Supporting Information

for

APPLICATION OF SO_3H SILICA GEL TO DEPROTECTION OF SILYL ETHERS.

Hideaki Fujii,[1] Takaaki Yamada,[1] Kohei Hayashida,[1] Miki Kuwada,[1] Atom Hamasaki,[2] Kazunori Nobuhara,[3] Sumio Ozeki,[2] Hiroshi Nagase[1,*]

[1] School of Pharmacy, Kitasato University, 5-9-1 Shirokane, Minato-ku, Tokyo 108-8641, Japan

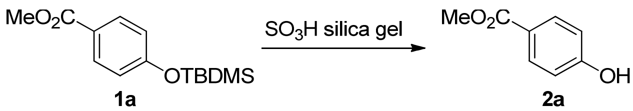
[2] Department of Chemistry, Faculty of Science, Shinshu University, 3-1-1, Asahi, Matsumoto, Nagano, 390-8621, Japan

[3] FUJI SILYSIA CHEMICAL LTD., 1846, 2-Chome, Kozoji-Cho, Kasugai-Shi, Aichi, 487-0013, Japan

Search of an optimal solvent

The desilylation of the test compound **1a** was conducted using various solvents (Table S1). Although the reactions went to completion under every reaction condition, the reaction rate was fastest in heptane. Therefore, we chose heptane as the optimal solvent. Dichloromethane may possibly be used as a solvent, but its reaction rate was the slowest among the tested solvents.

Table S1. Desilylation of **1a** in the presence of various solvents



Entry	Temp (ºC)	Time (h)	AcOEt	Tol	Heptane	MeOH	CH_2Cl_2
1	25	14	B	B	A	NT	B
2	25	50	A	A	NT	NT	A[a]
3	50	1	B	B	A	B	B
4	50	2	B	B	NT	A	B
5	50	3	A	A	NT	NT	A[b]

A: The starting material was disappeared in the TLC analysis.
B: The starting material was detected by the TLC analysis.
NT: not tested
[a] 3 days
[b] 10 h

The ^{1}H NMR spectra of the crude products in the desilylation of 1i under various reaction conditions

^{1}H NMR Spectra (300 MHz, benzene-d_6) of aryl TIPS ether **1h**, the corresponding phenol derivative **2c**, and the crude products obtained under indicated reaction conditions are shown (**Figures S1-S6**). Using far longer reaction time and/or higher reaction temperature decreased the amount of silyl residues in the crude products and ultimately almost no residues were detected.

Figure S1 ^{1}H NMR spectrum of aryl TIPS ether **1h**

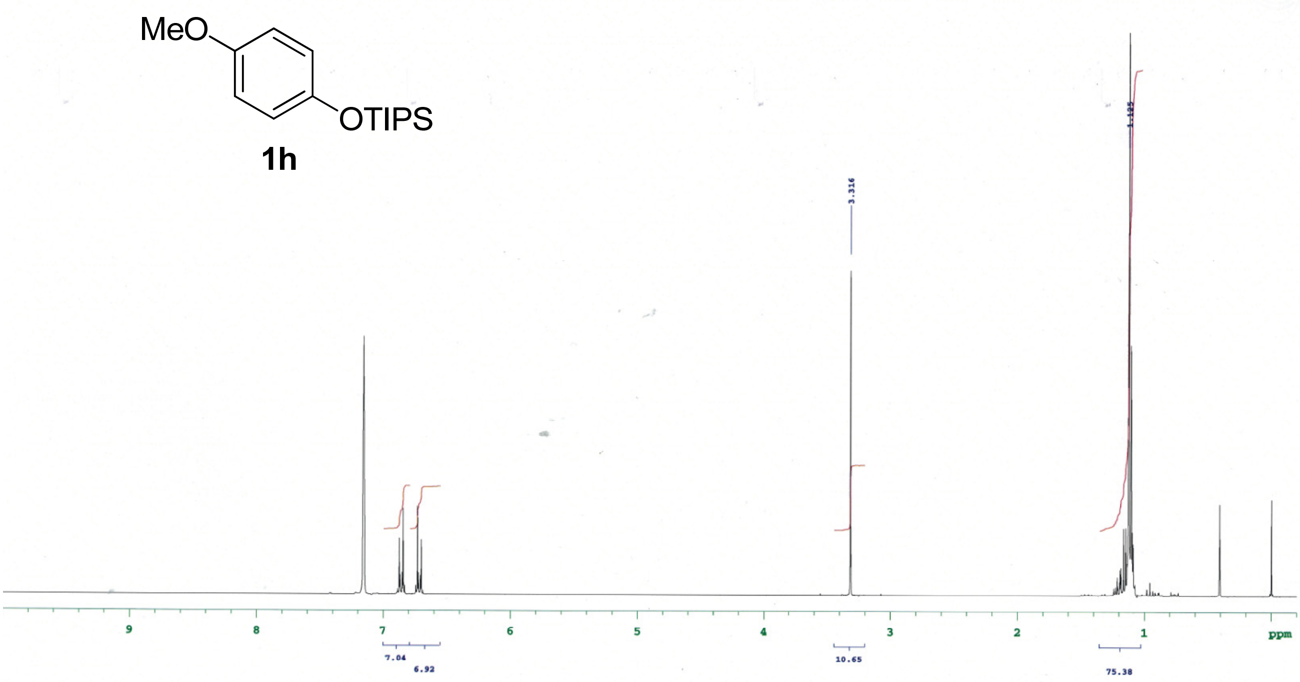


Figure S2 ^{1}H NMR spectrum of phenol derivative **2c**

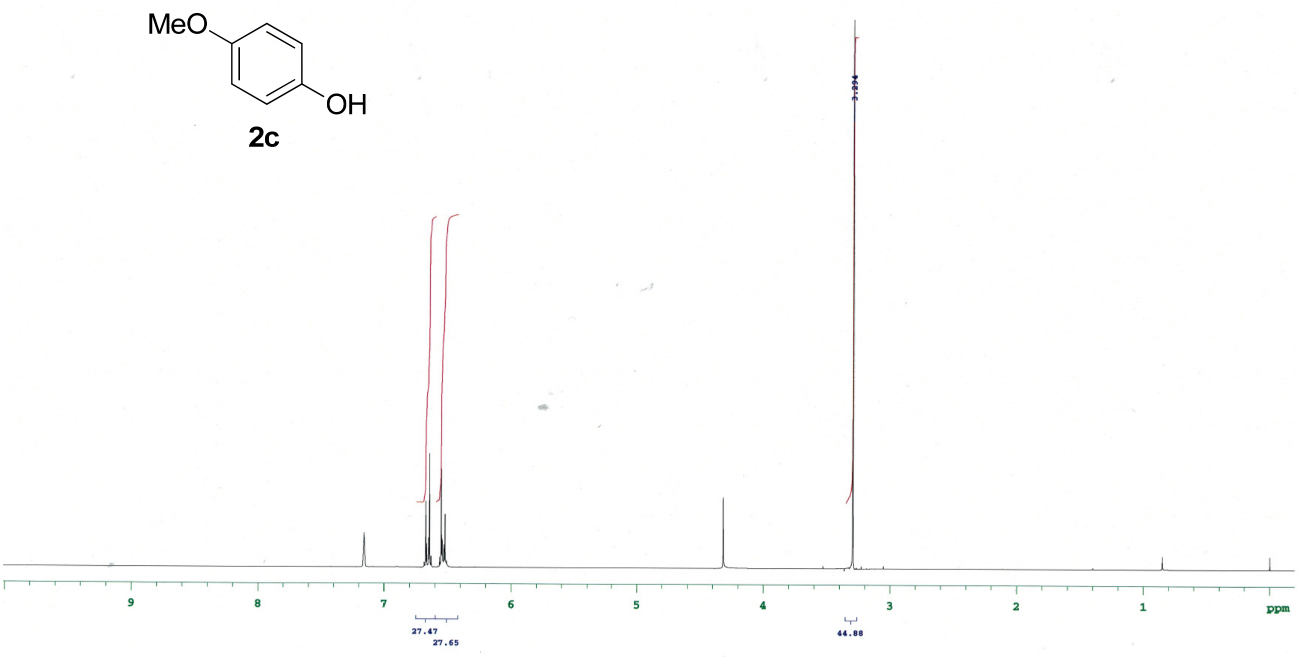


Figure S3 [1]H NMR spectrum of the crude product obtained by the reaction at 50 ºC for 2 h

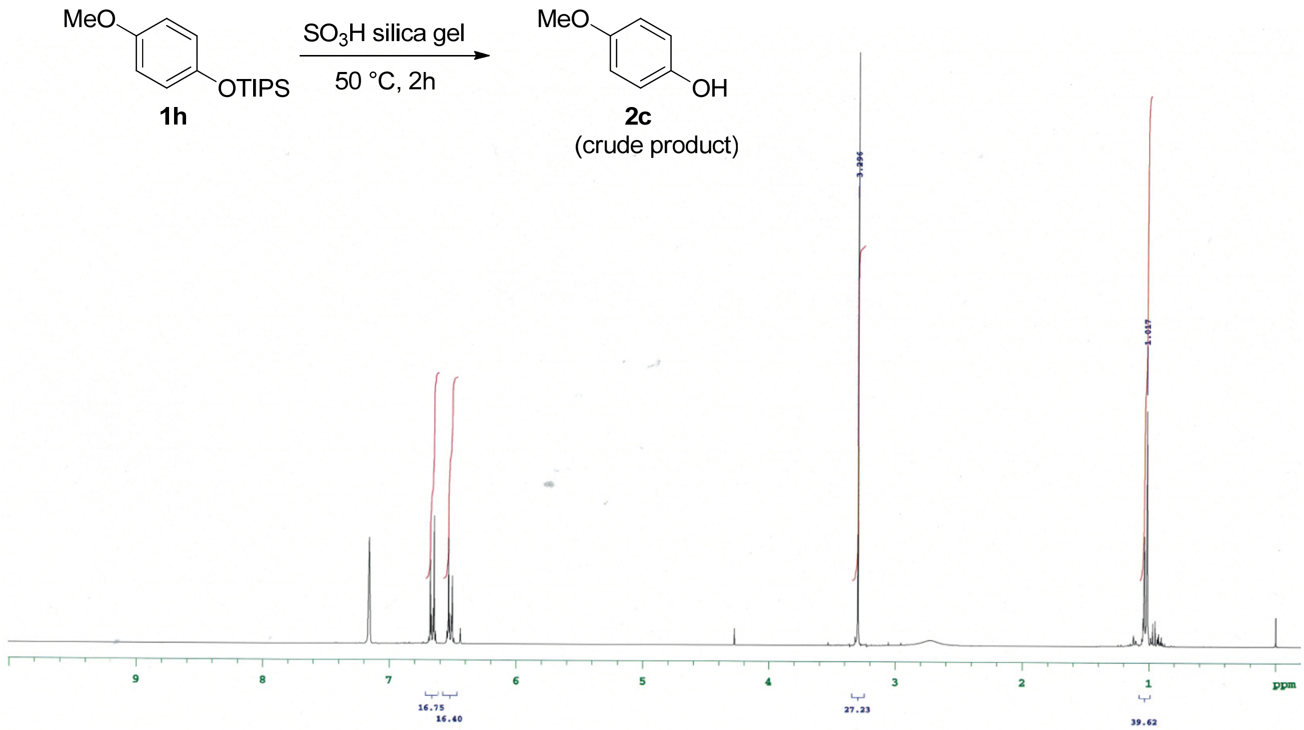


Figure S4 [1]H NMR spectrum of the crude product obtained by the reaction at 100 ºC for 2 h

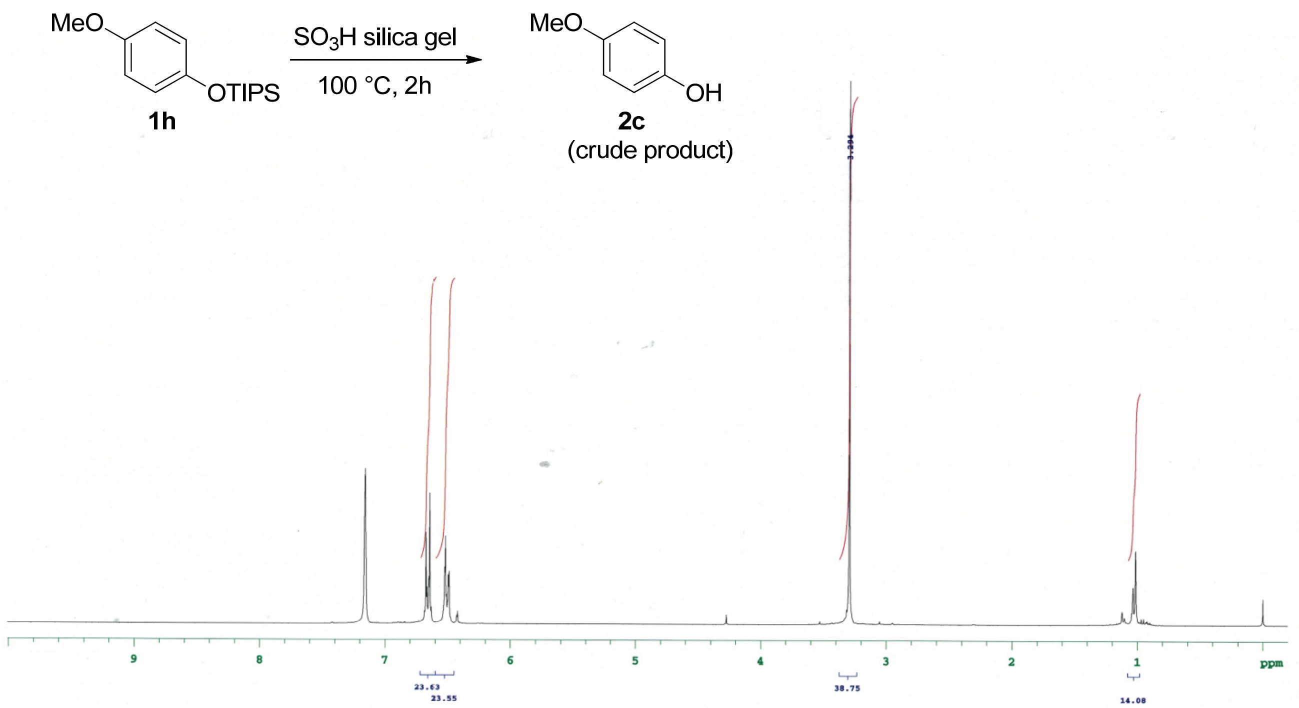


Figure S5 ^{1}H NMR spectrum of the crude product obtained by the reaction at 100 ºC for 10 h

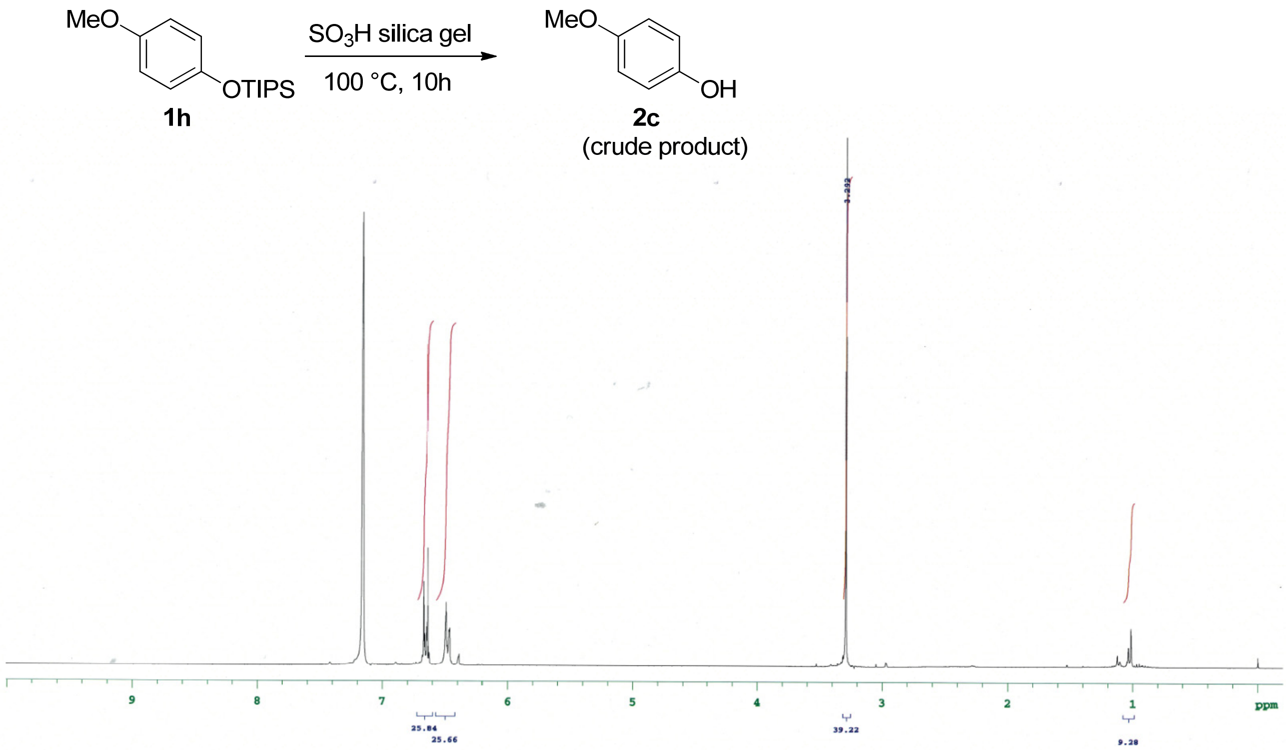


Figure S6 ^{1}H NMR spectrum of the crude product obtained by the reaction at 100 ºC for 24 h