Supporting Information for:

**Oxidative Aromatization of 9,10-Dihydroanthracenes
Using Molecular Oxygen Promoted by Activated Carbon**

Natsuki Nakamichi, Hirotoshi Kawabata, and Masahiko Hayashi*
Department of Chemistry, Faculty of Science, Kobe University, Kobe 657-8501, Japan
mhayashi@kobe-u.ac.jp

CONTENTS

1. General methods
2. Typical procedure for the aromatization
3. Typical procedure for measurement of ICP-AES
4. Compound characterization
5. Analytical data of ICP-AES

General methods. All melting points were uncorrected. ^1H and ^{13}C NMR spectra (400 and 100.6 MHz, respectively) were recorded using Me_4Si as an internal standard in CDCl_3 . ICP-AES were measured on a Varian Liberty Series II. Substituted 9,10-dihydroanthracenes were synthesized by reducing the corresponding anthracenes using Na in EtOH; Bass, K. C. *Organic Syntheses*; Wiley: New York, 1973; Collect. Vol. 5, p 398.

Typical procedure for oxidative aromatization: The 200 mL three-necked flask was charged with 9,10-dihydroanthracene (**1**) 2.0 g (11.1 mmol), xylene¹² (15 mL), and the 1.0 g (50 weight%) of activated carbon (Darco[®] KB, Aldrich Inc.) under an oxygen atmosphere using balloon. The whole was warmed to 120 °C and stirred for 12 h at this temperature. After confirmation of the consumption of starting material by TLC analysis, activated carbon was filtered off using Celite. After washing with toluene, the filtrate was evaporated and the resulting solid was purified by recrystallization to give the desired anthracene (**2**) 1.7 g (86%) as a yellow crystal. mp 216 °C (lit.¹³ 218 °C).

Typical procedure for measurement of ICP-AES: All reagents used in this study were of analytical or special grade.

Wet digestion method: An accurately weighed activated carbon (1.0 g) was digested with 20 mL of 2 M HNO_3 . After shaking for 24 h at room temperature, activated carbon was removed by filtration. Then, 5 mL of filtrate was diluted with water to 50 mL. The resulting solution was analyzed by ICP-AES (Varian Liberty Series II).

Dry digestion method: An accurately weighed activated carbon (1.0 g) was heated for 6 h at 600 or 800 °C. After cooling to the room temperature, 2 mL of 12 M HCl was added and heated gently. The insoluble material was filtrated and the filtrate was diluted with water to 100 mL. The resulting solution was analyzed by ICP-AES.

anthracene

mp 216 °C; ^1H NMR: δ 7.4—7.5 (m, 4H), 8.0 (m, 4H), 8.42 (s, 2H); ^{13}C NMR: δ 125.3, 126.2, 128.1, 131.7.

2-methylantracene

mp 208—210 °C; ^1H NMR: δ 2.55 (s, 3H), 7.31 (d, 2H), 7.4—7.5 (m, 2H), 7.8 (s, 1H), 7.91 (d, 1H), 7.9—8.0 (m, 2H), 8.32 (s, 1H), 8.38 (s, 1H); ^{13}C NMR: 22.0, 124.9, 125.1, 125.2, 125.9, 126.3, 127.9, 128.0, 128.1, 128.2, 130.3, 131.2, 131.8, 132.0, 134.9.

2-ethylantracene

mp 154—157 °C; ^1H NMR: δ 1.40 (t, 3H), 2.85 (q, 2H), 7.33 (d, 1H), 7.4—7.5 (m, 2H), 7.76 (s, 1H), 7.9—8.0 (m, 3H), 8.35 (s, 1H), 8.38 (s, 1H); ^{13}C NMR: δ 15.1, 29.2, 124.9, 125.0, 125.2, 125.4, 125.9, 127.3, 128.1, 128.2, 130.6, 131.3, 131.9, 132.1, 141.1.

9-methylantracene

mp 73—76 °C; ^1H NMR: δ 3.10 (s, 3H), 7.4—7.5 (m, 4H), 7.99 (d, 2H), 8.28 (d, 2H), 8.33 (s, 1H); ^{13}C NMR: δ 13.9, 124.6, 124.8, 125.2, 125.3, 129.0, 130.0, 130.1, 131.4.

9-phenylantracene

mp 153—155 °C; ^1H NMR: δ 7.3—7.4 (m, 2H), 7.4—7.5 (m, 4H), 7.5—7.6 (m, 3H), 7.70 (dd, 2H), 8.05 (d, 2H), 8.50 (s, 1H); ^{13}C NMR: δ 125.1, 125.3, 126.5, 126.8, 127.4, 128.3, 130.2, 131.2, 131.4, 137.0, 138.9.

Analytical data of ICP-AES

Element	Darco [®] KB (ppm)	Darco [®] KB-B (ppm)	Darco [®] G-60 (ppm)
Li	34.5	41.5	0.32
B	6.61	20	4.93
Na	1750	1753	268
Mg	86.9	54.80	409
Al	831	1130	156
Si	200	130	302
P	3931	3710	197
K	517	575	43.1
Ca	605.5	423	544.0
Ti	38.05	30.52	73.2
Cr	6.9	5.80	— ^b
Mn	9.99	4.1	6.5
Fe	299	98.3	175
Cu	16.77	29.8	6.88
Zn	5.36	31.8	37
Sr	12.3	7.07	6.01
Y	0.67	— ^a	— ^b
Sn	12	13	— ^b
Ba	14.6	7.76	22.6
Eu	0.23	— ^a	— ^a
Ho	0.45	— ^b	0.6
Er	9.33	7.83	18.3
W	5.59	4.06	— ^b

^a Under the level of determination limit (10 σ method). ^b Under the level of detection limit (3 σ method).

Darco[®] KB: The following elements were not observed above the level of determination limit (10 σ method); V, Co, Ni, As, Rb, Mo, Ru, Pd, In, Nd, Tb, Dy. The following elements were not observed above the level of detection limit (3 σ method); Be, Ga, Se, Zr, Rh, Ag, Cd, Sb, La, Ce, Sm, Pr, Gd, Yb, Re, Os, Ir, Pt, Au, Pb, Bi.

Darco[®] KB-B: The following elements were not observed above the level of determination limit (10 σ method); Ni, Y, Zr, Mo, Ru, Pd, Ag, In, Sb, Pr, Nd, Eu, Tb, Pt, Pb. The following elements were not observed above the level of detection limit (3 σ method); Be, V, Co, Ga, As, Se, Rb, Rh, Cd, Sb,

La, Ce, Sm, Gd, Dy, Ho, Yb, Re, Os, Ir, Au, Bi.

Darco[®] G-60: The following elements were not observed above the level of determination limit (10 σ method); Be, Co, Ni, Se, Zr, Ru, Pd, In, Nd, Eu, Tb, Pt. The following elements were not observed above the level of detection limit (3 σ method); V, Cr, Co, Ga, As, Rb, Y, Mo, Rh, Ag, Cd, Sn, Sb, La, Ce, Pr, Sm, Gd, Dy, Yb, W, Re, Os, Ir, Au, Pb, Bi.