Aza-Wittig access to chiral imidazol(in)es

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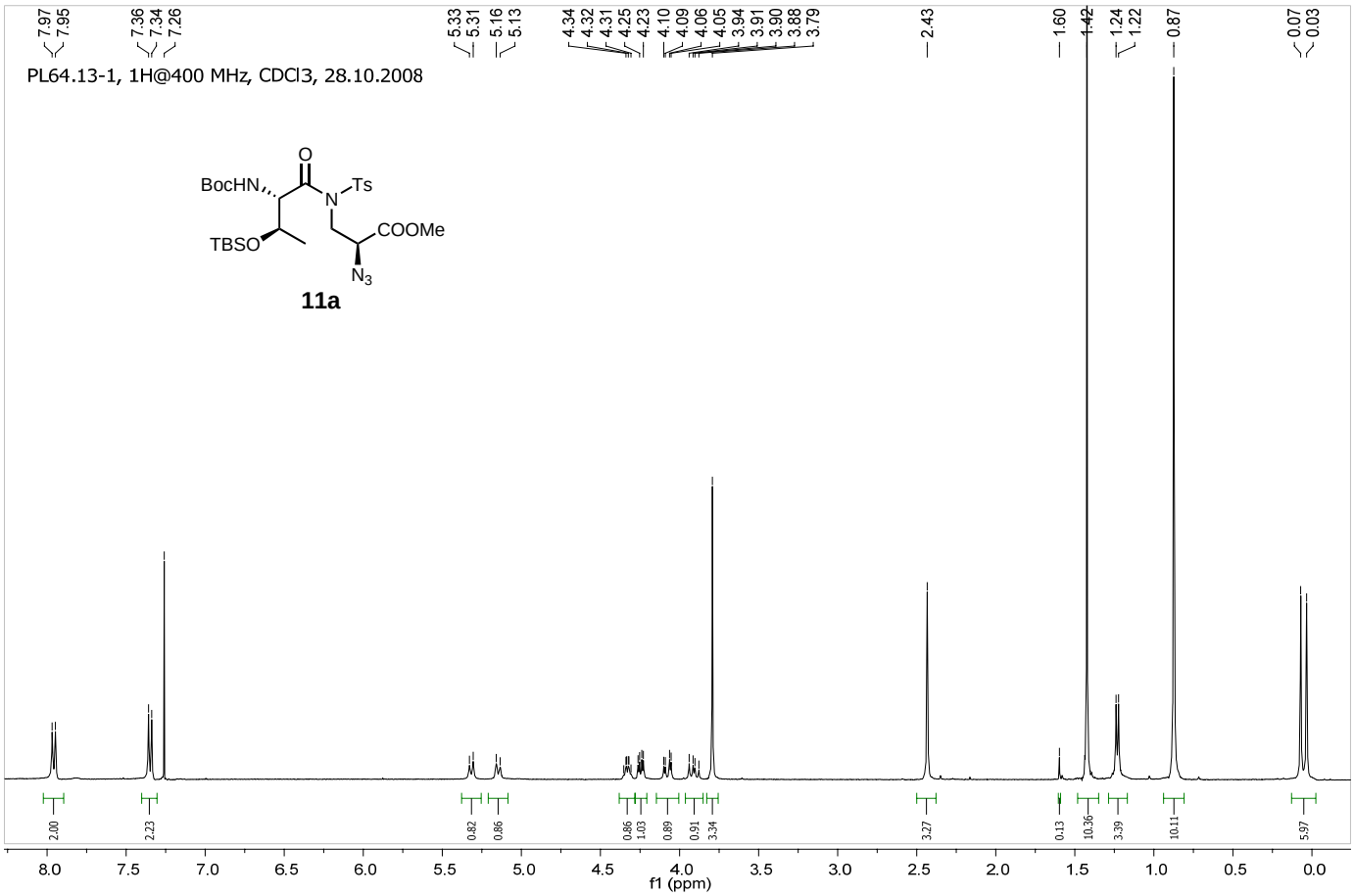
Electronic Supporting Information (10 pages)

1. General procedure for sulfonamide acylation: The carboxylic acid donor (1.1 equiv.) was dissolved in dry CH_2Cl_2 (1 M) and cooled to 0°C. HATU (1.1 equiv) and predried Cs_2CO_3 (1.0 eqiv) were added. The suspension was diluted with CH_2Cl_2 (to 0.25 M) and stirred for 15 minutes. A solution of the sulfonamide (1.0 equiv) in CH_2Cl_2 (0.25 M) and more predried Cs_2CO_3 (3 equiv.) were added. The reaction mixture was stirred at 0°C until conversion was complete (1-4 h, TLC control). The mixture was partitioned against 5% citric acid (final pH 5), the organic layer concentrated and the product retrieved by silica gel chromatography (*n*-hexane/EtOAc).

2. General procedure for aza-Wittig ring closure: The *N*-acylated sulfonamide was dissolved in THF or 2,6-lutidine (20 mM) and cooled to –20 °C. PPh_3 (1.5 equiv.) was added and the mixture was heated to 80 °C. After complete conversion (1-6 h, TLC control) the solvent was removed in vacuo and the product was purified by silica gel chromatography (*n*-hexane/EtOAc).

3. ^{1}H-NMR spectra of compounds **11a-g**, **12a-g**, **13a-c**, and **16** (9 pages).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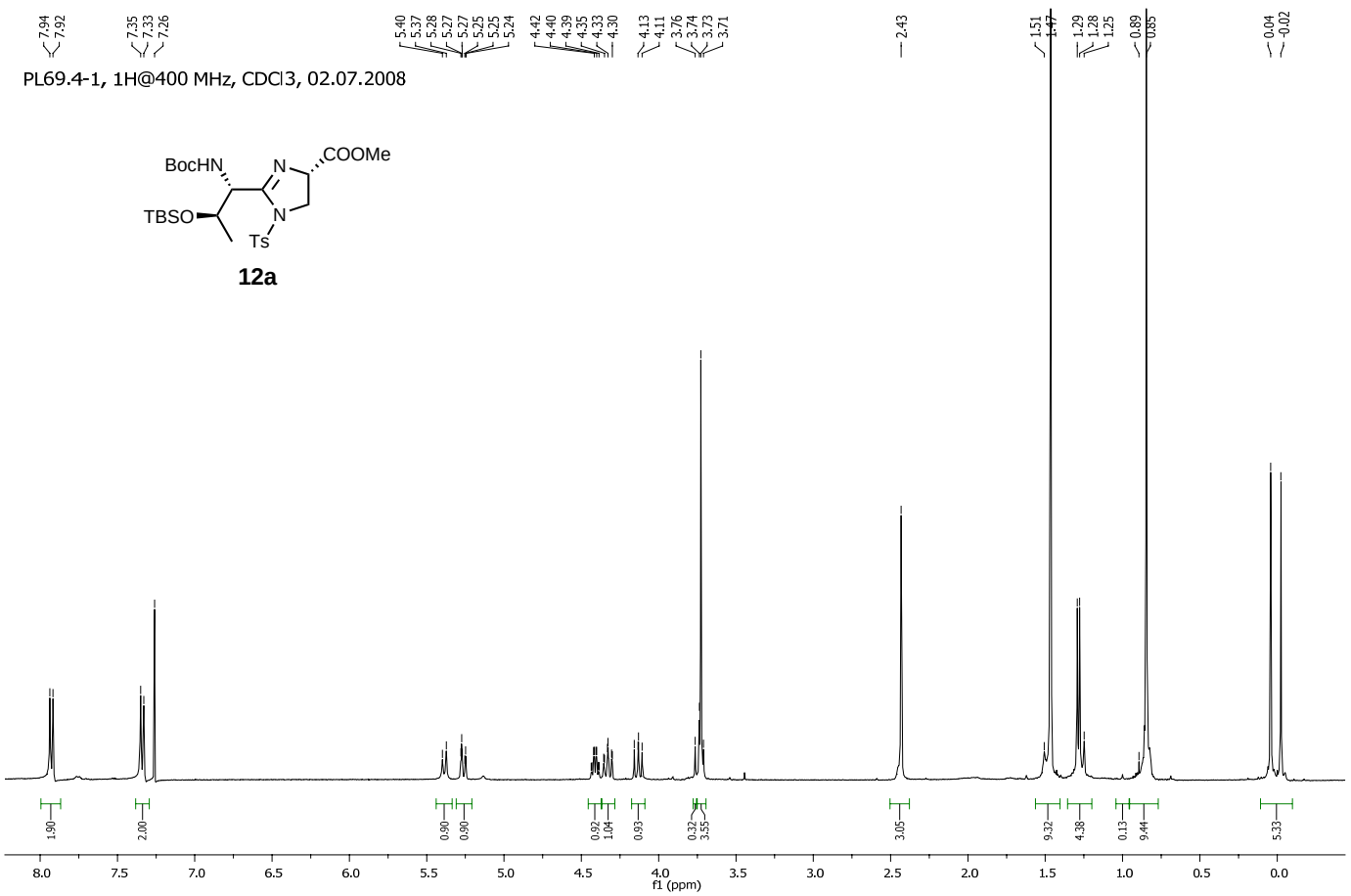
O
N
Ts
TBSO
COOMe
N3
11a
f1 (ppm)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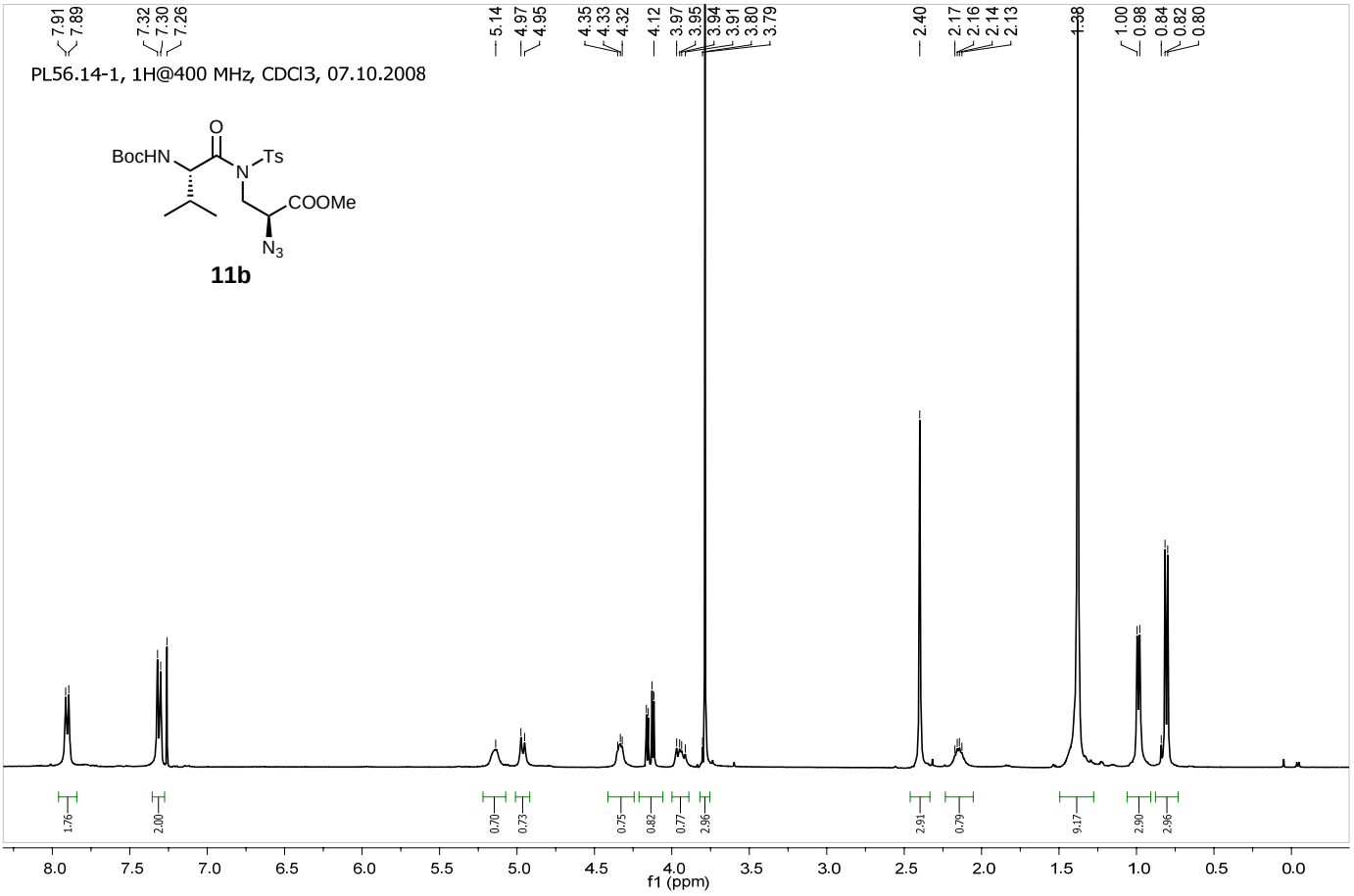
COOMe
TBSO
N
Ts
12a
f1 (ppm)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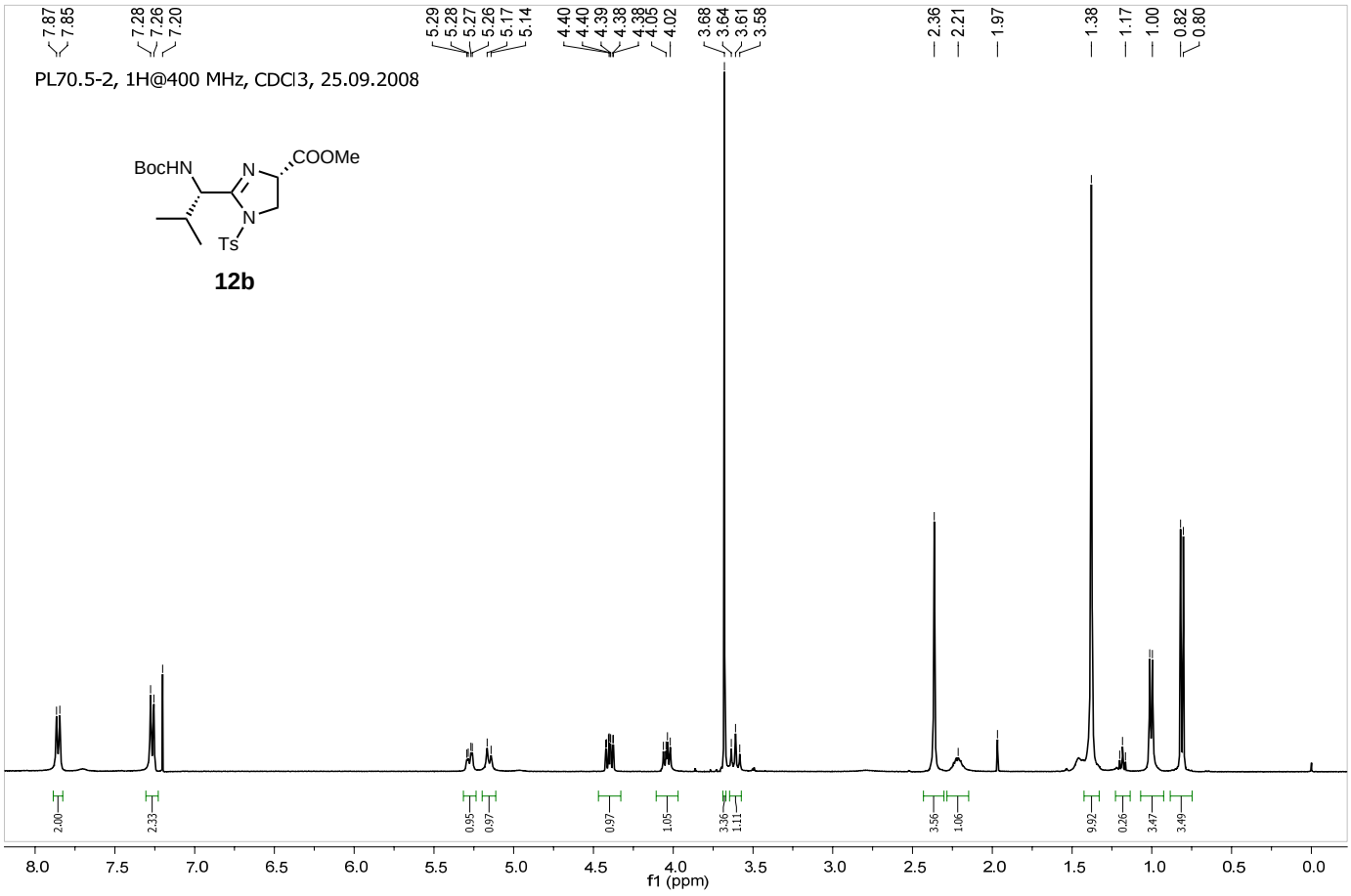
N
Ts
COOMe
N3
11b
f1 (ppm)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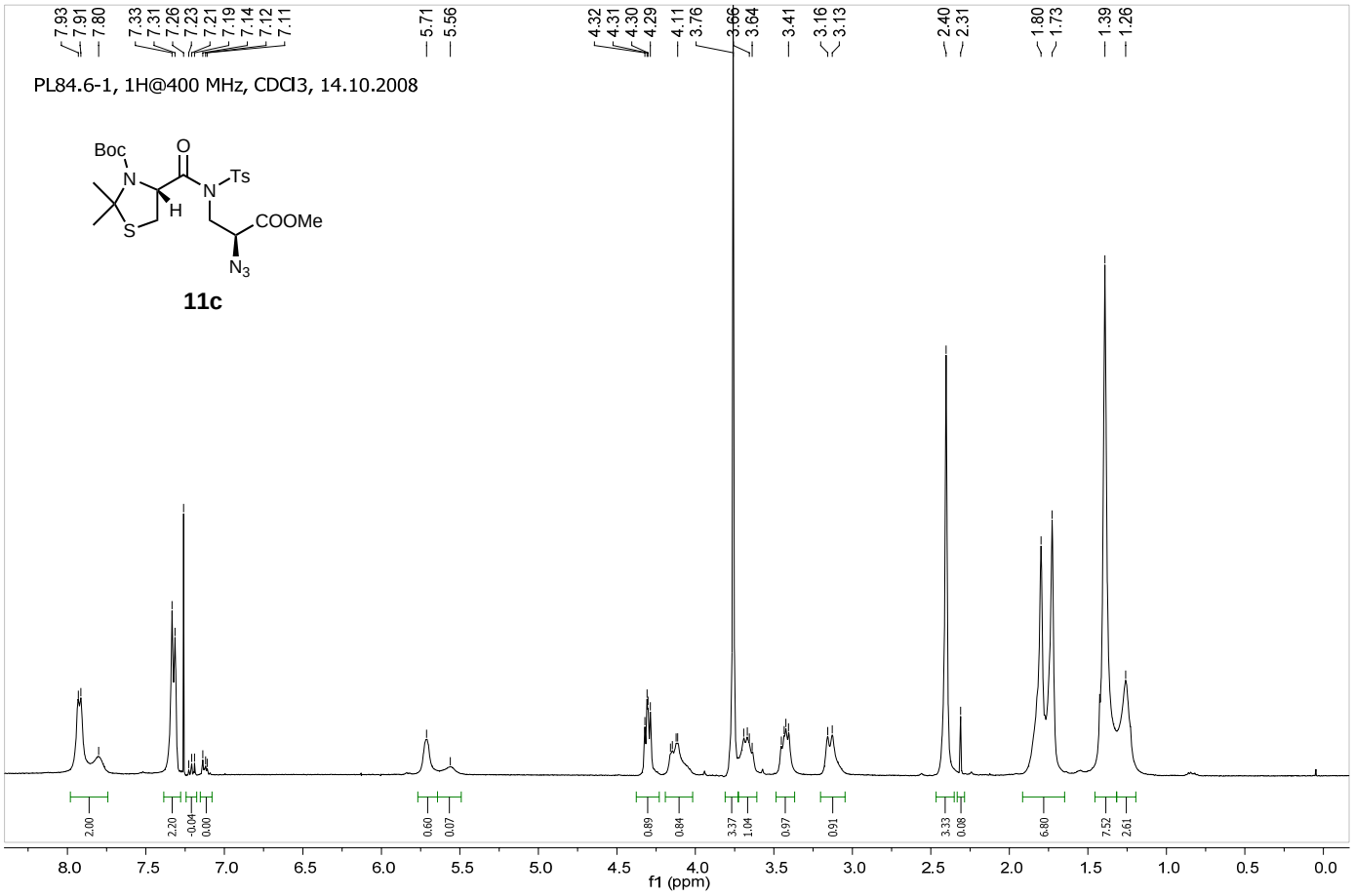
COOMe
N
Ts
12b
f1 (ppm)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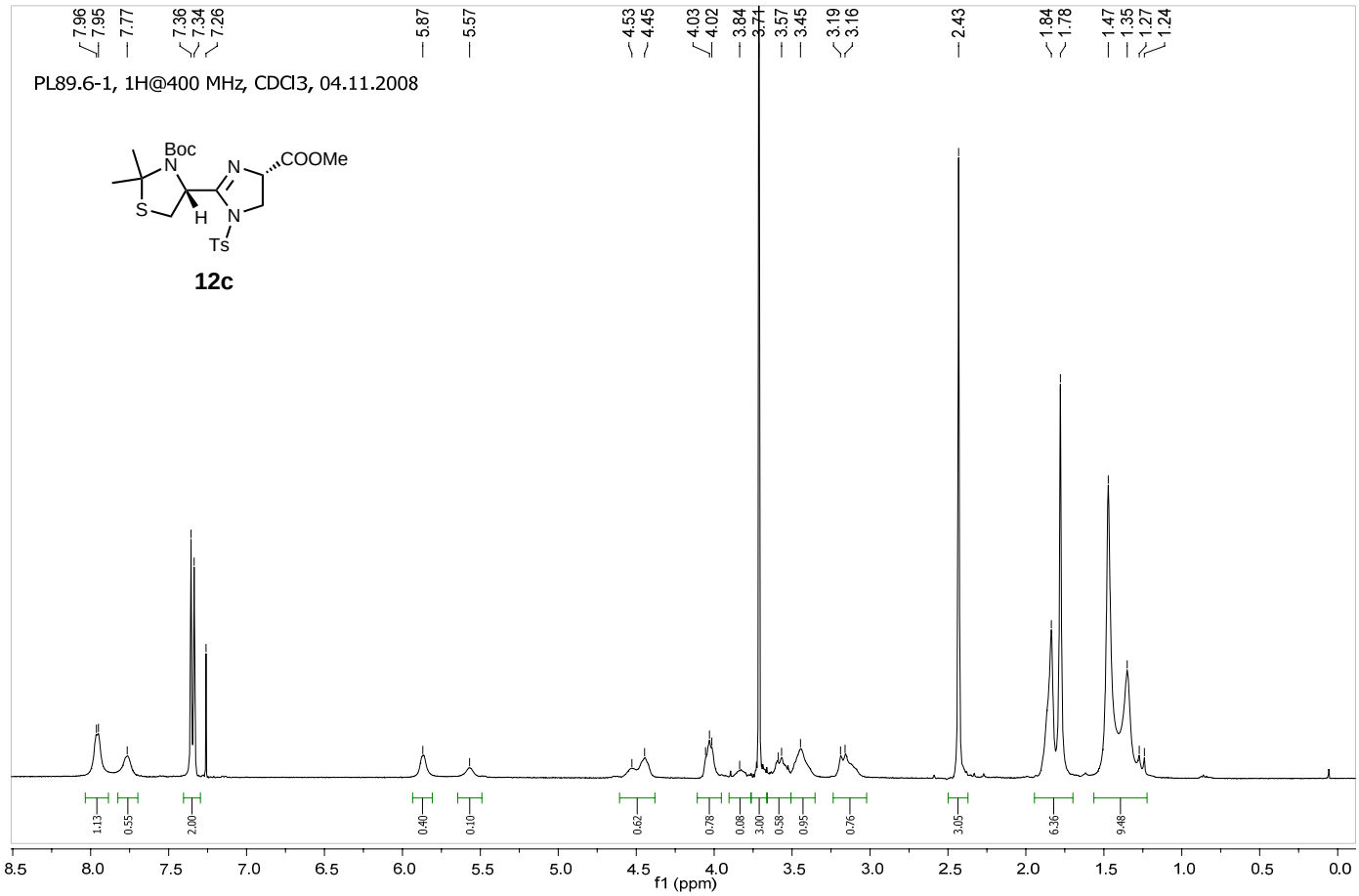
N
Ts
N
H
S
COOMe
N3
11c
f1 (ppm)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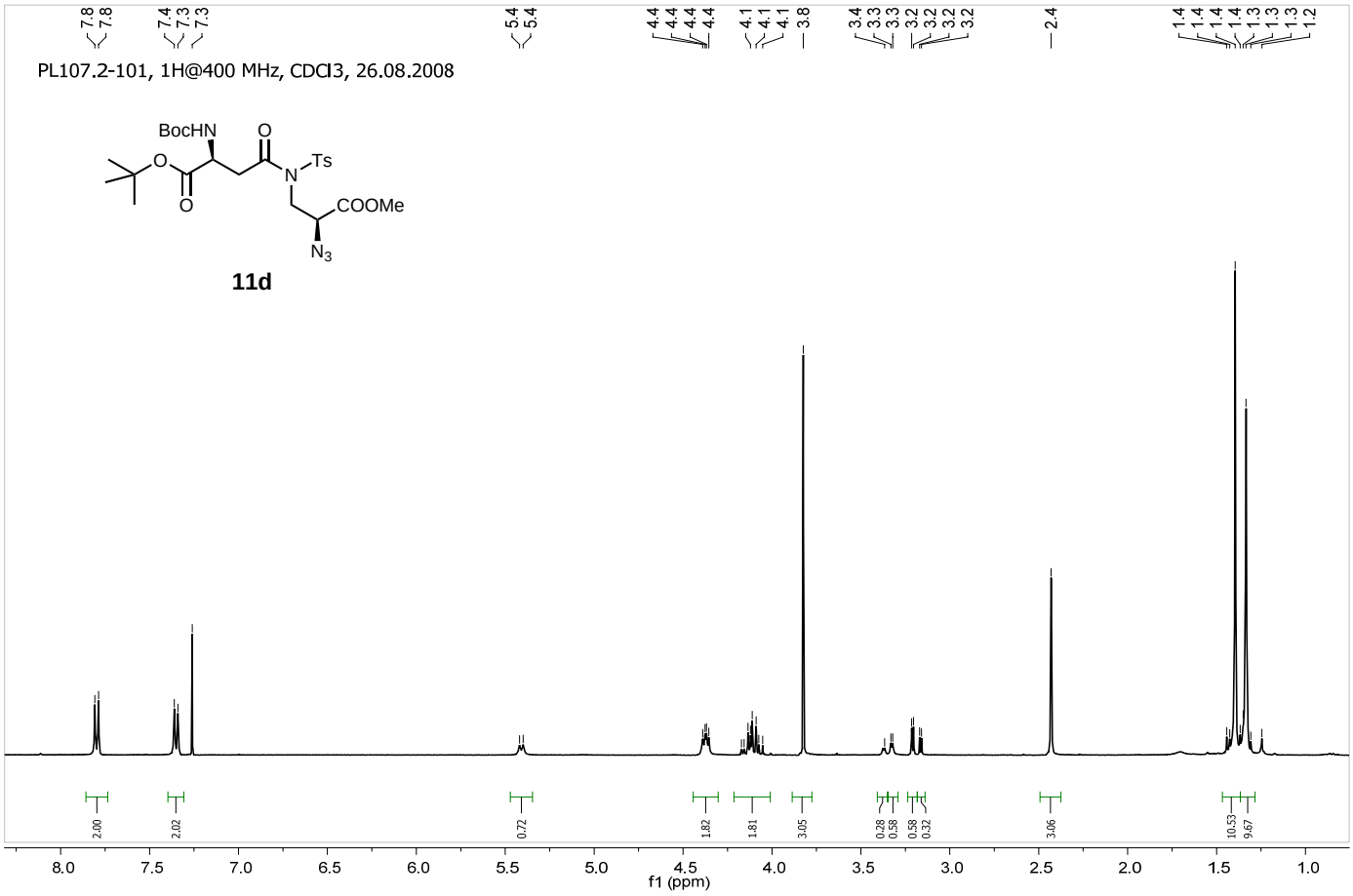
Boc
N
N
COOMe
S
H
N
Ts
12c
f1 (ppm)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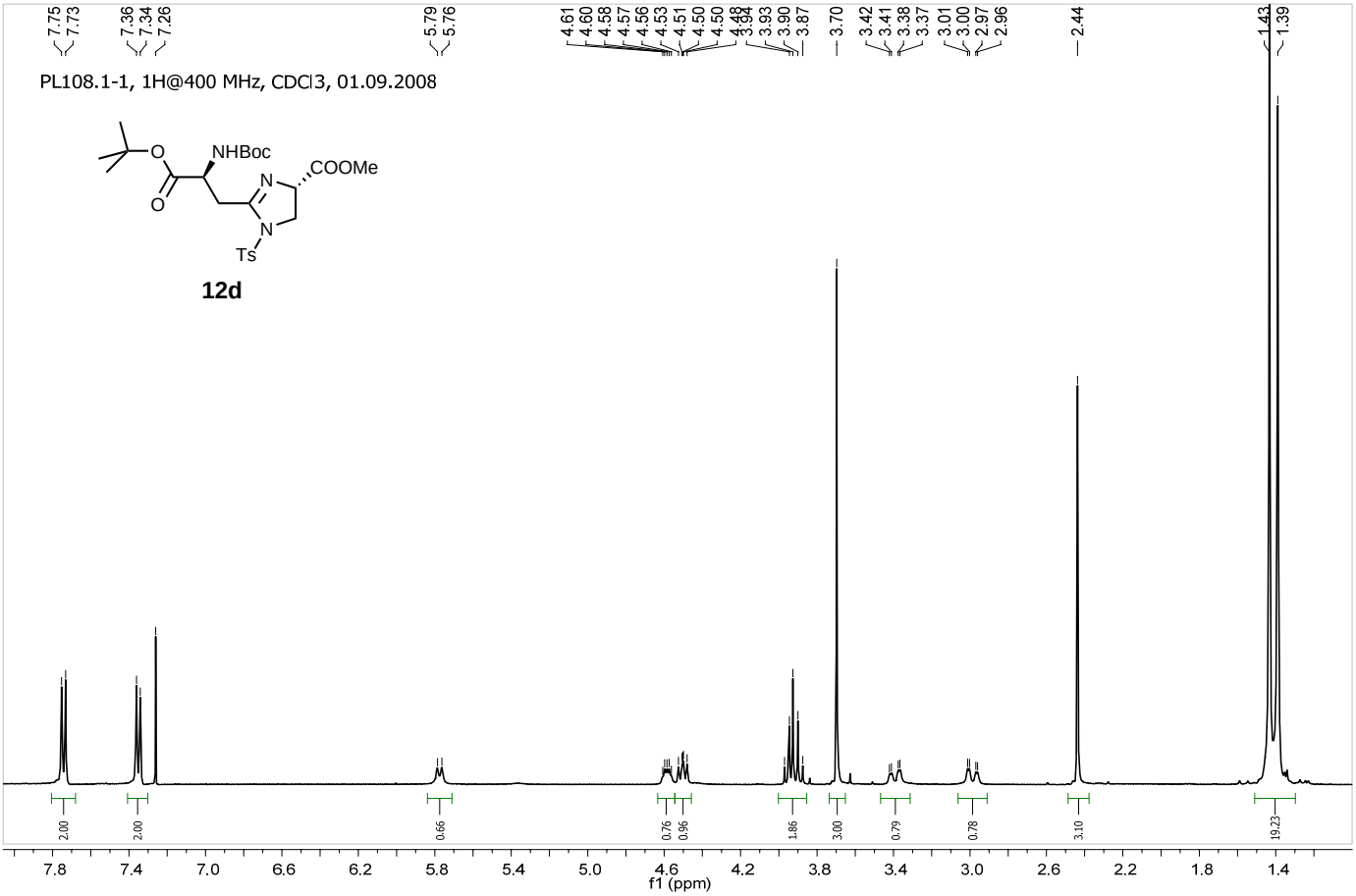
O
O
O
N
Ts
COOMe
N3
f1 (ppm)

11d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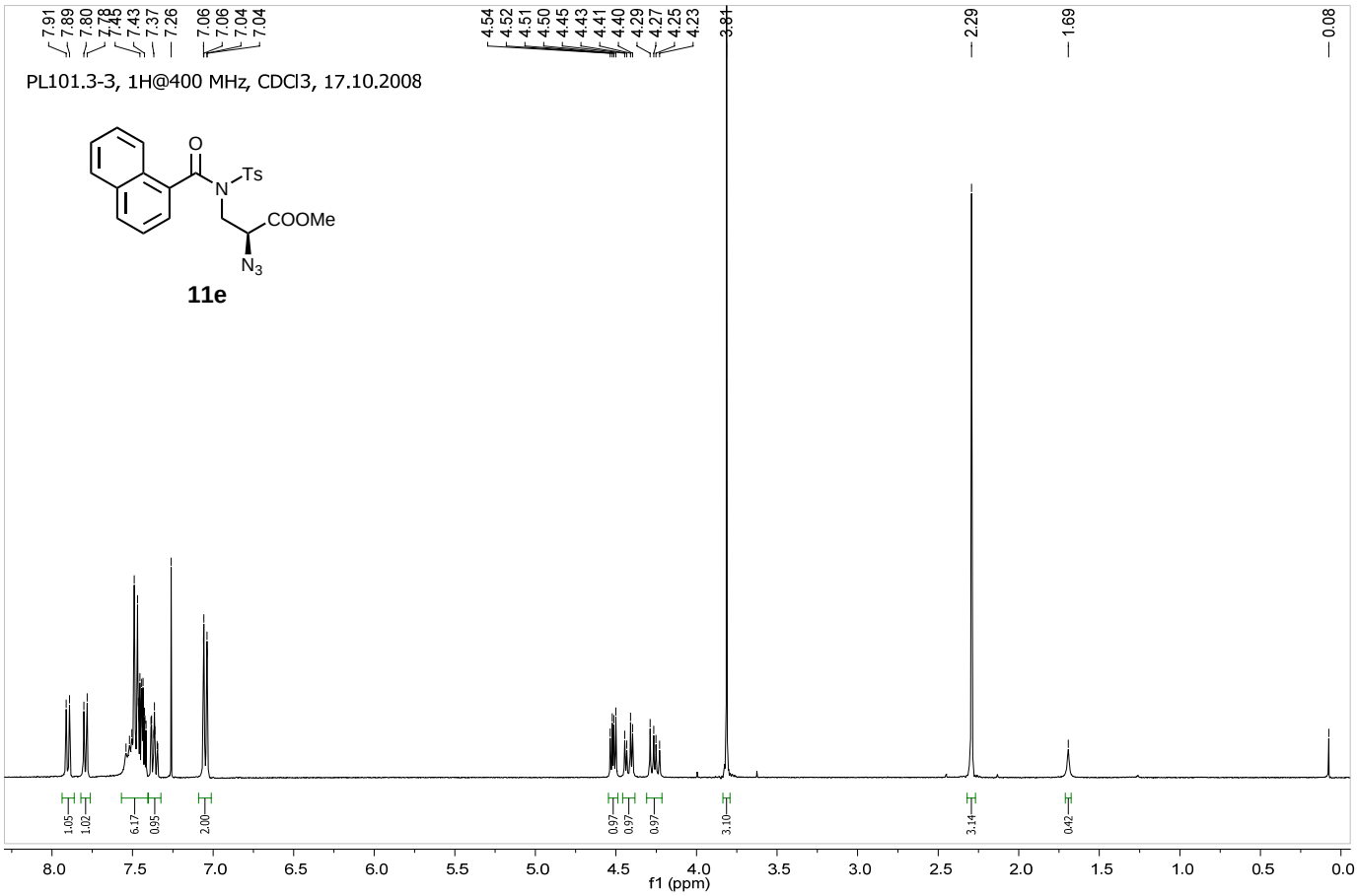
O
O
NHBoc
N
COOMe
N
Ts
f1 (ppm)

12d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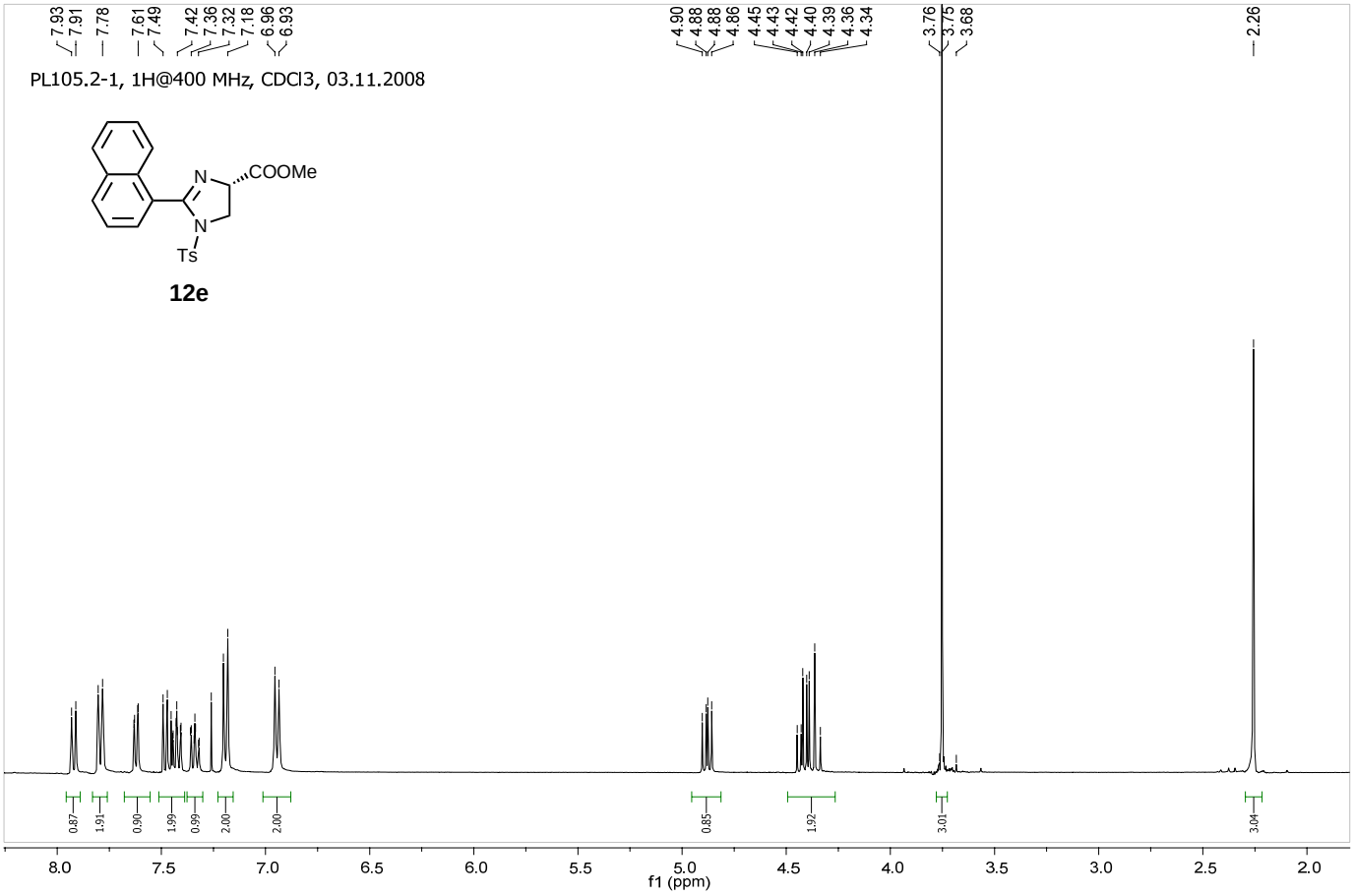
N
Ts
COOMe
N3
11e
f1 (ppm)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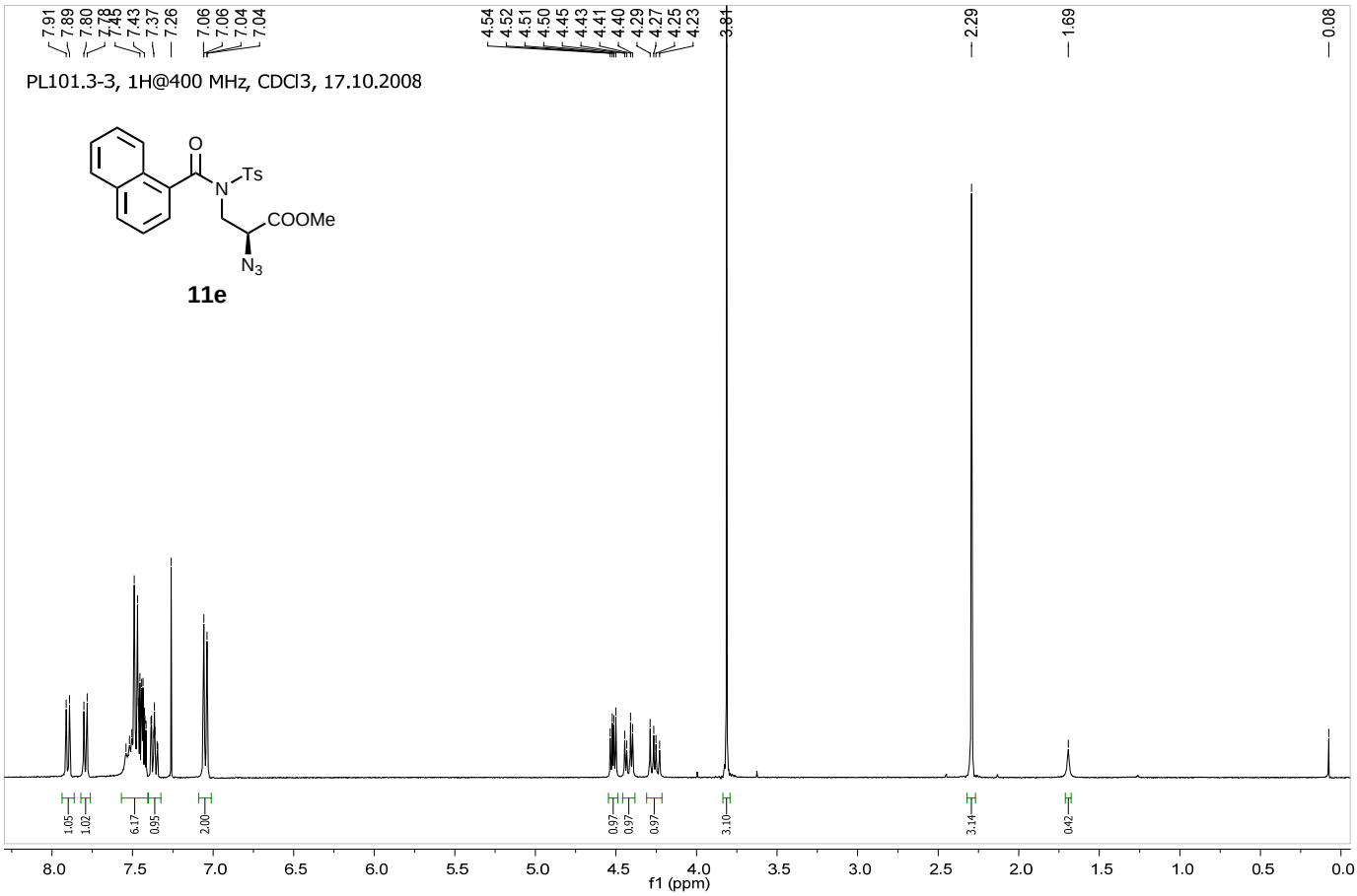
N
COOMe
N
Ts
12e
f1 (ppm)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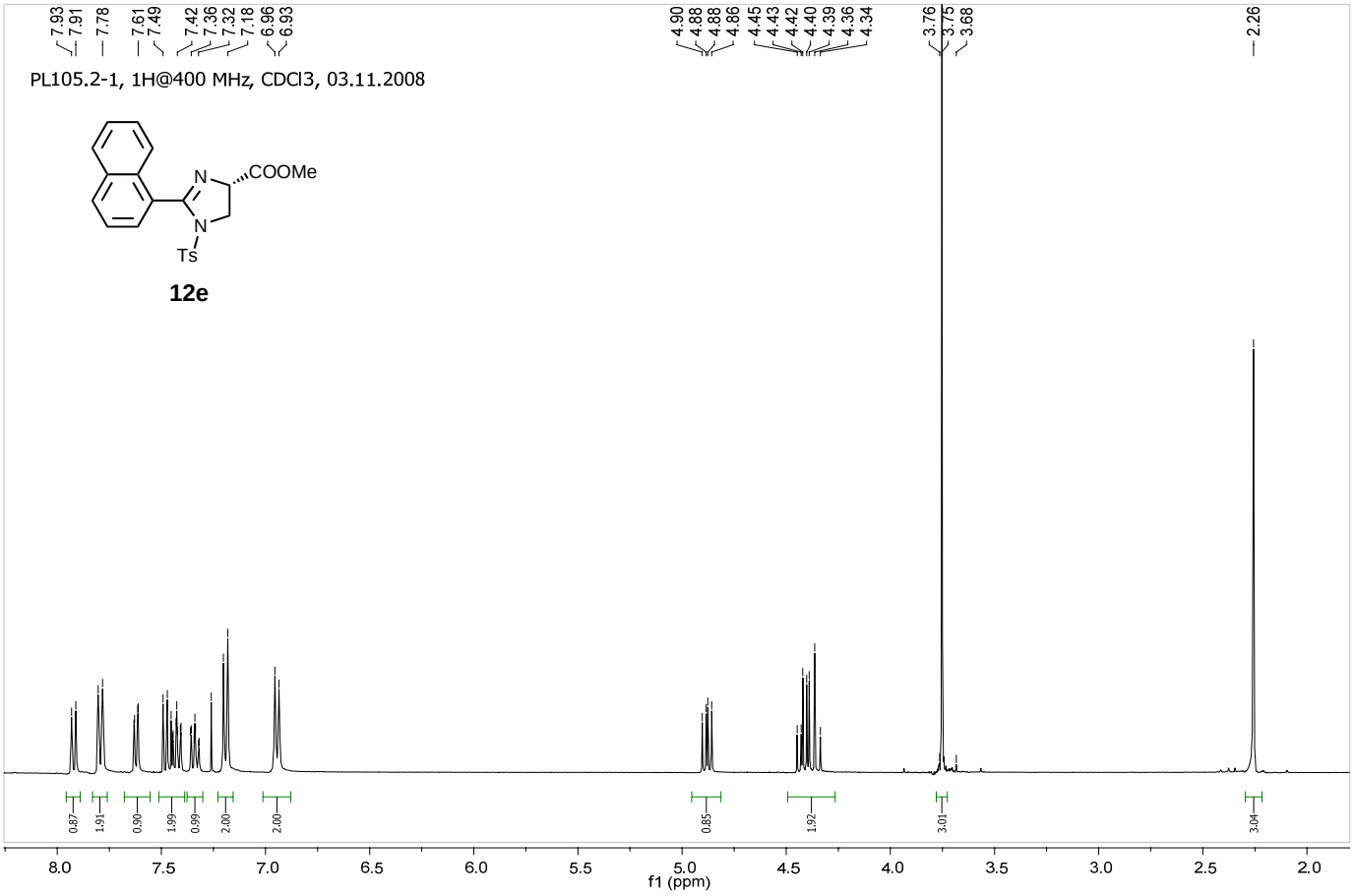
O
N
Ts
COOMe
N3
11e
f1 (ppm)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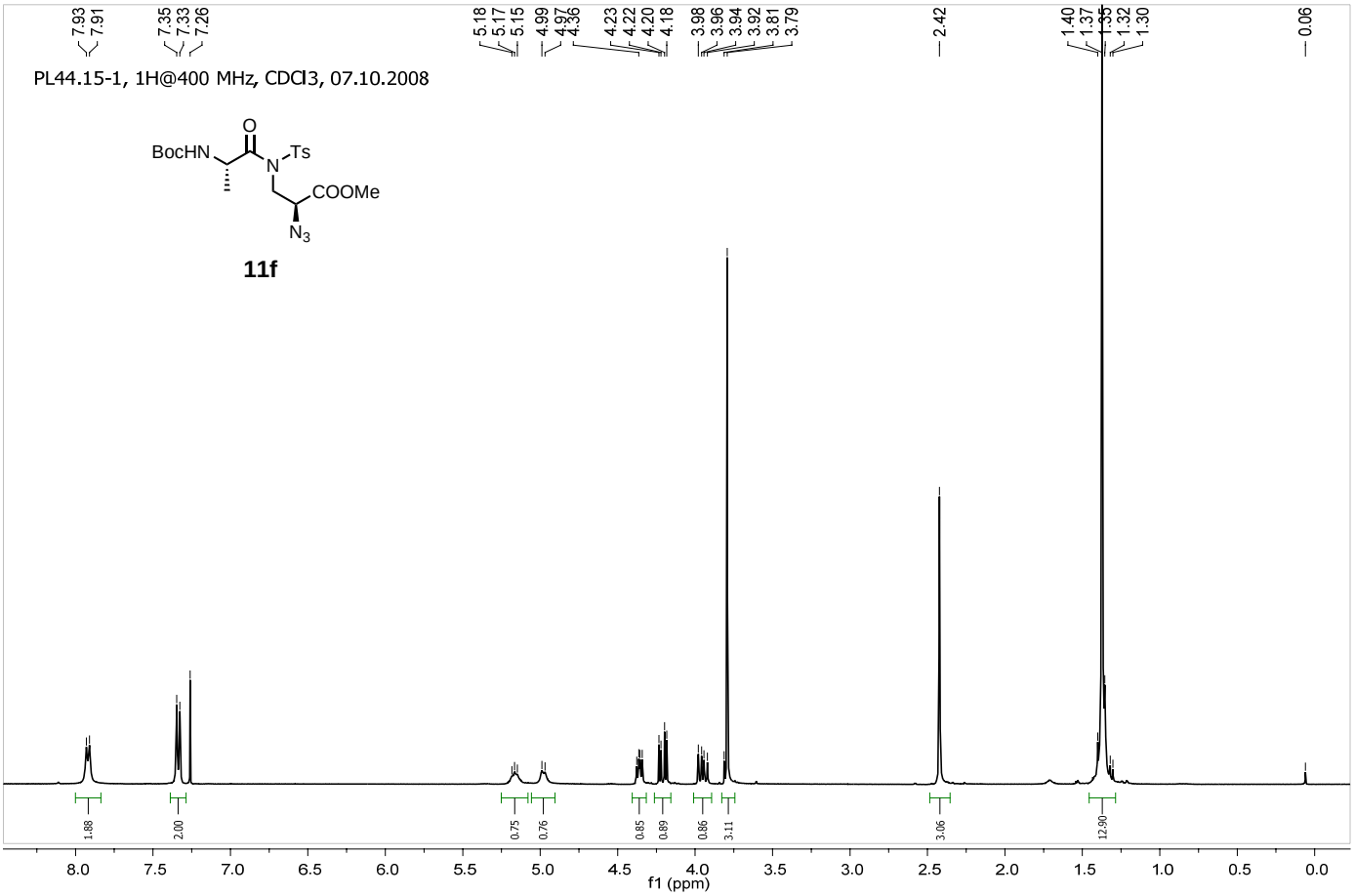
N
COOMe
N
Ts
12e
f1 (ppm)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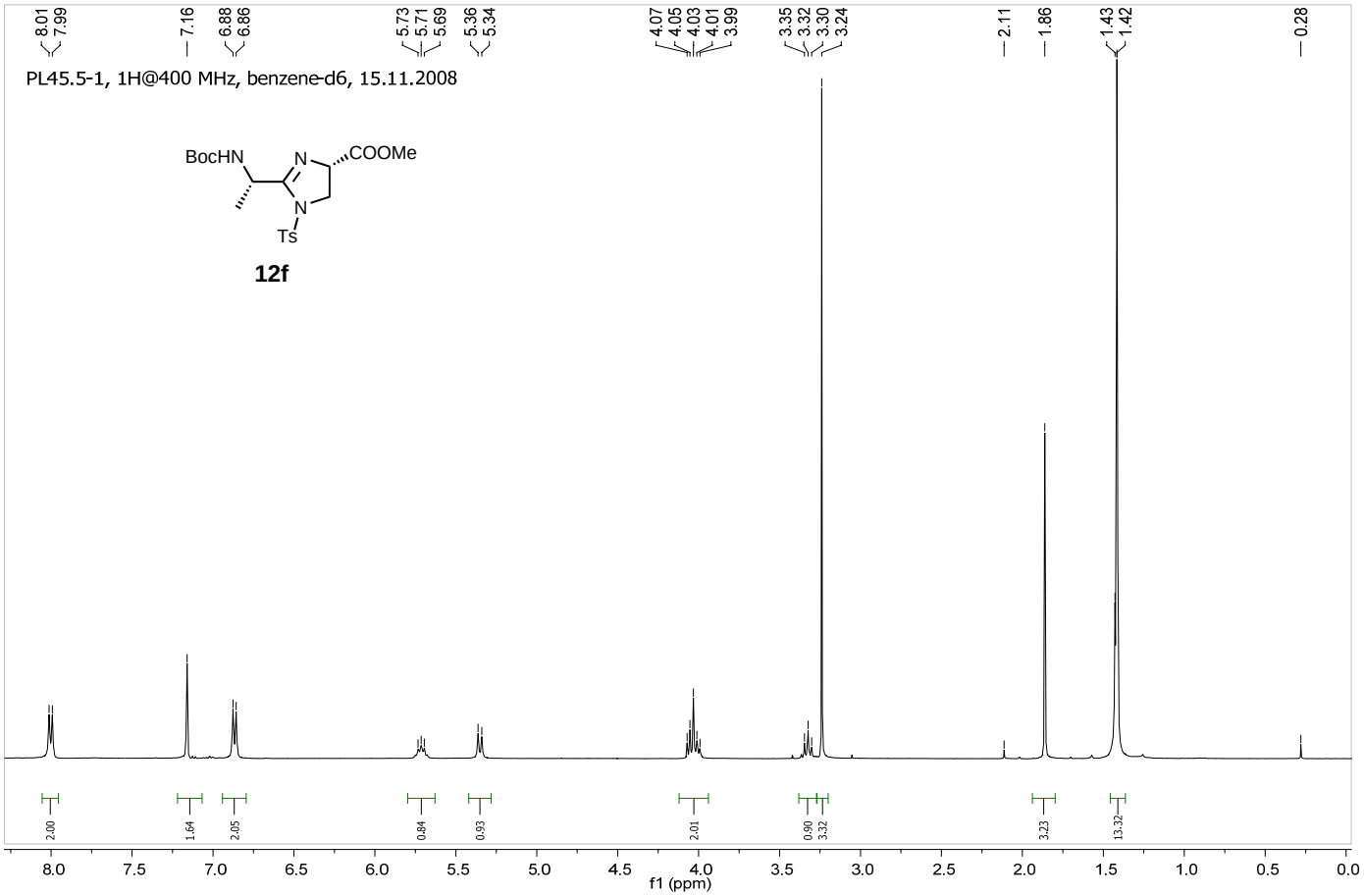
BocHN
O
N
Ts
COOMe
N3
11f
f1 (ppm)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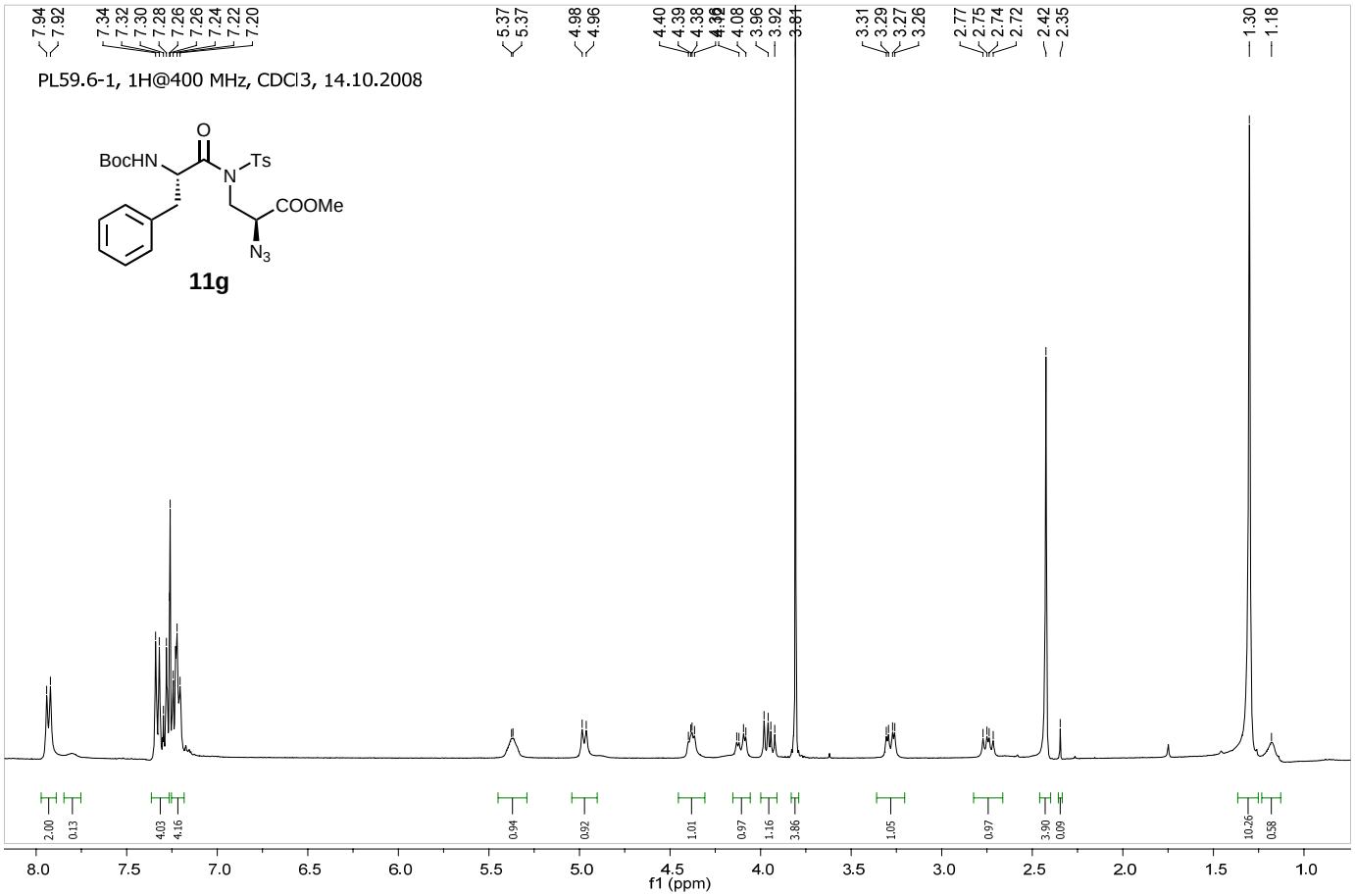
BocHN
N
COOMe
N
Ts
12f
f1 (ppm)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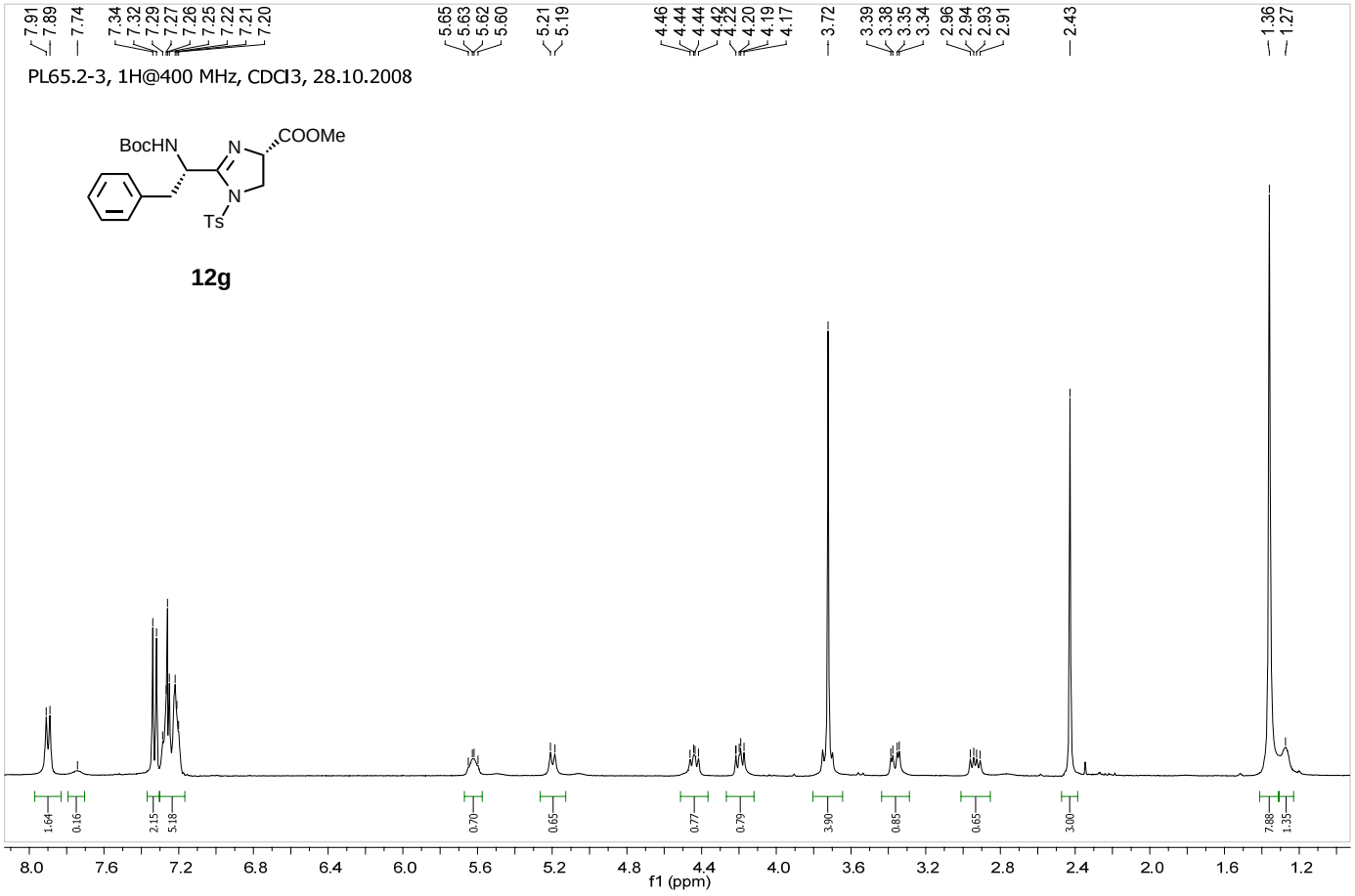
BocHN
O
N
Ts
COOMe
N3
11g
f1 (ppm)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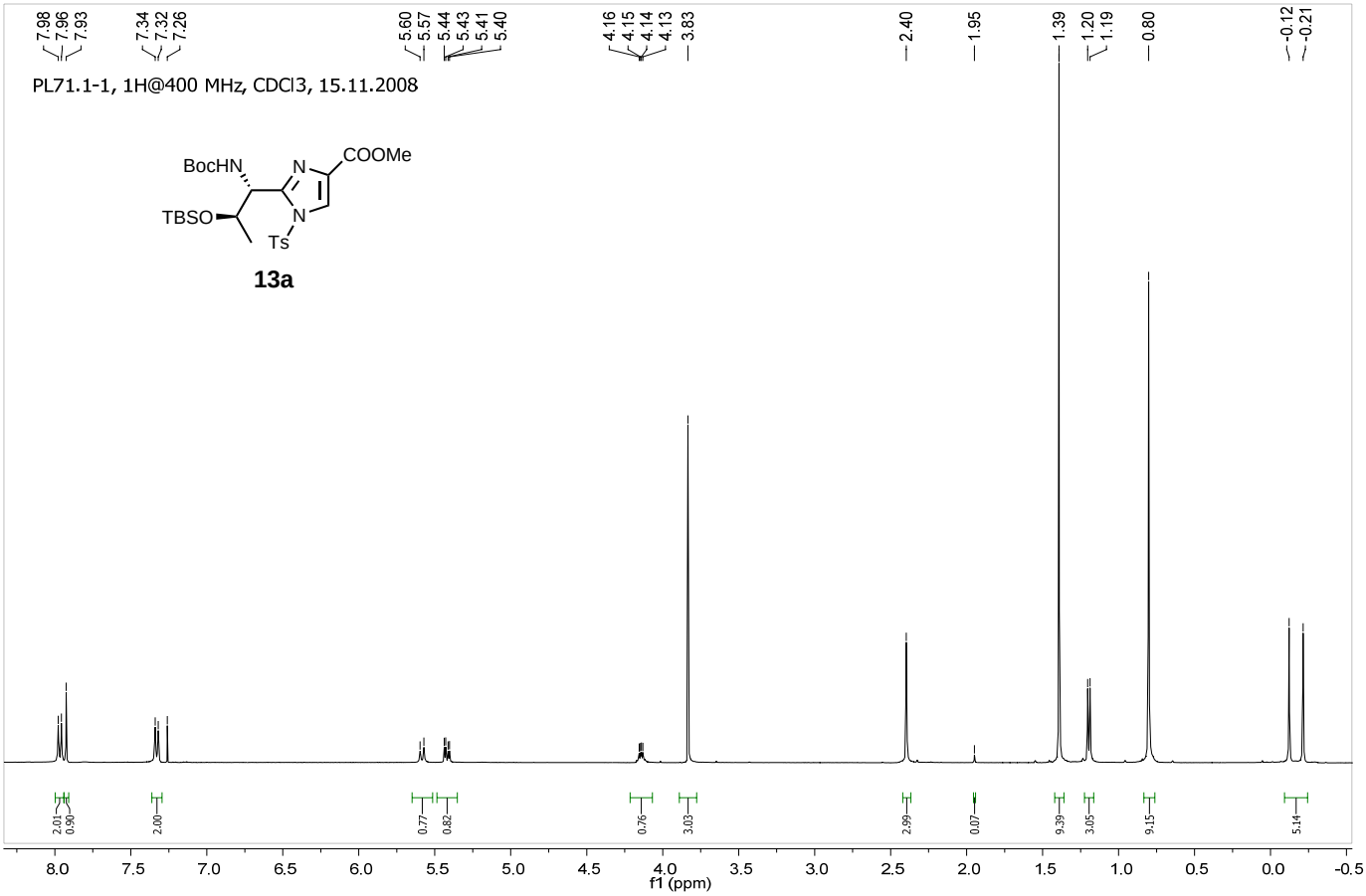
COOMe
N
Ts
12g
f1 (ppm)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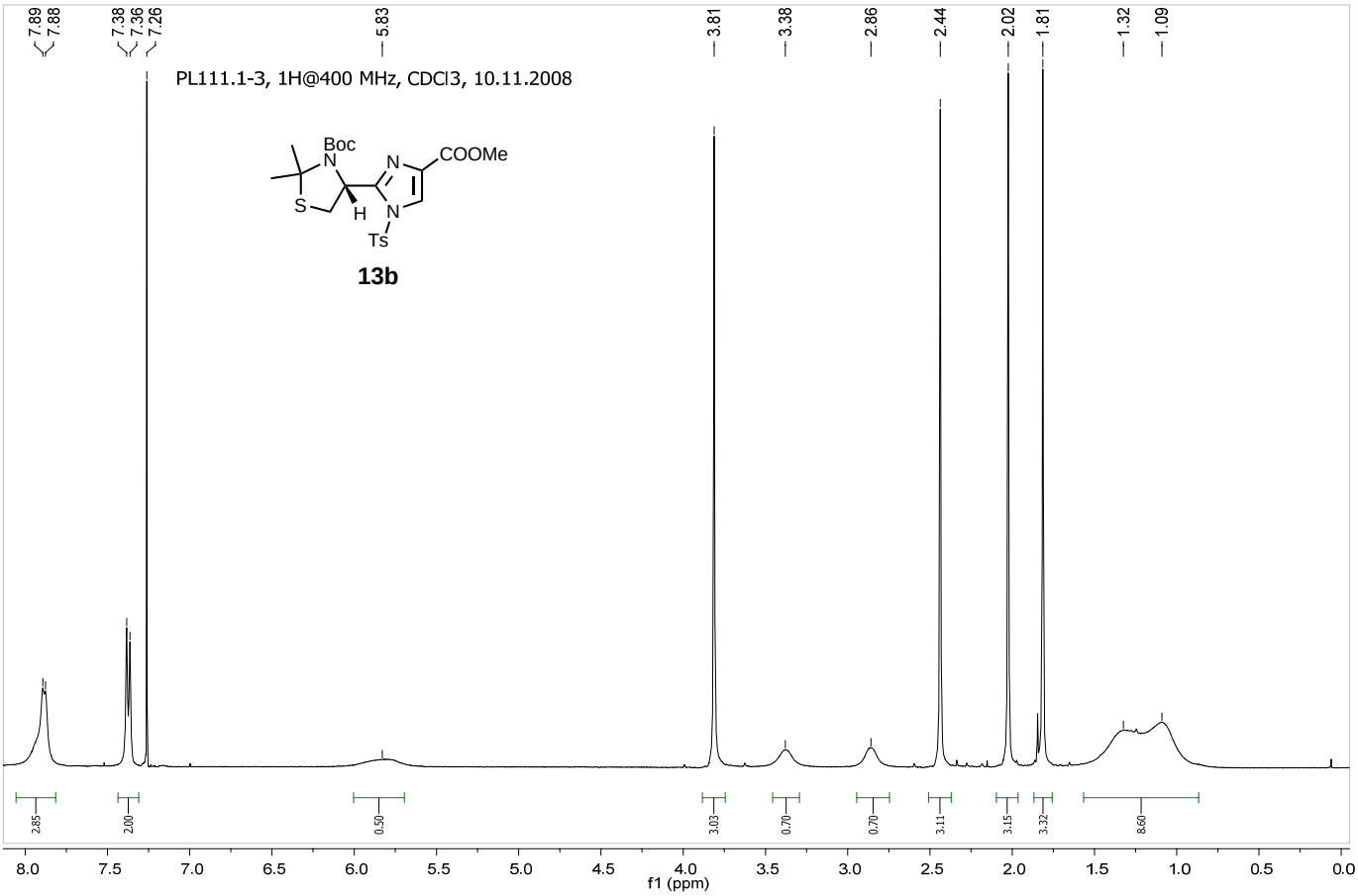
BocHN
TBSO
N
COOMe
N
Ts
13a
f1 (ppm)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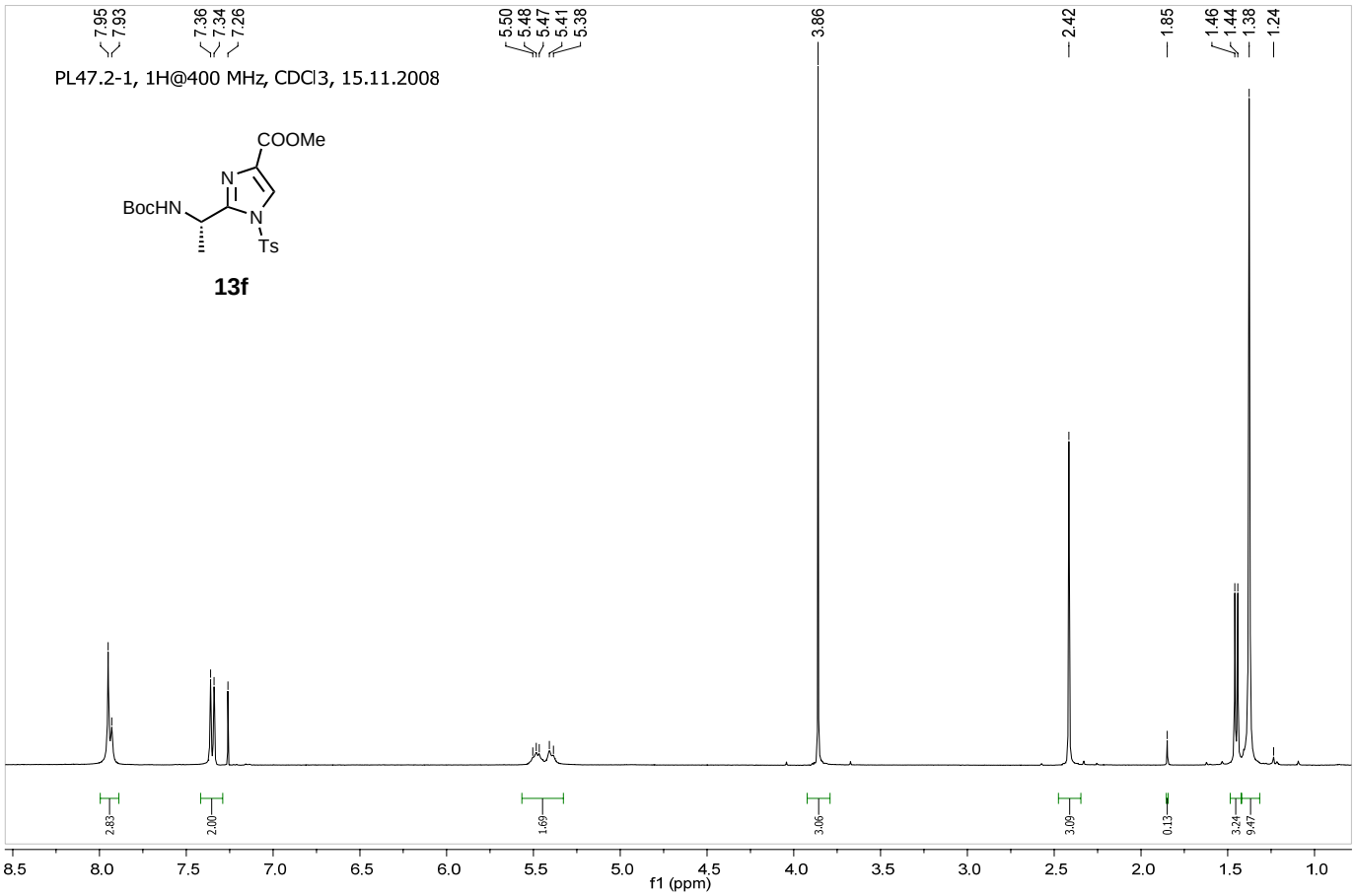
N
S
H
N
N
COOMe
Ts
13b
f1 (ppm)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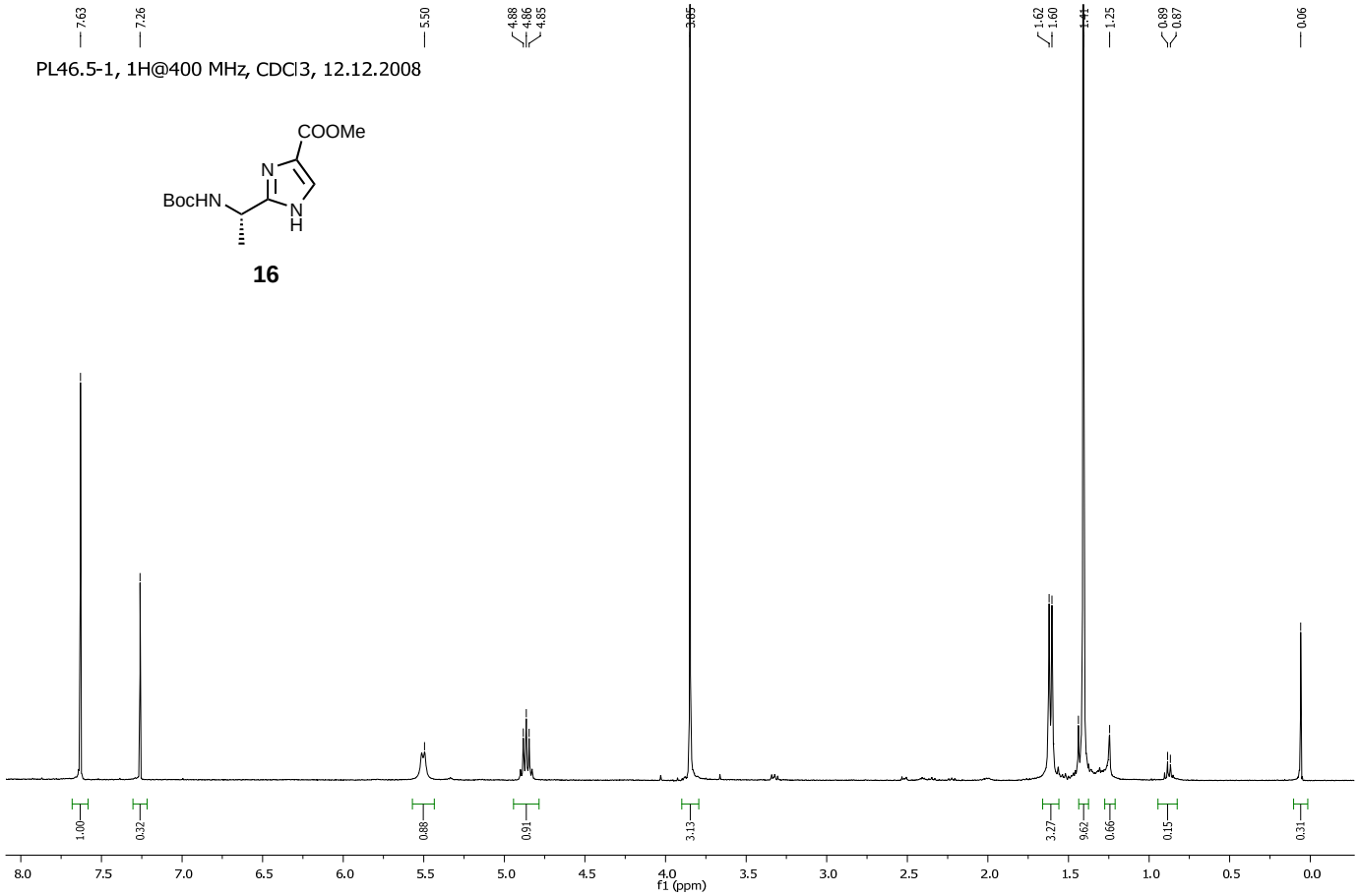
N
BocHN
N
Ts
13f
7.95
7.93
7.36
7.34
7.26
5.50
5.48
5.47
5.41
5.38
3.86
2.42
1.85
1.46
1.44
1.38
1.24
2.83
2.00
1.69
3.06
3.09
0.13
3.24
9.47
f1 (ppm)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COOMe
N
BocHN
N
H
16
7.63
7.26
5.50
4.88
4.86
4.85
3.85
1.62
1.60
1.41
1.25
0.89
0.87
0.06
1.00
0.32
0.88
0.91
3.13
3.27
9.62
0.66
0.15
0.31
f1 (ppm)