STEPS Students Report

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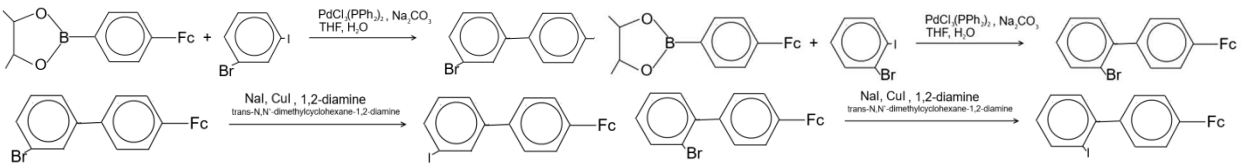
At the beginning in this work we tried to synthetize two substances – 4-ferrocenyl-3′-iodo-1,1′-biphenyl (meta-$FcPh_2I$) and 4-ferrocenyl-2′-iodo-1,1′-biphenyl (ortho-$FcPh_2I$). Both of them can be used for construction of monolayers onto hydrogen-terminated silicon electrodes. These monolayers can be used for a construction of molecular scale devices. Assembled monolayers chemically immobilized on substrate surfaces are promising materials for creating of molecular scale devices because they allow us to detect electric signals through substrates working as electrodes. In particular, a method of immobilizing molecules on a hydrogen-terminated silicon substrate is suitable for an electronic material due to a faster transfer without an oxide layer.

In previous researches, electron transfer between redox moieties and hydrogen-terminated interfacial silicon surface through a few kinds of structural anchors was investigated. As one of the anchors, aryl group directly linking to silicon atoms is expected to show great advantage in controlling of the geometric composition and accelerating of an electron transfer. Concerning to the later advantage, it originates in π-σ conjugation over the aryl group and silicon atoms. In other words, the electron transfer behavior is possibly controlled by a position of a substituent on the phenylene anchor or surface orientations of the silicon electrode which alter a shape of the π-orbital or the σ-orbital respectively.

In our synthesis we used reactions



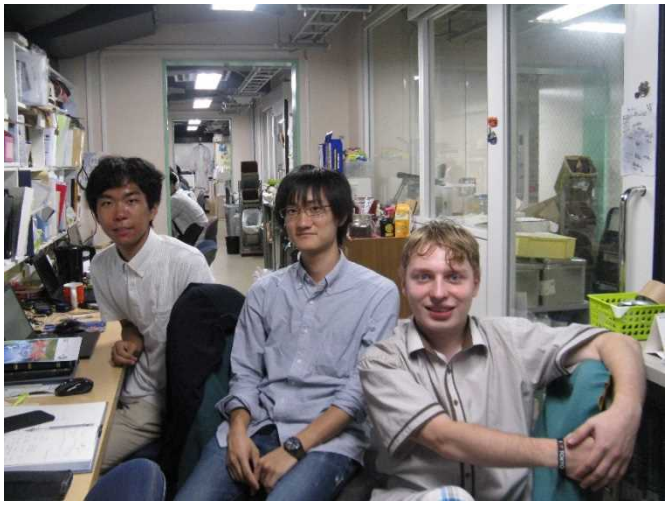
In first time when we tried to synthetize our substances we did not received desired product because of spoiled precursor 4-(4,4,5,5-tetramethyl-1,3,2-dioxoborolan-2-yl) phenylferrocene. We tried to get it twice, however have not any good results. Then we tested precursors by mass-spectroscopy and NMR-spectroscopy. It was showed by mass-spectra and NMR that instead of 4-(4,4,5,5-tetramethyl-1,3,2-dioxoborolan-2-yl) phenylferrocene we used 4-iodophenylferrocene. It turned out that iodine and 4,4,5,5-tetramethyl-1,3,2-dioxoborolan-2-yl have almost the same molecular weight, 126.90 g/mol and 126.97 respectively, therefore 4-

(4,4,5,5-tetramethyl-1,3,2-dioxoborolan-2-yl) phenylferrocene and 4-iodophenylferrocene have almost same results in mass-spectra.

Then we synthetized 4-ferrocenyl-3′-iodo-1,1′-biphenyl from 3-bromo-4′-ferrocenyl-1,1′-biphenyl. Used time for synthesis was 18 hours. Then it was purified by column chromatography with silica-gel and hexane solution of dichloromethane and recrystallization from ethanol. After every step we checked purity by NMR. We stopped purification of the product after getting NMR spectra without too high excess peaks. Yield 48.6 mg, 55.4%. Measured melting point was found to be equal 141.8-145^{0}C.

After it we purified 4-ferrocenyl-4′-iodo-1,1′-biphenyl (para-$FcPh_2I$) that we had unrefined by column chromatography with dichloromethane and recrystallization from different solvents such as ethanol, mixture of hexane with dichloromethane. Furthermore, thin-layer chromatography with mixture of hexane and dichloromethane was applied. After every step we checked purity by NMR. However every time we found in spectra little amounts of impurities or solvents: hexane, dichloromethane, and ethanol. To remove these amounts of solvents we used drying with vacuum pump. When we got substance pure enough we stopped purification and measured melting point. Melting point was equal to 212.3-214.0 ^{0}C.

In addition, I would like to express gratitude to organizers of STEPS program. It was a great



experience for me, because I could meet a whole new world of Japanese science and culture. Moreover, I was able to communicate with people not from Japan only, however also from other countries such as America, China, India, Philippines and New Zealand. I improved my language skills, both English and Japanese. Memories about time that I have spent in my internship in Tokyo University will stay in my heart forever. Therefore, I hope that in future I will have new opportunity to visit Japan.