SUPPORTING INFORMATION FOR

Phase Vanishing Methodology for Efficient Bromination, Alkylation, Epoxidation and Oxidation Reactions of Organic Substrates

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EXPERIMENTAL

The melting point for the new compound **13** is uncorrected. All solvents were collected from a Grubbs-type solvent purification system and kept under 4 Å molecular sieves. 1, 2-Dibromoethane was dried and distilled over CaH₂ and kept over 4 Å molecular sieves. ¹H and ¹³C NMR spectra were recorded on a Varian VRX-300 NMR spectrometer. Silica gel (J. T. Baker, 40-140 mesh) was used for column chromatography. High resolution mass spectra were recorded on a KRATOS MS-50 spectrometer. All chemicals were purchased from Aldrich or Fisher.

Bromination of aromatic compounds with bromine:

To perfluorohexane (1.5 mL) in a test tube was slowly added Br₂ (10 mmol). After 5 minutes, when all the bromine had settled down to the bottom of the tube, a solution of one of the aromatic compounds **1a-d** (2 mmol) in dichloromethane (1.5 mL) was carefully added to form the uppermost layer. The test tube was covered with Al foil and kept at room temperature for 1-3 days. After this period, the remaining two layers were separated and the dichloromethane layer was washed with aqueous sodium thiosulfate solution, then with water, and then it was dried over MgSO₄ followed by evaporation of

the solvent. The products **2a-d** were purified by chromatography over silica gel using 5 % ether in hexane for **2a-b** and 10% ether in hexane for **2c-d** as eluent.

Bromination of allylic and propargylic alcohols with phosphorus tribromide:

To perfluorohexane (5 mL) in a test tube was slowly added PBr_3 (10 mmol). After 5 minutes, when all the PBr_3 had settled down to the bottom of the test tube, a solution of one of the alcohols **3a-d** (30 mmol) in ether (15 mL) was carefully added to form the uppermost layer. The test tube was covered with Al foil and the bottom layer was stirred at room temperature with a magnetic stirring bar for 12 h. The remaining two layers were separated and the ether layer was washed with cold water, then with cold aqueous sodium bicarbonate followed by cold water again. The ether solution was dried over MgSO_4 and following evaporation of the solvent, the products **4a-d** were purified by atmospheric distillation.

Alkylation of a phenol derivative with dimethyl sulfate:

To perfluorohexane (1.5 mL) in a test tube was slowly added a solution of 1, 2-dibromoethane (1.5 mL) and Me_2SO_4 (2 mmol). After 5 minutes, when two layers remained, salicylic acid (2 mmol) and NEt_3 (4 mmol) was dissolved in acetone (1.5 mL) and the solution was carefully added to form the uppermost layer. The tube was covered with Al foil and the bottom layer was stirred with a magnetic stirring bar at room temperature for 2 days. The bottom layer consisting of the reaction product dissolved in 1,2-dibromoethane/acetone was separated from the fluorous phase, the dibromoethane/acetone layer was evaporated on a rotary evaporator and then the residue was purified by chromatography over silica gel. The product **5** was eluted by 10% ether in hexane.

Preparation of epoxides from alkenes with *m*-CPBA:

To perfluorohexane (1.5 mL) in a test tube was added *m*-CPBA (3 mmol) dissolved in dibromoethane (2 mL). After 5 minutes, when all of the dibromoethane solution had settled to the bottom of the tube, one of the alkenes **6a-e** (2 mmol) dissolved in dichloromethane (1.5 mL) was carefully added to form the uppermost layer. The tube was covered by Al foil and the bottom layer was stirred with a magnetic stirring bar at room temperature for 2 days. The dibromoethane/dichloromethane lower layer containing the reaction product was separated and washed with sodium bicarbonate solution (3 x 25 mL), then with water, and then it was dried over MgSO₄. The products **7a-e** were purified by chromatography over silica gel using 5% ether in hexane for **7a** and 3% ether in hexane for **7b-e** as eluent.

Preparation of N-oxides:

To perfluorohexane (1.5 mL) in a test tube was added *m*-CPBA (3 mmol) in dibromoethane (2 mL). After 5 minutes when the dibromoethane solution had settled down to the bottom of the test tube, one of the amines (2 mmol) in dichloromethane (1.5 mL) was added carefully to form the uppermost layer. The tube was then covered with Al foil and the bottom layer was stirred with a magnetic stirring bar at room temperature for 3 days. The dibromoethane/dichloromethane layer was evaporated and the products were purified by chromatography over silica gel using methanol for **8a** and ether for **8b** as eluent.

Preparation of sulfones and a sulfoxide from sulfides:

To perfluorohexane (1 mL) in a test tube was added *m*-CPBA (2 mmol) in dibromoethane (1.5 mL). After 5 minutes, when the dibromoethane solution had settled down the bottom

of the test tube, one of the sulfides (1 mmol) dissolved in dichloromethane (1 mL) was carefully added to form the uppermost layer. The tube was covered with Al foil and the bottom layer was stirred with a magnetic stirring bar at room temperature for 2-3 days. The dibromoethane/dichloromethane layer was separated and after evaporation, the products were purified by chromatography over silica gel using 50% ether in hexane for **9**, **10**, **12** and ether for **11**, **13** as eluent.

All the compounds synthesized herein are listed below, with literature citations (given in the ms) for their synthesis except for **13**. For compounds **7a**, **7d**, **10** and **12**, ^1H and ^{13}C NMR spectral data were recorded in the literature (cited in the ms) with which our data are in good agreement. For compounds **2a-d**, **4a-d**, **5**, **7b**, **7c**, **7e**, **8a**, **8b**, **9** and **11**, such data were found neither in the ms reference given nor elsewhere in the literature. However, our spectra are entirely consistent with their formulations.

2, 4, 6-Tribromo phenol^{4b,c,e,h} (**2a**): ^1H NMR (CDCl_3 , 300 MHz): δ 5.87 (s, 1H), 7.58 (s, 2H)

^{13}C NMR (CDCl_3 , 75.5 MHz): δ 110.6, 112.8, 134.4, 149.1

2, 4, 6-Tribromo-3, 5-dimethyl phenol^{4e,f} (**2b**): ^1H NMR (CDCl_3 , 300 MHz): δ 2.58 (s, 6H), 5.99 (s, 1H)

^{13}C NMR (CDCl_3 , 75.5 MHz): δ 24.9, 109.5, 117.7, 137.1, 148.2

2, 4, 6-Tribromo aniline^{4d,g} (**2c**): ^1H NMR (CDCl_3 , 300 MHz): δ 4.56 (s, 2H), 7.50 (s, 2H)

^{13}C NMR (CDCl_3 , 75.5 MHz): δ 108.9, 133.9, 141.5

2, 4, 6-Tribromo *N*-methyl aniline^{4d,g} (2d): ¹H NMR (CDCl₃, 300 MHz): δ 2.58 (s, 3H), 4.07 (brs, 1H), 6.21 (m, 1 H), 7.02 (m, 1H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 30.7, 107.9, 109.8, 111.8, 131.4, 134.2, 145.2

Propargyl bromide^{5a-b} (4a): ¹H NMR (CDCl₃, 300 MHz): δ 2.52 (t, *J* = 2.7 Hz, 1H), 3.86-3.87 (d, *J* = 2.7 Hz, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 13.2, 74.6, 78.5

1-Methyl propargyl bromide^{5a-c} (4b): ¹H NMR (CDCl₃, 300 MHz): δ 1.89-1.92 (d, *J* = 6.8 Hz, 3H), 2.62-2.63 (d, *J* = 2.3 Hz, 1H), 4.50-4.60 (m, 1H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 27.3, 30.0, 74.3, 83.9

Allyl bromide^{5d-e} (4c): ¹H NMR (CDCl₃, 300 MHz): δ 3.92-3.95 (d, *J* = 7.3 Hz, 2H), 5.13-5.16 (d, *J* = 10 Hz, 1H), 5.31-5.35 (d, *J* = 10 Hz, 1H), 5.96-6.10 (m, 1H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 33.5, 119.7, 134.8

Crotyl bromide^{5f-g} (4d): ¹H NMR (CDCl₃, 300 MHz): δ 1.68-1.70 (m, 3H), 3.88-3.90 (m, 2H), 5.61-5.70 (m, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 17.3, 33.1, 127.3, 130.9

2-Methoxy benzoic acid^{6a-c} (5): ¹H NMR (CDCl₃, 300 MHz): δ 3.94 (s, 3H), 6.85-6.99 (m, 2H), 7.42-7.48 (m, 1H), 7.81-7.84 (m, 1H), 10.76 (s, 1H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 52.5, 112.6, 117.8, 119.4, 130.1, 135.9, 161.8, 170.8

Styrene epoxide^{7h} (7a): ¹H NMR (CDCl₃, 300 MHz): δ 2.79-2.82 (dd, *J* = 5.5, 2.6 Hz, 1H), 3.13-3.17 (dd, *J* = 5.5, 4.0 Hz, 1H), 3.86-3.88 (dd, *J* = 4.0, 2.6 Hz, 1H), 7.28-7.37 (m, 5H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 51.3, 52.5, 125.6, 128.3, 128.6, 137.8

Cyclohexene epoxide^{7f,g,k-n} (**7b**): ¹H NMR (CDCl₃, 300 MHz): δ 1.24-1.43 (m, 4H), 1.78-1.97 (m, 4H), 3.12 (s, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 19.7, 24.7, 52.4

Cycloheptene epoxide^{7f,k-m} (**7c**): ¹H NMR (CDCl₃, 300 MHz): δ 1.11-1.58 (m, 6H), 1.85-1.90 (m, 4H), 3.03 (t, *J* = 2.9 Hz, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 24.2, 28.7, 30.8, 55.7

cis-Cyclooctene epoxide^{7j} (**7d**): ¹H NMR (CDCl₃, 300 MHz): δ 1.17-1.58 (m, 10H), 2.05-2.09 (m, 2H), 2.83 (t, *J* = 3 Hz, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 25.7, 26.4, 26.6, 55.6

1-Octene epoxide^{7m,o} (**7e**): ¹H NMR (CDCl₃, 300 MHz): δ 0.88 (m, 3H), 1.27-1.51 (m, 10H), 2.42-2.44 (dd, *J* = 5.0, 2.9 Hz, 1H), 2.70-2.73 (dd, *J* = 5.0, 4.0 Hz, 1H), 2.85-3.89 (m, 1H)

¹³C NMR (CDCl₃, 300 MHz): δ 13.8, 22.3, 25.7, 28.9, 31.5, 32.3, 46.8, 52.1

Pyridine N-oxide^{8c-h} (**8a**): ¹H NMR (CDCl₃, 300 MHz): δ 7.04-7.05 (m, 3H), 7.95-7.97 (m, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 125.8, 138.7

8-Hydroxy quinoline N-oxide^{8d-e} (**8b**): ¹H NMR (CDCl₃, 300 MHz): δ 6.99-7.2 (m, 3H), 7.41-7.46 (m, 1H), 7.73-7.76 (m, 1H), 8.20-8.22 (m, 1H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 115.0, 116.9, 120.5, 129.9, 130.7, 132.4, 134.7, 154.1

3(Methyl propanoate)phenylsulfone^{9i,l} (**9**): ¹H NMR (CDCl₃, 300 MHz): δ 2.73 (t, *J* = 7.5 Hz, 2H), 3.41 (t, *J* = 7.5 Hz, 2H), 3.61 (s, 3H), 7.53-7.68 (m, 3H), 7.87-7.90 (m, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 27.8, 51.6, 52.4, 128.3, 129.6, 134.2, 138.6, 170.5

4(Butan-2-one)phenylsulfone^{9k} (10): ¹H NMR (CDCl₃, 300 MHz): δ 2.11 (s, 3H), 2.86

(t, *J* = 7.5 Hz, 2H), 3.32 (t, *J* = 7.5 Hz, 2H), 7.50-7.64 (m, 3H), 7.83-7.86 (m, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 29.9, 35.9, 50.5, 127.9, 129.5, 134.0, 138.9, 203.8

3(Propane nitrile)phenylsulfone^{9h,l} (11): ¹H NMR (CDCl₃, 300 MHz): δ 2.88 (t, *J* = 7.5

Hz, 2H), 3.38 (t, *J* = 7.5 Hz, 2H), 7.56-7.73 (m, 3H), 7.89-7.91 (m, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 12.1, 51.1, 116.3, 128.3, 129.8, 134.8, 137.6

3(Cyclohexanone)phenylsulfone^{9k} (12): ¹H NMR (CDCl₃, 300 MHz): δ 1.55-2.16 (m,

2H), 2.17-2.42 (m, 4H), 2.43-2.57 (m, 2H), 3.23-3.34 (m, 1H), 7.55-7.57 (m, 3H), 7.60-

7.66 (m, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 23.6, 23.8, 40.5, 40.6, 62.5, 129.1, 129.6, 134.4, 136.8,

206.7

3(Cyclohexanone)phenylsulfoxide (13): ¹H NMR (CDCl₃, 300 MHz): δ 1.77-1.98 (m,

2H), 2.11-2.36 (m, 2H), 2.80-3.09 (m, 1H), 3.12-3.23 (m, 2H), 4.14-4.30 (m, 2H), 7.5-

7.70 (m, 3H), 7.72-7.85 (m, 2H)

¹³C NMR (CDCl₃, 75.5 MHz): δ 23.6, 23.8, 40.5, 40.6, 62.5, 129.1, 129.6, 134.4, 136.8,

206.6. HRMS (M⁺): calculated for C₁₂H₁₄O₂S 222.07145, found 222.07175. m.p. 161 °C.

STANDARD 1H OBSERVE

STANDARD OBSERVE

STANDARD III OBSERVE

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 PULPROG : s2pul
 RO : 20 Hz
 SFO1 : 299.9422137 MHz
 SW : 13.3359 ppm
 SW_h : 4000.000 Hz
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 YMIN_a : -116125.1484375

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7.2598

4a

STANDARD OBSERVE

STANDARD H1 OBSERVE

4c

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DSPFVS    : 0
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SFO1      : 299.9422137 MHz
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YMAX_a    : 107777.5781250
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STANDARD 1H OBSERVE

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STANDARD 1H OBSERVE

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 NUC1 : ¹H
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 SW : 13.3359 ppm
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 TD : 29952
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STANDARD 1H OBSERVE

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STANDARD 1H OBSERVE

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 SW : 13.3359 ppm
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7.2600

STANDARD 1H OBSERVE

7.2600

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STANDARD 1H OBSERVE

*** Current Data Parameters ***

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EXPNO : 1

PROCNO : 2

*** Acquisition Parameters ***

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DATE_d : Mar 20 2003

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DECNUC : 1H

DIGTYP : 16Bit

DS : 1

DSPFVS : 0

EXP : 0

FW : 4400.00 Hz

INSTRUM : varian

NS : 16

NUC1 : 1H

O1 : 400.00 Hz

PULPROG : s2pul

RO : 20 Hz

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TD : 29952

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YMIN_a : -134426.2500000

STANDARD 1H OBSERVE

*** Current Data Parameters ***

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PROCNO : 2

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DATE_d : Mar 24 2003
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STANDARD III OBSERVE

STANDARD 1H OBSERVE

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8b

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 NS : 16
 NUC1 : 1H
 O1 : 400.00 Hz
 PULPROG : s2pul
 RO : 20 Hz
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 SW_h : 4000.000 Hz
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STANDARD 1H OBSERVE

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7.6560
7.6523
7.6479
7.6393
7.6316
7.6202
7.6108
7.6072
7.5575
7.5323
7.5127
7.5083
7.2600

STANDARD 1H OBSERVE

*** Current Data Parameters ***

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 DECNUC : ¹H
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 DSPFVS : 0
 EXP :
 FW : 4400.00 Hz
 INSTRUM : varian
 NS : 16
 NUC1 : ¹H
 O1 : 400.00 Hz
 PULPROG : s2pul
 RO : 20 Hz
 SFO1 : 299.9422137 MHz
 SW : 13.3359 ppm
 SW_h : 4000.000 Hz
 TD : 29952
 YMAX_a : 115218.9218750
 YMIN_a : -115218.9218750

3.4079
 3.3831
 3.3575
 2.8142
 2.7910
 2.7889
 2.7641

7.9193
 7.9169
 7.8920
 7.7305
 7.7264
 7.7028
 7.6808
 7.6772
 7.6202
 7.5966
 7.5937
 7.5693
 7.2600

STANDARD 1H OBSERVE

STANDARD 1H OBSERVE

*** Current Data Parameters ***

NAME : nj242h2.win
 EXPNO : 1
 PROCNO : 2

*** Acquisition Parameters ***

AQ_mod : qsim
 BF1 : 299.9418137 MHz
 BF2 : 299.9414137 MHz
 BYTORDA : 0
 DATE_1 : 12:04:54
 DATE_d : Apr 17 2003
 DE : 115.7 usec
 DECIM : 1
 DECNUP : 1H
 DIGTYP : 16Bit
 DS : 1
 DSPFVS : 0
 EXP :
 FW : 4400.00 Hz
 INSTRUM : varian
 NS : 16
 NUC1 : 1H
 O1 : 400.00 Hz
 PULPROG : s2pul
 RO : 20 Hz
 SFO1 : 299.9422137 MHz
 SW : 13.3359 ppm
 SW_h : 4000.000 Hz
 TD : 29952
 YMAX_a : 144102.1093750
 YMIN_a : -144102.1093750

13

170.6426

135.9672

134.1936

129.3321

128.7444

77.2300

76.8062

76.3824

68.2371

58.5318

33.5546

27.3326

26.9622

*** Current Data Parameters ***

NAME : nj242c2.win

EXPNO : 1

PROCNO : 2

*** Acquisition Parameters ***

AQ_mod : qsim

BF1 : 75.4277928 MHz

BF2 : 299.9414137 MHz

BYTORDA : 0

DATE_t : 12:09:18

DATE_d : Apr 17 2003

DE : 115.7 usec

DECIM : 1

DECNUC : 1H

DIGTYP : 16Bit

DS : 4

DSPFVS : 0

EXP : 0

FW : 19600.00 Hz

INSTRUM : varian

NS : 32768

NUC1 : 13C

O1 : 300.00 Hz

PULPROG : s2pul

RO : 20 Hz

SFO1 : 75.4280928 MHz

SW : 233.4104 ppm

SW_h : 17605.634 Hz

TD : 60032

YMAX_a : 122118.2187500

YMIN_a : -122118.2187500

Manual Peak Matching Report For Accurate Mass Determination

Theoretical mass	Experimental mass	PFK matching mass	Deviation*
222.07145	222.07175	218.98562	1.4 ppm

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass} - \text{theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only ^{12}C , ^1H , ^{16}O , ^{14}N etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

SPEC: fin030265.dat (02-JUN-03 14:36:40)

Samp: 242

Conn: 70 eV EI

Oper: kh

Base: 40.08

Peak: 1000.0 mmu

Scan 2 @ 0.17 min (EI +Q3MS LMR UP LR)

Study: ms services

Masses: 35.01 > 650.00

Intensity: 73720

Scans: 1 > 158

Client: Jana

#Peaks: 269

RIC: 559739

2.9E+04

Jan 2 14:50:43 2003 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98