Diborane Dilution Procedure for HF-CVD Reactor - 26/08/2010

Pressure Units

* Diborane regulator
  + High pressure gauge
    - Two sets of units (bar and psi)
  + Low pressure gauge
    - Reads positive and negative pressures with respect to

atmospheric pressure

* + - Positive pressure: two sets of units (bar and psi)
    - Negative pressure: two sets of units (bar and o)
* Hydrogen regulator
  + High pressure gauge
    - Two sets of units (bar and lb/in2)
  + Low pressure gauge
    - Two sets of units (bar and lb/in2)
* Chamber pressure gauge
* Torr
* Conversion factor
  + 1 standard atmosphere (1 atm) = 1.01325 bar
  + 1 atm = 760 Torr
  + 1 atm = 760 millimetres of mercury (760 mmHg)
  + 1 atm = 30 inches of mercury (30 in Hg or 30o)
  + 1 pound per square inch (1 psi or 1 lb/in2)  0.069 Bar

See schematic diagram of gas system at end of document for details on valve names, etc

#### Section 1: Preparing the System

Ensure CVD chamber is up and running

* Check pump is working and both wide and narrow bore pipes are fully open
* Check exhaust line plumbed in
* Check vent valve is closed
* Check that pressure gauge is open and reading zero (ignoring any offset)

Pump down system including diborane reservoir

(M1, MFC3, M3, R1, R2, R3, R4, D5, D6, D7 and D8 open, keep valves MFC1, MFC2, MFC4, M2, R6 and R7 closed)

When pumped down (may take a few hours) close valves M3, D5, D6, D7, R1, R2, R4 and regulator D3.

#### Section 2: Adding  5 % Diborane Premix to Reservoir

Open main cylinder valve D1

# Check pressure on gauge D2

bar (e.g. 73 bar)

Psi (e.g. 1100 bar)

# Check pressure on gauge D4

bar (e.g. 1 atm – 0.5 bar)

o (e.g. 1 atm – 15o) 1o = 1 in Hg (inch of mercury)

Leave regulator D3 closed

Check that valve M3 and MFC3 and D7, D8 are closed.

Open valve D5

Gauge D4 should fall to zero

(1 atm – 1.0 bar)

(1 atm – 3.0o)

Open regulator D3 slowly until gauge D4 reads 1 atm – 0.5 bar (1 atm – 15o)

## Note some leak through MFC3

# Read gauge D4

bar (e.g. 1 atm – 0.5 bar)

o (e.g. 1 atm – 15o) 1o = 1 in Hg (inch of mercury)

### Open valve R1

# Open valve R2

# Open valve R3

### Allow for a few seconds for a system to reach equilibrium

**Reading 1 (required for calculations)**

Final gauge D4 reading

bar (e.g. 0.5 bar)

o (e.g. 1 atm – 15o) 1o = 1 in Hg (inch of mercury)

### Close valve R1

### Close valve R2

### Close valve R3

**Close valve D1**

# Now pump out excess diborane from lines.

# Open by-pass valve M3, and D7, D8, MFC3.

# Open valve D3 fully.

(Open valve M3 first to keep gas pressure down in the section of gas line from D5 to MFC3/M3).

Diborane will discharge through chamber, expect a peak pressure  100 Torr through the chamber.

D2 = 50 (to 60)bar/750 psi

# D4 = 2.6 bar + 1 atm, 28 psi +1 atm

# Pump down to vacuum: check gauges D2, D4 and the chamber pressure gauge

(expect pump down to take  2 minutes)

#### Section 3: First Addition of Hydrogen to Reservoir

# Check valve H1 is open, and valve D7is closed.

Gauge H2 = 147 bar, 2133 lb/in2

# Gauge H4 = 2 bar, 29 lb/in2

Open valve H5

# Open valve R5.

Open valve D6 (gauge D4 1 atm – 0.375 bar, 1 atm – 10o)

*(note: gauge D2 will not register- pressure too low)*

# Check regulator D3 fully open.

# Gauge D4 reading.

bar (e.g. 1.6 bar +1 atm)

o (e.g. 24 psi +1 atm)

Open valve D5 (MFC3 will shoot off the scale temporarily).

# Open valves R1, R2.

Open valve R3 for three seconds.

Close valves R1, R2 and R5.

**Reading 2 (required for calculations)**

# Final gauge D4 reading

bar (e.g. 1.6 bar +1 atm)

o (e.g. 24 psi +1 atm

### Now pump out excess hydrogen from lines.

Open valve M3, MFC3, D6, D7.

### Hydrogen will discharge through chamber, expect a peak pressure  40 Torr through the chamber.

### Pump down to vacuum: check gauges D2, D4 and the chamber pressure gauge

(expect pump to take  2 min)

After fully pumped down, close valve D5

Leave valve M3, MFC3, D6, D7 open.

#### Section 4: Reducing Pressure in the Reservoir

### Open valves R1, R2, R4

### D4 = 1.3 bar, 18 psi

Open valve D5 gently until gauge D4 falls to desired pressure, then close valve D5:

**Reading 3 (required for calculations)**

# Final gauge D4

bar (e.g. 1 atm – 0.5 bar)

o (e.g. 1 atm – 15o) 1o = 1 in Hg (inch of mercury)

### Close valves R1, R2, R4.

### Now pump out excess mixture from lines

### Open gauge D5 fully

### Excess mixture will discharge through chamber, expect a peak pressure  3 Torr through the chamber

### Pump down to vacuum: check gauges D2, D4 and the chamber pressure gauge

(expect pump down to take  2 minutes)

Close valves D6, D7.

#### Section 5: Second Addition of Hydrogen to the Reservoir

### Close D5, D3

# Open R5

Open D6, D3 fully (note gauge D2 will not register – pressure too low)

### Gauge D4 reading

bar (e.g. 1.6 bar +1 atm)

o (e.g. 24 psi +1 atm)

Open valves R1 and R2

### Open D5

### Open valve R3 for three seconds.

**Reading 4 (required for calculations)**

# Final gauge D4 reading

bar (e.g. 1.6 bar +1 atm)

o (e.g. 24 psi +1 atm

### Close valves R1, R2 and R5

### Now pump out excess mixture from lines

### Open valve D7

### Hydrogen will discharge through the chamber, expect a peak pressure  40 Torr through the chamber

### Pump down to vacuum: check gauges D2, D4 and the chamber pressure gauge

(expect pump down to take  2 minutes)

### Close valve D5

#### Section 6: Reducing the pressure in the Reservoir

### Open valves R1, R2, R4

### D4 = 1.3 bar, 18 psi

Open valve D5 gently until gauge D4 falls to desired pressure, then close valve D5:

**Reading 5 (required for calculations)**

# Final gauge D4

bar (e.g. 1 atm – 0.5 bar)

o (e.g. 1 atm – 15o) 1o = 1 in Hg (inch of mercury)

### Close valves R1, R2, R4

### Now pump out excess mixture from lines

Open gauge D5 fully

### Excess mixture will discharge through chamber, expect a peak pressure  3 Torr through the chamber.

### Pump down to vacuum: check gauges D2, D4 and the chamber pressure gauge

(expect pump down to take  2 minutes)

Close valve D7

#### Section 7: Third Addition of Hydrogen to Reservoir

### Close D5, D3

# Open R5

# Open D6, D3 fully

### Gauge D4 reading

bar (e.g. 1.6 bar +1 atm)

o (e.g. 24 psi +1 atm)

(note gauge D2 will not register – pressure too low)

Open valves R1 and R2

Open D5

### Open valve R3 for three seconds.

**Reading 6 (required for calculations)**

# Final gauge D4 reading

bar (e.g. 1.6 bar +1 atm)

o (e.g. 24 psi +1 atm

### Close valves R1, R2, R5, H5

### Now pump out excess mixture from lines

Open valve D7

### Hydrogen will discharge through the chamber, expect a peak pressure  40 Torr through the chamber

Pump down to vacuum: check gauges D2, D4 and the chamber pressure gauge

(expect pump down to take  2 minutes)

Leave valves D3, D4, D6, D7 and D8 pumping down

Open MFC3 (to maximum)

**Section 8: Long Term Storage of the Diborane Regulator.**

Rather than to leave the diborane regulator under vacuum, which might cause damage to the diaphragm, fill it with 1 atm of hydrogen. Ensure the cylinder is open.

Open H5

Close D5

Open R5

Gauge D4 shows  1 atm

Close R5, H5, D6 and D3

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Calculation:

Mole fraction of fully diluted diborane = 

Where *R* is the initial mole fraction of diborane in the cylinder (0.0475), and *pD1* is the pressure of the first fill of the diborane mixture (usually 0.5 bar) and *pH1* is the pressure of the first fill of hydrogen dilutant (normally 2.6 bar), and *r* means the reading numbers mentioned in the script.

So typically, mole fraction of diborane after 3 equal dilutions is:



When this is passed through the MFCs at a ratio of diborane:H2 of 0.5% (1:200), then the actual mole fraction of diborane in the chamber is ~1.7 ppm.



Image is also online on the Budgie intranet.