Supplement S4: Detailed amounts of reactants and solvents in the synthesis of 17-hydroxymethyl-17-methyl-18-nor-5-androst-13-en-3-ols

Table S4-1: Reactants and solvents used in the methenylation of Etiocholanolone (**5**) or androsterone (**5a**)

|  |  |  |
| --- | --- | --- |
|  | **6** | **6a** |
| Reagent |  |  |
| Nysted (20 %) | 14.2 mL | 13.4 mL |
| THF | 7.0 mL | 7.0 mL |
| TiCl4 | 360 µL | 330 µL |
| Precursor Steroid | **5** | **5a** |
| Amount of Steroid | 43.0 mg | 394 mg |
| HCl (2 M) | 15 mL | 25 mL |
| Ice-cold water | 15 mL | 25 mL |
| Diethyl ether | 60 mL | 100 mL |

Table S4-2: Reactants and solvents used for epoxidation

|  |  |  |
| --- | --- | --- |
|  | **7** | **7a** |
| Reagent |  |  |
| Precursor Steroid | **6** | **6a** |
| DCM | 5 mL | 20 mL |
| KHCO3 | 44.5 mg | 440 mg |
| m-CPBA | 31.0 mg | 370 mg |
| H2O | 10 mL | 20 mL |
| DCM | 20 mL | 50 mL |