7/3 cont.

· fill w/ methane

· use "Raman hydrate" "Methane only" tube + turn on hood

· on gas side, finger-tight + 7/16 wrench

· rotate regulator to 200 psi (only 1 turn max)

5:26 pm -10.40 psig

· open Valve A3

→ ~~220~~ 223 psig

· close valve A3

· rinse w/ methane multiple times (2x)

0 psig → 223 psig → 0 ...

· keep fill + deliver valves open during, open valve A3 + valve & B3

final fill = 224.4 psig

· Open regulator to vent off methane

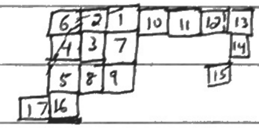
7/5 = ~~D05~~ D06   2108 psig, 14.54 MPag, U 6.6 °C, L 6.72 °C, A 24.8 °C   Pump = 2100 psig V_g = 5.490618 mL

· Look around + montage/map area (JPG + BMV, Paint)

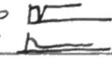
(0,0,0)

B

(6137.3, -4799, 0)



P, T



· Pressure dropped as T was brought down

MT 01 (~~MON01~~)	8000 / 10000	3000 / 5000	-6818.2 / -6818.2	· Dark region @ top of MT · Bright, kind of sparkly stuff
MT02	6200 / 8200	3000 / 5000	" / "	· Left of MT01 · Sparkly stuff, Interface
MT03	~~6400~~ 6200 / 8200	~~"~~ 4800 / 6800	~~"~~ " / "	· Sparkly, below 02
9:00 MT04	4400 / 6400	4800 / 6800	" / "	· Interface
MT05	4400 / 6400	6600 / 8600	" / "	· Also interface
MT06	4400 / 6400	3000 / 5000	" / "	· Small more "hydrate"
MT07	8000 / 10000	4800 / 6800	" / "	
MT08	6200 / 8200	6600 / 8600	" / "	
MT09	8000 / 10000	6600 / 8600	" / "	
MP04	4400 / 6400	4800 / 6800	" / "	250 nm steps, 9x9, Raster, 2 sec, 100 pwr, 1 acc

UR - majority of hydrate & small vapor peak larger

· contains mixture of peak intensities, including 1:1

MT10	9800 / 11800	3000 / 5000	" / "	· Sparkling + smooth hydrate interface
MT11	11600 / 13600	3000 / 5000	" / "	
MT12	13400 / 15400	3000 / 5000	" / "	~~Hydrate H_2O interface~~
MT13	15200 / 17200	3000 / 5000	" / "	· Left edge of cell
MT14	15200 / 17200	4800 / 6800	" / "	
MT15	13400 / 15400	6600 / 8600	" / "	
MT16	4400 / 6400	8400 / ~~8~~10400	" / "	
MT17	2600 / 4600	8400 / 10,400	" / "	

, Map across interface

MP04

↓

1 2 3 4 5 6 7 8 9

1 S L E S S
2 S S S S L
3 S S ~~E~~ E S L
4 S V L S S S L
5 V V V S E E E S L
6 V E S S S S S S
7 E S S E E S S S
8 V V S S V S S S S S
9 N S S S S S S S S

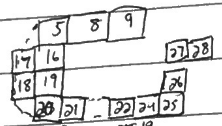
Messed up chart, BUT

· likely vapor (CH_4) boundary

· could be indicative of non-equilibrium

Blowing air → T up + pressure spikes + hydrate formation

7/5 cont



MT18 2600 10200 -6818.2
4600 12200 -6818.2

MT19
4400 10200 "
6400 12200 "

MT20
4400 ~~4600~~ 12000 "
6400 ~~6600~~ 14000 "

MT21
6200 12000 "
8200 14000 "

MT22
8000 12000 "
10000 14000 "

MT23
8000 13800 "
10000 15800 "

MT24 9800 12000 "
11800 14000 "

MT25
11600 12000 "
13600 14000 "
– Top left of cell

MT26
11600 10200 "
13600 ~~12000~~ 12200 "

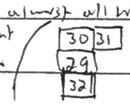
MT27
11600 8400
13600 10400

MT28
13400 8400 "
15400 10400 "

11:36 MT29 = MT04
4400 4800
6400 6800
Boundry shifted - lowering T → formation

MP29
1 ~~sec~~ 100 pwr 1 acc
Now almost all hydrate

Bottom right part of cell →



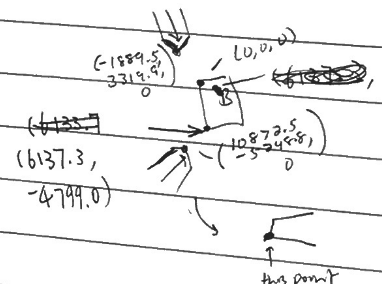
MT30
4400 3000 "
6400 5000 "

MT31
6200 3000 "
8200 5000 "

MT32
4400 6600
6400 8600

~~MP30~~
12:00 " " "
3:32
9×9, 1 ~~sec~~ LR, 100pwr, 1acc, 2 sec LR
• Small ... or even most

3D01 X, 6150 X, 2400 -6818.2
4:08 Y, 5750 Y, 5850 -6838.2 11×5×5 1 sec, 100pwr, 1acc
25um
• Spotty pore

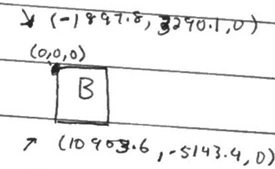


7/6 D07 14.54MPag 2168 psig, 4.43°C U
5.57°C L
24.61°C A
9:45 AM

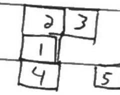
Pump B
2000psi + 5.5479 mL
A
459.4 psi + 1.0015 mL

- A few pressure drops overnight

• 7.19°C upper after morning



MT01 = MT04 + MT29 from D06
4400 4800 -6951
6400 6800 -6951



' Same as MT 29-32 D06

MT02
4400 3000 "
6400 5000 "

MT03
6200 3000 "
8200 5000 "

MT04
4400 6600 "
6400 8600 "

• Map areas, 2D + 3D

MP01
4400 4800 " 250um step
6400 6800 " 9×9 Raster
2 sec, 100pwr, 1acc

MP02
4400 3000 " "
6400 5000 " "
2 sec, 100 pwr, 1 acc

MP01 = same area as ~~MP04 and MT29~~ D06

S = small cage pk.
E = ~1:1 ratio
L = lg. cage pk.

	1	2	3	4	5	6	7	8	9
1	E	E	S	L	S	S	S	E	S
2	E/S	E	S	E	E	S	S	E	L
3	E/L	E	E	S	S	L	L	S	L
4	L	S	S	L	E	E	S	L	L
5	L	E	S	L	E	L	L	S	E
6	S	~~E~~	S	S	L	L	L	S	E
7	—	S	S	S	-	L	S	S	S
8	E	E	E	S	S	S	S	E	L
9	S	S	L	S	L	E	L	L	S

3D01 6000 5750 -6951
6250 5850 -6971 1 sec, 100pwr, 1acc
• Same pore as 3D01 from D06

11:50
T upper Thermistor = 9.27°C

MT05 8000 6600 -6971
10000 8600 -6971

3D02 9500 7000 -6971
9750 7100 -6991 11×5×6 1 sec, 100 pwr, 1acc

MP04 4400 7350 -6822.5
4700 7500 -6822.5 13×7 Raster Nothing, not hydrate pore

1:00
move

7/7 Hydrate Meeting

- Methane-Ethane-H_2O diagram - JC
  - C2+ = Methane+Ethane?
- Thermogenic hydrate C2+ compared to biogenic mostly methane
- More Ethane = more profitable
- Degrees of freedom in phase diagrams
- Lower P = more ethane hydrate in comparison
- Make sure > methane-ethane than H_2O? → est. whether cage preference or SII
  - or use cage ratios to det. SII or preference

2PM

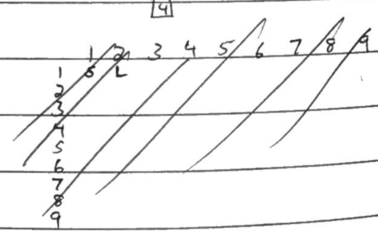
D08 14.51 MPag, 2105.8 Psig, 3.88°C (U), 4.24°C (L), 24.4°C (A) Pump B 2100 psi + 8.573 mL, A 457.3 psi, 1.00 mL

↓ (-1899.6, 3305.9, 0)
(0,0,0) [B]
↑ (10872.4, -5230.4, 0)

A few pressure drops overnight (1 group of drops)

MT01	4400 / 6400	4800 / 6800	-6795.2 / -6795.2	Clearer than yesterday
MT02	4400 / 6400	3000 / 5000	"	
MT03	6200 / 8200	3000 / 5000	"	
MT04	4400 / 6400	6600 / 8800	"	
MP01	4400 / 6400	4800 / 6800	"	Raster 250 um, 9×9

[2][3] / [1] / [4]



Lower brightness ↑ for future reference

- Mixture of peaks, fewer equal peaks

MP02	4400 / 6400	3000 / 5000	"	2 sec LR
MT05	8000 / 10000	6600 / 8800	"	
3D01	6000 / 6250	5700 / 5800	-6795.2 / -6815.2	

MP06 = for poster from MT01 250 um
5 sec., 100 pwr, 1 acc 9×9
4400 / 6400 4800 / 6800 -6795.2 / -6795.2

5:00 PM end

14.53 MPag, 2106.8 Psig, 5.61°C, 5.75°C, 24.67°C

5.567 mL

7/8 D09 14.53 MPag, 2107 psig, 3.38°C (U), 3.9°C (L), 23.87°C (A) B. 2100 psig, 5.601 mL

4:30PM

A 452.3 psig, 1.0015 mL

- 2 minor P drops overnight, P drop when moved cart, like ↑ increased

(-1949.5, 3295.6, 0)
↙
(0,0,0) [B]
↗ (10988.5, -5005.7, 0)

MT01	4400 / 6400	4800 / 6800	-6890 / -6890		
MT02	4400 / 6400	3000 / 5000	"		
MT03	6200 / 8200	3000 / 5000	"		
MT04	4400 / 6400	6600 / 8800	"		
MP01	4400 / 6400	4800 / 6800	"	9×9 Raster	2 sec LR / 1 sec UR

[2][3] / [1] / [4]

- Still mixture of peaks, though more large peak greater intensity

MP02	4400 / 6400	3000 / 5000	"	9×9 Raster	"
MT05	8000 / 10000	6600 / 8800	"		
3D01	6000 / 6250	5800 / 5900	-6890 / -6900	1 sec UR / 2 sec LR	25 um, 11×5×5

6:00 PM end

- Close to eq., looks similar to yesterday

Tomorrow - same MT and mappings

- 3D of more pores

~~7/9 D13 14.52 MPag, 2105 Psig, 4.0°C (U), 4.43°C (L), 23.66°C (A), B 2100 psig, 5.61 mL, A 400 Psig, 1.00149 mL~~

12:45 PM

7/11 - D13 Still Pump error CRC Check

14.53 MPag, 2106.84 Psig, 4.8°C (U), 4.53°C (L), 24.27°C (A)
B 2100 Psig, 5.62 mL
A 452 Psig, 1.00149 mL

↘ (-1871.1, 3316.8, 0)

(0,0,0) [B]

↗ (10860, -5296, 0)

MT01	4400	4800	-6905.3				
	6400	6800	-6905.3				
MP01	4400	4800	"	9x9 Raster	2 sec LR	~ 6184	small peaks larger
	6400	6800	"	250 um	1 sec UR	~ 10181	even peaks
MT02	4400	3000	"				
	6400	5000	"				
MP02	4400	3000	"	9x9 Raster			
	6400	5000	"	250 um			
MT03	6200	3000	"				
	8200	5000	"				
MT04	4400	6600	"				
	6400	8600	"				
MT05	8000	6600	"				
	10000	8600	"				
3D01	6100	5750	-6905.3 ~~13x7x5~~	13x7x5	2 sec ~~UR~~ LR		
	6400	5900	-6925.2	25 um	1 sec UR		

3: • Possible coating of grains, including those underneath

4: • Forgot to save mappings

3D02	6100	3500	-6889.4	9x9x5	2 sec LR
	6300	3700	-6909.4		1 sec UR

• whitish spot in middle correspond w/ small peak more intense
(very faint white)

2:45 PM

MP06 - within 3D02 - across pore

6100	3630	-6909.4	1 um		1 sec UR
6300	3631	-6909.4	201x2 Raster		

• maybe equilibrium first by grains

7/12 D13 14.52 MPag, 2106.7 Psig, 4.30°C / 4.12°C, 23.91°C
B 2100 Psig, 5.633 mL
A 449.3 Psig, 1.00149 mL

12:45 PM • Pressure drops overnight still

↘ (-1876.3, 3343.6, 0)

(0,0,0) - [B]

↗ (10902.8, -5221.8, 0)

MT01	4400	~~4800~~ 4800	-6759.6		~~Sparkly~~ Sparkly regions in montage
	6400	6800	-6759.6		
MP01	4400	4800	-6831.3		
	6400	6800	-6831.3		
MT02	4400	3000	"		
	6400	5000	"		
MP02	4400	3000	"	9x9 raster	Edge of cell slower equilibrium?
	6400	5000	"		
MT03	6200	3000	"		
	8200	5000	"		
MT04	4400	6600	"		
	6400	8600	"		
MT05	8000	6600	"		
	10000	8600	"		
3D01	6100	5750	-6877.5	13x7x5	1 sec LR
	6400	5900	-6897.5	25 um	1 sec UR

• still coating grains

3D02	6100	3500	-6882.2			
	6300	3700	-6902.2			
~~3D02~~ 3D06	"	"	-6882.2	UR .5 sec		Deeper 3D02
	"	"	-6927.2	13x7x10		
3D07	"	"	-6882.2			
	"	"	-6982.2	.1 sec		Even deeper

• Still has SL~~[illegible]~~ region in middle of pore

• Edge of grain large cage more intense, but intensity decreases as go deeper

MP05 - into pore

8000	6600	-6803.1	9x9 raster
10000	8600	-6803.1	

• Some small cage Z intensity - is it possible @ equilibrium w/ small cage > intense @ middle of pore?

• Maybe large cages 6 fewer fits w/ SiO_2 of beads

3:30 PM end

7/13 - D14

↘ (-1986.9, 3243.9, 0)

(0,0,0) [B]

2 pm ↗ (11033.8, -4886.9, 0)

MT01 4400 4800 -6938.4
6400 6800 -6938.4

MP01 4400 4800 " 9x9 Raster 250 nm
6400 6800 "

MT02 4400 3000 "
6400 5000 "

MP02 4400 3000 "
6400 5000 "

MT03 6200 3000 "
8200 5000 "

MT04 4400 6600 "
6400 8600 "

MT05 8000 6600 "
10,000 8600 "

3D01 5900 5750 -6958.4 11x9
6150 5950 -6958.4 25 nm deep

3D02 5900 3600 -6958.4 25 nm 2903.3 peak
6150 3750 -6978.4 20 nm deep vs. normal 2902

3D06 ~~MP06~~ 6200 ~~7400~~ 7500 -6915.2 ~~-6905.1~~ 10 nm ~~15x14~~ 31x31x2 0.2 sec UR 2902.8, ~~2913.0~~ 2913.01
6500 ~~6700~~ 7800 ~~-6905.1~~ -6965.2

• Sparkling region, many small ... peaks more intense, See coating of SS + bubbles are here greater 2/5 ratios

MT06 5000 6500 -6905.1
7000 8500 -6905.1

MT07 6800 7000 -6915.2
inside MT06 6800 8000 -6915.2

• Maybe not @ equilibrium yet

Klenborg

Priest

3D08 6900 8700 -6919.6 36x31x2 ~~50~~ 150 nm deep .2 sec UR 2902.36
7200 9000 -7069.6 10 nm 2912.66

MT08 6500 8500 -6916.6
7000 9500 -6916.6

MT09 7000 8000 -6916.6
8500 9500 -6916.6

3D09 X 7650 X 8050 -6916.6 2902.82 Green coating, like 3D06 White around granules too
Y 8750 Y 9000 -7066.6 2912.65

• Pressure drops over night still

7/14, D15

~~↘ (-1888, 3324, 0)~~ ↘ ~~(-1725.6, 3410.3, 0)~~ (-1889.4) 3331.9, 0)

~~(0,0,0) [B]~~ (0,0,0) [B]

~~↗ (10917.8, -5164.6, 0)~~ ↗ ~~(11021.6, -5106.4, 0)~~ (10925.8, -5186, 0)

~~MT01 4400 4800~~
~~6400 6800~~

~~3D06 6200 8100 7550 -6818.2 31x31x2 2902.94~~
~~6400 7750 -6968.2 0.2 sec UR 2912.76~~

MT06 ~~6350~~ 6500 7000 -6818.2
~~7000~~ 8500 8000 -6818.2

MT09 7500 8000 ~~-6883.6~~ -6892.2
8500 9000 ~~-6883.6~~ -6892.2

~~3D04 7450 7950~~
~~7750~~ ~~6924.2~~ ~~6974.2~~

3D02 6800 7300 ~~6924.2~~ 41x31x2
6900 7600 -6874.2

-6818.2
-6868.2

3D07 6350 7300 -6818.2 56x41x2
6900 7700 -6868.2

= 3D06 today = shifted yesterday

3D09 7900 8600 ~~-6883.6~~ -6892.2 41x21x2
8300 8800 ~~-6933.6~~ ~~-6942.2~~ -7042.2

3D10 8165 8725 -6898.8 .1 sec 21x31x5
8185 8750 -6918.8 20 nm deep 5x

3D11 8150 8740 -6898.8 51x2x8
8200 8741 -6918.8 0.1 sec

MP12 7350 8350 -6872.7 MT12 7246.9 8484 Changed color MP12 + MT12
7500 8700 -6872.7 7660.6 8735.9

MT01 4400 4800 -6872.7
6400 6800 -6872.7

MP01 " " " 9x9 Raster 25 nm 1 sec U + LR
" " "

MT02 4400 3000 "
6400 5000 "

MP02 " " "
" " "