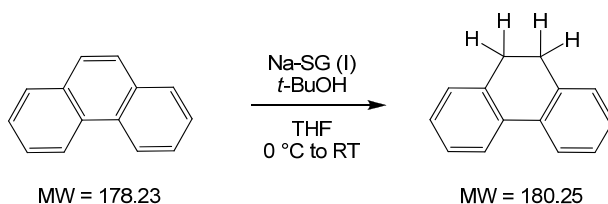


Birch Reduction of Phenanthrene with Na-SG (I) (KAP-A-31)



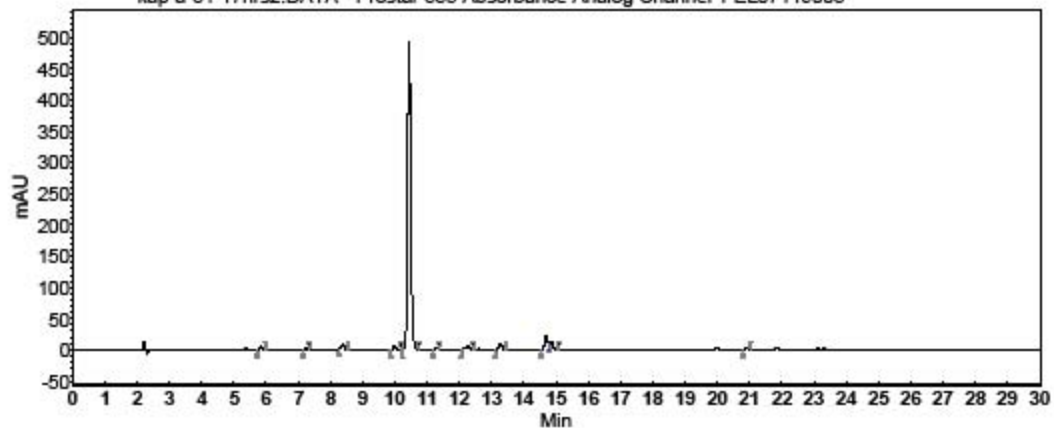
Procedure: A 25 mL 3-necked round bottomed flask was fitted with a stirring bar, septum, nitrogen inlet and a Teflon-coated digital thermometer. The reaction vessel was flame-dried while assembled (temperature reached 150 °C) under a nitrogen purge and then cooled to room temperature. Na-SG (I) (256 mg, 3.92 mmol, 3.5 equiv.; H₂ assay = 35% Na) and phenanthrene (100 mg, 0.56 mmol) were added to the reaction vessel followed by anhydrous THF (5.6 mL added via syringe). The resulting slurry was cooled to 0 °C (reaction appears green) and treated with anhydrous *t*-butanol (0.18 mL, 1.92 mmol, 1.7 equiv.; added via syringe). The reaction mixture (now appears light brown) was allowed to slowly warm to room temperature while stirring under nitrogen over 17 hours. The reaction was quenched by the addition of 20 mL of water (H₂ evolution observed) and extracted three 10 mL portions of ethyl acetate. The combined ethyl acetate extracts were washed twice with 10 mL of brine, dried over anhydrous Na₂SO₄, filtered and concentrated in vacuo to afford 90 mg (89%) of crude 9,10-dihydrophenanthrene (R_t = 10.22 min) as light amber oil which is 86% pure by HPLC and contains 1% phenanthrene (R_t = 10.00 min).

HPLC method: Inject 5 uL of a 2mg/mL solution in THF eluting with 60-100 % MeCN in H₂O over 15 minutes at 1.5 mL/min on a Varian Microsorb-MV 100-5 C-18 column (250x4.6 mm x ¼”).



Auto60-100 (30 min)49BONNIE3/18/20082:14:08 PM

kap-a-31-17hrs2.DATA - Prostar 335 Absorbance Analog Channel 1 EL07119003



Time [Min]	Area % [%]
5.85	0.760
7.25	0.452
8.39	1.144
10.00	1.080
10.44	86.376
11.28	0.379
12.28	1.411
13.27	1.625
14.69	3.885
14.87	2.324
20.91	0.563
Total	100.000