

Supporting Information

for

**A SELF-SUPPORTED PALLADIUM–
BIPYRIDYL CATALYST FOR THE SUZUKI–
MIYAURA COUPLING IN WATER**

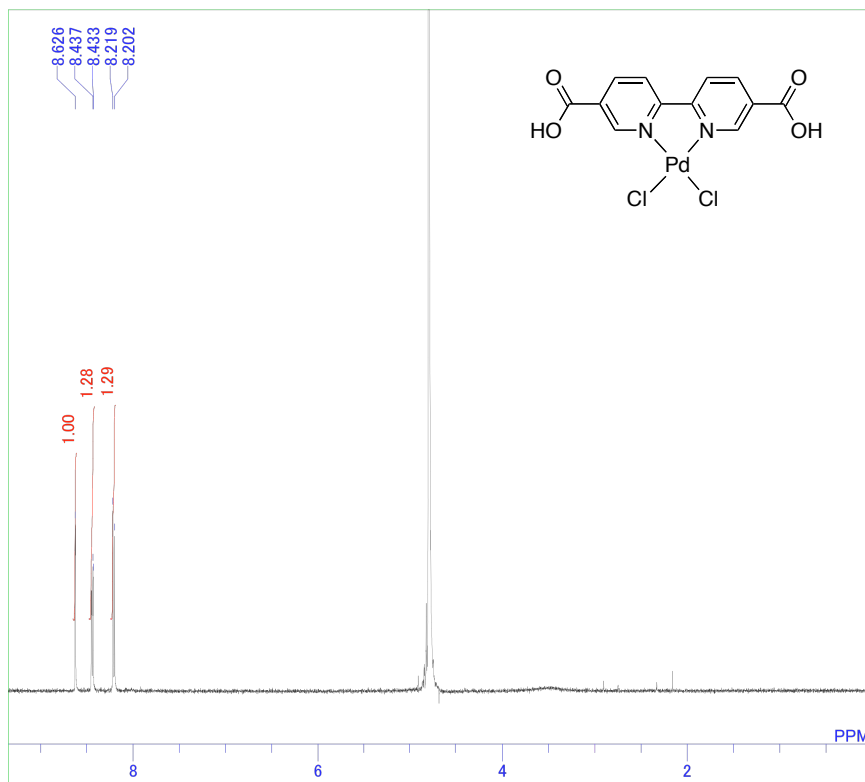
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E-mail: uo@ims.ac.jp

¹H NMR spectrum of PdCl₂BPy

C:\Documents and Settings\ALPHA\Desktop\UozumiG\Osako\PdBPY-COOH\TO1129.als

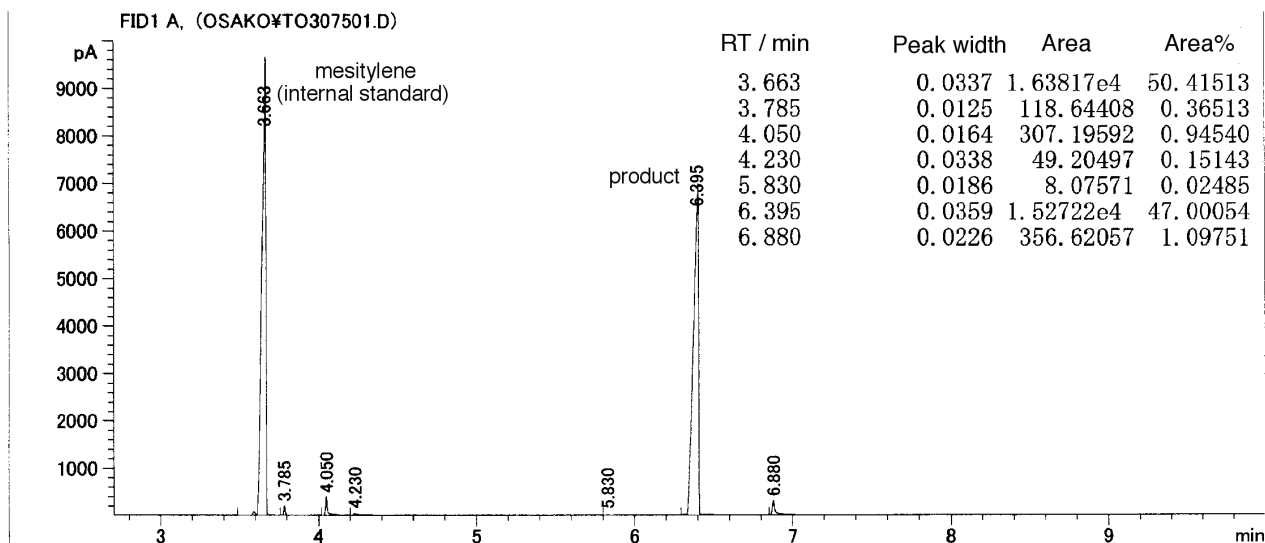


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56.80 usec
0.20000 msec
10.0000 msec
16384
16384
16
1
10000.00 Hz
5000 Hz
40.00 usec
1.6384 sec
2.0000 sec
16
21
0.00 Hz
0.00
0.00
90.00
100.00
SINGL
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1H
500.00 MHz
162410.00 Hz
50 usec
511
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20
228
768
12 Hz

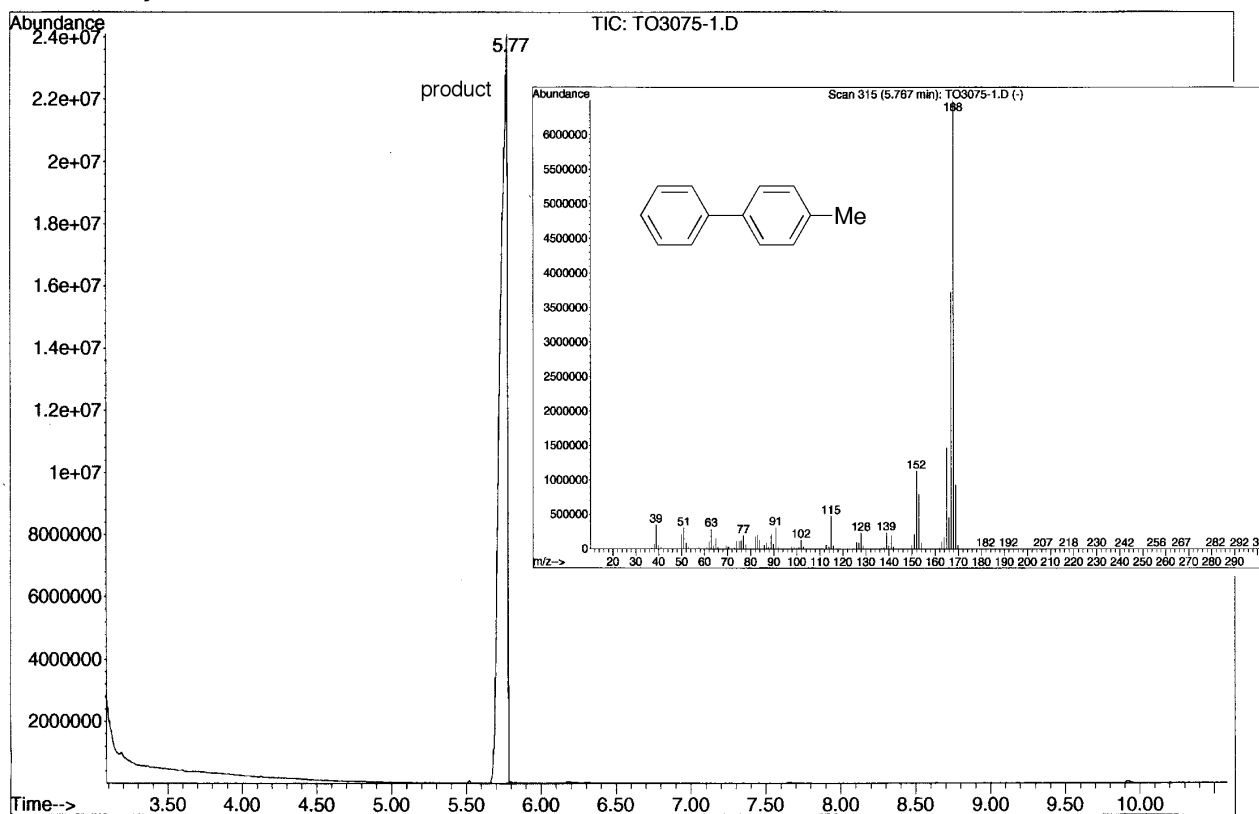
GC and GC-MS analyses for the Suzuki-Miyaura Coupling of phenyl iodide with 4-methylphenylboronic acid (Table 1, entry 1).

Product: 4-phenyltoluene [CAS: 644-08-6]

GC Analysis

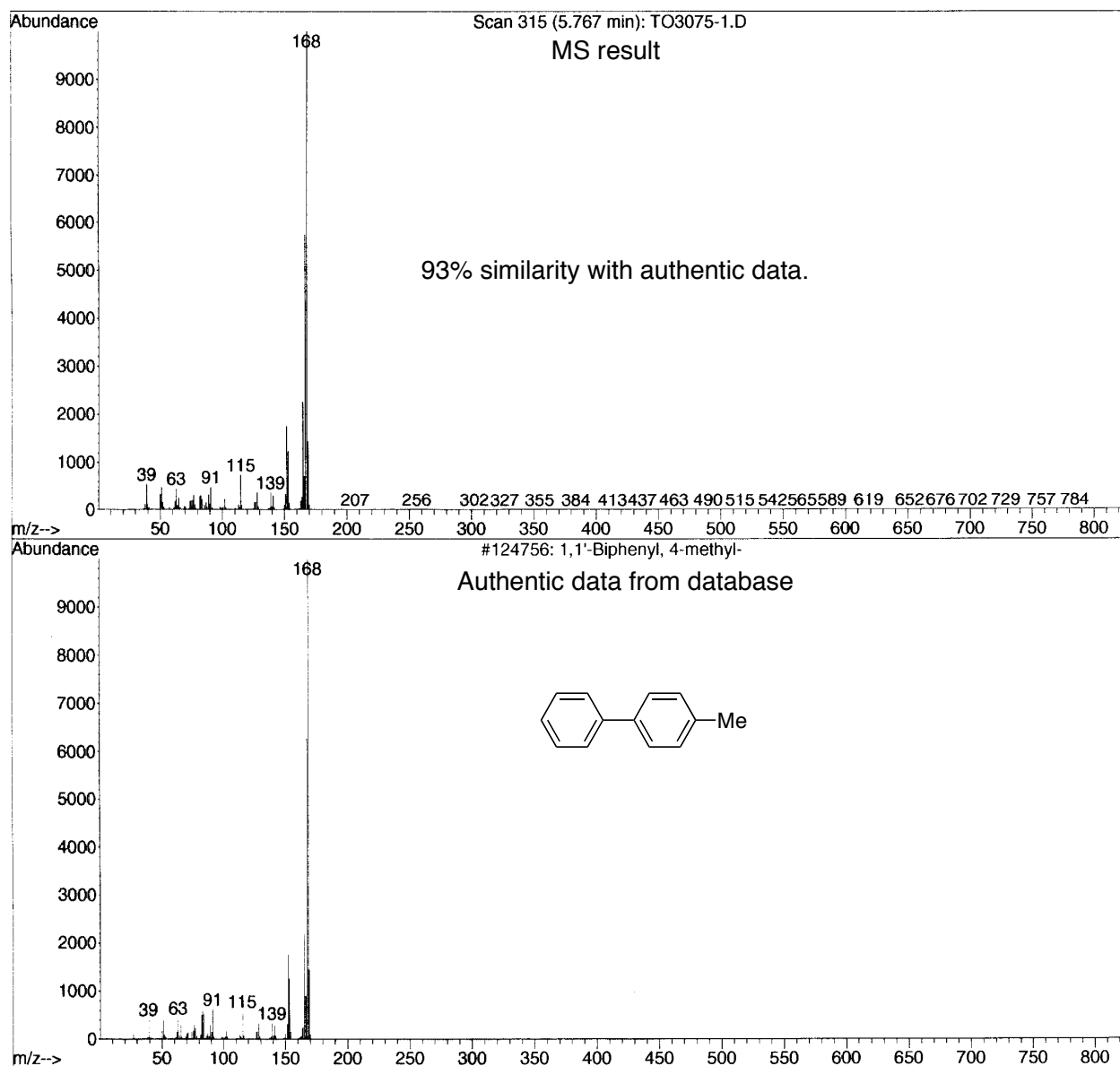


GC-MS Analysis



Comparison with NIST Mass Database Library

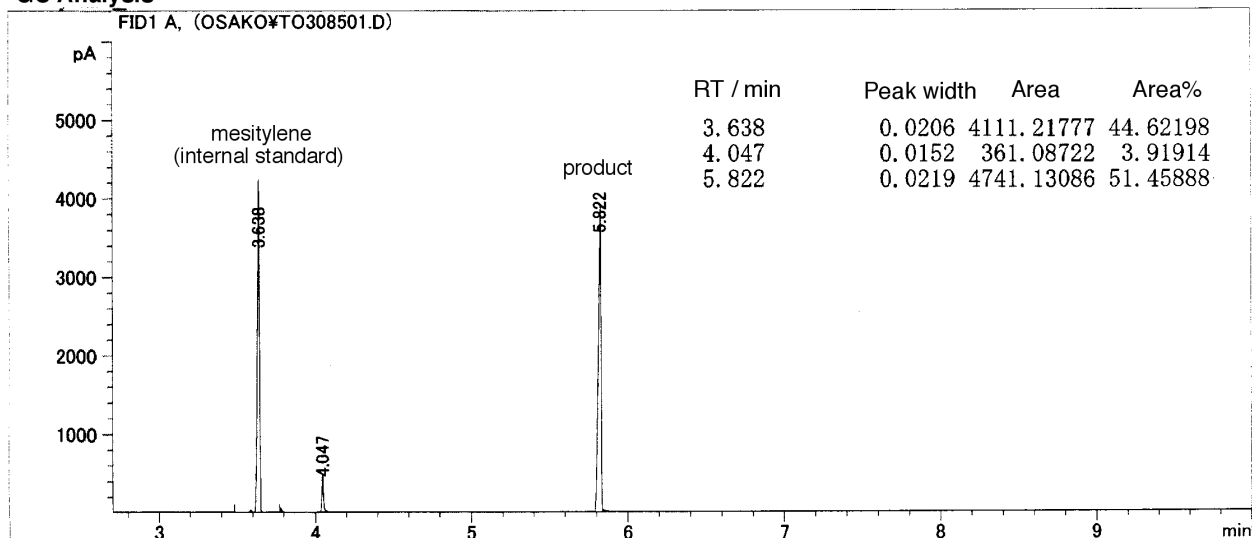
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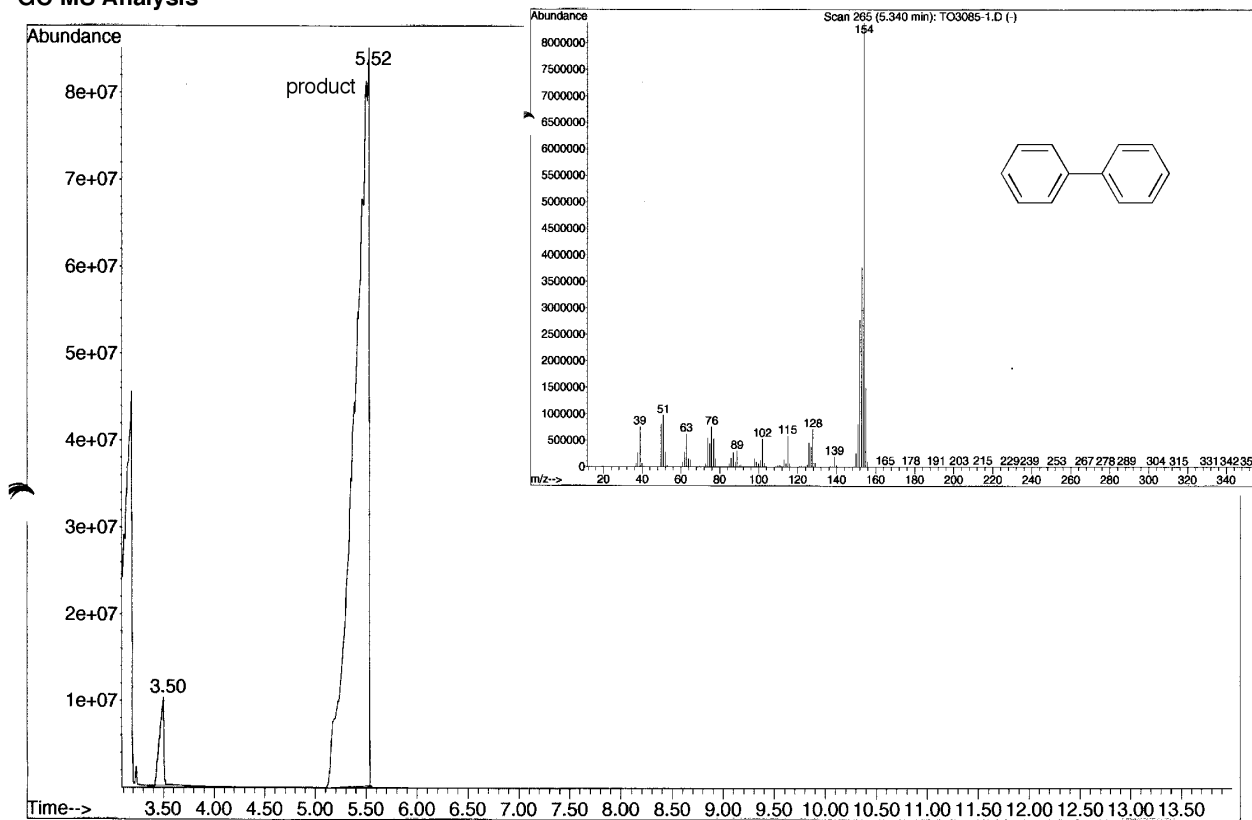
GC and GC-MS analyses for the Suzuki-Miyaura Coupling of phenyl iodide with phenylboronic acid (Table 1, entry 3).

Product: biphenyl [CAS: 92-52-4]

GC Analysis

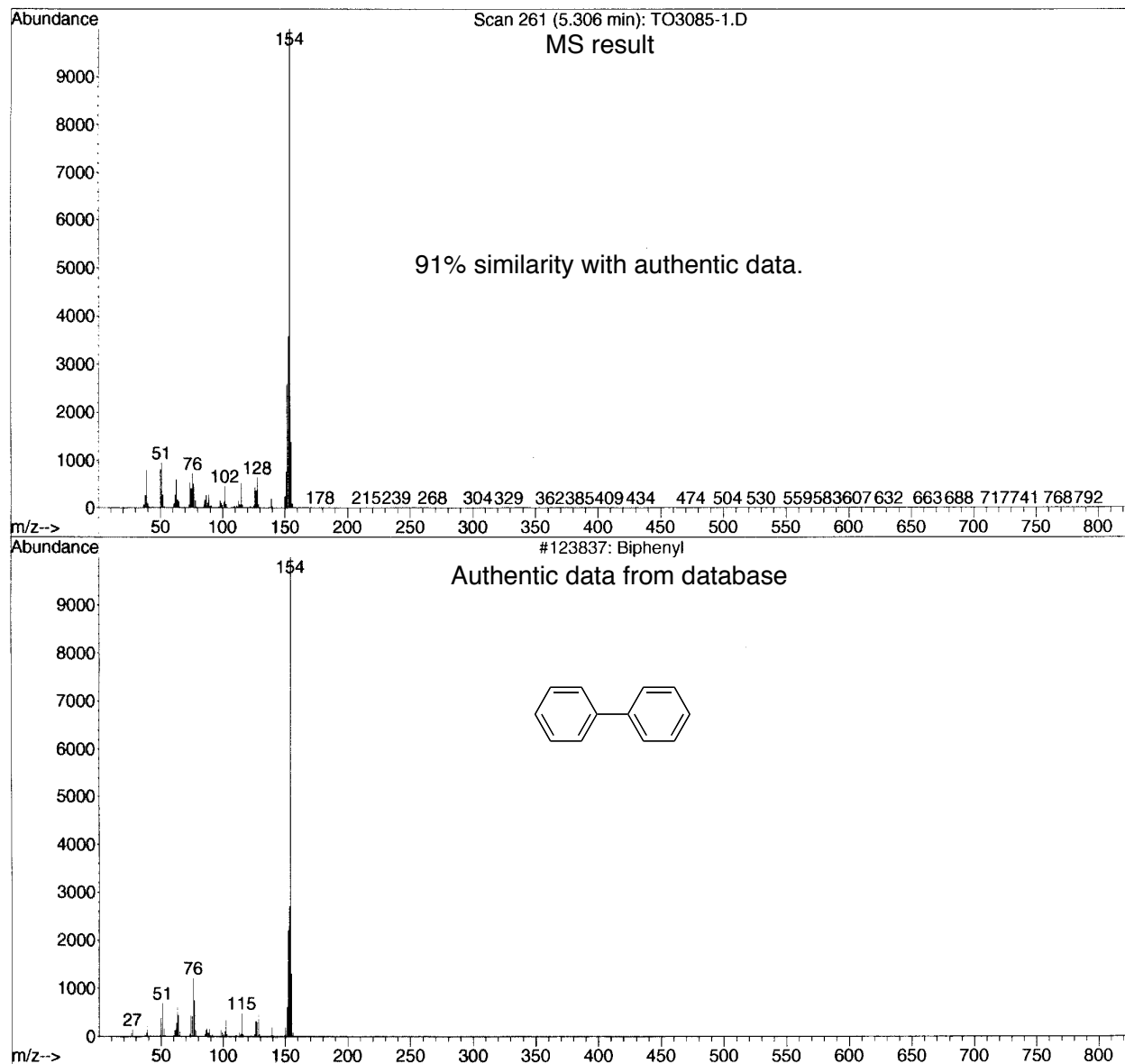


GC-MS Analysis



Comparison with NIST Mass Database Library

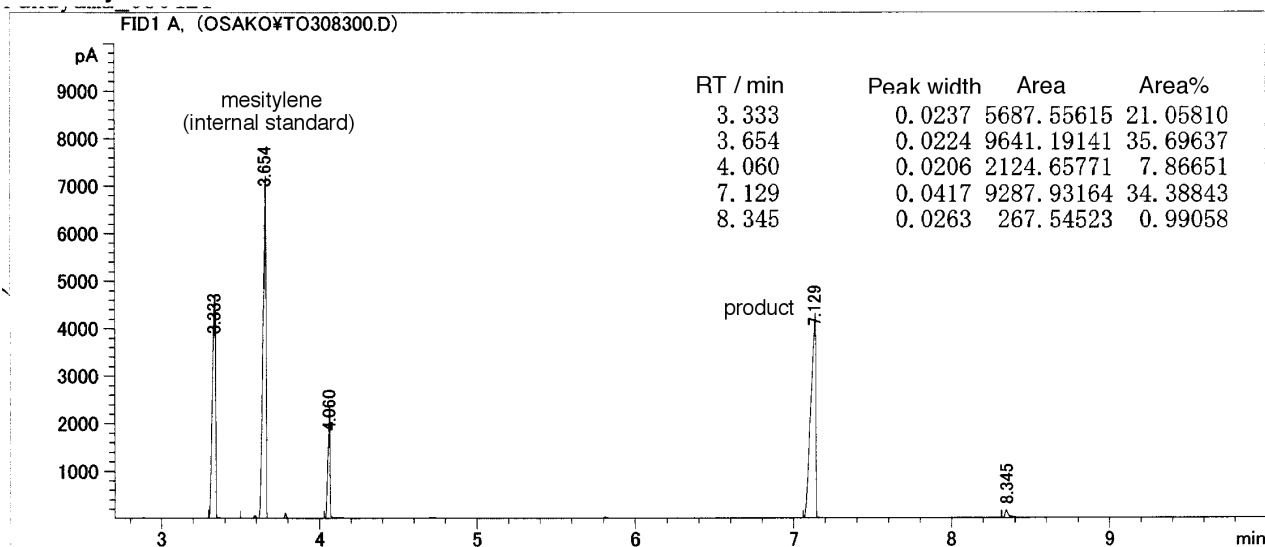
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Quality : 91
ID : Biphenyl



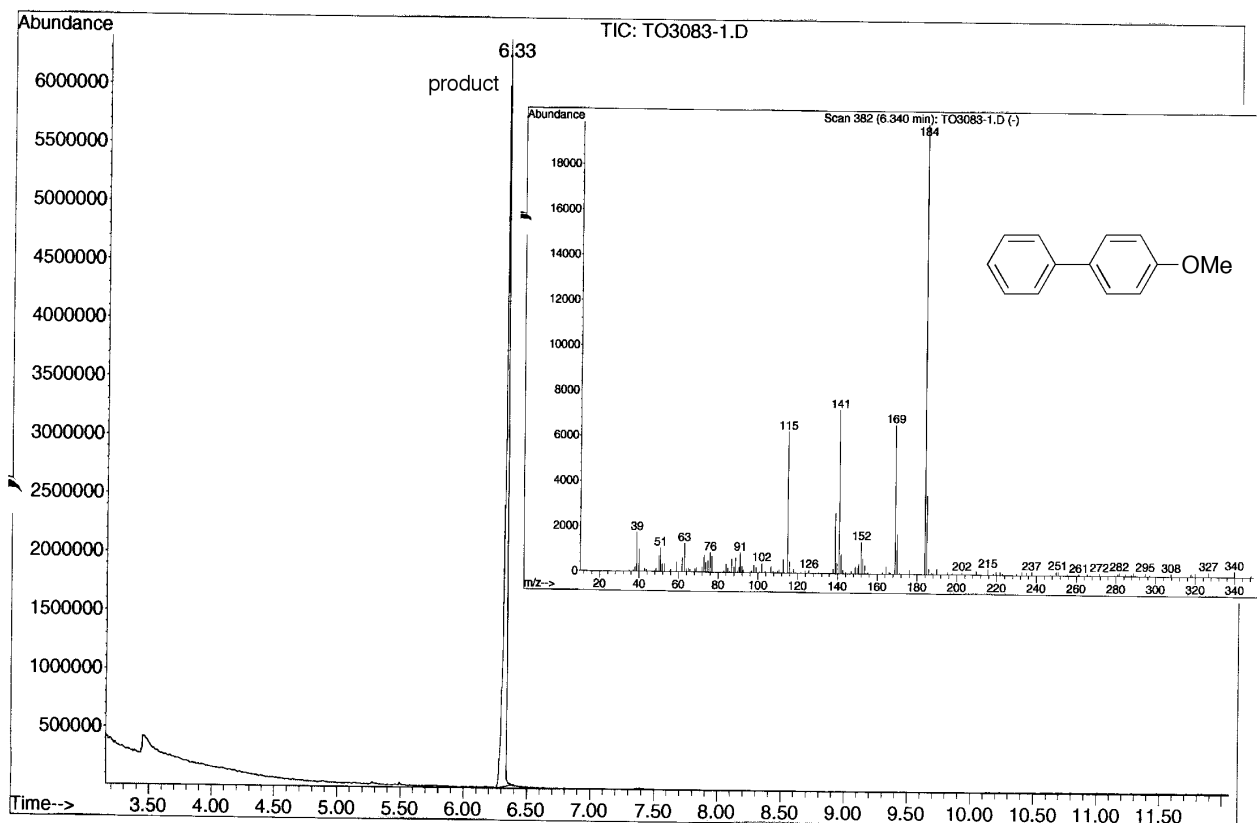
GC and GC-MS analyses for the Suzuki-Miyaura Coupling of phenyl iodide with 4-methoxyphenylboronic acid (Table 1, entry 5).

Product: 4-methoxybiphenyl [CAS: 613-37-6]

GC Analysis

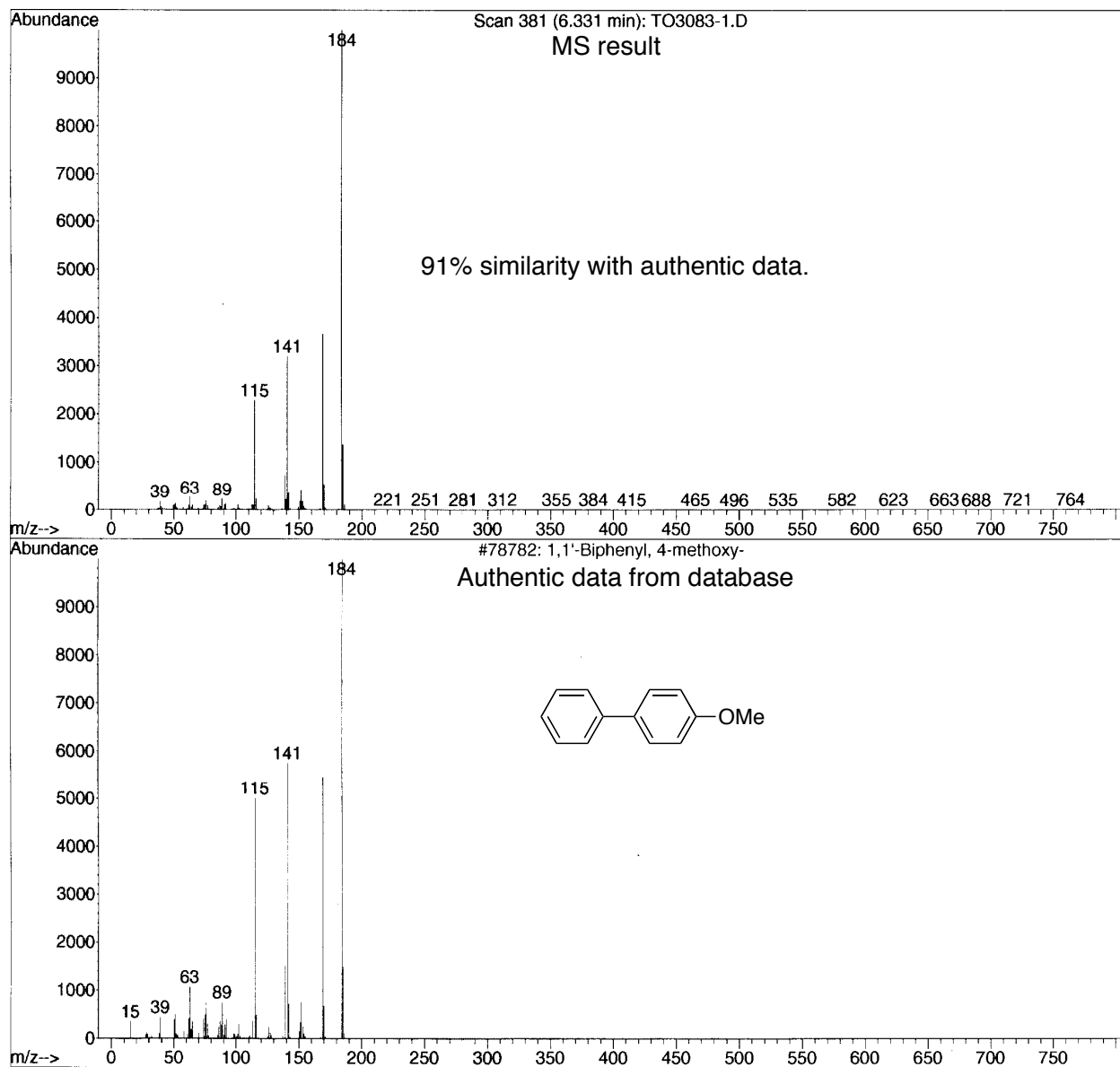


GC-MS Analysis



Comparison with NIST Mass Database Library

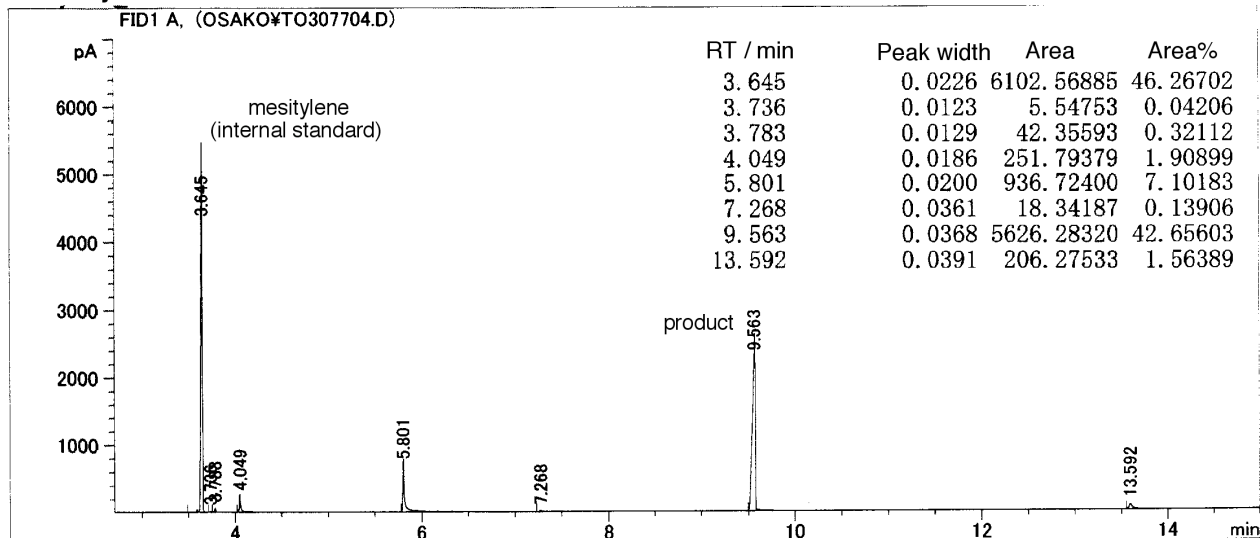
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Quality : 91
ID : 1,1'-Biphenyl, 4-methoxy-



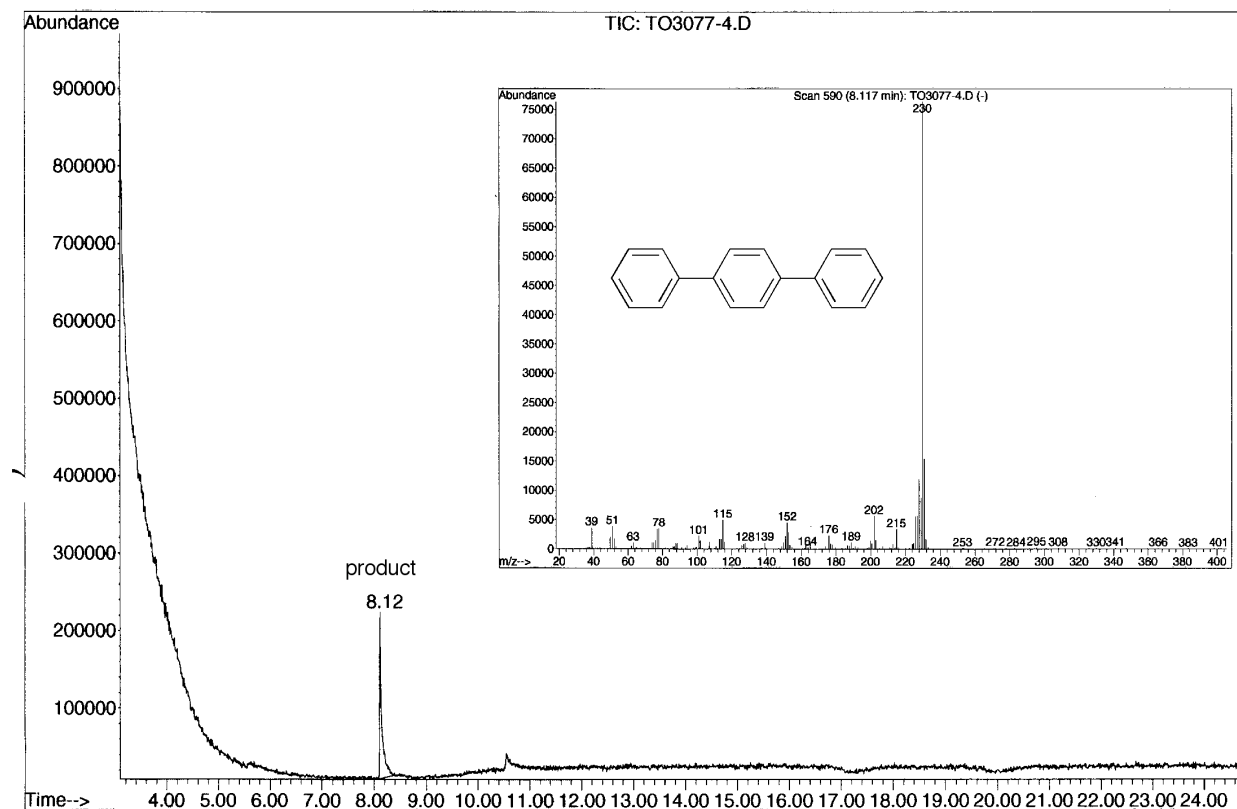
GC and GC-MS analyses for the Suzuki-Miyaura Coupling of phenyl iodide with 4-biphenylboronic acid (Table 1, entry 7).

Product: *p*-terphenyl [CAS: 92-94-4]

GC Analysis

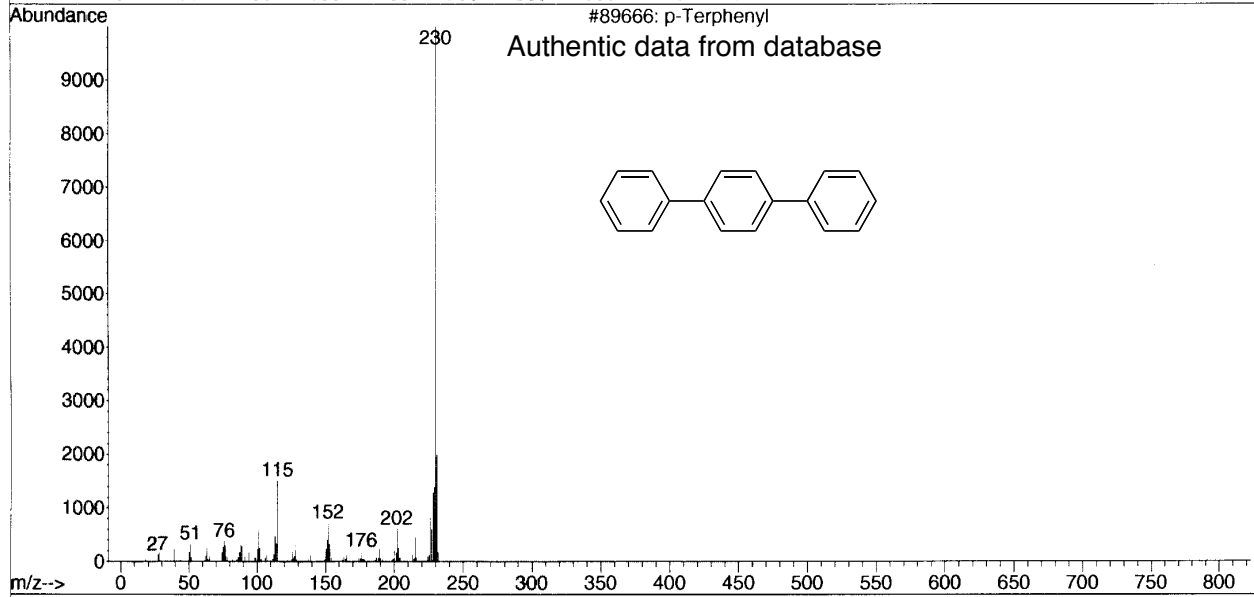
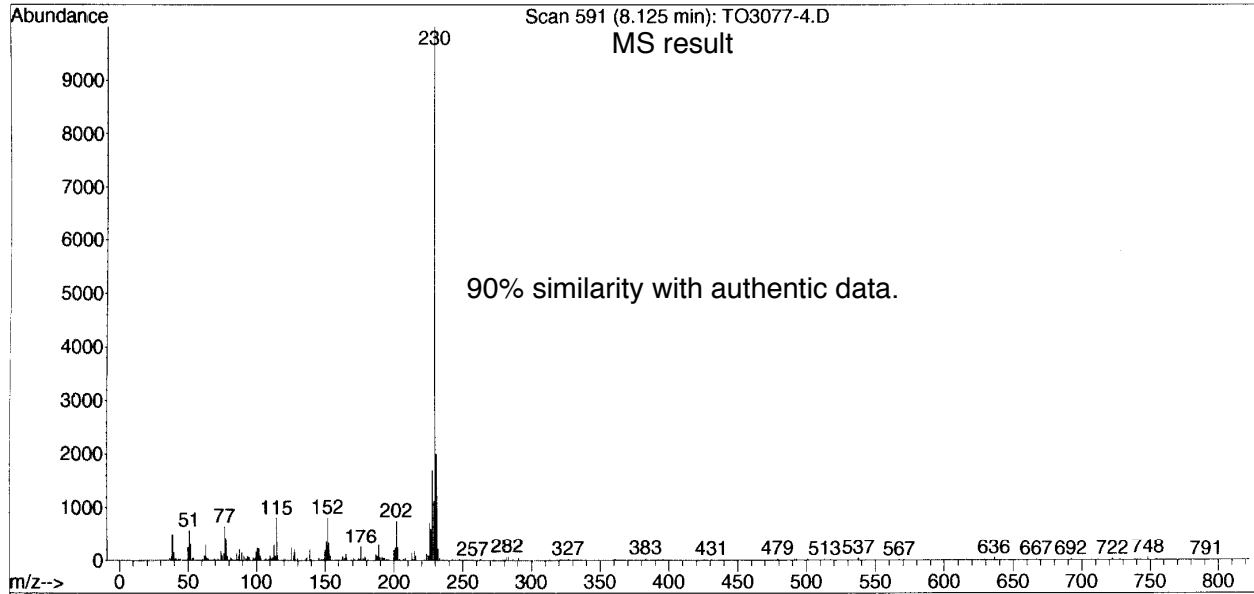


GC-MS Analysis



Comparison with NIST Mass Database Library

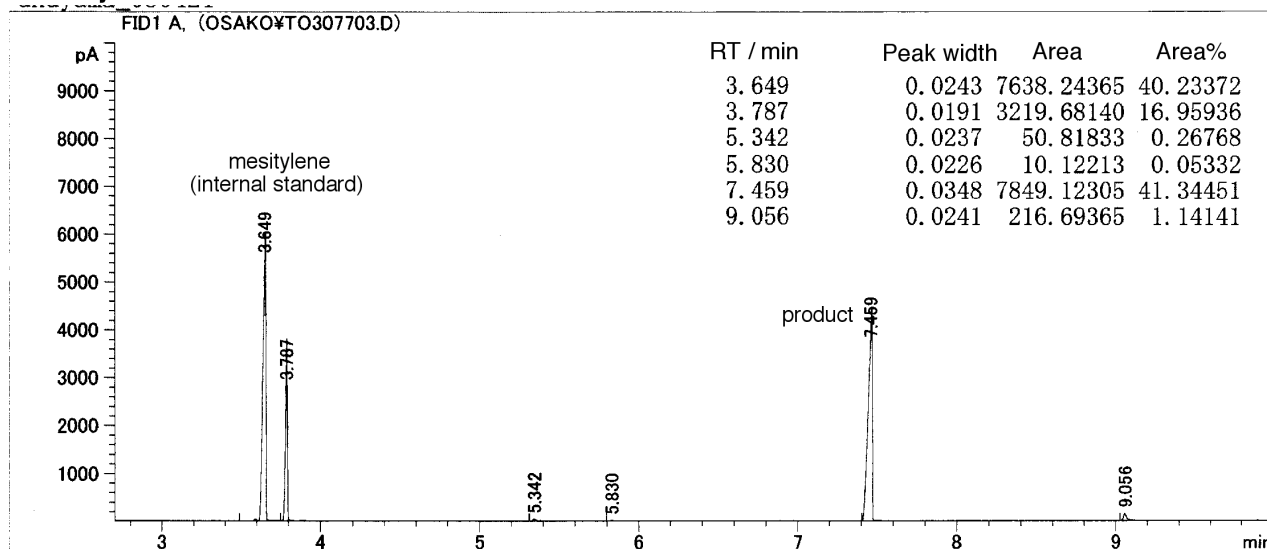
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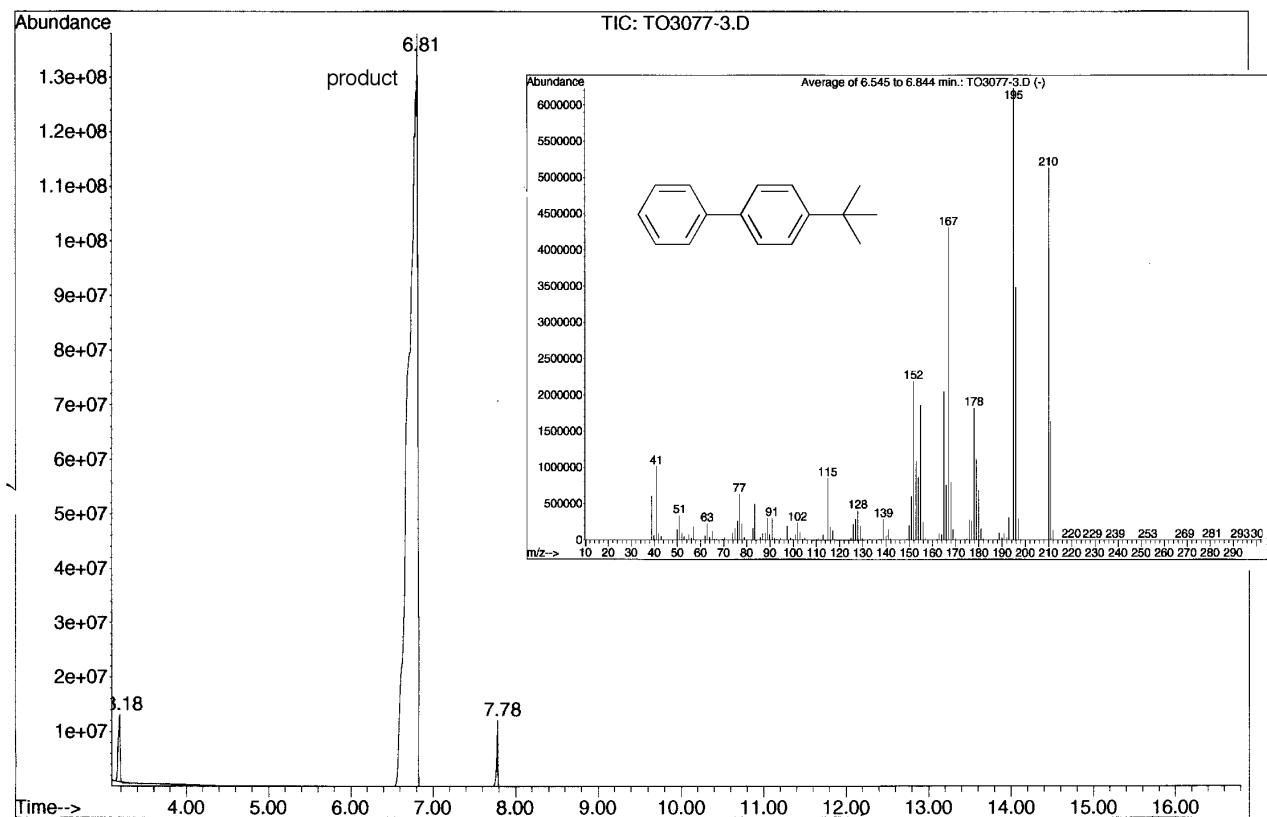
GC and GC-MS analyses for the Suzuki-Miyaura Coupling of phenyl iodide with 4-*tert*-butylphenylboronic acid (Table 1, entry 9).

Product: 4-*tert*-butylbiphenyl [CAS: 1625-92-9]

GC Analysis



GC-MS Analysis

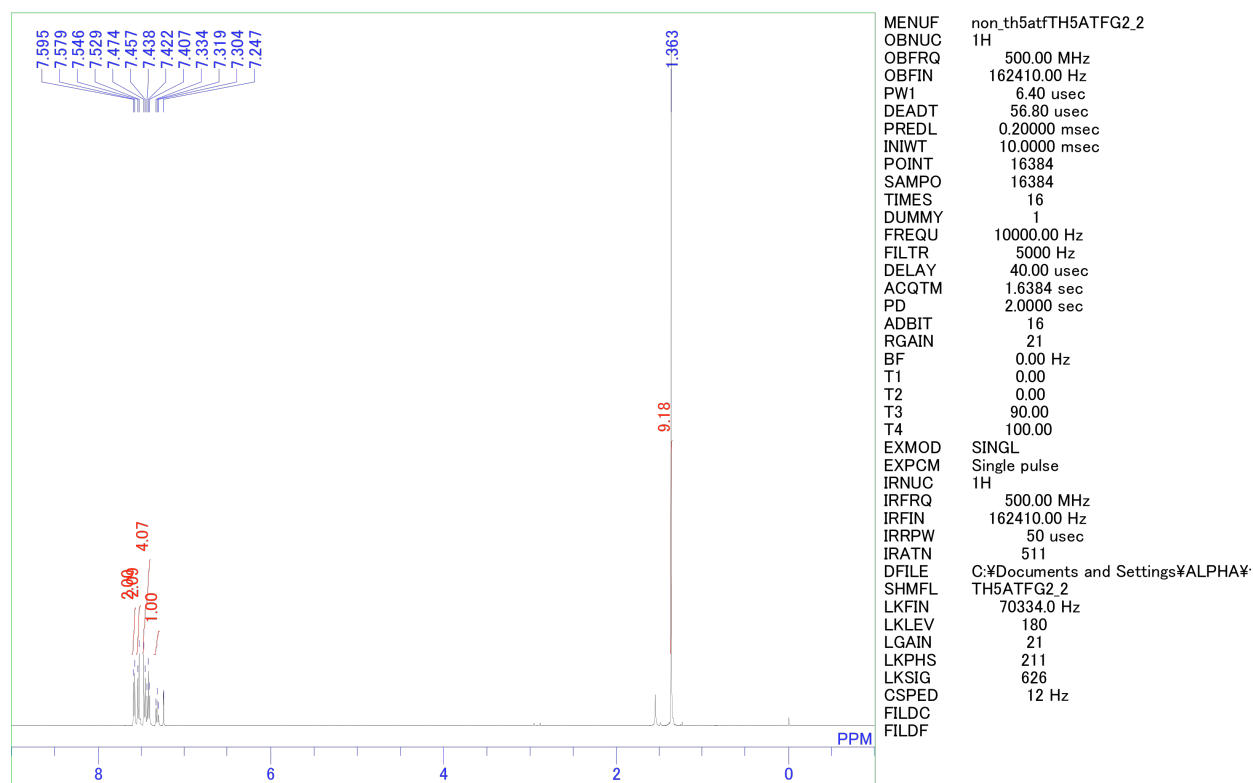


Authentic MS data of this product was not available in NIST data. Instead, ^1H and ^{13}C NMR analyses were performed. After the workup, the resulting solid was purified by silica gel column chromatography on silica gel (eluent: hexane). Isolated yield: 190 mg, 90%.

^1H NMR (500 MHz, CDCl_3): δ 7.59 (d, $J = 8.0$ Hz, 2H), 7.54 (d, $J = 8.5$ Hz, 2H), 7.47 (d, $J = 8.5$ Hz, 2H), 7.42 (t, $J = 7.5$ Hz, 2H), 7.32 (t, $J = 7.5$ Hz, 1H), 1.36 (s, 9H); ^{13}C NMR (126 MHz, CDCl_3): δ 150.2, 141.0, 138.2, 128.6, 126.9, 126.9, 126.7, 125.6, 34.5, 31.3.

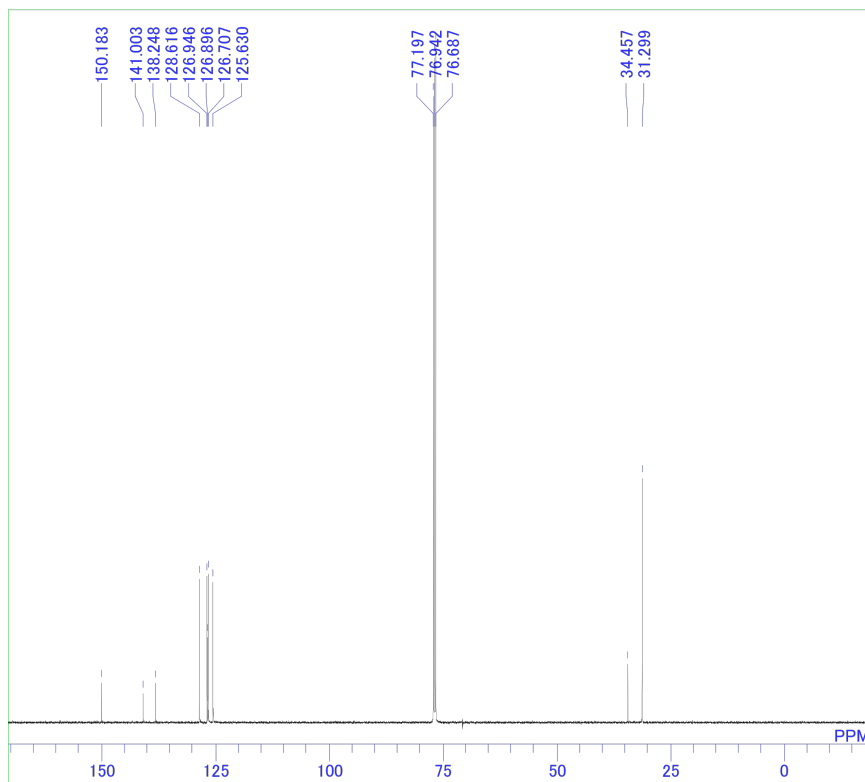
^1H NMR spectrum of 4-*tert*-butylbiphenyl

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$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of 4-*tert*-butylbiphenyl

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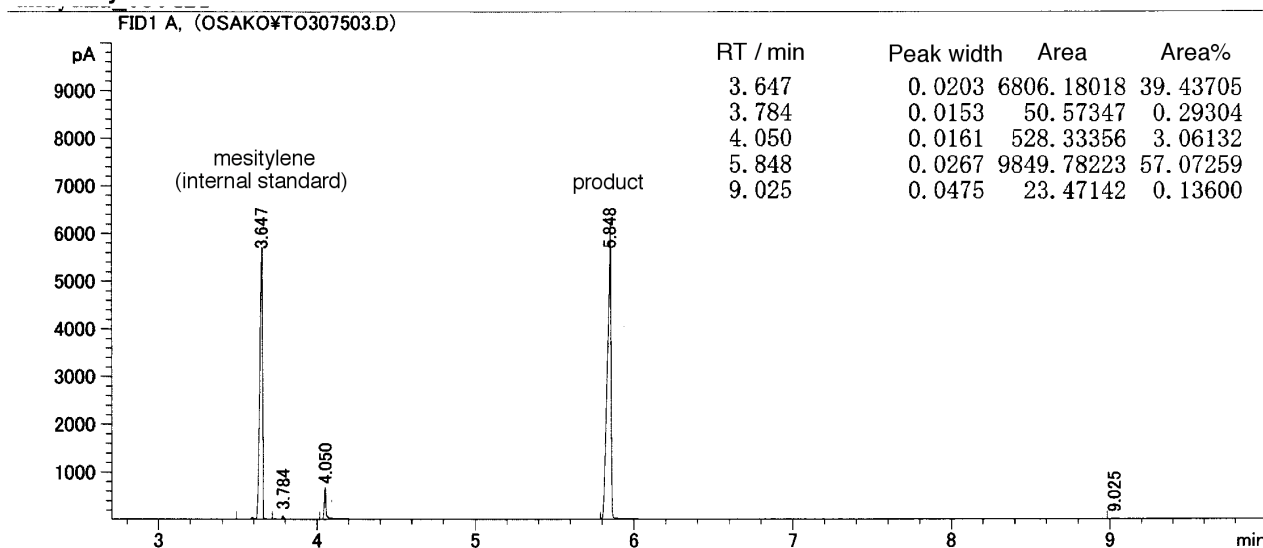


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PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 32768
SAMPO 32768
TIMES 25000
DUMMY 1
FREQU 33898.30 Hz
FILTR 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 1.0000 sec
ADBIT 16
RGAIN 27
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
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FILDF

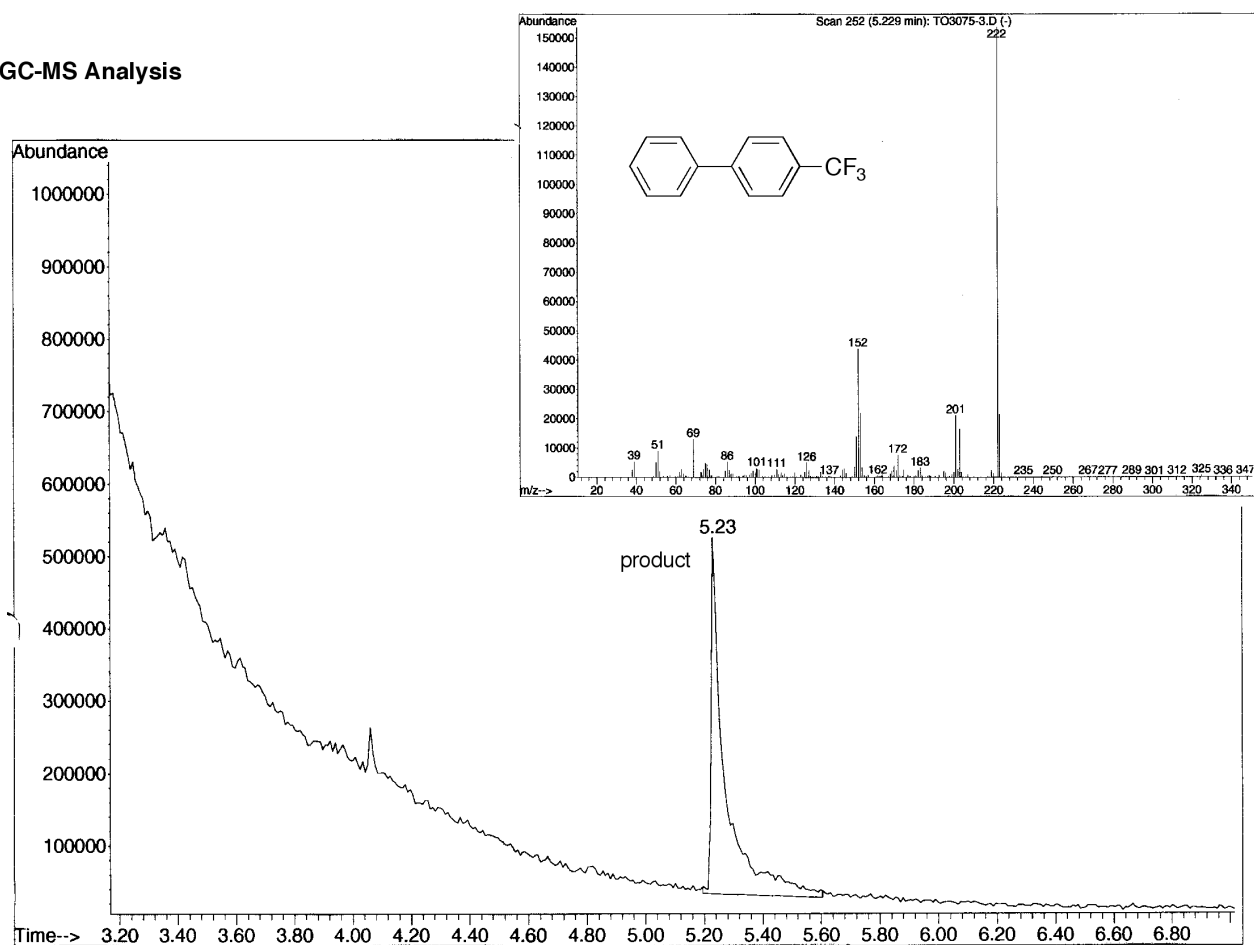
GC and GC-MS analyses for the Suzuki-Miyaura Coupling of phenyl iodide with 4-(trifluoromethyl)phenylboronic acid (Table 1, entry 11).

Product: 4-(trifluoromethyl)biphenyl [CAS: 398-36-7]

GC Analysis



GC-MS Analysis

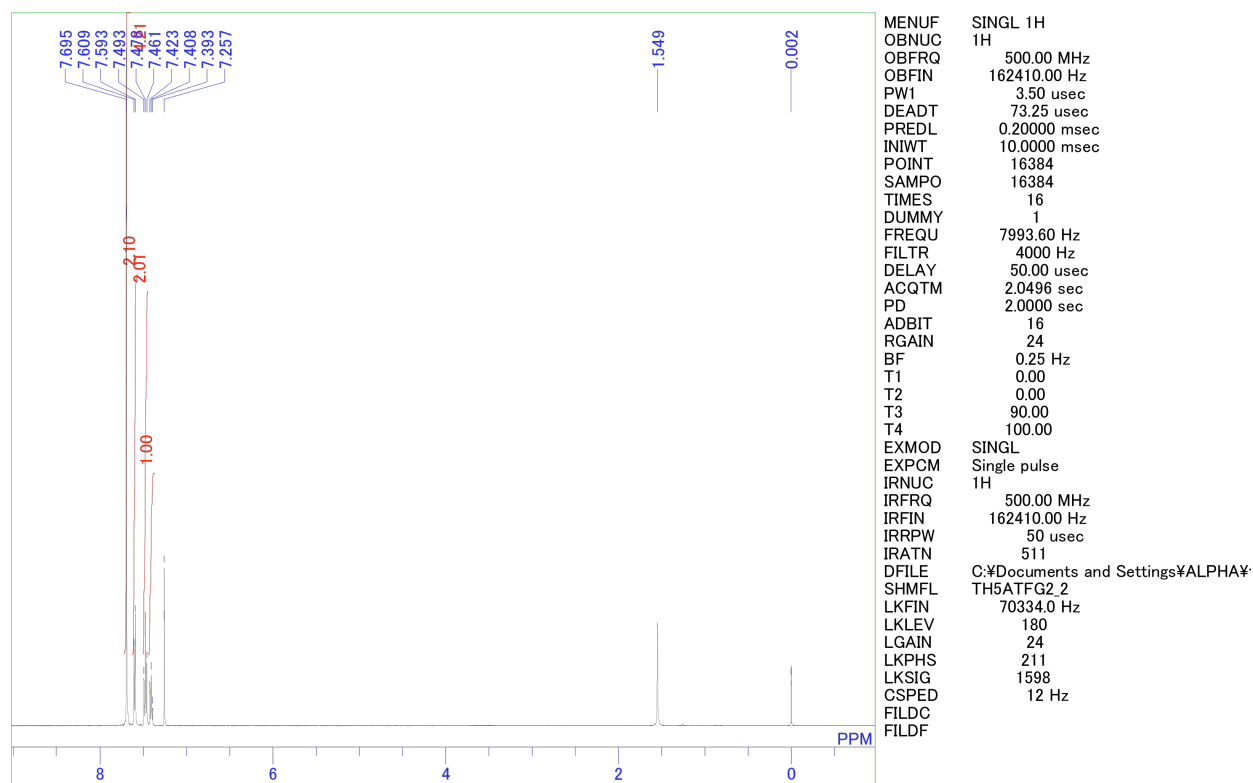


Authentic MS data of this product was not available in NIST data. Instead, ^1H and ^{13}C NMR analyses were performed. After the workup, the resulting solid was purified by silica gel column chromatography on silica gel (eluent: hexane:ethyl acetate = 9:1). Isolated yield: 184 mg, 85%.

^1H NMR (500 MHz, CDCl_3): δ 7.70 (s, 4H), 7.60 (d, $J = 7.5$ Hz, 2H), 7.48 (t, $J = 7.5$ Hz, 2H), 7.41 (t, $J = 7.5$ Hz, 1H); ^{13}C NMR (126 MHz, CDCl_3): δ 144.7, 139.8, 129.3 (q, $J = 33.2$ Hz), 129.0, 128.2, 127.4, 127.3, 125.7 (q, $J = 4.1$ Hz), 124.3 (q, $J = 271.9$ Hz).

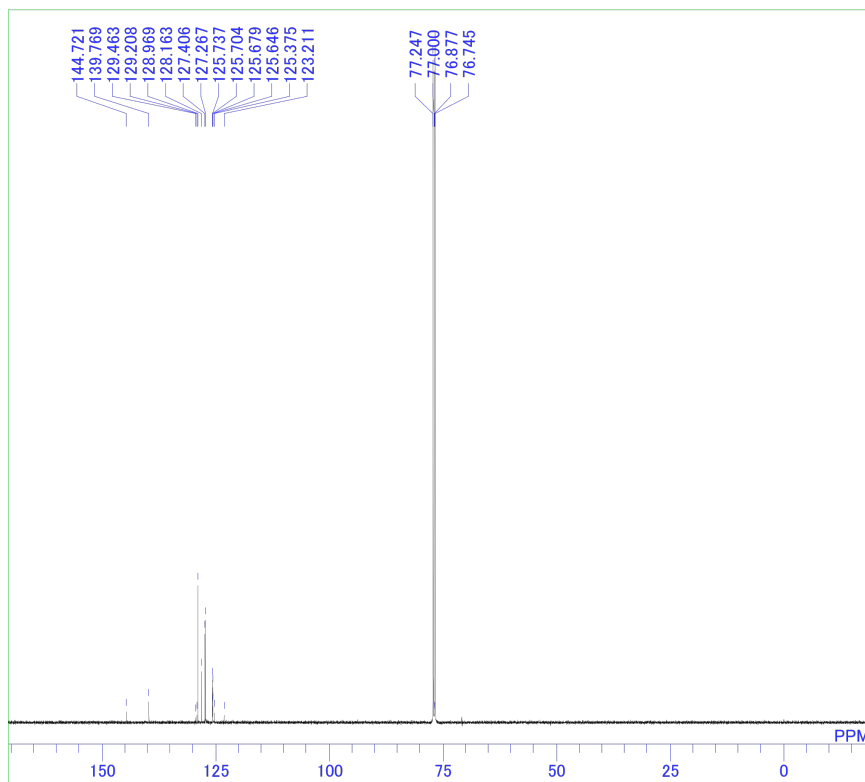
^1H NMR spectrum of 4-(trifluoromethyl)biphenyl.

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$^{13}\text{C}\{^1\text{H}\}$ NMR of 4-(trifluoromethyl)biphenyl.

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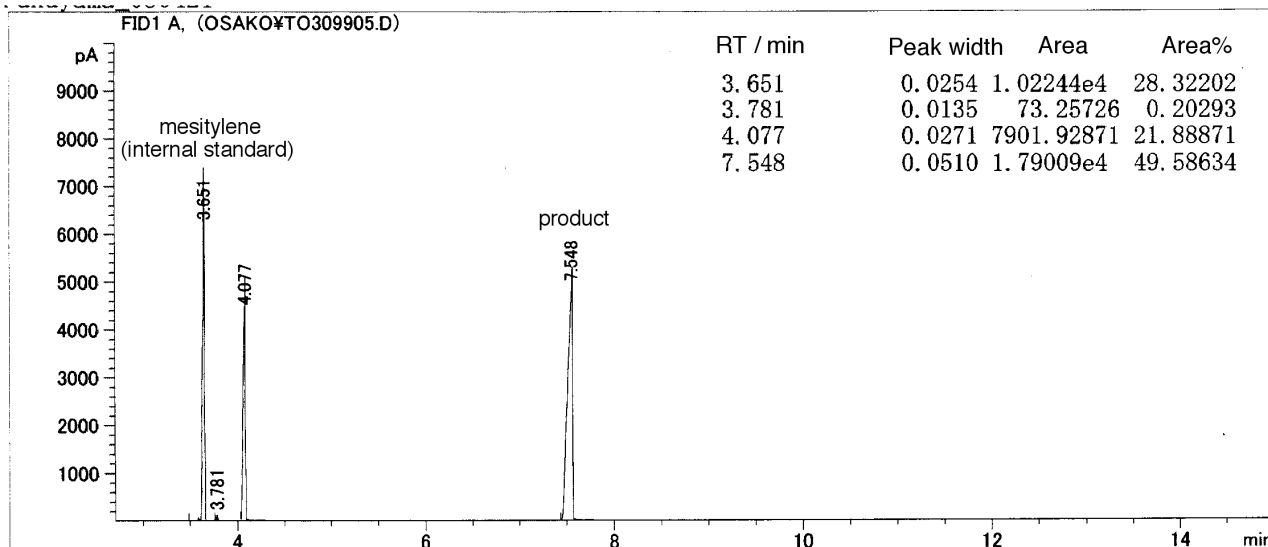


MENUP bcm
OBNUC 13C
OBFRQ 125.65 MHz
OBFIN 127958.00 Hz
PW1 5.75 usec
DEADT 10.00 usec
PREDL 0.20000 msec
INIWT 10.0000 msec
POINT 32768
SAMPO 32768
TIMES 23000
DUMMY 1
FREQU 33898.30 Hz
FILTR 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 1.0000 sec
ADBIT 16
RGAIN 27
BF 0.40 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD SINGL
EXPCM Single pulse
IRNUC 1H
IRFRQ 500.00 MHz
IRFIN 162410.00 Hz
IRRPW 50 usec
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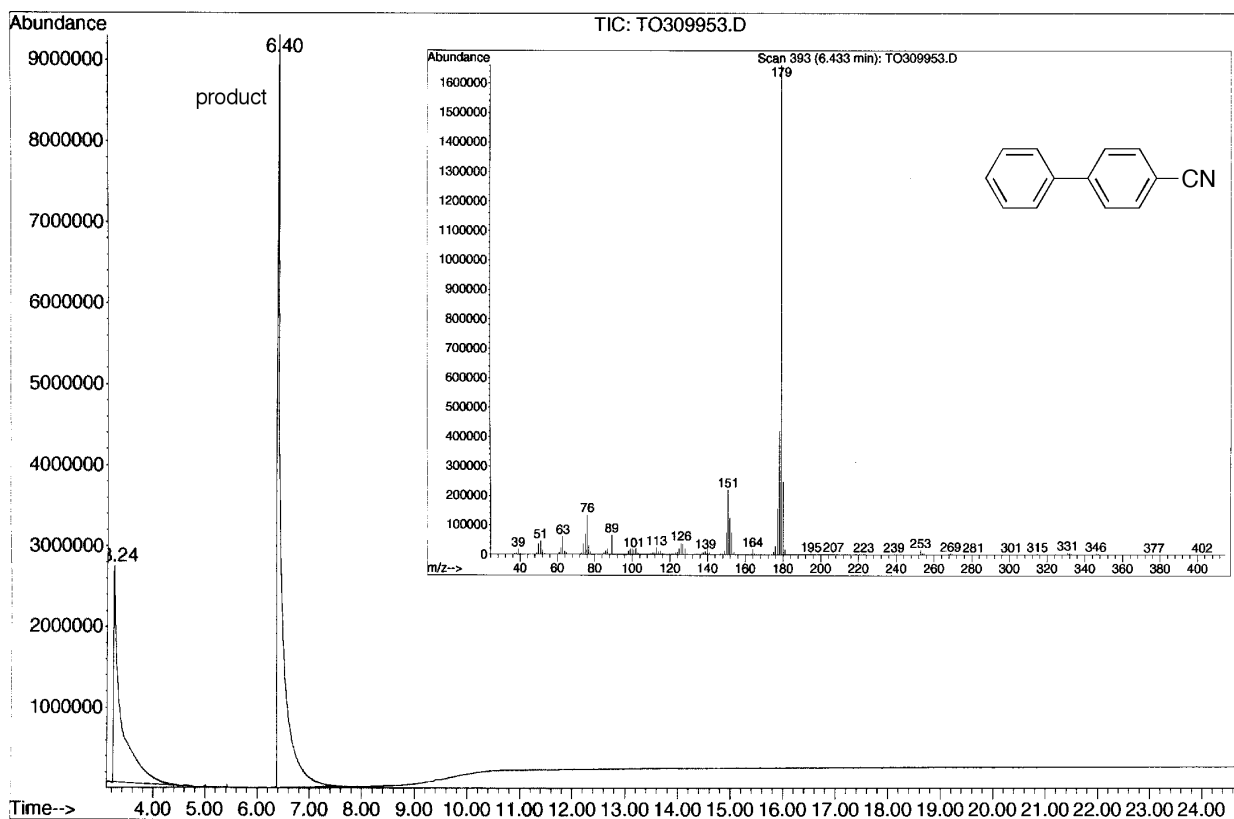
GC and GC-MS analyses for the Suzuki-Miyaura Coupling of phenyl iodide with 4-cyanophenylboronic acid (Table 1, entry 13).

Product: 4-phenylbenzonitrile [CAS: 2920-38-9]

GC Analysis

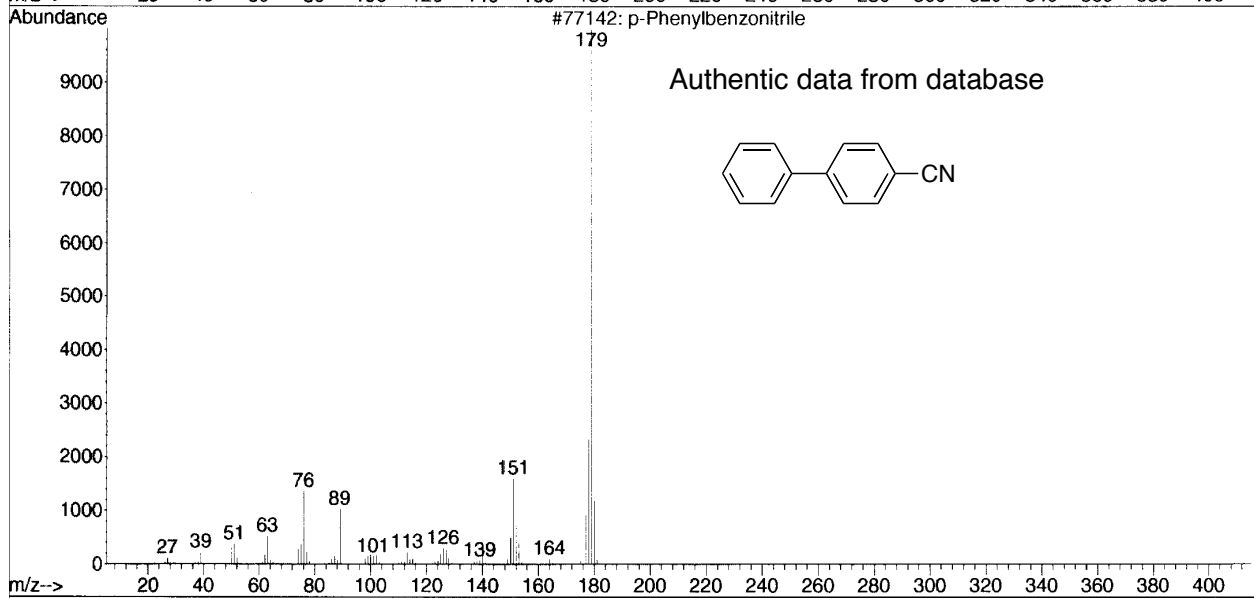
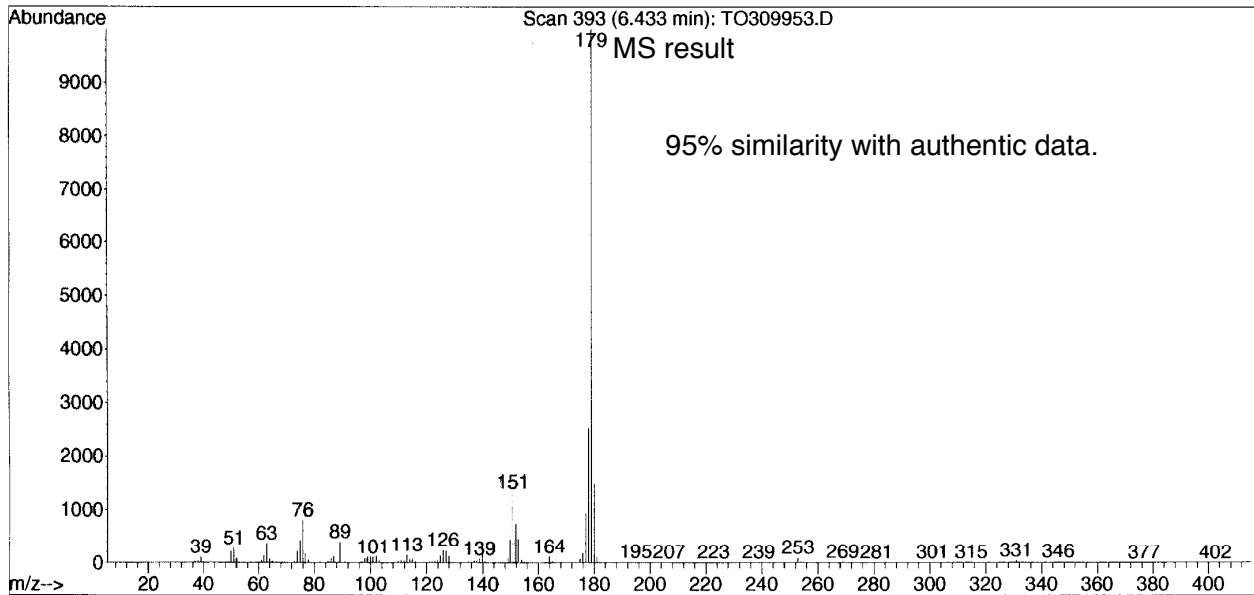


GC-MS Analysis



Comparison with NIST Mass Database Library

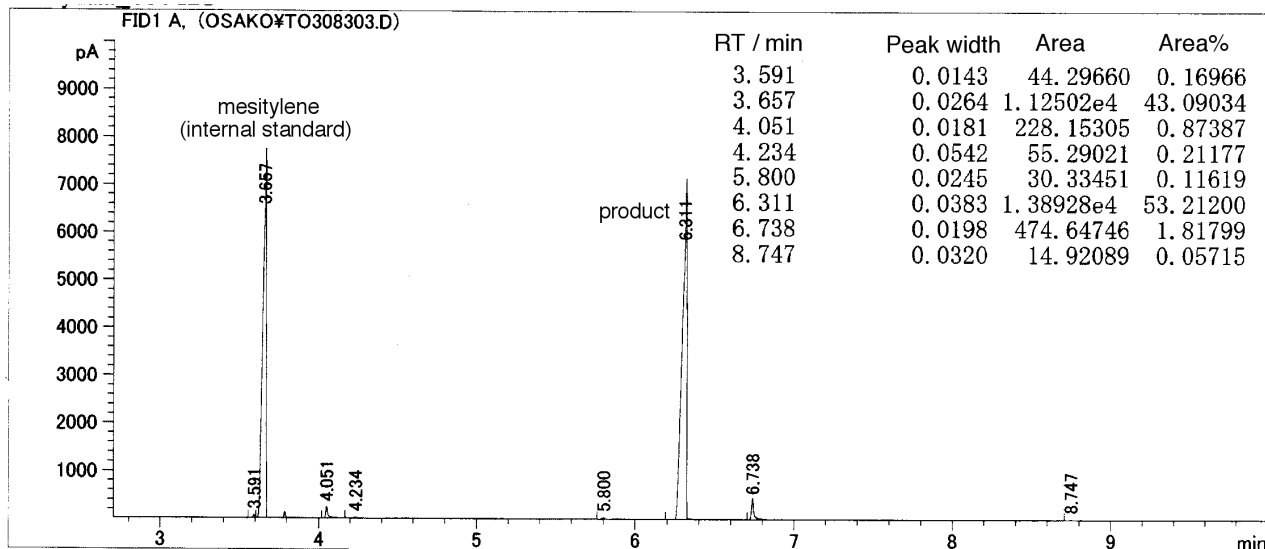
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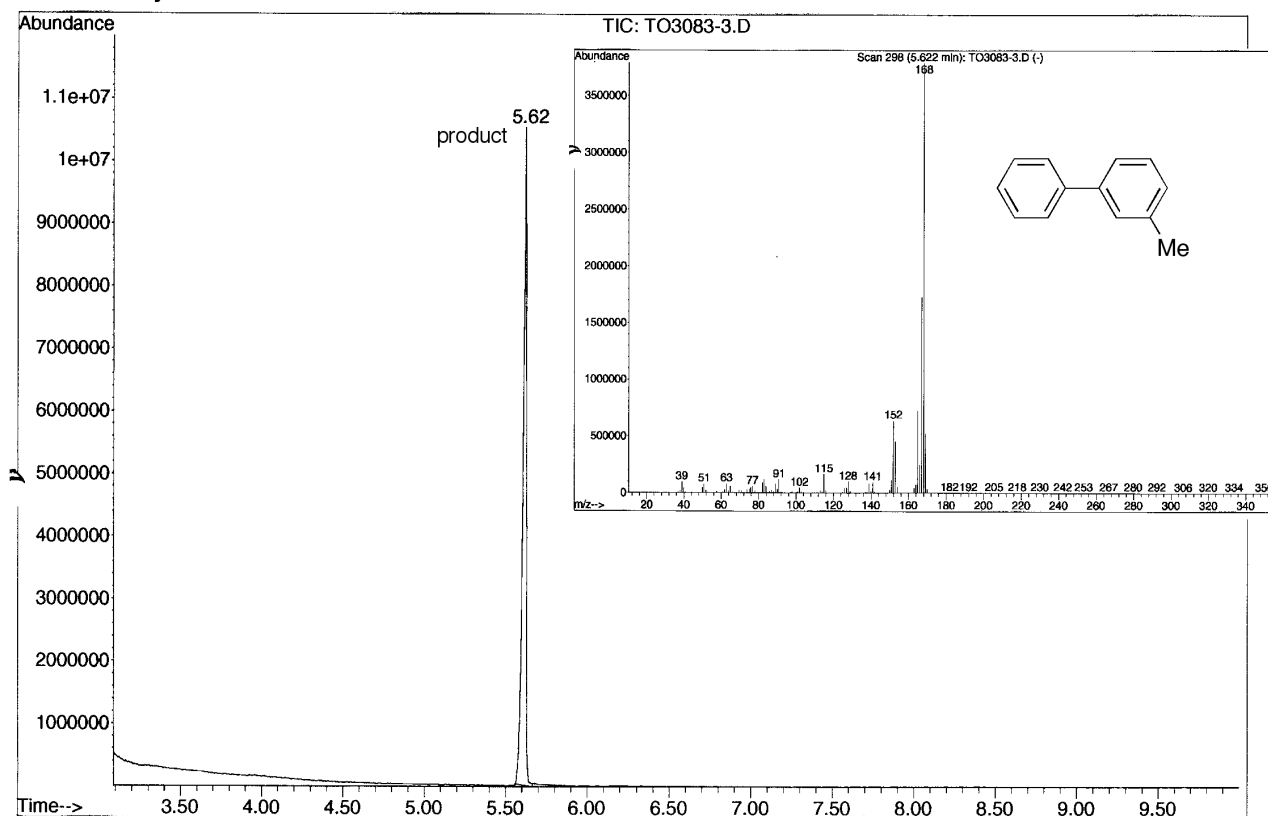
GC and GC-MS analyses for the Suzuki-Miyaura Coupling of phenyl iodide with 3-methylphenylboronic acid (Table 1, entry 15).

Product: 3-methyltoluene [CAS: 643-93-6]

GC Analysis

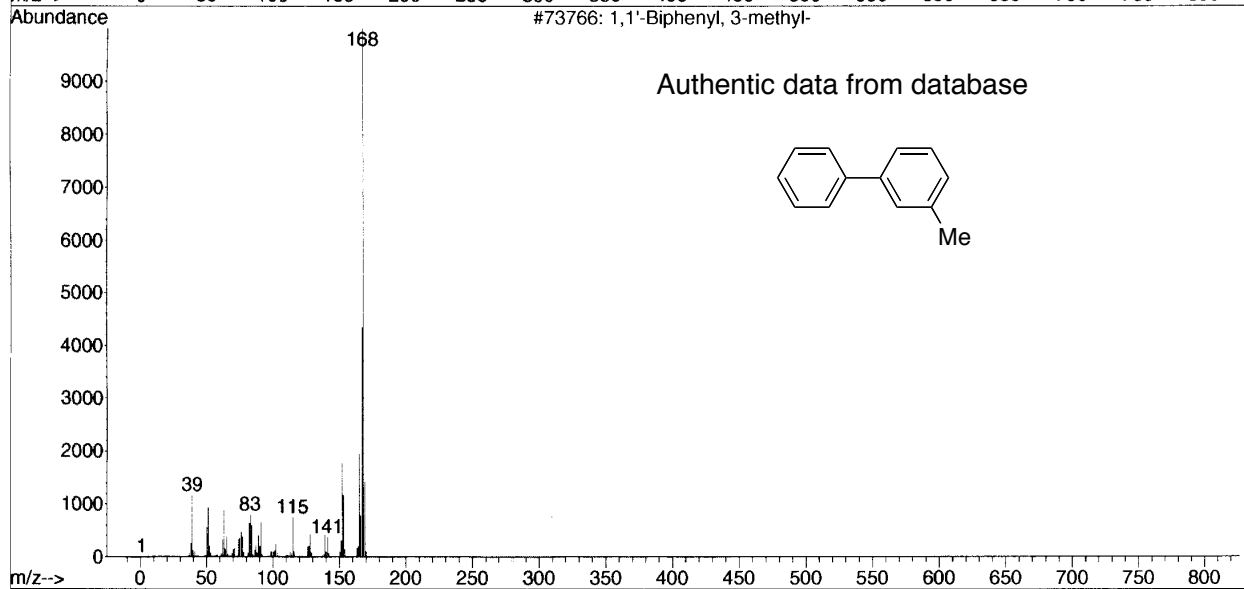
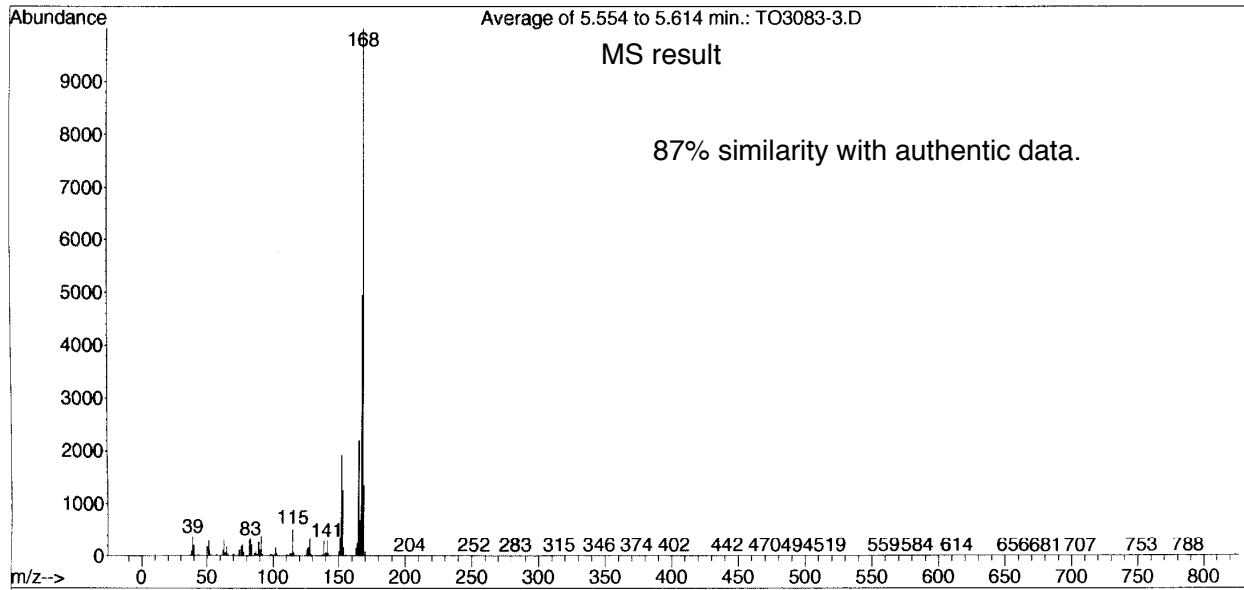


GC-MS Analysis



Comparison with NIST Mass Database Library

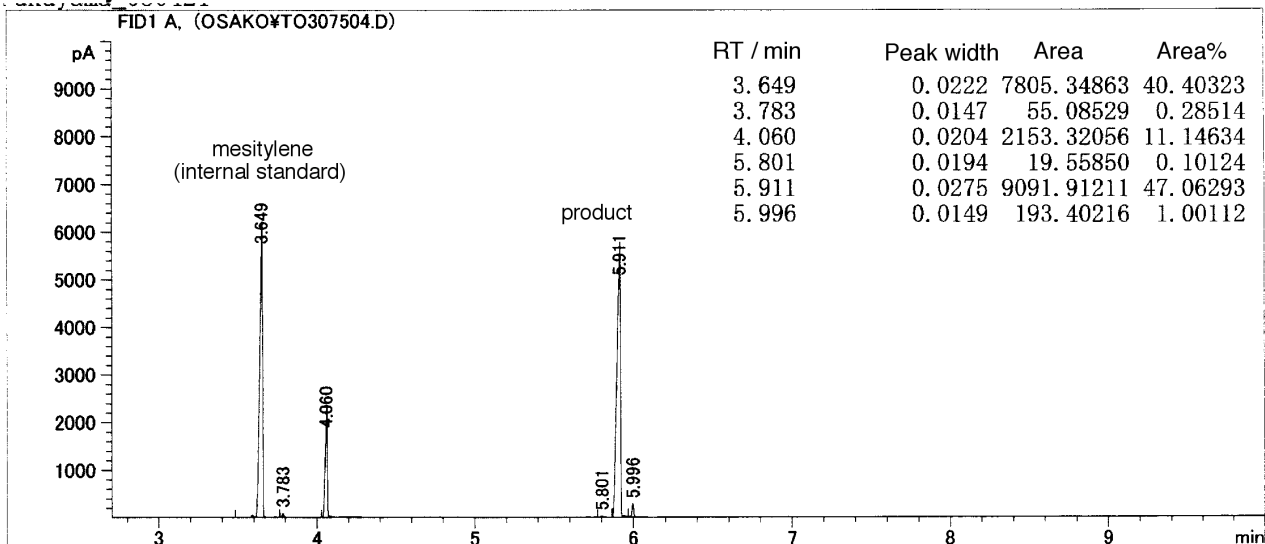
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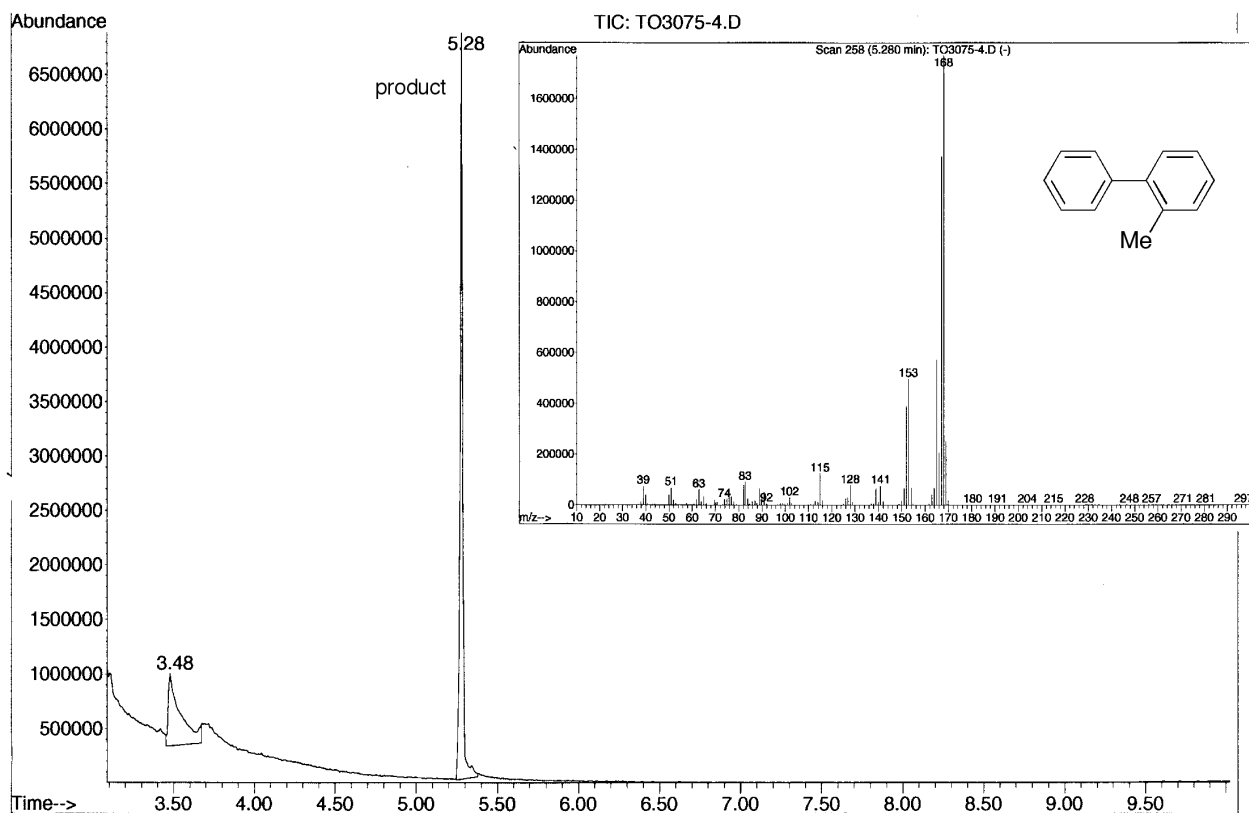
GC and GC-MS analyses for the Suzuki-Miyaura Coupling of phenyl iodide with 2-methylphenylboronic acid (Table 1, entry 17).

Product: 2-methyltoluene [CAS: 643-58-3]

GC Analysis

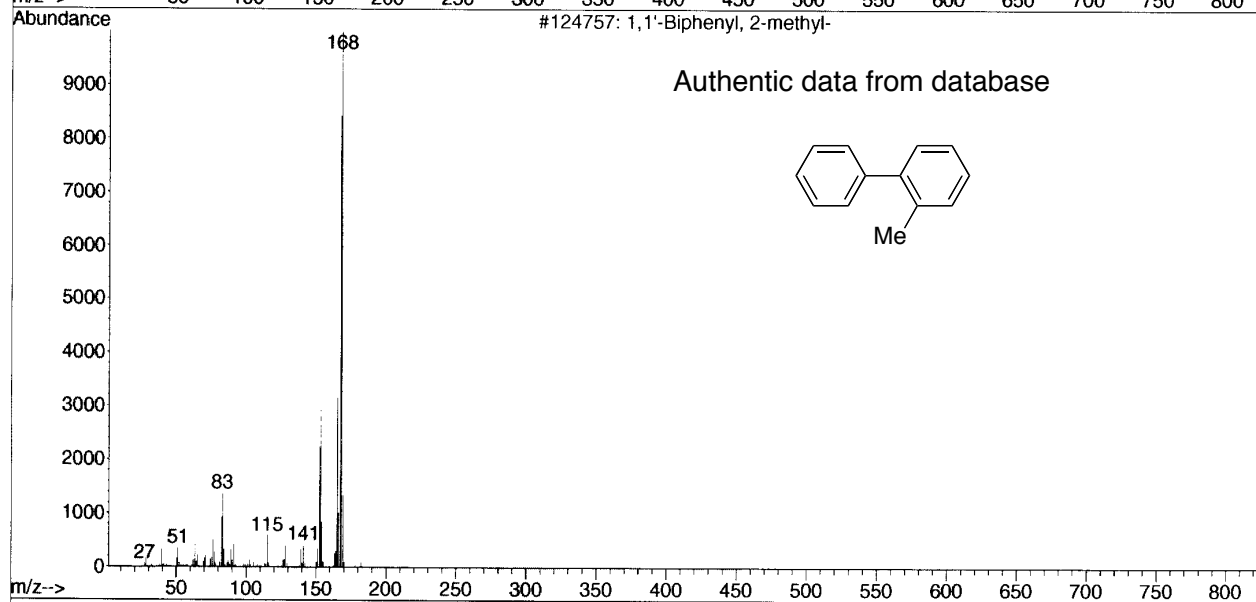
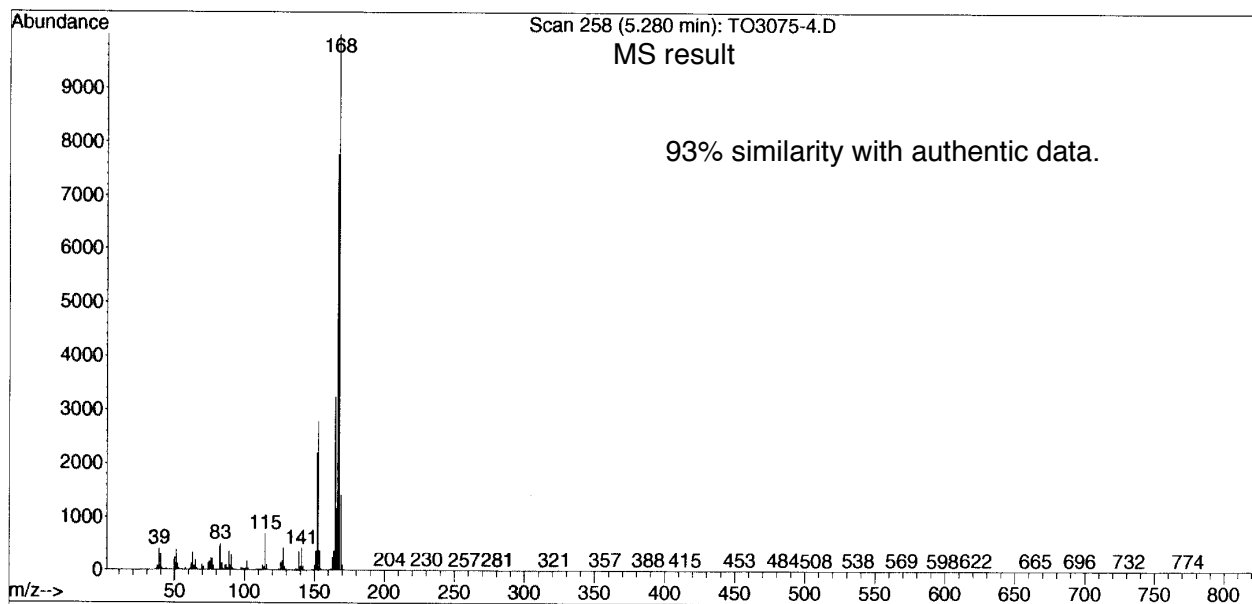


GC-MS Analysis



Comparison with NIST Mass Database Library

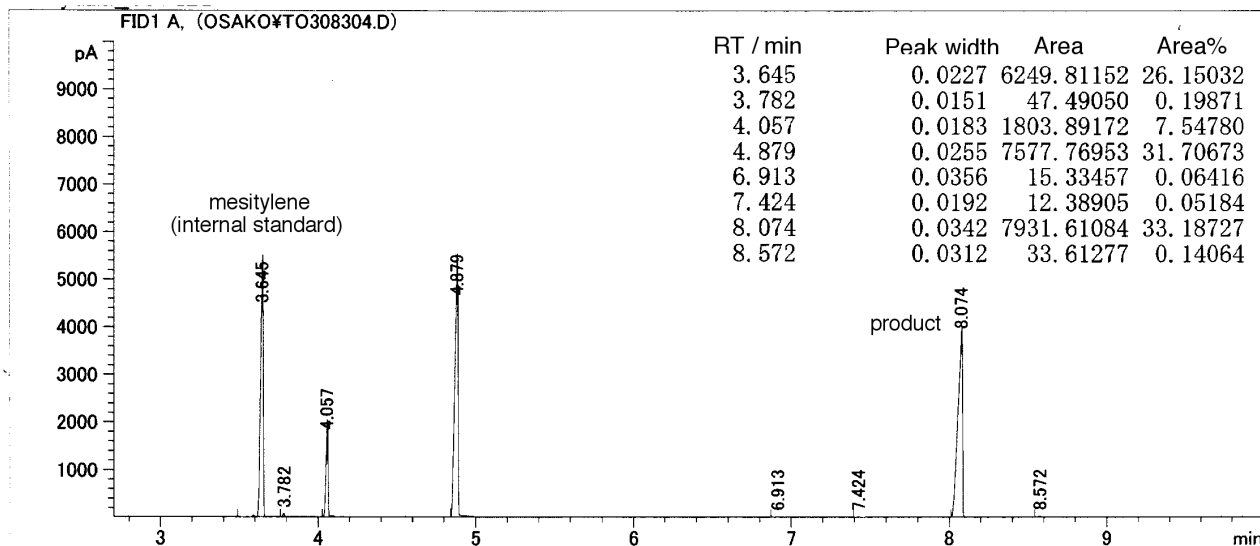
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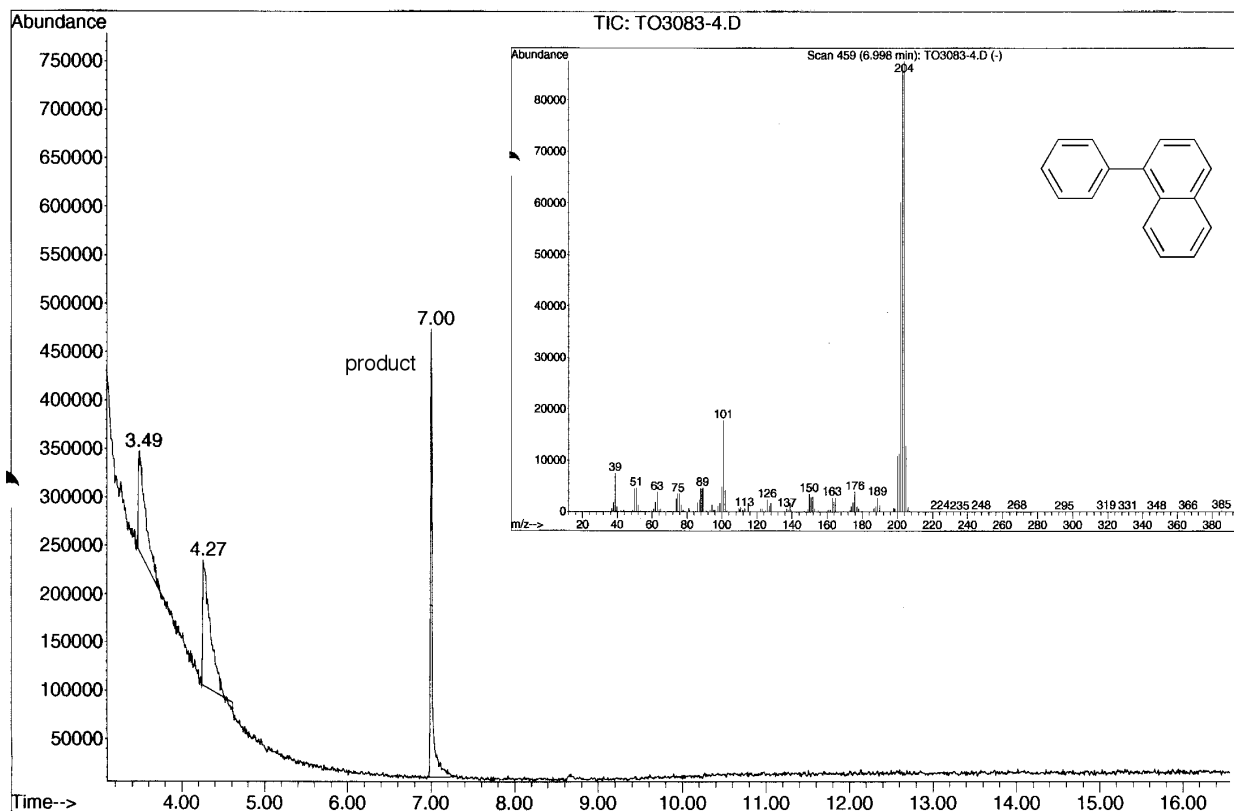
GC and GC-MS analyses for the Suzuki-Miyaura Coupling of phenyl iodide with 1-naphthylboronic acid (Table 1, entry 19).

Product: 1-phenylnaphthalene [CAS: 605-02-7]

GC Analysis



GC-MS Analysis



Comparison with NIST Mass Database Library

Library Searched : C:\Database\Nist98.1
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ID : Naphthalene, 1-phenyl-

