INFORMATION CLEARANCE REVIEW AND RELEASE APPROVAL

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Title: Validation Testing for Automated Solubility Measurement Equipment Final Report	Information Category: ☐ Abstract ☐ Journal Article ☐ Summary ☐ Internet ☐ Visual Aid ☐ Software ☐ Full Paper ☒ Report ☐ Other
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VALIDATION TESTING FOR AUTOMATED SOLUBILITY MEASUREMENT EQUIPMENT FINAL REPORT

J. S. Lachut
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EXECUTIVE SUMMARY

Laboratory tests have been completed to test the validity of automated solubility measurement equipment using sodium nitrate and sodium chloride solutions (see test plan WRPS-1404441, “Validation Testing for Automated Solubility Measurement Equipment”).

The sodium nitrate solution results were within 2-3% of the reference values, so the experiment is considered successful using the turbidity meter.

The sodium chloride test was done by sight, as the turbidity meter did not work well using sodium chloride. For example, the “clear” turbidity reading was 53 FNU at 80 ˚C, 107 FNU at 55 ˚C, and 151 FNU at 20 ˚C. The sodium chloride did not work because it is granular and large; as the solution was stirred, the granules stayed to the outside of the reactor and just above the stir bar level, having little impact on the turbidity meter readings as the meter was aimed at the center of the solution. Also, the turbidity meter depth has an impact. The salt tends to remain near the stir bar level. If the meter is deeper in the slurry, it will read higher turbidity, and if the meter is raised higher in the slurry, it will read lower turbidity (possibly near zero) because it reads the “clear” part of the slurry.

The sodium chloride solution results, as measured by sight rather than by turbidity instrument readings, were within 5-6% of the reference values.

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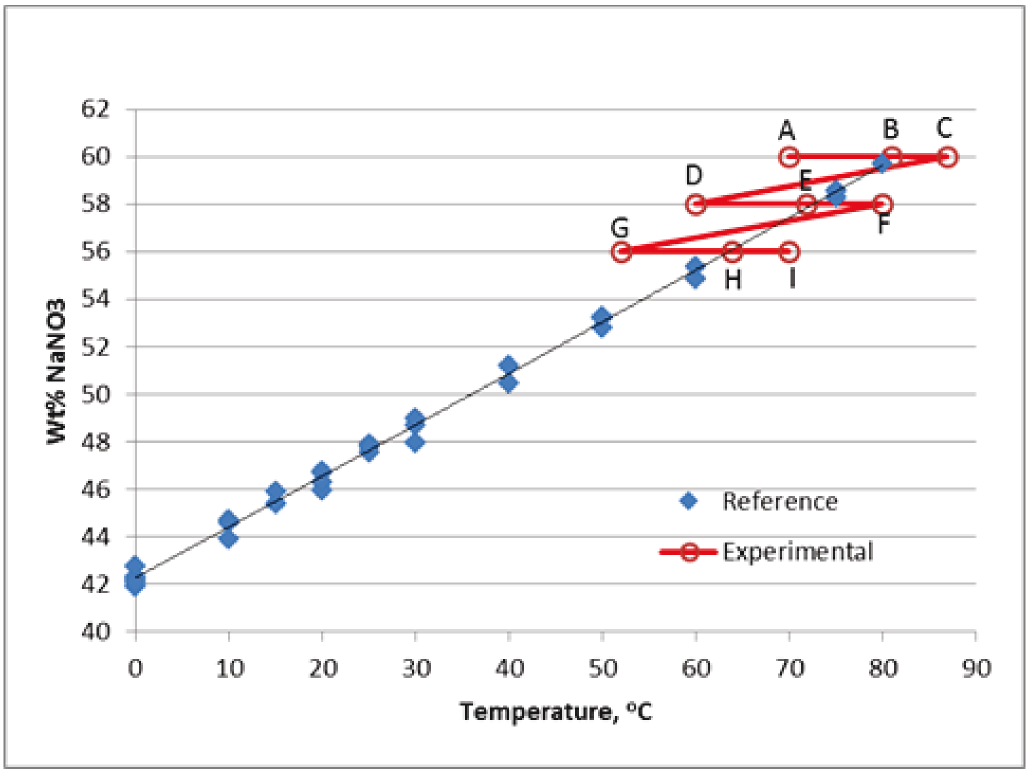
Units

°C	degrees Celsius
°F	degrees Fahrenheit
FNU	Formazin Nephelometric Unit
g	gram
mL	milliliter
%	percent
wt%	weight percent

1 BACKGROUND

The 222-S Laboratory developed a standalone apparatus for measuring solubility that includes a "reactor" (a round bottom flask with multiple penetrations), a turbidity meter to monitor the amount of solids in the slurry, a computer to monitor conditions and control reagent addition, a pump for adding reagent to the reactor, a water circulator for temperature control, and thermocouples to monitor the temperature of the slurry in the reactor and the water in the circulator. The computer tracks the time, date, turbidity of the slurry, slurry temperature, water bath temperature, and amount of stock solution added to the reactor. It also marks the nucleation temperature and the dissolution temperature, based on turbidity changes. The data generated by this automated equipment in testing performed according to WRPS-14-04441, "Validation Testing for Automated Solubility Measurement Equipment," intended to validate the equipment and procedures, is the subject of this report.

Figure 1-1. Sodium Nitrate Solubility in Pure Water



Sodium nitrate solubility is strongly dependent on temperature. In this testing, a 60 wt% sodium nitrate solution was heated to approximately 80 ˚C until all the solids were dissolved (turbidity near zero). The computer then triggered the circulator to cool the solution until the sodium nitrate precipitated (based on a sudden increase in turbidity), corresponding to point A in Figure 1-1 above. At this point, the computer prompted the water circulator to begin heating the solution slowly; this continued until the last remaining crystals dissolved (turbidity returned to near zero). This corresponds to point B and a little bit beyond point C in Figure 1.1. In this process, Point B represents the point at which the turbidity goes to "zero," but the heating continued a little beyond that point, to point C, before water was added to lower the wt% sodium nitrate. The computer recorded the dissolution temperature (point B) based on the sudden drop in turbidity reading. This dissolution temperature generated one point for the plot of temperature vs. wt% sodium nitrate. At point C, the computer triggered the pump to add a small amount of water to the reactor, lowering the wt% sodium nitrate in the slurry, cooling the solution, and causing the salt to precipitate again. These steps were repeated to generate additional data points (points D, E, and F and G, H, and I in Figure 1-1) that occurred at lower temperatures and concentrations than the first data point. This action of temperature/turbidity generates the sinusoidal curve of turbidity over time.

Jacob McCoskey provided the following explanation of the turbidity change needed to trigger the computer to mark the temperature: the nucleation temperature and dissolution temperature are determined using an algorithm that requires some user set value for change in turbidity to overcome the inherent noise of the meter (Attachment). For example, when looking for the nucleation temperature, the software notes when the turbidity increases and stores the time and temperature of the turbidity increase. If the turbidity continues to the user-defined increase in turbidity, say 10-30 FNU, then the algorithm retrieves the nucleation temperature when the turbidity first began to increase.

In the same e-mail, Mr. McCoskey also explained why all temperature readings are reported to the nearest tenth of a degree +/- 1 ºC: an El-1034 chip is used to measure the temperature, and its reported accuracy is +/- 0.4 ºF (LabJack Measurement & Automation, Queried 11/2/2015, EI-1034 Datasheet, http://labjack.com/support/ei-1034/datasheet). Also, the turbidity meter has a reported accuracy of +/- 0.1 FNU, which would have some effect on the detection of the nucleation temperature and dissolution temperature. The turbidity slope must be quantified to determine the effect on temperature (TSS HT sc TriClamp, Suspended Solids TriClamp inline Sensor, Queried 11/2/2015, [TSS sc Suspended Solids Family of Sensors Data Sheet], http://www.hach.com/tss-ht-sc-triclamp-suspended-solids-triclamp-inline-sensor/product-downloads?id=7640284926&callback=pf.). Mr. McCoskey indicated that he would report a range for each temperature with the number reported to the tenth of a degree at most, but to the nearest whole number to be conservative, knowing the system. This would cover most known analytical error and likely the noise.

Sodium chloride solubility is not very dependent on temperature. In this testing, dissolution was achieved by addition of water, not temperature change. A 30% sodium chloride solution was heated to 80 ˚C. Water was added in small increments while maintaining the temperature at 80 ˚C. Water addition continued until the turbidity was near zero (as measured by sight). This slurry composition provided a point on the wt% sodium chloride vs. temperature plot. A sufficient quantity of sodium chloride was added to return the composition to 30 wt% sodium chloride. The slurry was cooled to 70 ˚C, and the previous steps were repeated to generate a second data point. The slurry was then cooled to 60 ˚C, and in increments of 5 ˚C, down to 20 ˚C to generate the solubility curve.

2 TESTING

2.1 SODIUM NITRATE

American Chemical Society reagent grade sodium nitrate was used along with reagent water. Three separate runs of a 60% solution were made up and tested. 150 g of sodium nitrate and 100 g of water were weighed out and placed into the reactor. The temperature of the reactor was increased to approximately 80 °C, and when all other operational aspects of the system were acceptable, the experiment was run. The resulting data plots are shown in Figure 4-1. The data points are reported in Tables 3-1 through 3-5.

Calculations were done as follows: Run 1 (see Table 3-1):

Equation 1: 150.02 g $NaNO_3$/150.02 g $NaNO_3$+ 100.01 g H_2O = 60 wt% $NaNO_3$ (first concentration)

Equation 2: 150.02 g $NaNO_3$/150.02 g $NaNO_3$+ 100.01 g H_2O + (641.879 g H_2O – 639.701 g H_2O) = 59.5 wt% $NaNO_3$ (second concentration)

Equation 3: 150.02 g $NaNO_3$/ 150.02 g $NaNO_3$+ 100.01 g H_2O + 2.178 g H_2O + (639.701 g H_2O – 637.498 g H_2O) = 59 wt% $NaNO_3$

2.2 SODIUM CHLORIDE

American Chemical Society reagent grade sodium chloride was used along with reagent water. Three separate runs of a 30% sodium chloride solution were made up and tested. 45 g of sodium chloride and 105 g of water were weighed out and placed into the reactor. The temperature of the reactor was increased to approximately 80 °C and reduced stepwise to 20 °C as described in Section 1. The experiments were run manually, with the endpoint determined by eyesight and the water addition performed by an automated pump setup and a balance. The resulting data plots are shown in Figure 4-2.

Calculations were done as follows: Run 1 (see Table 3-6):

Equation 4: 45 g NaCl/45 g NaCl + 105 g H_2O + 21.237 g H_2O = 26.3 wt% NaCl

Equation 5: 45 g NaCl + 6.37 g NaCl/ 45 g NaCl + 105 g H_2O + 21.237 g H_2O + 6.37 g NaCl + 20.735 g H_2O = 25.9 wt% NaCl

NOTE: The grams of water added must be multiplied by 0.3 (30%) in order to maintain the 30 wt% NaCl. In Equation 5 above, 21.237 g H_2O was added to dissolve the 30 wt% NaCl solution at 80 °C. Therefore, 21.237 g H_2O x 0.3 = 6.37 g NaCl was added to the solution.

Equation 6: 45 g NaCl + 6.37 g NaCl + 6.22 g NaCl/ 45 g NaCl + 105 g H_2O + 21.237 g H_2O + 6.37 g NaCl + 20.735 g H_2O + 6.22 g NaCl + 20.209 g H_2O = 25.6 wt% NaCl

3 TABLES

Table 3-1. Sodium Nitrate Solubility – Run 1

12/22/2014				**$NaNO_3$ Solubility - Run 1**	
				150.02 grams $NaNO_3$ + 100.01 grams H_2O	
Weight % $NaNO_3$	*Temperature Range (°C +/-1 °C)*			*Dissolution Temperature (°C +/- 1 °C)*	*Balance Reading*
	Start	→	*End*		*(grams)*
60	85.1	→	85.1	85.1	644.007
59.5	85.1	→	83.1	83.1	641.879
59	83.1	→	80.5	80.5	639.701
58.5	80.5	→	78	78	637.498
58	78	→	76.1	76.1	635.403
57.5	76.1	→	74.4	74.4	633.257
57	74.4	→	72.3	72.3	631.071
56.6	72.3	→	70.3	70.3	628.901
56.1	70.3	→	68	68	626.748
55.7	68	→	66.1	66.1	624.562
55.2	66.1	→	64.3	64.3	622.42
54.8	64.3	→	62.4	62.4	620.237
54.4	62.4	→	60.6	60.6	618.128
54	60.6	→	58.4	58.4	616.051
53.5	58.4	→	56.7	56.7	613.925
53.2	56.7	→	55	55	611.838
52.8	55	→	53	53	609.695
52.4	53	→	51.4	51.4	607.54
52	51.4	→	49.6	49.6	605.455
51.6	49.6	→	47.8	47.8	603.311
51.3	47.8	→	46	46	601.219
50.9	46	→	44.4	44.4	599.094
50.5	44.4	→	42.6	42.6	596.951
50.1	42.6	→	40.9	40.9	594.839
49.8	40.9	→	39.4	39.4	592.667
49.4	39.4	→	37.8	37.8	590.547
49.1	37.8	→	36.4	36.4	588.43
48.8	36.4	→	35	35	586.331
48.4	35	→	33.5	33.5	584.22
48.1	33.5	→	32.1	32.1	582.081
47.8	32.1	→	30.5	30.5	579.929
47.4	30.5	→	29.2	29.2	577.84
47.1	29.2	→	27.8	27.8	575.744
46.8	27.8	→	NA	NA	573.594

Table 3-2. Sodium Nitrate Solubility – Run 2

12/29/2014					**$NaNO_3$ Solubility - Run 2**					
					150.04 grams $NaNO_3$ + 100.07 grams H_2O					
Weight % $NaNO_3$		*Temperature Range (°C +/-1 °C)*				*Dissolution Temperature (°C +/- 1 °C)*			*Balance Reading*	
		Start	→	*End*					*(grams)*	
60		86.6	→	86.6		86.6			573.64	
59.2		86.6	→	82.6		82.6			570.486	
58.5		82.6	→	80		80			567.377	
57.8		80	→	77		77			564.213	
57.1		77	→	73.7		73.7			561.034	
56.4		73.7	→	70.8		70.8			557.872	
55.8		70.8	→	68.1		68.1			554.693	
55.1		68.1	→	65.9		65.9			551.628	
54.5		65.9	→	64.6		64.6			548.424	
53.9		64.6	→	62.3		62.3			545.278	
53.3		62.3	→	NA		NA			542.138	

Table 3-3. Sodium Nitrate Solubility – Run 3

12/30/2014					**$NaNO_3$ Solubility - Run 3**			
					150.13 grams $NaNO_3$ + 100.05 grams H_2O			
Weight % $NaNO_3$		*Temperature Range (°C +/-1 °C)*			*Dissolution Temperature (°C +/- 1°C)*		*Balance Reading*	
		Start	→	*End*			*(grams)*	
60		87	→	87	87		541.833	
59.3		87	→	83.3	83.3		538.683	
58.5		83.3	→	80.2	80.2		535.533	
57.8		80.2	→	77.3	77.3		532.353	
57.1		77.3	→	74.3	74.3		529.175	
56.4		74.3	→	70.9	70.9		526.011	
55.8		70.9	→	68.2	68.2		522.828	
55.1		68.2	→	65.4	65.4		519.637	
54.5		65.4	→	62.2	62.2		516.478	
53.9		62.2	→	59.4	59.4		513.333	
53.3		59.4	→	56.3	56.3		510.172	
52.7		56.3	→	53.4	53.4		506.998	
52.1		53.4	→	50.9	50.9		503.89	
51.5		50.9	→	47.7	47.7		500.708	
51		47.7	→	45.2	45.2		497.512	
50.4		45.2	→	42.8	42.8		494.434	
49.9		42.8	→	40.4	40.4		491.236	
49.4		40.4	→	37.8	37.8		488.149	
48.9		37.8	→	35.4	35.4		485.029	
48.4		35.4	→	33.3	33.3		481.835	
47.9		33.3	→	31.5	31.5		478.76	
47.4		31.5	→	29.3	29.3		475.549	
47		29.3	→	26.8	26.8		472.468	
46.5		26.8	→	24.8	24.8		469.332	
46.1		24.8	→	22.8	22.8		466.221	
45.6		22.8	→	20.9	20.9		463.136	
45.2		20.9	→	19	19		459.957	
44.8		19	→	17.4	17.4		456.784	
44.4		17.4	→	15.7	15.7		453.707	
44		15.7	→	13.9	13.9		450.594	
43.6		13.9	→	12.2	12.2		447.48	
43.2		12.2	→	10.4	10.4		444.295	
42.8		10.4	→	7.9	7.9		441.144	

Table 3-4. Sodium Nitrate Solubility – Run 4

1/7/2015				**$NaNO_3$ Solubility - Run 4**	
				150.01 grams $NaNO_3$ + 100.02 grams H_2O	
Weight % $NaNO_3$	*Temperature Range (°C +/-1 °C)*			*Dissolution Temperature (°C +/- 1 °C)*	*Balance Reading*
	Start	→	*End*		*(grams)*
60	75.5	→	75.5	75.5	568.25
59.5	75.5	→	77	77	566.127
59	77	→	79.4	79.4	564.031
58.5	79.4	→	79.4	79.4	561.929
58	79.4	→	79.4	79.4	559.852
57.6	79.4	→	76.4	76.4	557.746
57.1	76.4	→	74.5	74.5	555.627
56.7	74.5	→	72.3	72.3	553.557
56.2	72.3	→	70.3	70.3	551.467
55.8	70.3	→	68.3	68.3	549.279
55.3	68.3	→	66.5	66.5	547.194
54.9	66.5	→	64.2	64.2	545.017
54.5	64.2	→	62.4	62.4	542.879
54.1	62.4	→	59.6	59.6	540.785
53.7	59.6	→	57.6	57.6	538.707
53.3	57.6	→	55.9	55.9	536.598
52.9	55.9	→	53.8	53.8	534.546
52.5	53.8	→	51.8	51.8	532.471
52.1	51.8	→	49.6	49.6	530.413
51.7	49.6	→	48.2	48.2	528.309
51.4	48.2	→	46.5	46.5	526.235
51	46.5	→	44.8	44.8	524.147
50.6	44.8	→	43.1	43.1	522.054
50.3	43.1	→	41.5	41.5	519.965
49.9	41.5	→	39.9	39.9	517.845
49.6	39.9	→	38.3	38.3	515.76
49.2	38.3	→	36.7	36.7	513.657
48.9	36.7	→	35.2	35.2	511.547
48.6	35.2	→	33.7	33.7	509.455
48.3	33.7	→	32.4	32.4	507.381
47.9	32.4	→	31	31	505.299
47.6	31	→	30.1	30.1	503.21
47.3	30.1	→	28.6	28.6	501.101
47	28.6	→	27.2	27.2	498.975
46.7	27.2	→	25.9	25.9	496.892
46.4	25.9	→	24.6	24.6	494.789
46.1	24.6	→	23.2	23.2	492.627
45.8	23.2	→	21.9	21.9	490.477

Table 3-5. Sodium Nitrate Solubility – Run 5

1/21/2015				**$NaNO_3$ Solubility - Run 5**	
				150.00 grams $NaNO_3$ + 100.01 grams H_2O	
Weight % $NaNO_3$	*Temperature Range (°C +/-1 °C)*			*Dissolution Temperature (°C +/- 1 °C)*	*Balance Reading*
	Start	→	*End*		*(grams)*
60	77.8	→	77.8	77.8	702.193
59.5	77.8	→	82.8	82.8	700.124
59	82.8	→	81.2	81.2	698.066
58.5	81.2	→	81.4	81.4	696.007
58.1	81.4	→	80.7	80.7	693.956
57.6	80.7	→	78.6	78.6	691.906
57.2	78.6	→	76.3	76.3	689.845
56.7	76.3	→	74.2	74.2	687.794
56.3	74.2	→	71.8	71.8	685.731
55.8	71.8	→	69.3	69.3	683.655
55.4	69.3	→	67.2	67.2	681.608
55	67.2	→	65.3	65.3	679.545
54.6	65.3	→	63.3	63.3	677.484
54.2	63.3	→	61.2	61.2	675.426
53.8	61.2	→	59	59	673.366
53.4	59	→	56.8	56.8	671.294
53	56.8	→	55	55	669.226
52.6	55	→	53.1	53.1	667.16
52.2	53.1	→	51.1	51.1	665.09
51.9	51.1	→	48.9	48.9	663.029
51.5	48.9	→	47	47	660.975
51.1	47	→	45.3	45.3	658.908
50.8	45.3	→	43.6	43.6	656.852
50.4	43.6	→	42	42	654.781
50.1	42	→	40.3	40.3	652.726
49.7	40.3	→	38.6	38.6	650.664
49.4	38.6	→	37	37	648.616
49.1	37	→	35.5	35.5	646.548
48.7	35.5	→	34	34	644.489
48.4	34	→	32.6	32.6	642.431
48.1	32.6	→	31.1	31.1	640.366
47.8	31.1	→	29.9	29.9	638.315
47.5	29.9	→	28.3	28.3	636.256
47.2	28.3	→	27	27	634.193
46.9	27	→	25.7	25.7	632.131
46.6	25.7	→	24.4	24.4	630.071
46.3	24.4	→	23	23	628.003
46	23	→	21.8	21.8	625.96
45.7	21.8	→	20.7	20.7	623.905
45.4	20.7	→	18.8	18.8	621.855
45.1	18.8	→	NA	NA	619.786

Table 3-6. Solubility Data (wt% $NaNO_3$) for Sodium Nitrate

Temperature (^oC)	Weight% $NaNO_3$								
	Reference Mulder 1864	**Reference Berkeley 1904**	**Reference Chretien 1929**	**Reference Shpunt 1941**	**Run 1 12/22/14**	**Run 2 12/29/14**	**Run 3 12/30/14**	**Run 4 01/07/15**	**Run 5 01/21/15**
10	44.7	44.6	-	43.9	-	-	43.2	-	-
15	-	-	-	-	-	-	*44.2*	-	-
20	46.7	46.8	-	46.0	-	-	*45.4*	-	*45.6*
25	47.6	47.9	47.9	47.9	-	-	46.5	46.4	*46.8*
30	48.7	49.0	-	48.0	47.8	-	*47.6*	47.6	47.8
40	50.5	51.2	-	-	*50.0*	-	49.9	49.9	50.1
50	52.8	53.3	53.3	-	52.0	-	*51.9*	52.1	*52.0*
60	54.9	55.4	-	-	*54.2*	-	*54.1*	54.1	*54.0*
70	-	-	-	-	56.6	*56.2*	*56.2*	56.2	*56.0*
75	-	-	58.6	-	*57.8*	*57.4*	*57.4*	*57.3*	*56.9*
80	59.7	59.7	-	-	59.0	58.5	58.5	-	*57.9*

Note: All italic numbers in the table above present in Runs 1-5 are estimated values based on results in Tables 3-1 through 3-5.
Note: Reference values are taken from Solubilities of Inorganic and Metal Organic Compounds (Linke and Seidell 1965).

Table 3-7. Sodium Chloride Solubility – Run 1

1/22/2015	**NaCl Solubility - Run 1**		45 grams NaCl + 105 grams H_2O	
Temperature (°C)	*Balance Reading (* grams*)*		*Amount of Water Added (mL)*	*NaCL added (grams)*
	Start (Turbid)	*End (Clear)*		
80	618.782	597.545	21.237	6.37 (for 70° run)
70	597.545	576.81	20.735	6.22 (for 60° run)
60	576.81	556.601	20.209	6.06 (for 55° run)
55	556.601	540.265	16.336	4.9 (for 50° run)
50	540.265	524.747	15.518	4.66 (for 45° run)
45	524.747	508.882	15.865	4.76 (for 40° run)
40	508.882	493.887	14.995	4.5 (for 35° run)
35	493.887	480.96	12.927	3.88 (for 30° run)
30	480.96	468.869	12.091	3.63 (for 25° run)
25	468.869	455.815	13.054	3.92 (for 20° run)
20	455.815	444.115	11.7	NA

Table 3-8. Sodium Chloride Solubility – Run 2

1/26/2015		**NaCl Solubility - Run 2**	45 grams NaCl + 105 grams H_2O	
Temperature (°C)	*Balance Reading (grams)*		*Amount of Water Added (mL)*	*NaCL added (grams)*
	Start (Turbid)	*End (Clear)*		
80	790.662	766.463	24.199	7.26 (for 70° run)
70	766.463	744.868	21.595	6.48 (for 60° run)
60	744.868	725.459	19.409	5.82 (for 55° run)
55	725.459	705.993	19.466	5.84 (for 50° run)
50	705.993	689.052	16.941	5.08 (for 45° run)
45	689.052	673.088	15.964	4.79 (for 40° run)
40	673.088	658.183	14.905	4.47 (for 35° run)
35	658.183	644.684	13.499	4.05 (for 30° run)
30	644.684	630.205	14.479	4.34 (for 25° run)
25	630.205	616.95	13.255	3.98 (for 20° run)
20	616.95	601.606	15.344	NA

Table 3-9. Sodium Chloride Solubility – Run 3

1/28/2015		**NaCl Solubility - Run 3**	45 grams NaCl + 105 grams H_2O	
Temperature (°C)	*Balance Reading (grams)*		*Amount of Water Added (mL)*	*NaCL added (grams)*
	Start (Turbid)	*End (Clear)*		
80	788.006	764.175	23.831	7.15 (for 70° run)
70	764.175	740.127	24.048	7.21 (for 60° run)
60	740.127	719.03	21.097	6.33 (for 55° run)
55	719.03	699.017	20.013	6.00 (for 50° run)
50	699.017	681.628	17.389	5.22 (for 45° run)
45	681.628	664.761	16.867	5.06 (for 40° run)
40	664.761	649.733	15.028	4.51 (for 35° run)
35	649.733	635.23	14.503	4.35 (for 30° run)
30	635.23	623.257	11.973	3.59 (for 25° run)
25	623.257	611.722	11.535	3.46 (for 20° run)
20	611.722	599.367	12.355	NA

Table 3-10. Solubility Data (wt% NaCl) for Sodium Chloride

Temperature (^{0}C)		Weight % NaCl		
	Reference	Run 1 1/22/15	Run 2 1/26/15	Run 3 1/28/15
80	27.5	26.3	25.8	25.9
70	27.3	25.9	25.7	25.4
60	27	25.6	25.7	25.4
55	--	25.8	25.4	25.3
50	26.8	25.6	25.4	25.3
45	--	25.4	25.3	25.2
40	26.6	25.3	25.3	25.2
35	--	25.4	25.2	25.1
30	26.5	25.3	25.1	25.2
25	26.4	25.1	25	25.1
20	26.4	25.1	24.8	25
15	26.3	--	--	--
10	26.3	--	--	--
0	26.3	--	--	--

Note: Reference values are taken from Linke and Seidell 1965.

4 FIGURES

Figure 4-1. Sodium Nitrate Solubility

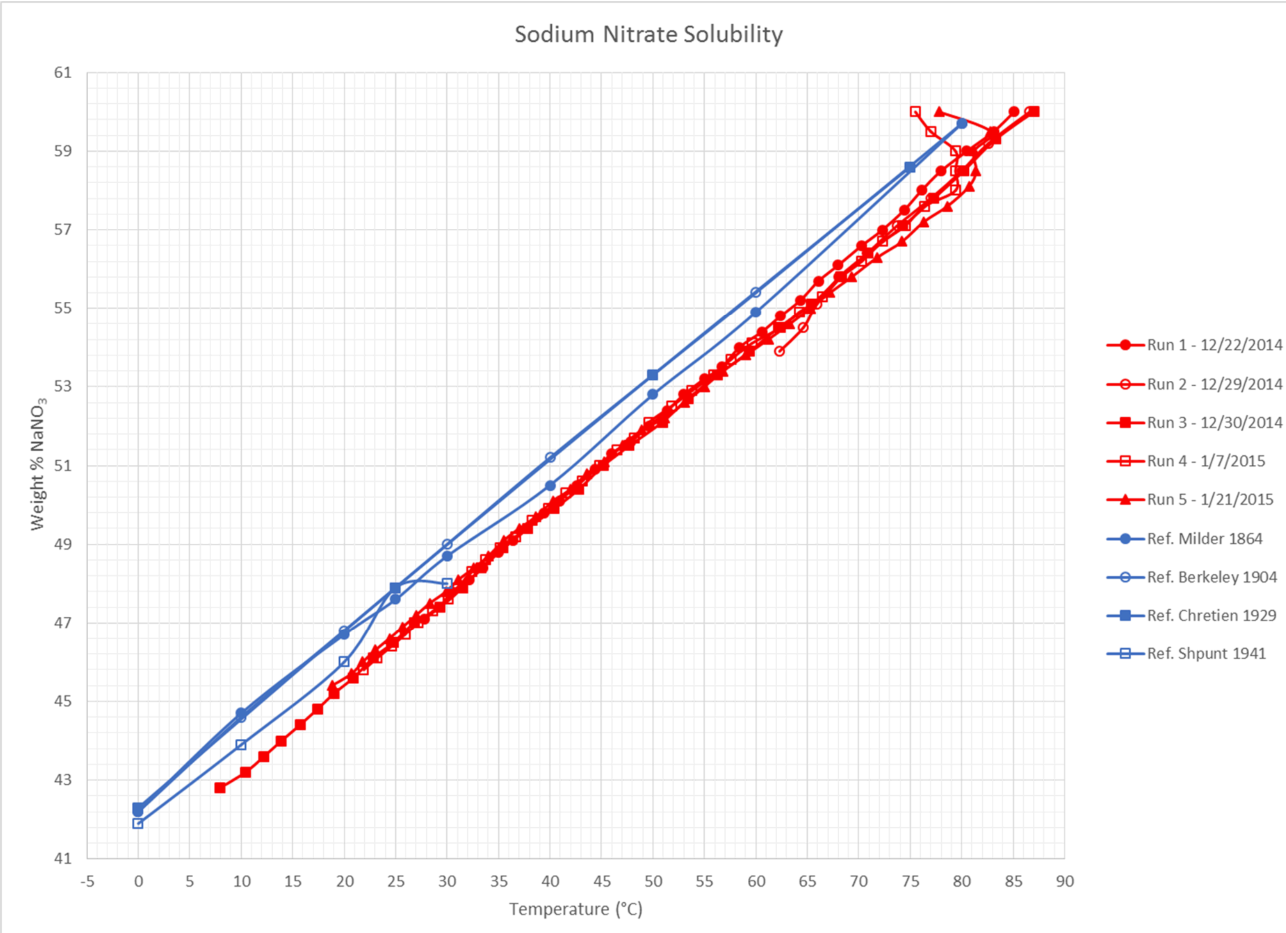
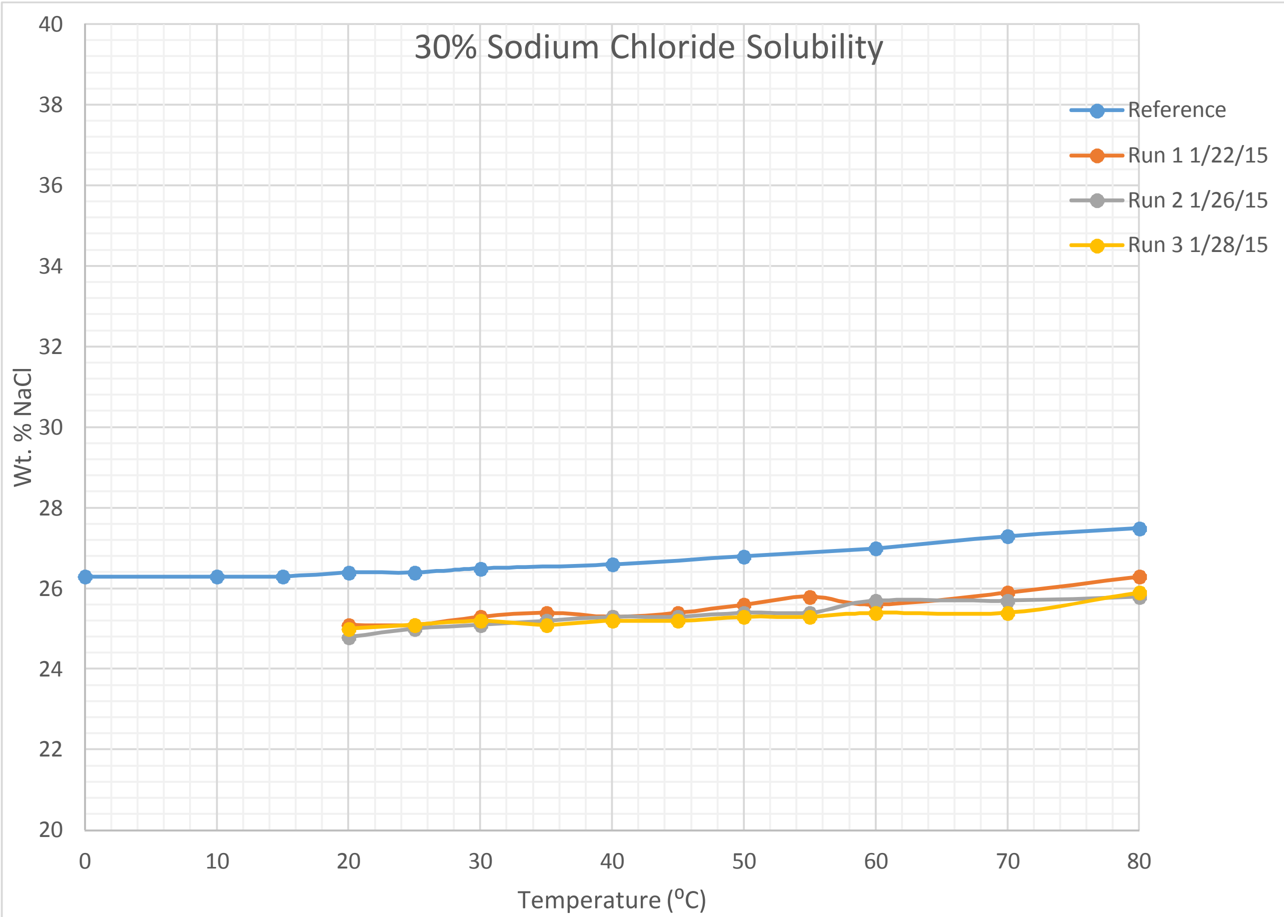


Figure 4-2. 30% Sodium Chloride Solubility



5 CONCLUSIONS

The conclusion based on the laboratory findings thus far is that the turbidity meter works well with compounds that have finer particle size and disperse well in water. The sensor has problems with denser, coarser, and more granular compounds because it cannot capture the entire picture of what is occurring; it only senses what is going on directly in front of the detector, which is located at the bottom of the probe. Perhaps using a turbidity meter that could detect from the bottom or the sides of the reactor would alleviate this problem, but that would assume the detector would be able to measure turbidity through glass. Other possibilities are to improve mixing by mechanical agitation, changing the shape of the reactor, and improving the baffling design. Another possibility would be to measure particle size in-situ instead of turbidity. Particle size would be large initially and then decrease as the salt dissolves.

Error analysis was not done and propagated errors were not calculated. Water addition to the sodium nitrate solution was done by computer as it was driven by the turbidity meter readings. Water was added by means of a peristaltic pump. The water is stored in an Erlenmeyer flask that is placed on a three-place balance. As water is pumped into the reactor, it gets pumped out of the Erlenmeyer flask and the weight goes down.

Sodium chloride addition was made after turbid solutions went clear, and the process was repeated in order to maintain the 30 wt% salt solution. The amount of salt to be manually added was calculated by multiplying the water weight difference between turbid and clear readings by 0.3 and rounding the answer to 0.01 g. The error associated with the salt additions is approximately 0.05 wt%.

The sodium nitrate solution results were within 2-3% of the reference values, so the experiment is considered successful using the turbidity meter.

The sodium chloride solution results, as measured by sight rather than by turbidity instrument readings, were within 5-6% of the reference values.

The scope of the test plan was to determine if the turbidity meter would work in a sodium nitrate matrix and a sodium chloride matrix using reference data as a baseline. The acceptance criteria is +/- 10%, and that has been met. Therefore, further testing involving solubility kinetics and data collection will need to be captured in another test plan with other target conclusions. This may include the purchase of commercial equipment as the need for solubility testing becomes more valued and involved.

6 REFERENCES

Linke, W. and A. Seidell, 1965, Solubilities of Inorganic and Metal Organic Compounds, 4th Edition, Volume 2, American Chemical Society, Washington DC.

LabJack Measurement & Automation, Queried 11/2/2015, EI-1034 Datasheet, http://labjack.com/support/ei-1034/datasheet.

TSS HT sc TriClamp, Suspended Solids TriClamp inline Sensor, Queried 11/2/2015, [TSS sc Suspended Solids Family of Sensors Data Sheet], http://www.hach.com/tss-ht-sc-triclamp-suspended-solids-triclamp-inline-sensor/product-downloads?id=7640284926&callback=pf.

WRPS–1404441, 2014, "Validation Testing for Automated Solubility Measurement Equipment," (interoffice memorandum from J. S. Lachut to K. L. Pierson, November 25), Washington River Protection Solutions LLC, Richland, Washington.

ATTACHMENT

EMAIL FROM J. MCCOSKEY, 4-06-201515
"DRAFT OF SOLUBILITY TEST FINAL REPORT"

From: Mc coskey, Jacob K
Sent: Monday, April 06, 2015 8:49 AM
To: Lamothe, Margaret E; Lachut, James S
Subject: RE: Draft of Solubility Test Final Report

All,

The NT and DT are determined using an algorithm that requires some user set value for change in turbidity to overcome the inherent noise of the meter. For example, when looking for the NT, the software notes when the turbidity increases and stores the time and temperature of the turbidity increase. If the turbidity continues to the user defined increase in turbidity, say 10-30 FNU, then the algorithm retrieves the NT when the turbidity first began to increase.

For this system, I don't think that the temperature should be reported to the hundreds place. A EI-1034 chip is used to measure the temperature and its reported accuracy is +- 0.4F (http://labjack.com/support/ei-1034/datasheet). Also the turbidity meter has a reported accuracy of +- 0.1 FNU, which would have some effect on the NT and DT temperature (The turbidity slope must be quantified to determine the effect on temperature) (http://www.hach.com/tss-ht-sc-triclamp-suspended-solids-triclamp-inline-sensor/product-downloads?id=7640284926&callback=pf). I do recall that the turbidity meter had a lot of noise from the 4-20mA I/O and that error should be calculated into the reported temperatures. The 4-20 I/O was to be replaced with a digital I/O, which would have eliminated this noise. If it was changed to digital I/O, then the algorithm in place should be altered to make the system more accurate. All things being said I would report a range for each temperature with the number reported to the tenths of a degree at most (I would report it to the nearest whole number to be conservative knowing the system). For the case below, I would report 40. 3 C +- 1C. This would cover most known analytical error and likely the noise. The data should be interrogated to quantify the error from noise.

Jake

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