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ISSUED TO Dr. Anne-Marie LEJEUNE
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Project: CNWRA 20.1402.461

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This notebook is a continuation of scientific notebook # 508. It documents work performed to study the interaction of a basaltic dyke with the proposed Yucca Mountain nuclear waste repository.	
Project number: 20.01402.461	
All the experiments reported in this notebook are performed by Dr. A.-M. Lejeune	
Initial entry by Dr. A.-M. Lejeune 18.12.02	
Note: All entries by Dr. A.-M. Lejeune are dated using the European format (day. month. year) 18.12.02	

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GRA cups weighing 25.03.02

Cups 56 to 72 taken out of the oven at 6H44 - $T_{B57} = 96°C$

Start weighing cups at 6H55 using the B57 OHAUS scale - $T = 24°C$

Cup	m(g)
56	12.230
56'	11.048
56''	13.116
57	7.669
58	12.722
58'	13.079
59	7.295
60	16.360
60'	17.337
61	14.381
61'	9.061
62	8.726
63	14.505
64	9.604
65	19.267
65'	5.783
66	11.00718
67	11.663
67'	12.753
68	6.771
69	8.018
69'	7.493
69''	7.440
69'''	7.155
70	5.347

4 "cm-size-bubbles"

1 2cm-bubble

3 "cm-size" bubbles

Cup	m(g)
71	6.060
71'	8.207
71''	6.183
71'''	7.033
72	5.576

Weighing done at 7H09

All cups 56 to 72 plus cup 73 in the B57 oven at 7H10

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Cups 38' to 55, cup 73 and cup GRB taken out of the oven at 7H38
$T_{B57} = 92°C$

At 7H41, T = 24°C, cup 73 = 7.586 g
Cup 73 back in the B57 oven at 7H42

At 7H54, T = 24°C, start weighing cups with the B57 OHAUS scale

Cup	m (g)	Comments
38'	16.952	One bubble half the size of the cup
38''	17.956	One bubble half the size of the cup
39	8.014	One bubble 1/4 size of the cup, surface entirely covered by ::
40	13.717	One bubble 2/3 cup size
40'	15.744	Two centimetric bubbles
41'	15.187	One bubble 1/4 cup size
42	11.631/2	Two "1-cm" bubbles
42'	13.939	Two "2-cm" bubbles
43	19.429	One "3-cm" bubble, one "2-cm" bubble
44	13.026	Two "2-cm" bubbles
44'	15.302	One "3-cm" bubble
45	9.832	Three "1-cm" bubbles
46	9.441	One "1-cm" and two "0.5-cm" bubbles
47	24.964	One "2-cm" bubble
48	12.314	Two "1-cm" and one "3-cm" bubbles
50	17.702	One "2-cm" bubble

Checked experiment EOS-GRA45 at 8H05, then carried on weighing cups.

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Cup	m (g)	Comments
50'	20.937	One "3-cm" and one "2-cm" bubbles
51	13.142	One bubble covering 2/3 of the cup
52	17.915	Four "1-cm" bubbles
52'	18.986	Two "2-cm" and one "1-cm" bubbles
52"	18.058	
53	10.350	One "2-cm" bubble
54	20.503	One "1-cm" and one "1.5-cm" bubbles
54'	24.155	One "3-cm" and one "1-cm" bubbles
54"	10.975/6	Two "1.5-cm" bubbles
55	9.901	
GRB	16.598/9	

8H12
T = 24°C

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Done at 8H25.

All cups (38' to 55 and GRB) back in the B57 oven at 8H27.

Cup 73 out of the oven at 8H28.
At 8H30, Cup 73 = 7.398g - Back in the oven at 8H33.

Cups 20 to 38 taken out the B57 oven at 8H28.
Cup 41 out of the oven at 8H35.
Cups 20 to 26' back in the oven at 8H35.

GRB trays 1 and 2 taken out of the B57 oven at 8H35.

GRB tray 1 leaks from the bottom ⇒ GR spillage on the oven bottom

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Start weighing cups 27 to 38 at 10H30 - T = 24°C

Cup	m (g)	Comments
27	6.455	
28	11.254	One big bubble 3/4 of the cup surface
29	13.389	
30	17.636	One big bubble 2/3 of the cup surface
31	23.483	One big bubble 3/4 of the cup surface
32	19.645	One big bubble 3/4 of the cup surface
33	24.581	One bubble 1/4 of the cup surface
34	24.388	Two bubbles ~ 2 cm in Ø
34'	19.820	One big bubble 2/3 of the cup surface
35	23.726/7	One big bubble 1/2 of the cup surface
35'	17.988	
36	3.935/6	
36'	12.880	One "3-cm", One "2-cm" and one "1-cm" bubbles
36"	11.945	One big bubble 1/2 of the cup surface
37	4.958	
38	17.089	
41	14.065	

All cups (27 to 38 and 41) back in the B57 oven at 10H44.

Cup 73 out of the oven at 10H43 - T_{B57} = 96°C
At 10H45, Cup 73 = 7.210 g - Back in the oven at 10H46

Cup 73 out of the oven at 12H44 - T_{B57} = 96°C
At 12H52, T = 23.5°C, Cup 73 = 7.138 g - Back in the oven at 12H53

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PGS density measurements 25.03.02

The pycnometer was left switched on over Sunday

At 9H00

T = 24°C
Humidity = 52 %
$m_{cup+PGS}$ = 120.38 g ⇒ m_{PGS} = 93.503 g
PGS still contains 8 bubbles but Ø ≤ 1 mm.

P_1 (psi)	P_2 (psi)	V (cm³)	ρ (g/cm³)	
18.970	9.866	65.485	1.428	
18.417	9.586	65.622	1.425	
18.559	9.661	65.641	1.424	
19.008	9.903	65.785	1.421	rezeroed pycno
18.434	9.599	65.696	1.423	
18.921	9.853	65.703	1.423	
18.858	9.830	65.876	1.419	
18.462	9.621	65.829	1.420	
17.683	9.211	65.754	1.422	
18.473	9.633	65.941	1.418	→ discussion with MV
17.452	9.086	65.665	1.424	
18.522	9.644	65.682	1.424	
18.957	9.871	65.690	1.423	
18.680	9.729	65.730	1.423	
18.407	9.577	65.553	1.426	
17.741	9.234	65.619	1.425	
19.218	10.010	65.744	1.422	
18.755	9.774	65.835	1.420	
18.790	9.792	65.831	1.420	
19.050	9.928	65.839	1.420	
18.740	9.766	65.832	1.420	
19.158	9.982	65.800	1.421	
18.660	9.725	65.844	1.420	

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P_1 (psi)	P_2 (psi)	V (cm³)	ρ (g/cm³)
19.039	9.921	65.817	1.421
19.082	9.942	65.793	1.421
18.768	9.781	65.839	1.420
19.165	9.987	65.823	1.421

Done at 10H20

After pycno measurements, $m_{cup+PGS} = 120.37\,g$

DGS 35 density measurements 25.03.02

At 12H05 sample prepared on 22.03.02 (see page ...)
$m_{cup} = 26.877\,g$
$m_{cup+DGS35} = 111.44\,g$
Bubbly layer at the surface from having shaken the sample in the bottle ⇒ wait to do the density measurements until no bubble is left

At 16H55, three 1-mm bubbles left ⇒ pierce them

$T = 24°C$
Humidity = 52%
$m_{cup+DGS35} = 111.38\,g$ at 16H55

$m_{cup+DGS35} = 111.27\,g$ at 17H55

↳ $m_{DGS35} = 84.393\,g$

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P_1 (psi)	P_2 (psi)	V (cm^3)	ρ (g/cm^3)
18.356	9.665	67.599	1.248
18.820	9.914	67.680	1.247
18.409	9.701	67.742	1.246
19.045	10.030	67.637	1.248
18.163	9.558	67.503	1.250
18.802	9.906	67.705	1.246
18.816	9.909	67.630	1.248
18.690	9.853	67.809	1.245
18.741	9.882	67.846	1.244
18.404	9.687	67.542	1.250
	9.688	67.559	1.249
17.766	9.358	67.666	1.247
18.472	9.730	67.668	1.247
18.867	9.941	67.718	1.246
18.624	9.820	67.840	1.244
18.852	9.932	67.699	1.247
18.958	9.991	67.753	1.246
18.889	9.949	67.657	1.247
18.785	9.896	67.687	1.247
18.793	9.903	67.735	1.246
18.476	9.733	67.684	1.247

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~~M~~ 18H40

no bubble in the DG835 sample
T = 24°C
Humidity = 52%
$m_{cup + DG835}$ = 111.20 g

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GRA sample preparation BATCH 23 25.03.02

	m (g)	Time	Comments
1000 ml PYREX flask	336.2	11H23	P_5 = 983.0/982.9 mbar T_2 = 21.5°C
+ GR	451.5	11H25	+ 115.3 g GR
+ acetone	520.3	11H26	+ 68.8 g acetone
+ GR	534.1	11H27	+ 13.8 g GR
+ magnet	571.6	11H28	⇒ stirring
34.8 wt%	575.8	12H45	P_5 = 983.0 mbar - T = 20.6°C ⇒ cup 74 and EOS-GRA46
	500.0	13H15	
	500.0	20H15	P_4 = 986.213 mbar P_5 = 988.2 mbar
On 26.03.2002	500.1	6H30	⇒ cup 76 and EOS-GRA47

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EOS-GRA46 25.03.02

GRA sample = batch 23

System used = 1000 ml KIMAX flask

At 12H50, T = 23.5°C

empty flask = 267.64 g
flask + GRA = 330.01 g
cup 74 (empty) = 0.959 g
cup 74 + GRA = 15.517 g

Flask set in bath at 12H52 and left to equilibrate in temperature.

Cup 74 put in the B57 oven at 12H53.

At 13H20

P_4 = 981.5 mbar
P_5 = 983.3/4 mbar
T_0 = 0.90 mV
T_1 = 22.5°C
T_2 = 20.5°C
T_3 = 22.4°C

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Time	P_{EOS} (mbar)	P_{atm} (mbar)	T_0 (mV)	T_1 (°C)	T_3 (°C)	Comments
3.65	900	983.3	0.90	22.5	22.4	
17.24	800	-	-	-	-	
33.15	700	-	-	-	-	
48.12	600	983.3/4	-	-	-	
1'03.90	500	-	-	-	-	
1'30.56	400.5	983.3	-	-	-	
2'08.53	300.0	983.3/4	-	-	-	Couple of bubbles + surface degassing features
3'13.12	227.0	-	-	22.4	-	
16'19.40	224.0/1	983.4/5	0.89	-	22.3/4	Adjust P_{EOS}
17'56.18	216.7	-	-	-	22.2	Bath on - T_{set} = 22.5°C
19'19.71	210.2	983.5	0.90	22.5/6	22.6	Adjust P_{EOS} Bath off
21'00.77	210.8	983.4/5	-	22.7	-	Lower P_{EOS}
23'12.33	208.1/2	-	-	-	-	
31'02.62	206.6	-	0.91	22.6	22.5	
33'31.97	206.3	-	-	-	-	
1H23'13"	203.5	-	0.89	22.3	22.1/2	
2H12'27"	202.5	983.9	0.88	22.0/1	21.9	
2H33'59"	202.6/7	984.4	-	22.0	-	
2H40'20"	202.7/8	984.4/5	0.87	-	21.8	
2H41'45"	206.0	984.4	-	21.9	19.4	Bath on
2H43'00"	203.6	984.4/5	0.88	22.2	19.6	
3H17'35"	202.2/3	984.9	0.91	22.8	22.7	

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Time	P_{EOS} (mbar)	P_{atm} (mbar)	T_0 (mV)	T_1 (°C)	T_3 (°C)	Comments
3H23'20"	202.1/0	984.8/9	0.91	22.8	22.7	
3H34'15"	202.0	984.9	-	22.9	22.8	Bath turned off Shake flask a bit ⇒ P_{EOS} increases up to 206 mbar but no bubble forms.
3H35'30"	205.2	984.9	0.91	22.9	22.8	
3H59'00"	202.2	985.1	0.90	22.7	22.7	
4H34'34"	202.8	985.4/5	0.89	22.5	22.4	
5H25'50"	202.6/7	986.3/4	0.91	22.9	22.9	Bath off
5H38'50"	203.1/0	986.7	-	-	22.8/7	
5H57'00"	203.9	987.3	-	22.8	22.7	Roll GRA sample on flask ⇒ P_{EOS} increases up to 204.6 mbar then decreases
5H58'00"	204.2/3	987.2/3	0.91	22.7	22.7/6	
6H35'45"	205.4	987.8	0.89/0.90	22.5	22.4	
6H42'10"	205.5	987.9	0.89	22.4/5	22.4	T_a = 20.8°C

⇒ experiment stopped at 20H00 after 6H42'15"

At 20H05, T = 22°C

flask after EOS-GRA46 = 322.38 g
cup 75 (empty) = 0.963 g
cup 75 + GRA = 24.079 g (82)
cup 75' (empty) = 0.960 g
cup 75' + GRA = 19.576 g

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DGS 20 density measurements 25.03.02

At 19H05

sample prepared on 22.03.02
$m_{cup} = 26.877\,g$
$m_{cup+DGS20} = 128.47\,g \Rightarrow m_{DGS20} = 101.593\,g$
Humidity = 52%
T = 24°C

P_1 (psi)	P_2 (psi)	V (cm³)	ρ (g/cm³)
18.320	10.192	76.737	1.324
18.922	10.534	76.846	1.322
18.886	10.512/1	76.816	1.323
19.050	10.603	76.812	1.323 → adjust zero
18.581	10.334	76.687	1.325 → adjust zero
18.747	10.437	76.853	1.322
18.790	10.467	76.946	1.320
18.634	10.381	76.960	1.320
17.272	9.623	76.973	1.320
19.086	10.628	76.887	1.321
18.667	10.388	76.783	1.323
18.829	10.489	76.950	1.320
18.896	10.515	76.777	1.323
18.710	10.411	76.769	1.323
	10.412	76.784	1.323
19.156	10.680	77.084	1.318

At 19H45, after pycno measurements $m_{cup+DGS20} = 128.45\,g$

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Cups 73 and 74 taken out of the oven at 20H02.

At 20H10, T = 22°C | Cup 73 = 7.006 g
Cup 74 = 11.080 g

Cups 73 to 75' put in the B57 oven at 20H11 - T_{B57} = 96°C

At 20H15

P_4 = 986.3/4 mbar
P_5 = 988.2 mbar
T_0 = 0.89 mV
T_1 = 22.5°C
T_2 = 20.9°C
T_3 = 22.4°C

T_{bath} = 22.0°C
T_{set} = 22.5°C

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Prepare DGS5 for density measurements tomorrow.

At 20H20

m_{cup} = 26.877 g
$m_{cup+DGS5}$ = 106.17 g ⟹ left in peace overnight to let the bubbles go out

Go to Academy.

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Tuesday 26 March 2002 At 6H00 41F

At 6H30
P_4 = 992.112 mbar
P_5 = 994.0/1 mbar
T_0 = 0.90 mV
T_1 = 22.7°C
T_2 = 18.0°C
T_3 = 22.6°C

At 6H48, cups 73 to 75' taken out of the oven - T_{B57} = 96°C

At 6H56, T = 20°C

Cup	m (g)
73	6.919
74	10.860/1
75	19.458
75'	15.691

All cups (73 to 75') back in the oven at 6H58.

Cup 76 (see next page) put in the B57 oven at 6H55 and taken out at 7H27 - T_{B57} = 96°C

At 7H34, T = 20°C, Cup 76 = 13.671 g
Cup 76 back into the oven at 7H35.

Cup 76 taken out of the B57 oven at 10H00 - T_{B57} = 96°C
At 10H20, T = 20.5°C, Cup 76 = 13.000 g
Cup 76 back into the oven at 10H22.

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EoS-GRA47

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GRA sample = batch 23
System used = 1000 ml KIMAX flask

At 6H50, T = 20°C

cup 76 (empty) = 0.955 g
cup 76 + GRA = 17.683 g
empty flask = 255.06 g
flask + GRA = 347.98 g

Flask set in bath at 6H53 and cup 76 put in the B57 oven at 6H55.

At 7H00

P_4 = 992.4/5 mbar
P_5 = 994.4/5 mbar
T_0 = 0.91 mV
T_1 = 22.7°C
T_2 = 18.1°C (room temperature)
T_3 = 22.6/7°C

Time	P_{EoS} (mbar)	P_{atm} (mbar)	T_0 (mV)	T_1 (°C)	T_3 (°C)	Comments
4.69	900	994.5	0.91	22.7	22.6	
32.51	800	-	-	-	-	
49.10	700	994.4/5	-	-	-	
1'08'69	597	994.5	-	-	-	
1'32'57	500	-	-	-	-	
1'55.17	400	994.5/6	0.90	-	-	
2'31.45	300.2	-	-	-	-	
3'10.35	200.0	994.6	-	-	-	

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Time	P_{EOS} (mbar)	P_{atm} (mbar)	T_0 (mV)	T_1 (°C)	T_3 (°C)	Comments
3'29.48	190.0	994.6	0.90	22.7	22.6	
3'48.79	183.0	-	-	-	-	
4'18.35	174.0	994.6/7	-	-	-	
4'40.76	169.0	-	-	-	-	
5'26.26	162.0	-	-	-	-	
6'07.86	158.0	-	-	-	-	
6'41.01	155.4	-	-	-	-	Adjust P_{EOS}
7'06.60	157.4	-	-	-	-	
15'24.57	152.2	994.7/8	-	-	-	
19'43.51	151.9	994.6/7	-	22.8/7	-	

At 20'08", tilt the flask ⇒ bubbling and P_{EOS} increases up to 184.7 mbar then starts decreasing at 20'38.51 when the bubbling stops

20'50.45	180.0	994.6/7	0.89	22.8/7	22.6	
21'35.01	171.0	-	-	22.7	-	
22'06.07	168.0	994.7/8	-	-	-	Only surface features ≈ No visible ∴
22'59.35	165.0	-	-	-	-	
33'40.29	151.3	994.7/6	-	22.7/8	-	Adjust P_{EOS} a tad
34'24.63	154.0	994.6/7	-	-	-	
36'57.86	153.8	-	-	22.8	-	
48'16"	151.7	995.1	-	-	-	

↳ tilt flask ⇒ bubbling but not much. P_{EOS} increases up to 163.4 mbar then starts decreasing at 49'16"

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Time	P_{EOS} (mbar)	P_{atm} (mbar)	T_0 (mV)	T_1 (°C)	T_3 (°C)	Comments
49'43"	162.1	995.1/2	0.89	22.8	22.6	
54'16"	157.2	995.2/3	-	-	-	
55'45"	156.0	995.3/4	-	-	22.6/7	
1H03'56"	151.3/2	995.5	-	-	22.7	
1H11'41"	151.0	995.4/5	-	-	-	
1H14'30"	150.9/151.0	-	0.90	-	-	Surface degassing wrinkles
1H29'15"	150.4	995.2/3	0.90	22.8	22.7	Adjust P_{EOS}
1H35'10"	153.3/4	995.4/5	-	-	-	
2H27'45"	151.5	995.6/7	-	22.9	22.7/8	Bath off
↳ tilt flask ⇒ P_{EOS} increases up to 158 mbar. A few bubbles form but not many. P_{EOS} starts decreasing at 2H28'43"						
2H29'13"	156.1	995.5/6	0.90	22.8/9	22.7/8	
2H29'41"	154.6	995.6	-	-	-	
2H30'17"	153.5	-	-	22.8	-	
2H44'40"	151.5/6	995.7	0.89	22.6	22.5	Bath on - T_{set} = 22°C
2H51'45"	151.3	-	-	-	-	Surface smoother: less but longer wrinkles instead of
2H58'15"	151.0	995.7	0.89	22.6	22.5	
3H07'05"	150.6/7	995.6	-	22.5	-	
3H19'20"	150.0/1	995.5/6	-	-	-	

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Time	P_{EOS} (mbar)	P_{atm} (mbar)	T_0 (mV)	T_1 (°C)	T_3 (°C)	Comments
3H49'30"	149.4/5	995.1/2	0.89	22.5	22.4/5	Adjust P_{EOS}
4H24'15"	154.0/1	995.6/5	0.88	-	22.5	T_2 = 20.4°C
↳ Tilt flask ⇒ P_{EOS} increases and reaches up to 159.0/1 mbar at 4H25'08". Very few bubbles.						
4H25'35"	157.7	995.6	0.88/0.89	22.5/6	22.5	
4H26'35"	155.6	-	0.88	22.5	-	
4H47'10"	152.3/4	995.3	-	22.6	22.6	
4H55'00"	152.1/0	995.3/2	-	22.7	-	T_2 = 21.0°C T_{bath} = 22.7°C T_{set} = 22.0°C
5H01'35"	151.9	995.2/3	0.88	22.7	22.6	
5H05'15"	151.7/8	-	-	-	-	
6H29'10"	151.3/4	994.0	0.91	23.2	23.1	Bath off
6H54'00"	151.0	993.3/4	0.89	22.9	22.8	T_2 = 20.8°C
7H29'25"	151.5/6	993.1	0.88/0.87	22.6	22.5	
↳ Tilt flask ⇒ a few bubbles (4 or 5 only). P_{EOS} increases up to 156.3 mbar then decreases from 7H30'41"						
7H32'33"	152.7/8	993.0/1	0.87	22.5	22.4/5	Bath on T_{set} = 21.8°C T_{bath} = 22.7°C
7H41'20"	151.1/2	992.9	0.88	22.6	22.5	
8H58'15"	149.7/8	992.3	0.90	23.1	23.0	Bath off
9H55'00"	150.2	-	0.87	22.5	22.4/5	T_2 = 21.3°C
↳ stop experiment after 9H55'30" at 17H00						

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At 17H02, T = 24°C

flask after EOS-GRA47 = 331.31 g

cup 77 (empty) = 0.962 g
cup 77 + GRA = 26.229 g

cup 77' (empty) = 0.963 g
cup 77' + GRA = 14.070 g

Both cups 77 and 77' put in B57 oven at 17H05 - T_{B57} = 95°C

Cup 76 taken out of the oven at 17H05.
At 17H07, Cup 76 = 12.655 g - Cup back in the oven at 17H09.

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DGS5 density measurements 26.03.02

At 8H35 | $m_{cup} = 26.877 g$
$m_{cup+DGS5} = 106.17 g \Rightarrow m_{DGS5} = 79.293 g$
Two 1-mm in diameter bubbles

⇒ load sample in the pycnometer and start measurements at 8H45

P_1 (psi)	P_2 (psi)	V (cm³)	ρ (g/cm³)	
18.638	9.218	56.579	1.401	
18.650	9.229	56.678	1.399	
19.488	9.652	56.835	1.395	
18.810	9.326	57.025	1.390	
18.975 / 18.976	9.411	57.087 / 57.077	1.389 / 1.389	
18.560	9.194	56.867	1.394	
18.022	8.928	56.877	1.394	
18.610	9.220	56.891	1.394	
19.329	9.578	56.925	1.393	
17.885	8.854	56.751	1.397	Adjust zero
18.348	9.092	56.927	1.393	
19.157	9.495	57.024	1.391	
17.905	8.873	56.938	1.393	
18.541	9.192	57.013	1.391	
18.533	9.173	56.716	1.398	
19.025	9.416	56.706	1.398	
19.009	9.418/9.419	56.897/56.916	1.394/1.393	
18.499	9.167	56.930	1.393	

At 9H30, after the pycno measurements, $m_{cup+DGS5} = 106.15 g$

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DGS 25 density measurements 26.03.02

At 9H55 | $m_{cup} = 26.877\,g$
$m_{cup+DGS25} = 126.80\,g \Rightarrow m_{DGS25} = 99.923\,g$

Start density measurements at 10H54.

P_1 (psi)	P_2 (psi)	V (cm³)	ρ (g/cm³)
19.481	10.854	76.977	1.298
18.655	10.401	77.089	1.296
18.592	10.369	77.137	1.295
19.029	10.619	77.233	1.294
18.886	10.535	77.168	1.295
17.908	9.990	77.177	1.295
18.197	10.142	77.031	1.297
18.590	10.373	77.217	1.294
18.739	10.453	77.168	1.295
17.885	9.970	77.061	1.297
17.408	9.704	77.060	1.297
19.264	10.751	77.245	1.294
18.814	10.492	77.125	1.296
18.886	10.536	77.184	1.295
18.371	10.241	77.063	1.297

At 11H25, after the pycno measurements, $m_{cup+DGS25} = 126.77\,g$

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DGS10 density measurements 26.03.02

At 11H55 | sample prepared on 22.03.02
$m_{cup} = 26.877\,g$
$m_{cup+DGS10} = 153.88\,g$

Let sample resting for a while to allow the bubbles to escape.
Pierce the remaining bubbles at 13H50.

At 13H55 | $T = 24°C$
Humidity = 51%
$m_{cup+DGS10} = 153.86\,g \Rightarrow m_{DGS10} = 126.983\,g$

P_1 (psi)	P_2 (psi)	V (cm³)	ρ (g/cm³)	
18.790	11.572	92.344	1.375	
19.500	12.016	92.425	1.374	re-zeroed
18.185	11.206	92.429	1.374	
18.861	11.621	92.410	1.374	
18.093	11.155	92.504	1.373	
19.088	11.737	92.573	1.372	re-zeroed
18.434	11.358	92.411	1.374	
18.887	11.652	92.597	1.371	
18.795	11.586	92.481	1.373	
17.677	10.890	92.390	1.374	
18.758	11.565	92.504	1.373	re-zeroed
18.840	11.610	92.434	1.374	
17.694	10.908	92.490	1.373	re-zeroed
18.242	11.239	92.402	1.374	
18.634	11.486	92.471	1.373	

At 14H25, after the pycno measurements, $m_{cup+DGS10} = 153.85\,g$

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DGS15 density measurements 26.03.02

At 14H45 | sample prepared on 22.03.02
$m_{cup} = 26.877\,g$
$m_{cup+DGS15} = 148.13\,g$

At 19H25, $m_{cup+DGS15} = 148.11\,g \Rightarrow m_{DGS15} = 121.233\,g$

Measurements started at 19H55.

P_1 (psi)	P_2 (psi)	V (cm^3)	ρ (g/cm^3)
19.048	11.538	89.905	1.348
19.577	11.866	90.000	1.347
19.380	11.744	89.967	1.348
18.582	11.259	89.948	1.348
18.810	11.399	89.972	1.347
18.620	11.285	89.987	1.347
18.674	11.317	89.978	1.347
19.330	11.714	89.971	1.347
19.329		89.978	1.347
18.954	11.492	90.046	1.346
18.494	11.210	90.005	1.347

At 20H20, after pycno measurements, $m_{cup+DGS15} = 148.09\,g$

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VACUUM GAUGES

26.03.02

At 21H05, put the ASG used for measuring P_1 (tonnel), P_4 (EOS cell) and P_5 (atmospheric) on the same line to compare their readings

P_1 (mbar) AN 007630 SN 995000176	P_4 (mbar) AN 008236 SN 005000055	P_5 (mbar) AN 008466 SN 0050000371
992.5/4	994.1/2	993.5/6
841.0/1	842.7/8	842.2/3
753.2/1	754.8/9	754.4/5
661.2/3	662.9/663.0	662.6/7
574.3/4	575.8/9	575.6/7
435.2/3	436.5/6	436.6/5
349.2	350.4/5	350.5/6
231.6/5	232.6/7	232.8/9
135.4	136.4	136.8/7
17.4	18.3	18.7
28.3/4	29.2/3	29.6/7
62.0/61.9	63.0/62.9	63.3/4
95.0	96.0	96.4
160.6	161.7	162.0
208.8	209.9	210.2
270.7/6	271.7/8	271.9
372.8	374.1	374.1
459.5	460.8	460.8
502.5	503.9	503.8
549.8	551.2	551.0
608.4	609.9	609.6
692.9	694.5	694.1
719.0	720.6	720.2

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P_1 (mbar) AN 007630 SN 995000176	P_4 (mbar) AN 008236 SN 005000055	P_5 (mbar) AN 008466 SN 005000371
799.2	800.9	800.4
862.2	863.9	863.4
902.5/4	904.0/1	903.4/5
929.4/5	931.1/2	930.5/6
965.6/7	967.5/4	966.7/8
992.5	994.2/3	993.5/6

Done at 21H20

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GRA cups weighing 26.03.02

All cups taken out of the oven at 19H10. Weighing started at 21H40
T = 22°C

Cup	m(g)		Cup	m(g)		
1	8.826/7	Tray 1	19	13.544		
2	19.995		19'	11.725	Tray 1	21H54
3	17.357		20	7.330/1	Tray 2	T=22°C
4	17.821/2		20'	16.159		
4'	19.732		21	15.664		
5	12.789		21'	15.691		
6	17.955		22	15.276		
7	8.386		22'	16.994		
8	24.867		23	9.401		
9	7.337		24	16.865		
10	20.754/3		24'	15.900		
11	18.291		25	8.643		
12	5.349/5.350		26	11.237/8		
12'	15.034		26'	11.703/4		
13	17.949/50		27	6.449		
13'	18.955		28	11.234		
14	13.390		29	13.372		
14'	15.242		30	17.612		
15	8.727/8		31	23.459/8		
16	13.938		32	19.625		
16'	7.895		33	24.561		
17	13.824		34	24.362		
17'	18.181		34'	19.799		
18	18.823		35	23.703		
18'	16.292		35'	17.969		

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Cup	m2(g)			Cup	m2(g)	
36	3.930			56	12.223	Tray 4
36'	12.865			56'	11.039/8	
36''	11.929			56''	13.104	
37	4.950			57	7.659	
38	17.071	Tray 2	22H05	58	12.703	
38'	16.934	Tray 3	T= 22°C	58'	13.059/60	
38''	17.937/8			59	7.282	
39	8.003/4			60	16.340	
40	13.697			60'	17.316/7	
40'	15.728			61	14.359/60	
41	14.047/8			61'	9.051	
41'	15.169			62	8.714	
42	11.626			63	14.466	
42'	13.929			64	9.575	
43	19.413/2			65	19.183/4	
44	13.017			65'	5.766	
44'	15.292			66	10.959	
45	9.825/6			67	11.607	
46	9.432			67'	12.682	
47	24.937			68	6.746	
48	12.303			69	7.982	
50	17.684			69'	7.466/5	
50'	20.910/1			69''	7.414	
51	13.122/3			69'''	7.129	
52	17.892			70	5.330	
52'	18.962/3			71	6.035	→ restart scale
52''	18.039/8			71'	8.162/3	
53	10.337			71''	6.151	
54	20.478			71'''	6.999	
54'	24.124			72	5.543	
54''	10.961			73	6.886	
55	9.889			74	10.790	
GRB	16.593	Tray 3	22H20 T= 22°C			

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Cup	m(g)
75	19.160 } still soft (mass finger in)
75'	15.477
76	12.611
77	23.685 (a tad of GRA on the side of the cup)
77'	12.557

All cups weighed by 22H35. $T_{room} = 22°C$

All cups back in the B57 oven at 22H38 - $T_{B57} = 100°C$

At 22H40, { GRB tray 2 = 3915.9 g
GRB tray 1 + alu foil = 3423.3 g

had a leak

Both trays stored in the volcanology group cabinet in building 51.

Recycling GRA taken out of the B51 oven at 22H54.

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GRA cups weighing 27.03.02

Cups weighed by Melissa Hill with scale s/n 2883 - T = 23.5°C
No time given for when cups were taken out of the oven and put back in.

Cup	m (g)
73	6.869
74	10.744
75	19.009
75'	15.368
76	12.409
77	22.509
77'	12.047

Asked Melissa to leave the cups in the oven for degassing, check that the oven temperature never exceed 110°C and to weigh all the cups every 2 or 3 weeks until I return to SeoRI to carry on the experimental programm.

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From Page No. GRA cups weighing by MH and BH

Date	s/n	Date	s/n	Date	s/n	Date	s/n
4-Apr-02	2883	18-Apr-02	2883	9-May-02	N45601	11-Jul-02	N45601
T (°C)	23.2	T (°C)	23.3	T (°C)	23.0	T (°C)	23.0
Cup #	Weight (g)	Cup #	Weight (g)	Cup #	Weight (g)	Cup #	Weight (g)
1	8.823	1	8.821	1	8.814	1	8.814
2	19.978	2	19.969	2	19.957	2	19.942
3	17.344	3	17.338	3	17.328	3	17.315
4	17.811	4	17.794	4	17.779	4	spill
5	12.775	5	12.768	5	12.761	5	12.747
6	17.935	6	17.920	6	17.888	6	17.870
7	8.377	7	8.377	7	8.372	7	8.367
8	24.840	8	24.820	8	24.801	8	24.770
9	7.333	9	7.327	9	7.328	9	7.339
10	20.740	10	20.722	10	20.708	10	spill
11	18.279	11	18.254	11	18.246	11	spill
12	5.348	12	5.348	12	5.348	12	spill
13	17.929	13	17.918	13	17.900	13	17.795
14	13.373/4	14	13.366	14	13.339	14	13.330
15	8.719	15	8.719	15	8.719	15	8.713
16	13.924	16	13.913	16	13.905	16	13.883
17	13.813	17	13.804	17	13.789	17	13.781
18	18.808	18	18.792	18	18.785	18	spill
19	13.533	19	13.526	19	13.521	19	spill
20	7.326	20	7.326	20	7.324	20	7.316
21	15.659	21	15.642	21	15.626	21	15.608
22	15.268	22	15.252	22	15.235	22	15.220
23	9.399	23	9.398	23	9.388	23	9.383
24	16.848	24	16.833	24	16.813	24	16.795
25	8.641	25	8.638	25	8.626	25	8.620
26	11.226	26	11.225	26	11.219	26	11.208
12'	15.025	12'	15.008	12'	14.988	12'	14.974
13'	18.944	13'	18.928	13'	18.909	13'	spill
14'	15.223	14'	15.211	14'	15.190	14'	15.176
16'	7.887	16'	7.887	16'	7.885	16'	7.886
17'	18.149	17'	18.129	17'	18.127	17'	18.104
18'	16.274	18'	16.261	18'	16.252	18'	16.236
19'	11.713	19'	11.713	19'	11.701	19'	spill
20'	16.146	20'	16.132	20'	16.116	20'	16.105
21'	15.678	21'	15.658	21'	15.641	21'	15.631
22'	16.984	22'	16.955	22'	16.937	22'	16.917
24'	15.889	24'	15.875	24'	15.860	24'	15.843
26'	11.690	26'	11.685	26'	11.674	26'	11.660
4'	19.722	4'	19.713	4'	19.695	4'	spill
27	6.433	27	6.433	27	6.428	27	6.418
28	11.201	28	11.152	28	11.098	28	11.071
29	13.336	29	13.286	29	13.240	29	13.213

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4-Apr-02	2883	18-Apr-02	2883	9-May-02	N45601	11-Jul-02	N45601
T (°C)	23.2	T (°C)	23.3	T (°C)	23.0	T (°C)	23.0
Cup #	Weight (g)	Cup #	Weight (g)	Cup #	Weight (g)	Cup #	Weight (g)
30	17.551	30	17.477	30	17.430	30	17.395
31	23.368	31	23.254	31	23.175	31	23.130
32	19.522	32	19.449	32	19.410	32	19.369
33	24.456	33	24.347	33	24.272	33	24.220
34	24.281	34	24.146	34	24.082	34	24.023
35	23.603	35	23.485	35	23.422	35	spill
36	3.925	36	3.924	36	3.917	36	3.913
37	4.943	37	4.943	37	4.928	37	4.927
38	17.014	38	16.943	38	16.905	38	16.875
39	7.945	39	7.927	39	7.914	39	7.884
40	13.609	40	13.568	40	13.518	40	13.458
41	13.975	41	13.926	41	13.886	41	13.858
42	11.586	42	11.567	42	11.555	42	11.533
43	19.328	43	19.290	43	19.257	43	19.221
44	12.968	44	12.942	44	12.923	44	spill
45	9.782	45	9.770	45	9.751	45	9.731
46	9.392	46	9.372	46	9.359	46	9.337
47	24.775	47	24.705	47	24.632	47	24.556
48	12.248	48	12.216	48	12.198	48	12.172
50	17.582	50	17.524	50	17.495	50	17.451
51	13.051	51	13.018	51	12.997	51	12.974
52	17.786	52	17.730	52	17.697	52	17.655
53	10.286	53	10.256	53	10.242	53	10.219
54	20.351	54	20.288	54	20.241	54	20.185
55	9.853	55	9.832	55	9.819	55	9.803
56	12.156	56	12.135	56	12.115	56	12.095
57	7.615	57	7.602	57	7.599	57	7.586
58	12.613	58	12.581	58	12.558	58	12.523
59	7.242	59	7.236	59	7.227	59	7.218
60	16.214	60	16.163	60	16.137	60	16.105
61	14.224	61	14.186	61	14.164	61	14.136
62	8.642	62	8.630	62	8.617	62	8.594
63	14.298	63	14.252	63	14.222	63	14.186
64	9.459	64	9.442	64	9.422	64	9.404
65	18.885	65	18.823	65	18.773	65	18.716
66	10.805	66	10.775	66	10.752	66	10.726
67	11.430	67	11.396/7	67	11.373	67	11.347
68	6.676	68	6.662	68	6.658	68	6.636
69	7.889	69	7.879	69	7.866	69	7.850

03.04.03

To Page No.

Witnessed & Understood by me, | Date | Invented by | Date

Recorded by | 03.04.03

Project No. 20_1402.461
Book No. 555 TITLE

From Page No.

Date	s/n	Date	s/n	Date	s/n	Date	s/n
4-Apr-02	2883	18-Apr-02	2883	9-May-02	N45601	11-Jul-02	N45601
T (°C)	23.2	T (°C)	23.3	T (°C)	23.0	T (°C)	23.0
Cup #	Weight (g)	Cup #	Weight (g)	Cup #	Weight (g)	Cup #	Weight (g)
70	5.284	70	5.281	70	5.274	70	5.267
71	5.978	71	5.973	71	5.970	71	5.958
72	5.481	72	5.472	72	5.471	72	5.464
73	6.722	73	6.707	73	6.698	73	spill
74	10.502	74	10.454	74	10.431	74	10.407
75	18.402	75	18.306	75	18.251	75	18.189
76	11.973	76	11.929	76	11.911	76	spill
77	21.338	77	21.228	77	21.179	77	21.102
34'	19.732	34'	19.627	34'	19.585	34'	19.545
35'	17.906	35'	17.846	35'	17.793	35'	17.756
36'	12.828	36'	12.781	36'	12.749	36'	12.728
36"	11.897	36"	11.860	36"	11.826	36"	11.806
38'	16.842	38'	16.781	38'	16.745	38'	16.695
38"	17.832	38"	17.747	38"	17.682	38"	17.625
40'	15.626	40'	15.573	40'	15.521	40'	15.431
41'	15.068	41'	15.005	41'	14.950	41'	14.885
42'	13.887	42'	13.863	42'	13.847	42'	13.826
44'	15.236	44'	15.200	44'	15.174	44'	15.150
50'	20.779	50'	20.715	50'	20.677	50'	20.622
52'	18.849	52'	18.802	52'	18.764	52'	18.712
52"	17.940	52"	17.884	52"	17.843	52"	17.800
54'	23.962	54'	23.884	54'	23.833	54'	23.766
54"	10.912	54"	10.883	54"	10.866	54"	10.841
56'	10.984	56'	10.964	56'	10.945	56'	10.928
56"	13.028	56"	12.994	56"	12.974	56"	12.950
58'	12.964	58'	12.936	58'	12.914	58'	12.889
60'	17.170	60'	17.118	60'	17.085	60'	16.964
61'	8.983	61'	8.962	61'	8.953	61'	spill
65'	5.712	65'	5.707	65'	5.696	65'	5.689
67'	12.482	67'	12.433	67'	12.403	67'	12.381
69'	7.382	69'	7.370	69'	7.360	69'	spill
69"	7.332	69"	7.319	69"	7.310	69"	7.292
69"'	7.053	69"'	7.042	69"'	7.034	69"'	spill
71'	8.066	71'	8.053	71'	8.046	71'	8.031
71"	6.088	71"	6.086	71"	6.073	71"	6.069
71"'	6.930	71"'	6.918	71"'	6.908	71"'	6.894
75'	14.888	75'	14.829	75'	14.792	75'	14.756
77'	11.490	77'	11.444	77'	11.411	77'	11.389
GRBristol	16.579	GRBristol	16.567	GRBristol	16.560	GRBristol	16.544

03.04.03

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	03.04.03

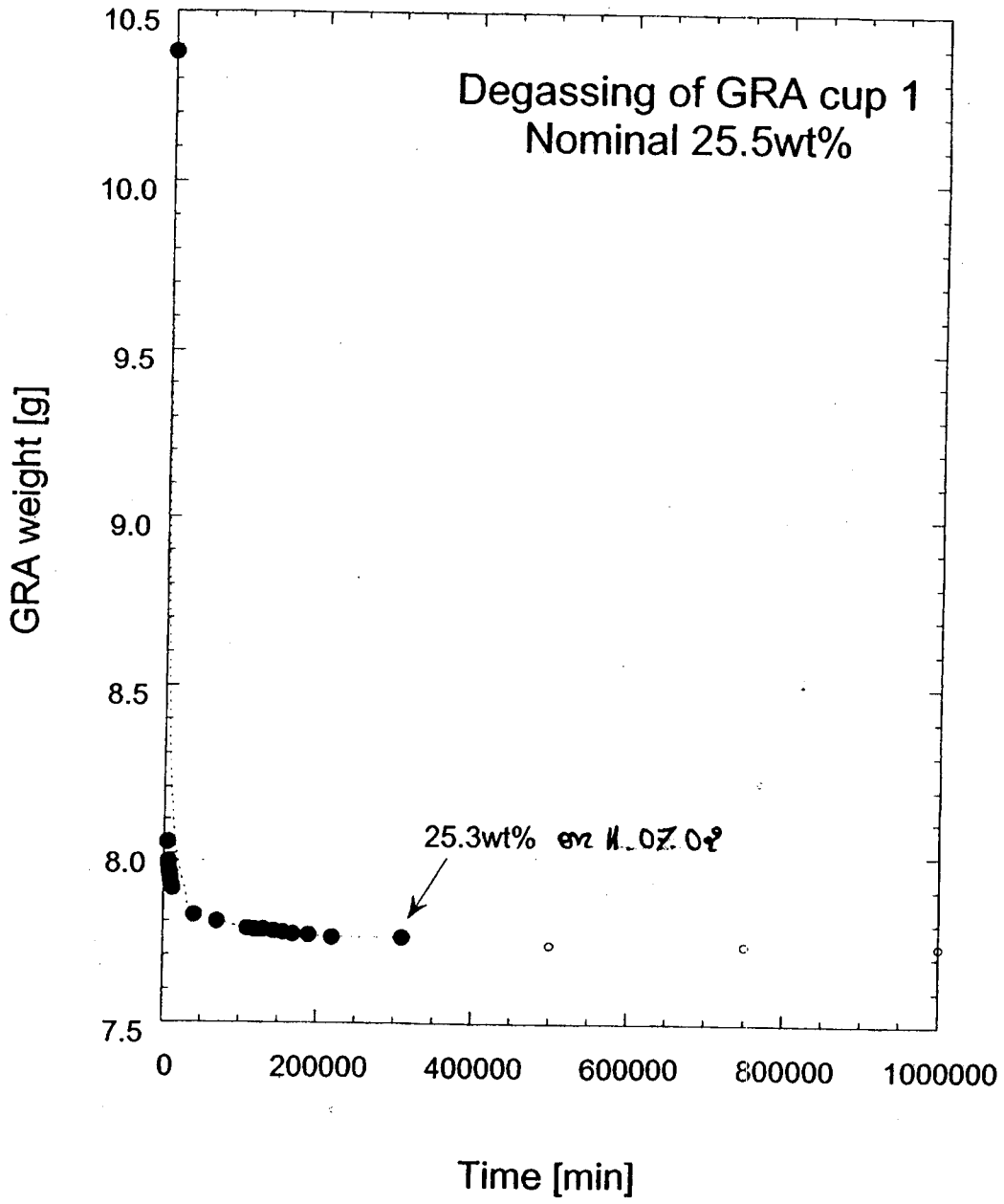
TITLE

Project No. 20.1402.461

Book No. 555

From Page No.

Example of degassing over time (up to 11 July 2002)



03.04.03

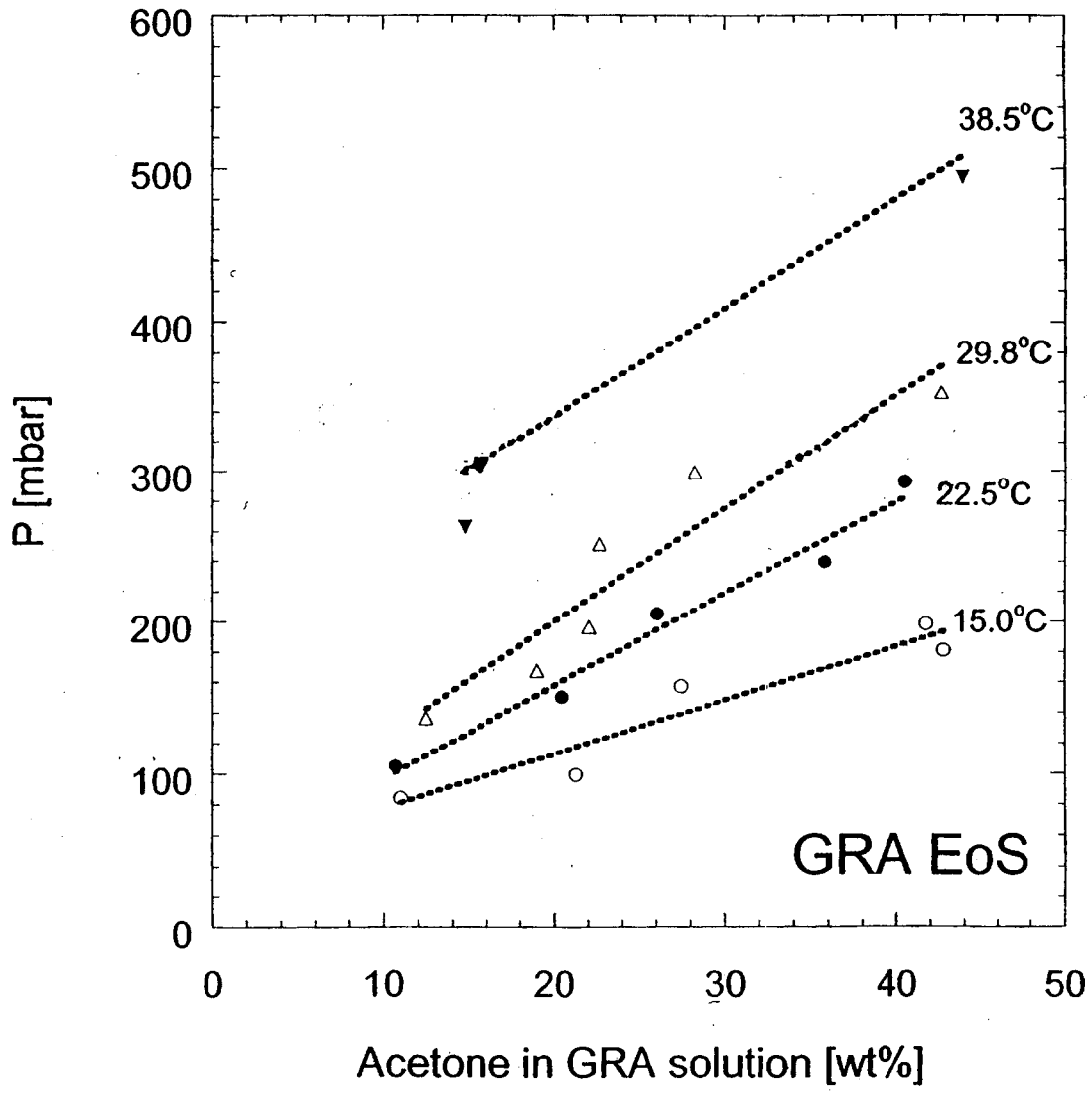
To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	03.04.03

Project No. 90.1402.461
Book No. 555 TITLE

From Page No.

Very early GRA EoS summary (12 July 2002)

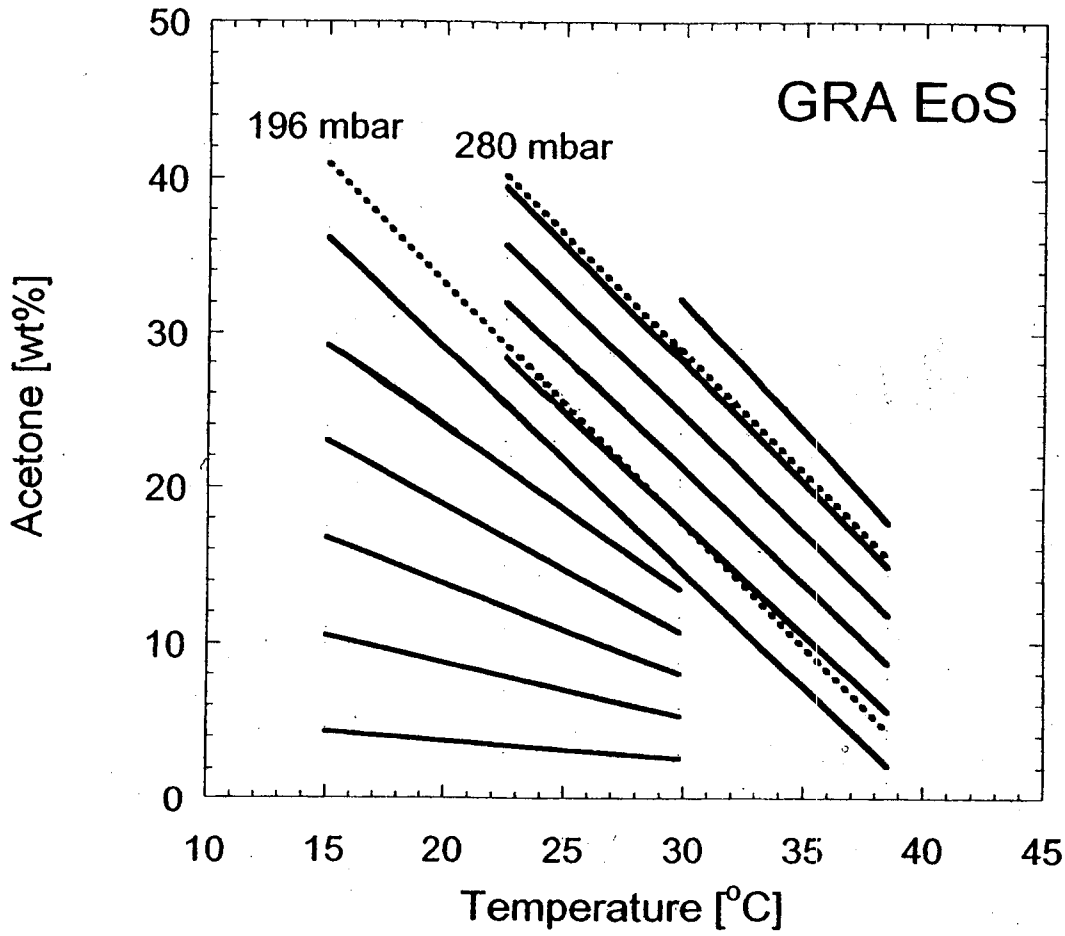


03.04.03

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	03.04.03

From Page No.



03.04.03

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by	03.04.03

Project No. 80.140-2401
Book No. 555 TITLE

GRA equation of states (23 July 2002)

Warning: This file contains temporary raw data. The samples are still currently degassing. The values below giving the acetone contents are not final and will have to be readjusted in a few months time. AML 23 July 2002

T=15.0°C

Run	P[mbar]	Acetone[wt%]
38	198.6	41.73
39	99.2	21.27
40	157.7	27.45
41	181.5	42.74
44	84.4	10.98

T=22.5°C

Run	P[mbar]	Acetone[wt%] Flask	cup
17	402.1		38.29
18	239.5		35.77
19	302.3		38.22
20	500.2		34.73
21	581.5		46.44
27	293.0		40.48
36	346.4	40.91	40.45
37	450.1	37.59	37.10
45	105.0	10.69	
46	205.5	26.05	25.48
47	150.2	20.44	20.29

T=29.8°C

Run	P[mbar]	Acetone[wt%]
28	196.3	22.06
29	497.9	41.92
30	136.5	12.45
31	167.7	19.00
32	299.4	28.25
33	401.3	29.44
34	251.4	22.65
35	352.9	42.64

T=38.5°C

Run	P[mbar]	Acetone[wt%]
11	694.6	34.51
12	497.0	33.56
13	303.3	15.73
14	594.2	31.00
15	407.7	22.38
16	262.8	14.73
25	496.1	36.97
26	494.6	43.88

03.04.03

Data given to Omma Bokhave for using in Rich numerical modelling

To Page No.

Witnessed & Understood by me, | Date | Invented by | Recorded by | Date 03.04.03

37
Rae
4/10/2003

I have reviewed scientific notebook 555 and find it in compliance with QAP-001. There is sufficient information regarding procedure used for conducting the research and acquiring and analyzing the data so that another qualified scientist could repeat the activity or activities recorded in this scientific notebook

H. L. McKague 04/10/03

H. Lawrence McKague
GLGP Element Manager

H L McKAGUE
04/10/03

UNIVERSITE PIERRE & MARIE CURIE
LA SCIENCE A PARIS

University of BRISTOL

TEMPORARY VERSION

Scientific Notebook n° 555

Volume II
AMLJ 16/2/04

Dr. Anne-Marie Lejeune

Project n° 20.01402.461

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CNWRA / SwRI

All the experiments and data reported in this notebook are performed and recorded by Dr. A.-M. Lejeune.

All entries are dated using the standard European format (day. month. year)

Travel to Bristol: Paris → Le Havre on 20.12.03
then Le Havre → Portsmouth → Bristol on 21.12.03

Sunday 21.12.03 — unload eqpt in the lab

Monday 22.12.03 — sort out eqpt and lab
meeting w/ RSJS

Tuesday 23.12.03

Order new gasmask and stopwatch from RS
(old gasmask doesnot allow breathing anymore!
and stopwatch has gone for a walk!!!)
Got consumables from Faculty Store (acetone,
paper towel, ...)

Wednesday 24.12.03

Meeting w/ Trish Cobb about administration and
finances of the Texas (CNWRA) contract/grant.

Set up the lab and experiment in the GFD lab's
cold room ⇒ run EOS expts at T between
5 and 10°C.

27.12.03

27.12.03

19H30

12.2 mbar
8.7 °C
981.5 mbar
2 flasks

(8°C)

GRA batch

570.9 g flask with

604.9 g + magnet (magnet = 34.7 g)

+ GR 724.8 g

+ acetone 856.6 g

+ GR 877.0 g

+ bouchon 1154.3 g at 981.2 mb

19H40

21H15 weight when all dissolved = 876.5 g

27.12.03

28.12.03

① not stirring: sample looks allright (clear, all gas dissolved) no

11H00 875.6 g (some above condensed on stopper) → stirring at level 5

room 5°C
(5.3°C on thermometer)
↳ once in room T ↗ 5.7-5.8°C @ 11H15

prepare EOS expt

① check all ASG and pick up 3 for the expts.

11H20

Edwards Serial No	Sw/l Cal Lab Am (Cal on Aug 19, 2003)	by PWC Patm	
use → 9.12.03 005000371	008466	991.3	/4
005000055	008236	~~998.3~~	/4
005000176	007630	997.3	/4 cable damaged → line 3
	↳ due on 19/02/04		
005000333	008464	995.617	→ line 2
005000334	008463	995.617	→ line 1

11H35 Troom 6.6°C

28.12.03

check balance room → OK

11H50 Modify set up : 2P, same sample

12H00 load sample in flasks

Flask 1 323.4 g
↳ 401.6
↳ 429.6

Flask 2 319.9 g
422.6

Sample preparation flask
875.2 g
+ stopper
1153

let set at T in room (7.3 °C)
7 °C on door

P_1	995.617
P_2	996.0 /
P_3	997.3

12H40

while opening balance room door spill some from 2

17.4470 g

clean 2 → 32.3752 g

28.12.03

(2) 28.12.03

T = 26° C

cup B1 — Empty cup = 9.5 g 61 g
↳ cup + GRA = 20.0820

cup B2 — 9.526 6 g ↳ 36.5007 g ↓ fast

12/10

Let evaporate on to p of oven
before putting them inside @ → 920 C

<u>Cups nomenclature:</u>

From now consider all the cups in Texas as T cups (T1, T2, ... to T77')

Label Bristol cups as B cups (B1, B2, etc...) and cups from expts done in Paris as P cups (P1, P2, ...)

28.12.03

12H20

Time	P_1	P_2	P_3	T
0	995.6/5	995.8/7	997.1/2	6.8
0	–	–	–	
13.07	886			
34.35	698	415		7.0
1'00.26	500			
1'18.54	400			no variable
1'40.01	350			
1'53.04	334			
2'11		↓		
2'20.57	318	900		
2'38.29	290	800		
2'52.35	260	672	–	7.2
3'19.95	263	600	997.2	7.2/7.1
3'48.57	261.8	500	–	7.1
4'08.83	261.3	441	997.2/3	–
4'30.76	260.8	359	997.2	–

28.12.03

(3)

4'58.29	260.5	280	–	–	
5'10.89	260.6	235.2	997.3	–	
5'36.32		235.1			tiny bubbles but very few (< 1mm coalescing
6'15.07	260.4	210	–	–	
6'34.07	260.6	193	997.3/4	–	
6'51.48	260.3/4	180.3	997.2	–	
7'12.79	260.4/3	158.0	–	–	
7'38.51	210	157.7	997.2/3	–	↓ P1
7'52.19	170.2	157.6	997.2/1	–	7.0/1 rotating
8'39.29	161.9/8	157.8	997.3	7.2	↙ P1
9'08.04	140.1	157.8	997.3/4	7.2/3	P2 bubbles
9'58.17	109	157.8	997.3	7.3	P1 start
10'22.04	102.8	–	–	–	occasional tiny bubbles bursting @ surface in P1

28.12.03

11' 14.63 105 158./157.9 997.2/3

when shaking F1
rapid bubbling
of a few bars
(< 1 mm ⌀)
stop very rapidly
P1 ↑ then ↓

11/58.04 100.7 157.8/7 997.2/3 7.2

shake both
P1 & P2 ↑ then ↓
by a few bars

13/13 54 100.4 157.7/8 997.3 7.2

nothing obvious in F1

in F2 < 10 bubble coming
from 1 → 2 mm ⌀
at surface

microscopic ° ° ° → coalescing

14.29.76 97.4 158.0 997.3 7.3

14/46.10 adjust P1

96.9 28.10.03

(4)

15'08.26	98.8	158.3/4	997.4	7.4
16'00.19	98.9	158.6	997.3/4	7.4
	98.9	158.4	—	—

(7°C at door)

(2H37 16'36.19)

↳ cups B1 & B2 in oven at 12 MG7

29'00	99.0/1	158.7	997.6/7	7.1

shake
↳ nothing
but P1 & P2 ↗ slightly by 1

28.12.03

30' ↓	P1 & P2			
30'47.23	95.8	158.7/8		
31'05.38	94.7	140.7		
31'12.00	94.7	128	→	few °° when shaking F2 & P2 ↗
32'09.29	92	130.8	997.7	
32'30	90.1	–	–	" " F1 & P1 ↗
33'08.45	88.8	129.9	–	7.4
33'41.89	86.6	129.7/8	997.6	–
34'01.89	85.3	129.8	–	–
34'26.12	84.1	–	997.7	–
34'43.29	83.1	130.0	– 8	–
35'25.00	81.4	129.8/9	–	–

28.12.03

(5)

36'00.67 79.5 129.8/9 997.7

36'35.29 80.1 131.7/6 997.7/8

7.6°C

Shake both F
↳ bubbles (more in F)
& P pt
slightly

37'54.86 84.6 131.8 997.8/9

39'48.48 81.5 131.0 997.7/8

41'00 80.4/3 130.3/4 997.7

Cups B1 and B2 taken out of the oven at.
13H02 has been
cup B1 = 14.8930 g
cup B2 = 28.8480 g ↓ bench
pesé à 13H05
↳ oven @ 13H07

28.12.03

48 76.5/6 131.7/6 997.7 7.3°C

↳ readjust P1 ↑ 95 then ↓

49 18 80.5/6 130.4 997.8/9 7.5°C

↳ aftershaking ↳ 85.2 ↳ 131.2

50 30 82.6 130.8 997.7/8

1405 50 76.2/3 130.5/6 998.1

1H32 78.5/6 131.5/6 998.2/3 7°C

1H11'50

1H38 cups decreasing w/ g up to 3-4 cm

1H32 79.4/5 132.4 999.4 7°C

2H12'30

28.12.03

15 H32 3 H19'00 20.9 133.5 1000

7.4°C

shade F1 & F2

31.2 133.8/9

3/13'30 66.2 134.1/2 1000

28.12.03

22

(6) 28/12/03

cupsB1 & B2 out of oven @ 15H35 (still bubbling)
for washing

pesé @ 15H41

cupB1	=	12.7480 g
cupB2	=	22.6018 g

with Balance OHAUS model GA200D
calibrated by serial 3072
SCC on
19/02/03 due in Feb04

(Calibration by: www.trade-web.co.uk
01376 521486)

Cups back in oven @ 15H45

3H25/30"	80.9
15H45	134.0
	1000.0
	7.4°C

28.12.03

7)

17H35 cupsB1 & B2
out of oven

17H36

5H16'40'' | 88.1
134.5/6
1001.0

T = 7.6 °C

A1 & A2 → 91.7
shaken 134.8

5H18'10'' | 90.6/5
135.0
1001.0
7.6° C

Pesée cupsB1 = 12.5895 g
17H46 B2 = 22.3191 g
26°C

↳ back in oven @ 17H49

98.12.03

17H49

5H29'30

81.8
135.0
1001.0
7 on door

5H39'00

81.7/8
135.3/4
1001
6/7 on door
7.1°C

Shake a few ⚘ in F2

5H40'50

84.2/1
135.5/6
1001
7.3°C

5H45'30

82.4/5
135.3
1001.0
7.2°C

~~M~~ 18H17 5H56'30"
28.12.03

82.1/0
135.5/6
1001.0
7°C @ door

→ leave it overnight

29/12/03

9H 55

21H 100
35

80.4
131.415
997.3/4

pole 8
8.8°C

P in F1 ↗ 81.7
shake F1 ⟹ degas (bubbles form rapidly)

P in F2 ↗ 132.2/3 stable GRA

switched light off

21H 3810

80.9
132.3
997.3/4

bubbling

29.12.03

26

29/12/03

10 H02 cups B2 out of oven

↳ weighing 10 H13 { cup B1 12.2126 g
cup B2 21.5576 g
26°C

↳ back in oven @ 10 H15

10 H03 80.5

21H42'30 " 132.1
997.3/4

F1 still bubbling a bit

10 H15 80.4/5

21H55'00 132.1 8.2°C
997.3/4

F1 shaken bubbly
F2 nothing

P stable 80.5
132.2/3

21H56'35 | 80.5
131.8/9
997.4
8.2

Severn Science Ltd (01454) 414723
8 Brunel Way
Thornbury
Bristol BS35 3UR

29.12.03

10 H 35

22 H 14 100

80.5
132.2/3
997.4
7.9

↗ 80.8 aftershaking and bit bubbling
22 14 45 back at 80.5

22 H 51'00

80.4/5
132.3
997.4/3
7.3

leave lightson

shake F1 F2
↳ nothing
↙
bit bubbling
P1 ↗ 80.6

23 H 07'00

80.5
132.4/3
997.4/5

7.5°C

shake F1 → bubbling bit
23.07'30 ↗ 80.7

23 H 08'00

80.5
132.2/3
997.4/5
7.5

lightoff

29.12.03

23 13 50

80.5/6
132.2
997.4

24H + 15'30

80.9
132.8/9
997.9 /998.0
6.3°C

light on
shake ↗ 81.1
few :)

24H + 35'00

81.1/2
133.0/132.9
998.2
6.7°C

light on

shake fl ⟹ no bubble
but fl ↗ 81.5

13H00 41'00

6.9
81.1/2 132.9 998.2/3

light on

29.12.03

13H20	24H59'	81.4
		132.3
		998.5
		7.1° C light off

Pl 1 81.6 when shaking but no ∞

24H59'40"	81.3/4
	132.3/4
	998.4
	7.1°C

24H + 1H29	81.8
	132.6
	998.9
	6.50° C light off back on

29.12.03

At 13 H 52 6.7°C

24H + 1 H 3200

81.9
133.6/7
998.9

stop 1H32.25

13H53
Finished

P_1 = 997.0/996.9
P_2 = 997.1/2
P_3 = 998.9

Weights after expts:

Flask 2 = 417.7g

Flask 1 = 395.4g

Room set at 4
↳ 20°C kitchen

29.12.03

29.12.03

New GRA batch:

12H55

Flask 1 570.1 g

magnet 35.8 g

stopper 277.5 g

Flask + magnet	605.9 g	116 g GR
+ GR	721.9 g	232.1 g acetone
+ acetone	954.0 g	→ 66.68%
+ stop	1231.5 g	13H00 stirring

13H17 almost all dissolved

1231.4 g w/ stopper

953.9 g without stopper

13 H 50 all dissolved | 1231.3 g w/ stopper
953.4 g

29.12.03

flask 2

cup	m (g)
B3	2.5180
	19.1594
14 H00 B4	2.512 8
26° C	33.0696
B5	2.5240
	37.2296

9'55"

Time the loss of acetone per cup
⇒ evaluate the precision of first weighing when sample just poured from flask into cup

18.8596	0.0
8466	11.7
8207	35.12
8100	45.22
8006	54.2
7900	1'04

36.9270	0.00
9197	5.02
9126	10.20
9025	17.33
8944	23.43
8816	32.93
8722	40.17
8639	46.73
8517	56.33
8407	1'04.93

36.8268	1'16.17
8195	1'22.14
8069	1'32.58
7978	1'40.20
7897	1'46.93
7807	1'54.58
7718	2'02.17
7629	2'09.80
7545	2'17.14
7447	2'25.36
7359	2'32.96
7267	2'41.05
7139	2'52.39

36.7015	3'03.11
6943	3'09.43
6866	3'16.14
6807	3'21.39
6744	3'27.02
6663	3'34.11
6546	3'44.55
6426	3'55.36
6341	4'03.02
6270	4'09.33

29.12.03

cupsB1 x B2 out 14H15

7.0

Flask 1 395.4

should be flask 1

cupB6 2.5235 g

cupB7 2.5117 20.3060 g

19.4431 10'06

~50"

Cup B6 weight loss with time (at room T)

cupB6

20.2853	0.00			
2849	7.17	20.2698	2'10 87	
2838	15.73	2688	2'19 70	
2827	23.67	2680	2'27 17	
2815	32.43	2674	2'33.17	
2804	40.61	2665	2'42 43	
2795	47.64	2658	2'48 77	
2781	58.04	2653	2'54.39	
2773	1'04.80			
2760	1'15 70			
2753	1'21 58			
2741	1'31 96			
2732	1'39 61			
2721	1'49 61			
2710	1'59.17			

26°C

29.12.03

14H25 cup B1 12.1782 g
B2 21.4782 g

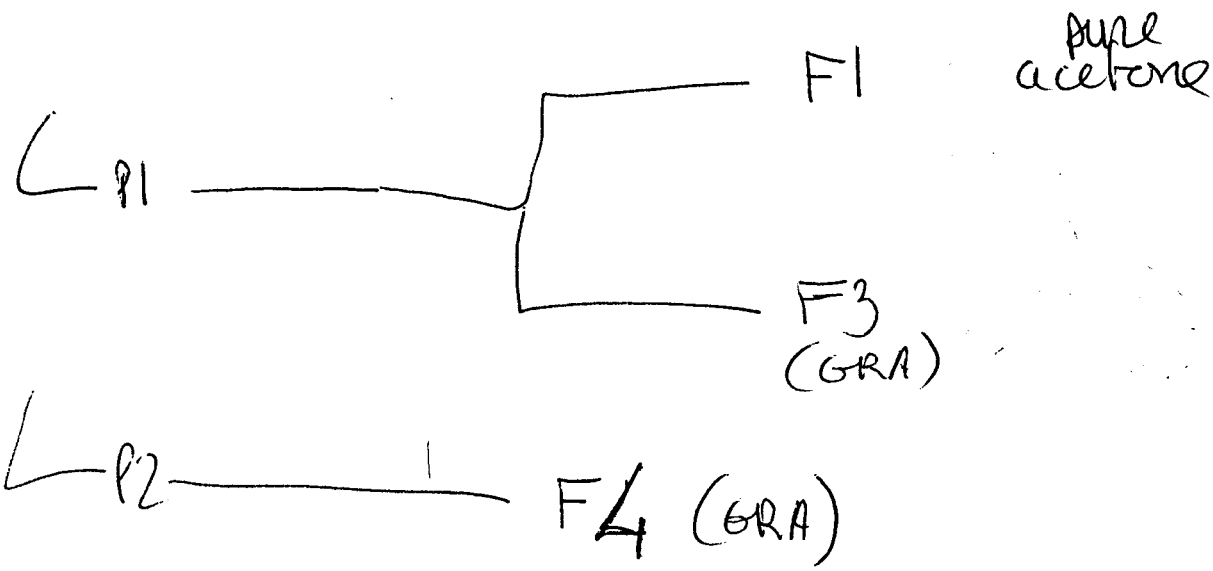
All cups in oven @ 14H27

↳ out @ 14H46

29.12.03

14H43

997.9 mbar
998.110 mbar
999.718 mbar
7.2°C



4H50

	empty	flask + GRA	
Flask 3	317.4 g	466.4 g	
Flask 4	328.0 g	431.0 g	
Flask 1	323.4 g	448.4 g	→ pure acetone

Flask prep		
	stopper	1231.0 g
	no stopper	953.3 g

29.12.03

In cold room at 7.6°C @ 14H55

998.2/3
998.2/3
999.9/1000.0

14H57 cup B8 2.5196
36.0800

new sample cup B9 2.5352
38.6189

15H07

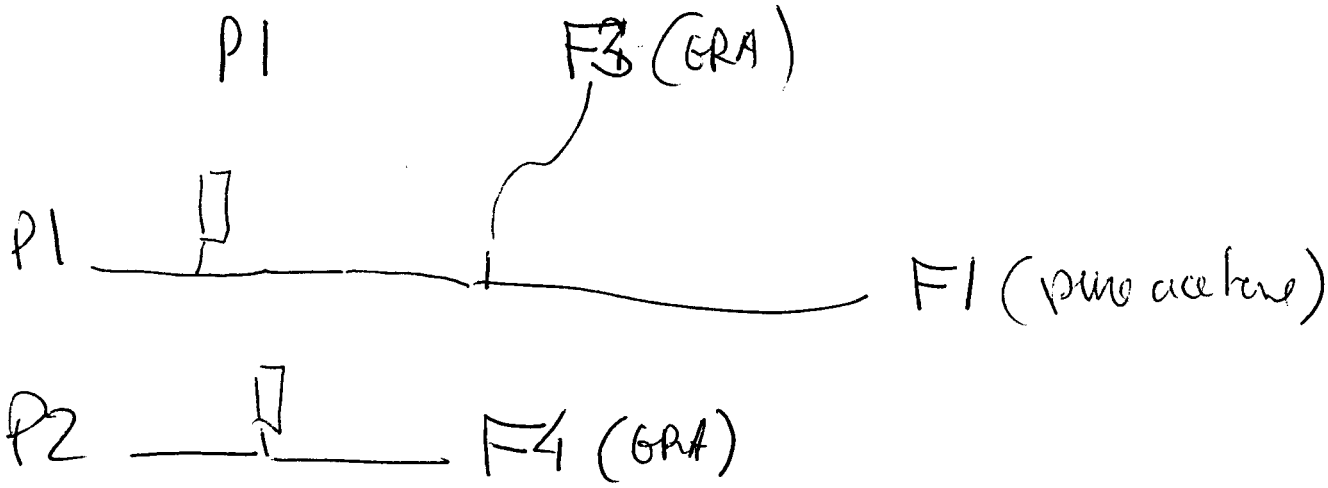
cup	m(g)
B1	12.1779
B2	21.4770
B3	13.8876
B4	26.3564
B5	29.9520
B6	19.0952
B7	18.3061

↳ in oven @ 92°C 15H11

29.12.03

15 H17

7.6°C
998.718
998.9
1000.0



Tried to install drivers and connect ASG controller on laptop but they won't "talk" to each other. Phoned Severn Science to ask advice from their technical service ⇒ on holiday until 05.01.04 Try to contact them by email in early January.

29.12.03

15 H 24

998.9/999.0
999.0/1
1000.0
8.4

Time	P1
8.29	900
22.64	797
38.88	700
50.52	650
1'01.36	600.2
1'17.67	530
1'26.00	529.516

→ P2

1'44.39

	P1	
1'57.12	529	799
2'11.96	528.5	698
2'25.02	–	600
2'35.29	–	563.2
2'47.61	499	563.1
3'03.80	430	–

T(°C)
8.3

29.12.03

3'26.02	374.1/0	563.1	1000
3'46.80	340	–	–
4'02.73	300.4	–	–
4'22.73	270	–	–
4'43.55	244	–	–
4'58.58	230	–	–
5'08.43	229	530	–
5'22.58	220	450	–
5'33.36	215	450.4	–
5'48.11	209	450.4	–
6'01.61	396	199	–
6'22.39	187	350	–
6'40.06	180	324.5	–
6'48.77	174	300	–

8.3

29.12.03

7'14.89	161	265.2/3	1001	
7'31.58	165.8	210	–	
7'47.77	164.2	158.1	–	
8'03.29	163.6	145 → bub		
8'16.52	163	136		
8'46.29	140	136	acetone no	
9'20.00	131	132	1001 → no: but yes in F3	
9'54.55	126.0	134.1	–	
	↳ rapid bubbling F3 after shaking			
11'06.89	121.0	132/131.9	1001	8.1°C
12'30.55	121.0	132.1	–	
13'05.08	118.1/0	131.9	–	–
13'32.46	117.5/6	131.8	–	8.0

29.12.03

Cups out of oven @ 15H53

Weighing @ 16H00

26°C

Cup	m (g) 16H00	m (g) 18H54
B1	12.1744	12.1584
B2	21.4697	21.4338
B3	12.9192	12.4032
B4	22.2035	20.9655
B5	25.2185	23.6810
B6	18.2485	17.4453
B7	17.3493	16.7237
B8	16.4739	14.9828
B9	17.7678	16.0010

↳ Back in oven @ 16H04

Out at 18H45 back at 18H58

29.12.03

14/45.02	116.0	132.6/7		headset P
15/40.11	115.8	133.9/8		
16/38.58	115.0	132.9		
26/19.36	109.6	132.3/2	1001	light over off 7.3°C

F4 shaken ↗ bubbling 134.4

F3 shaken ↗ 113.9

28/14.77	112.2	134.4	1001	
29/16.89	110.4	134.3/4	–	(7°C cardoon)
34/03.11	108.2/1	133.7	–	
40/38	106.4/5	133.5	–	
49/41	105.1/0	133.5/4	–	16H14
2H25.09	101.9/102.0	134.7	1003	7°C

F4 ↗ 136.1 a few but

F3 ↗ 105.3 no apparent ∞

29.12.03

2H41'30"

102.2
134.8/9
1003.0
7.3°C / 7.4°C

Shaking

2 42 22

104.6
136.7
1003
7.3/4°C

3H23'30

102.5
136.3
1004
6.8/9°C

18H45

19H00

102.6
136.0/135.9
1004

3H36'41"

29.12.03

3H55'00 | 102.617
19H19 | 136.3
1004.0
7°C

shale ↗ | 138.3
109.0

3H58'00 | 104.87
136.8
1004
7°C

29.12.03

30/12/03

①

9H13 17H49'30"

68.1/2
142.2/1
1010.0
8.4°C

F1 all acetone evaporated
shake F2 lamb
→ 83.0 adjust P
→ 89.2

17H52'30"
93.4
142.7
1010
8.6°C

17H53 40"
86.0
143.7
1010

light off

17H55'00" adjust P1 & P2
10"
82.9
134.9/135
1010

30.12.03

17H55'50"

80.5
135.0/134.9
1010

light off

17H56'50"

79.2
136.6/7
1010

17H58'15"

77.4/3
135.3/4
1010

Cups out of oven @ 9H20

Weighing @ 9H30 26° C

cup	m(g)	m(g) @ 14H08
B1	12.0925	12.0801
B2	21.2733	21.2385
B3	12.0085	11.9756
B4	20.1996	20.1316
B5	22.7183	22.6446
B6	16.7977	16.7414
B7	16.1314	16.0770
B8	14.3989	14.3547
B9	15.3953	15.3472

back in oven @ 9H36

out of oven @ 14H06

back @ 14H16

30.12.03

(2) 30/12/03

18H04'00" | 73.1/2
134.8/9
1010
8.2/3°C

18H13'30" | 66.3/4
135.1
1010
8.0°C

readjust P1 & P2
↳ so that doesn't ↓ further

18H14'50" | 67.3/4
136.9/137.0
1010

18H24'35" | 65.7 136.5 1010

↳ go & adjust P1

18H27'00" 65.3 136.5/6 1010 7.9°C
→ readjust P1
18H28'00 71.2 136.8/7 1010 7.9

@ 9H51 am

30.12.03

10H06 18H42'50" | 69.8
136.8/9
1010
7.6°C

18H50'50" | 70.0/69.9
137.0/136.9
1010
7.5°C

18H58'50" | 70.0/1
137.0
1010
7.3/4°C

→ T set back up to 6°C

19H07'30" | 70.1/2
136.9
1010

~~11~~ 10H34 19H10'15" | 70.3/2 137.0/136.9 1010 7.2°C

12H08 20H44'45" | 71.2/3
135.4/5
1010
7.5

↳ down to 4°C

30.12.03

③ 30/12/03

20 H 53'00 71.3 135.6 1010 7.4/5

F4 shaken P2 ↗ 136

F3 shaken bubbling ↗ 77.9

20H55'00 77.1 136.1/2 1010

Neco GRA sample @ 12H19

	m(g)
flask 1	570.1
magnet	35.9
stopper	277.4
A + magnet	606
+ GR	709
+ acetone	939.9
+ stopper	1217.4

14H19
939.4
1217.2 (ace here on stopper)

stirring @ 12H23

all dissolved @ 13H34

30.12.03

21 H 01' 20"

71.6/7
134.9/135
1010

FT still degassing

~~Ar~~ 13 H 34

light on

22H10'45"

71.9 → shaken 91 ↗ 78.6 bubbling
136.1 → stable
1010
6.5°C

22 H40' 20"

72.1 → still bubbling when shaken
135.4
1010
6.8/9

22H53'00"

72.3/4 ↗ 76.8 still bu
135.7/8 stable
1010
6.9

30.12.03

(4) 30/12/03

23H11'00" | 72.7
135.0
1010
6.7°C

decide to ↓ P2 slightly

shake for a while F1 & F2 → stable
↳ still degas

23H15'50" 6.9°C | 73.1/0
130.1
1010.0

23H54'40" | 73.2 → still degassing when stirred
128.7
1011.0
7.1°C

~~Mr~~ 15H22, New batch seems a bit cloudy (due to T?)
↳ stirring back on

24H00'00" | 73.5
130.3/4
1011.0
7.1°C

24H10'10" | 73.5
129.8
1011.0
7.2°C

↗ 75.6 when stirring still bubbling
30.12.03

15H41

24H17'50"

73.7/8	
130.2/3	
1011.0	
7.2/3	light off

shake F1/F2

still but less

24H. 20'00"

74.3/2
130.5/6
1011
7.3

light off @ 15H44

(24H 20'30")

17H34

24 + 2H 10'30"

75.3/4
131.6/7
1012.0
6.0/1

stirred F1 still bubbles forming but less

much more viscous

P_1 ↑ 76.0

30.12.03

(5) 30/12/03 light back on

26 H 18' 40"

75.6
131.6
1012.0
6.2°C

26 H 22' 40"

@ 17H46

75.5/6
131.2/3
1012.0
6.2°C

26 H 40' 40"

75.8/9
130.4
1012.0
6.4°C

stirred F1 → P1 ↗ 76.3
Little bobble (~ 3-4) only

@ 18 H 23

27 H 09' 40"

76.4/5
131.1/0
1013.0

stirred F1 → P1 76.9
still a few bubbles
6.6°C

30.12.03

New batch @ 18H33

1217.1 w/stopper
939.1

slightly cloudy
(∵ white pts) insuspension
↳ discard ???
↳ allright on stirred

Prepare new batch @ 18H35

	m(g)
Flask 2	554.9
magnet	34.8
stopper	279.5
F2 + m	589.7
+ GR	689.1
+ acetone	898.1 (897.3)
+ stopper	1177.6

99.4 GR
208.2 acetone
67.69%

→ stirred from 18H39

19H00 all dissolved stopper off

897.7

seem a bit cloudy
(a few particles in susp)
↳ Cow staining
blackish

30.12.03

(6) 30/12/03

27H16'40"

76.7/8
132.1
1013.0
6.7°C

stirred → P_1 ↑ 76.9

readjust P_1 a bit (↓)

27H 21' 40"

75.3
132.5
1013.0
6.9°C

27H 34'40"

75.5 ↗ 76.0 when stirring (still ↗)
131.9/132.0
1013.0
6.9°C

light off @ 19H00 (27H36'40)

27H39'00

75.6/7
132.3
1014

30.12.03

Ar 19 H 24

28 H 00' 40"

76.2/3
132.1
1014.0
6.8/9

↓ slightly P1 to 73.9
a few °° formed when stirring
↗ P1 a bit → 74.3 no °° any more when stirring

28 H 04' 00

74.4/3
133.7
1014.0
6.9

Consider stopping F2

28 H 11' 40"

74.3/4 stirred ⇒ P stays ok no °°
133.5/6 ⇒ ↓ to 132.9
1014
7°C

Ar 19 H 50

28 H 26' 40"

74.5/6
133.9
1015/1014
6.0

stirring → 74.0 a few °° but not unmeasurable

decide to ↓ P2 → 132.1

30.12.03

28H 29' 40"
74.8
133.
1015
4

28H 49' 20"
75.1
134.0
1015
7.1

readjust P_1 at P_2
73.6 → 132.6
shake → ∴

28H 52' 30"
74.1/0
133.1
1015
7.2 ⟹ light off

28H 56' 15"
74.0
133.0/1
1015

29H 09' 00
74.2
132.7
1015
@ 20H 33

30.12.03

29H 19' 30"
74.3 1015
132.8/9 6.9°C

stirred F1 → 74.5/4

OK

29H31'20"

at 20H55

74.5
133.6/5
1015/1016
6.7/6.8°C

29H39'20"

74.6
133.8/9
1016

29H44'50"

74.8
134.0/1
1016/ 6.8°C

29H45'50"

74.8
133.9
1016
6.8°C
6.9°C

stop pump after @ 29H46' → at H10

1014.0 mbar = P1
1014.0 mbar = P2
1016.0 mbar = P3

30.12.03

30/12/03

(8)

21H12

F4 = 425.7 g
F3 = 379.7 g
F1 = 323.4 g

weights after expts.

Tcold room set back up to 4°C

21H16 - 26°C

cup	empty cup (g)	cup + GRA (g)
B10	2.5835	+F4 32.2808 (18 11)
B11	2.5343	+F4 39.7722
B12	2.5468	22.5575
B13	2.5382	+F4 27.8473 (-2 min)
B14	2.5423	7.4034

done 21H23

30.12.03

30.12.03

30/12/03

9

All new aps in oven @ 21H30
Tray C out of oven @ 21H30

set new run

Flask 2	w/ stopper	1177.2 g	sample looks OK
	without stopper	897.3 g	

	empty		+GRA
F1	= 323.4 g	→	468.7 g
F2	= 319.9 g	→	444.5 g → in room at 21H35

↳ cupBIS | 2.5565
32.6932 21H37

m(g)	Time		
32.6137		32.4498	1'48.36
32.6044	06'05	4397	1'55.11
5895	15.67	4303	2'01.49
5797	22.02	4214	2'07.52
5674	30.02	4114	2'14.33
5565	37.27	4007	2'21.52
5443	45.20	3914	2'27.87
5244	58.33	3814	2'34.77
5124	1'06.36	3714	2'41.55
5004	1'14.29	3597	2'49.52
4897	1'21.61	3509	2'55.27
4795	1'28.49	3416	3'01.93
4646	1'38.43	3294	3'10.23
		3137	3'21.23

in oven @ 920C @ 21H45
30.12.03

62

Start run @ 21 H 48

1015
1015
1016
7.7°C

6'/84 | 738
740

15'/68 | 499
503

27'/06 | 327
328

37.49 | 238
237

1'40.81 | 124.1
145
7.8°C
1016

179
200
47.97

149
185
58 06 bubbles

F1 | 130 boil 1'10 15
F2 | 157

30.12.03

(10)

30/12/03

2/105.18
118.6
138.5
1016
7.8/9

1/20.12
118
135
1016
7.8
start adj

115.4
132
2/41.18

114.2/1 → gentle boiling when shaken
130.7 → 131.8/7 boiling
2/56.71
1016
7.8

118.4/5
133.3
1016
7.8
4/20.77

30.12.03

125.9
128.1 4'59.62

125.2
127.2 5'33.62
7.8°C

125.6
128.3 6'12.81
1016/1017
7.8

boil stop when P2 reach 130

124.5
127.1 7'33.81
1017

123
126 8/18 //18 less bubbling when shaking

30.12.03

(11)
30/12/03

119.4
120.0
1017

10'49.15

119.5
121.0
1017

fluctuating fairly & ↓

14'23.74

113.3
111.7
1017
7.3

20'56.65

22 H10 FI GRA prep w/stopper 1216.7
939.0

26'56"93
117.5
120.1/2
1017

30.12.03

22H19

111.1
110.7
1017
7.2°C

30'47.74

light off for night
(Tset backup to
6 from 4)

33'42.56

113.1
116.011
1017

22H23

37'21"65
@ 22H26

117.5
120.1
1017

30.12.03

31/12/03

①

② 10H00

12H10'50"

89
92 fluctuating over a range of ~ 5 mbar each
1023
4.1°C

5°C on door

Both samples still fluid

(guess ~ 40% acetone

no bubble forming when shaking

Note: exhaust pipe sec° filled up w/ acetone → drained → control on P & bit better but still fluc°

12H14'26"

104
100
1023
4.6

Set up to 8°C so that doesn't cool down room

door open

stop the stop watch by inadvertance

add +10' to time (4)

restart stopwatch

20"

98.7
83
1023
4.8

@ 10H07

Note that ~~the tubing~~ transparent polypropylene connecting the vacuum controller P2 to ~~AEG~~ is flatten ⇒ could impair w/ pumping & maintaining P2 ± constant

⇒ decide to change it & replace it by vacuum tubing

close valves leading to F1 & F2 @ 50'

4130"

1023
98.7
1023
5.3

4'40" valves reopened

31.12.03

⟹ P1 & P2 stable now that tubing has been replaced

⟹ order more vacuum tubing & replace everywhere

readjust P1 & P2

6'30"

102.2 ↗ 102.6 when stirring
90.9 ↗ 94.1
1023
5.5

F2 slightly more viscous than F1

livelighton

10'40"

102.0
90.211 ↓ ⟹ adjust P2
1023
5.4

31.12.03

10H 18 samplestirred looks OK (no clouds)

31/12/03

(2)

14'56"
102.2/3 ↗ 103.4 after stirring (no bubble)
90.1 ↗ 95.7 " " " "
1023
5.4

16'06"
102.3
91.6 ↓
1023
5.4

leave door open

All cups out of oven @ 10H24 and let cool down to room temperature.

20'00"
102.6/5
89.8
1023
5.3/4

Readjust P2 to ↑ it slightly

30'00"
102.9/103.0 ↗ 104.7 when stirring
91.5/4 ↗ 96.8
1023
5.6

31.12.03

weighing cups @ 10H40 – 26°C

cups	m (g)
B1	12.0361
B2	21.1178
B3	11.8765
B4	19.9232
B5	22.3928
B6	16.5826
B7	15.9238
B8	14.2331
B9	15.2105
B10	13.6354
B11	16.5159
B12	19.4305
B13	11.9971
B14	6.5656
B15	12.9939

back in oven @ 10H49 – 92°C

42'30"

103.615
91.617
1023
6.1°C

31.12.03

31/12/03

(3)

Stirred prepared sample

F1 w/ stopper	1216.3 g
without	938.2 g

Decide to add more gum resin.

+ GR → 982.6 g 10 H52

1260.6 g w/ stopper

GR 147.4 g
230.9

→ 61%

→ stirring

46' 20"

103.7/8
92.0/91.9
1023
6.2

Shut door @ 10 H54 (lab room ~ 17°C)
leave light on

47' 30"

103.8/9
94.2
1023.0
6.2

49' 40"

103.9
94.5/6
1023
6.7

31.12.03

59140"

104.3/4
94.4
1023
6.5

(9 on door)

T set
↳ from 8 back down to 6
@ 11H07

11H07

GR all dissolved
but a few
~~one~~ small bits

1260.5 w/stopper
982.4

back to stirring

1H02'30"

104.5
94.413
1023.0
6.8

↗ 107.5
↗ 100.6

when stirring

1H17'40"

105.110
92.8/9
1023.0
6.5

↗ 108.8
↗ 99.5

when stirring

31.12.03

31/12/03

1H 28' 50"

105.4/5
93.2/1
1022.0
6.9/7.0 → switch light off 11H36

2H 01' 00"

106/105.9 ↗ 109.1
96.6 ↗ 101.6 when stirring
1022
6.8

Light off

At 12H31, 2H24'00"

105.8/9
94.8/9
1022
6.5°C ⇒ light on drain pump tubing

2H46'30"

106.2/1 ↗ 108.2
95.0/94.9 ↗ 97.9
1021
6.8°C

31.12.03

At 12H50 FT + stop

1260.4
989.4

all GR dissolved

2H49'45"

106.3
95.3
1021.0
6.8

2H52'45"

106.4
94.9/95.0
1022.0

At 13H43, 3H36'40"

106.7/8
96.3
1021.0
7.7 → light off

dropped stopwatch
↳ restart after Δt = 10"

10'00"

106.8/9
97.1/2
1021.0
7.6°C

15'00"

106.9/8 ↗ 110.1
97.3 ↗ 98.0
1021
7.6°C

At 13H59

31.12.03

31/12/03

1H00'45" | 106.8/9 ↗ 109
98.0/97.9 ↗ 98.4) when stirring (no ∞)
14H45 | 1020
6.9

Cleaning flasks

15H02

1H18'40" | 106.8/9
96.8
1020

1H31'50" | 106.7/8
97.8/9
1020

~~M~~ 15H16 | Fl + stopper 1260.3
without 982.3
T = 11°C | + GR 1133.1
+ stopper 1411.1

GR 147.4 + 150.8 = 298.2
230.9
→ 43.64%

→ stirring on SM8 (bigger stirrer than SM1)

31.12.03

15 H 22

1H 38'10"

106.6 ↗ 108.6
97.9 ↗ 98.4
1020
6.8/9

light off

17H30

3H 47'00"

105.5 ↗ 108.7 no ∴
96.7 OK no ∴
1017.0
7.2

3H48'30"

106.1
96.5
1017
7.2

Tset from 6 ↓ 5°C

At 17 H41 all GR dissolved

1410.9 g
1132.9 g
(some acetone in stopper)
↳ stirring on SM1

3H57'30"

105.4
96.5/6
1017

31.12.03

31/12/03

3H59'00"

105.3/4
96.6
1017.0
7.3°C

Tset down to 4°C

4H01'20"

105 3/4
95.8/9
1017.0

4H07'40"

105.2/3
95.8
1017.0

17H52

4H12'00"

105.1
96.1
1016.0

4H18'15"

104.8/9
96.3/4
1016
7.4°C

4H27'00

104.8/9
96.3
1016

31/9/03

4H 40'00" 104.5/6
96.0/1
at 18H24 1016.0
6.9°C

~~4H41~~ ↗ 106.9
↔ OK

4H41'20" 104.6/7
95.0
1015/1016
6.9

↳ 4H41'35" stop

Time run = 12H14'26"
+ ~10' (<)
+ 3H36'50"
+ 4H41'35"
19H 1H41'1'50"
20H43'
Start @ 21H48 on 30.12.03
Stop @ 18H25 on 31.12.03
18H25' + 2H12'
= 20H37'

18H27 F1 403.9 g
F2 373.0 g
after expts.

31.12.03

31/12/03

Flasks for next run:

flask 3 = 317.3 g (empty)

flask 4 = 327.9 g (empty)

18 H30 26°C

	cup	empty cup (g)	cup + GRA (g)
F1	B16	2.5664	23.4658
	B17	2.5635	29.9911
	B18	2.5620	28.2683
F2	B19	2.5629	30.4314
	B20	2.5322	9.0768

done @ 18H35

31.12.03

18H37 load F3 et F4 w/ GRA

Flask 3 = 317.4 g → 451.6 g

Flask 4 = 328.0 g → 545.9 g

F prep 1 + stopper | 1410.8 g
| 1132.9 g

in cold room at 7.4°C at 18H41

| 1013
| 1013
| 1015

↳

cup	empty (g)	? or 35.	+ GRA (g)
B 21	2.5527	42.	4910
B22	2.5669	? 42.	5687

@ 18H44

all new cups B16 to B22 in oven @ 18H47

31.12.03

18H49 | 1013
1013
1015
8.3

8140 | 652
640
1015

18!15 | 422
419

1100.77 | 166
156
8.4
1015

30.71 | 276
275

11484 F3 151.0 a few

40.30 | 210
210

1120.00 F4 139 a few

47.87 | 183
180
8.4
1015

135.1
8.4
F4 boiling

| 127
133

149 09

| 125
136
1015
8.4

2'23 87

3'09.68 | 117.7
132.5
1015

Light off

31.12.03

cupB23

2.5626

40.1192

@ 18 H54

Spilled in corridor cup sent
↳ direct to bin

10/26.33 | 116
119
1014

New cupB23 :

| 2.54 42 g
43 2950 g

@ 19 H04

left in balance room
@ 26°C

21'15'' | 109.0
112
1014
7.9

31.12.03

21H59 31/12/03

3H09'40"

82.6 ↗ 99.6 when stirring
64.5 ↗ 102.4 & bubbling
1010
7.0 w/ light on

$T_{set} = 4°C$

22H05 cup B23 37.2970 (loss ~ 6g in 3 hours)

↳ 22H07 in oven @ 92°C

22H09 cups weighing

cup	m(g)	cup	m(g)
B1	12.0154	B12	19.2014
B2	21.0630	B13	11.8550
B3	11.8415	B14	6.5262
B4	19.8404	B15	12.8735
B5	22.3021	B16	15.9581
B6	16.5197	B17	20.2891
B7	15.8621	B18	19.1857
B8	14.1869	B19	26.0973
B9	15.1590	B20	7.9194
B10	13.4834	B21	20.0506
B11	16.3093	B22	27.3050

Done @ 22H18

31.12.03

→ All cupsin oven @ 22 M21

31.12.03

3H32'00'

81.7/6
64.0
100g

22H30 3H40'15"

82.2 ↗ 96.2
63.2 ↗ 97.2
100g
7.3

light off
↳ set @ 70°C
60 min

3H42'00"

87.5 ↓
88.2 ↓
100g
7.3

3H44'40'

84.9/85.0
85.1/2
100g.0

31.12.03

31.12.03

01/01/04
①

11H02

16H12'40"

83.2/3 ↗ 85.6
73.5/6 ↗ 88.5 bubbling
1001.0
5.30°C
light on

16H14'00" ↓ P1

Cups out of oven @ 11H05

16H16'20"

62.0
77.6 → 6.00 from the a few seconds when stirred & P1/P2 ↗
1001.0
5.5

16H20'00"

71.7
80.8
1001.0

16H28'50"

79.3/2
82.2/3
1001
5.5/6

tubing #3 flatten
↳ P1 & P2 fluctuate

01.01.04

16H30'00" → decide to change tubing & replace by vacuum tubing

16H30'50" | 94.517
97.6
1001

16H31'00" valve off of F4 closed & pump off

↳ P let back in F3 to atm via vacuum controller no 3

16H31'30" | 450
100.5
1001

16H31'50" | 809
101.3
1001.0

16H32'00" | 999.7
102.0
1001.0
5.4°C

Weight
F3 = 423.2g
@ 16H33'30

↳ 16H35'50" | 999.718
109.112
1001.0

01.01.04

Change tubing.

Pump back @

11H26 ⇒ 16H36'20"

16H36'34"
350.0
110.3
1001.0
6.0°C

16H37'01"
120.6
111.0
1001.0
6.1°C
→
81.7
111.7 16H37'20"
1001.0
6.1°C

↳ open F4 valve 16H37'30"

16H37'37"
67.5
99.0
1001.0

16H37'52"
59.2
85.0
1001.0
6.2°C

01.01.04

01.01.04

16H38'26" | 46.0 → readjust P1
70.1
100.1
6.3

16H38'52" | 50.7/6 ↗ 84.6, 16H39'20" when stirring ↳ ∞
69.8/7
100.1
6.3

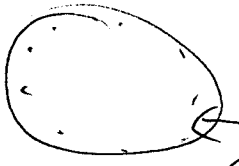
16H39'40" 80.2 continuous boiling

P2 ↗ 82.1 ∞ too

16H40'23" | 71.5 →
80.8
100.1
5.8

interface air/GRA

∞ all over surface of flask



at interface air/GRA

∞ essentially on side of flask

16H41'14" | 64.6 → stirring
76.7
100.1
5.8

16H42'06" | 67.6
76.5
100.1
5.8 → Tset cold room backup to 6°C

01.01.04

16H 43' 40''	61.6 69.8 1001	
16H 47' 10''	59.7/6 69.3/2 1002	→ F3 : GRA still boiling all over outside sample
16H 48' 30''	59.6 69.4 1002.0	

01.07.04

Weighing cups @ 11H 42 01/01/04

Cup	m (g)
B1	12.0034
B2	21.0311
B3	11.8220
B4	19.7937
B5	22.2482
B6	16.4823
B7	15.8280
B8	14.1601
B9	15.1280
B10	13.4196
B11	16.2055
B12	19.0886
B13	11.7971
B14	6.5095
B15	12.8175
B16	15.4806
B17	19.6280
B18	18.5709
B19	25.3040
B20	7.7379
B21	19.4102
B22	26.3459
B23	27.9825

Done @ 11H52

Back in oven @ 92°C 01.01.04
@ 11H58

11 H 54

17 H 03' 40"

55. 4/5 → still bubbles forming & bursting all over interface over surface
69. 5
1002
6.0

P2 ↗ 88.6 when stirring to remove pellicule made gasses @ surface a few 8° but not much

P1 ↗ 72.6 when stirring

17 H 06' 36"

68.0
76.6
1002.0
6.0

17 H 09' 20"

54.3 bubbling all over interface
69.5
1002

17 H 09' 45"

55. 1/2
69. 4
1001/1002

01.01.04

01/01/04

17H16'30"

54.3/2
69.6/7
1001
6.1

Readjust a bit P1 & P2 & P2 a bit lower than 69.6
so P1 doesn't

Stored F3 & F4

17H18'40"

55.1/0
68.2/3
1001
6.2

17H24'45"

55.3/4 → still boiling
67.5/6
1001

17H30'00"

55.7/6
67.7/6
1001

GR
fac C 2110.5 g 17H22 → stored in cool box 01.01.04

17H34'00"

55.1/2
67.8
1001.0

17H40'30"

55.4/5
68.2
1001

bubbling slowing down (↗)

17H51'00"

55.8/7 ↗ 63.2 when stirred
69.0 ↗ 93.0
1001.0
5.8

17H53'30"

58.4
79.8
1001
5.8

Set T cold room increase from 4 to 5°C
Light left on

17H56'10"
01.01.04

56.1/2 → still bubbles all over interface
67.8/9
1001.0

i 12H59 01/01/04

18H09/30"
55.8/9 still degassing °° all over interface
68.8/7 a few °°
1001.0
6.1°C → stirred → P_2 89.0

P_1 ↗ 64.9 when stirring
viscous but still liquid
(~ Golden syrup)
PGS is at ambient (220°C)

At 13H09
18H14/30"
55.2
68.6
1001.0
6.6°C
back to set P & stable more rapidly

20H10/50"
58.1/0 → fully foamed but still moving
71.8/9 → bubbles continuously a bit
1002.0
6.9°C → ↗ 92.6

15H03 20H13'10"
58.4
82.8 ↓
1002.
7.1 → light off

20H13'45"
58.2/1
71.4/3
1002/1003

01.01.04

15H15 20H25'50"

58.6
71.6/7
1003.0
6.9°C

shake F4 → bubbles P2 ↗

20H27'00"

58.5
71.5/4
1003.0
6.9°C

20H30'15"

58.6/5
71.3/4
1003

20H36'00"

58.6
71.9
1003.0
7.0

→ Tset down to 4°C (from 5°C)

20H39'30"

58.7/8
71.5/4
1003.0

01.01.04

01/01/04

20H45'00"

58.9
72.0 → P_2 ↑ 85.0 when stirring
1003.0
6.9

20H47'40"

59.0/58.9
71.8
1003.0

At 15H51

21H01'00"

59.3
72.4/5 ⟹ ↑82.2
1003
6.8°C

(light is off)

21H03'10"

59.3
73.6
1003
6.9°C

21H14'40"

59.6 → stable
72.4/5 ↗82.0 when stirred
1004
6.7°C

21H33'10"

59.6/7
73.0/72.9 Stir → P2 ↑ then ↓
1003
6.6°C → light on

01.01.04

21 H 35'00"
59.7
72.8/9
1003/1004
6.6/6.7

16 H 52

22 H 02' 30"
60.4/5
74.0 → 80.3 still 80
1004.0
6.8

22 H 03' 00"
60.3
73.9
1004.0
6.8
light off

22 H 21' 00"
60.7
74.2 ↗ 83.5
1004.0
6.6/6.7

22 H 22' 00"
60.6/7
74.3
1004
6.6/6.7

22 H 45' 00"
61.2
75.1/2
1005.0
6.4

01.01.04

01.01.04

(8)

~~Mt~~ 17H47

22H58'00"

61.1

74.4/5

1005.0

6.4

Lower P2 a bit & stir Flask

22H59'00"

61.0/1

71.5/6

1005.0

6.4

23H16'40"

61.5

72.1 ↗84.0 when stirred

1005.0

6.1

23H18'00"

@ 18H07

61.3

71.6/7

1005.0

6.1 → lighter

01.01.04

102

20H17

25H28'00"

62.6
74.1 ↗84.0 bubbling
1005/1006
7.0/7.1

↳ light off

20H43

25H53'00"

62.5
73.6 → stir ⇒ P2↗ >80
1006.0 bubblg
↳ consider turn leaves overnight
6.8

25H55'30"

62.5
73.4/5
1006.0
6.8

At 21H01

25H11'30"

62.817 ⇒ photos taken
73.7
1006.0
6.7

01.01.04

9

26H14'00" | 62.8
74.6
1006
6.7/8

↳ 26H14'23" close valve F4

26H14'40" 75.6 ← P2

26H14'50" stop F3

26H15'48" | 1004
71.3
1006

21H05

26H16'30" 72.4

26H17'40" 72.4/5 °° gently

stir ↗ 82.0 mbar

26H | 18' 30" 76.6
40" 74.5
50" 73.4 —— 1004 / 1005
19' 00" 72.5 7.0/1

21H09 F3 = 403.0g - 403.1g

(402.9 @ 26H27'50 21H17) 01.01.04
403.0

26 H 21' 50"

1009
72.2 /3
1006

26 H 27' 30"

1004.0
72.3/4
1006.0

26 H 30' 30"

1004.0
72.4 ↗ 80.6
1006.0
7.0

26 H 35' 00"

1004.0
73.6/7
1006.0

26 H 43' 20"

1004.0
73.2 ↗ 84 & 00
1006
7.2 ↗

29 H 01' 30"

1004/1005
73.6
1006
7.2

27 H 59

29 H 09' 40"

1004
73.4 ↗ 78.2 still so
1006
7.4/3

Room 26 Bar
4 bar night (light)

01.01.04

01.01.04

22H00 | 27H11'50" | 1004
73 4
1006

Leave the expt running over night until tomorrow

02/01/04 (1)
8H34

24H + 13H44'30" | 1012.0
85.4/5
1013.0
8.9 → lower T

light off → T cold room set down to 3°C

13H55'20" | 1012.0
85.4/5 ↗ 88.4 still bub when shined
1014.0
8.7

T cold room set down to 0°C at 8H47

24H + 14H | 1012.0
84.8
1014.0

02.01.04

24H + 14H19'40" | 1012.0
85.7
1014.0
8.7/8.3

cups out of oven
@ 8H48

↳ weighing @ 9H15 26°C

cup	m(g)
B1	11.9812
B2	20.9759
B3	11.7915
B4	19.7239
B5	22.1627
B6	16.4257
B7	15.7800
B8	14.1243
B9	15.0852
B10	13.3546
B11	16.1108
B12	18.9738
B13	11.7434
B14	6.4892
B15	12.7505
B16	15.2377
B17	19.3167
B18	18.2612
B19	24.8506
B20	7.6555
B21	19.0985
B22	25.8745
B23	27.3573

done @ 9H24
back in oven at 92°C
@ 9H26

02.01.04

Yr 9 H 27

02/01/04 107 (2)

24H + 14H 37' 45"

1013
86.415
1014
7.9°C a few bubbles when stirred

24H + 14H 45' 40"

1013.0
85.6
1014.0
7.8°C

24H + 14' 56' 30"

1013
86.617
1014

24H + 15H 15' 45"

1013
87.1
1015
7.3

24H + 15H 22' 30"

1013
86.112
1015
7.4

10H12

↳ modify by −2°C Tset.

02.01.04

108

1013.0
86.4
1015.0
7.2/3

24H
15H 32' 10"
at 10H22

11H 26

24+
16H37' 20"

1014.0
88.0
1015
6.3/4 door open

~~Air~~ → Portable
no bubble

11H28

24H
16H 38' 50"
6.7/8
87.5/6 → 16H39 drop
1014
1015

1014

02.01.04

mF4 = 485.0 g @ 11H31

11H33

cups B24 | 2.5416 g
| 36.8495 g

B25 | 2.5500 g
| 39.5579 g

→ oven 11H34

In an ideal world
(not having to be in Portsmouth by 14H30 to catch the 15H30 ferry to Le Havre!!!), it would have be nice to let running F4 longer but @ a lower pressure (until this evening for example, at lowest P_2 reached on 01.01.04)

02.01.04

hours for 02.01.04

8:10 → 12:00
16:00 → 18:30
19:30 → 20:30

Will have to order vacuum tubings (to replace all the tubings in the experimental setup) and one 2l flask.

02.01.04

06/01/04 Set up EOS expt in UPMC's lab inside the flume cabinet (= "flotte")

17H12 Test system

1014.0 → 13.4/5
1012 → 10.9
1013.0
24.7°C

17H30

1014
1012
1013

nettoyage flasks

18H10

1014
1012
1013
24.9

switched on @ 8:00 this morning.

T_{four} = 98°C avec consigne @ 92°C
↳ consigne @ 88°C

18H25

1014
1012
1013
23.9 (vitre baissée)

four = 94°C
avec T_{set} = 88°C

06.01.04

:N2

06/01/04
②

	T	P		
acetone boiling point	23°C	27900	Pa	28292
	24°C	30123	Pa	29444
	25°C	31358	Pa	31317

18455 (pump lb)

237 lb
1014
1012
1013

T {from oven = 94°C

06.01.04

07/01/04

①

10H15

1013
1011
1013
23.5 (hotte left on ~~the~~ + light) night

New sample: GRA

flask 579
magnet 34.8
stopper 278.7

F+M 602.7
+GR 706.2
+acetone 870.2
↳ stirring

103.5 g GR
163.1 g acetone
61.18 wt%

G.S.3

10H25

870.0
+ stopper 1148.7

DH20

1013
1011
1013
24.3 Thermo + en hauteur

10H35

1013
1011
1013
24.2

1148.5
869.7 almost all dissolved

07.01.04

1DH59 869.6 g

$T_{fom} = 94°C$

10H17 | 1148.3 g
869.3 g

| 1013
1011
1012 mbar

Look at available balances:

Mettler AE 260 Delta Range

Mettler AE 260 Delta Range N° serie: M85851 (cal. 04/04/03 next. 04/2005)

up	empty PMMP	empty Magie	+ GRA
P1 = 26	2.5471	2.5469	31.6692
P2 = 27	2.5515	2.5513	37.0551

↳ in oven @ 11H25 @ 94°C

11H10

		empty	+GRA
Flask 1	=	279.9	342.0 341.9 → 363.0
Flask 2	=	267.6	368.0

boomt

Note flask 1 has GRA on neck's inside

could biaise weighing flask method

07.01.04

11H22

1013.0
1011.0
1012.0
24.4/5

11H31

1013.0
1011.0
1012.0
24.5/6

P1

901.8 6.15
805.0 18.07
736.6 30.93

P2 ↓ after 50/09

910.6 1'02.71
808.9 1'15.21
693.9 1'33.30
567.3 1'51.43 733.8 P1

P1

647.6
566.6 2' 11.21

07.01.04

539.5
566.2
1012 | 2'33.74

462.4
566.0 | 2'54.06

349.8
565.8 | 3'24.30 Hmm ↓ P2

348.6
468.6 | 3'47.47

347.3/4
355.1 4'13.21

P2 309 → kt 00 4'37'09

270.9 4'55.33
P1 = 346.1

256.1 5'10.59
346.0 24.6°C
1012

07.01.04

07/01/04

(3)

5'30 ↓ P1

289.45
253.9
1012

6'02.40

250.7
253.1
1012

6'45.71

when stirred, P ↗ rapidly
not much ∘∘
(very fluid)

228.7
252.8/9
1012
24.6

7'00.00

208.5
252.7
1012.0
24.6

8'45.30

207.6
252.6/7
1012
24.6

10/05 65

07.01.04

Cups out of oven @ 11 H 44

Pesée @ 11H54

26 = P1	16.4898	Magie
27 = P2	19.9059	
11H55		
26 = P1	16.4895	PMMP
27 = P2	19.9043	

back in oven @ 11H56

206.2
252.7
1012
24.6° C

13' 18" 30 @ 11H45

205.0
252.6
1012
24.5°C

24' 46" 45

adjust P1 to make sure air ↓ can't carry air

210.0
252.6
1012
24.5

25' 50" 00

adjust P1

206.112
252.6
1012
24.5

26' 10" 00

07.01.04

07/01/04

(4)

acetone = AnalaR from BDH (VWR International Ltd Poole UK)

CH_3COCH_3 = 58.08 g/mol

purity ≥ 99.5 %

Lot K32632306 345
Prod 100035 R

28' 45" 00"	205.7 252.5/4 1012.0 24.4/5 °C
34' 20" 00"	205.4/3 252.5 1012.0 24.5
46' 20"	204.8/9 ↗ 223.7 when stirred 252.3/4 ↗ 257.3 when stirred 1012.0 24.5
47' 45"	204.5/6 252.3 1012 24.5

no go

12H01

07.01.04

120

Cups out of oven @ 12H21

weight @ 12H31

cup 26=P1 15.6825

cup 27=P2 18.4746

back in oven @ 12H 33

49'49"

203.3/2 → adjust P1 a bit (↑)
252.3
1012
24.5

50'15"

206.5
252.4
1012
24.5

58'50"

209.0
252.2
1012
24.6/5

At 12H34

1H01'30"

208.8
252.1
1012
24.6

1H05'50"

209.0/208.9
252.1/2
1012
24.9

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07/01/04
(S)

1H 25'45"

206.7/8
251.7/8
1011
24.6

↗ 226.2 when stirred
↗ 257.2 when stirred
back in < 30 sec
at 252.0

1H 27'00

211.8
251.8
2011
24.6

1H 27'20"

203.2
251.8
1011

1H 27'35"

202.4
251.9
1011
24.6

At 13H00

1H 29'00"

200.8/9
251.8
1011.0
24.6

1H 32'00"

200.3/4 ⇒ ↗ P1
251.6/7
1011
24.4

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1H33'00''

201.3
251.7
1011.0
24.4/3

1H55'30''

200.3/4
251.1/2
1010
24.7

2H14'40''

199.5 ⟹ readjust P1
250.8/7
1010.0
24.8

At 13H52

200.6/5
250.6/7
1010.0
24.7

2H20'30''

At 16H48

197.9/8 ↗ 226.4 when stirring
249.8/9 ↗ 258.2 when stirring
1009.0
25.2

5H15'50''

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(6)

SH17'30"	199.1/2 250.2/3 1009.0 25.2
SH22'30"	197.9/198.0 250.0 1009.0 23.9/24.0 (thermometer placed underneath flask

cups out of oven @ 16H49

weighed @ 16H56 cups | 26 = P1 14.9673
27 = P2 17.4874

back in oven @ 94° C @ 16H58

16H58	SH27'00"	197.6/7 250.0 1009 24.2
	SH39'15"	197.6/7 249.9 1009 24.4
	SH45'20"	198.6/5 250.0 1009.0

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17 H36

6 H04'20"	198.5/6 249.9/250.0 1009.0 24.6°C
6 H 11'50"	197.5/4 ⇒ ↑ P1 249.7/8 1009.0
6 H20'00"	198.4/5 249.8/9 1009.0 24.7°C
6 H25'00"	198.9/198.8 249.8/7 1009.0 24.7°C

@ 17H57

↳ leave for night

6H51'30"	197.7.8 249.7/6 1009 24.6

18H24

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21 H 47' 40" (1)

188.2/3
241.3
1001.0
23.8/9

~~Kh~~ 9 H 37 22 H 00' 00"

188.4/3
241.0/1 ↗ 244.0 back down to 22 H 00'
1001.0
23.9

241.7 31"
241.4 38"
241.2 56"

→ stirred

P1 ↗ 191.9 + a few bubbles 22 H 01' 30"
191.5 22 H 01' 45"
190.4 — 02' 04"
189.5 — 18" bubbles continue forming
188.8 — 45" " "

T = 23.9/24.0

22 H 03' 50"

188.7/6
241.3/4
1001.0

Cups out of oven @ 9 H 35

cup 26 = P1 14.5859
cup 27 = P2 17.0049

back in oven @ 9 H 39

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22H 08'00"

188.6/5 bubbling has stopped
241.1/2
1001.0
23.9/24.0

22H 38'00"

187.5/6
240.3/4
1000.0
24.0

Readjust P1 & P2 so that they don't carry on ↓

22H 39'00

188.3/4 ↗ 189.2 when ~~stirred~~ (no °°)
241.1 ↗ 243.6 when stirred
1000.0
24.0

22H 40'20"

189.1
241.2
1000.0
24.1

22H 40/~~4~~0"

188.9/189.0
241.1/2
1000
24.1

22H 47'20"

10 M19

188.4/3
240.8
1000
24.1

⇒ readjust P1 & P2
⇓

22H 48'40"

189.0
241.6/7
1000.0
24.1

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(2)

23H19'10" | 187.6
240.8
999.5
24.0

adjust P1 & P2 (↑)

23H39'20"

@ 11H12

188.0/187.9
241.7/8
999.5
24.1

23H50'50" | 187.9/8 ↗ 191.7 (no ∞)
241.5/4 ↗ 243.7
999.2/3
24.1/2

23H52'20" | 189.2/1 & ↓
241.5/6
999.2
24.2

23H52'40" | 188.4/5
241.4
999.1
24.2

23H53'00" | 188.2
241.4
999.1/2
24.2

24H03'30" | 187.9
241.4
999.2/3

readjust a bad P1 (↑)

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24 H 14' 45"

188.4 (↑ 189 & ↓)
241.1/2
998.9
24.3

24 H 15' 30"

188.3/2
241.1/2
999.0
24.3

24 H 16' 40"

188.1/2
241.2/3
999.0
24.3

14 N52 = 24 H + 3 H 19' 50"

183.6/7
236.4/5
994.4/5
24.6.7

24 H + 3 H 29' 30"

188.2/3 ↑ 186.3 a few
237.6/7 ↑ 241.0
994.4/5
24.6

24 H + 3 H 33' 30"

188.5/6
240.2/3
994.4/5
24.6

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↑ P1 & P2 then
~~sound~~

08/01/04

(3)

15H24

188.4/5 ↗ 190.7
239.9 / 240.0 ↗ 241.6 } no ∴
994.2/3
24.5/6

24H +
3H52'10"

24H +
3H54'00"

189.9
240.1/0
994.1
24.6

24H 3H59'00"

188.6
240.1/0
994.3/4
24.6

15H50

24H + 4H18'20"

189.9 / 190.0 stable (∴)
240.5 ↗ 242.5
994.7
24.5

24H 4H28'20"

188.5/4
240.0
994.4/3
24.6

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24H + 4H 37'00"

188.4/5
239.9 readjust P2 (↗)
994.2
24.6

24H + 4H 37'30"

188.5/6
241.0
994.2/3
24.6

24H + 5H 10'00"

189.5/6 stable when stirred
241.1 ↗ 242.8
994.6
24.7

1 ou 2 bulles when stirred but stable

AM 10UP

24H + 6H 02'45"

188.5/4
240.6/7
994.5
24.8/9

readjust P1/P2

24H + 6H 04' 10"

190.1/2
242.0
994.4/5
24.8/9

N 17/146

24H + 6H 14'10"

189.9 / 190.0
241.9 / 242.0
994.5
24.8/9

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08/01/04

(4)

New GRA batch =

F	567.9 g
M	34.7 g
S	279.1 g

18 H 50

F + M	602.7 g
+ GR	795.3 g
+ acetone	925.4 g
+ S	1204.6 g

192.6 g GR
129.1 g acetone
40.13%

→ stirring

24 H + 6 H 19' 30"

189.8/7
241.6
994.2/3
24.8/9

24 H + 6 H 30' 10"

189.4/5
241.6/7
994.5
24.7/8

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138

18H11

+6H39'45"

189.4/5
241.7/8
994.4
24.8°C

1859
H

188.8/9 → 189.4 when stirred - no °°
240.8 → 242.6 - no °°
24H
7H22/30'
993.7/8
24.9

7H28'55'

190.2/3
241.1
993.8/9
24.9

7H29'30"

189.1
241.1/0
993.8/9
24.9

M 19H02

stirring has stopped
still undissolved bits of GR

F+S 1204.3g
F 925.0g

↳ stirring

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(5)

7H31'15"

188.8/9
241.0 → 189.5
993.8 → 241.2
24.8

on own
($\Leftrightarrow P_{atm}$ & T?)

7H32'00"

189.1/2
241.0
993.8
24.8

Cups {26/27, P1/P2} out of oven at 19H05

7H33'40"

188.9 → 189.6 on its own
241.0/240.9
993.8/9
24.7/8

7H51'50"

189.1/0
241.1/2
994.1
24.9

17H24 all GR dissolved

1204.2 g
924.8 g

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7H53'00"

189.0
241.3/2
994.0/1
24.9

19H26 aup

26 = P1	14.5076
27 = P2	16.9112
P3	2.5549
P4	2.5597
P5	2.5711
P6	2.5969
P7	2.5369

19H29
7H57'30"

189.0
241.2/1
994.0
24.8

7H59'35"

189,3
241.1/2
993.9
24.8

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08/01/04
(6)

19H 33

8H01'00"

188.8/9
241.1
993.9/994.0
24.8

24H 8M 01' 30" ⟹ stop pump 19 H34

188.8/9
241.1/2
994.0/993.9
24.8/7

⟹ pesée
@ 19H36

F1 = 319.8 g
F2 = 325.1 g
325.0 g

994.7/8
992.5
993.9
mbar

5

08.01.04

Cups prepared @ 19H38

	empty (g)	+ GRA (g)
P3	2.5550 /49	19.4689
		↳ Flask 1
P4	2.5596	54.3805 (↓ flask)
		↳ Flask 2

Timing of acetone loss in cup P4:

54.2298	0
2114	6.73
1923	14.02
1716	22.17
1556	28.49
1399	34.89
1245	41.33
1033	50.20
0904	55.73
0747	1'02.64
0552	1'11"08
0305	1'22.17
0146	1'29.43
53.9999	1'36.39
9868	1'45.17
53.9647	1'52.80
53.9461	2'01.70
9297	2'10.02
9092	2'20.08
8892	2'30.29
8701	2'40.19
8588	2'46.17
8297	3'01.52

↳ all 4 cups in oven @ 19H47

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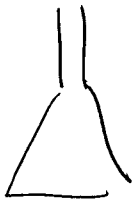
08/01/04

7

Note: on GRAX from cups method: the more GRA solution in cup, the more precise the method is
However, the more time it takes to pour the GRA in the cup. Therefore the higher is the acetone loss during the cup preparation & the less precise the method is ⟹ check/calculate precision

At 19H51 F+S 1203.8
(F should be 924.4g but forgot to weigh!!)

994.6/7
992.4/5
993.8/9
24.7



→ F1 323.3 g ↳ 410.3 = P1 ↳ Run P3

→ F2 324.1 g ↳ 423.1 = P2 ↳ Run P4

All set up @ 19H56

↳ cups

P5	2.5711/12	28.2698
P6	2.5970/69	45.5773
P7	2.5369/70	43.7794

Done @ 20H01

↳ in oven @ 20H05

08.01.04

20H06

994.9 / 995.0
992.5
994.0 / 993.9
24.8/9

↳ 20H07 start

~ 700 mbar 7'07 = time
~ 500 mbar 14.97
24.9 °C
994.2 mbar

320 30.94

230
285 45.00
994.1/2
24.9

P_1 = 199 mbar
P_2 = 252 mbar 59'31

192 → surface fremt 1'12''16
237
24.9
994.1/2

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(8)

181 mbar
235 mbar 1'36.91
24.9°C
994.1/2

176.5
235.0
24.8 1'53.63

168.7
234.7 2'16.59
24.817

157.0 → P ↑ 193 & boiling
234.3 → nothing => decrease P
994.1/2 2'56.50
24.617

185.5
193.4 4'11.72
994.1
24.5

182.1
188.4 4'51.34
994.1/2
24.5

08.01.04

5' 40".00

190.9
191.9
994.1

6' 30"

209.1/2
208.3/4
994.1
24.1/2

10' 00" 69

223.2
222.4
994.1
23.7

bay
(door open)

cups out of oven @ 20H19 ⟹ abort (too liquid might spill out samples)
cups left in the oven

13' 10" 00

232.4
231.7
993.9

leaks somewhere ???
or oversaturation?
No! liquid in pump

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08/01/04

⑤ → ~~try~~ to remove liquid acetone w/ no success

→ plan a cold trap maybe

15' 00"	238.1/2
	237.4
	994.0/1
	23.8/7

26' 10"	261.3/4
	260.6/7
	993.9
	24.0

make sure ASG up so no liquid acetone inside

@ 20H35 ⇒ shake pump/tubings to see where liquid acetone might be
↳ leave overnight ⇒ cannot see apart

foam inside pump tubing

& from pumping noise

⇒ next time when better idea of GRA X at that T acetone
start w/ less enriched soln

bad feelings about it but leave it overnight

08.01.04

Take from Bristol bottle
the Conc° of the acetone used
for the Bristol run

$$\%_F = 1 - \frac{m_{GRA_i}}{m_{GRA_F}} (1 - \%_i)$$

Run	Initial acetone content (wt%)	GRA m_i (g)	GRA m_f (g)	P (mbar)	T (°C)	$\%_F$ acetone
P1	61.18 (163.1/266.6) cups P1/P2	83.2	39.9	189.0	24.8	19.05% cup P3
P2		100.4	57.(4)/5	241.2	24.8	32.21% cup P4.
P3	40.13 (129.1/321.7) cups P5/P6/P7	87.0	—	(Flask imploded)		—
P4		99.0	71.1	169.2	24.8	16.64% cup P8
P5	39.06 (60.5/154.9) cups P9/P10	84.8	82.2	281.5	24.8	37.59% cups P11/P12/P13

At 24.8°C, acetone boiling pr ~ 315 mbar

a bit oversaturated
exp. was short, probably too short (<7 hrs)

P6	33.875 (54.2/160) cups P14	67.6	49.7	123.7	24.8	10.06% no cup
P7		57.1	48.1	207.4	24.8	21.50% cups 15/16
P8	57.01 (147.2/258.3) cups P17/18	102.5	98.9	293.6	24.9	55.45% cups 19/20/21
P9		88.3	84.6	285.1	24.9	55.13% cups P22/23/24
08.01.04 P10	62.79 (178.7/284.6) cups P25/P26	107.4	78.6	261.6	24.7	49.6% cups P29/P30/P
P11		98.4	50.3	231.7	24.7	27.01%

09/01/04

Bad surprise = flask F1 (run P3) has blown up
This morning

Hotte & eqpt completely splashed w/ GRA and covered
↳ photos

It's a mess !!!

10H57
1002.0
1000.0
1001.0
23.8°C

clean flask F2 from projection; once clean,
m_{F_2} = 412.5 at 12H24

cups out of oven at 15H44

1003
1001
1002

"nettoyage hotte" for hours : only acetone can removed the splashed GRA inside the fume cabinet. Long and exhausting task which will take several hours (a few days).

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Pesée à 16 H 05

Cup	m (g)
P1 (26)	14.4168
P2 (27)	16.7938
P3	17.0719
P4	40.7199
P5	18.8936
P6	30.3117
P7	29.2430

Back in oven @ 16H14

At 21H45, flask F2 = 412.7178 g

Flask F2 is still OK to be used for a run.

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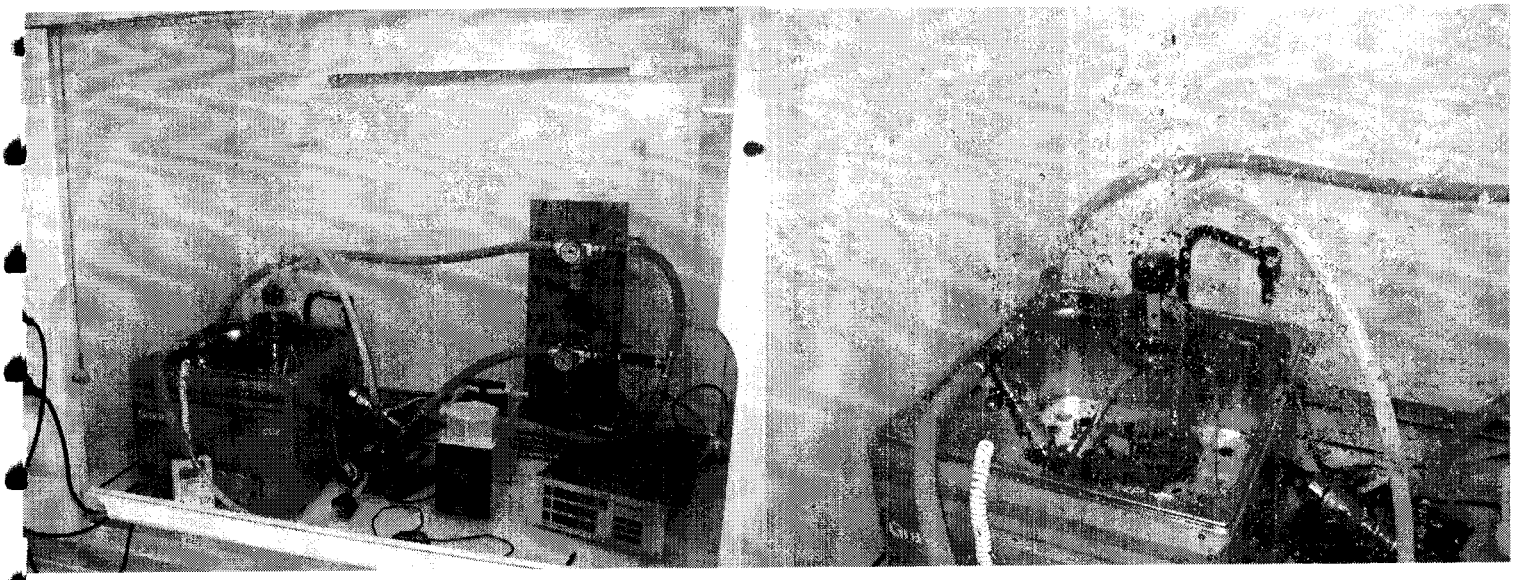

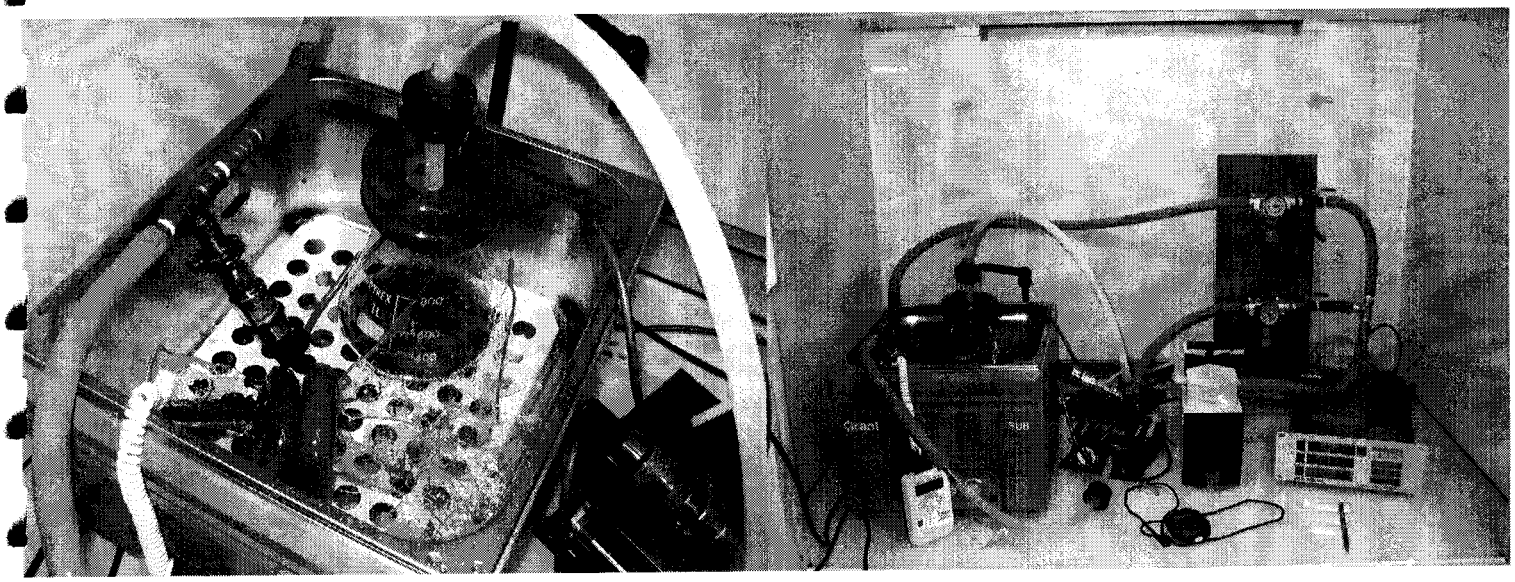

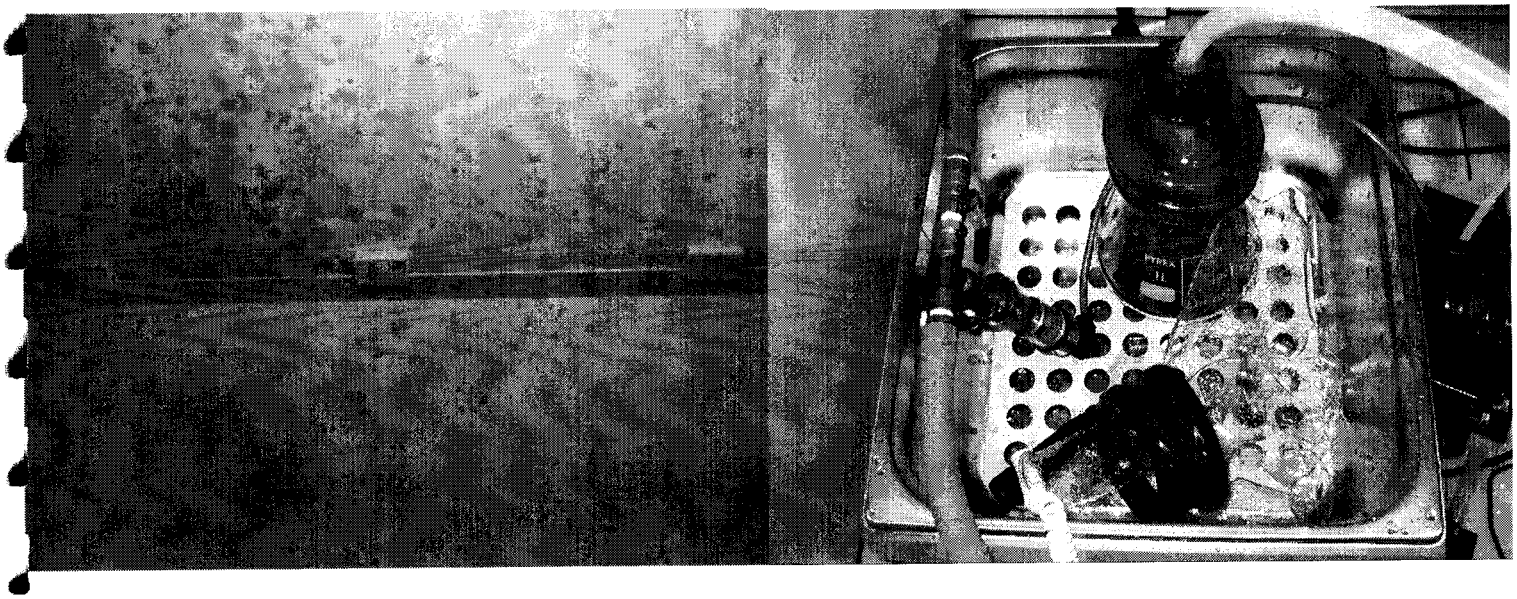

Flask F1 has blown up overnight.
Flask F2 is OK.
Fume cabinet and eqpt splashed with GRA.

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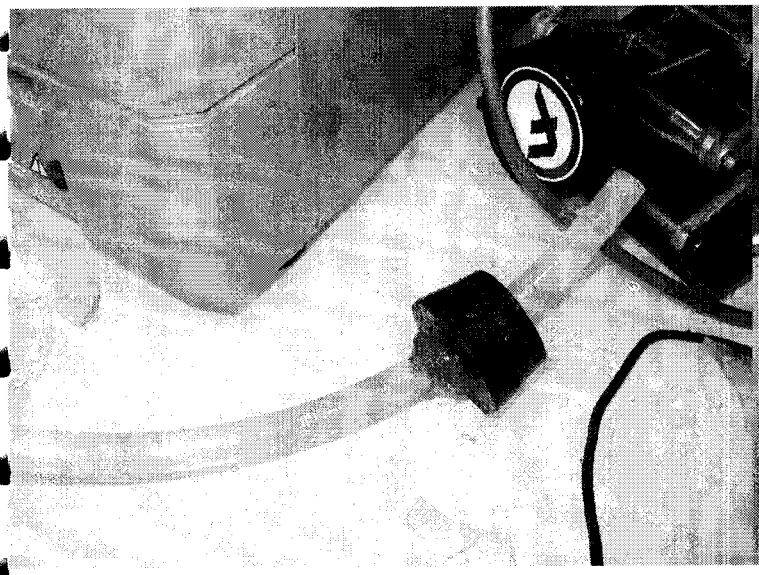
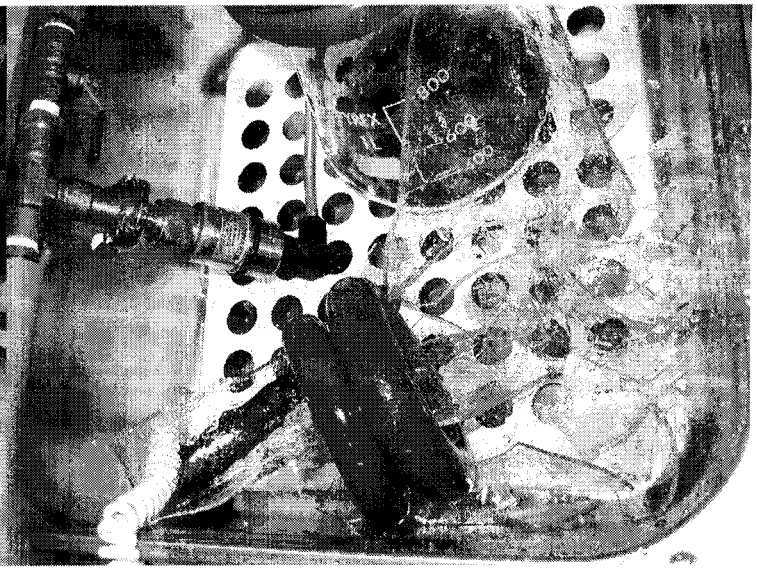
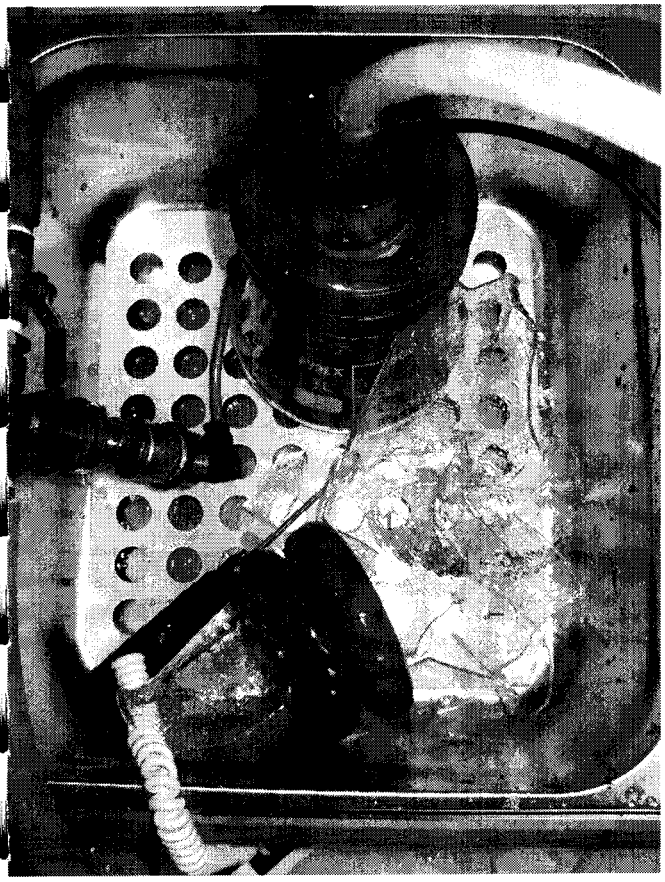
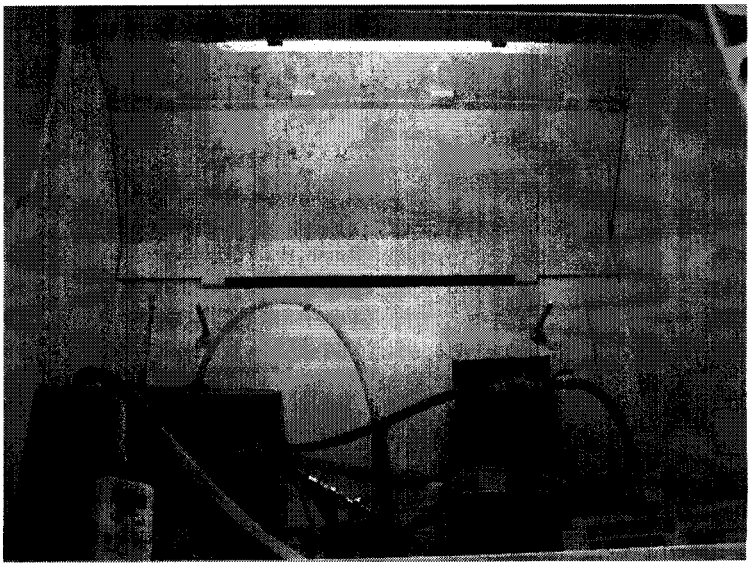
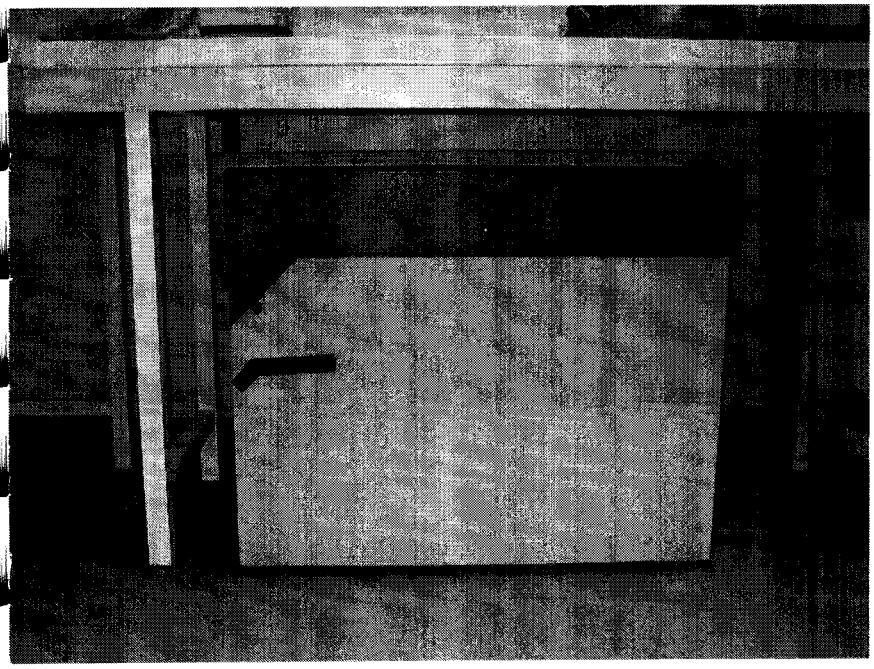
09.01.04











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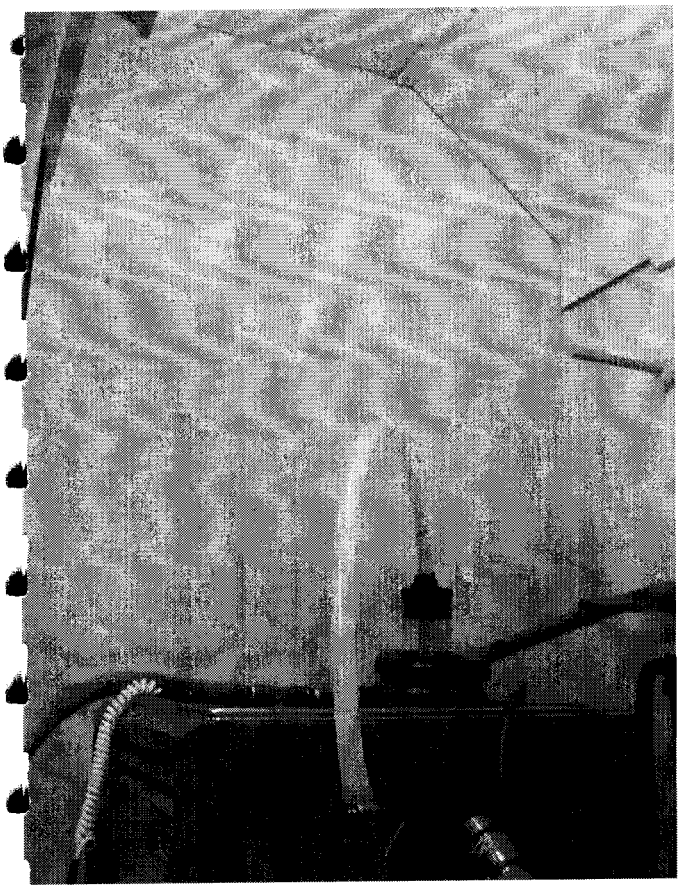

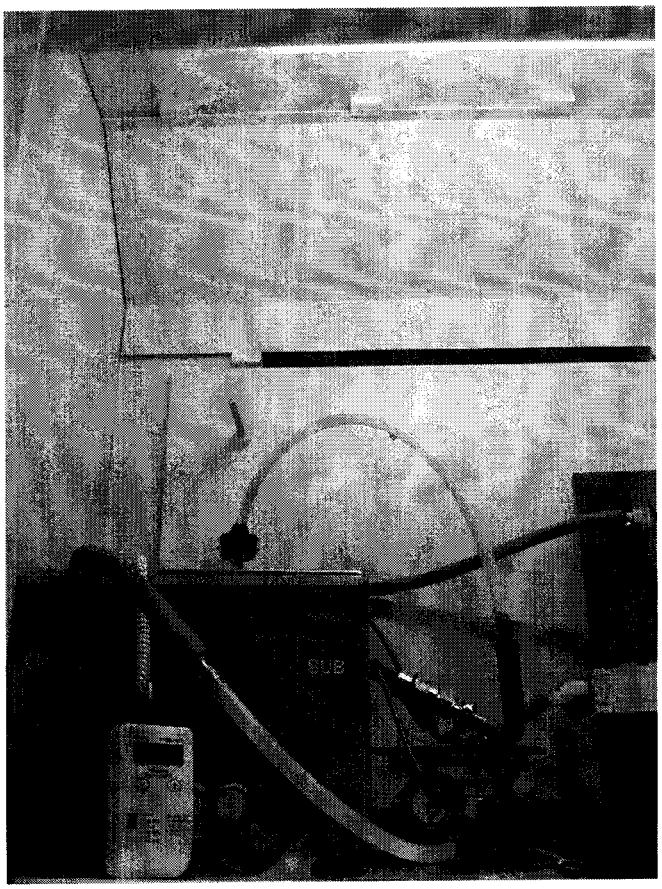

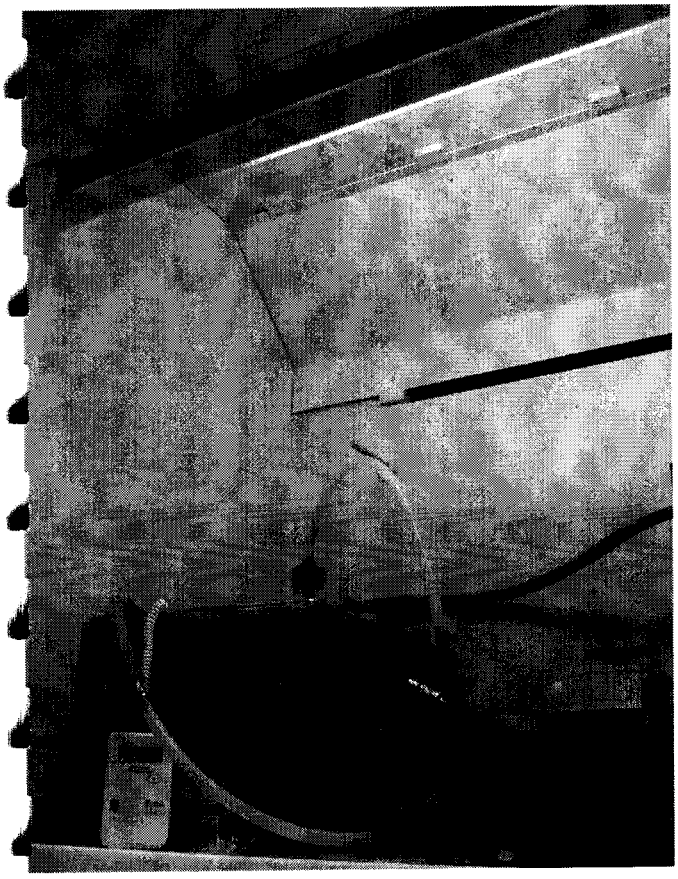

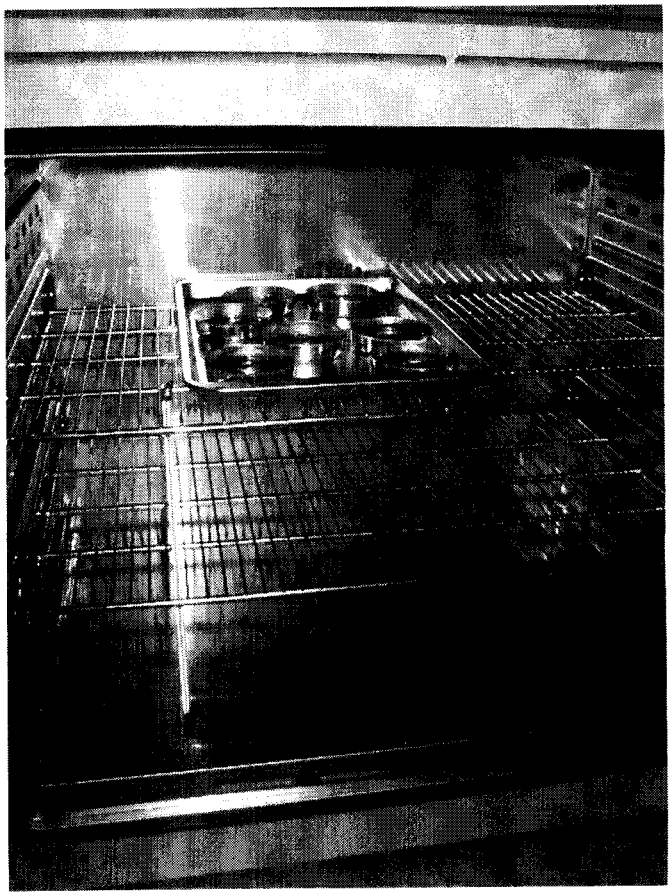

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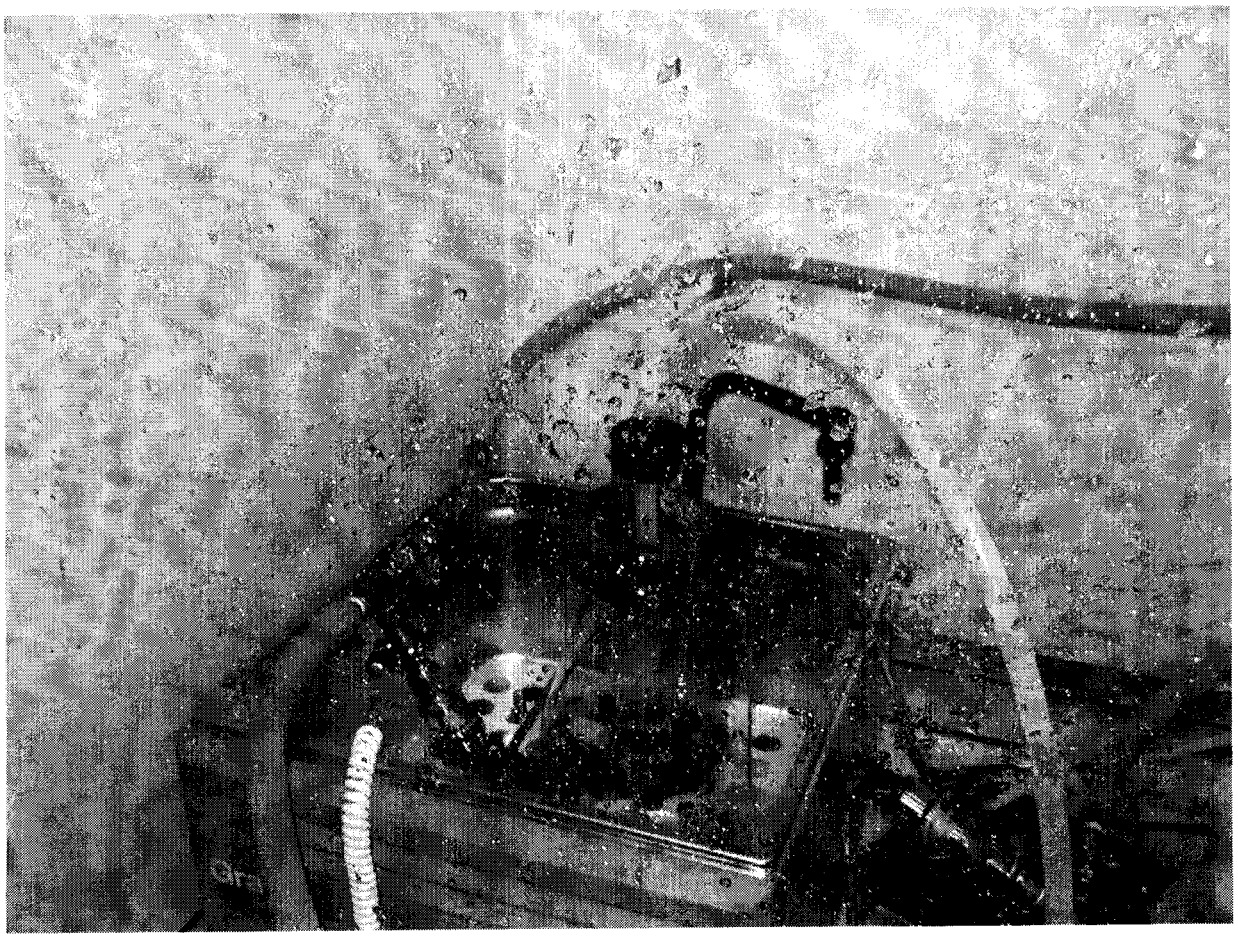

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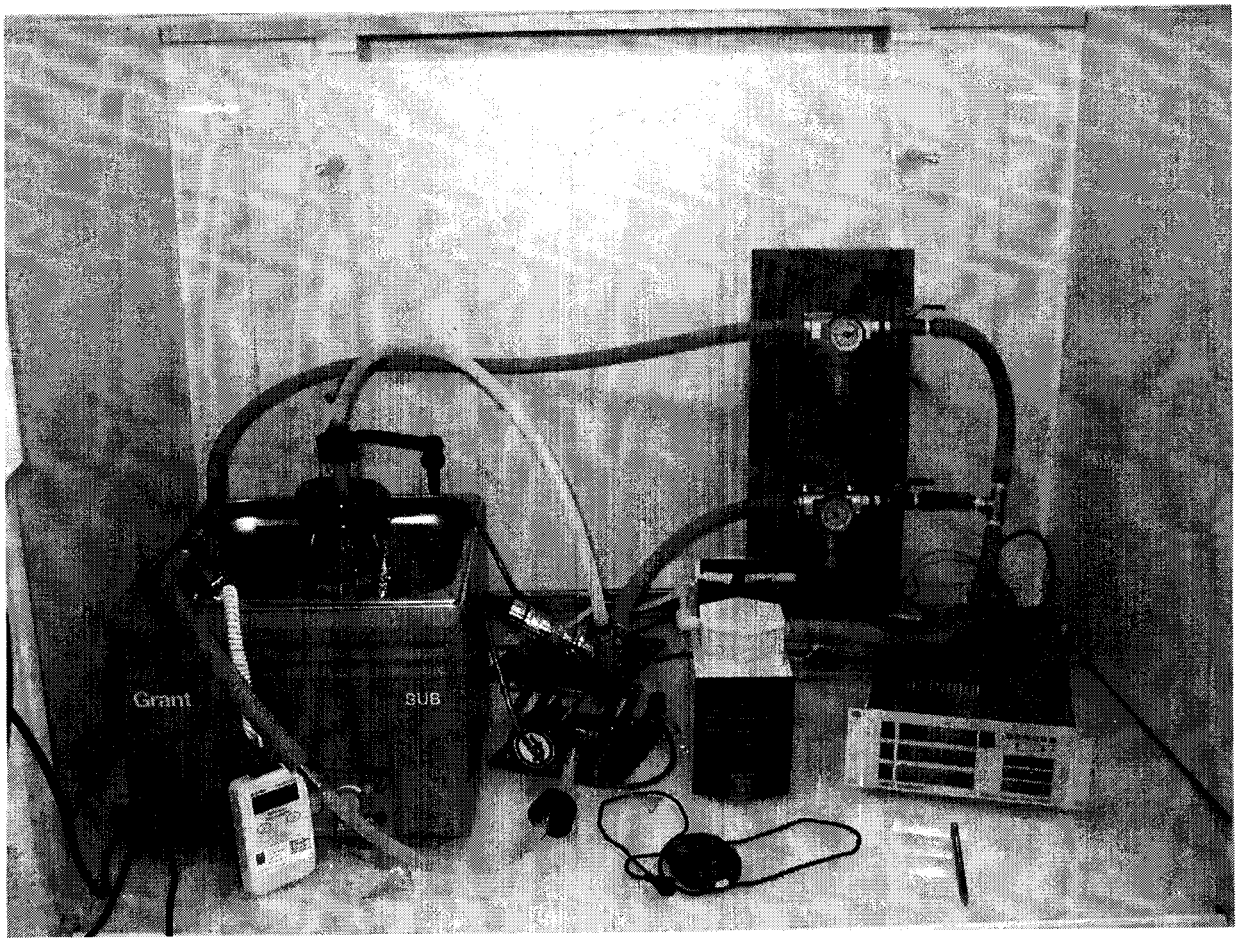

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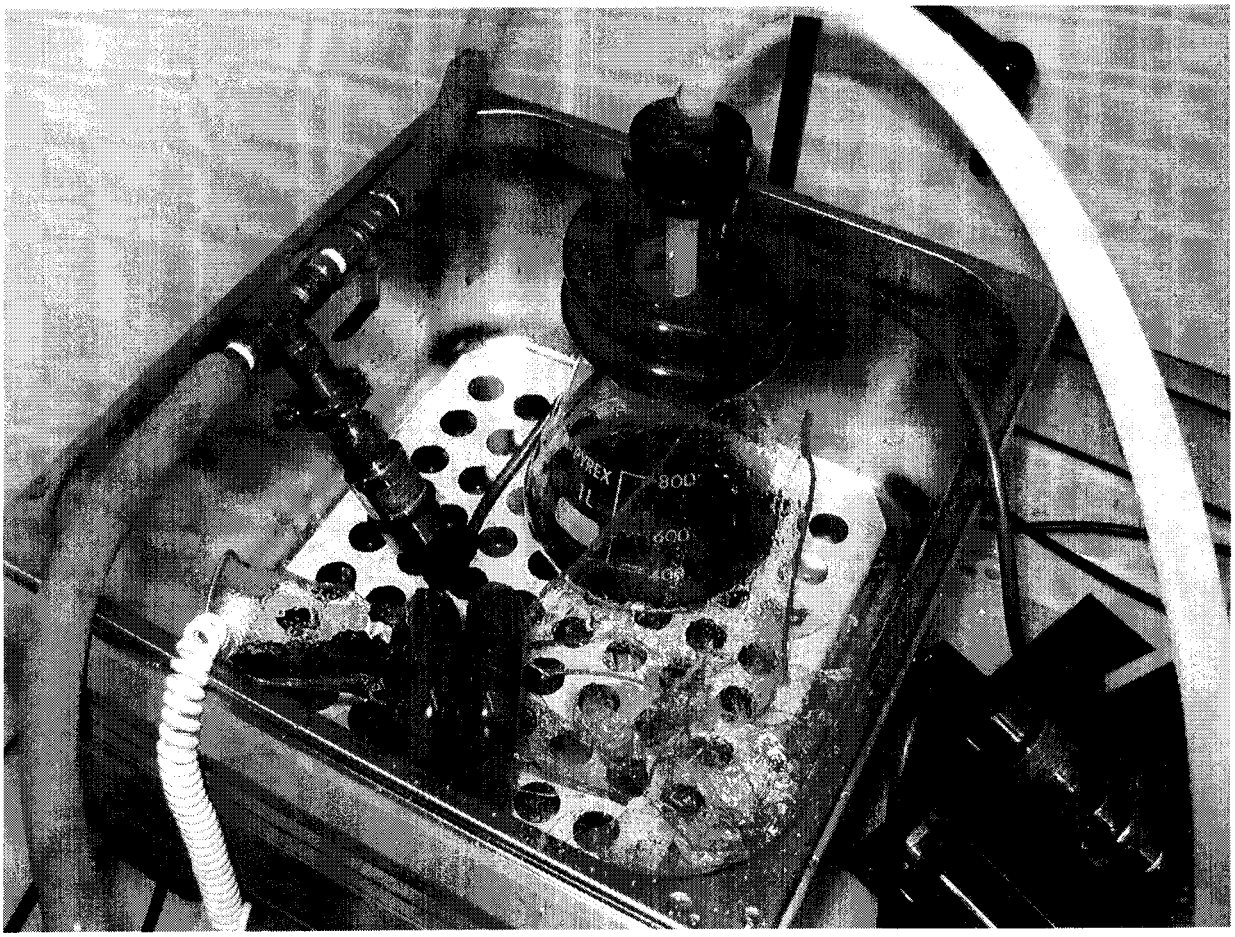
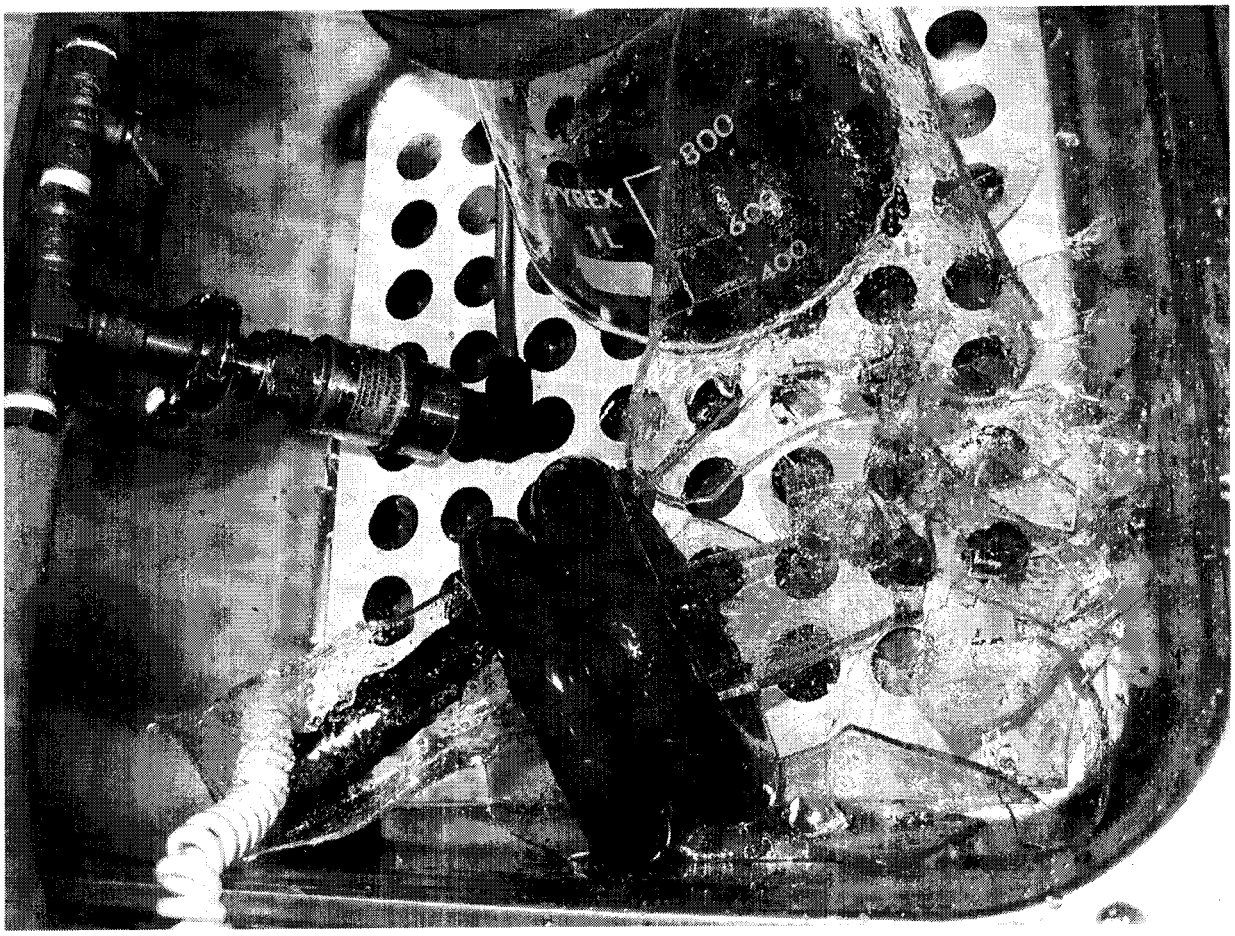


09.01.04

09.01.04



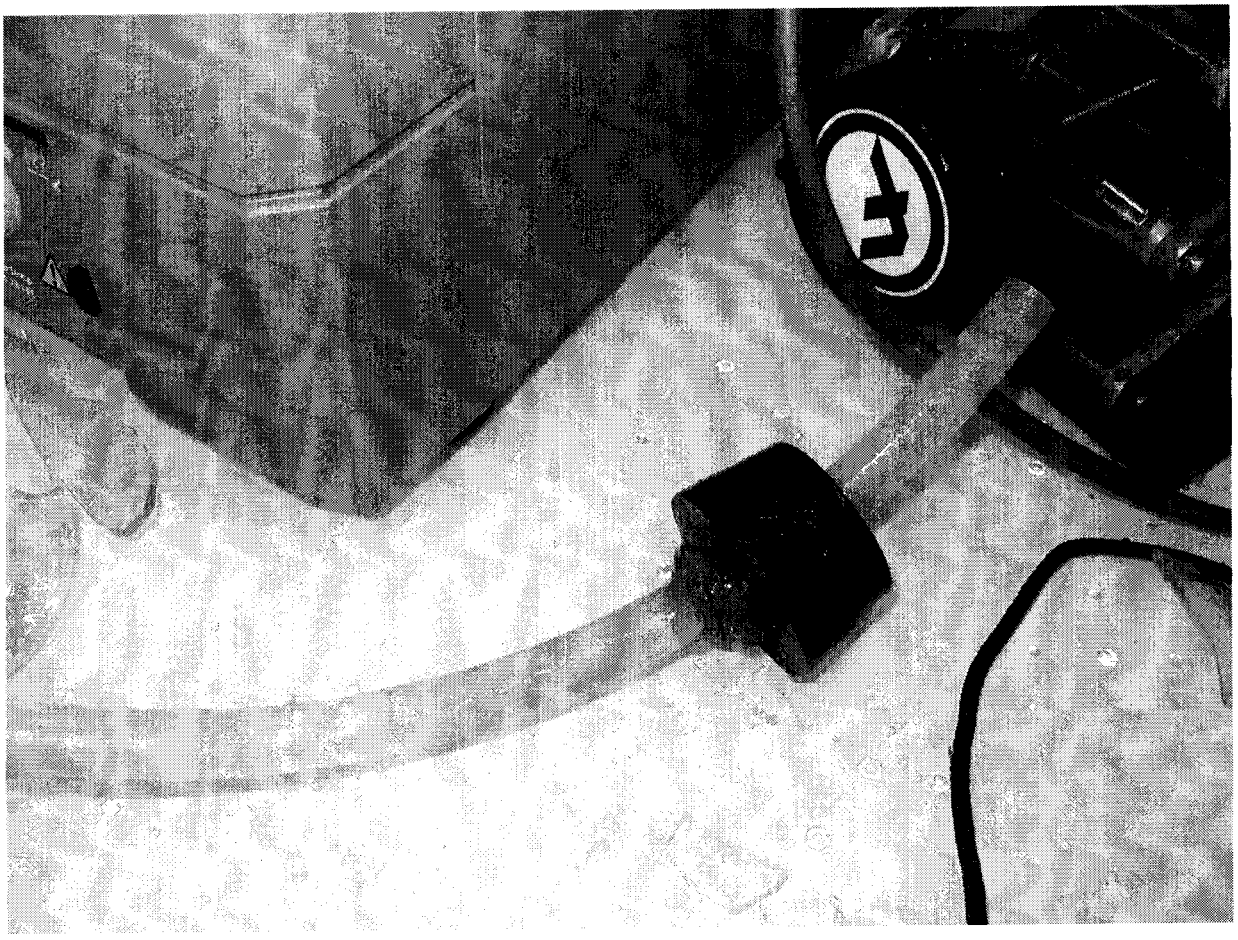
09.01.04

Flask F1 has imploded rather than blown up. It didn't bear the pressure difference between the room ($P_{atm} \sim 994$ to 1002 mbar overnight) and inside the flask ($P_1 < 260$ mbar overnight).

This flask was purchased from the Bristol Faculty Store. Its quality / strength might not be as good as the one of the flasks I order from VWR.

09.01.04

09.01.04



09.01.04

09.01.04

Consumables purchased from the UPMC faculty store

Total € 55.67 (net)

09.01.04

Le 09.01.2004 C142 8

~~Ref. 1263~~
~~Acetone pour analyse 2.5 l x 3~~

Ref. 414
2 rouleaux de papier essuyage "ECO"
€ 7.73 x 2.

Ref. 2251
1 lot de 4 savon p/antiseptie rapide et fréquente
€ 24.16

Ref. 562
tuyau pour vide 8/22 3 m
€ 3.83 x 3

Ref. 2103
fiole Erlen de 1 l x 1
€ 4.56

163

09.01.04

le 09.01.2004 C 147 9

Ref. 1147

Acetone pour analyses 5 l X1

€ 13,83

10/01/04

(1)

Cups out of oven @ 18H40

Weighing @ 19H05

Cup	m(g)
P1 (26)	14.3528
P2 (27)	16.7058
P3	16.8469
P4	40.0779
P5	18.6519
P6	29.8534
P7	28.8252

Back in oven @ 19H08

19H24

1010
1008
1009
23.415

10.01.04

19H31

23.7/8
1010
1008
1009

F2 = 412.3g

19H37

23.5/6
1010
1008
1009

Restart run P4 with flask F2

pressure inside flask F2

P_1 (mbar)	Time
830	4.64
755	20.14
709.8	35.64
670.0	48.83
650.5	55.27
618.0	1'07.20
580.5	1'17.08
552.5	1'30.61
469.0	1'45.17
410.0	1'58.93

P_1 (mbar)	Time
389.5	2'07.87
344.3	2'20.43
318.0	2'29.58
280.6	2'43.43
249.4	2'54.02
220.5	3'12.93
202.7	3'31.73
189.6	6'06.80

↳ ↗ 201.7 when stoned (a few sec)

10/01.04

10/01/04
(2)

22.8 °C
195.5 mbar
1008 mbar
1009 mbar
7' 09.33

7'50"55	192.9	1008	1009	23.0
8' 17.64	191.5	–	–	–
10'16'08	189.0	–	–	–
13'42'20"	188.1	–	–	

~ 17' stirred from 188.1 ↗ 193.4 when stirred

191.1 – 191.6
1008
1009
18'30"08

190.2 → 190.6
1008
1009
23'37"27

~ 25' try to lower P : stuck @ ~ 190 mbar due to degassing
↗ slightly (up to ~ 200) then ↓ goes as down as 182 (∴) decide to ↗ again

188.9
1008
1009
23
28'52.17

10.01.04

31'/03"02 | 188.0 → 188.6
1008
1009

↗ P

stined => P ~~[illegible]~~ 10.01.04
↗ K :)

42'35" | 193.4 → 193.8
1008
1009
23.0/22.9 → toilet retire

toilet remis @ 47'10"

47'20" | 193.2 → 193.6
1007
1009
23.1/23.0

50'55" 193.1 → 193.5 1002 1009 23.0/23.1
↗ P

56'00" 193.3 → 193.9 1007 1009 23.0

10.01.04 @ 20H35 leave overnight

11.01.04
①

14H 18'00

209.3 → 209.7 at 9 HSS
999.0/1
1000
23.4

started P ↑ 211.2, no ∴

14H 25'15"

212.0 → 212.4
999.0
1000
23.1

⇒ decide to ↓P

14H 25'50" 205.1 → 205.9 — —

14H 26'20" 197.7 — — —

14H 26'50" 191.1 → 194.3 — — 23.1/2

started no ∴

P cannot be ↓ further & also keep fluctuating. why??? is the ASG still OK??
sample doesn't seem to dose anymore
could ASG have been affected by blow 10.01.04
⇒ change ASG to check decide to

No difference observed with a different ASG.

Look for other causes ⟹ to better investigate the problem, decide to stop the run and check the whole setup.

11.01.04

11.01.04
(2)

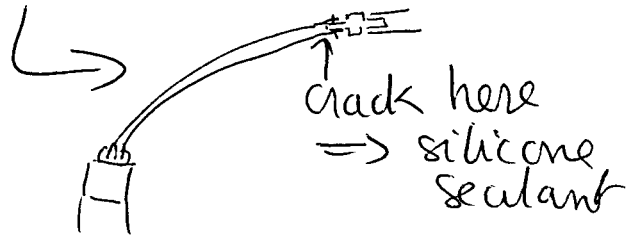
14H29'30' 190.6 → 193.4 999.0 1000.0 23.1/0

pump stopped at 14 H 29 / 41' (time recorded by stopwatch)

⇒ actually w/ valve close P ↑ fast

⇔ leak !!!

somewhere



pump on 14H31'40 P at 905 mbar when start repumping

14H31' 50" 916
14H31' 55" 315
14H32' 00" 220
05" 164
PI → 80 mbar

Problem is sorted out (will have to replace the tubing through)

14H 32' 35" 160
14H 33' 10" 201.3

11.01.04

14H 33' 20" 187.7

14H 33' 30" 176 ⟹ one big bubbles stirred ⟹ boiling ⟹ foam

23.2°C
999.1
1000.0

14H 34' 50" 202.1
23.3

14H 35' 25" 198.0 ↓
999.0
1000
23.3

14H 37' 20"

191.213
999.1
1000
23.1

stirred ⟹ P ↗ 205.0, still fluid
recheck sample & remove :0

11.01.04

11.01.04
(3)

14 H 40' 10"

195.0 / 194.9
999.1
1000
23.1

still w/ ⚬⚬

Try to ↓ P

14 H 40' 50"

195.4/5
—
—
23.0/1

doesn't ↓ due to leak
add more siliconesealant

14 H' 43' 10"

168.1/0
999.0
1000
23.1

stirred ⇒ ⚬⚬ nucleating everywhere rapidly

& P ↗ 176.8

14 H 44' 45"

23.2/1
166.7/8
999
1000

14 H 45' 25"

166.3/2
23.1
999.0/1
1000

degassing gently everywhere
leave it alone for a while

11.01.04

14H55'30"
@ 10H34

163.4
999.0
1000
22.8

switch off extract
to allow
T to ↗

15H00'00"

162.3
999.0/1
1000.0
23.1/2

stirred ⇒ P ↗ 174.5
& ∞ more
vigourous

15H00'55"

171.0
23.2/3

999.1/0 1000

15'01'30"

23.3
165.2
999.0
1000.0

15'02'00"

23.3
999.0
1000.0

162.4/5

11.01.04

11.01.04 (4)

15H02'30" | 162.0/1
999.0
1000.0
23.3

15H03'30" 161.7 - - -

15H05'40" 162.6 999.0/1 1000.0 23.3/4

15H10'40" 161.2/1 - - 23.2

15H52'35" | 158.7/6
998./9 / 999.0
1000
24.1

stirred => ∴ & P ↗ 168.8

15H53'40" 167.1 - - 24.3

15H07'15" | 158.1/2
998. 9/8
1000.0
24.8

stirred => P↗ 165.4 & ∴

T ↗ 24.9 restart extrac 11.01.04

176

11 H 47

16 H 09' 00"

162.1/2
998.9 / 999.0
1000.0
24.9 / 25.0

16 H 15' 00"

157.8
999.0
1000.0
23.8/7 → stop extrac°
effective 16H16'55"

16 H 17' 00"

23.9/8
999.0
1000.0
159.4/5

16 H 51' 20"

157.6
999.5
1000.0
25.1 → restart extrac°
(effective @ 16H51' 15")

Started P ↗ 167.5 & ⁝⁝

16 H 53' 30"

163.2
999.4
1000
24.9
& ↓

11.01.04

11.01.04

(5)

16H55'50" | 158.0/157.9
999.4
1000
23.8

↳ stop extra ? (effectively stop @ 16H57 47

16H57'20" | 157.2/3
999.4/5
1000
23.8

17H22'05" | 157.0/1 ↗ 172 ↘
999.5
1000
25.1/2

17H47'30" | 25.7/8 → restart "hotte"
157.2 @ 13H26 (17H49'30") ~ 2 min after
999.9/8
1001

stirred ⇒ ∘° & P ↗

11.01.04

17 H 48'00"

161.7
999.8
1001.0
25.2

18 H 20'30"

157.1
999.8
1001.0
23.4

↗ 168.6 when stirred
+ ∞

moved thermometer

18 H 23'00"

164.6
999.8
1000.0 /1001.0
23.7

18 H 27'40"

23.1
157.7
999.8
1000.0

18 H 38'30"

156.8 ↗ 163.5 when stirred + a few ∞
23.6/7
999.8
1000.0

↳ another bottle

18 H 40'45"

163.4/5
999.7/8
1000.0
23.5/6

18 H 48'00"

23.9
156.7/6
999.6
1000

↳ stirred

11.01.04

11.01.04
(6)

19 H 05'30"

156.8
999.9/1000.0
1001.0
24.4

→ stirred
→ P ↗ 162.7
& ∞

19H 15' 45"

156.9 999.9/1000.0 1001.0 24.6

→ stirred
P ↗ 165.0

At 15H20

19 H 41'35"

156.9
999.7
1000.0
25.0

P ↗ 165.8
T 25.0/1
↳ hotter on

Cleaning

19 H 45'50"

158.1/0
999.7
1000.0

24.5°C

11.01.04

15H31	19H53'10"	156.8/9	P ↗ 166.8 when stirred
		999.4	
		1000.0	
		23.8/9	→ stop bottle

Cleaning !!

20H 19'30"	157.6/7
	1000.0
@ 15H59	1001.0
	25.2

→ restart bottle

→ stirred → P ↗ 166.4 + ∴

20H 25'30"	159.3/4	20H 28'30"	159.1
	1000.0		1000.0
	1001.0		1001.0
	24.6		24.2

↳ stirred still bubbles P ↗ 164.0

11.01.04

11.01.04

7

cups out of oven @ 16H05

20H39'30"

159.5
1000.0
1001.0
23.617

timed

P ↗ 164.5

20H47'10"

160.2
1000.0
1001.0
23.6 ⟹ switch off

Weighing cup @ 16H28

cup	m(g)
P1 (26)	14.3204
P2 (27)	16.6617
P3	16.7659
P4	39.8204
P5	18.5585
P6	29.6837
P7	28.6518

Back in oven @ 16H31 @ 94°C

11.01.04

check
whitman.edu/geology/winter
↳ course on continental volcanism

16H32 20H53'20" | 160.2
1000.0
1001.0
24.4 ⟹ ouverture baie

stoned P ↑ 166.0 + a few ∴ (but less than B4)

At 17H00 after 21H21'30" | 160.1
1000.0
1001.0
25.0/1

stoned ⟹ P ↑ 163.7
+ a few ∴

not instantaneously but as ∴ burst
P max reached ~ 10 s
after stoning stopped

11.01.04

11.01.04

(8)

21H25'40"
162.0
1000.0
1001.0
25.0

21H35'00"
161.1
1000.0
1001.0
25.0/1

21H39'30"
160.9/161.0
1000.0
1001.0
25.1
hotte back on

22H01'00"
161.2/1
1000.0
1001.0/1002.0
23.1 => stop hotte

stirred => a few :00 P↗ 163.5

22H17'00"
162.2
1000.0
1001.0
24.3/4

~~A~~ 18H07
24.6/7
164.3
1000.0
1001.0

stirred =>
P↗ 165.9 no :00

11.01.04

22 H 35' 45"

164.718
1000.0
1001.0
24.7

=> open Nicole's office window to regulate room temperature

22 H 44' 00"

164.617
1000.0
1001.0
24.413

=> close bay window of the fume cabinet

22 H 50' 15"

164,516
1000.0
1001.0
24.8

stirred => pg 165.9
@ 22 H 53' 30

=> new sample @ 18 H 33

F	567.9 g
M	34.8 g
S	279.5 g
F + M	602.6 g
+ GRA	677.3 g
+ acetone	707.6 g
+ S	987.1 g done @ 18 H 36

11.01.04

stirred manually => then w/ SM1

11.01.04
9

~~M~~ 18H38

23H00'30"

165.6A
1000.0
1001.0
24.5

=> close a bit bay window

<u>sample prep</u>

prosec ? on flask walls

18H43 707.2 g = $m_{Flask + GRA}$

add acetone to clean walls

↳ 740.2 g

+ stopper 1019.6

11.01.04

186

At 18H46

23H07'40"

165.4
999.9 / 1000.0
1001.0
24.7/6

At 18H55

23H17'00"

165.4/5
1000.0
1001.0
24.1 ⇒ close Nicole's office window

11.01.04

11.01.04
(10)

18H55
23H17'00"

165.415
1000.0
1001.0
24.1

⇒ close Nicole's office window

19H10
23H32'00"

166.112
999.5/6
1000.0
24.6/7

Weighing new GRA batch
1019.6 g w/ stopper
739.9 g
add
+ GR 759.6
+ Stopper 1039.2

19H12

not all dissolved @ 19H20

adjust a bad P
23H39'40"

166.1/0
999.5/6
1000.0
24.6/7

⇒

23H41'20"

165.9
999.5/6
1000.0
24.5/6

11.01.04

ouvre bay
v 23H45' stirred → P↗ 168.7

At 19H30

23H52'40" | 168.7
999.5
1000.0
24.5/6

At 19H32 sample prep | 1038.9
759.3

not all GK dissolved

At 19H38

24H00'00" | 168.7 adjust a bit
999.1/0
1000.0
24.6 → close bag

24H2'30" | 168.9
999.2/3
1000.0
24.8/9

11.01.04

11/01/04
(11)

24 H 4' 40"

167.3
999.2
1000.0
24.7/8

24 H 8' 00"

167.4/5
999.3/4
1000.0
24.7/8

24 H 19' 00"

168.6/7
998.7/8
999.8/9
24.6 → close bay & adjust P

sample pop:
@ 19 H 58 allm st all dissoved (except one small bit)
1038.8
758.9
1038.6

24 A 21' 00"

168.2
998.9/999.0
1000.0
24.7

11. 01. 04

At 20H03

168.3/4
998.8
999.9 /1000
24.6

24H24'30"

24H 33' 40"

169.2
999.3/4
1000.0
24.8

adjust P a tad

24H 44' 00"

170.5
999.1/2
1000
24.8

stop
@ 20H26

pump very hot

At 20H24

1001.0
999.1/2
1000.0
24.8

$m_{GRA} = 395.2 - 324.1 = 71.1\ g$

At 20H25

Flask = 395.2 g

11.01.04

11/01/04 (12)

~~At~~ 20H26	empty cup m (g)	m (g) cup + GRA
cups		
P8	2.5207	27.9067

↳ in oven @ 20H28

sample prep | 758.7g (same as stopper)
| 1038.2g all dissolved

@ 20H30 ⟹ stirring overnight

Pump very hot + P ↑ gently but can't be ↓ ⟹ leak } ⟹ leak in system ⇓ pump has overworked

⇓

leave it to rest for the night

~~At~~ 20H30 | 1001.0
| 998.9
| 1000.0 24.1°C

11.01.04

At 21H03

23.9
1001.0
998.9 / 999.0
1000

pump cooling down

Cup P8 out @ 21H04

At 21H08 $m_{Cup\ P8} = 26.8287$

back in oven @ 21H10

pump warmish but OK

Will only start next run tomorrow morning to allow the pump to rest.

11.01.04

12.01.04

1

995.817
993.6
994.7
23.1°C

At 10 H05

sample mop 1037.5
757.5

1 → 279.9 g
+ GRA 364.7 g

cup	P9	2.5202	at 10H15
		28.3844	
	P10	2.5151	in oven
		31.8162	@ 10H17

cup P8 out @ 10H17

12.01.04

At 10H17 | 995.0/1
992.9/8
994.0
23.9/24.0

Manip @ 280 mbar

Start @ 10H19

P_1 (mbar)	Time
731	3'16
509	8.63
465	5.28
429	35.25
388	45.78
349	51.82
330	1'02.41
291	1'10.59
293.0	1'27.53
291	1'45.00
297.0	1'57.31

:: adjust

12.01.04

2'16.82 | 291.3
992.7/8
993.8
23.6/5

2'44.82 289.7

3'01.34 288.3 stirred P ↗ 298

+ (microbubbles)

3'43.41 | 288.2
992.7/6
993.8
23.5

up P8 @ 10H24 25.1159 g

back in oven @ 10H25

| 286.2/3
992.4/5
993.5
23.1

5'53.09

12.01.04

9'56"25
@ 10H30

286.3/2
992.2
993.3/4
23.3

~~stirred~~ P ↑ 290
no :-
back to 286.1
rapidly

11'15"00

285.7/8
992.1/2
993.3
23.3

1H22'40"

282.2/3
987.2/3
988.4/5
23.7

⟹ stirred

~~11~~ 11H59

1H39'40"

282.5/4
987.4/3
988.5
23.7

⟹ stopped bottle
stirred P reste stable

12.01.04

12.01.04

(3)

1H49'10"

281.9
986.7/8
987.8
23.9/8

New sample

at 12H15

F	567.9
M	34.8
S	279.9
F+M	602.7
+GR	659.6
+ acetone	717.7
+ S	997.6

+ 56.9 GR
+ 56.7 acetone
+ 37.1 GR
- 1.1 acetone
+ 41.8 GR

~ 34.4%

At 12H17

1H57'40"

281.7/6
986.4/3
987.5/4
24.0

12.01.04

cups P9 & P10 out of oven @ 12 H18

Pesée à 12 H22

cup	m(g)
P9	20.1895 g
P10	22.7075 g

Back in oven @ 12 H24.

~~th~~ 12H24	281.5
	986.1/2
2H04'30"	987.2/3
	24.2

~~th~~ 4H55	
4H35'45"	280.4.5
	984.6/7
	985.7/8
	27.0/1

too hot
⟹ hotter on
(pump is hot)
+ lower P later on

4H42'50"	280.9
	985.1/2
	986.3
	26.6

12.01.04

12.01.04
(4)

Sample prep 996.2
716.3

15H01 + GR 753.4 g
+ S 1033.3 → stirring

cups P8/P9/P10 out of oven @ 15H05

4H47'00" | 281.1/0
985.3/4
986.4/5
26.4/5

4H51'00" | 281.4
985.4/5
986.6
26.3

5H32'45" | 282.0/1
987.7
988.8/9
25.0

12.01.04

16H01

5H42'00"

282.0/283.0
988.2/3
989.4
25.0

stirred P ↗ 296.0

weighing @ 16H04

cups	m(g)
P8	24.9848
P9	19.8380
P10	22.3252

Back in oven @ 16H05

5H45'20"

284.5/6
988.5/6
989.6/7
24.9/25.0

6H15'50"

285.4/5
990.4
991.5
24.5/6

stop heat & ↓ P

12.01.04

12.01.04

(5)

16 H 38

sample prep | 1032.2 g
752.3 g

+ GR 764.1 → stirring

6 H 22' 40" | 283.3/4
990.9
991.9 / 992.0
24.6

6 H 28' 30" | 24.6/7
282.6/5
991.2/3
992.3

6 H 34' 00" | 282.0
991.5
992.6
24.7

stirred P 1 283.1

6 H 38' 30" | 281.2
991.7
992.8
24.8

at 16 H 58

12.01.04

202

Sample prep | 1043.9
763.9 not all dissolved

6H40'00 | 281.0
991.7
992.8
24.8

↳ stop @ 6H40'10'
at 17H00

994.2/3
991.7/8
992.8/8

$\boxed{\text{Flask 1} = 362.1\ g}$

↳ cups P11/P12/P13

12.01.04

12.01.04

(6)

cup	m (g)	+ GRA
P11	2.5228	21.4081 (71)
P12	2.5234	19.1988
P13	2.5232	42.7502

↳ in oven @ 17H14

At 17H45 pump cold

996.213
993.9 / 994.0
995.0
24.3°C

Sample prep 17H50

1043.6
763.7 not all dissolved

12.01.04

% acetone pour manip P5 (sample made on 11.01.04)

74.7 g GR
30.3 g acetone
− 0.4 g acetone
+ 33 g acetone
− 0.3 g acetone
+ 19.7 g GR
− 2.1 g acetone

⇒ 94.4 g GR
60.5 g acetone

⇒ 39.06 %

Cups P11/P12/P13 out of oven @ 17H27

New sample prep (1043.2 g
763.3 g) a few bits not dissolved yet

Weighing cups @ 18H37

P11 15.8851 g
P12 14.2514 g
P13 31.7975 g

Back in oven @ 18H39

12.01.04

2.01.04
(7)

% acetone pour manip P6

56.9 g	GR	58.1 g	acetone
37.1 g		−1.4 g	
11.8 g		−1.1	~ 54.2
105.8		1.4	

~ % 33.875

140 mbar

ou (215 / 220) mbar

14 19H45
load flasks w/ GRA :

Flask 1 315.8g
erlen
↳ 372.9 g

Flask 2 267.6g
↳ 335.2 g

sample prop
1042.8
762.7

↳ ou P14 2.5213g
@ 19H56 20.8065
in oven @ 19H58

12.01.04

19H58

999.6
997.5/4
998.4
23.6/7

20H05

1000.0/999.9
997.7
998.7/8
24.4

→ on

P_1 (mbar)	Time
710	4'38
506	12'67
411	24.54

43"45

373
869

1'02.23

322
757
24.2
998.8

1'17.38

301.2
757.6
998.8

1'35.38

282
680

1'51.54

276
572

2'08.29

272.6
493.6

2'30.89

270.2
376.8

12.01.04

12.01.04

(8)

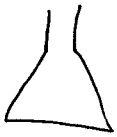
3'08.17
271.3
269.0
998.8
24.0/23.9

3'50.67
208 → a few ∴
268.3

4'25.04
173.0
268.1

P1

P2



4'51.10
171.9
232.3
998.8
23.9/24.0

5'52.07
145.9 stirred ⇒ boiling P↗163.0
231.7 → stirred then ↓
↳ P↗234.0 no ∴

7'26.76
148.0
231.7
998.7/8 —

12.01.04

208

7'55.01 | 143.1
231.6 ⟹ Lower P

8'20.45 | 140.3/4
225, 4/5

8'46.26 | 139.1/2
220.0/1
– 23.8

9'27.32 | 138.9
220.1
998.8 23.8

10'04.13 | 138.7
219.8/9 ↗ a bit Ps
998.7 23.8

11'18'25 | 139.7/6
220.7/8 stuned ⟹ P ↗ 223
998.8/9 23.8

11'50.95 | 139.0
220.9
998.8/9
23.8

12.01.04

12.01.04

9

stirred flask

P ↑ from 139 to 167 mbar boiling

13/08.42	144.0
	221.2
	998.9/999.0
	23.6

All caps removed from oven @ 20 H 18

4/05.83	139.3
	221.0/220.9
	998.9
	23.5/6

25/50.01	138.8/9 ⇒ ↑ a bit P, stirred P ↑ 163
	220.9/8
	998.9
	23.1

28/06.19	139.4/5 → ↑ to ~160 when stirred boiling
	220.8/9 → ↑ by ~ 2 mbar only when stirred
	998.6
	23.2

12.01.04

210 Pesées @ 20H24

Cup	m (g)
P1 (26)	14.2883
P2 (27)	16.6218
P3	16.7029
P4	39.6090
P5	18.4895
P6	29.5291
P7	28.5104
P8	24.8886
P9	19.5866
P10	22.0198
P11	15.6329
P12	14.0407
P13	31.1357
P14	17.0105

Back in oven @ 20H40

35 | 142.35

139.415
220.413
998.415
23.1

@ 20H40

12.01.04

12.01.04

(10)

38'31.07

138.7/8
220.2
998.2/3
23.2

48'45'

138.1/2 ⟹ ↗ 165 when stirred & shaken
220.1
998.2/3
23.0/22.9

@ 20H54

decrease a bit P2

51'15"

138.5
218.1/0
998.2
23.2

55'00"

137.9
218.0
998.3
23.2

59'50"

137.4/5
218.2/3
998.4/3
23.5

21H10

1H05'00

137.3/4
218.1
998.3
23.6

Good stir before closing 21H15

12.01.04

212

XA
21 HIS

1409/30"

23.6
137.3 —> still bubbling gently
218.1/2
998.4

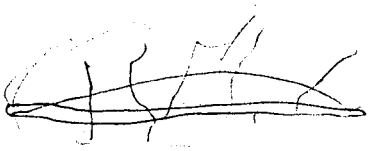
12.01.04

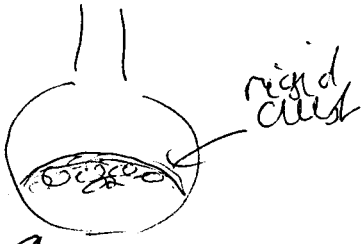
13.01.04 (1)

8H21

12H16'45''

125.9/126.0
207.6/5
987.1/0
23.7





12H29'00''

125.9/126.0
207.8/9 987.3
23.6

GRA forms a soufflé-like dome with an upper rigid crust and viscous interior.

stirred Flask P_2 ↗ 208.5 no 80

⟹ Marc De Raffelis 0688969290 to use and borrow digital camera belonging to sedimentology lab.

12H46'50''

126.8/7
208.4/3 ⟹ ↗ 208.8 when stirred
987.9
23.5

soufflé returned
↳ still viscous

shake
↳ P9.

133.8

13.01.04

12H52

128.3/4
208.6/7
988.1
23.6

@ 8H57

Cups out of oven @ 8'H59

13 H 09'20"

126.4/5
208.6
988.2/3
24.6

Weighing @ 9H38

P1(26)	14.2789
P2(27)	16.6082
P3	16.6840
P4	39.5 397
P5	18.467 9
P6	29.4756
P7	28.4622
P8	24.7062
P9	19.283 3
P10	21.6580
P11	15.1503
P12	13.6361
P13	30.0170
P14	15.6559

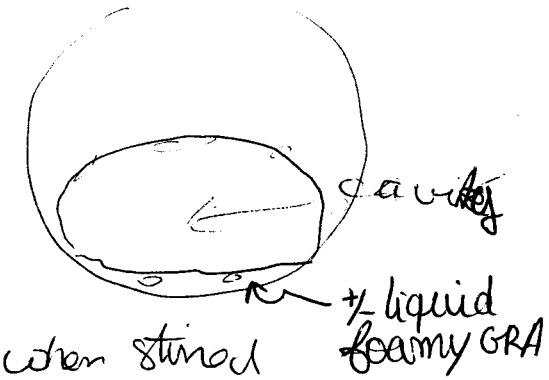
→ done @ 9H45
Back in oven @ 9H46

13.01.04

13.01.04
②

13H41'30"

126.7/8
209.2
988.6/7
24.5/6

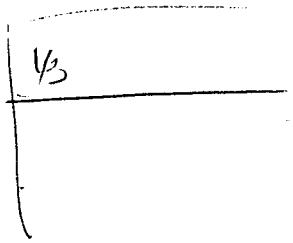


↗ 210.5 when stirred

At 10H11

126.9/127.0
209.2/3
988.9
24.5

baisser un peu bouvitré



At 10H40
14H35'30"

127.0
210.0/1
988.9/989.0
25.0/24.9

big bubble has collapsed

P ↗ 129.8 after moving block
big bubble start to reinflate

(very faint smell of acetone
light
in the room)

13.01.04

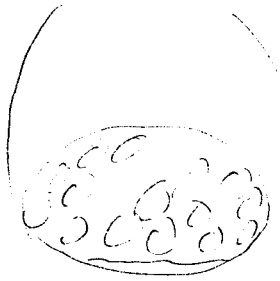
14H42'50''

127.3/2
209.3/4
988.9
24.8/9

dose 1/8

15H04'40''

126.5/6
209.2/3
988.9
24.8/9



15H15'30''

127.0/126.9
209.1
988.8
24.8

→ ↑ 127.6 when moved

bottle bag removed

flask

15H22'30''

127.0/126.9
209.2
988.7/8
24.8/9

15H27'30''

126.9/127.0
209.1/2
988.7/8
24.8

16H06'00''

126.2/3
208.6/5
988.4
24.8/7

13.01.04

13.01.04
(3)

At
12 H 19

16 H 14'20" | 126.1/0
208.4/5
988.1/2
24.9/25.0 → hotter on

16 H 28'00" | 125.7/8
208.6
987.7
24.4/5

hotter stop @ 16 H 30'00" effectively & buy open

16 H 32'40" | 125.8 ⇒ ↗ 126.1
208.1/2 ⇒ ↗ 208.9 when started back in a very few seconds to 208.0/1
987.7
24.6/5

16 H 34'50" | 125.8/9
207.9/8
987.6/5
24.6

16 H 37'30" | 125.9
207.7
987.4/5
24.6/7

13.01.04

17H02'20"	125.5
@ 14H07	207.5
	987.2/3
	24.7

17H05'00"	125.4/5
	207.4/3
	987.1/2
	24.7/8

17H13'00"	125.2/1
	207.4
	987.2/3
	24.8

17H17'00"	125.3/4
	207.5
	987.4
	24.8

17H21'30"	125.2/3 ⟹ P ↗ 125.5 when shaken
	207.4/5 ⟹ P ↗ 208.3
	987.3
	24.9/25.0 → hotter

both back initial to values within few sec

17H24'00"	124.9/125.0
	207.5/4
	987.1/2
	25.0/1

13.01.04

3.01.04
(a)

17H26'30"	125.0 207. 5 987.3 /2 24.9
17H27'30"	125.0/1 207.5/4 987.3/2 24.9/8
17H30'00"	124.8/9 207.4/5 987.3/2 24.8
17H30'50"	124.9/125.0 207.5/4 987.2/3 24.9/8
17H32'40"	124.9 207.4/5 987.2/3 24.8
17H52'00" @ 13H56	124. 8/7 207.3/2 986.8/9 24.4/ 3 => stop hotte @ 13H57

13.01.04

220

17H53'00"

124.9/125.0 ⟹ bubble reformed

207.8

987.0/986.9

24.5

après ouverture bay window

(hotte stopped effectively @ 17H53'40")

P ↗ 208.3

P ↗ 125.2

17H55'00"

125.0/124.9

207.3/4

986.9/987.0

24.5/6

18H01'20"

124.7/6

207.0/1

986.9

24.6

18H15'30"

124.5/6

207.2

987.0

24.6/7

13.01.04

13.01.04
(5)

18H23'00"
124.3/4
207.2
987.0/1
24.7

18H24'00"
124.3/4
207.2
987.0
24.7/8 → 24.8/9 ⇒ open office Nicole's door
+ start ~~air~~conditioning in flume cabinet

18H34'30"
124.3/4 → stable
207.5/4 → ↗208.6
987.2/3
24.4 ⇒ arrêt hotte
@ 14H39

18H36'30"
124.3/4
207.7
987.2/3
24.7/8

18H39'00"
124.4/5
207.6/7
987.3/4
24.7

13.01.04

18 H 52	124. 13 207. 6/7 987. 4/5 24. 8
19 H 12' 30"	124. 1/0 207. 5/6 987. 4 24. 9

→ hôte on (effectively) @ 15 H 18 barafo mn)

19 H 29' 40"	24. 7 124. 1 207. 4 987. 3
19 H 35'	124. 1/0 207. 2 987. 1/2 24. 8
19' 49'	123. 7/6 207. 5/4 987. 1 24. 9

13.01.04

→ arrêt @ 19 H 49' 30" @ 15 H 55

⟹ bulle dégonflée complète

13.01.04

(6) Pesé flask 1 363.9 g

@ 15H56

↳ cup P15 | 2.5739 g = empty P15 cup
| 25.4007 g = cup P15 + GRA

cup P16 | 2.5790 g = empty P16 cup
↳ 9.0667 g = cup P16 + GRA

@ 15H59

↳ in oven @ 16H00

988.712
986.213
987.314

Pesé flask 2 317.3 (19H57'00")

sample rotating

no cup as sample is not retrievable

(m_{Flask2} @ 20H04 = 317.3 g)

13.01.04

224 Cups K/15/16 out of oven @ 16H25

988.8/9
986.4/3
987.5
25.1 (in bottle)

Weighing @ 16H33

Cups P14 = 15.5451
P15 = 23.6669
P16 = 8.3903

↳ back in oven @ 16H35

All Cups out of oven @ 19H33

At 19H54

992.7/8
990.3/4
991.4
24.7

Cups Weighing @ 19H55

Cups	m(g)
P16	8.1367
P15	22.54 77
P14	15.5106
P13	29.7165
P12	13.5323
P11	15.0249
P10	21.5153
P9	19.1647
P8	24.6014
P7	28.4268
P6	29.4397
P5	18.4510
P4	39.484 4
P3	16.6690
P2(27)	16.5968
P1(26)	14.2710

Done @ 20H02
Cups back in oven @ 20H03

13.01.04

15.01.03

Travelled yesterday from Paris to Bristol

Cups out of oven @ 19H53
let cool down to ambient covered with paper sheets.
Weighing from 20H07 @ 27°C

Cup	m (g)
B1	11.8644
B2	20.6751
B3	11.6570
B4	19.4094
B5	21.7895
B6	16.1892
B7	15.5708
B8	13.9611
B9	14.9068
B10	13.1667
B11	15.8452
B12	18.6285
B13	11.5909
B14	6.4324
B15	12.5593
B16	14.8889
B17	18.7917
B18	17.7838
B19	24.0825
B20	7.5403
B21	18.6014
B22	25.0901
B23	26.4469
B24	29.8508
B25	32.0705

Done @ 20H17
Back in oven @ 20H19

15.01.04

<u>Note</u>: When cooling down from ~ 94°C to room temperature, GRA tends to crack. Tiny GRA chips may "jump" out of the cups and be lost, being therefore a source of error. To prevent this to happen, cover the cups with paper sheets as soon as they are taken out of the oven and left to cool down to room temperature.

GRA chips may also get lost during rapid heating from T_{room} to 94°C $\Longrightarrow$ leave the paper sheets on top of the cups during the first ~ 15-20 minutes of heating then remove them.

15.01.04

18.01.04
①

Back in Paris from Bristol yesterday.

At 12H17 24.4°C

F 317.3g = $m_{empty\ flask}$

At 12H20 all cups out of oven (T_{oven} = 94°C, T_{set} = 88°C) and covered w/ paper sheets

(P12 une écaille cassée)

Weighing @ 12H52

cup	m(g)
P1 (26)	14.2125
P2 (27)	16.5191
P3	16.5682
P4	39.1146
P5	18.3364
P6	29.2003
P7	28.1990
P8	24.1676
P9	18.7848
P10	21.0728
P11	14.7174
P12	13.2781
P13	28.9185
P14	15.1942

Cup	m(g)
P15	21.2117
P16	7.8233

Done @ 13H00

Back in oven @ 94°C @ 13H01

18.01.04

228

GRA

Sample prep At 13H02

F	567.9
M	34.8
S	281.0
F+M	602.8
F+M+GR	665.5
+ acetone	814.2
+ S	1095.2

62.7 GR
148.7 acetone
↳ 70%
↳ add GR

↳ stirring @ 13H05

At 13H09

1019.0
1016.0
1017.0
25.3/2

bottom

At 13H46

24.9
1019
1017
1018

all dissolved GR

1094.8
813.7

↳

+GR → 862.8 → stirring

18.01.04

8.01.04
(2)

At 14H25

861.4g all GR dissolved
1019
1016
1017
24.9

111.0g GR
147.2g acetone
↳ ~57%

Plan 2 runs → ~310 mbar (should be < 315 mbar)
→ ~295 mbar

Then ~ 230 mbar

At 14H40

24.9
1019
1016
1017

Same on stopper
1142.2
GRA Flask 861.0g

At 14H44

Flask 1 315.7 → 418.2

Flask 2 324.1 → 412.4

18.01.04

cup	empty	+ GRA
P17	2.5719	38.0698
P18	2.5636	26.0427

Done @ 14H5

↳ in oven @ 14H52

Rt 14H53

24.9°C
1019
1017
1018

P1 790 3'16 sec
551 13.75 sec
546 25.59 sec

P2 ↓ from 47.47 sec
737 53.00 sec
641 1'06.72

1'30.88

552.3/4
630.8/9
1018
25.011

18.01.04

18.01.04
③

1'43.94
553.6/7
557.3
1018

2'04.41
554.3/5
450
1018
25.0

2'42.34
404.1
435
1018
25.0/1

3'12.57
348.0
434.8/9
1018
25.0/1

3'42.28
346.8 → a few ∘∘
343.1
1018
25.0/1

4'21.07
347.3
298.1
1017/1018
25.0

4'55.07
319.5/6
298.3/4
1018/1017
24.9/25.0
bay window half open

5'48.94
313.9
298.1
1018/1017
25.0/24.9

when flasks stirred a few ∘ in F1 & P_1 ↗ 316.8
P_2 ↗ 299.3

7'51.28
311.0
298.0/297.9
1018
25.1/2

close bay

18.01.04

11'44.50	312.8/9 298.0/1 1018.0 25.0/1

@ 15H05

Cups 17 & 18 out of oven @ 15H06

16'03.48	313.0/312.9 298.2/1 1018.0 25.1

stop coatch stopped !!! @ 16'06 restart straigh awat

open bay half way

Weighing cups @ 15H12

P17	24.3974
P18	15.1156

& ↓ fast

Back in oven @ 15H13

3'50"	313.1/2 298.4 1018 25.0

18.01.04

18.01.04
(4)

15'40"00

313.5 → ↗ 314.6 and a few ∴
298.6/7 → ↗ 299.3 when stirred
1018
24.8

25'10"00

313.6
298.8/9
1018.0
24.8

36'40"00

313.7/8
299.1
1018
24.8

Cups P17 & P18 out of oven @ 15H47
cup 18 dropped on floor @ ~15H57 but OK as solid but soft ⇒ no GRA loss
Weighing @ 15H58

cup	m(g)
P17	20.4235
P18	14.2978

↳ back in oven @ 15H59

51'40"

313.6/7
299.3
1018
24.9

56'50"

313.6/7
299.3/4
1018
24.9/25.0

18.01.04

1H17' 20"
313.9/314.0
299.7/8
1019.0
24.8

Readjust P_1 & P_2 so that they are a bit lower

1H19' 10"
310.1
297.3/4
1019.0
24.8

1H24' 00"
307.6/7
297.4/5
1019
24.8

1H45' 20"
302.5/4 → readjust P1 (↑)
297.5
1019
24.8/9

1H47' 20"
305.6/7
297.5/6
1019
24.9

Cups P17 & P18 out of oven @ 16H56
weighing @ 17H05
cupP17 = 19.9436
cupP18 = 14.0072
back in oven
@ 17H06

18.01.04

18.01.04

(5)

At 17H07

1H56'30"

305.5/6
297.6/5
1019.0
24.9

At 17H40, 2H30'45"

302.6/7 → adjust P1 (↑)
298.2/3
1019
24.8

2H49'10"

303.9/3040
298.5
1019/1020
24.7/8

At 18H27

3H16'50"

302.4/5 → adjust P1 (↑)
298.6/7
1020
24.7/8

3H34'30"

304.0/1
298.8/9
1020.0
24.7

18.01.04

4H14'20"

at 19H24

303.2/1 → ↗ P1 a bit
299.3 → lower P2 a bit
1020
24.7/6

4H20'30"

305.8
294.5/6
1020
24.7

4H28'15"

305.6
294.6/7
1020
24.7/8

4H41'45"

305.4/5
294.7/6
1020
24.6/7

At 19H52

5H03'30"

305.3/2
294.8/7
1020
24.6/7

18.01.04

18.01.04
(6)

pump hot

~~A~~ 20H32	305.415
	295.213
SH22/45''	1021.0
	24.617

Leave expt running overnight.

GRA sample % acetone initial

GR	acetone
62.7	148.7
48.3	-0.5
	-0.6
	-0.4
111g	147.2g

⇒ 57.01%

18.01.04

18.01.04

19.01.04
①

~~At~~ 7H43

298.8
291.718
1017
23.718 → stop heater

after 16H34'25"

Cups P17 & P18 out of oven @ 7H45

~~After~~ 16H42'10"

298.7
291.7
1017
24.0 → stirred

⇒ P_1 ↗ 298.9
P_2 ↗ 291.8/9

Weighing cups @ 7H54

cup P17	19.0256
cup P18	13.4549

↳ back in oven @ 7H55

19.01.04

At 16 H45'40"	298.6/7 291.6 1017.0 24.1	open bay window
17 H02'30"	299.2/3 291.1/2 1016 24.3	adjust P
17 H06'30"	300.8/9 291.1/2 1016 24.4	
17 H08'30"	300.5 291.0/1 1016.0 24.4/5	
17 H18'50"	299.4 290.8/9 1016 24.5/6	
17 H58'10"	296.6/7 → adjust 290.7 1016.0 24.9/25.0	

19.01.04

19.01.04
(2)

18H 14' 40"
299.7/6
290.7
1016
25.1 → holle on

@ 9H25

18H 22' 50"
298.718
290.6
1016.0
24.5

20h 00 32
293,9
294,0
289,6/7
1015
24,9/25,0 °C

18h 43' 36"
2970
2901
1015,0
24,8/9°

20h23 56
293,5/4
289 6/7
1015,0
25,3°C

19h 11 20
295,5
290,1
1016,0
24,8/9°

20h 43' 40
293,0
289,5/6
1015,0
24,4°C

19h39' 25"
294,5
289,9
1015,0
24,8/9

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242

After 21h17'00"

At 12H27

292.3
289.1/0
1015.0
24.9/25.0

21H22'00"

292.0/1 ⟹ adjust P1 ↗ to 299.6 to 300.5
289.0/288.0
1014.0
25.0/24.9

21H24'15"

302.8/9
289.1
1014
24.9/25.0

21H25'00"

303.6/7
289.1/0
1014
24.9/25.0

24H3'50"

292.0/1
285.3/4 @ 15H14
1011
25.0

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19.01.04
(3)

24H13'40"

291.7/8
285.4/5
1011
24.4

→ stop hotte & open buy window
adjust P1

24H15'00"

296.2
285.6/5
1011
24.6

24H19'10"

294.4/5
285.3/4
1011
24.8/9

24H21'00"

294.1/0
285.3/4
1011
25.0

24H26'00

293.6/7
285.2
1011
25.1

→ stop runs @ 24H26'00
at 15H35
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Kr 15H43

1012
1010
1011
25.1

F2 408.7
F1 414.6

after expts

Cups prepared from 15H45

	empty (g)	+GRA (g)	Sample
P19	2.5604	33.2649	F1
P20	2.5768	31.3385	F1
P21	2.5839	36.0135	F1
P22	2.5230	26.2614	F2
P23	2.5239	30.6445	F2
P24	2.5208	31.8515	F2

Done @ 15H51

Cups P17 to P24 out of oven @ 16H55

1011
1009
1010
25.3°C

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19.01.04

④ Weighing @ 18H10

cup	m (g)
P17	18.8883
P18	13.3641
P19	18.1571
P20	17.2008
P21	19.6981
P22	14.6434
P23	16.9379
P24	17.6800

All back in oven @ 18H13

$P_1 = 1011$ mbar
$P_2 = 1008$ mbar
$P_3 = 1009$ mbar
$T = 25.6°C$

19.01.04

New GRA sample @ 18H38

F	567.9	
M	34.8	
S	280.4	
F+M	602.6	
+ GR	708.5	
+ acetone	888.1	

105.9 g GR

179.6
− 0.9
178.7 g acetone

⇒ 62.79%

↳ stirring @ 18H42

18H59 all GR dissolved
GRA sample ready

887.6 g

230
265

19.01.04

At 19H11

887.2g

a few drop (some on stopper)

Flasks	empty	+GRA	pressure
F1	315.8	423.2	⇒ P1 / run
F2	324.1	422.5	⇒ P2 / run

Done @ 19H13

At 19H14

cups		empty cup	+GRA
P25	=	2.5229 →	41.4983
P26	=	2.5022 →	30.9599

↳ Done @ 19H16

In oven @ 19H18

Cups P19 to P24 out @ 19H18

At 19H21

1010
1007
1008
25.516 → hotter on

Cups P25/P26 out of oven @ 19H 19.01.04

At 19 H25

1010
1007
1008
25.3/4

3/77

850
813
1008
25.3

15.81

497
577
1008
25.3

31'06

418.5
551.5
1008
25.3

58.65

424.8
430.0
1008
25.2/3

1'19.12

427.2
364.4
1008
25.1

1'29.97

428.2/3
361.7/6
1008
25.0

1'46.65

400.1
361.1
1008
25.0

1'59.97

381.5/6
361.1
1008
24.9

2'24.47

356.6/7
360.9
1008
24.8

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19.01.04
(6)

3'10.21	281.0 360.5 1008 24.7

3'28.74	275.7/8 360.5 1008 24.7

→ stir ⇒ bubbles & P ↗

4'12.09	274.1 360.4 1008 24.7

4'51.30	274.9 313.2/3 1008 24.7

5'32.81	274.9 270.8 1008 24.7/8

6'07.62	274.9 245.0 1008 24.7/8

→ hotter on bay window halfway door open

6'41"30	275.8 232.7 1008 24.6

7'02.87	275.8/9 242.6/7 1008.0 24.6

Stirred flasks
P1 & P2 ↗ then ↓
no boiling but
a few ∴

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8'22.40	275.9 241.9 1008 24.6/7

at ~ 19H34

Cups P25 & P26 out of oven @ 19H35

9'48.74	277.0 241.5/6 1008 24.6

16'03.21	277.9/8 241.2/3 1008 24.2/3

22'52.87	278.0 241.2 1008 24.2

→ pump inside bottle

24'24.43	278.0 241.3/4 1008 24.3

19.01.04

Weighing @ 19H51

Cup	m(g)
P19	17.8780
P20	16.9274
P21	19.3662
P22	14.4128
P23	16.6759
P24	17.3683
P25	22.5688 and ↓
P26	16.1208

Back in oven @ 94°C
@ 19H54

28'55.71

278.011 → P1 ↑ 280.6
241.213 → P2 ↑ 245.7 } when stirred
100g
24.213

30'09.65

278.8
241.4
100g
24.4

30'28.74

277.9
241.5
100g
24.4

19.01.04

34'30"00	277.9/278.0
	241.2/1
	1008.0
	24.2

50'10"	277.6
	241.1/2
	1008
	24.2/3

45'15"	277.7/8
	241.1/0
	1008
	24.1

Cups P25/P26 out of oven @ 20H12

Weighing @ 20H21

cupP25	19.9353
cupP26	15.0364

Back in oven @ 20H23

57'45"	277.3/4
	241.1/0
	1008
	24.1

At 20H37	277.0
	241.0
1H12'00"	1008
	24.0

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19.01.04
(8)

Hr 20H 49
after 1H 24'20"

27.8/9
240.9 / 241.0
1008.0
24.1

↳ bave overnight

450
2'05.37
100 m uaw)

19.01.04

19. 01. 04

20.01.04

①

12H23

T° 24,5
P1: 273.4 → 274,7 (Agitation)
P2 = 239,9 → 241.7 (Agitation) Glucke neprek
P3: 1006.0

12H58'40"

273.3
240.2/3
1006
24.4 (bay closed)

13H19' 15"

273.1
240.3
1006.0
24.2 → bay half open

decrease P1 & P2 to ~265 & 230 mbar

13H20'40"
~8H46

266.5/4
230.5
1006
24.0

stop hotte (effective at 13H22'40"

13H22'50"

265.1/0
230.4/5
1006.0
24.0

Note smell of acetone has reappeared (→ degassing
20.01.04

Fiddling w/ P_1 & P_2 to adjust

13 H 24' 50"

266.0/265.9
231.1/2
1006
24.1

Cups P18 to P26 and P17 out of oven @ 8 H 51

13H 26' 00"

264.9/265.0 → ↗ 268.2 when stirred no :(
231.0/230.9 → ↗ 234.3 when stirred no :(
1006
24.2
smell of acetone

13H 38' 00"

263.6/7
230.9/231.0
1006
24.3

13 H 48' 50"

263 0/1 → adjust (↗ a tad)
231.1/2 → adjust (↘ a tad)
1007
24.4

13 H 50' 50"

263.7
230.6/7
1007
24.5

20.01.04

20.01.04
②

Weighing from @ 9H 18

Cup	m (g)
P17	18.7578
P18	13.2836
P19	17.1831
P20	16.2825
P21	18.6142
P22	13.8752
P23	16.0385
P24	16.7094
P25	18.1903
P26	14.0075

Done @ 9H23
Back in oven @ 9H24

13H58'50"	263.1
	230.6/7
	1007.0
	24.6

14H09'00"	262.6/5
	230.7
	1007
	24.7/8

→ hotte on (effective @ 14H 11'00"

14H10'00"	264.9/265.0
	230.8/9
	1007
	24.8/9

20.01.04

14H 12'00"

265.3
230.8/9
1007
24.6/7

14H 12'40"

264.9
230.8/7 → P_2 ↗ 234.7 when stirred, no ∴ but GRA
1007.0
24.6/5

less fluid than sample in F1 and than initial sample

14H 13' 45"

264.7/8 → P_2 ↗ 269.1 when stirred
230.9
1007
24.5

. no ∴
. fluid

~~14~~ 9H40 F2 = 317.2g

from mm

(yesterday 317.2g @ 9H00)

14H15' 40"

265.2
230.6/9
1007
24.3/4

20.01.04

0.01.04
(3)

14H32'10"	264.0/263.9 230.6/7 1007 24.0
14H45'30"	263.6 → readjust P_1 (↗) 230.7/6 1007 23.9/24.0 → stop holle
14H51'00"	265.7/8 → ↗ 267.4 230.8/7 → ↗ 233.4 1007 24.1/2 @ 10H17
15H02'40"	265.2/1 230.7/8 1007.0 24.4
15H16'40"	264.5/6 → ↗ 268.7 230.9/8 → ↗ 233.5 1007 24.6/7
15H21'30"	264.6/5 230.9 1007 24.7

20.01.04

15H48'30"

263.1
231.3/4
1007
25.2/3 → hotte on

15H51'40"

262.8/9 → adjust a bad (262.9/263.0)
231.2
1007
25.0

15H53'30"
@ 11H24

263.2/1
231.1/2
1007
24.8

16H00'00"

262.9/8
231.1/2
1007
24.4/5 → hotte off (@ 16H01'40"

16H02'00"

262.7
231.1/2
1007
24.5 open bay window

16H26'30"
11H53

262.4/3 → adjust a bad (still smell of acetone)
231.3/4
1007
25.1 → hotte on

20.01.04

20.01.04
(4)

→ But has slightly ↗ P_1

8 H27'00"	P_2 ↗	234.1	from	231.3/4	when stirred
	P_1 ↗	267.5	from	263.2	

At 11H55,

16H30'30"	263.9 231.3/2 1007 25.0/24.9
16H52'15"	262.3 231.0/230.9 1007 24.7

(Bay window closed & pump inside)

P_2 ↗ 233.2
P_1 ↗ 266.6 when flasks stirred

16H54'00" at 12H20	263.6/7 231.2 1007 24.7/8
16H54'30"	263.0/262.9 231.2 1007 24.7/8

20.01.04

262

17H12'30"

261.8/7 → adjust a bad
230.9
1002
24.6/5

17H14'30"

262.9/8
231.1/0
1007.0
24.6

17H20'00"
@ 12H45

262.8
231.1/0
1007.0
24.5/6 → stop holte

17H44'40"

261.9/262.0
230.9/8
1007.0
24.8/9 →

~~holte ok~~ is
~~bay~~ window is closed
pump inside
lab door open
N's door closed

P_1 ↑ 232.4 from 230.9/8
P_2 ↑ 266.2

17H47'00"

263.0
231.5
1007.0
24.9

at 13H13

20.01.04

20.01.04

(5)

#63

17H49'20"

262.2/3
231.2/3
1007
24.9/25.0 → hotte on

17H51'00"

261.7/8
231.1/2
1007 ← hotte starts @ 17H51'10"
24.9

bay half open

adjust P_1

17H59'00"

262.7/6
231.3/4
1007
24.6

18H02'45"

262.1/2
231.3/4
1007
24.5/6

18H17'15"

262.2/3 → P_2 ↗ 265.7
231.2/3 → P_1 ↗ 232.0
1007
24.3/4

18H44'00"

261.8 → adjust
230.7/6
1007
24.1

20.01.04

264

18H58'30"

261.8/9
230.7/6
1007.0
24.1/2

19H05'00"

261.5/6
230.6/7
1007
24.1/2

@ 14H31

19H11'00"

261.3 → adjust
230.6
1007
24.2

At 14H43

19H17'30"

261.7/6
230.5/6
1007
24.2

20H35'10"

260.1 → readjust a bad
230.7/6
1007
24.1/2 → stop hotte openbaar

at 16H01

20H47'00"

260.7/8
231.2
1007
24.5

20.01.04

20.01.04

(6)

20H56'00

260.9/261.0 → ↗ 264.7
231.0/1 → ↗ 232.0
1008.0
24.7

At 16H36

21H10'30"

261.3/4
230.8/9
1008
24.9 → holta on

21H16'00"

261.1/2
230.8/9
1008
24.8

21H16'40"

261.1/2
230.9/231.0
1008
24.8/7

21H20'45'

261.0/1
230.9/8
1008.0
24.8

20.01.04

21 H 31' 20"

261.0/260.9
231.0/230.9
1008
24.7

21 H 40' 10"

260.9
230.9/8
1008
24.6/7 → ferma hotte

21 H 53' 40"

261.1/0 → ↗ 265.7
230.9/231.0 → ↗ 232.3
1008
25.0 → hotte

~~Mr~~ 17 H 30

22 H 04' 00"

260.5
230.9/231.0
1008
24.7

22 H 11' 35'

260.4/5
231.1/2
1008
24.7

20.01.04

20.01.04

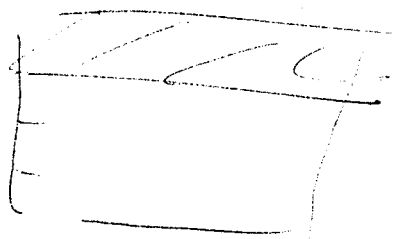
(7)

22H24'00"

260.7 → adjust
231.0/1
1008
24.7

22H28'00"

266.1 → readjust
231.3/4
1008
24.7



bottom

22H35'00"
@ 18H01

260.3/4 → ↗264.5
231.0/230.9 → ↗231.8
1008
24.7

22H37'30"

260.6/7
231.2
1008
24.7

22H42'20"

259.9 → adjust ⟹ 260.5/6
231.0/1
1008
24.7

20.01.04

268

23H 24'00

262.5
231.2/3
1008
24.5/6 → stop hotte

23H 33'00

262.5/6
231.0/1
1008
24.7/8

23H 36'10"

262.4
231.0/1
1008
24.8/9 → hotte on

23H 38'00"

↓
hotte starts

262.3/4
231.1/2
1008
24.9

23H 51'00"

261.9/8
231.2/3
1008
24.6 → hotte off

24H 2'00"

20.01.04

261.7/8
231.3/4
1008.0/1009
25.0/24.9 → hotte on

20.01.04

(8)

All cups out of oven @ 19H29

Weighing started @ 19H48

Cup	m (g)
P1 (26)	14.1936
P2 (27)	16.4938
P3	16.5364
P4	38.9986
P5	18.2961
P6	29.1123
P7	28.1227
P8	24.0772
P9	18.7222
P10	20.9923
P11	14.6639
P12	13.2380
P13	28.7950 → 19H54
P14	15.1441
P15	21.1132
P16	7.8040
P17	18.6902
P18	13.2470
P19	17.0309
P20	16.1359
P21	18.4580
P22	13.7625
P23	15.9023
P24	16.5647
P25	18.0058
P26	13.8662

Done @ 19H58

Back in oven @ 94°C

@ 19H59

20.01.04

24 H 04' 00"

bottom

261.7/8
231.3/4
1008/1009
25.1

24 H 15' 20"

261.4/5
231.4/5
1009
25.0

bouse window
mere

24 H 20' 20"

261.3
231.7/6
1009
24.6/7

@ 19 H 46
open buy mare

24 H 34' 10"

261.4/5
231.8/7
1009
24.7

@ 19 H 59

24 H 39' 40"

261.5 → ↗ 264.5
231.8/9 → ↗ 232.0/1
1009
24.7/8

24 H 42' 45"

261.6/7
231.6/7
1009
24.7

20.01.04

9.01.04
(9)

24 H 51' 10"
@ 20 H 17

261.5
231.6/7
100g
24.6/7 → open fully bar & switch off heater

25 H 05' 20"

261.6/7
231.9/232.0
100g
24.9 → heater on

25 H 12' 00"

261.3/2
231.9/8
100g
24.8

25 H 24' 30"

261.0/1
231.8/7
100g.0
24.7/8

26 H 37' 00"

261.3
231.7/8
100g
24.7/8

26 H 40' 50"

261.9
231.8/9
100g
24.7/8

20.01.04

272

25H47'45" | 261.617
231.718
1009
24.7

@ 21H13

25H55'00" | 261.617
231.718
1009
24.7

stop
@ 25H55'05"

@ 21H21

| 1010
1008
1009
24.7

At 21H23

F2 374.4/3

↳ cups

	empty	+GRA
P27	2.5264	33.1048
P28	2.5409	8.4138

At 21H28 F1 = 394.4 g

↳ cups

P29	2.5271	31.9856
P30	2.5201	34.8197
P31	2.5388	14.7484

Done @ 21H32 → moven @ 21H33

20.01.04

20.01.04
(10)

At 21H37 317.2g

RunP6

Cups out of oven @ 21H59
P27 to P31

Weighing @ 22H06 @ 23.7°C

Cups	m(g) 22H06	m(g) 22H31
P27	29.3804	28.9575
P28	7.4585	7.3794
P29	20.8233	20.0765
P30	22.4398	21.7448
P31	9.6398	9.5370

still warm!

Done @ 22H08
↳ back in oven @ 22H08

Out @ 22H27
↳ weighed @ 22H31

↳ Back in oven @ 22H35
⇒ home!!!

20.01.04

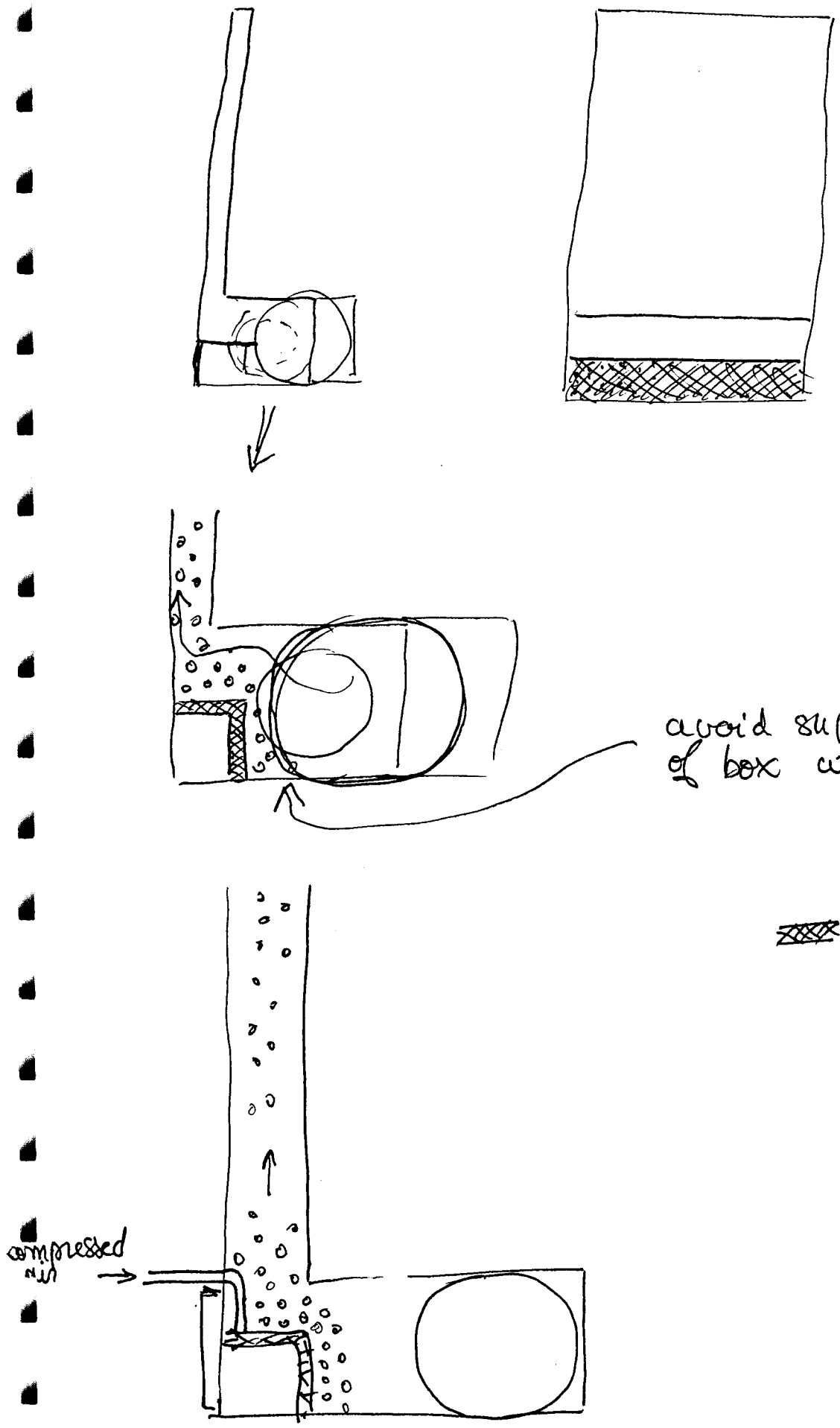
Comments / thoughts on future GRA tunnel runs:

Large GRA samples are long and difficult to prepare
Setting up the H-S cell and cleaning after a GRA run is a long and quite exhausting task (need/have to wear a fullface mask due to sensitivity to acetone).
GR is also expensive (~ £900 for 50 kg). One cannot afford, regarding time and cost, to run tests which parameters would have not been carefully thought and chosen. There are still many unknown: the GRA EOS is still not well constrained (have to wait for the final results of all the EOS runs) and one needs to know more about the physical effect of expanding bubbles during decompression. One way of addressing the latest question would be to runs a few tests with bubbles-bearing golden syrup. To do so, one needs a system which can generate air or nitrogen bubbles at the bottom of the H-S cell. One could for example make a "bubble-box" from Porvair material and have compressed air or nitrogen injected inside the box. The air would be forced out of the box through the Porvair producing bubbles in the liquid residing inside the dike part of the cell.

29.01.04

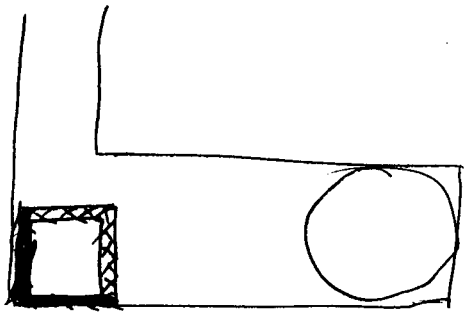
Bristol workshop → bubbles box

10H00 ↓ 11H45

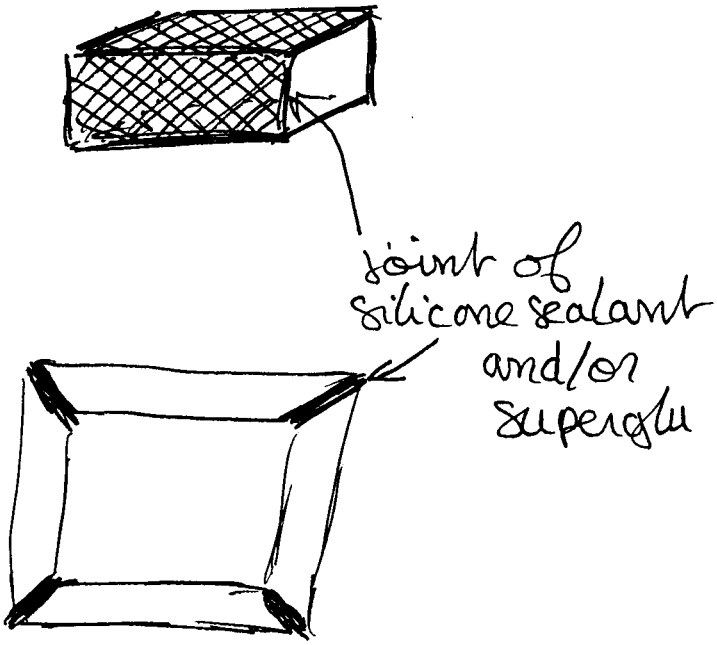


22.01.04

6 faces closed box ⟹ to avoid air leak



- 2 sides made of Perspex
- 4 sides made of aluminium or polypropylene or anything else which is acetone resistant

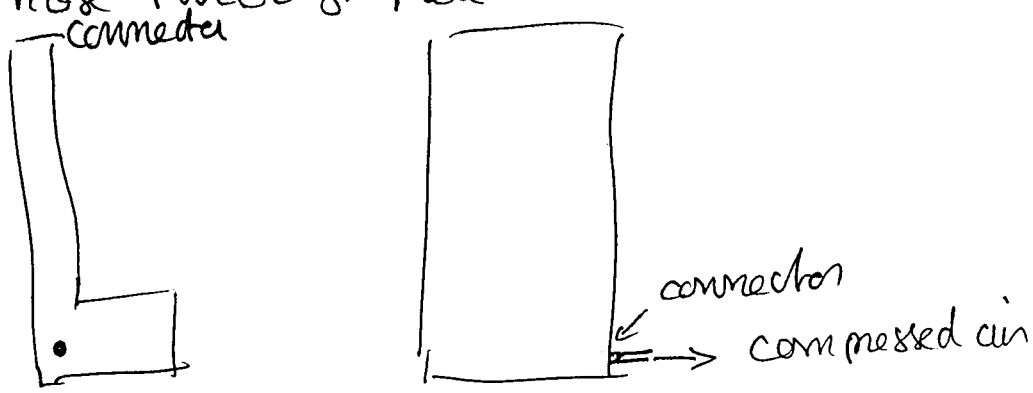


22.01.04

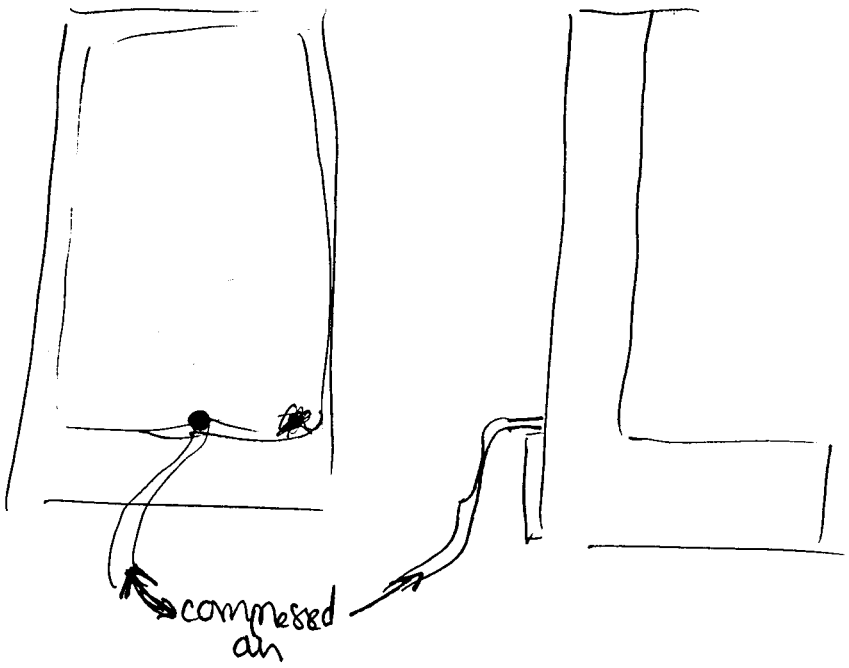
Connection to compressed air

several options

1) hose through Hele-Shaw cell side



2) hose through glass plate



22.01.04

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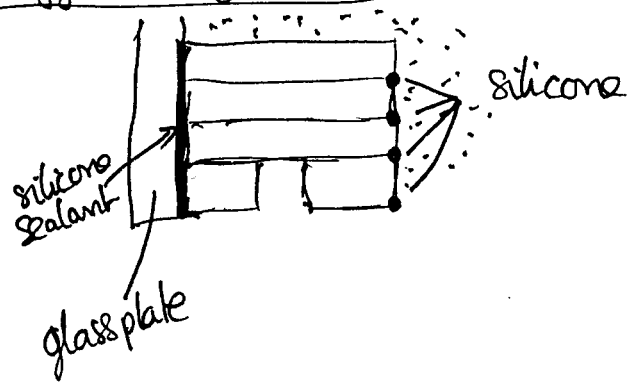
Air distribution in bubbles box

1) point distribution? or 2) line distribution?

might give a more homogeneous bubble distribution in dike

Suggestion for line



Need to discuss with Mike Dury ASAP about the bubble-box and its design.

22.01.04

Back in Bristol on 23.01.04 with EOS eqpt.

Things to do:

- order thermometers from RS + check validity date on mask filters. If necessary, order filters
- Fisher order
  - butyl gloves
  - vacuum tubings
  - rigid tubings → none available
  - stoppers (green)
- Faculty store
  - acetone (2.5 l) × 4
  - rigid tubings → none available
  - nitrile gloves (box of 100)
- Hawthorns order = Golden syrup
- contact workshop regarding bubble box, but first test in tube filled with GS (ask Jerry / Mike): could Porvair be blocked by GS ??
- weigh all the GRA cups

26.01.04

- Contact ASAP Severn Science to connect ASG controller onto PC (as do not manage to have a PC reading data from controller)

26.01.04

28/01/04

Cups B1 to B25 out of oven @ 19H17
(T in oven = 92°C)

Weighing @ 19H48 - T = 27°C

Cup	m (g)
B1	11.8285
B2	20.6011
B3	11.6208
B4	19.3270
B5	21.6987
B6	16.1216
B7	15.5015
B8	13.9119
B9	14.8487
B10	13.1169
B11	15.7796
B12	18.5463
B13	11.5563
B14	6.4191
B15	12.513
B16	14.8246
B17	18.709
B18	17.7038
B19	23.9578
B20	7.5207
B21	18.5287
B22	24.9522
B23	26.3177
B24	29.6871
B25	31.8915

19H53

D

6

Done @ 19H57
Back in oven @ 20:00 (w) T oven = 50°C & light off
(Cold room = 6°C)

28.01.04

282
31.01.04 Check ΔT in cold room.

Cold room 13H00 $T_{set} = 5°C$ light off

↳ $T_{door} = 7°C$

16H00 $T_{door} = 7°C \rightarrow 6.3°C = T_{room}$

17H45 $T_{door} = 6°C \rightarrow 5.7°C = T_{room}$

↳ T_{set} increased by 1°C from 5°C to 6°C

Room left w/ light off.

31.01.04

05.02.04

At 19H00

Lightoff
$T_{set} = 5\,°C$
$T_{door} = 7\,°C$
$T_{Thermo} = 5.9/6.0\,°C$ (in the middle of the room)

↳ leave light on

21 H00

$T_{door} = 6$
$T_{Thermo} = 5.5/5.6$

05.02.04

05.02.04

07.02.04
D

Cold room T= 5.6 @ 16H20
↳ Tset increase by 1°C to 6°C from 5°C

At 16H35
1010 mbar Sn 005000333
1010 mbar Sn 005000334
1012 mbar Sn 005000176
6.8

At 16H45 T_{room}=7.0/1°C w/ light on
At 17H07 T_{room}=7.1°C

At 17H27
1010
1010
1012
7.1/2°C

→ New GRA sample
F 570.1
M 36.1
S 277.1
17H28
95.1g GR
165.4 ace

F+M 606.1
+GR 701.2
+acetone 866.6 +S 1143.7 → stirring @ 17H33

07.02.04

Note Dec Run 03 acetone used BDH AnalaR Prod 100035R

Lot K32632306 345

CH_3COCH_3 = 58.08 g/mol

from VWR International Ltd
Poole BH15 1TD U.K.
Tel. 01202 669700

Purity ≥ 99.5%

Test date 29.10.03

Minimum shelf life 31.10.08

New for Feb 04

Lot K30222706205

Test date 21.01.02

shelf life 31.01.07

17H45 6.8°C 1010 / 1010 / 1012

⟹ Runs @ 100 mbar
90 mbar

07.02.04

07.02.04
(2)

At 18H05 | 1009
1010
1011

95.1g GR
165g acetone
↳ 63.44%

imploded F+S 1143.5
F 866.2

	empty	+GRA	
F2	319.9	402.9 (some on walls)	⇒ P2 RumB10
F3	317.4	499.8	⇒ P1 Run B9

↳ in cold room @ 18H09 T=7.1°C (light on)

At 18H14
T=27°C

cups	empty	+ GRA
B 26	2.5661	23.6640
B 27	2.5572	32.22.23
B 28	2.5724	10.2808

In oven @ 18H20 @ 92°C

07.02.04

At 18H20

1009
1010
1011 when thermo
10.1/2 in flask
9.8 outside flask
↳ 8.8

(@ door
= 8°C
from 6°C
T set to 5°C

18H21 start run

P2	P1	t	P3	T
1009	905.7	9.35	1011	8.8
–	699.6	28.79	–	9.0
–	430.1	1'01.73	–	8.8
–	360.6	1'27.57	–	8.7
897	354	1'59.07		climb on
288.0	641.2	2'25.92	–	8.5/6
473.7	233.2	2'53.92	–	8.4/5
282	196	3'20.01	–	8.3
251.1	156.0	3'36.54	–	8.2
	↳ bubbles @ 143			8.1

07.02.04

07.02.04
(3)

			T(°C)
137/138	250.5	4'13.23	7.8
↳ boiling gently			
136.0	160.1	500.89	7.8/9
	↳ boiling	when passed the BS	
125.9	123.4	1011	555.23 8.0
126.1/0	123.5/4	1011	632.67 8.0
	↳ leave it for a while switch light off		
123.2	120.7	1011	7'11.10
121.4/5	119.5	–	9'39.67 8.1
	no °°	(°° when stirred & P ↗)	

Room set to 4°C @ 18H35

~~M~~ 20H05 ~~D~~ H44/10 T=5.7/8

F3 is OK
100g
100g
1010

Stop run as F4/05

(RA up to ceiling!!! blown up in cold room)

in column holder
Note same as flask (photos on 13.02.04)

07.02.04

Cups out of oven @ 20H10

At 20H20

B26	11.1315
B27	14.7559
B28	5.6994

Back in oven @ 20H23

⇒ cleaning cold room with acetone

Done @ 20H45

At 20H47

1007
1007
1009 / 1010
5.4

Restart run with F3

670 4'33
420 12'06
362.6 1007 1010 22'03 5.5°C
open door (strong smell

288 — — — 56.24
273.5 1'07.37 5.6°C
210.5 — — 1'31.84 —
179.3 — — 1'47.71 —
150.0 1007 1009 2'12.12 5.7
126.4 — — 2'34.03 5.7
115.0 2'56.03

07.02.04

07.02.04
(4)

109.8 1007 1009 3'13.12 5.7/8

106.1 3'36.40 - - 5.8 adjust P_1 (↗)

107.5/6 3'54 11/21 - - -

stirred → bubbles (gentle but rapid bubbling

P_1 ↗ 112.3

107.9 - - 5.9 4'50.97

107.0/106.9 - - - 5'22.65 still bubbling

106.6 - - - 5'54.81 adjust P1

107.2 - - 5.9°C 6'26.49

107.2 - - - 7'28.49

Cups out @ 20H58

107.0 - - - 9'00.49

Cups @ 21H04 - 27°C B26 | 11.0775
B27 | 14.6704
Back in oven @ 21H06 B28 | 5.6788

107.3
1008
1010

19/11.03 5.8°C

07.02.04

35'00.00 107.4/3 1007 1009 5.8/9°C

stirred P1 ↑ 109.5
bubbles but no boiling

Tset cold room ↑ from 4 to 5°C
@ 36/20'00"
21 H24
↳ leave overnight
w/ light off

37'00.00 5.9/6.0°C 107.6/7 1007 1009/1010
↳ light off

07.02.04

08.02.04
①

Cups out of oven @ 10H04

~~At~~ 10H05 13H17'00"

118.6
1015
1017
6.7/8 w/ light off (doors = 5

stir → no change in P, no ∘ ∘
lower P1 to ~ 100

13H18'55"

100.1/2
1015
1017
6.7

stir P1 ↗ 112.7 then ↓

13H20'15"

103.8/7
1015
1017
6.7

Weighing @ 10H09 T = 27°C

Cup	m(g)
B26	10.69865
B27	14.1485/6
B28	5.565 0

Back in oven @ 10H12

13H25'00"

98.2/1
1015
1017
6.6/7

read first P1 (↗ a bad)

08.02.04

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13 H30'15'' | 98.7 (=> P1 ↗
1015
1017
6.6/6.7°C

13H31'00 | 100.4
1015
1017
6.6/6.7°C

~~At~~ 12 H08, 15H20'20'' | 102.0/101.9
1016.0
1018
6.5

stirr => no ∞ but P1 ↗ 106.3

15H22'10'' | 102.3/4
1016
1018
6.5

15H25'45'' | 102.2
1016
1018
6.6/5 (after clim stopped
(Tclou = 5°C
Tset = 5)

15H29'40'' | 102.1/2
1016
1018
6.5 leave lights on

08.02.04

08/02/04

(2)

At 18H54

16H06'20" | 100.3/4 | 1017 | 1019 | 6.7°C | ↓ a bit (6 at door)

16H07'00" | 100.4/3 | — | —

17H01'10" | 100.3 | 1017 | 1020/1019 | 6.7/8 | light has been left on | no °° when stirred but P₁ ~ 104

At 15H12

18H23'50" | 101.3 | 1018 | 1020 | 6.8/9 → ↓P₁ a tad to 100.5

stirred => | no °° | very fluid | P₁ ↗ 104.9 | then back down to

18H25'40" | 100.7/8 | 1018 | 1020 | 6.9°C

18H36'00" | 100.5/6 | 1018 | 1020/1021 | 6.8

08.02.04

18H50'00" | 1019
1021
6.8/7

100.9

15H58, 19H10'20" | 101.2/3
1019
1021
6.9

↓ Plaqad

19H10'50" | 100.1/0
1019
1021
6.9

19H23'15" | 100.1/2
1019
1021
6.8/9

08.02.04

08/02/04

(3)

19 H 56'20"	101.0/100.9 1020 1022 6.9	→ ↓ a bad
19 H 57'00"	100.3 1020 1022 6.9	stir → fluid no °° P_1 ↗ 105.0
19 H 58'00"	100.9 1020 1022 6.9	
19 H 59'00"	100.5/6 1020 1022 6.9	
20 H 00'00"	100.5/5 1020 1022 7.0	
20 H 33'00"	101.1 → ↓ 1021 1023 6.9	→ 100.1
20 H 37'20"	100.1 1021 1023 6.8	stirred → P_1 ↗ 104.1 no °°

08.02.04

20 H 39'40	100.2 1021 1023 6.8
21 H 09'40"	100.5/6 1021 1024 6.7
21 H 38'10"	101.4 → ↓ 1022 1024 6.7
21 H 38'50"	100.2/3 1022 1024 6.7
22 H 38'30"	100.9 → P_1 ↗ 103.1 when stirred 1023 1025 6.7

08.08.04

08.02.04

(4)

23H21'20"

101.5 → P, ↗ 103.3 when stirred then ↓
1024
1026
6.7

At 20H10 new batch

F	570.0
M	36.2
S	277.2
F+M	606.2
+ GR	760.1
+ acetone	914.9
+ S	1192.1

↳ stirring on SM8 @ 20H15

23H28'00"

101.7 → ↓ a tad
1024
1026
6.7

23H29'00"

100.415
1024
1026
6.7

08.02.04

23H 46' 20"

100.8
1024
1026
6.7

24H 19' 15"

101.4
1025
1027
6.7

New GRA sample ready @ 21H07

F + GRA + M + S 1191.7
——— − S 914.3

↳ stirring overnight on SM8 (speed 150)

24 H 20' 45"

101.4
1025
1027
6.7

↳ leave overnight

08.02.04

09.02.04

AT 6H45, 24H + 9H57'50"

104.9	↗ 105.2 when stirred (more viscous than yesterday)
1028	
1030	
6.8/6.9	

↓ Pl a head

24H+ 9H59'20"

100.3
1028
1030
6.8/6.9

AT 6H48

New GRA Flask + S		1191.3
———	-S	913.4

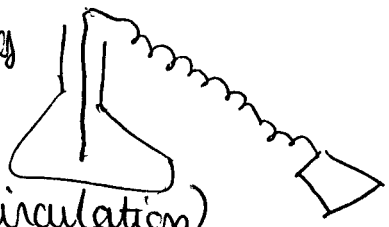
(same on stopper)

↳ left stirring on SM1

24H+ 10H02'00"

100.4
1028
1030
6.9

Note: thermo in flask (like sample) to minimise fluctuations due to switching on/off of cold room air condition-ning unit (air circulation)



09.02.04

302 24H
+10H16'40"

100.1/2
1028
1030
7°C/6.9

10H22'20"

100.2/1
1028
1030
6.9

P_1 ↗ 100.9 when stirred

10H25'40"

100.2/3
1028
1030
6.9/8

Cups out of oven @ 7H17

10H30'00"

100.3
1028
1030
6.8

Weighing @ 7H26 @ 27°C
Back in oven @ 7H29

Cup	m(g)
B26	10.5682
B27	13.9544
B28	5.5355

09.02.04

09.02.04
(2)

10 H 42' 00" | 100.4 (↗ 101.2 when stirred)
1028
1031
6.8/6.9

10 H 43' 30" | 100.4
1028
1030
6.8

7 HSS GRA flask 913.5

↳ left on SM8

11 H 09' 20" | 100.5/6 ↓ P1 a bad
1028/1029
1031
6.8/6.9

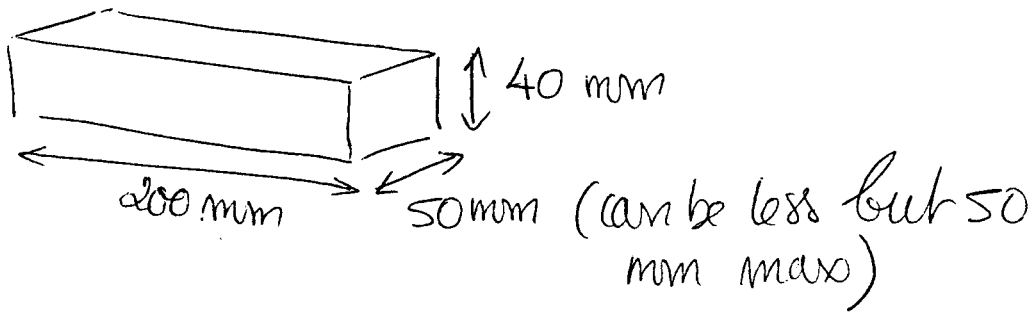
11 H 10' 20" | 100.3
1029
1031
6.8/6.9

09.02.04

304

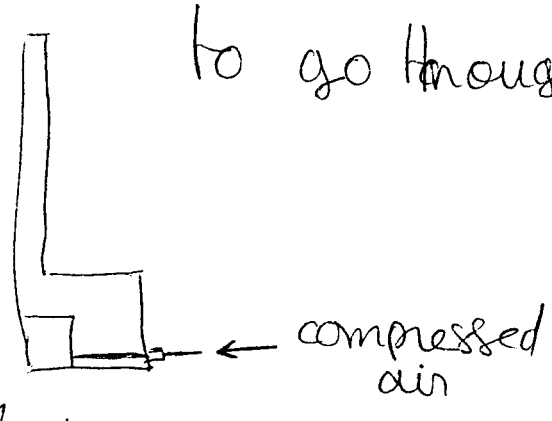
Discussed w/ Mike the "bubble box" for the bottom of the Hele-Shaw cell

⟹ will be ready for April 5, 2004



Top & back side have to be made of Perspex. The others can be made of Perspex or aluminum or any acetone resistant material (e.g. polypropylene but not perpex)

Hole will be drilled at back of HS cell to allow tubing to go through



will have a plug-in connector

09.02.04

GEOLOGY DEPARTMENT WORKSHOP WORK PROPOSAL.

DATE:	09. 02. 2004
NAME:	Anne-Marie Lejeune
TEL:	5243
ROOM:	66
EMAIL:	amlejeun@cor.jussieu.fr
SUPERVISOR:	

FUNDING:

The workshop is required to recharge for any materials supplied.
For NON-DEPARTMENTAL or GRANT FUNDED projects a charge is made for time spent, current rate £20per hour.

Please supply where applicable a:-

GRANT CODE	AY 5336

or a:-

BUDGET CODE	

to which these charges can be made.

Materials		Authorised
Time		Lejeune
Total	~£200	

Brief description of proposed work:

Bubble-box for the Hele-Shaw cell as discussed.

Porvair material provided by AML.

PLEASE RETURN THE FORM TO THE WORKSHOP PIGEON HOLE IN THE COMMON ROOM. YOU WILL BE CONTACTED AS SOON AS POSSIBLE AFTER RECEIPT OF THIS REQUEST. TO DISCUSS YOUR PROPOSAL.

09.02.04

09.02.04

09.02.04

③

Time(+24H)	P_1	P_2	P_3	T
11 H 10' 30"	100.3	1029 / 1028	1031	
14 H 33' 30"	100.4	1029	1031	
18 H 41' 30"	99.8 - 99.9	1028 / 1029	1031	
19 H 14' 00"	99.8/7	1028	1031	6.7
(16 H 02)	↗ 100.1 when stirred			
19 H 15' 00"	100	1028	1031	6.7
20 H 13' 40"	99.9/100	1028	1031	6.6/6.7
	↗ 100.2 when stirred			
20 H 14' 40"	100.0/1	1028	1031	6.6/6.7
20 H 21' 30"	100.0/99.9	1028	1031	6.6/6.7
22 H 20' 45" @ 19 H 08	99.7	1028	1030	6.8
22 H 40' 00"	99.5	1028	1030	6.7
	↗ 99.8 when stirred			
41'	99.6	1028	1030	6.7
stop @ 24H + 22 H 41' 09 @ 19 H 30	1028	—	—	—

09.02.04

308

clean flask from GRA projections
w/ acetone

At 19H32 $m_{Flask3} = 372.5\,g$

At 19H35

cup B29	=	2.56 28
+ GRA	=	29 0306
cup B30	=	2.5627
+ GRA	=	10.2220

in oven @ 19H39

↳ after run $\boxed{25.41\%}$

⟹ next runs @ $\begin{cases} 90 \text{ mbar} \\ 94 \text{ mbar} \end{cases}$

09.02.04

Then @ 70 mbar
and < 60 mbar

09.02.04
(4)

At 20H23

1028
1028
1030

Flask + GRA + S = 1191.1
——— − S = 912.8 (same as S)

	empty	+ GRA	
F3	317.5	406.0	→ P2 Run B12
F4	328.0	432.1	→ P1 Run B11

in room @ 6.5°C
@ 20H25

→ cups @ 20H39 − T = 27°C

	empty	GRA	
B31	2.5443	28.6325	
B32	2.5513	30.5325	→ in oven
B34	2.5154	28.2761	@ 20H48 09.02.04
B33	2.5681	24.7531	

Cups B29 & B30 out of oven @ 20H47

P | 1028
1028
1030

At 20H59 | 1028
1028
1030
6.9 → start (decrease P_1 & P_2 slowly & gently)

			P_1	P_3	
P_2	730.0	5.02	1028	–	6.8
	688.3	17.02	1030	–	6.8

P_1	P_2	P_3	T	t	
881.5	687.4	1030	6.718	1'14.36	start ↓ P_1
743.6	687.2	–	–	1'47.61	after 60"
598.3	687.0	–	6.7	2'12.49	
597.5	597.8	–	6.718	2'53.17	
470.5	597.5	–	6.7	3'33.17	
470.0/1	566.1/2	–	–	4'01.87	
469.9	403.0/402.9	–	–	4'52.52	
333.1	402.5	–	–	5'27.05	
09.02.04 332.4	323.4	1029	6.8	5'53.14	

09.02.04

(5)

P_1	P_2	P_3	T	t
202.1	322.6	1030	6.7	7'03.61
201.3	237.3	–	–	7'39.70
201.0	160.3	–	–	8'22.02
138.7 a few	159.6 a few			8'47.70
9'04.33				
127.5	159.3	–	6.718	9'29.30
113.3	116.0	1030	6.7	10'57.89
108.0	107.8	–	6.718	11'46.00
105.8	101.3	–	–	12'32.20

when stirred, both boil fast but gently but only on rim

& P ↗

109.7	105.9	–	–	14'19.49

Cups out of oven @ 21H15
B31 to B34

109.2	105.2	–	–	16'18.73

P_1 & P_2 oscillating a bit
check → no leak (valves closed)
close 1 valve, can pump down
pump stabilize & can be
liquid acetone exhausting from pump

09.02.04

21'27.02 106.0 103.9 1029 6.7/8
3/2

Cups weighting @ 21 H 23 - 27°C

Cup	m (g)
B29	25.1848
B30	8.9075
B31	19.7929
B32	20.7488
B33	16.5764
B34	19.5288

→ back in oven @ 9 H 28

28'34.27 105.9 103.7/8 1029/1030 6.4

31'11.89 109.9 107.6 1030 6.5

pump has nice but irregular noise ⟹ presence of liquid acetone

42'30 106.2/3 103.8/9 1030 6.5 @ 21 H 43

Cups out of oven @ 21 H 43

1H06'00" 106.3/4 104.2 1026 6.4

09.02.04

09.02.04
(6)

Weighing @ 22H07

Cup	m (g)
B29	25.1296
B30	8.8930
B31	18.5254
B32	19.7408
B33	16.0761
B34	18.3423

→ Back in oven @ 22H11

1H11'50" 105.8/9 103.5/6 1031 6.415

Cups out @ 22H36

1H37'00" 102.8/9 100.8 1030 6.4

→ 106.0 ↳ 103.4

depends on if liquid acetone blocks

09.02.04

Weighing @ 22H50 - 17°C

Cup	m (g)
B29	25.0316
B30	8.8688
B31	17.8675
B32	19.0439
B33	15.6590
B34	17.6856

→ back in oven @ 22H53

1H54'40"	109.2/3	106.8	1030	6.3 (down = 7)
2H40'40"	105.2/3	102.7/8	1030	6.2/6.3

~~Hr~~ 23H41 → heme

2H41'50"	104.4/5	102.1/2	1030	6.2/6.3

still lizaeline

09.02.04

02.04

(1)

Cups out of oven @ ‖ H13

At ‖ H14

14 H15'00"

45.5/6
42.3
1027
6.4

P2 ↗ 75.0 when stirred
(skin removed upper degassed
↳ bubbling

(and P1 also ↗ 78.0)

close P1 valve @ 14 H16'40"
to help stab. P2

14 H17'15"

84.6
72.1/0
1027
6.4

10.02.04

316

still quite fluid

P2 | 109.5
90.0
1027
6.4/3

14H19'44''

14H20'10'' 65.7 ↑ P2

14H20'45'' 76.6

14H21'00'' 71.6 | P_1 = 120.0
1027
6.3/4 bubbling
with valve closed

14H21'45'' 68.4 ↓ bubbling

14H22'20'' | 131.1
70.1/2 still
1027
6.4

14H22'50'' | 136.9
69.8
1027
6.4/5 → adjust P_2 then close P_2 valve

14H23'20'' | 141.2
70.5 (102)
6.4/5

10.02.04

:02.04

317

(2) P_1 ↓

14H23'45" | 87 bubbles
76.5
1027
6,5

14H24'30" | 67.3
66.6 no
1027
6,5

14H24'45" open valve P2

14H25'00" | 68.110
76.0
1027
6.5

10.02.04

318

14 H 25 30''

64.2
70.9 a few °°
1027
6.5

leave it for while while by degos

14 H 26' 50''

64.0
70.9
1027
6.5

14 H 27' 20''

63.5 → 63.9
70.5
1027
6.5

14 H 44' 00''

56.4 – 56.8
69.8
1027

10.08.04

10.02.04

(3) Weighing cups @ 11H35 T = 27°C

Cup	m(g)
B29	23.8080
B30	8.5830
B31	16.4783
B32	17.5409
B33	14.5443
B34	16.3536

Use Balance
||
OHAUS
GA200D
serial # 3072

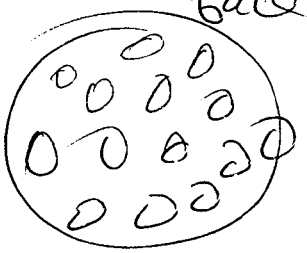
↳ back in oven @ 11H59

15H00'35'

50.7
70.0
1027
6.3

oscillating w/ liquid acetone in pump

and as bubbling on whole surface



Readjust P_1 carefully as don't want P_1 ↓ too much and to stabilize P_1

15H03'40"

50.817
69.9/70.0
1027
6.4

readjust P_2

10.09.04

15 H04' 20" | 50.8/9 → 51.2
70.3/4 ⟹ stirred
1027
6.4
↳ P_2 ↗ 76.3
and ooo

15H06' 20" | 60.9 after shaking P_1
71.0
1027
6.4

15H07 100" | 55.5 bubbling
70.7
1027
6.4

15H07' 50" | 50.8/9
70.8/7
1027
6.4

15H37' 30" | 50.6/7 ⟹ degassing on all over
70.5 over surface
1026 ↳ will eventually foam
6.6/6.5

10.02.04

10.02.04

(4) 15H40'20" | 51.1/2
71.1/2
1026
6.5/6

15H49'10" | 51.2 → foaming
71.2/1
1026
6.3

@ 12H49

17H31'30" | 38.9 readjust P1
71.2
1025
6.5/6

→ all foamed up flask completely filled up

→ but unfoamed bit bottom

17H32'45" | 40.0
71.4/5
1025
6.6

17H33'20" | 40.76 adjust
71.3
1025
6.6

17H33'50" | 41.2/1 → sudden ↗ to 54.7 then ↓
71.3
1025
6.6

10.02.04

322

17H 34'50"

40.7
71.0/70.9
1025
6.6

17H 35'30"

40.6/9
71.1 ↗ 76.5 when stirred & ∴
1025
6.6

17H 40'20"

48.8
71.6
1025
6.5

17H 41'00"

40.2
71.2/3
1025
6.5/6

19H 02'00"

40.2
72.6
1025

10.02.04

0.02.04 (5)

20 H 38' 20"
41.0
73.2
1025
6.5

@ 17 H 37

20 H 39' 00"
40.8/9
72.3 → ↑ 76.7 when stirred (and 90°)
1025
6.5

20 H 39' 50"
40.9
72.9
1025
6.5 → ↑ Tset by 1 from 5 to 6°C & switch light off

20 H 41' 00"
40.9
72.8/9
1025
6.5

Cups out of oven @ 20 H 12

23 H 13' 40"
41.7
74.2
1024
7.5 (@ door → Tset from 6 to 5°C

10.02.04

23H15'00"

41.9/42.0
74.3
1024/1025
7.5

23H34'45"

41.9 → photos
74.0 → ↗ 76.1 when stirred & ∞
1024
7.0

23H37'00"

41.7/6
74.2/1
1024
7.0/7.1

23H37'30"

41.7
73.5
1024
7.0

23H45'40"

41.8
73.5/4
1024
6.8/6.9

23H50'10"

41.9/42.0
73.3
1024
6.8

10.02.04

10.02.04
(6)

20 H 39 weighing - 27° C

Cup	m (g)
B 26	10.4882
B 27	13.8211
B 28	5.5184
B 29	23.5895
B 30	8.5348
B 31	16.3456
B 32	17.4004
B 33	14.4335
B 34	16.2231

→ back in oven @ 20H44

At 21 H 22,

24 H 22' 30"
42.110
74.2 ⇒ ↗ 75.7 when stirred + °°
1024
6.7

24 H 46' 30"
42.112
74.213
1024
6.4

25 108' 30"
42.211
74.0/73.9 (still ↘ few °° when stirred)
1024
6.5

10.02.04

326

25H28'10"

42.011
73.2 ↗ 75.3 when tired + ⁰⁰
1024
6.5

25 H 30'00"
at 22 H 29

42.0
73.3
1024
6.5

→ home! Sweet home

(me ago home)

10.02.04

10. 02. 04

328
11.02.04
(0)

	empty	+ GRA	after run		
F3	317.5	406.0	371.2 17.03%	P_2	Run B12
F4	328.0	432.1	~ 6.85%	P_1	Run B11

Starting GRA

GR	760.1 - 606.2
acetone	914.9 - 760.1
	914.3
	913.4
	913.5
	912.8

Plan 2 next runs

~ 50 mbar	needs	~ GRA 20
~ 110 mbar	needs	~ GRA 60

11.02.04

11.02.04
①

~~At~~ 17H21 44H21'25"

46.4
77.3
1026
6.4

↗ Tset from 5 to 6

Cups out of oven @ 17H22

24H+
20H41'40"

46.4
76.7
1026
6.8

↘ P_1 & P_2 to closer values from yesterday's

Note in F4, foam has collapsed (same on tubing)

Tset from 6 to 5

24H+
20H44'10"

43.0/1
72.6 → slowed P_2 ↗ 73.4 + bubbles
1026
6.9/7.0

24H+
20H45'50"

42.8/9
71.8 → b° ⇒ ↗ P_2 a bit
1026
7.2

11.02.04

24H +
20H 48'40"

43.011
73.011 → still degassing
1026
6.9

Cups weighing @ 17H51

Cup	m(g)
B26	10.468 4
B27	13.7849
B28	5.5136
B29	23.3236
B30	8.4754
B31	16.2004
B32	17.2464
B33	14.3197
B34	16.0725

Back in oven @ 17H55

24H +
20H56'00"

43.415
73.1
1026
7.4

24H + 21'04'40"

43.617
73.7 ↗ 74.3 as from stirred + ⚠
1026
6.1 ⇒ Tset back to 6°C

11.02.04

11.02.04
②

Start new sample: ~GRA 20 @ 17H57

F	570.1
M	36.1
S	277.5
F+M	606.2
+GRA	704.7
+ acetone	736.6 → storing on SM8

24H + 21H07'20"	43.5
	73.718
	1026
	6.2

24H + 21H20'30"	43.5
	73.0
	1026
	7.1 → light off

24H+21H28'50"	43.7
	73.4
	1026
	7.0

w/ light off & $T_{set} = 6°C$
($T_{door} = 7°C$ 11.02.04

Hr 19H17 still GR to dissolved in flask

24 H + 22 H 17'30"

43.718
73.2
1026
7.2 → Tset down to 5°C

24 H + 22 H 18'30"

43.8
73.2
1026
7.2 → ↗ 75.2 & °° when stirred

24H + 23 H 20' 40"

43.7
73.6 ↗ 74.6 + °° when stirred
1026
6.3

Tset ↗ 6°C & light off

24 H 23 H 23'00"

43.5/6
73.5
1026
6.4

11.02.04

1.02.04

(3)

Hr 20H23 GR not completely dissolved but getting there

$m_{F_{new}}$ = 735.8 g
ORA

24H + 23H25'00"	43.6/7
	72.9/73.0
	1026
	6.6A

Hr 21H01, GR not all dissolved

48H 2'30"	44.0
	73.0
	1027
	6.8

⟹ stop 48H3'00"

$Ne \rightleftharpoons P_2$

48H5'	1024	→ Tset 5°C
	89.4	
	1027	(8°C)
48H6'	1024	
	86.6	
	1027	

heavy higher until Tset back to 6.5°C

11.02.04

→ photos

~~Mt~~ 21H07 F4 = 384.1

but some GRA on tubing

~~48~~ H 9'00'' | 1024
86.5
1027
8°C

~~48~~ H 19'20'' | 1024
85.6
1026
7.6

empty cup ⊔ = 2.6

+ ▭ = | 5.1 g
5.0753

Then clean bit of tubing w/ acetone

11.02.04

11.02.04
(4)

48H 31'00" | 1024
85.6/7 → has one big bubble
1027
7.4

↳ photos

48H 36'30" | 1024
84.5/4
1026/1027
7.3 → light off

At 21H56, GRA flask still one or 2 bits to dissolved fully

48H58'00" | 1025/1024
84.2
1027
6.9/7.0

Decrease P2 to 74.6 → ∴ forms

49H 00'00" | 1025
74.1
1027
6.9

11.02.04

49H04'00"

1025
74.0/73.9 still ∘∘ brining
1027
7.0

49H07'20"

1025
73.9
1027
7.0

New GRA sample @ 22H07

F+S 1013.4 g
F 735.6 g still have a few bits to dissolve (thin ones ∘ ∘)

49H09'00"

1025
74.1/74.0
1027
7.0

11.02.04

1.02.04 (5)

49 H 29' 40"

1025
73.9
1027
7.0

~~Ar~~ 22H30 New GRA sample almost ready

49H 47' 40"

1025
73.7/6
1027
7.0

(still one bubble every now and then)

Back to run B

⇒ cup & ~~tubing~~ have been respectively washed & soaked in acetone, then dried in oven @ 92°C

~~Ar~~ 22H51

~~empty~~ clean cup	2.5635 g	(2.5g)
cup + tubing	4.6543 g	(4.6g)
tubing	2.0909 g	(2.1g)

(original weights on page 334)

49 H 55' 00"

1025
73.6/7
1027
7.0

11.02.04

New GKA sample @ 22 H 56

F+S	1013.2
F	735.3

· still on thin flake to dissolve

↳ back on SM8 to stirring

49 H 58' 30"

1025
73.8/9
1027
7

50 H 18' 00"

1025
73.6
1027
7

~~M~~ 23 H 31

50 H 32' 00"

1025
73.5/6
1027
6.8/9

→ tried stirring but very viscous P_2 stable no ☺

11.02.04

11.02.04

(6)

50H49'00" | 1025
73.5
1027
6.9

At 23H49, new GRA ready

F+S 1013.2
F 735.2

↳ prepare flask F4 @ 23H50

empty 328.0
+GRA 406.4 ⟹ cups @ 23H54

50H52'30" | 1025
73.615
1027
7.0

por 2°.

→ T=27°C

	empty	+ GRA
B35	2.5215	30.6465 (30.65)

t=0 30.6254
t=10" 30.6238
20 30.6222
30 30.6206
40 30.6192

t=50 30.6176
60 30.6163
70 30.6148
80 30.6135
90 30.6121
100 30.6110
110 30.6096
120 30.6082

11.02.04

340

cups B35 in oven @ 92°C @ 0400:

11.02.04

12.02.04

(7)

51H02'00" | 1025
73.6/5
1027
6.8

(T&t = 5 / light on)

51H08'00" | 1025
73.5
1027
6.8

51H20'30" | 1025
73.5/6
1027
6.7/8

Cup B35 out of oven @ 0H09

At 0H20, F4 = 406.3

Cup B35 @ 0H22 = 29.9776g and ↓
T = 28°C

↳ back in oven @ 0H24

51H24'45" | 1025
73.7
1027
6.7/8

still a few H_2O but not many

F4 put in cold room @ 0H25 to ⇌ in T

12.02.04

At 0H45

51 H46'00" | 1025
73.4 → a few ⁰
1027
7.0/6.9 ↑ a tad P2

51 H47'50" | 1025 74.2/1 1027 6.9°C

Start preparing new GRA soon ~ GRA50

At 0H47, B35 cup out of oven

51H56'40" | 1025
73.6
1027
6.8

At 0H59, B37 = 28.1763g
28°C

↳ back in oven @ 1H01

52 H02'00" | 1025
73.5/6
1027

12.02.04

12.02.04
(2)

New GRA

@ 1H02

F	570.1g
M	36.2
S	277.7
F+M	606.2
+GR	667.4
+ acetone	747.3
+ S	1025.0

→ stirring on SM8 @ 1H06

52H07'30"	1025 73.4 1027 6.7
52H33'50"	1025 73.3 1027 6.7

@ 1H33

At 1H35, New GRA ready

F+S	1024.7
F	746.9

12.02.04

↳ stirring on SM1

52H35'40"

1025
73.718
1027
6.7

Cup B35 out of oven @ 1H36

↳ weighed @ 2H02

T = 28°C

$m_{B35} = 27.4498$

Back in oven @ 2H03

52H44'50"

1025
73.2/3
1027
6.718

53H00'50"

1025
73.2
1027
6.7

53H04'30"

1025
73.3
1027
6.8

53H19'40"

1025
73.0/729 → 9 to 73.4 53H20'20"
1027
6.8

12.02.04

12.02.04
③

~~At~~ 2H27

1025
73.3/4
1027
6.7

53H28'15"

~~At~~ 2H28 Meco GRA

F+S 1024.4
F 746.6/5

~~At~~ 2H29

1025
73.4
1027
6.8

53H30'00"

53H31'00"

1025
73.4
1027
6.7/6.8

stop run
@ 2H30

↳ 2H31

1025
1025
1027
6.7/8

F3 = 371.2g
w/ P_2
(~ 17.28%)

12.02.04

At 2H34 T = 28°C

	empty	+ GRA (viscous)
B36	2.5300	18.2970

↳ in oven @ 2H37

Clean flask & let dry on top oven

At 2H45

Cup B36 out of oven

1025
1025
1027

At 3H11

New GRA F+S	1024.	(some GRA on stopper)
F	746.	

F3 empty = 317.8
F3 + GRA = 399.5

↳ in room to ⇌ in T = 6.5°C while preparing cups

Note some GRA on tubing @ 3H15 ⇒ cut tubing & clean

↳ @ 3H17

$m_{tubing+GRA} = 0.7260$

clean tubing = 0.6920

12.02.04

12.02.04
(4)

T = 27.5 °C

~~AA~~ 3H 17 , Cup B36 = 18.0001

cup	empty	+GRA
B37	2.5315	27.4550 ↓ fast
B38	2.5306	20.3920
B39	2.5205	11.2052

cups B26 to B39
↳ in oven @ 3H24

~~AA~~ 3H25

1025
1025
1027
6.5

⟹ start runs @ 3H27

F3 ⟷ P2
F4 ⟷ P1

(P2)

965.7	5'09	6.5	–	–
818.9	21.78	T = 6.5/6	–	–
746.2	39.82	6.6	–	–
582.3	1'00.47	6.6/7	–	–
447.7	1'26.34	6.6	–	–

12.02.04

1'59.41	1025
	364.3
	1027
	6.6

→ close P2 valve & start ↓ P1

2'24.09 364.0

P1

885.4	2'55.09	377.3	1027	6.7
769.2	3'15.88	380.0	–	–
594.3	3'38.57	382.6	–	–
475.0	3'59.25	384.6	–	–
399.8	4'19.57	386.0	–	–
324.0	4'46.91	386.9	–	–

closed P_1 valve, open P_2 and ↓ P2

339.2	5'35.59	211.0	–	–
349.8	6'24.28	146.9	–	–

close P_2 open P1

324.5	6'44.19	153.7	–	–
205.8	7'13.91	157.5	–	–

8' 0447 bubble @ ~95 mbar
huge one forms! (whole surface covered

94.9	8'32.88	159.2	–	6.6

12.02.04

12.02.04

(5)

P1		P2		
95.2	9'29 41	159,8	1027	6,6
↳ bubble all over the place				
94,5	10'02.85	160	–	–

open P2 10'14.19

95.7	146.5	10'20"30	–	–
95	126.5	10'51.66	–	–
	↳ no °°			
94,9/95	120.9	11'15.37	–	–
	↳ no °°	@ 116 mbar		
95.5 after shaking	112.7	12'04.16	–	–
	↳ still no °° even when shaken (not stirred			

P1 ↑ when stirred

96.0	107.7	14'49.82	1027	6.6

when stirring P2, P2 ↑ but no °°

95.8	106.4	15'10.41	–	6.7
95.8	108.7	16'01.44	–	–

↳ bubbling less vigorous => consider ↘ P1

12.02.04

350

P1	P2	t	P3	T(°C)
95.4/3	109.1	17'19.59	1027	6.7/6.8
87.0	109.0	17'52.82	–	6.7/6.8
80.0 ↳ ~~boiling~~ restart	–	18'00'20	–	–
71.6	108.9	18'33.50	–	6.8
68.6	108.8	18'59.16	–	–
↗ 70.3 with boiling	↗ 111.3 when stirred		–	6.7/8
stirred ↗ 82.9	108.6 adjust P_2 (↗)	21'09.57	–	6.7/8
72.7	109.4	21'54.41	–	6.7/8
67.6	109.8	22'24.69	–	–
65.0	109.7/8	22'45.41	–	–
66.4	109.4	23'59.03	–	–
65.2/3 ↳ still boiling	108.9 ↳ adjust P_2 (↗)	30'41.37	–	6.7
65.1 ↗ 81.7 when stirred	112.3	31'30.19	–	–
80.0	111.5/6	32'34.31	–	6.7/8
75.2	111.4 ↳ ↗ 112.4 when stirred	32'57.72	–	12.02.04

12.02.04
(6)

Cups B36 to B39 out of oven
@ 3H51

Weighing @ 3H55 – T = 27.5°C

Cup	m(g)
B36	17.3528
B37	15.4149
B38	11.6325
B39	6.9212

↳ back in oven
@ 3H58

69.7 111.2 33'37.78 1026/1027 6.7/8
64.3/2 110.7 36'34.17 1027 —

restart stop watch @ 4H08 !!! (3H27 ≙ t=0)
⇒ add 41' to new time)

63.8 110.617 21.39 1027 6.6
↳ bubbles coalescing & expanding ↑ to 4cm ⌀!
stirred ⇒ P_2 ↑

12.02.04

63.8 111.4/3 1027 5'20.08 6.7

↳ still bubbles fairly big

Cups B37 to B39 out @ 4H14

62.3 111.0 1027 30/35.27 6.5

↳ 2 photos + stirring

65.8 111.4 – 32/29.39 –

Weighing cups @ 4H48 — T = 27°C

cups	m(g)
B37	15.1597
B38	11.4515
B39	6.860 7

→ back in oven @ 4H52

Bit of tubing from F3 = 0.6920

45'10''

62.2 → foaming as ↑ w/ degassing
111.1 → no °° when stirred but P_2 ↑
1027
6.5

12.02.04

12.02.04 (7)

57'20" 61.4 Foaming 110.7 1027 6.6

1 H00'00" 61.8 111.3/4 1027 6.6

↳ home! @ 5H08 am

12.02.04

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12.02.04
(8)

P_1	P_2	P_3	Time	T (°C)
53.0	111.2	1028.0	5:58:23 (= 10h04:00) mentre Thierry	
53.3 53.4	111.0	1028.0	8:03:23 (12:14:00 mentre thierry)	
53.4	110.9	1028	8H46:55	6.5/6.6
52.9/53.0	111.5/6	1028	8H50/30' @ 12H58 (mentre AML)	6.6
55.3/2	111.9	1028	9H09'50"	6.6
53.6/5	111.8/9	1028	9H25'55"	6.5
53.3	111.5	1028	9H50'20"	6.5
52.9	111.1	1028	13H04' 10"	6.7

all foamed up (⇒ photo)

turned around flask ⇒ still viscous material between bubbly foam

↗ 112.6 when stirred, still very fluid, no bubbles then ↓

↗ 53.2 when looked around

↗ 112.3 when stirred

↳ foam & bubbles w/ viscous walls

12.02.04

P_1	P_2	P_3	time	T
53.8/9	112.4	1028	13H06'15"	6.7
53.5/6	112.3	1028	13H12'30"	6.8
53.5	112.5 112.6	1028.0	13:36:~~23~~ (17:47:00 marie Thierry)	
53.4	111.6 ↳ 112.3 when stirred	1029	14:55'00" 19H04	6.7
53.9	112.3	–	14H56'00"	6.7
53.5/6	111.2/1	1028 1029	19H18'30"	6.8
54.1	112.3	1028	19H21'00"	6.8

↳ home @ 23H28

12.02.04

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12.02.04
(9) (inside page 8)

13H03 cups B36 → B39 out of oven

Weighing @ 13H21 - T = 27°C

Cup	m(g)
B36	16.1896
B37	14.1174
B38	10.8212
B39	6.5877

B38 fell on floor but OK (no loss)

↳ back in oven @ 13H34

12.02.04

13.02.04
①

P_1	P_2	P_3	Time 24H 5:47:23	T(°C)
54.8	112.6 112.7	1029.0	(9:58:00 Thierry)	
53.9	111.4	1027/1028	36H 22'20"	6.6/6.7
53.9/54.0	111.5/4	1028	(@ 16H31) 36H42'50"	6.6

→ only ↗ to 111.7 when stirred

Cups B26 to B39 out of oven @ 16H53

53.8/9	111.4	1028	36H57'40"	6.6/7
53.7	110.9/111.0	1027	37H17'20"	6.6
53.7/8	111.2/3	1027	37H 27'40"	6.9
53.1	111.3/4	1027	37H30'40"	6.8
53.6/7	110.6	1027	37H37'40"	6.9
53.6	110.8/7	1027	37H55'00"	6.8
53.4/5	110.6/7	1027	37H59'00"	6.8/9
53.4/5	110.8	–	38H08'10"	6.8
53.3/2	110.5	–	38H14'40"	6.8
53.3	110.3/2	–	38H24'00"	6.8
53.2/1	110.3/2	–	38H28'20"	–
53.1/2	110.2	–	38H29'30"	6.8
53.1	110.2/3		38H30'00"	

13.02.04

→ stop @ 18H38

Cups weighing @ 17 H 28 - T = 27°C

cup	m(g)
B 26	10.4373
B 27	13.7283
B 28	5.5057
B 29	23.0558
B 30	8.4189
B 31	16.0742
B 32	17.1053
B 33	14.2210
B 34	15.9446
B 35	25.0349
B 36	15.8378
B 37	13.8607
B 38	10.6389
B 39	6.5363

Done @ 17H34
Back in oven
@ 17H35

13.02.04

~~18~~ 18H39

1025
1025
1027
6.8

no cups ⟸ F_{P_1} = 392.0 (all foamed up)

↳ even some on tubing

F_3 (P_2) = 376.3

↓

cups @ 18H43 - T = 27°C

cups	empty	+ GRA
B40	2.5266159	31.5468
B41	2.5528	27.4223

(decreasing fast

↳ in oven @ 92°C @ 18H48

out of oven @ 19H04

Weighing @ 19H17 - T = 27°C

B40 = 25.2298 and ↓
B41 = 21.7788 and ↓

Back in oven @ 19H18

13.02.04

360

Load acetone in F3

F3 + acetone = 416 g

↳ in cold room @ 19H02

at 7.2/7.3°C

P_1 = 1025 mbar
P_2 = 1025 mbar
P_3 = 1027 mbar

At 19H19

1025
1025
1027
6.8

At 21H08

1024
1024
1027
6.3

Acetone boiling point?

~114-115 mbar
P oscillate
not stable
and warm ↓

6.5 114.2/3

↳ 113.8

13.02.04

6.5°C 113.5 mbar

13.02.04
(3)

21H09

1025/1029
1024/1025
1027
6.5° C

Cups B40 & B41 out of oven @ 20H48

Weighing @ 21H19 - T = 27° C

B40 = 22.4979
B41 = 19.6172

back in oven
@ 21H22

Cups out of oven @ 23H04

Weighing @ 23H36 - T = 28°C

B40 = 22.0268
B41 = 19.2212

↳ back in oven at 92°C
@ 23H38

13.02.04

F3

empty 317.8
BE 399.5
AE 376.3

F4

392.0

78.7g acetone
61.2g GR

110.2 mbar 38.90%

Contact for A&G:

Severn Science Ltd

01454 414 723

13.02.04

Have also packed today the H-S cell and all its equipement, accessories and tools to bring them to Paris $\Rightarrow$ will set up the expt on the PMMP lab, as after all might not run the GRA tunnel tests at 5°C (need to compare the benefit of higher η at low T with a lower P_{sat} $\Rightarrow$ might be better to run GRA tests at room T. Can only decide when all EOS data will be available). Will also run in Paris bubbly GS tests when bubble-box is ready.

13.02.04

14.02.04

①

All cups out of oven @ 8H26
Let them cool down to room T

Weighing @ 09H05 - T = 27°C

	Cup	m(g)		Cup	m(g)
Tray 1	B1	11.8062	Tray 2 @ 09H09	B14	6.4112
	B2	20.5360		B15	12.4884
	B3	11.5976		B16	14.7870
	B4	19.2666		B17	18.6533
	B5	21.6271		B18	17.6507
	B6	16.0763		B19	23.8502
	B7	15.4580		B20	7.5095
	B8	13.8819		B21	18.4743
	B9	14.8165		B22	24.8579
	B10	13.0886		B23	26.2297
	B11	15.7379		B24	29.5758
	B12	18.4930		B25	31.7635
	B13	11.5350		B26	10.4275

Done @ 9H13

14.02.04

14.02.04
(2)

	Cup	m (g)	
Tray 3	B27	13.7169	@ 9H17
	B28	5.5040	
	B29	22.9991	
	B30	8.4085	
	B31	16.0521	
	B32	17.0788	
	B33	14.2045	
	B34	15.9229	
	B35	24.9366	
	B36	15.7811	
	B37	13.8249	
	B38	10.6190	
	B39	6.5304	
	B40	21.3338	
	B41	18.6483	Done @ 9H22

other cups → 7.1760 (reddish brown spots)
→ 25.1846

All cups back in oven @ 9H28

a few * * *
pale coiled
clear GR

14.02.04

→ home for packing

→ Ferry from Portsmouth to le Havre @ 15H30 !!!

left home @ 12:30
arrived @ P&O checking @ 14:50

Didn't manage to load all the capt in the car. Have to do one short return trip to pick up the H-S cell.

14.09.04

$\boxed{\text{GPA EoS}}$ = a few thoughts

14.02.04

(3)

1) run a test for several days
(or two in //)
and every now and then weigh the flask

⟹ m ↑ ? ? ?
→ time

Hopefully m ↑ ↘ as it equilibrates
→ time

but have to check if this is the case or not

↳ run w/ a very fluid compo as starting material (> ~ 60%)

↳ aim for
1) ~ 40%
2) ~ 20%
3) ~ 60%

↓

leave @ room T and P atm for a long time in closed flask

2) Rerun B5 for longer time

14.02.04

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$$(m_i - \cancel{m_{cup}}) - (m_F - \cancel{m_{cup}})$$

Cup

$$\% \text{ acetone} = \frac{m_{acetone}}{m_{GR+acetone}} = \frac{m_{GRA_i} - m_{GRA_f}}{m_{GRA_i}}$$

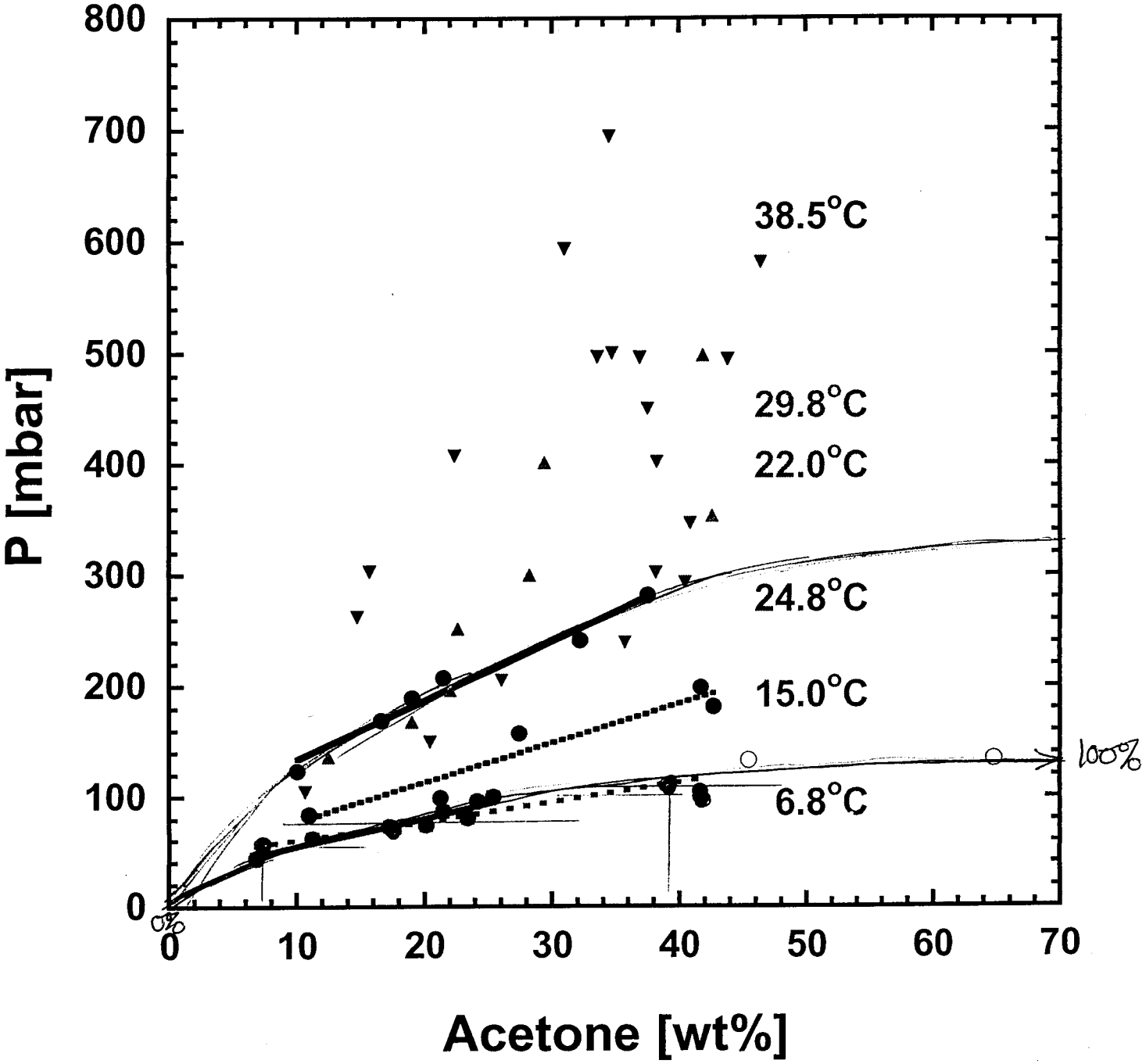
$$= \frac{m_{cup_i} - m_{cup_F}}{m_{cup_i} - m_{empty\ cup}} \times 100$$

Flask

$$\% \text{ acetone} = 1 - \frac{m_{TOT_i}}{m_{TOT_F}} (1 - \% \text{ acetone}_i)$$

14.02.04

GRA system



14.02.04

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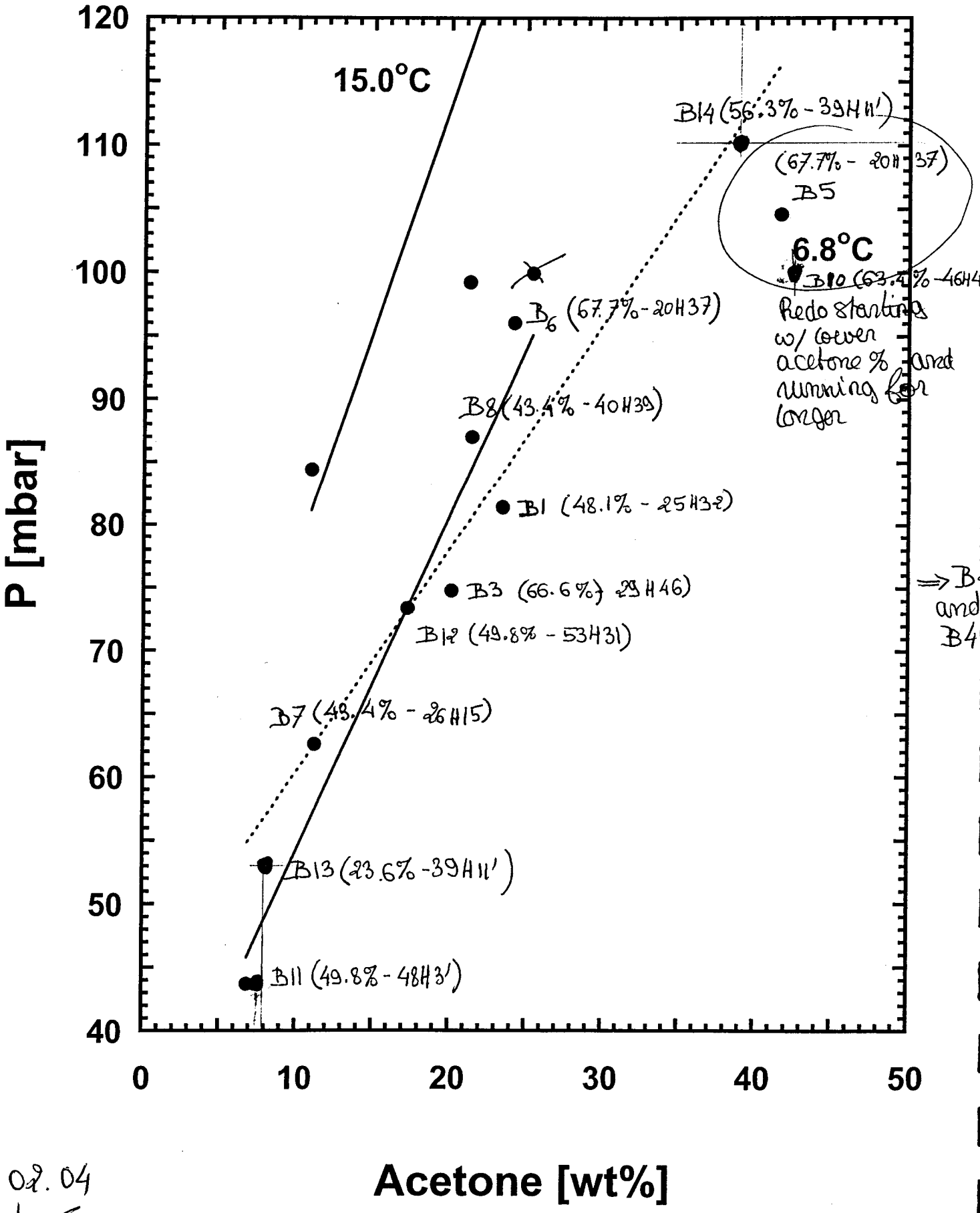
14.02.03

GRA system



14.02.04

In same Excel ~~spreadsheet~~ book

Summary table EOS runs

GRA sample # + date prepared
batch

Run #	Start date	Duration [min]	Initial A%	P [mbar]	T [°C]	Final A%	Comments

+

Cups before runs	Cup A%	Cups after runs	Cup A%

Summary GRA batches

One ~~spreadsheet~~ book per Temperature

Batch #	Preparation Date

$$\frac{m_{F+m+GRA} - m_{F+m+GR}}{m_{F+m+GRA} - m_{F+m}} \times 100$$

$m_{flask+magnet}$	m_{F+M+GR}	$m_{F+m+GR+acetone}$	% acetone	Cups #	Cup A%

Runs	Run date

Summary GRA cups

Cup #	Date	Batch or Run #	$m_{empty\ cup}$	initial $m_{cup+GRA}$	$m_{cup+GRA}$ @ ≠ time	A%

14.02.04

Runs summary (temporary results)

Run #	Initial acetone % (w/ cups)	Before run: m empty flask	Before run: m flask + GRA	After run: m flask + GRA	% acetone after run (w/ cups)
B9	63.44 (165/260.1)	317.4	429.8	←	Flask has
B10	Cups B26/B27/B28	319.9	402.9	372.5	42.30 (B29/B30)
B11	49.80 (152.7/306.6)	328.0 (F4)	432.1	384.1 + 0.421	7.55
B12	cups B31/B32 B33/B34	317.5 (F3)	406.0	371.2 + 0.034	17.33 (B36)
B13	23.64 (30.5/129) Cup B35	328.00 (F4)	406.4	392.0 + ~1	7.90
B14	56.25 (78.7/139.9) cups B37/B38 and B39	317.8 (F3)	399.5	376.3	38.9 (B40/B41)

14.02.04

P (mbar)	T (°C)	Duration
imploded →		
99.6	6.7	46H41'
43.6 (P1)	6.8	48H3'
73.4 (P2)	6.8	53H31'
53.1 (P1)	6.8	39H11'
110.2 (P2)	6.8	39H11'

14.02.04

Make a new excel spreadsheet for the cups with each cup having the following info displayed:

Cup #	
Date	
GRA batch and/or run#	
$m_{empty\ cup}$	$\leftarrow m_{cup}$
Time (min)	$m_{cup+GRA}$ (g)
0	- $\leftarrow m_i$
-	-
⋮	
-	- $\leftarrow m_f$
% A	

with $\% A = \dfrac{m_{acetone}}{m_{GRA}} = \dfrac{m_i - m_f}{m_i - m_{cup}} \times 100$

14.02.04

18.02.04

Cups out of oven
@ 9:30 (T = 92°C)

Pesé à 13H27

	Cup	m (g)
Tray 1	P1	14.0956
	P2	16.3714
	P3	16.3955
	P4	38.4943
	P5	18.1389
	P6	28.7935
	P7	27.8194
	P8	23.7692
	P9	18.5216
	P10	20.7392
	P11	14.5237
	P12	13.1181
	P13	28.3453 → 13H34
Tray 2	P14	15.0014
	P15	20.8527
	P16	7.7479
	P17	18.1606
	P18	12.9437
	P19	16.4074
	P20	15.5656
	P21	17.7554
	P22	13.3213
	P23	15.3464
	P24	15.9529
	P25	17.2710
	P26	13.3522 → 13H40

18.02.04

Tray 3

Cup	m(g)
P27	25.2015
P28	6.8620
P29	17.6268
P30	19.0914
P31	8.8147

→ done @ 13H43

→ Back in oven @ 92°C
@ 13H 50

Check the validation dates of the ASG calibration.

Phoned today Severn Science to discuss about connection between ASG controller and a PC ⇒ bring controller and all accessories (ASG, cables) plus PC to Bristol and phone from there to take appointment w/ service engineer (will leave message on answerphone on 20.02.04 to make arrangements).

18.02.04

SOUTHWEST RESEARCH INSTITUTE™

6220 Culebra Road, P.O. Drawer 28510
Institute Quality Systems
Institute Calibration Laboratory
Phone: 210-522-5215 Fax 210-522-3692

Certificate of Calibration

Submitted By:	DIV20	**Work Order:**	444055030
Address:	B189	**Date Issued:**	Aug 19, 2003
Contact:	BRITT HILL	**Calibration Date:**	Aug 19, 2003
Manufacturer Model:	EDWARDS D35726000	****Calibration Due:**	Feb 19, 2004
Description:	PRESSURE TRANSDUCER	**Calibration Location:**	Bldg. 64
Serial No:	005000176	**Environment:**	Temp. 68.0°F Hum. 40 %RH
Asset No:	007630	***As Found:**	IN TOLERANCE
Procedure:	CLCP-PI-001, JUN/99	***As Left:**	IN TOLERANCE

This certificate documents traceability to the National Institute of Standards and Technology (NIST) and the International System of Units (SI). The Laboratory quality system conforms to ISO/IEC 17025, 1999 and ANSI/NCSL Z540-1-1994 which are equivalent to relevant requirements of the ISO 9000-1994 series of standards. This certificate may not be reproduced, except in full, without the written approval of the Southwest Research Institute Calibration Laboratory. The results of this calibration relate only to the individual instrument described above. This certificate shall not be used to claim product endorsement by the American Association for Laboratory Accreditation (A2LA) or any agency of the U. S. Government.

Uncertainty evaluation includes the item under test and is calculated in accordance with the ISO "Guide to the Expression of Uncertainty in Measurement" (GUM). The uncertainty represents an expanded uncertainty using a coverage factor of k=2 to approximate a 95% confidence level. The calibration process provides a Test Uncertainty Ratio (TUR) of less than or equal to 25% (4:1) of the test limit unless otherwise stated in remarks or an attachment.

*The client has sole responsibility for determination of in/out of tolerance or compliance/noncompliance. An in/out of tolerance opinion is provided for your convenience based only on the Test Instrument (TI) reading(s) and limits as reported. The reported uncertainty relates only to the results at the time of calibration and does not imply any short or long term stability of the TI.

**Calibration interval is determined by the client and does not assure the instrument will remain within tolerance until this date. Any number of factors may cause the instrument to be out of tolerance before the next calibration date.

Remarks: None

Standards Used

Asset	Manufacturer	Model	Description	Cal Due
002079	RUSKA	6200	PRESSURE GAUGE	Mar 20, 04

Approved by: Walt Hill
Metrology Group Leader
m:\Nona2la1.rpt Rev date 15, August 02

Measurements by: Perry Carpenter
Metrology Technician

18.02.04

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Southwest Research Institute
Calibration Laboratory
Calibration Report

Work Order	444055030	Mfr	Edwards	Technician	PWC
Asset No.	007630	Model	D35726000		
Serial No.	5000176	Type	Pressure Indicator	Cal Date	19-Aug-03
Remarks:					

Function/Range	Test Point	TI Reading	Difference	+/-Limit	+/-Uncertainty	Found/Left
Pressure	mBar	mBar	mBar	mBar	mBar	Result
	0.000	0.0000	0.000	2.0000	0.197	Pass
	250.000	249.7000	-0.300	2.0000	0.197	Pass
	500.000	499.3000	-0.700	2.0000	0.197	Pass
	750.000	749.4000	-0.600	2.0000	0.197	Pass
	1000.000	999.2000	-0.800	2.0000	0.197	Pass
	750.000	749.4000	-0.600	2.0000	0.197	Pass
	500.000	499.4000	-0.600	2.0000	0.197	Pass
	250.000	249.6000	-0.400	2.0000	0.197	Pass
	0.000	-0.2000	-0.200	2.0000	0.197	Pass

END OF REPORT

18.02.04

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Institute Quality Systems
Institute Calibration Laboratory
Phone: 210-522-5215 Fax 210-522-3692

Certificate of Calibration

Submitted By: DIV20
Address: B189
Contact: BRITT HILL
Manufacturer Model: EDWARDS D35726000
Description: PRESSURE TRANSDUCER
Serial No: 005000055
Asset No: 008236
Procedure: CLCP-PI-001, JUN/99

Work Order: 444055031
Date Issued: Aug 19, 2003
Calibration Date: Aug 19, 2003
****Calibration Due:** Feb 19, 2004
Calibration Location: Bldg. 64
Environment: Temp. 68.0°F Hum. 40 %RH
***As Found:** IN TOLERANCE
***As Left:** IN TOLERANCE

This certificate documents traceability to the National Institute of Standards and Technology (NIST) and the International System of Units (SI). The Laboratory quality system conforms to ISO/IEC 17025, 1999 and ANSI/NCSL Z540-1-1994 which are equivalent to relevant requirements of the ISO 9000-1994 series of standards. This certificate may not be reproduced, except in full, without the written approval of the Southwest Research Institute Calibration Laboratory. The results of this calibration relate only to the individual instrument described above. This certificate shall not be used to claim product endorsement by the American Association for Laboratory Accreditation (A2LA) or any agency of the U. S. Government.

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**Calibration interval is determined by the client and does not assure the instrument will remain within tolerance until this date. Any number of factors may cause the instrument to be out of tolerance before the next calibration date.

Remarks: None

Standards Used

Asset	Manufacturer	Model	Description	Cal Due
002079	RUSKA	6200	PRESSURE GAUGE	Mar 20, 04

Approved by: Walt Hill
Metrology Group Leader
m:\Nona2la1.rpt Rev date 15, August 02

Measurements by: Perry Carpenter
Metrology Technician

18.02.04

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Southwest Research Institute
Calibration Laboratory
Calibration Report

Work Order	444055031	Mfr	Edwards	Technician	PWC
Asset No.	008236	Model	D35726000		
Serial No.	500055	Type	Pressure Transducer	Cal Date	19-Aug-03

Remarks:

Function/Range	Test Point	TI Reading	Difference	+/-Limit	+/-Uncertainty	Found
Pressure	mBar	mBar	mBar	mBar	mBar	Result
	0.000	0.8000	0.800	2.0000	0.197	Pass
	250.000	251.0000	1.000	2.0000	0.197	Pass
	500.000	501.3000	1.300	2.0000	0.197	Pass
	750.000	751.6000	1.600	2.0000	0.197	Pass
	1000.000	1001.0000	1.000	2.0000	0.197	Pass
	750.000	751.5000	1.500	2.0000	0.197	Pass
	500.000	501.2000	1.200	2.0000	0.197	Pass
	250.000	251.1000	1.100	2.0000	0.197	Pass
	0.000	0.7000	0.700	2.0000	0.197	Pass

END OF REPORT

18.02.04

Southwest Research Institute
Calibration Laboratory
Calibration Report

Work Order	444055031	Mfr	Edwards	Technician	PWC
Asset No.	008236	Model	D35726000		
Serial No.	5000055	Type	Pressure Transducer	Cal Date	19-Aug-03

Remarks:

Function/Range	Test Point	TI Reading	Difference	+/-Limit	+/-Uncertainty	Left
Pressure	mBar	mBar	mBar	mBar	mBar	Result
	0.000	0.1000	0.100	2.0000	0.197	Pass
	250.000	250.3000	0.300	2.0000	0.197	Pass
	500.000	500.4000	0.400	2.0000	0.197	Pass
	750.000	750.7000	0.700	2.0000	0.197	Pass
	1000.000	1000.0000	0.000	2.0000	0.197	Pass
	750.000	750.5000	0.500	2.0000	0.197	Pass
	500.000	500.4000	0.400	2.0000	0.197	Pass
	250.000	250.3000	0.300	2.0000	0.197	Pass
	0.000	0.1000	0.100	2.0000	0.197	Pass

END OF REPORT

18.09.04

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Institute Calibration Laboratory
Phone: 210-522-5215 Fax 210-522-3692

Certificate of Calibration

Submitted By:	DIV20	**Work Order:**	444055028
Address:	B189	**Date Issued:**	Aug 19, 2003
Contact:	BRITT HILL	**Calibration Date:**	Aug 19, 2003
Manufacturer Model:	EDWARDS D35726000	****Calibration Due:**	Aug 19, 2004
Description:	PRESSURE TRANSDUCER	**Calibration Location:**	Bldg. 64
Serial No:	005000333	**Environment:**	Temp. 68.0°F Hum. 40 %RH
Asset No:	008464	***As Found:**	IN TOLERANCE
Procedure:	CLCP-PI-001, JUN/99	***As Left:**	IN TOLERANCE

This certificate documents traceability to the National Institute of Standards and Technology (NIST) and the International System of Units (SI). The Laboratory quality system conforms to ISO/IEC 17025, 1999 and ANSI/NCSL Z540-1-1994 which are equivalent to relevant requirements of the ISO 9000-1994 series of standards. This certificate may not be reproduced, except in full, without the written approval of the Southwest Research Institute Calibration Laboratory. The results of this calibration relate only to the individual instrument described above. This certificate shall not be used to claim product endorsement by the American Association for Laboratory Accreditation (A2LA) or any agency of the U. S. Government.

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**Calibration interval is determined by the client and does not assure the instrument will remain within tolerance until this date. Any number of factors may cause the instrument to be out of tolerance before the next calibration date.

Remarks: None

Standards Used

Asset	Manufacturer	Model	Description	Cal Due
002079	RUSKA	6200	PRESSURE GAUGE	Mar 20, 04

Approved by: Walt Hill
Metrology Group Leader

Measurements by: Perry Carpenter
Metrology Technician

m:\Nona2la1.rpt Rev date 15, August 02

18.09.04

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Southwest Research Institute
Calibration Laboratory
Calibration Report

Work Order	444055028	Mfr	Edwards	Technician	PWC
Asset No.	008464	Model	D35726000		
Serial No.	5000333	Type	Pressure Transducer	Cal Date	19-Aug-03
Remarks:					

Function/Range	Test Point	TI Reading	Difference	+/-Limit	+/-Uncertainty	Found
Pressure	mBar	mBar	mBar	mBar	mBar	Result
	0.000	1.9000	1.900	2.0000	0.197	Pass
	250.000	251.4000	1.400	2.0000	0.197	Pass
	500.000	500.9000	0.900	2.0000	0.197	Pass
	750.000	750.6000	0.600	2.0000	0.197	Pass
	1000.000	1000.0000	0.000	2.0000	0.197	Pass
	750.000	750.5000	0.500	2.0000	0.197	Pass
	500.000	500.9000	0.900	2.0000	0.197	Pass
	250.000	251.4000	1.400	2.0000	0.197	Pass
	0.000	1.9000	1.900	2.0000	0.197	Pass

END OF REPORT

18.02.04

Southwest Research Institute
Calibration Laboratory
Calibration Report

Work Order	444055028	Mfr	Edwards	Technician	PWC
Asset No.	008464	Model	D35726000		
Serial No.	5000333	Type	Pressure Transducer	Cal Date	19-Aug-03
Remarks:					

Function/Range	Test Point	TI Reading	Difference	+/-Limit	+/-Uncertainty	Left
Pressure	mBar	mBar	mBar	mBar	mBar	Result
	0.000	0.1000	0.100	2.0000	0.197	Pass
	250.000	249.7000	-0.300	2.0000	0.197	Pass
	500.000	499.1000	-0.900	2.0000	0.197	Pass
	750.000	748.7000	-1.300	2.0000	0.197	Pass
	1000.000	998.4000	-1.600	2.0000	0.197	Pass
	750.000	748.8000	-1.200	2.0000	0.197	Pass
	500.000	499.2000	-0.800	2.0000	0.197	Pass
	250.000	249.6000	-0.400	2.0000	0.197	Pass
	0.000	0.1000	0.100	2.0000	0.197	Pass

END OF REPORT

18.02.04

18.02.04

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Institute Quality Systems
Institute Calibration Laboratory
Phone: 210-522-5215 Fax 210-522-3692

Certificate of Calibration

Submitted By:	DIV20	**Work Order:**	444055029
Address:	B189	**Date Issued:**	Aug 19, 2003
Contact:	BRITT HILL	**Calibration Date:**	Aug 19, 2003
Manufacturer Model:	EDWARDS D35726000	****Calibration Due:**	Aug 19, 2004
Description:	PRESSURE TRANSDUCER	**Calibration Location:**	Bldg. 64
Serial No:	005000371	**Environment:**	Temp. 68.0°F Hum. 40 %RH
Asset No:	008466	***As Found:**	IN TOLERANCE
Procedure:	CLCP-PI-001, JUN/99	***As Left:**	IN TOLERANCE

This certificate documents traceability to the National Institute of Standards and Technology (NIST) and the International System of Units (SI). The Laboratory quality system conforms to ISO/IEC 17025, 1999 and ANSI/NCSL Z540-1-1994 which are equivalent to relevant requirements of the ISO 9000-1994 series of standards. This certificate may not be reproduced, except in full, without the written approval of the Southwest Research Institute Calibration Laboratory. The results of this calibration relate only to the individual instrument described above. This certificate shall not be used to claim product endorsement by the American Association for Laboratory Accreditation (A2LA) or any agency of the U. S. Government.

Uncertainty evaluation includes the item under test and is calculated in accordance with the ISO "Guide to the Expression of Uncertainty in Measurement" (GUM). The uncertainty represents an expanded uncertainty using a coverage factor of k=2 to approximate a 95% confidence level. The calibration process provides a Test Uncertainty Ratio (TUR) of less than or equal to 25% (4:1) of the test limit unless otherwise stated in remarks or an attachment.

*The client has sole responsibility for determination of in/out of tolerance or compliance/noncompliance. An in/out of tolerance opinion is provided for your convenience based only on the Test Instrument (TI) reading(s) and limits as reported. The reported uncertainty relates only to the results at the time of calibration and does not imply any short or long term stability of the TI.

**Calibration interval is determined by the client and does not assure the instrument will remain within tolerance until this date. Any number of factors may cause the instrument to be out of tolerance before the next calibration date.

Remarks: None

Standards Used

Asset	Manufacturer	Model	Description	Cal Due
002079	RUSKA	6200	PRESSURE GAUGE	Mar 20, 04

Approved by: Walt Hill
Metrology Group Leader

Measurements by: Perry Carpenter
Metrology Technician

m:\Nona2la1.rpt Rev date 15, August 02

8.02.04

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Southwest Research Institute
Calibration Laboratory
Calibration Report

Work Order	444055029	Mfr	Edwards	Technician	PWC
Asset No.	008466	Model	D35726000		
Serial No.	5000371	Type	Pressure Transducer	Cal Date	19-Aug-03
Remarks:					

Function/Range	Test Point	TI Reading	Difference	+/-Limit	+/-Uncertainty	Found
Pressure	mBar	mBar	mBar	mBar	mBar	Result
	0.000	2.0000	2.000	2.0000	0.197	Pass
	250.000	252.0000	2.000	2.0000	0.197	Pass
	500.000	501.8000	1.800	2.0000	0.197	Pass
	750.000	751.6000	1.600	2.0000	0.197	Pass
	1000.000	1001.0000	1.000	2.0000	0.197	Pass
	750.000	751.6000	1.600	2.0000	0.197	Pass
	500.000	501.6000	1.600	2.0000	0.197	Pass
	250.000	251.7000	1.700	2.0000	0.197	Pass
	0.000	1.7000	1.700	2.0000	0.197	Pass

END OF REPORT

18.02.04

Southwest Research Institute
Calibration Laboratory
Calibration Report

Work Order	444054029	Mfr	Edwards	Technician	PWC
Asset No.	008466	Model	D35726000		
Serial No.	5000371	Type	Pressure Transducer	Cal Date	19-Aug-03
Remarks:					

Function/Range	Test Point	TI Reading	Difference	+/-Limit	+/-Uncertainty	Left
Pressure	mBar	mBar	mBar	mBar	mBar	Result
	0.000	0.0000	0.000	2.0000	0.197	Pass
	250.000	250.0000	0.000	2.0000	0.197	Pass
	500.000	500.0000	0.000	2.0000	0.197	Pass
	750.000	750.0000	0.000	2.0000	0.197	Pass
	1000.000	999.8000	-0.200	2.0000	0.197	Pass
	750.000	750.0000	0.000	2.0000	0.197	Pass
	500.000	500.0000	0.000	2.0000	0.197	Pass
	250.000	250.1000	0.100	2.0000	0.197	Pass
	0.000	0.2000	0.200	2.0000	0.197	Pass

END OF REPORT

18.02.04

18.02.04

18.02.04

ASG Sn 005000176
Am 007630

and ASG Sn 005000055
Am 008236

which were last calibrated on August 19, 2003 at SwRI Cal Lab by PWC are now due, on Feb 19 2004 for calibration and need to be sent by Fed Ex w/ the controller to SwRI. (*)
ASG Sn 005000176 was last used on 13.02.04.
These 2 ASG won't be used until calibrated again.

(*) the alternative would be to have them calibrated by the manufacturer BOC-Edwards (= Severn Science in Bristol, U.K.). Check with Britt if QA would be happy w/ that solution, which would be faster and probably cheaper. It would also allow the ASG to be fully checked by the manufacturer after several years of usage ⟹ something that would be very useful

18.02.04

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22.02.04 Back in Bristol on 20.02.04 to pick up the H-S cell.

① All cups out of the oven @ 11H40 => load can

Weighing @ 13H58 - 27°C

	Cup	m (g)
Tray 1	B1	11.7942
	B2	20.5099
	B3	11.5862
	B4	19.2376
	B5	21.5958
	B6	16.0573
	B7	15.4388
	B8	13.8674
	B9	14.8008
	B10	13.0752
	B11	15.7191
	B12	18.4697
	B13	11.5252
Tray 2	B14	6.4071
	B15	12.4767
	B16	14.7705
	B17	18.6323
	B18	17.6317
	B19	23.8231
	B20	7.5046
	B21	18.4538
	B22	24.8149
	B23	26.2008
	B24	29.5327
	B25	31.7119
	B26	10.3684

@ 14H03

Done @ 14H07

22.02.04

22.02.04

(2)

	Cup	m (g)	
Tray 3	B 27	13.6292	@ 14 H 11
	B 28	5.4906	
	B 29	22.663 9	
	B 30	8.3477	
	B 31	15. 9320	
	B 32	16.9351	
	B 33	14.1208	
	B 34	15.8063	
	B 35	24.6075	
	B 36	15.6078	
	B 37	13.7261	
	B 38	10.5605	
Tray 4	B 39	6.5134	@ 14 H 15
	B 40	20.6656	
	B 41	18.0870	
	GR1	25.1867	
	GR2	7.1778	

Note: Talked w/ Severn Science over the phone on 22.02.04 regarding ASG controller. Will have to spend a few days working at their office in April.

All back in oven @ 92°C @ 14 H 18

Crossing from Portsmouth to Le Havre tonight.

22.02.04

Date: Mon, 01 Mar 2004 13:39:27 -0600
From: Brittain Hill <bhill@cnwra.swri.edu>
Reply-To: bhill@swri.org
To: Anne-Marie Lejeune <amlejeun@ccr.jussieu.fr>
Subject: GRA cups

Anne-Marie - I have FINALEMENT weighted the old GRA cups, which have been in the drying oven (90C) for over a year. Attached is the excel file with the data. One of the cups went missing. But at least this time, the nosy kids kept their fingers out of the oven and didn't spill a bunch of the cups.

One unusual thing was that nearly all the cups had a flaky top layer, much like a thin glass rind that was inflated over the main surface. Looks like outgassing had raised a bubbly layer, which then burst. For many of these cups, there were flakes of GRA glass shards that landed outside the cup. May have been from circulation of air in the oven, or glass fracturing itself. I've marked with "flakes" the samples that clearly had flakes of GRA around the cups.

Many of the low-volume samples seemed to have gained a bit of weight. Some of these were next to more completely filled cups that had lots of flakes at the surface, so it is possible that some of the flakes deposited in the surrounding cups, with small weight cups being more sensitive to the additions. Perhaps these weight increases give us a measure of uncertainty from "flying flakes", in that any cup could have weight fluctuations in this range due to flakiness. Ugh.

About the only sample that didn't flake was the GRA from BRISTOL, which remained a nice, clear glass throughout. There was essentially no weight change in this sample.

Bottom line is I'm not sure how useful these long-term data will be, given the potential for mass loss due to flake formation. Hopefully you can extrapolate the acetone degassing curve from your existing data, with these last measurement being confirmatory rather than baseline for the curve fitting.

Hope your eutectics went better last week!

Britt

--

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In Magma Veritas

[Part 2, Application/MSEXCEL 59KB.]

[Unable to print this part.]

01.03.04

From Britt (01.03.2004)

s/n	Date	s/n	Date	s/n	Date	s/n	Date
2883	18/04/2002	2883	09/05/2002	N45601	11/07/2002	N45601	01/03/2004
23.2	Temp (C)	23.3	Temp (C)	23	Temp (C)	Temp (C)	23
Weight(g)	Sample #	Weight(g)	Sample #	Weight(g)	Sample #	Weight(g)	Sample #
8.823	1	8.821	1	8.814	1	8.814	1
19.978	2	19.969	2	19.957	2	19.942	2
17.344	3	17.338	3	17.328	3	17.315	3
17.811	4	17.794	4	17.779	4	spill	4
12.775	5	12.768	5	12.761	5	12.747	5
17.935	6	17.92	6	17.888	6	17.870	6
8.377	7	8.377	7	8.372	7	8.367	7
24.84	8	24.82	8	24.801	8	24.770	8
7.333	9	7.327	9	7.328	9	7.339	9
20.74	10	20.722	10	20.708	10	spill	10
18.279	11	18.254	11	18.246	11	spill	11
5.348	12	5.348	12	5.348	12	spill	12
17.929	13	17.918	13	17.9	13	17.795	13
13.373/4	14	13.366	14	13.339	14	13.330	14
8.719	15	8.719	15	8.719	15	8.713	15
13.924	16	13.913	16	13.905	16	13.883	16
13.813	17	13.804	17	13.789	17	13.781	17
18.808	18	18.792	18	18.785	18	spill	18
13.533	19	13.526	19	13.521	19	spill	19
7.326	20	7.326	20	7.324	20	7.316	20
15.659	21	15.642	21	15.626	21	15.608	21
15.268	22	15.252	22	15.235	22	15.220	22
9.399	23	9.398	23	9.388	23	9.383	23
16.848	24	16.833	24	16.813	24	16.795	24
8.641	25	8.638	25	8.626	25	8.620	25
11.226	26	11.225	26	11.219	26	11.208	26
6.433	27	6.433	27	6.428	27	6.418	27
11.201	28	11.152	28	11.098	28	11.071	28
13.336	29	13.286	29	13.24	29	13.213	29
17.551	30	17.477	30	17.43	30	17.395	30
23.368	31	23.254	31	23.175	31	23.130	31
19.522	32	19.449	32	19.41	32	19.369	32
24.456	33	24.347	33	24.272	33	24.220	33
24.281	34	24.146	34	24.082	34	24.023	34
23.603	35	23.485	35	23.422	35	spill	35
3.925	36	3.924	36	3.917	36	3.913	36
4.943	37	4.943	37	4.928	37	4.927	37
17.014	38	16.943	38	16.905	38	16.875	38
7.945	39	7.927	39	7.914	39	7.884	39
13.609	40	13.568	40	13.518	40	13.458	40
13.975	41	13.926	41	13.886	41	13.858	41

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s/n	Date	s/n	Date	s/n	Date	s/n	Date
2883	18/04/2002	2883	09/05/2002	N45601	11/07/2002	N45601	01/03/2004
23.2	Temp (C)	23.3	Temp (C)	23	Temp (C)	Temp (C)	23
Weight(g)	Sample #	Weight(g)	Sample #	Weight(g)	Sample #	Weight(g)	Sample #
11.586	42	11.567	42	11.555	42	11.533	42
19.328	43	19.29	43	19.257	43	19.221	43
12.968	44	12.942	44	12.923	44	spill	44
9.782	45	9.77	45	9.751	45	9.731	45
9.392	46	9.372	46	9.359	46	9.337	46
24.775	47	24.705	47	24.632	47	24.556	47
12.248	48	12.216	48	12.198	48	12.172	48
17.582	50	17.524	50	17.495	50	17.451	50
13.051	51	13.018	51	12.997	51	12.974	51
17.786	52	17.73	52	17.697	52	17.655	52
10.286	53	10.256	53	10.242	53	10.219	53
20.351	54	20.288	54	20.241	54	20.185	54
9.853	55	9.832	55	9.819	55	9.803	55
12.156	56	12.135	56	12.115	56	12.095	56
7.615	57	7.602	57	7.599	57	7.586	57
12.613	58	12.581	58	12.558	58	12.523	58
7.242	59	7.236	59	7.227	59	7.218	59
16.214	60	16.163	60	16.137	60	16.105	60
14.224	61	14.186	61	14.164	61	14.136	61
8.642	62	8.63	62	8.617	62	8.594	62
14.298	63	14.252	63	14.222	63	14.186	63
9.459	64	9.442	64	9.422	64	9.404	64
18.885	65	18.823	65	18.773	65	18.716	65
10.805	66	10.775	66	10.752	66	10.726	66
11.43	67	11.396/7	67	11.373	67	11.347	67
6.676	68	6.662	68	6.658	68	6.636	68
7.889	69	7.879	69	7.866	69	7.850	69
5.284	70	5.281	70	5.274	70	5.267	70
5.978	71	5.973	71	5.97	71	5.958	71
5.481	72	5.472	72	5.471	72	5.464	72
6.722	73	6.707	73	6.698	73	spill	73
10.502	74	10.454	74	10.431	74	10.407	74
18.402	75	18.306	75	18.251	75	18.189	75
11.973	76	11.929	76	11.911	76	spill	76
21.338	77	21.228	77	21.179	77	21.102	77
19.722	4'	19.713	4'	19.695	04'	spill	04'
15.025	12'	15.008	12'	14.988	12'	14.974	12'
18.944	13'	18.928	13'	18.909	13'	spill	13'
15.223	14'	15.211	14'	15.19	14'	15.176	14'
7.887	16'	7.887	16'	7.885	16'	7.886	16'
18.149	17'	18.129	17'	18.127	17'	18.104	17'

01.03.04

s/n	Date	s/n	Date	s/n	Date	s/n	Date
2883	18/04/2002	2883	09/05/2002	N45601	11/07/2002	N45601	01/03/2004
23.2	Temp (C)	23.3	Temp (C)	23	Temp (C)	Temp (C)	23
Weight(g)	Sample #	Weight(g)	Sample #	Weight(g)	Sample #	Weight(g)	Sample #
16.274	18'	16.261	18'	16.252	18'	16.236	18'
11.713	19'	11.713	19'	11.701	19'	spill	19'
16.146	20'	16.132	20'	16.116	20'	16.105	20'
15.678	21'	15.658	21'	15.641	21'	15.631	21'
16.984	22'	16.955	22'	16.937	22'	16.917	22'
15.889	24'	15.875	24'	15.86	24'	15.843	24'
11.69	26'	11.685	26'	11.674	26'	11.660	26'
19.732	34'	19.627	34'	19.585	34'	19.545	34'
17.906	35'	17.846	35'	17.793	35'	17.756	35'
12.828	36'	12.781	36'	12.749	36'	12.728	36'
11.897	36"	11.86	36"	11.826	36"	11.806	36"
16.842	38'	16.781	38'	16.745	38'	16.695	38'
17.832	38"	17.747	38"	17.682	38"	17.625	38"
15.626	40'	15.573	40'	15.521	40'	15.431	40'
15.068	41'	15.005	41'	14.95	41'	14.885	41'
13.887	42'	13.863	42'	13.847	42'	13.826	42'
15.236	44'	15.2	44'	15.174	44'	15.150	44'
20.779	50'	20.715	50'	20.677	50'	20.622	50'
18.849	52'	18.802	52'	18.764	52'	18.712	52'
17.94	52"	17.884	52"	17.843	52"	17.800	52"
23.962	54'	23.884	54'	23.833	54'	23.766	54'
10.912	54"	10.883	54"	10.866	54"	10.841	54"
10.984	56'	10.964	56'	10.945	56'	10.928	56'
13.028	56"	12.994	56"	12.974	56"	12.950	56"
12.964	58'	12.936	58'	12.914	58'	12.889	58'
17.17	60'	17.118	60'	17.085	60'	16.964	60'
8.983	61'	8.962	61'	8.953	61'	spill	61'
5.712	65'	5.707	65'	5.696	65'	5.689	65'
12.482	67'	12.433	67'	12.403	67'	12.381	67'
7.382	69'	7.37	69'	7.36	69'	spill	69'
7.332	69"	7.319	69"	7.31	69"	7.292	69"
7.053	69'''	7.042	69'''	7.034	69'''	spill	69'''
8.066	71'	8.053	71'	8.046	71'	8.031	71'
6.088	71"	6.086	71"	6.073	71"	6.069	71"
6.93	71'''	6.918	71'''	6.908	71'''	6.894	71'''
14.888	75'	14.829	75'	14.792	75'	14.756	75'
11.49	77'	11.444	77'	11.411	77'	11.389	77'
16.579	GRBristol	16.567	GRBristol	16.56	GRBristol	16.544	GRBristol

01.03.04

Will have to take the "flakiness" effect into account for all the Texas cups (T1 to T77'). and estimate an error bar.

01.03.04

All cups out of oven @ 11H26 (T_{oven} = 94°C
T_{set} = 88°C)

Weighing at 13H55

	Cup	m(g)	
Tray 1	P1	14.0691/2	
	P2	16.3361	
	P3	16.3661	
	P4	38.3865	
	P5	18.0912	
	P6	28.7102	
	P7	27.747817	
	P8	23.6954	
	P9	18.4747	
	P10	20.6813	
	P11	14.4923	
	P12	13.0932	
	P13	28.2854/5	
Tray 2	P14	14.9698	14H03
	P15	20.7943/4	
	P16	7.7358	
	P17	18.1019	
	P18	12.9068	
	P19	16.3579	
	P20	15.5184	
	P21	17.703 4	

03.03.04

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	Cup	m(g)
	P22	13.2795
	P23	15.3008
	P24	15.8946
	P25	17.2141
	P26	13.3187
Tray 3	P27	25.0821
	P28	6.8468
	P29	17.5644
	P30	19.0324
	P31	8.7934

@ 14H09

↳ All Back in oven @ 94°C

@ 14H12

Note from Chung Choi in Bristol: A service engineer from Southern Counties Calibration will be visiting the Dpt on 11.03.04 to carry out the annual servicing and calibration of balances.
Last time, I used the Bristol balance (OHAUS model GA 200 D - serial 3072) was on 14.02.04 while it was still under valid calibration.

03.03.04

5.03.2004

①

All cups out of oven @ 10H08 (T_{oven} = 94°C)

Weighing @ 11H30

	Cup	m (g)
Tray 1	P1 (26)	14.0577
	P2 (27)	16.3186
	P3	16.3494
	P4	38.3282
	P5	18.0685
	P6	28.6702
	P7	27.7096
	P8	23.6572
	P9	18.4545
	P10	20.6550
	P11	14.4805
	P12	13.0837
	P13	28.2522
Tray 2	P14	14.9566
	P15	20.7690
	P16	7.7306
	P17	18.0762
	P18	12.8920
	P19	16.3359
	P20	15.4983

@ 11H37

15.03.04

15.03.2004

(2)

402

	cup	m (g)	
	P21	17.6759	
	P22	13.2648	
	P23	15.2782	
	P24	15.8736	
	P25	17.1820	
	P26	13.3022	
Tray 3	P27	25.0270	@ 11H44
	P28	6.8404	
	P29	17.5313	
	P30	18.9972	
	P31	8.7870	

Done @ 11H45

All cups
↳ Back in oven @ 94°C
@ 11H47

15.03.04

24.03.04
(1)

All cups out of oven @ 11H35

↳ covered up w/ papers to avoid loss and/or contamination by "jumping" GR chips when cooling

Weighing @ 15H23

	Cup	m(g)
Tray 1	P1	14.0468
	P2	16.3050
	P3	16.3327
	P4	38.2780
	P5	18.0525
	P6	28.6414
	P7	27.6742
	P8	23.6257
	P9	18.4383
	P10	20.6401
	P11	14.4701
	P12	13.0751
	P13	28.2251
Tray 2	P14	14.9453 @ 15H30
	P15	20.7508
	P16	7.7260
	P17	18.0563
	P18	12.8800

24.03.04

24.03.04

(2)

404

Cup	m (g)
P19	16.3190
P20	15.4828
P21	17.6542
P22	13.2533
P23	15.2628
P24	15.8575
P25	17.1616
P26	13.2880
P27	24.9836
Tray 3 P28	6.8355
P29	17.5087
P30	18.9711
P31	8.7799

@ 15H35

All cups back in oven @ 15H38 with papers covering them to avoid loss of chips.

24.03.04

02.04.04

① 405

All cups out of oven @ 10H58
let cool down to ambiant w/ paper sheets covering to prevent flakes loss
weighing @ 12H00

	Cup	m(g)	
	P1	14.0378	
	P2	16.2934	
	P3	16.3210	
	P4	38.2457	
	P5	18.0357	
	P6	28.6196	
	P7	27.6495	
	P8	23.6027	
	P9	18.4250	
	P10	20.6276	
	P11	14.4625	
	P12	13.0682	
	P13	28.1939	
Tray 2	P14	14.9335	12H08
	P15	20.7351	
	P16	7.7226	
	P17	18.0413	
	P18	12.8718	
	P19	16.3038	
	P20	15.4688	

02.04.04

02.04.04
②

	Cup	m(g)	
	P21	17.6360	
	P22	13.2446	
	P23	15.2503	
	P24	15.8454	
	P25	17.1364	
	P26	13.2779	
Tray 3	P27	24.9496	12H15
	P28	6.8319	
	P29	17.4901	
	P30	18.9496	
	P31	8.7746	

All cups back in oven @ 12H19 @ 94°C
w/ paper sheet on top to prevent flakes loss.

02.04.04

09.04.04

①

Cups out of oven @ 11H10

Weighing @ 17H25

	Cup	m(g)
	P1	14.0309
	P2	16.2847
	P3	16.3117
	P4	38.2147
	P5	18.0253
	P6	28.6027
	P7	27.6322
	P8	23.5811
	P9	18.415 3
	P10	20.6133
	P11	14.4575
	P12	13.0633
	P13	28.1775
← 17H31	P14	14.9245
	P15	20.7225
	P16	7.7197
	P17	18.0264
	P18	12.8641
	P19	16.2914
	P20	15.4582

09.04.04

408

09.04.04

②

Cup	m(g)
P21	17.6247
P22	13.2361
P23	15.2392
P24	15.8340
P25	17.1221
P26	13.2698
17H37 →	
P28	6.8296
P27	24.9217
P29	17.4762
P30	18.9318
P31	8.7717

All Back in oven @ 17H41
at 94°C
w/ sheet on top

09.04.04

13.04.04

① Crossed the Channel this morning from Le Havre to Portsmouth

All B-caps out of oven at 17H45 (summertime)
↳ 16H45 wintertime

Paper sheets to cover them up & prevent loss of chips.

Weighing @ 18H25 - $T_{room} = 27°C$
w/ OHAOS GA 200 (last calibrated by SCC on 11.03.2004)

13.04.04

13.04.04

(2) 410

	Cup	m (g)
Tray 1	B1	11.768 8
	B2	20.4231
	B3	11.558 1
	B4	19.1567
	B5	21.4864
	B6	16.0147
	B7	15.3932
	B8	13.8309
	B9	14.757 6
	B10	13.0438
	B11	13.0439
	B12	18.4051
	B13	11.500 2
Tray 2	B14	6.3986
	B15	12.4453
	B16	14.7240
	B17	18.5736
	B18	17.57 69
	B19	23.7382
	B20	7.4896
	B21	18.3868
	B22	24.7180
	B23	26.090 3

@ 18H35

13.04.04

13.04.04
(3)

	Cup	m (g)	
	B24	29.3707	
	B25	31.5400	
	B26	10.2771	→ 18 H 41
Tray 3	B27	13.4950	→ 18 H 45
	B28	5.4752	
	B29	22.35	19
	B30	8.2663	
	B31	15.8048	
	B32	16.7957	
	B33	14.0021	
	B34	15.6745	
	B35	24.4405	
	B36	15.4306	
	B37	13.6512	
	B38	10.5244	
Tray 4	B39	6.5067	
	B40	20.5143	
	B41	17.9732	
	cup test 1	7.1788	
	cup test 2	25.1843	→ Done @ 18 H 52

All back in oven covered w/ paper sheet @ 18 H 55
(cracking noise)
Paper sheets removed @ 19 H 25

13.04.04

Sent the floppy disk containing the Edwards drivers for the ASG controller to Severn Science on 19.04.04, as there might be a problem with it and/or some files.

After several discussions and tests, it turns out that there were several pbs:

1) some files on the floppy were corrupted preventing a proper installation of the driver

2) the ASG controller was not properly configured

3) the communications PORT of my laptop is not working at all (tried to uninstall and reinstall, but didn't help ⇒ might be a loose connection inside the laptop)

A new floppy was given by Severn Science. Tested on one of the GFD lab computer ⇒ the ASG data are read by the computer HOORAY!

22.04.04

How to configure the AS6 controller:

To enter the user menu, press (Esc)

(Esc) → user menu
↳ ▼ to system [Enter]
▼ to comms [enter]

1) ▼ to mode [Enter]
(should be 1)
▼ to choose | 1 cont
| 0 print

2) ▼ to protcl [Enter]

| baud = 7 (9600)
| parity = 0
| stop = 1

Check that there are the same values on PC
(system manager
↳ port communications
PORT = 9600)

| Port settings (on PC) | bits per second = 9600
| | data bits = 8
| | parity = none
| | stop bits = 1
| | flow control = none

22.04.04

Once controller and PC are configured, start in folder "BOC-Edwards":

EHV1 serial driver - com 1

to check if the coms are working.

| R repeat instructions
| Y one shot

For continuous recording, choose <u>R</u>

22.04.04

Once the coms have been tested to work on one of the GFD lab computer, drive back to Thornbury to leave the controller and the 2 out-of-calibration ASG with Severn Science for calibration (cost = £45/ASG)

The ASG are : { Sn 005000176 An 007630
Sn 005000055 An 008236

Will send the 4 other ASG from Paris for calibration next week, as drive back and cross the Channel tomorrow to Le Havre.

22.04.04

$T_{oven} = 94°C$ at 12:00 and then 13:45 after having increased slightly T_{set} by ~2°C at 11:30 yesterday.

22.04.04

28.04.04

Sent today the 4 remaining ASG by Chronopost International to Severn Science for calibration.

Edwards serial #	SUPA CalLab No	Cal due on
005000371	008466	19.08.04
005000333	008464	19.08.04
005000334	008463	18.08.04

This last ASG has not been used since SUPA CalLab said it was only valid over a limited P range.
Asked Severn Science to check it.

Time estimate for the calibration ~ 1-2 weeks

28.04.04

28.04.04

Date: Wed, 28 Apr 2004 13:52:28 +0100
From: rstone <rstone@severnscience.co.uk>
To: Anne-Marie LEJEUNE <amlejeun@ccr.jussieu.fr>
Cc: Sarah Davies <Sarah@severnscience.co.uk>
Subject: RE: Parcel with ASG to calibrate

Dear Anne-Marie,
Thank you for your e-mail, as soon as the gauges arrive we have one of our engineers service and calibrate them.

Best Regards

Rod

-----Original Message-----
From: Anne-Marie LEJEUNE [mailto:amlejeun@ccr.jussieu.fr]
Sent: Wednesday, April 28, 2004 01:41
To: rstone
Subject: Parcel with ASG to calibrate

Dear Mr. Stone,

I have just sent to your attention the 4 active strain gauges that I had left in Paris and which also need to be calibrated. You should receive them by the end of this week.

I have sent them with Chronopost International (http://www.chronopost.com). The tracking number is:

XL 123 167 029 FR

Best regards,

Anne-Marie

Dr. Anne-Marie LEJEUNE
Universite Pierre et Marie Curie
Laboratoire de Petrologie, Modelisation des Materiaux et Processus
4 place Jussieu
75252 Paris cedex 05 France

Tel. +33 (0)1 44 27 22 41 Messages +33 (0)1 44 27 50 44
Fax +33 (0)1 44 27 39 11

EU Volcano Dynamics Research Training Network website:
http://gfd.gly.bris.ac.uk/euvolcanonet/intro.htm

28.04.04

28.04.04

06.05.04 42

①

All cups out of oven @ 20H00

21H00

	Cup	m(g)	
	P1	14.0116	
	P2	16.2614	
	P3	16.2853	
	P4	38.1310	
	P5	17.9972	
	P6	28.5474	
	P7	27.5811	
	P8	23.5374	
	P9	18.3865	
	P10	20.5801	
	P11	14.4394	
	P12	13.0480	
	P13	28.1297	
Tray2	P14	14.9035	@ 21H11
	P15	20.6880	
	P16	7.7114	
	P17	17.9941	
	P18	12.8439	
	P19	16.2601	

06.05.04

422

06.05.04

(2)

	Cup	m (g)	
	P20	15.4293	
	P21	17.5901	
	P22	13.2138	
	P23	15.2068	
	P24	15.8055	
	P25	17.0864/5	
	P26	13.2496	
Tray3	P27	24.8669	@ 21H18
	P28	6.8225	
	P29	17.4382	
	P30	18.8870/1	
	P31	8.7627	

All cups back in oven @ 21H20

Paper sheets off @ 21H45

06.05.04

28/05/04

① 423

All cups out of oven @ 14 H50.
Same procedure.
Weighing started @ 18 H44

Cup	m(g)
P1	14.0046
P2	16.2507
P3	16.2720
P4	38.0814
P5	17.9810
P6	28.5180
P7	27.5476
P8	23.5140
P9	18.3720
P10	20.5650
P11	14.4327
P12	13.0415
P13	28.1035
P14	14.8936
P15	20.6684
P16	7.7083
P17	17.9773
P18	12.8358
P19	16.2450

28.05.04

28/05/04

(2)

Cup	m (g)
P20	15.4174
P21	17.5736
P22	13.2048
P23	15.1952
P24	15.7933
P25	17.0740
P26	13.2430
P27	24.8428
P28	6.8215
P29	17.4229
P30	18.8724
P31	8.7617

↳ done @ 19H00

Back in oven @ 19H01 (same procedure)
@ 94/95°C

Paper sheets removed @ 19H26

ASG still not back from calibration. Contact Severn Science next week.

28.05.04

Date: Tue, 1 Jun 2004 12:44:53 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: rstone <rstone@severnscience.co.uk>
Subject: ASG calibration

Dear Rod,

I just got back from abroad and was wondering how did the ASG calibration go ? Would you please let me know when you ship the gauges and controller so that I can inform the University parcel office to keep an eye open.
Thank you.

On the address where the gauges and controller have to be sent, please make sure to write Case 110:

Anne-Marie LEJEUNE
Universite Pierre et Marie Curie
Laboratoire PMMP
Case 110
4 place Jussieu
75252 Paris cedex 05
France

Best regards,

Anne-Marie

On Wed, 28 Apr 2004, rstone wrote:

> Dear Anne-Marie,
> Thank you for your e-mail, as soon as the gauges arrive we have one of
> our engineers service and calibrate them.
>
> Best Regards
>
> Rod
>

01.06.04

> -----Original Message-----
> From: Anne-Marie LEJEUNE [mailto:amlejeun@ccr.jussieu.fr]
> Sent: Wednesday, April 28, 2004 01:41
> To: rstone
> Subject: Parcel with ASG to calibrate
>
>
>
> Dear Mr. Stone,
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> I have just sent to your attention the 4 active strain gauges that I had
> left in Paris and which also need to be calibrated. You should receive
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> I have sent them with Chronopost International
> (http://www.chronopost.com). The tracking number is:
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> XL 123 167 029 FR
>
> Best regards,
>
> Anne-Marie
>
> _____
>
> Dr. Anne-Marie LEJEUNE
> Universite Pierre et Marie Curie
> Laboratoire de Petrologie, Modelisation des Materiaux et Processus
> 4 place Jussieu
> 75252 Paris cedex 05 France
>
> Tel. +33 (0)1 44 27 22 41 Messages +33 (0)1 44 27 50 44
> Fax +33 (0)1 44 27 39 11
>
> EU Volcano Dynamics Research Training Network website:
> http://gfd.gly.bris.ac.uk/euvolcanonet/intro.htm
> _____

01.06.04

Date: Tue, 1 Jun 2004 16:09:35 +0100
From: rstone <rstone@severnscience.co.uk>
To: Anne-Marie LEJEUNE <amlejeun@ccr.jussieu.fr>
Cc: Sarah Davies <Sarah@severnscience.co.uk>
Subject: RE: ASG calibration

Hi Anne-Marie,
I hope you had a good visit abroad, was it business or pleasure ?.

We have finished the gauges and we are just waiting for the engineer sign the certificates, he is on holiday at present and is due back on the 7/6/04. We found one ASG's (transducer) to beyond repair, you will receive a quote for the cost of a replacement ASG.

Best Regards

Rod

-----Original Message-----
From: Anne-Marie LEJEUNE [mailto:amlejeun@ccr.jussieu.fr]
Sent: Tuesday, June 01, 2004 11:45
To: rstone
Subject: ASG calibration

Dear Rod,

I just got back from abroad and was wondering how did the ASG calibration go ? Would you please let me know when you ship the gauges and controller so that I can inform the University parcel office to keep an eye open.
Thank you.

On the address where the gauges and controller have to be sent, please make sure to write Case 110:

Anne-Marie LEJEUNE
Universite Pierre et Marie Curie
Laboratoire PMMP
Case 110
4 place Jussieu
75252 Paris cedex 05
France

Best regards,

Anne-Marie

01.06.04

On Wed, 28 Apr 2004, rstone wrote:

> Dear Anne-Marie,
> Thank you for your e-mail, as soon as the gauges arrive we have one of
> our engineers service and calibrate them.
>
> Best Regards
>
> Rod

Will have to wait for another week to get the ASG back :(

01.06.04

Date: Wed, 2 Jun 2004 11:20:31 +0100
From: Sarah Davies <Sarah@severnscience.co.uk>
To: amlejeun@ccr.jussieu.fr
Subject: Quotation traceable calibration
Parts/Attachments:
1 Shown ~6 lines Text (charset: ISO-8859-1)
2 52 KB Application, "13866.doc"

<<13866.doc>>
Please find quotation for traceable calibration for your active gauge controller and 6 x active strain gauges.

Best regards
Mandy Hook

[Part 2, "13866.doc" Application/MSWORD 69KB.]
[Cannot display this part. Press "V" then "S" to save in a file.]

02.06.04

02.06.04

8, Brunel Way
Thornbury
Bristol
BS35 3UR
England

Tel: 00 44 (0) 1454 414723
Fax: 00 44 (0) 1454 281680
E-mail : **mhook@severnscience.co.uk**

Severn SCIENCE

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- ✓ High vacuum equipment
- ✓ Cryo pumps and coolers
- ✓ Analytical Instrumentation

To : Dr Anne-Marie Leyeune

From : Mandy Hook

To : Universit'e Pierre & Marie Curie

Pages : 1 of 1

Fax :

Date : 1/6/04

Ref : Traceable calibration

cc :

Service/Repair of High Vacuum Equipment

Equipment : Active Gauge Controller / 6x ASG
Serial Number : Various

Our Ref : 13866 & 13839
Your Order Number : GL31976

5 x Traceable calibration @ £115.00	£575.00
Carriage – delivery only	£64.67

	£639.67

1 x Active strain gauge will need replacing

All prices quoted are exclusive of VAT
Payment Terms : Against pro-forma invoice or nett 30 days if on account

Cost of New Equipment for comparison: Active strain gauge D357-27-000 £394.00

Please confirm acceptance of quotation with a covering purchase order

Best regards

Mandy Hook
Service Administrator

NB : Quotation Valid for Two Weeks From Above Date

02.06.04

02.06.04

02.06.04
433

Date: Wed, 2 Jun 2004 14:17:06 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: Sarah Davies <Sarah@severnscience.co.uk>
Cc: Rod Stone <rstone@severnscience.co.uk>
Subject: Re: Quotation traceable calibration

Dear Mandy,

Thank you for your quotation. I confirm that I am accepting it. I will not replace the damaged ASG for the time being, as I only need 3 to run my experiments.

I left the original purchase order (number GL31976) to Rod Stone when I brought the ASG and controller to your company. You should send your invoice using the order number GL31976 to the address written on the purchase order and which is:

University of Bristol
Department of Earth Sciences
Wills Memorial Building
Queen's Road
Bristol BS8 1RJ

Best regards,

Anne-Marie

Dr. Anne-Marie LEJEUNE
Universite Pierre et Marie Curie
Laboratoire de Petrologie, Modelisation des Materiaux et Processus
Case 110
4 place Jussieu
75252 Paris cedex 05 France

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Fax +33 (0)1 44 27 39 11

EU Volcano Dynamics Research Training Network website:
http://gfd.gly.bris.ac.uk/euvolcanonet/intro.htm

On Wed, 2 Jun 2004, Sarah Davies wrote:

> <<13866.doc>>
> Please find quotation for traceable calibration for your active gauge
> controller and 6 x active strain gauges.
>
> Best regards
> Mandy Hook

02.06.04

02.06.04

Date: Wed, 2 Jun 2004 14:24:48 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: rstone <rstone@severnscience.co.uk>
Subject: RE: ASG calibration

Hi Rod,

Thank you for your message. The trip was in La Reunion, a French island in the Indian Ocean, closed to Madagascar and Mauritius :-) I went for work and was very lucky that there was an eruption of the Piton de la Fournaise, the active volcano, while out there :-) so it was also pleasure.

I have just emailed back Mandy Hook to confirm that I accept the quotation. I won't replace for the time being the damaged ASG.

Thank you for your help.

Best regards,

Anne-Marie

Anne-Marie LEJEUNE
Universite Pierre et Marie Curie
Laboratoire de Petrologie, Modelisation des Materiaux et Processus
Case 110
4 place Jussieu
75252 Paris cedex 05 France

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EU Volcano Dynamics Research Training Network website:
http://gfd.gly.bris.ac.uk/euvolcanonet/intro.htm

On Tue, 1 Jun 2004, rstone wrote:

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> We have finished the gauges and we are just waiting for the engineer
> sign the certificates, he is on holiday at present and is due back on
> the 7/6/04. We found one ASG's (transducer) to beyond repair, you will
> receive a quote for the cost of a replacement ASG.
>
> Best Regards
>
> Rod

02.06.04

436

> -----Original Message-----
> From: Anne-Marie LEJEUNE [mailto:amlejeun@ccr.jussieu.fr]
> Sent: Tuesday, June 01, 2004 11:45
> To: rstone
> Subject: ASG calibration
>
>
>
> Dear Rod,
>
> I just got back from abroad and was wondering how did the ASG calibration
> go ? Would you please let me know when you ship the gauges and controller
> so that I can inform the University parcel office to keep an eye open.
> Thank you.
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> Case 110
> 4 place Jussieu
> 75252 Paris cedex 05
> France
>
> Best regards,
>
> Anne-Marie
>
>
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> On Wed, 28 Apr 2004, rstone wrote:
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> > Thank you for your e-mail, as soon as the gauges arrive we have one of
> > our engineers service and calibrate them.
> >
> > Best Regards
> >
> > Rod

02.06.04

10.06.04
①

All cups out of oven @ 14H22 - T = 94°C

Weighing @ 19H17

Cup	m(g)
P1	14.0038
P2	16.2491
P3	16.2687
P4	38.0652
P5	17.9793
P6	28.5091
P7	27.5353
P8	23.5069
P9	18.3688
P10	20.5628
P11	14.4327
P12	13.0414
P13	28.0939 → 19H24
P14	14.8920
P15	20.6618
P16	7.7080
P17	17.9727
P18	12.8341
P19	16.2407
P20	15.4136

10.06.04

438
10.06.04
(2)

Cup	m (g)
P21	17.5679
P22	13.2025
P23	15.1921
P24	15.7908
P25	17.0711
P26	13.2437
P27	24.8339
P28	6.8235
P29	17.4192
P30	18.8683
P31	8.7654

→ 19H29

↳ Back in oven @ 19H32

Paper sheets removed @
Fly to Bristol tomorrow (11.06.04)

10.06.04

Received today a rather unpleasant news about the ASG calibration: SCORI QA group has decided that it is not acceptable for the manufacturer to perform the calibration!!! Bull!

12.06.04

0:35 French time on 11.06.04

Date: Thu, 10 Jun 2004 16:35:15 -0500
From: Brittain Hill <bhill@cnwra.swri.edu>
Reply-To: bhill@swri.org
To: Anne-Marie Lejeune <amlejeun@ccr.jussieu.fr>
Subject: URGENT URGENT URGENT

Anne-Marie - I have just been through a very disagreeable meeting with our QA group. I will not belabour the details, but there are three things I really need you to do:

1) Please send me a copy of whatever you have for your scientific notebook. I absolutely must have something to show the auditors, as this is being used as an example of how negligent I have been with regards to QA procedures under my control.

2) The QA group has now decided that it is not acceptable for the manufacturer (i.e., Severn Science) to perform the calibration. This calibration must be done with one of _their_ approved suppliers, or in our Calibration Lab. If the Edwards gauges are with you, please box them up along with the ASG controller and DHL express ship them to me as quickly as you can. If the gauges and controller are in Bristol, please call/email Thierry/Jerry and have them express ship them to me as soon as possible. Once an "approved" supplier has calibrated the gauges, I will express ship them back to you.

3) Please let me know your decision regarding the email I sent on 1 June 2004. It is attached below. I don't know if there is enough time left to get you on the books as a consultant, before our 12 July meeting, but I'm still willing to try if you will send me the necessary paperwork. Otherwise, please clearly let me know that you are no longer willing or able to participate in our work, so that I can try and deal with the very large hole your absence will create in the program. Again, our QA people are asking nasty questions about why these experiments are being done by someone who has no documentation of current (not past) qualifications and evaluation of Conflict of Interest for work in Paris.

Merci beaucoup-
Britt

12.06.04

-------- Original Message -------- REGARDING THE GAUGE CALIBRATION
Subject: News
Date: Wed, 05 May 2004 10:11:40 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@ccr.jussieu.fr>
To: Brittain Hill <bhill@swri.edu>

...
Gauges: I was in Bristol for two weeks over the Easter break and have spent a fair amount of time at the company called Severn Science, who works for BOC Edwards. I have bought the ASG equipment from them. The communication problem between the ASG controller and PC computers is now sorted out. In fact there were 3 different kinds of problems which I wouldhave never been able to sort out without their help (one being that Edwards had sent a software with corrupted library files ...). We spent numerous hours sorting out the problems one by one. People at Severn Science were truly great and won't charge anything for the time.

Calibration: I have left the controller and the 2 gauges which needed calibration to Severn Science. They will ship them back to Paris this month. When gauges are calibrated, they use the controller which usually runs the gauges and calibrate the controller at the same time. The main cost of the calibration is the controller calibration (115 GBP per controller), each additionnal gauge costing 15 GBP to calibrate. So I have shipped the other gauges which I had left in Paris (the ones which calibration runs out in August) to Severn Science. That way all the ASG will be up to date and there won't be twice the cost of 115 GBP for the controller. Hope this is allright with you. Also Severn Science working for Edwards, I am quite pleased to have the ASG checked by them after all the usage they had. Severn Science will send the calibration data sheets so that I can stick them on the labbook and give copies to SwRI callab.

12.06.04

All cups out of oven @ 16H07 - $T_{oven} = 94°C$

Weighing from 18H49 - $T_{room} = 23°C$

	Cup	m(g)
Tray 1	B1	11.7480
	B2	20.3815
	B3	11.5424
	B4	19.1125
	B5	21.4398
	B6	15.9771
	B7	15.3568
	B8	13.8015
	B9	14.7263
	B10	13.0194
	B11	15.6360
	B12	18.3581
	B13	11.4819
Tray 2	B14	6.3924
	B15	12.4256
	B16	14.7023
	B17	18.5267

12.06.04

442

12.06.04

(2)

	Cup	m(g)
	B18	17.5417
	B19	23.6775
	B20	7.4822
	B21	18.3561
	B22	24.6644
	B23	26.0293
	B24	29.2893
	B25	31.4607
	B26	10.2608
Tray 3	B27	13.4427
	B28	5.4743
	B29	22.2169
	B30	8.2483
	B31	15.7569
	B32	16.7453
	B33	13.9589
	B34	15.6253
	B35	24.3666
	B36	15.3788
	B37	13.6238
	B38	10.5100
Tray 4	B39	6.5048
	B40	20.4626
	B41	17.9344
	GRA	25.1836
	GRA	7.1812

12.06.04

Weighing done @ 19H07 - $T_{room} = 23°C$

Back in oven @ 19H10 @ 94°C

Paper sheets removed @ 19H57

12.06.04

13.06.04

Date: Mon, 14 Jun 2004 20:16:19 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: Brittain Hill <bhill@cnwra.swri.edu>
Subject: Re: URGENT URGENT URGENT

Britt,

Just sent you a 12 pages fax from Bristol. Hope you received it OK. Will be back in Paris on Friday. Will copy the labbook _as it is_ and mail the photocopies, then later will copy neatly the notes and send a neat version.

Thanks and sorry for all the delays and hassles.

Very best wishes,

Anne-Marie

On Thu, 10 Jun 2004, Brittain Hill wrote:

> Anne-Marie - I have just been through a very disagreeable meeting with
> our QA group. I will not belabour the details, but there are three
> things I really need you to do:
>
> 1) Please send me a copy of whatever you have for your scientific
> notebook. I absolutely must have something to show the auditors, as this
> is being used as an example of how negligent I have been with regards to
> QA procedures under my control.
>
> 2) The QA group has now decided that it is not acceptable for the
> manufacturer (i.e., Severn Science) to perform the calibration. This
> calibration must be done with one of _their_ approved suppliers, or in
> our Calibration Lab. If the Edwards gauges are with you, please box them
> up along with the ASG controller and DHL express ship them to me as
> quickly as you can. If the gauges and controller are in Bristol, please
> call/email Thierry/Jerry and have them express ship them to me as soon
> as possible. Once an "approved" supplier has calibrated the gauges, I
> will express ship them back to you.
>
> 3) Please let me know your decision regarding the email I sent on 1 June
> 2004. It is attached below. I don't know if there is enough time left to
> get you on the books as a consultant, before our 12 July meeting, but
> I'm still willing to try if you will send me the necessary paperwork.
> Otherwise, please clearly let me know that you are no longer willing or
> able to participate in our work, so that I can try and deal with the
> very large hole your absence will create in the program. Again, our QA
> people are asking nasty questions about why these experiments are being

14.06.04

> done by someone who has no documentation of current (not past)
> qualifications and evaluation of Conflict of Interest for work in Paris.
>
> Merci beaucoup-
> Britt
>
>
> -------- Original Message -------- REGARDING THE GAUGE CALIBRATION
> Subject: News
> Date: Wed, 05 May 2004 10:11:40 +0200 (DFT)
> From: Anne-Marie LEJEUNE <amlejeun@ccr.jussieu.fr>
> To: Brittain Hill <bhill@swri.edu>
>
> ...
> Gauges: I was in Bristol for two weeks over the Easter break and have
> spent a fair amount of time at the company called Severn Science, who
> works for BOC Edwards. I have bought the ASG equipment from them. The
> communication problem between the ASG controller and PC computers is now
> sorted out. In fact there were 3 different kinds of problems which I
> would have never been able to sort out without their help (one being that
> Edwards had sent a software with corrupted library files ...). We spent
> numerous hours sorting out the problems one by one. People at Severn
> Science were truly great and won't charge anything for the time.
> Calibration: I have left the controller and the 2 gauges which needed
> calibration to Severn Science. They will ship them back to Paris this
> month. When gauges are calibrated, they use the controller which usually
> runs the gauges and calibrate the controller at the same time. The main
> cost of the calibration is the controller calibration (115 GBP per
> controller), each additionnal gauge costing 15 GBP to calibrate. So I
> have shipped the other gauges which I had left in Paris (the ones which
> calibration runs out in August) to Severn Science. That way all the ASG
> will be up to date and there won't be twice the cost of 115 GBP for the
> controller. Hope this is allright with you. Also Severn Science working
> for Edwards, I am quite pleased to have the ASG checked by them after
> all the usage they had. Severn Science will send the calibration data sheets
> so that I can stick them on the labbook and give copies to SwRI callab.

14.06.04

Date: Mon, 14 Jun 2004 14:23:30 -0500
From: Brittain Hill <bhill@cnwra.swri.edu>
Reply-To: bhill@swri.org
To: Anne-Marie LEJEUNE <amlejeun@ccr.jussieu.fr>, Anne-Marie.Lejeune@upmc.fr
Subject: Re: URGENT URGENT URGENT

Hola Anne-Marie - Yes, I received the fax. Several things still are needed:

1) I need to have a current copy of your cirriculum vitae. I only have a several-year old version in our files.

2) I need to clarify with you the hours listed in the statement of work. Since you are on a full-time appointment at Paris, are you able to bill us for these hours? If you will be unable to bill us, then I would prefer to take this expense out of the SoW because there are budget problems here and any reduction I can make is a good thing.

3) Are the travel expenses accurate? These were just my estimates - If these costs are overestimated, please let me know. I am being told to reduce costs in IA, and any reductions we can make now will help me convince Wes that we really need to do this work.

I am glad you bought a refundable ticket. I will do all that I can to get the very large amount of paperwork approved within the next 3 weeks. But at this very late date, I can make no promises about what I can accomplish regarding your consultancy in time for our July meeting.

I am trying to get some additional info from QA about some documentation Severn Sciences could possibly provide that would satisfy QA about the calibrations. I will let you know as soon as possible, but you can wait on sending the gauges back to me for another day or so until I resolve this. Good news, though, about the other gauges that weren't out of calibration until August 04. If you're going to run experiments in the interim, please use these "in calibration" gauges until I resolve this mess. Were any of the gauges out of calibration, according to Severn?

In haste-
Britt

Anne-Marie LEJEUNE wrote:

> Britt,
>
> Just sent you a 12 pages fax from Bristol. Hope you received it OK. Will
> be back in Paris on Friday. Will copy the labbook _as it is_ and mail the
> photocopies, then later will copy neatly the notes and send a neat
> version.
>
> Thanks and sorry for all the delays and hassles.
>
> Very best wishes,
>
> Anne-Marie

14.06.04

14.06.04

Date: Mon, 14 Jun 2004 14:39:06 -0500
From: Brittain Hill <bhill@cnwra.swri.edu>
Reply-To: bhill@swri.org
To: Anne-Marie LEJEUNE <amlejeun@ccr.jussieu.fr>, Anne-Marie.Lejeune@upmc.fr
Subject: Re: URGENT URGENT URGENT

OK, got a straight answer from QA. I need you to contact whoever did the calibration at Severn Science and ask them if they have any accreditation or certification for the ISO 17025 Standard. This is an international standard for calibrations. If Severn has that accreditation, please ask them to fax a copy of that accreditation to you with all possible haste. Then, please send this to me along with the calibration certificates. Evidence of ISO 17025 accreditation will be acceptable for our QA, which means the Severn calibration is OK by QA. I know, what bollocks, but it's the morasse I'm ensnared it.

Murky Buckets-
Britt

14.06.04

14.06.04

Date: Wed, 16 Jun 2004 00:59:24 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: bhill@swri.org
Cc: Anne-Marie.Lejeune@upmc.fr
Subject: AML's CV

Hola Britt,

On Mon, 14 Jun 2004, Brittain Hill wrote:

> Hola Anne-Marie - Yes, I received the fax. Several things still are needed:
>
> 1) I need to have a current copy of your cirriculum vitae. I only have a
> several-year old version in our files.

Here is a CV attached as a word file named CVAML_June2004.doc. Hope you receive it OK.

> 2) I need to clarify with you the hours listed in the statement of work.
> Since you are on a full-time appointment at Paris, are you able to bill us for
> these hours? If you will be unable to bill us, then I would prefer to take
> this expense out of the SoW because there are budget problems here and any
> reduction I can make is a good thing.

I am able to bill you, but if you need to reduce the budget, that's fine with me as long as the travel expenses between Paris and Bristol can be covered. I will try and keep them as low as possible.

> 3) Are the travel expenses accurate? These were just my estimates - If these
> costs are overestimated, please let me know. I am being told to reduce costs
> in IA, and any reductions we can make now will help me convince Wes that we
> really need to do this work.

I need to have a look in details. When I am in Bristol, I do not need to rent a car (I am using my own) and there is no accommodation expenses as long as I keep my house over there. If I need to meet Onno, I can suggest him to meet in Paris :-) I will have a look at the travel expenses as soon as I can. This week, I am helping Thierry to sort out a new Bristol bill, with copies of all the invoices and receipts. We should soon be up to date.

> I am glad you bought a refundable ticket. I will do all that I can to get the
> very large amount of paperwork approved within the next 3 weeks. But at this
> very late date, I can make no promises about what I can accomplish regarding
> your consultancy in time for our July meeting.

Do you really want me to attend this meeting ? You, Andy and Steve can very well talked on my behalf. If you need to cut down the budget, that would reduce the cost of travel a lot. My

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return flight costed about 1200 euros. The cost of cancellation and refund is 100 euros (10 days before the departure date). Also I got the ASG back late (last Thursday) and really need to spend time in the lab as soon as I am back in Paris from Bristol. I am more keen on catching up with the experiments to try as much as possible to have them all completed by mid-summer.

> I am trying to get some additional info from QA about some documentation
> Severn Sciences could possibly provide that would satisfy QA about the
> calibrations. I will let you know as soon as possible, but you can wait on
> sending the gauges back to me for another day or so until I resolve this. Good
> news, though, about the other gauges that weren't out of calibration until
> August 04. If you're going to run experiments in the interim, please use these
> "in calibration" gauges until I resolve this mess. Were any of the gauges out
> of calibration, according to Severn?

I had already put away the 2 ASG which were due for calibration last April. I have 3 others which are only due for August 2004. According to Severn Science/BOC Edwards, these 5 gauges are calibrated and working properly. A 6th gauge, which had been found as working in a limited range by SwRI cal lab, has been labelled as 'faulty' by Severn Science and can just be binned. This faulty gauge has never been used anyway since it was returned by SwRI cal lab as 'working in a limited range'.

TTFN and thank you.

Volcanologiquement,

Anne-Marie

[Part 2, "" Application/OCTET-STREAM (Name: "CVAML_June2004.doc") 94KB.]
[Cannot display this part. Press "V" then "S" to save in a file.]

16.06.04

Date: Wed, 16 Jun 2004 01:01:39 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: bhill@swri.org
Cc: Anne-Marie.Lejeune@upmc.fr
Subject: Re: URGENT URGENT URGENT

OK will phone Severn Science tomorrow morning (today !) and ask. AML

On Mon, 14 Jun 2004, Brittain Hill wrote:

> OK, got a straight answer from QA. I need you to contact whoever did the
> calibration at Severn Science and ask them if they have any accreditation or
> certification for the ISO 17025 Standard. This is an international standard
> for calibrations. If Severn has that accreditation, please ask them to fax a
> copy of that accreditation to you with all possible haste. Then, please send
> this to me along with the calibration certificates. Evidence of ISO 17025
> accreditation will be acceptable for our QA, which means the Severn
> calibration is OK by QA. I know, what bollocks, but it's the morasse I'm
> ensnared it.
>
> Murky Buckets-
> Britt

16.06.04

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All cups taken out of the oven at 13H42.
T = 94° C

Weighing from 18H23 T = 23°C

	Cup	m(g)
Tray 1	B1	11.7454
	B2	20.3742
	B3	11.5401
	B4	19.1070
	B5	21.4334
	B6	15.9720
	B7	15.3508
	B8	13.7969
	B9	14.7219
	B10	13.0163
	B11	15.6310
	B12	18.3517
	B13	11.4806
Tray 2	B14	6.3926
	B15	12.4233
	B16	14.6994

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(2)

	Cup	m(g)
	B17	18.5201
	B18	17.5360
	B19	23.6693
	B20	7.4818
	B21	18.3520
	B22	24.6576
	B23	26.0182
	B24	29.279 2
	B25	31.448 2
	B26	10.2610
Tray 3	B27	13.4354
	B28	5.4768
	B29	22.2017
	B30	8.2478
	B31	15.7510
	B32	16.7397
	B33	13.9547
	B34	15.6195
	B35	24.3585
	B36	15.3744
	B37	13.6231
	B38	10.510 4

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Tray 4

Cup	m(g)
B39	6.506 3
B40	20.4580
B41	17.9322
GRA1	25.1862
GRA2	7.1828

Done @ 18H41

All cups back in the oven @ 94°C @ 18H44 all covered w/ paper sheets to prevent the loss of any GRA wee flakes while heating up.

Paper sheets removed @ 19H19

16.06.04

16.06.04

Date: Wed, 16 Jun 2004 15:09:10 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: Rod Stone <rstone@severnscience.co.uk>
Subject: ASG calibration

Dear Rod,

The company, which is sponsoring the experiments I am doing, has a Quality Assurance Department. They can be quite difficult people ! They are asking if Severn Science has "accreditation or certification for the ISO 17025 Standard". I was told that it is an international standard for calibration. If Severn Science has it, which I guess you have, could you please fax a copy to me ?

If you can fax it this afternoon, you can use the University of Bristol number: 0117 925 3385

If you can only fax it from tomorrow, please use the University of Paris number: 00 33 1 44 27 39 11

I will also need a copy of the data spreasheets of the ASG calibrations as well as signed certificates.

I will have to send copies to the Southwest Research Institute in Texas. If I cannot provide evidence of the ISO 17025 accreditation, then they want me to ship the ASG to Texas for calibration by their own lab and I am not keen on doing it. I trust more your lab than theirs !

Thank you very much for your help. And sorry to bother you with all this.

Very best regards,

Anne-Marie

Anne-Marie LEJEUNE
Universite Pierre et Marie Curie
Laboratoire de Petrologie, Modelisation des Materiaux et Processus
Case 110
4 place Jussieu
75252 Paris cedex 05 France

Tel. +33 (0)1 44 27 22 41 Messages +33 (0)1 44 27 50 44
Fax +33 (0)1 44 27 39 11

EU Volcano Dynamics Research Training Network website:
http://gfd.gly.bris.ac.uk/euvolcanonet/intro.htm

16.06.04

On Mon, 14 Jun 2004, Brittain Hill wrote:

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> calibration at Severn Science and ask them if they have any accreditation or
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> for calibrations. If Severn has that accreditation, please ask them to fax a
> copy of that accreditation to you with all possible haste. Then, please send
> this to me along with the calibration certificates. Evidence of ISO 17025
> accreditation will be acceptable for our QA, which means the Severn
> calibration is OK by QA.

16.06.04

Date: Wed, 16 Jun 2004 15:16:30 +0100
From: rstone <rstone@severnscience.co.uk>
To: "Anne-Marie Lejeune (E-mail)" <amlejeun@ccr.jussieu.fr>
Subject: FW: ASG calibration

Hi Anne-Marie,

Please find attached reply to enquiry regarding the accreditation.

Best Regards
Rod

-----Original Message-----
From: Andrew Hilton
Sent: Wednesday, June 16, 2004 03:11
To: rstone
Subject: RE: ASG calibration

We do not have this accreditation. Our calibration procedures are documented under our ISO 9000 system and the calibration equipment / procedures are in accordance with ISO/DIS 3567.

-----Original Message-----
From: rstone
Sent: 16 June 2004 14:27
To: Andrew Hilton
Subject: FW: ASG calibration

Andy,

Can we help ?

Rod

-----Original Message-----
From: Anne-Marie LEJEUNE [mailto:amlejeun@ccr.jussieu.fr]
Sent: Wednesday, June 16, 2004 02:09
To: rstone
Subject: ASG calibration

Dear Rod,

The company, which is sponsoring the experiments I am doing, has a Quality Assurance Department. They can be quite difficult people ! They are asking if Severn Science has "accreditation or certification for the ISO 17025 Standard". I was told that it is an international standard for calibration. If Severn Science has it, which I guess you have, could you please fax a copy to me ?

If you can fax it this afternoon, you can use the University of Bristol number: 0117 925 3385

16.06.04

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If you can only fax it from tomorrow, please use the University of Paris number: 00 33 1 44 27 39 11

I will also need a copy of the data spreasheets of the ASG calibrations As well as signed certificates.

I will have to send copies to the Southwest Research Institute in Texas. If I cannot provide evidence of the ISO 17025 accreditation, then they want me to ship the ASG to Texas for calibration by their own lab and I am not keen on doing it. I trust more your lab than theirs !

Thank you very much for your help. And sorry to bother you with all this.

Very best regards,

Anne-Marie

Anne-Marie LEJEUNE
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Laboratoire de Petrologie, Modelisation des Materiaux et Processus
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4 place Jussieu
75252 Paris cedex 05 France

Tel. +33 (0)1 44 27 22 41 Messages +33 (0)1 44 27 50 44
Fax +33 (0)1 44 27 39 11

EU Volcano Dynamics Research Training Network website:
http://gfd.gly.bris.ac.uk/euvolcanonet/intro.htm

On Mon, 14 Jun 2004, Brittain Hill wrote:

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> calibration at Severn Science and ask them if they have any accreditation or
> certification for the ISO 17025 Standard. This is an international standard
> for calibrations. If Severn has that accreditation, please ask them to fax a
> copy of that accreditation to you with all possible haste. Then, please send
> this to me along with the calibration certificates. Evidence of ISO 17025
> accreditation will be acceptable for our QA, which means the Severn
> calibration is OK by QA.

16.06.04

Date: Wed, 16 Jun 2004 16:40:46 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: Brittain Hill <bhill@swri.edu>
Subject: FW: ASG calibration (fwd)

Hola Britt,

Please find below the reply from Severn Science. Hope this helps.

They do not have the requested accreditation. Their calibration procedures are documented under their ISO 9000 system (BOC Edwards) and the calibration equipment / procedures are in accordance with ISO/DIS 3567.

AML

---------- Forwarded message ----------
Date: Wed, 16 Jun 2004 15:16:30 +0100
From: rstone <rstone@severnscience.co.uk>
To: "Anne-Marie Lejeune (E-mail)" <amlejeun@ccr.jussieu.fr>
Subject: FW: ASG calibration

Hi Anne-Marie,

Please find attached reply to enquiry regarding the accreditation.

Best Regards
Rod

-----Original Message-----
From: Andrew Hilton
Sent: Wednesday, June 16, 2004 03:11
To: rstone
Subject: RE: ASG calibration

We do not have this accreditation. Our calibration procedures are documented under our ISO 9000 system and the calibration equipment / procedures are in accordance with ISO/DIS 3567.

16.06.04

-----Original Message-----
From: rstone
Sent: 16 June 2004 14:27
To: Andrew Hilton
Subject: FW: ASG calibration

Andy,

Can we help ?

Rod

-----Original Message-----
From: Anne-Marie LEJEUNE [mailto:amlejeun@ccr.jussieu.fr]
Sent: Wednesday, June 16, 2004 02:09
To: rstone
Subject: ASG calibration

Dear Rod,

The company, which is sponsoring the experiments I am doing, has a Quality Assurance Department. They can be quite difficult people ! They are asking if Severn Science has "accreditation or certification for the ISO 17025 Standard". I was told that it is an international standard for calibration. If Severn Science has it, which I guess you have, could you please fax a copy to me ?

If you can fax it this afternoon, you can use the University of Bristol number: 0117 925 3385

If you can only fax it from tomorrow, please use the University of Paris number: 00 33 1 44 27 39 11

I will have to send copies to the Southwest Research Institute in Texas. If I cannot provide evidence of the ISO 17025 accreditation, then they want me to ship the ASG to Texas for calibration by their own lab and I am not keen on doing it. I trust more your lab than theirs !

Thank you very much for your help. And sorry to bother you with all this.

Very best regards,

Anne-Marie

16.06.04

Date: Wed, 16 Jun 2004 17:10:35 -0500
From: Brittain Hill <bhill@cnwra.swri.edu>
Reply-To: bhill@swri.org
To: Anne-Marie Lejeune <amlejeun@ccr.jussieu.fr>, Anne-Marie Lejeune <Anne-Marie.Lejeune@upmc.fr>
Subject: [Fwd: RE: [Fwd: FW: ASG calibration (fwd)]]

Anne-Marie - I'm sorry to say that you need to send me the pressure transducers as soon as possible, as the Severn calibration just won't be acceptable to our QA group. Yes, I agree that this doesn't make sense. But that is the way it is with them. Selah.

Please send me whatever needs to be sent for calibration. I think the controller is needed, along with the transducers. Since this has to be done, perhaps it would be best to see if the other pressure sensors are coming due for calibration in the next couple of months. I think the small PCB(?) sensors (in the glass tube) were done last August, just before I sent them to you. If you think you'll be done before they fall out of calibration, then don't bother. But if there's a chance that you'll still be doing an experiment or two with them in August, it would be best to send them along with the ASG transducers and get everything calibrated while the equipment is down.

You did mention that only 2 of the ASG transducers were officially out of calibration, but the others were good until August 2004. If you think you can get the experiments done before those transducers fall out of official calibration, then we should hold off on shipping anything and see if you can get the experiments done before the calibration expires. Otherwise, ship all the gauges that need calibration to me and we'll get it done ASAP. Still, that will be several weeks delay.

Please use 1-2 day airfreight possible for these, to minimize delays. It doesn't have to be overnight, though.

Thank-you for understanding that it is just not my decision, but if we don't do this, they will declare all of the experiments unuseable.

Merci-
Britt

16.06.04

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-------- Original Message --------
Subject: RE: [Fwd: FW: ASG calibration (fwd)]
Date: Wed, 16 Jun 2004 13:11:46 -0500
From: Robert Brient <rbrient@swri.edu>
Reply-To: rbrient@swri.edu
Organization: CNWRA
To: brittain.hill@swri.org

Britt:

From what I can gather, ISO/DIS 3567 is a draft standard for calibrating vacuum or pressure measuring devices. It is probably equivalent to an ASTM standard method. This draft standard doesn't appear to be equivalent to ISO 17025 that addresses calibration systems.

Another consideration is that the supplier does not have a third party confirmation (by certification or accreditation).

I think that we are stuck with shipping it back.

Bob Brient
Director, Quality Assurance
Center for Nuclear Waste Regulatory Analyses
Southwest Research Institute
(210) 522-5537
(210) 522-6081 fax

-----Original Message-----
From: Brittain Hill [mailto:bhill@cnwra.swri.edu]
Sent: Wednesday, June 16, 2004 10:02 AM
To: Robert Brient
Subject: [Fwd: FW: ASG calibration (fwd)]
Importance: High

Bob - For calibration of the gauges in UK:

The manufacturer does their calibrations under ISO/DIS 3567. Is this an acceptable standard relative to ISO 17025?

If so, I'll have them send the certification. Otherwise, please let me know and I'll have the equipment shipped to us from UK.

Thanks-
Britt

16.06.04

-----Original Message-----
From: Andrew Hilton
Sent: Wednesday, June 16, 2004 03:11
To: rstone
Subject: RE: ASG calibration

We do not have this accreditation. Our calibration procedures are documented under our ISO 9000 system and the calibration equipment /procedures are in accordance with ISO/DIS 3567.

16.06.04

16.06.04

Date: Fri, 18 Jun 2004 12:17:45 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: bhill@swri.org
Subject: Re: [Fwd: RE: [Fwd: FW: ASG calibration (fwd)]]

Britt,

I know and I understand that it is not your decision. I am only very rude (as much as possible !) to the QA people but I also understand that they have a job to do. The cost of one of the calibrations should come from their budget though !

3 ASG are only due for calibration at SwRI on August 19, 2004. I prefer to keep and use these 3 ASG and the controller until that date. Then on August 19, 2004, I will ship the controller and the 5 ASG to SwRI for calibration. I will not use the 2 ASG which calibration ran out in April 2004. Is this allright ?

Thank you for trying to sort this out. ALl the best,

Anne-Marie

On Wed, 16 Jun 2004, Brittain Hill wrote:

> Anne-Marie - I'm sorry to say that you need to send me the pressure
> transducers as soon as possible, as the Severn calibration just won't be
> acceptable to our QA group. Yes, I agree that this doesn't make sense.
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> done, perhaps it would be best to see if the other pressure sensors are
> coming due for calibration in the next couple of months. I think the
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> before I sent them to you. If you think you'll be done before they fall
> out of calibration, then don't bother. But if there's a chance that
> you'll still be doing an experiment or two with them in August, it would
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> calibrated while the equipment is down.
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> You did mention that only 2 of the ASG transducers were officially out
> of calibration, but the others were good until August 2004. If you think
> you can get the experiments done before those transducers fall out of
> official calibration, then we should hold off on shipping anything and
> see if you can get the experiments done before the calibration expires.
> Otherwise, ship all the gauges that need calibration to me and we'll get
> it done ASAP. Still, that will be several weeks delay.
>
> Please use 1-2 day airfreight possible for these, to minimize delays. It
> doesn't have to be overnight, though.
>

18.06.04

> Thank-you for understanding that it is just not my decision, but if we
> don't do this, they will declare all of the experiments unuseable.
>
> Merci-
> Britt
>
> -------- Original Message --------
> Subject: RE: [Fwd: FW: ASG calibration (fwd)]
> Date: Wed, 16 Jun 2004 13:11:46 -0500
> From: Robert Brient <rbrient@swri.edu>
> Reply-To: rbrient@swri.edu
> Organization: CNWRA
> To: brittain.hill@swri.org
>
> Britt:
>
> From what I can gather, ISO/DIS 3567 is a draft standard for calibrating
> vacuum or pressure measuring devices. It is probably equivalent to an
> ASTM standard method. This draft standard doesn't appear to be
> equivalent to ISO 17025 that addresses calibration systems.
>
> Another consideration is that the supplier does not have a third party
> confirmation (by certification or accreditation).
>
> I think that we are stuck with shipping it back.
>
> Bob Brient
> Director, Quality Assurance
> Center for Nuclear Waste Regulatory Analyses
> Southwest Research Institute
> (210) 522-5537
> (210) 522-6081 fax
>
> -----Original Message-----
> From: Brittain Hill [mailto:bhill@cnwra.swri.edu]
> Sent: Wednesday, June 16, 2004 10:02 AM
> To: Robert Brient
> Subject: [Fwd: FW: ASG calibration (fwd)]
> Importance: High
>
> Bob - For calibration of the gauges in UK:
>
> The manufacturer does their calibrations under ISO/DIS 3567. Is this an
> acceptable standard relative to ISO 17025?
>
> If so, I'll have them send the certification. Otherwise, please let me
> know and I'll have the equipment shipped to us from UK.
>
> Thanks-
> Britt

18.06.04

> -----Original Message-----
> From: Andrew Hilton
> Sent: Wednesday, June 16, 2004 03:11
> To: rstone
> Subject: RE: ASG calibration
>
>
> We do not have this accreditation. Our calibration procedures are
> documented under our ISO 9000 system and the calibration equipment /
> procedures are in accordance with ISO/DIS 3567.

18.06.04

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18.06.04

Just received today by mail the certificates of calibration of the ASG by Severn Science (see following pages) and invoices.

Total cost of calibration = £751.69
or US $1375.47 ..... for nothing
as the manufacturer is not recognised by QA for being competent to calibrate its own products !!!

18.06.04

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Severn SCIENCE SOLUTIONS IN SCIENCE

CERTIFICATE OF CALIBRATION

Universite Pierre & Marie Curie
Laboratoire PMMP, Case 110
4 Place Jussieu
75252 Paris
Cedex 05

Order No:GL31976

Certificate No: 04174

Date: 29.05.04

EQUIPMENT Gauge: Controller (AGC) S/N. 996243404
Head: Strain Gauge (ASG) S/N. 005000334

PROCEDURE
Gauge head cleaned and calibrated against S.S.L Reference Gauge using a high vacuum pumping system, (constructed in accordance with the proposals stated in Draft International Standard ISO/DIS 3567) under controlled conditions. Full procedure outlined in S.S.L Publication IGK046. Refer also to Service Report No. 13866. The calibration references used in carrying out this work can be traced to National Standards.

MASTER GAUGE (Secondary Standard)
Capacitance Manometer (refer to Calibration Certificate N1120315V, NAMAS accreditation 0072).

SSL REFERENCE GAUGE (Tertiary Standard)
Edwards Capacitance Manometer 590 Serial No. 40224614

SSL Reference Gauge (mbar)	Test Pressure (mbar)	Customer Gauge (mbar)
0.9	0.95	1.0
3.0	3.09	3.1
6.0	6.05	6.1
9.0	9.06	9.1
20.0	20.06	20.1
30.0	30.01	30.1
60.0	60.09	60.2
90.0	89.94	90.2
300.0	299.69	300.1
700.0	699.42	699.8
1000.0	1000.0	1000.0

The uncertainty of the reference pressure is ± 0.5% at 1000 mbar and ± 2.5% at 10 mbar

Test Engineer

Authorisation

Severn Science Limited, 8 Brunel Way, Thornbury, Bristol BS35 3UR
Publication Reference: 1EW056-9

18.06.04

Severn SCIENCE SOLUTIONS IN SCIENCE

CERTIFICATE OF CALIBRATION

Universite Pierre & Marie Curie
Laboratoire PMMP, Case 110
4 Place Jussieu
75252 Paris
Cedex 05

Order No:GL31976

Certificate No: 04173

Date: 25.05.04

EQUIPMENT Gauge: Controller (AGC) S/N. 996243404
Head: Strain Gauge (ASG) S/N. 1005000371

PROCEDURE
Gauge head cleaned and calibrated against S.S.L Reference Gauge using a high vacuum pumping system, (constructed in accordance with the proposals stated in Draft International Standard ISO/DIS 3567) under controlled conditions. Full procedure outlined in S.S.L Publication IGK046. Refer also to Service Report No. 13866. The calibration references used in carrying out this work can be traced to National Standards.

MASTER GAUGE (Secondary Standard)
Capacitance Manometer (refer to Calibration Certificate N1120315V, NAMAS accreditation 0072).

SSL REFERENCE GAUGE (Tertiary Standard)
Edwards Capacitance Manometer 590 Serial No. 40224614

SSL Reference Gauge (mbar)	Test Pressure (mbar)	Customer Gauge (mbar)
0.9	0.95	1.0
3.0	3.09	3.1
6.0	6.05	6.1
9.0	9.06	9.1
20.0	20.06	20.1
30.0	30.01	30.1
60.0	60.09	60.2
90.0	89.94	90.2
300.0	299.69	300.3
700.0	699.42	700.3
1000.0	1000.0	1000.0

The uncertainty of the reference pressure is ± 0.5% at 1000 mbar and ± 2.5% at 10 mbar

Test Engineer

Authorisation

Severn Science Limited, 8 Brunel Way, Thornbury, Bristol BS35 3UR
Publication Reference: 1EW056-9

18.06.04

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Severn SCIENCE SOLUTIONS IN SCIENCE

CERTIFICATE OF CALIBRATION

Universite Pierre & Marie Curie
Laboratoire PMMP, Case 110
4 Place Jussieu
75252 Paris
Cedex 05

Order No:GL31976

Certificate No: 04171

Date: 29.05.04

EQUIPMENT Gauge: Controller (AGC) S/N. 996243404
Head: Strain Gauge (ASG) S/N. 995000176

PROCEDURE
Gauge head cleaned and calibrated against S.S.L Reference Gauge using a high vacuum pumping system, (constructed in accordance with the proposals stated in Draft International Standard ISO/DIS 3567) under controlled conditions. Full procedure outlined in S.S.L Publication IGK046. Refer also to Service Report No. 13839. The calibration references used in carrying out this work can be traced to National Standards.

MASTER GAUGE (Secondary Standard)
Capacitance Manometer (refer to Calibration Certificate N1120315V, NAMAS accreditation 0072).

SSL REFERENCE GAUGE (Tertiary Standard)
Edwards Capacitance Manometer 590 Serial No. 40224614

SSL Reference Gauge (mbar)	Test Pressure (mbar)	Customer Gauge (mbar)
0.9	0.95	0.9
3.0	3.09	3.0
6.0	6.05	6.0
9.0	9.06	9.0
20.0	20.06	20.0
30.0	30.01	30.0
60.0	60.09	60.0
90.0	89.94	90.0
300.0	299.69	299.7
700.0	699.42	699.4
1000.0	1000.0	1000.0

The uncertainty of the reference pressure is ± 0.5% at 1000 mbar and ± 2.5% at 10 mbar

Test Engineer

Authorisation

Severn Science Limited, 8 Brunel Way, Thornbury, Bristol BS35 3UR
Publication Reference: 1EW056-9

18.06.04

Severn SCIENCE SOLUTIONS IN SCIENCE

CERTIFICATE OF CALIBRATION

Universite Pierre & Marie Curie
Laboratoire PMMP, Case 110
4 Place Jussieu
75252 Paris
Cedex 05

Order No:GL31976

Certificate No: 04175

Date: 29.05.04

EQUIPMENT Gauge: Controller (AGC) S/N. 996243404
Head: Strain Gauge (ASG) S/N. 005000333

PROCEDURE
Gauge head cleaned and calibrated against S.S.L Reference Gauge using a high vacuum pumping system, (constructed in accordance with the proposals stated in Draft International Standard ISO/DIS 3567) under controlled conditions. Full procedure outlined in S.S.L Publication IGK046. Refer also to Service Report No. 13866. The calibration references used in carrying out this work can be traced to National Standards.

MASTER GAUGE (Secondary Standard)
Capacitance Manometer (refer to Calibration Certificate N1120315V, NAMAS accreditation 0072).

SSL REFERENCE GAUGE (Tertiary Standard)
Edwards Capacitance Manometer 590 Serial No. 40224614

SSL Reference Gauge (mbar)	Test Pressure (mbar)	Customer Gauge (mbar)
0.9	0.95	1.0
3.0	3.09	3.1
6.0	6.05	6.1
9.0	9.06	9.1
20.0	20.06	20.1
30.0	30.01	30.1
60.0	60.09	60.1
90.0	89.94	90.1
300.0	299.69	300.0
700.0	699.42	699.9
1000.0	1000.0	1000.0

The uncertainty of the reference pressure is ± 0.5% at 1000 mbar and ± 2.5% at 10 mbar

Test Engineer

Authorisation

Severn Science Limited, 8 Brunel Way, Thornbury, Bristol BS35 3UR
Publication Reference: 1EW056-9

18.06.04

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Severn SCIENCE SOLUTIONS IN SCIENCE

CERTIFICATE OF CALIBRATION

Universite Pierre & Marie Curie
Laboratoire PMMP, Case 110
4 Place Jussieu
75252 Paris
Cedex 05

Order No:GL31976

Certificate No: 04172

Date: 29.05.04

EQUIPMENT Gauge: Controller (AGC) S/N. 996243404
Head: Strain Gauge (ASG) S/N. 005000055

PROCEDURE
Gauge head cleaned and calibrated against S.S.L Reference Gauge using a high vacuum pumping system, (constructed in accordance with the proposals stated in Draft International Standard ISO/DIS 3567) under controlled conditions. Full procedure outlined in S.S.L Publication IGK046. Refer also to Service Report No. 13839. The calibration references used in carrying out this work can be traced to National Standards.

MASTER GAUGE (Secondary Standard)
Capacitance Manometer (refer to Calibration Certificate N1120315V, NAMAS accreditation 0072).

SSL REFERENCE GAUGE (Tertiary Standard)
Edwards Capacitance Manometer 590 Serial No. 40224614

SSL Reference Gauge (mbar)	Test Pressure (mbar)	Customer Gauge (mbar)
0.9	0.95	1.0
3.0	3.09	3.2
6.0	6.05	6.2
9.0	9.06	9.2
20.0	20.06	20.3
30.0	30.01	30.3
60.0	60.09	60.3
90.0	89.94	90.3
300.0	299.69	300.0
700.0	699.42	699.7
1000.0	1000.0	1000.0

The uncertainty of the reference pressure is ± 0.5% at 1000 mbar and ± 2.5% at 10 mbar

Test Engineer

Authorisation

Severn Science Limited, 8 Brunel Way, Thornbury, Bristol BS35 3UR
Publication Reference: 1EW056-9

18.06.04

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DELIVER TO

Universit'e Pierre et Marie Curie
Labaratoire PMMP,Case 110
4 Place Jussieu
75252 Paris Cedex 05
FRANCE

Se er
SCIENC

INVOICE

INVOICE / CREDIT TO

Bristol University
Department of Earth Sciences
Wills Memorial Building
Queens Road, Clifton
Bristol BS8 1RJ

SEVERN SCIENCE LIMITEI
8 Brunel Way, Thornbury, Bristol BS35 3UR,
Telephone: (01454) 414723 Facsimile: (0145
E-mail: sales@severnscience.co.uk
Wesite: www.severnscience.co.uk
Company Registration Number : 935827 En
VAT Registration Number: 138 5274 5.

ACCOUNT No.	REP.	YOUR ORDER NUMBER	DELIVERY DATE	DELIVERY REFERENCE	INVOICE DATE	INVOICE NUMBER	TRAN
B2098		GL31976	29th May 2004		29th May 2004	1003019	13839

PRODUCT	DESCRIPTION OF GOODS	UNIT	QUANTITY	PRICE	DISC %	NETT VAL
Traceable calibration			2			£230.00
Carriage			1			£64.67

ADDRESS CHANGE

Severn Science Limited
8 Brunel Way,
Thornbury, Bristol BS35 3UR

VAT CODE	GOODS	VAT RATE	VAT AMOUNT
	£294.67	17.5%	£51.57

INVOICE GOODS	INVOICE VAT	INVOICE T
£294.67	£51.57	£346.24

ds remain the property of Severn Science Limited until paid for in full.

18.06.04

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Severn Science Ltd. 8 Brunel Way,Thornbury Ind. Est. South Glos BS35 3UR
Tel : 01454-414723 Fax : 01454-281680 E-mail : service@severnscience.co.uk

Servicing Gauge & Gauge Heads

Ref No : 13839 Inv No : 1003019 Order No : GL31976 Date : 29th May 2004

AC No : B2098

Service At :

Customer Address
Universit'e Pierre et Marie Curie
Labaratoire PMMP,Case 110
4 Place Jussieu
75252 Paris Cedex 05
FRANCE

Invoice Address
Bristol University
Department of Earth Sciences
Wills Memorial Building
Queens Road, Clifton
Bristol BS8 1RJ

Contact : Anne-Marie Lejeune Tel No : 0117 9545441 Fax No : e-mail amlejeun@ccr.ju

Log Date : 23rd Apr 2004 Health & Safety Form : ✓ Date Received :

Reason for Repair: Service& calibrate

Cost Before Proceeding: yes Traceability ✓ Certification ✓

Manufacturer : Edwards Model : AGC 2 x ASG 0-1000 mbar Serial No : 996243404 995000176 & 005000055

Work Carried Out (If Necessary)

Gauge Head

Filaments : ☐
Lead : ☐
Test on Calibration Unit : ☐
Other Work : ☐

Gauge

Initial Test / Clean : ☐
Elec. Res. Checks : ☐
Other Work : ☐

Cal. Conds.	Cal. Fluid	T1 Deg. C	T2 Deg. C	TAV Deg. C	Atmos. Pressure
Trace	N2	21	21	21	1012

Remarks: One strain gauge needed repairing, this was carried out.

Travel Out	On-Site Labour	Travel Return	Engineer/Customer Signature	QA
	In-house		Steve Davies PP. A.W.Hall.	

Materials Code	Qty	Total	Materials Code	Qty	Total
Traceable calibration	2	£230.00	Carriage	1	£64.67

18.06.04

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DELIVER TO

Universit'e Pierre & Marie Curie
Laboratoire PMMP,Case110
4 Place Jussieu
75252 Paris Cedex 05
France

Severn SCIENCE

INVOICE

INVOICE / CREDIT TO

Bristol University
Department of Earth Sciences
Wills Memorial Building
Queens Road, Clifton
Bristol BS8 1RJ

SEVERN SCIENCE LIMITED
8 Brunel Way, Thornbury, Bristol BS35 3UR,
Telephone: (01454) 414723 Facsimile: (01454
E-mail: sales@severnscience.co.uk
Wesite: www.severnscience.co.uk
Company Registration Number : 935827 Eng
VAT Registration Number: 138 5274 55

ACCOUNT No.	REP.	YOUR ORDER NUMBER	DELIVERY DATE	DELIVERY REFERENCE	INVOICE DATE	INVOICE NUMBER	TRANS
B2098		GL31976	28th May 2004		28th May 2004	1003018	13866

PRODUCT	DESCRIPTION OF GOODS	UNIT	QUANTITY	PRICE	DISC %	NETT VAL
Traceable calibration			3			£345.00

ADDRESS CHANGE

Severn Science Limited
8 Brunel Way,
Thornbury, Bristol BS35 3UR

VAT CODE	GOODS	VAT RATE	VAT AMOUNT
	£345.00	17.5%	£60.38

INVOICE GOODS	INVOICE VAT	INVOICE TOT
£345.00	£60.38	£405.38

ds remain the property of Severn Science Limited until paid for in full.

18.06.04

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Severn Science Ltd. 8 Brunel Way,Thornbury Ind. Est. South Glos BS35 3UR
Tel : 01454-414723 Fax : 01454-281680 E-mail : service@severnscience.co.uk

Servicing Gauge & Gauge Heads

Ref No : 13866 Inv No : 1003018 Order No : GL31976 Date : 28th May 2004

AC No : B2098

Service At :

Customer Address
Universit'e Pierre & Marie Curie
Laboratoire PMMP,Case110
4 Place Jussieu
75252 Paris Cedex 05
France

Invoice Address
Bristol University
Department of Earth Sciences
Wills Memorial Building
Queens Road, Clifton
Bristol BS8 1RJ

Contact : Dr.Anne-Marie Lejeune Tel No : 0117 9545441 Fax No : 0117 9253385

Log Date : 5th May 2004 Health & Safety Form : ☐ Date Received :

Reason for Repair: Service
Cost Before Proceeding: ☐ Traceability ☐ Certification ☐

Manufacturer : Edwards Model : 4 x Active Strain Gauges Serial No : 500333-500371-500334 99500241

Work Carried Out (If Necessary)

Gauge Head

Filaments : ☑
Lead : ☒
Test on Calibration Unit : ☑
Other Work : ☐

Gauge

Initial Test / Clean : ☐
Elec. Res. Checks : ☐
Other Work : ☐

Cal. Conds.	Cal. Fluid	T1 Deg. C	T2 Deg. C	TAV Deg. C	Atmos. Pressure
Trace	N2	21	21	21	1012

Remarks: S/No. 995000241 will not zero and will need replacing.

Travel Out	On-Site Labour	Travel Return	Engineer/Customer Signature	QA
	In-house		Steve Davies PP. A.W.Hall	

Materials Code	Qty	Total	Materials Code	Qty	Total
Traceable calibration	3	£345.00			

18.06.04

Date: Fri, 18 Jun 2004 08:43:12 -0500
From: Brittain Hill <bhill@cnwra.swri.edu>
Reply-To: bhill@swri.org
To: Anne-Marie LEJEUNE <amlejeun@ccr.jussieu.fr>
Subject: Re: [Fwd: RE: [Fwd: FW: ASG calibration (fwd)]]

D'accord. Please continue to use the 3 ASG gauges that are in calibration until 8/19/04. Over the next several weeks, I think you can determine if you will be able to complete (as I hope) the experiments by 19 August. If it looks like you will need to do more experiments after 19 Aug, then you should try and send me/bring the gauges & controller to our meeting in DC. This will minimize the disruption in the experimental work.

Please make sure you note in the notebook that you are not (and have not been) using the officially "out-of-calibration" gauges in the experiments between 19 Feb 2004 and present. I know this will be audited, and if you make it clear that these gauges were not used, then the questions will be easy for me to answer.

Also, please do not forget to send me the copy of your scientific notebook tout suite, when you return to Paris. That should settle the snarling beasts for a while.

Thanks-
Britt

PS - you have made hotel reservations for DC at the Woodfin Suites in Rockville, yes?

Anne-Marie LEJEUNE wrote:

> Britt,
>
> I know and I understand that it is not your decision. I am only very rude
> (as much as possible !) to the QA people but I also understand that they
> have a job to do. The cost of one of the calibrations should come from
> their budget though !
>
> 3 ASG are only due for calibration at SwRI on August 19, 2004. I prefer to
> keep and use these 3 ASG and the controller until that date. Then on
> August 19, 2004, I will ship the controller and the 5 ASG to SwRI for
> calibration. I will not use the 2 ASG which calibration ran out in April
> 2004. Is this allright ?
>
> Thank you for trying to sort this out. ALl the best,
>
> Anne-Marie

18.06.04

> On Wed, 16 Jun 2004, Brittain Hill wrote:
>
> > Anne-Marie - I'm sorry to say that you need to send me the pressure
> > transducers as soon as possible, as the Severn calibration just won't be
> > acceptable to our QA group. Yes, I agree that this doesn't make sense.
> > But that is the way it is with them. Selah.
> >
> > Please send me whatever needs to be sent for calibration. I think the
> > controller is needed, along with the transducers. Since this has to be
> > done, perhaps it would be best to see if the other pressure sensors are
> > coming due for calibration in the next couple of months. I think the
> > small PCB(?) sensors (in the glass tube) were done last August, just
> > before I sent them to you. If you think you'll be done before they fall
> > out of calibration, then don't bother. But if there's a chance that
> > you'll still be doing an experiment or two with them in August, it would
> > be best to send them along with the ASG transducers and get everything
> > calibrated while the equipment is down.
> >
> > You did mention that only 2 of the ASG transducers were officially out
> > of calibration, but the others were good until August 2004. If you think
> > you can get the experiments done before those transducers fall out of
> > official calibration, then we should hold off on shipping anything and
> > see if you can get the experiments done before the calibration expires.
> > Otherwise, ship all the gauges that need calibration to me and we'll get
> > it done ASAP. Still, that will be several weeks delay.
> >
> > Please use 1-2 day airfreight possible for these, to minimize delays. It
> > doesn't have to be overnight, though.
> >
> > Thank-you for understanding that it is just not my decision, but if we
> > don't do this, they will declare all of the experiments unuseable.
> >
> > Merci-
> > Britt
> >
> > -------- Original Message --------
> > Subject: RE: [Fwd: FW: ASG calibration (fwd)]
> > Date: Wed, 16 Jun 2004 13:11:46 -0500
> > From: Robert Brient <rbrient@swri.edu>
> > Reply-To: rbrient@swri.edu
> > Organization: CNWRA
> > To: brittain.hill@swri.org
> >
> > Britt:
> >
> > From what I can gather, ISO/DIS 3567 is a draft standard for calibrating
> > vacuum or pressure measuring devices. It is probably equivalent to an
> > ASTM standard method. This draft standard doesn't appear to be
> > equivalent to ISO 17025 that addresses calibration systems.
> >

18.06.04

> > Another consideration is that the supplier does not have a third party
> > confirmation (by certification or accreditation).
> >
> > I think that we are stuck with shipping it back.
> >
> > Bob Brient
> > Director, Quality Assurance
> > Center for Nuclear Waste Regulatory Analyses
> > Southwest Research Institute
> > (210) 522-5537
> > (210) 522-6081 fax
> >
> > -----Original Message-----
> > From: Brittain Hill [mailto:bhill@cnwra.swri.edu]
> > Sent: Wednesday, June 16, 2004 10:02 AM
> > To: Robert Brient
> > Subject: [Fwd: FW: ASG calibration (fwd)]
> > Importance: High
> >
> > Bob - For calibration of the gauges in UK:
> >
> > The manufacturer does their calibrations under ISO/DIS 3567. Is this an
> > acceptable standard relative to ISO 17025?
> >
> > If so, I'll have them send the certification. Otherwise, please let me
> > know and I'll have the equipment shipped to us from UK.
> >
> > Thanks-
> > Britt
> >
> >
> >
> > -----Original Message-----
> > From: Andrew Hilton
> > Sent: Wednesday, June 16, 2004 03:11
> > To: rstone
> > Subject: RE: ASG calibration
> >
> >
> > We do not have this accreditation. Our calibration procedures are
> > documented under our ISO 9000 system and the calibration equipment /
> > procedures are in accordance with ISO/DIS 3567.

--

Dr. Brittain E. Hill, P.G. bhill@swri.org
Senior Research Scientist www.swri.org
CNWRA-Southwest Research Institute Ph:(210) 522-6087
6220 Culebra Rd. FAX: 210-522-5155
San Antonio, TX, USA 78238-5166 In Magma Veritas

18.06.04

18.06.04

Date: Fri, 18 Jun 2004 15:55:22 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: bhill@swri.org
Subject: Re: [Fwd: RE: [Fwd: FW: ASG calibration (fwd)]]

On Fri, 18 Jun 2004, Brittain Hill wrote:

> D'accord. Please continue to use the 3 ASG gauges that are in calibration
> until 8/19/04. Over the next several weeks, I think you can determine if you
> will be able to complete (as I hope) the experiments by 19 August. If it looks
> like you will need to do more experiments after 19 Aug, then you should try
> and send me/bring the gauges & controller to our meeting in DC. This will
> minimize the disruption in the experimental work.

Thanks Britt. I was considering bringing ths ASG and controller to DC, but I might prefer to use them in July until mid-August.

> Please make sure you note in the notebook that you are not (and have not been)
> using the officially "out-of-calibration" gauges in the experiments between 19
> Feb 2004 and present. I know this will be audited, and if you make it clear
> that these gauges were not used, then the questions will be easy for me to answer.

It has been done already a few months ago when you first contacted me about the 2 gauges. I wouldn't mind sending you these two now, but I do need the controller and so does the cal lab. So it is better to send everything (5 gauges and controller) together later. One point worth mentionning though: the 5 ASG were last calibrated all together. The 2 out-of-calibration gauges are such because I initially told Mack Wood at SwRI that I would like to have the gauges checked every 6 months to make sure that the data recorded were fine. But actually one calibration once a year was considered to be sufficient according to SwRI Cal Lab. The 3 other ASG are thus recorded as needing calibration once a year, which doesn't prevent me to ask for an earlier calibration if I feel that something might be wrong with one of them.

> Also, please do not forget to send me the copy of your scientific notebook
> tout suite, when you return to Paris. That should settle the snarling beasts
> for a while.

Started to make photocopies today. This is my notes as they currently are. I will make a neat version over the summer.

> Thanks-
> Britt
>
> PS - you have made hotel reservations for DC at the Woodfin Suites in
> Rockville, yes?

Forgot about it. Will do it right now.

Cheers,

AML

18.06.04

488

Date: Fri, 18 Jun 2004 16:25:18 +0200 (DFT)
From: Anne-Marie LEJEUNE <amlejeun@moka.ccr.jussieu.fr>
To: Brittain Hill <bhill@cnwra.swri.edu>
Subject: Hotel booked

Britt,

Just to confirm that I have booked my room at the Woodfin Suite Hotel Rockville (USD 109/night). The magic of internet :-)

I am faxing you the calibration sheets of the 5 ASG. Just received them in the mail.

Cheers,

Anne-Marie

Anne-Marie LEJEUNE
Universite Pierre et Marie Curie
Laboratoire de Petrologie, Modelisation des Materiaux et Processus
Case 110
4 place Jussieu
75252 Paris cedex 05 France

Tel. +33 (0)1 44 27 22 41 Messages +33 (0)1 44 27 50 44
Fax +33 (0)1 44 27 39 11

EU Volcano Dynamics Research Training Network website:
http://gfd.gly.bris.ac.uk/euvolcanonet/intro.htm

18.06.04

22.06.04
① 489

All cups out of the oven (T = 94°C) @ 9H00.
Left to cool down to room temperature, covered w/ paper sheets to prevent any loss of GR chips.

Weighing from 15H 22

	Cup	m (g)
Tray 1	P1	13.9997
	P2	16.2433
	P3	16.2624
	P4	38.0381
	P5	17.9724
	P6	28.4941
	P7	27.5163
	P8	23.4956
	P9	18.3614
	P10	20.5545
	P11	14.4281
	P12	13.0369
	P13	28.0803

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(2)

	Cup	m (g)
Tray 2	P14	14.8864
	P15	20.6511
	P16	7.7061
	P17	17.9652
	P18	12.8301
	P19	16.2341
	P20	15.4080
	P21	17.5598
	P22	13.1992
	P23	15.1864
	P24	15.7845
	P25	17.0663
	P26	13.2406
Tray 3	P27	24.8221
	P28	6.8235
	P29	17.4124
	P30	18.8609
	P31	8.7653

Done @ 15H35

All cups back in the oven @ 15H37 @ 94°C and covered up w/ paper sheets to prevent any loss of GR chips during cracking induced by rapid heating.

Paper sheets removed @ H. $T_{oven} = 94°C$

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All GRA cups out of the oven (T_{oven} = 94°C) @ 14H45. Let cool down to room temperature covered w/ paper sheets to prevent any loss of weight/GR ships while boiling-induced "cracking" occurs.

Weighing from 16H24

	Cup	m (g)
Tray 1	P1	13.9897
	P2	16.2320
	P3	16.2518
	P4	38.0123
	P5	17.9619
	P6	28.4747
	P7	27.4989
	P8	23.4800
	P9	18.3504
	P10	20.5437
	P11	14.4204
	P12	13.0301
	P13	28.0634
Tray 2	P14	14.8773
	P15	20.6340
	P16	7.7020

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(2)

	Cup	m (g)
	P17	17.9514
	P18	12.8203
	P19	16.2217
	P20	15.3961
	P21	17.5468
	P22	13.1892
	P23	15.1757
	P24	15.7733
	P25	17.0514
	P26	13.2306
Tray 3	P27	24.7997
	P28	6.8200
	P29	17.3983
	P30	18.8456
	P31	8.7604

All cups back in the oven, covered w/ paper sheets @ 16H39 - T_{oven} = 94/95°C

All paper sheets removed @ 18H00 - T_{oven} = 94°C

05.07.04

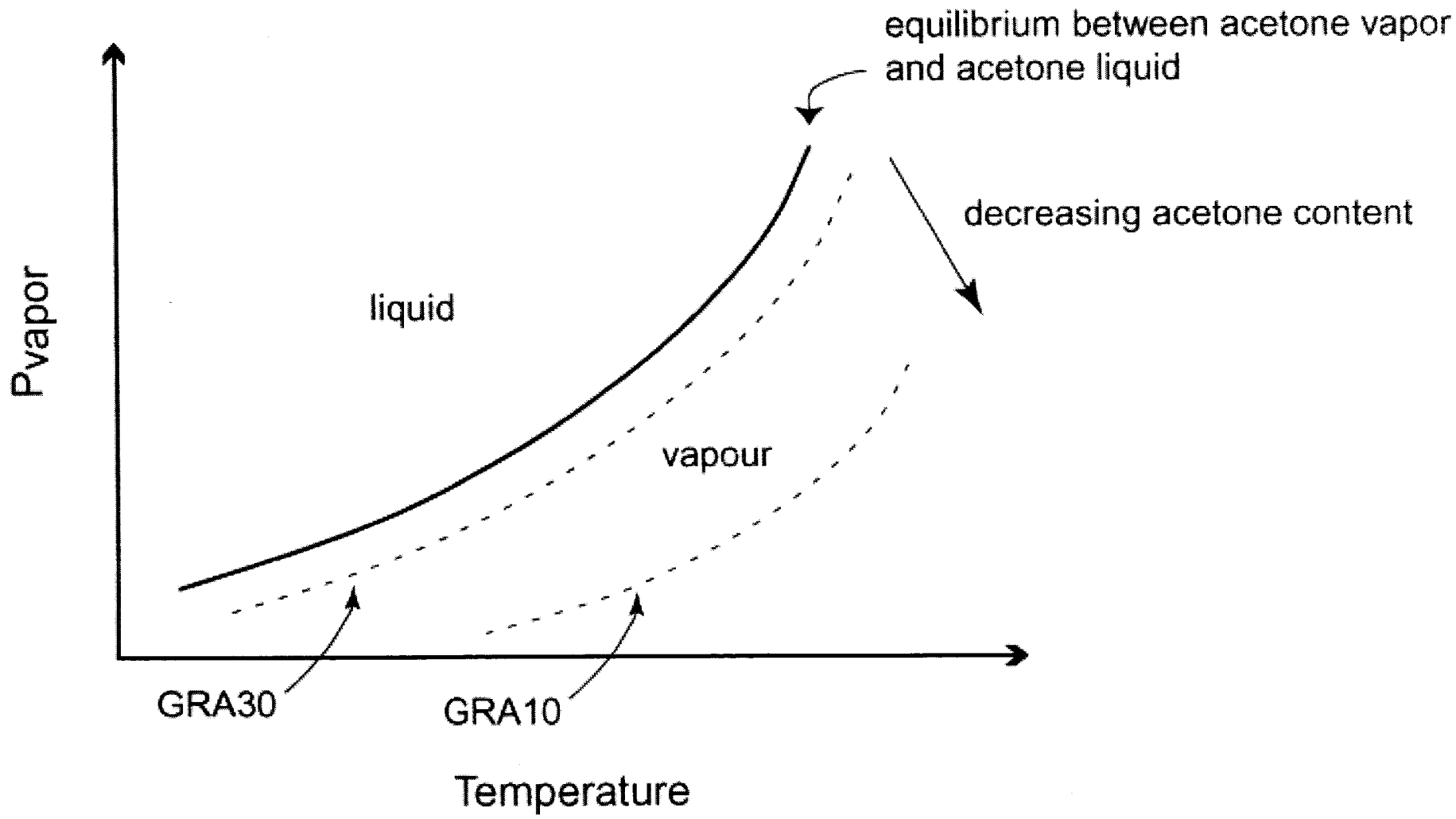
equilibrium between acetone vapor
and acetone liquid
decreasing acetone content
liquid
vapour
Pvapor
GRA30
GRA10
Temperature

Fig. 07.04

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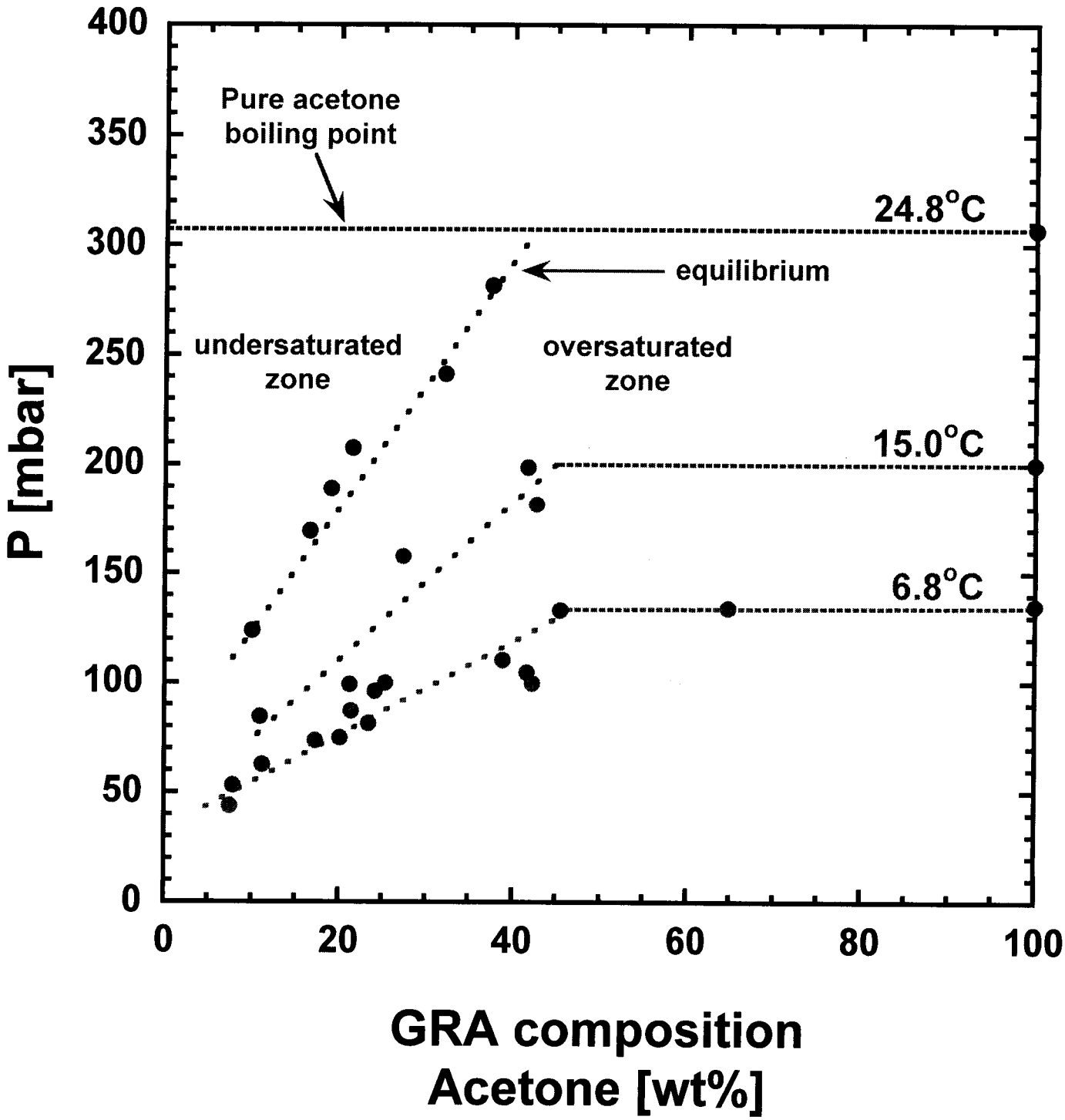
18.07.04

Summary of the GRA EoS data

T = 6.8°C		T = 24.8°C		T = 15.0°C	
GRA composition Acetone [wt%]	P [mbar]	GRA composition Acetone [wt%]	P [mbar]	GRA composition Acetone [wt%]	P [mbar]
23.43	81.40	19.05	189.00	41.73	198.60
20.14	74.80	32.21	241.20	21.27	99.20
41.68	104.60	16.64	169.20	27.45	157.70
24.18	96.00	10.06	123.70	42.74	181.50
11.26	62.60	21.50	207.40	10.98	84.40
21.44	87.00	37.59	281.50	100.00	200.00
25.41	99.90	100.00	307.00		
42.30	99.60				
7.55	43.60				
17.33	73.40				
7.90	53.10				
38.90	110.20				
100.00	135.00				
45.49	133.00				
64.80	133.90				

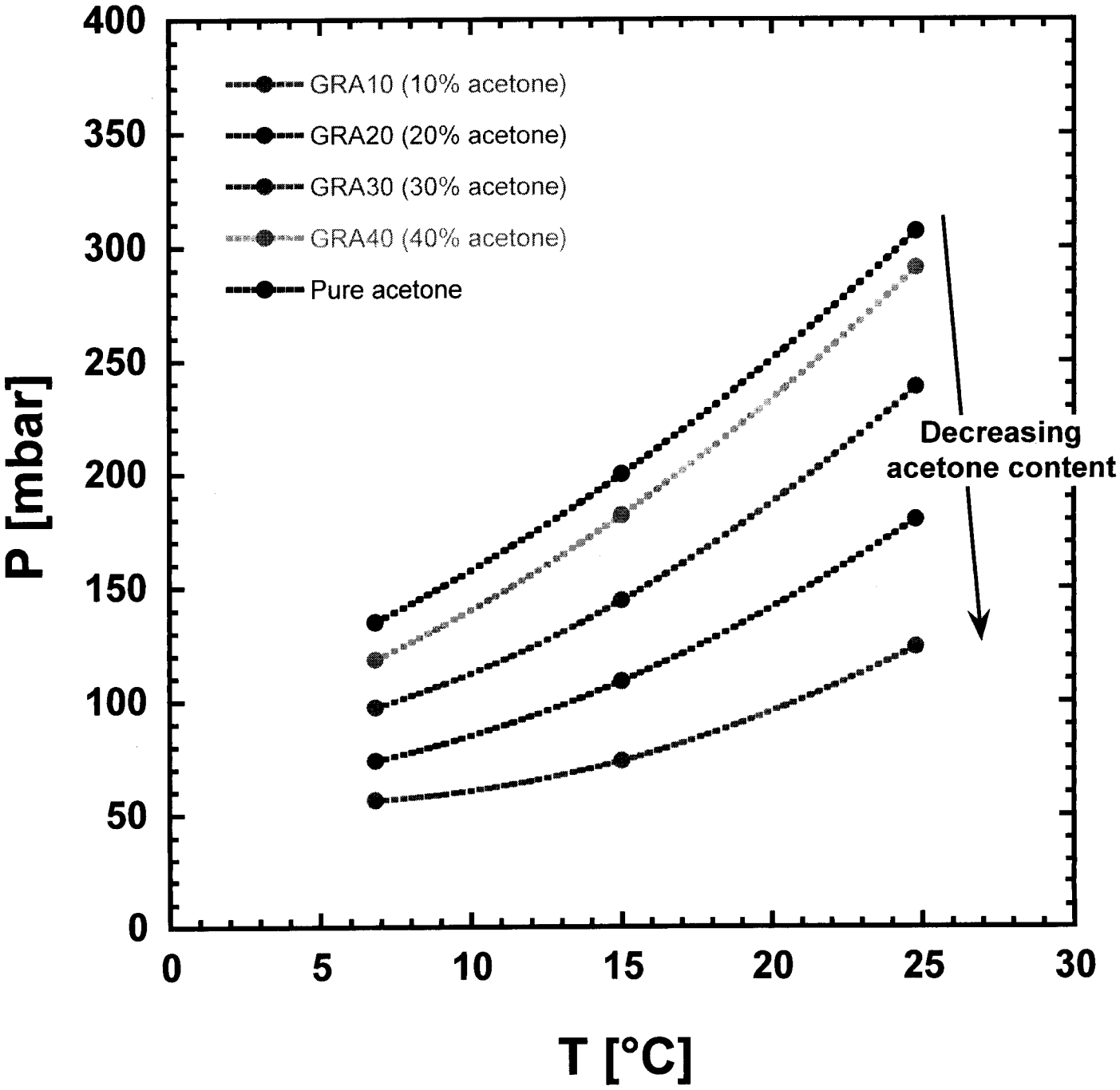
12.07.04

GRA system



19.07.04

GRA system



12.07.04

I have reviewed scientific notebook 555 and find it in compliance with QAP-001, except that it covers work through July 12, 2004. There is sufficient information regarding procedure used for conducting the research and acquiring and analyzing the data so that another qualified scientist could repeat the activity or activities recorded in this scientific notebook

H Lawrence McKague 10/27/04

H. Lawrence McKague
GLGP Element Manager

GEOSCIENCES AND ENGINEERING DIVISION

SCIENTIFIC NOTEBOOK REVIEW CHECKLIST RECORD

Scientific Notebook No. 555 Project Numbers: 20.1402.461

Accomplished

- [✓] 1. Initial entries per QAP-001
- [✓] 2. Dating of entries
- [✓] 3. Corrections (crossed out, one line through w/initials/date)
- [✓] 4. No White out used
- [✓] 5. Page number visible on copy or original notebook
- [✓] 6. In process entries per QAP-001
- [✓] 7. Figure information present
- [✓] 8. Text readable
- [NA] 9. Copyrighted material is identified
- [✓] 10. Permanent ink or type only
- [✓] 11. Signing of entries (not required on each page)
- [✓] 12. Electronic media in the scientific notebook properly labeled
- [✓] 13. NRC Supplementary Scientific Notebook Questions are addressed.
- [✓] 14. The independent, two person verification required by AP-019, Section 5.2.1.2(b) is complete

This notebook was recovered from periodic submittals for disaster recovery. Despite best efforts, we have not been able to obtain the original from Dr. LeJeune in France. We believe that little or no data were not recorded. RPBrient 12/11/07

Any discrepancies must be resolved before notebook closeout.

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I have reviewed this scientific notebook and find it in agreement with QAP-001.

Manager's Signature 12/28/07 Date

Attach this completed form to the last page of the notebook.

Form QAP-01 (03/2007)