = 0.55$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 263 [M$^+$] (61), 149 (11), 136 (100), 127 (8), 106 (19), 89 (21), 76 (12), 63 (5), 51 (4). Anal. calcd. for $C_7H_6NIO_2$: C, 31.96; H, 2.30; N, 5.33. Found: C, 31.88; H, 2.39; N, 5.27.

Diphenylmethyl iodide (2i). Colorless crystalline solid; yield: 0.74 g (84%); m.p.: 69–70°C (lit.[19] m.p.: 72°C); $R_f = 0.78$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 294 [M$^+$] (26), 217 (43), 167 (15), 127 (8), 105 (100), 91 (7), 77 (51), 65 (7), 51 (14). Anal. calcd. for $C_{13}H_{11}I$: C, 53.09; H, 3.77. Found: C, 53.12; H, 3.73.

α-Iodo-α-phenylacetophenone (2j). Colorless crystalline solid; yield: 0.79 g (82%); m.p.: 90–92°C (lit.[20] m.p.: 92–93°C); $R_f = 0.38$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 322 [M$^+$] (44), 245 (19), 217 (21), 195 (100), 127 (12), 118 (25), 105 (31), 91 (16), 83 (8), 77 (15), 65 (5), 51 (9). Anal. calcd. for $C_{14}H_{11}IO$: C, 52.20; H, 3.44. Found: C, 52.27; H, 3.38.

2-Iodomethyl-furane (2k). Oily; yield: 0.57 g (91%); $R_f = 0.69$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 208 [M$^+$] (29), 127 (41), 104 (4), 81 (100), 77 (6), 65 (11), 52 (6), 43 (15). Anal. calcd. for C_5H_5IO: C, 28.87; H, 2.42. Found: C, 28.81; H, 2.48.

2-lodomethyl-thiopliene (2l). Oily; yield: 0.598 (88%); $R_f = 0.79$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 224 [M^+] (48), 141 (11), 127 (32), 97 (100), 83 (23), 51 (19), 43 (8), 32 (6). Anal. calcd. for C_5H_5IS: C, 26.80; H, 2.25. Found: C, 26.75; H, 2.31.

4-lodomethyl-pyridine (2m). Yellow needle; yield: 0.56 g (85%); m.p.: 61–63°C; $R_f = 0.24$ (hexane:ethyl acetate, 5:4); EI MS: m/z (%) = 219 [M^+] (23), 127 (11), 93 (100), 78 (4), 67 (14), 66 (36), 51 (13), 39 (23). Anal. calcd. for C_6H_6IN: C, 32.90; H, 2.76; N, 6.39. Found: C, 32.86; H, 2.67; N, 6.44.

3-Iodo-1-phenyl-1-propene (2n). Colorless plates; yield: 0.62 g (85%); m.p.: 54–56°(lit.[10] m.p. 56–57°C); $R_f = 0.85$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 244 [M^+] (56), 167 (43), 127 (31), 117 (100), 106 (11), 91 (19), 77 (9), 57 (23), 43 (25). Anal. calcd. for C_9H_9I: C, 44.29; H, 3.72. Found: C, 44.24; H, 3.78.

***trans*-1-Iodo-2-butene (2o).** Oily; yield: 0.45 g (83%); $R_f = 0.53$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 182 [M^+] (36), 141 (11), 127 (15), 55 (100), 41 (23), 26 (11). Anal. calcd. for C_4H_7I: C, 26.40; H, 3.88. Found: C, 26.46; H, 3.81.

3-Iodoprop-1-yne (2p). Oily; yield: 0.41 g (82%); $R_f = 0.48$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 166 [M^+] (53), 141 (31), 127 (19), 39 (100), 25 (11). Anal. calcd. for C_3H_3I: C, 21.71; H, 1.82. Found: C, 21.78; H, 1.88.

1,4-Diiodo-2-butene (2q). Oily; yield: 0.74 g (80%); $R_f = 0.64$ (hexane:ethyl acetate, 5:1); EI MS: m/z (%) = 308 [M^+] (42), 282 (15), 181 (41), 167 (21), 141 (13), 54 (100), 40 (9), 26 (7). Anal. calcd. for $C_4H_6I_2$: C, 15.60; H, 1.96 Found: C, 15.67; H, 1.89.

ACKNOWLEDGMENT

We express our gratitude to the Deutsche Akademische Austauschdienst (DAAD, Bonn, Germany) for a sandwich-type scholarship granted to Safdar Hayat.

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Received in the Netherlands September 20, 2002