Oxidative and Reductive Carbodiazenylation of Nonactivated Olefins

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General information

Solvents and reagents were used as received. Infra-red spectra were recorded on a Fourier transform IR spectrometer. ^1H NMR were recorded on 250, 360 and 500 MHz spectrometers using CDCl_3 , CD_3OD and C_6D_6 as solvents referenced to TMS (0 ppm), CHCl_3 (7.26 ppm) or CHD_2OD (3.31 ppm). Chemical shifts are reported in parts per million (ppm). Coupling constants are in Hertz (J Hz). The following abbreviations are used for the description of signals: s (singlet), d (doublet), dd (double doublet), t (triplet), q (quadruplet), m (multiplet). ^{13}C NMR were recorded at 62.9 and 90.6 MHz in CDCl_3 , CD_3OD and C_6D_6 using CDCl_3 (77.0 ppm), CD_3OD (49.5 ppm) and C_6D_6 (128.0 ppm) as standard. Chemical shifts are given in parts per million (ppm). Mass spectra were recorded using electron impact (EI). Analytical TLC was carried out on Merck silica gel plates using short wave (254 nm) UV light and KMnO_4 to visualise components. Silica gel (Kieselgel 60, 40-63 μm , Merck) was used for flash column chromatography.

Titanium(III)-chloride (15% aqueous solution) was purchased from VWR-Prolabo.

General remark on the stability of carbodiazenylation products: Carbodiazenylation products which can undergo isomerization to the corresponding hydrazones should be purified by column chromatography immediately after work-up and should be kept away from acids. Traces of acid, for example from slightly acidid CDCl_3 , can cause significant isomerization. No degradation was observed over several months when the products were stored in pure state at 0°C .

Infrared-spectra

Acetic acid 3-phenyl-2-phenylazo-propyl ester (6a)

(KBr): $\tilde{\nu}$ = 3063 (w), 3028 (w), 2923 (w), 1743 (s), 1637 (w), 1592 (w), 1454 (m), 1231 (s), 1042 (m) cm^{-1} .

Acetic acid 3-(2-chloro-phenyl)-2-(2-chloro-phenylazo)-propyl ester (6b)

(KBr): $\tilde{\nu}$ = 3063 (w), 2951 (w), 1744 (s), 1640 (s), 1465 (m), 1444 (m), 1366 (m), 1230 (s), 1052 (s) cm^{-1} .

(1R,2S,3S,4S)- and (1S,2R,3R,4R)-Phenyl-(3-phenyl-bicyclo[2.2.1]hept-2-yl)-diazene (6c)

(KBr): $\tilde{\nu}$ = 3063 (w), 3026 (w), 2955 (s), 2870 (m), 1624 (w), 1602 (w), 1495 (w), 1479 (w), 1452 (m), 1118 (w) cm^{-1} .

Acetic acid 3-(4-carboxy-3-chloro-phenyl)-2-(4-carboxy-3-chloro-phenylazo)-propyl ester (6d)

(KBr): $\tilde{\nu}$ = 3428 (br), 2941 (m), 2369 (w), 1737 (vs), 1603 (s), 1386 (s), 1235 (vs), 1043 (m) cm^{-1} .

4-[3-Acetoxy-2-(4-methoxycarbonyl-phenylazo)-propyl]-benzoic acid methyl ester (6e)

(KBr): $\tilde{\nu}$ = 2950 (w), 1724 (s), 1610 (w), 1430 (m), 1282 (s), 1252 (m), 1110 (m) cm^{-1} .

2-[3-Acetoxy-2-(2-methoxycarbonyl-phenylazo)-propyl]-benzoic acid methyl ester (6f)

(KBr): $\tilde{\nu}$ = 2952 (w), 1731 (s), 1600 (m), 1434 (s), 1366 (w), 1258 (m), 1085 (m), 753 (w) cm^{-1} .

Acetic acid 3-(4-trifluoromethyl-phenyl)-2-(4-trifluoromethyl-phenylazo)-propyl ester (6g)

(KBr): $\tilde{\nu}$ = 2948 (w), 1736 (s), 1617 (m), 1327 (s), 1282 (s), 1258 (s), 1242 (s), 1165 (s), 1123 (s), 1064 (s), 1018 (m) cm^{-1} .

4-[2-(4-Methoxycarbonyl-phenylazo)-3-cyano-propyl]-benzoic acid methyl ester (6h)

(KBr): $\tilde{\nu}$ = 2950 (w), 2241 (w), 1718 (s), 1604 (m), 1431 (m), 1283 (s), 1180 (m), 1110 (m) cm^{-1} .

4-[2-(4-Methoxycarbonyl-phenylazo)-5-oxo-hexyl]-benzoic acid methyl ester (6i)

(KBr): $\tilde{\nu}$ = 2951 (w), 1720 (s), 1606 (m), 1435 (m), 1279 (s), 1180 (m), 1111 (m) cm^{-1} .

4-[3-Acetoxy-2-methyl-2-(4-methoxycarbonyl-phenylazo)-propyl]-benzoic acid methyl ester (6j)

(KBr): $\tilde{\nu}$ = 2989 (w), 2953 (m), 1746 (s), 1728 (s), 1610 (m), 1434 (s), 1278 (s), 1237 (m), 1111 (m), 1044 (m) cm^{-1} .

4-[3-Hydroxy-2-methyl-2-(4-methoxycarbonyl-phenylazo)-propyl]-benzoic acid methyl ester (6k)

(KBr): $\tilde{\nu}$ = 3505 (m), 2951 (m), 1722 (s), 1605 (m), 1435 (m), 1280 (s), 1180 (m), 1111 (m), 1053 (m) cm^{-1} .

3-(4-Methoxy-phenyl)-2-(4-methoxy-phenylazo)-2-methyl-propan-1-ol (6l)

(Film): $\tilde{\nu}$ = 3440 (s, br), 1605 (m), 1586 (w), 1513 (s), 1463 (w), 1441 (w), 1301 (w), 1249 (s), 1178 (w), 1145 (w), 1034 (m), 837 (m) cm^{-1} .

Acetic acid 3-(4-methoxy-phenyl)-2-(4-methoxy-phenylazo)-propyl ester (6m)

(Film): $\tilde{\nu}$ = 3001 (w), 2939 (m, br), 2837 (m), 1738 (s), 1603 (s), 1586 (m), 1514 (s), 1463 (m, br), 1421 (w), 1383 (w), 1365 (m), 1298 (m), 1248 (s, br), 1179 (m), 1145 (s), 1114 (w), 1034 (s, br), 984 (w), 839 (s) cm^{-1} .

4-(4-Methoxy-phenyl)-3-(4-methoxy-phenylazo)-butan-1-ol (6n)

(Film): $\tilde{\nu}$ = 3406 (w, br), 2937 (w), 2836 (w), 1604 (m), 1586 (w), 1513 (s), 1464 (w), 1442 (w), 1299 (w), 1249 (s), 1179 (w), 1146 (w), 1111 (w), 1033 (s), 837 (m) cm^{-1} .

6-(4-Methoxy-phenyl)-5-(4-methoxy-phenylazo)-hexan-2-one (6o)

(KBr): $\tilde{\nu}$ = 2958 (w), 2933 (w, br), 2892 (w), 2841 (w), 1712 (s), 1606 (m), 1585 (m), 1512 (s), 1464 (w), 1441 (w), 1416 (w), 1356 (w, br), 1320 (w), 1300 (w), 1251 (s), 1176 (m), 1146 (s), 1110 (w), 1029 (s), 841 (m), 814 (m) cm^{-1} .

Acetic acid 3-(4-methoxy-phenyl)-2-(4-methoxy-phenylazo)-2-methyl-propyl ester (6p)

(Film): $\tilde{\nu}$ = 2936 (w, br), 2836 (w), 1738 (s), 1604 (m), 1586 (w), 1514 (s), 1495 (w), 1463 (m), 1442 (w), 1383 (w), 1370 (w), 1297 (w), 1248 (s, br), 1178 (m), 1104 (w), 1036 (s), 838 (m) cm^{-1} .

3-(2,5-Dimethoxy-phenyl)-2-(2,5-dimethoxy-phenylazo)-2-methyl-propan-1-ol (6q)

(Film): $\tilde{\nu}$ = 3491 (m, br), 2995 (w), 2935 (s, br), 2833 (m), 1720 (w), 1607 (w), 1589 (w), 1504 (s), 1463 (s), 1455 (s), 1440 (m), 1428 (m), 1417 (m), 1311 (m), 1272 (s), 1222 (s), 1179 (m), 1155 (m), 865 (w) cm^{-1} .

(3-Phenyl-2-phenylazo-propyl)-carbamic acid *tert*-butyl ester (6r)

(Film): $\tilde{\nu}$ = 3352 (m, br), 3062 (w), 3027 (w), 2976 (s), 2929 (m), 1715 (s, br), 1603 (w), 1504 (s, br), 1454 (m), 1392 (m), 1366 (s), 1267 (w), 1251 (s), 1169 (s), 1098 (w), 1070 (w), 1030 (w), 1020 (w), 986 (w), 924 (w), 860 (w) cm^{-1} .

2-(3-Hydroxy-2-oxo-propyl)-benzoic acid methyl ester (17)

(Film): $\tilde{\nu}$ = 3439 (m, br), 2949 (w), 1712 (s), 1435 (m), 1404 (m), 1269 (s), 1132 (m), 1082 (s), 1056 (m), 1040 (m), 962 (w) cm^{-1} .

Acetic acid 2-[5-methoxy-3-(4-methoxy-phenyl)-1*H*-indol-2-yl]-ethyl ester (18)

(Film): $\tilde{\nu}$ = 3392 (m, br), 3032 (w), 2955 (m, br), 2833 (m), 2051 (w), 1890 (w), 1736 (s), 1612 (m), 1586 (m), 1570 (w), 1560 (w), 1509 (s), 1485 (s), 1459 (m), 1387 (w), 1364 (m), 1243 (s, br), 1176 (m), 1150 (m), 1115 (w), 1034 (s), 972 (w) cm^{-1} .

4-Acetoxy-3-(4-methoxy-phenylazo)-3-methyl-butyric acid (19)

(Film): $\tilde{\nu}$ = 2938 (m, br), 2358 (w), 1732 (s, br), 1716 (s), 1604 (m), 1587 (w), 1519 (m), 1495 (w), 1372 (m), 1252 (s, br), 1145 (m), 1104 (w), 1043 (m), 839 (m) cm^{-1} .

¹³C-NMR spectra

Acetic acid 3-phenyl-2-phenylazo-propyl ester (6a)

Date:

12 Mar 2006

Document's Title:

11

Spectrum Title:

OL-100-11

Frequency (MHz):

(f1) 62.902

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 2.1758

Spectral Width (ppm):

(f1) 239.426

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

300

Acq. Date:

Tue Nov 29 05:50:21 PM

Acetic acid 3-(2-chloro-phenyl)-2-(2-chloro-phenylazo)-propyl ester (6b)

Date:

12 Mar 2006

Document's Title:

11

Spectrum Title:

HMSW19a1
C13CPD300 C6D6 (C:\u) akbach 94

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

400

Acq. Date:

Thu Feb 16 06:25:35 PM

(1R,2S,3S,4S)- and (1S,2R,3R,4R)-Phenyl-(3-phenyl-bicyclo[2.2.1]hept-2-yl)-diazene (6c)

Date:

14 Aug 2006

Document's Title:

dl81anorbomen.mrc

Spectrum Title:

OL81-12b
C13CPD300 C6D6 (C:u) akbach 62

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

600

Acq. Date:

Fri Oct 28 01:38:28 PM

Acetic acid 3-(4-carboxy-3-chloro-phenyl)-2-(4-carboxy-3-chloro-phenylazo)-propyl ester (6d)

Date:

12 Mar 2006

Document's Title:

11

Spectrum Title:

OL-106-22
 C13CPD MeOD (C:\u) akbach 51

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

2000

Acq. Date:

Fri Jan 20 09:15:42 PM

4-[3-Acetoxy-2-(4-methoxycarbonyl-phenylazo)-propyl]-benzoic acid methyl ester (6e)

Date:

12 Mar 2006

Document's Title:

11

Spectrum Title:

HMSW20-21
 C13CPD300 C6D6 {C:\u} akbach 33

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

400

Acq. Date:

Mon Feb 20 12:10:15 AM

2-[3-Acetoxy-2-(2-methoxycarbonyl-phenylazo)-propyl]-benzoic acid methyl ester (6f)

Date:
12 Mar 2006

Document's Title:
11

Spectrum Title:
HMSW21-11
C13CPD300 C6D6 {C:\u} akbach 15

Frequency (MHz):
(f1) 90.564

Original Points Count:
(f1) 32768

Actual Points Count:
(f1) 32768

Acquisition Time (sec):
(f1) 1.5139

Spectral Width (ppm):
(f1) 239.003

Pulse Program:
ZGPG30

Temperature:
300

Number of Scans:
400

Acq. Date:
Fri Feb 17 06:47:21 PM

Acetic acid 3-(4-trifluoromethyl-phenyl)-2-(4-trifluoromethyl-phenylazo)-propyl ester (6g)

Date:

12 Mar 2006

Document's Title:

11

Spectrum Title:

HMSW25-11
C13CPD300 C6D6 (C:\u) akbach 72

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

400

Acq. Date:

Wed Feb 22 12:43:17 AM

4-[2-(4-Methoxycarbonyl-phenylazo)-3-cyano-propyl]-benzoic acid methyl ester (6h)

Date:

12 Mar 2006

Document's Title:

11

Spectrum Title:

HMSW22-11
C13CPD300 C6D6 {C:\u} akbach 40

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

400

Acq. Date:

Mon Feb 20 02:28:59 PM

4-[2-(4-Methoxycarbonyl-phenylazo)-5-oxo-hexyl]-benzoic acid methyl ester (6i)

Date:

12 Mar 2006

Document's Title:

11

Spectrum Title:

HMSW24-21
C13CPD C6D6 {C:\u} akbach 80

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

1024

Acq. Date:

Wed Feb 22 05:55:08 PM

4-[3-Acetoxy-2-methyl-2-(4-methoxycarbonyl-phenylazo)-propyl]-benzoic acid methyl ester (6j)

Date:

12 Mar 2006

Document's Title:

11

Spectrum Title:

HM131-11

Frequency (MHz):

(f1) 62.902

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 2.1758

Spectral Width (ppm):

(f1) 239.426

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

300

Acq. Date:

Fri Mar 10 04:10:55 PM

4-[3-Hydroxy-2-methyl-2-(4-methoxycarbonyl-phenylazo)-propyl]-benzoic acid methyl ester (6k)

3-(4-Methoxy-phenyl)-2-(4-methoxy-phenylazo)-2-methyl-propan-1-ol (6l)

Date:

15 Aug 2006

Document's Title:

11

Spectrum Title:

aw29
 C13CPD300 CDCl3 {C:\u} akbach 96

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZPGPG30

Temperature:

300

Number of Scans:

400

Acq. Date:

Tue Jul 11 05:32:51 PM

Acetic acid 3-(4-methoxy-phenyl)-2-(4-methoxy-phenylazo)-propyl ester (6m)

Date:

15 Aug 2006

Document's Title:

11

Spectrum Title:

HM-SW-68-11
C13CPD300 C6D6 {C:\u} akbach 74

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPC30

Temperature:

300

Number of Scans:

400

Acq. Date:

Tue Jul 25 06:01:41 PM

4-(4-Methoxy-phenyl)-3-(4-methoxy-phenylazo)-butan-1-ol (6n)

Date:

15 Aug 2006

Document's Title:

11

Spectrum Title:

HM-SW-65-21

C13CPD300 C6D6 (C:\u) akbach 50

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

400

Acq. Date:

Mon Jul 24 07:46:25 PM

6-(4-Methoxy-phenyl)-5-(4-methoxy-phenylazo)-hexan-2-one (6o)

Date:

15 Aug 2006

Document's Title:

11

Spectrum Title:

HM200-11

Frequency (MHz):

(f1) 62.902

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 2.1758

Spectral Width (ppm):

(f1) 239.426

Pulse Program:

ZGPC30

Temperature:

300

Number of Scans:

300

Acq. Date:

Wed Jul 19 06:57:27 PM

Acetic acid 3-(4-methoxy-phenyl)-2-(4-methoxy-phenylazo)-2-methyl-propyl ester (6p)

Date:

15 Aug 2006

Document's Title:

11

Spectrum Title:

HM-SW-63-11
C13CPD300 C6D6 (C:\u) akbach 61

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

400

Acq. Date:

Thu Jul 20 10:16:25 AM

3-(2,5-Dimethoxy-phenyl)-2-(2,5-dimethoxy-phenylazo)-2-methyl-propan-1-ol (6q)

Date:

15 Aug 2006

Document's Title:

11

Spectrum Title:

HM202-11
C13CPD300 C6D6 {C:\u} akbach 48

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

400

Acq. Date:

Mon Jul 24 06:00:41 PM

(3-Phenyl-2-phenylazo-propyl)-carbamic acid *tert*-butyl ester (6r)

Date:

15 Aug 2006

Document's Title:

HM205.mrc

Spectrum Title:

HM205-11
C13CPD C6D6 (C:\u) cs 2

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

1024

Acq. Date:

Thu Jul 27 12:04:09 AM

2-(3-Hydroxy-2-oxo-propyl)-benzoic acid methyl ester (17)

Date:

18 Sep 2006

Document's Title:

11

Spectrum Title:

HM-SW-94-12
C13CPD300 CDCl3 {C:\u} akbach 114

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

400

Acq. Date:

Thu Sep 14 05:43:34 PM

Acetic acid 2-[5-methoxy-3-(4-methoxy-phenyl)-1*H*-indol-2-yl]-ethyl ester (18)

Date:

15 Aug 2006

Document's Title:

11

Spectrum Title:

HM212-12
C13CPD300 CDCl₃ {C:\u} akbach 64

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPC30

Temperature:

300

Number of Scans:

400

Acq. Date:

Fri Aug 11 05:24:19 PM

4-Acetoxy-3-(4-methoxy-phenylazo)-3-methyl-butyric acid (19)

Date:

15 Aug 2006

Document's Title:

11

Spectrum Title:

HM213-12
 C13CPD300 CDCl₃ (C:u) akbach 65

Frequency (MHz):

(f1) 90.564

Original Points Count:

(f1) 32768

Actual Points Count:

(f1) 32768

Acquisition Time (sec):

(f1) 1.5139

Spectral Width (ppm):

(f1) 239.003

Pulse Program:

ZGPG30

Temperature:

300

Number of Scans:

400

Acq. Date:

Fri Aug 11 06:12:12 PM