

**Parker, Bill**

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**From:** Kellenberger, Bill  
**Sent:** Tuesday, October 28, 2003 9:22 AM  
**To:** Parker, Bill  
**Subject:** FW: NEED HELP IN ANALYZING.HG

Bill See below. Your input is needed tooooooo. Do we or you do the variance...Hit thru the ball.

-----Original Message-----

**From:** Kellenberger, Bill  
**Sent:** Tuesday, October 28, 2003 8:19 AM  
**To:** Price, John L.; Tenace, Laurie  
**Subject:** NEED HELP IN ANALYZING.HG

Hi All, See the attached letter and data from Jeff. We feel that they have a problem with all the negative results, etc. It also appears that an extension of the variance is asked for. I am not sure how we do this. What do you think on both issues. Let's have a conference call to discuss in house. Thanks. Hit thru the ball.

-----Original Message-----

**From:** Jeff A Kirk [mailto:JAKirk@onyxsp.com]  
**Sent:** Monday, October 27, 2003 3:37 PM  
**To:** Kellenberger, Bill  
**Subject:** Letter to Bill Kellenberger

Bill,  
Here is the quarterly report.

Jeff Kirk  
Onyx Special Services  
342 Marpan Lane  
Tallahassee, FL 32305  
866-877-8299  
Fax 850-878-3349

October 28, 2003

Mr. Bill Kellenberger  
Department of Environmental Protection  
Northwest District Office  
160 Governmental Center  
Pensacola, FL 32501-5794

RE: Onyx Special Services, Inc.  
342 Marpan Lane  
Tallahassee, FL 32305  
EPA ID# FL0000207449  
OGC File No. 01-1298

Dear Mr. Kellenberger:

This letter is being submitted to report the status of research and development activities under the variance issued by the Florida Department of Environmental Protection under the above referenced file number on January 4, 2002. This letter is also being submitted to request an extension of the variance for the purpose of conducting additional research and development activities.

Below is a summary of the results obtained from the additional time at maximum temperature tests. Attached is a detailed listing of every batch that was processed in the retort along with the associated analytical data, percent recoveries, and any maintenance activities contained in the maintenance log.

Trial 1, 2 additional hours at max. temp.	1 of 6	>99% recovery or <10 mg/kg total mercury
	4 of 6	>0 and <99% recovery and >10 mg/kg total mercury
	1 of 6	Negative percent recovery
Trial 1, 4 additional hours at max. temp.	3 of 6	>99% recovery or <10 mg/kg total mercury
	3 of 6	>0 and <99% recovery and >10 mg/kg total mercury
Trial 1, 6 additional hours at max. temp.	1 of 6	>99% recovery or <10 mg/kg total mercury
	5 of 6	>0 and <99% recovery and >10 mg/kg total mercury
Trail 2, 2 additional hours at max. temp	3 of 6	>99% recovery or <10 mg/kg total mercury
	1 of 6	>0 and <99% recovery and >10 mg/kg total mercury
	2 of 6	Negative percent recovery
Trial 2, 4 additional hours at max. temp	0 of 6	>99% recovery or <10 mg/kg total mercury
	2 of 6	>0 and <99% recovery and >10 mg/kg total mercury
	4 of 6	Negative percent recovery
Trial 2, 6 additional hours at max. temp	1 of 6	>99% recovery or <10 mg/kg total mercury
	1 of 6	>0 and <99% recovery and >10 mg/kg total mercury
	4 of 6	Negative percent recovery

The data would seem to suggest that the efficiency of the retort decreases with the additional time at maximum temperature; however, this conclusion is not consistent with the theory and logic of the trial. After receiving the results, a number of items were investigated and/or evaluated.

Area Investigated	Results
Retort temperature	The retort is equipped with an indicator light that is activated when the retort reaches the maximum temperature contained in the program controller. This light was activated on every batch indicating that the retort reached maximum temperature.
Retort Vacuum	All vacuum reading were found to be within the retorts normal operating range and there was no correlation between vacuum readings and percent recovery.
Laboratory Quality Control	Copies of quality control documents were obtained from the laboratory and all quality control measures were found to be within acceptable ranges.
Laboratory Sample Control	When phosphor powder is heated in the retort the powder undergoes a slight color change. All samples analyzed by the laboratory were shipped back to the facility and the samples were visually inspected. This inspection did not find any evidence of samples being mislabeled and the color changes were consistent between all samples.
Processed powder on regular program	Between the two trials several batches were processed using the regular program in the retort. All of the samples from this material met the requirement for a >99% recovery or <10 mg/kg final concentration.

Based on the laboratory data and the results of the investigation that we have conducted we can make the following conclusions.

1. When operated on the regular processing program the retort continues to meet the requirements of the variance.
2. The additional cook time results are not valid.
3. We cannot at this time identify the reason for the invalid results.

One theory as to why the results are not valid is when the retort is reprogrammed for the additional time at maximum temperature, some of the other programming is lost in the system. This could lead to the oven heating up too quickly and reducing the overall time the material spends in the oven. To conclusively answer this question we are proposing to purchase and install a data logging thermocouple on the retort. This device will monitor the temperature of the retort oven and log that temperature at a set interval. This will allow us to chart the length of time the retort is requiring to reach maximum temperature and the length of time at that temperature.

Mr. Bill Kellenberger  
October 28, 2003  
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We anticipate that it will take several weeks to obtain the new thermocouple and to have an adapter manufactured to allow the installation of the thermocouple. Once we have the thermocouple installed it will take several additional weeks to run the trial and obtain the analytical results.

The trial study to determine if processing the material for a longer period of time at the maximum temperature has taken longer than originally anticipated and the completing this trial for the third time with the data logging thermocouple will also take several additional months to complete. The time spent on this project has precluded us from attempting the two additional methods for increasing retort efficiency required by the variance. Based on these factors, Onyx Special Services, Inc. is requesting an extension of the variance issued January 4, 2002 under the above referenced file number.

If you have any questions please call Phillip Ditter at (262) 243-8908 or call me at (850) 878-2259.

Sincerely,

ONYX SPECIAL SERVICES, INC.

Jeff Kirk  
General Manager

Cc: Phillip Ditter

Date	Run Number	Vac. Reading	Max. Temp	Program	Conc. Before	Conc. After	% Recovery	Maintenance Activities
01/02/03	758	4	Yes	Regular				
01/03/03	757	4	Yes	Regular				Replaced bearing in vac pump
01/04/03	758	2	Yes	Regular				
01/05/03	759	2	Yes	Regular				
01/06/03	760	2	Yes	Regular				
01/07/03	761	4	Yes	Regular				
01/08/03	762	2	Yes	Regular				
01/09/03	763	2	Yes	Regular				
01/10/03	764	4	Yes	Regular				
01/11/03	765	4	Yes	Regular				
01/13/03	766	2	Yes	Regular				
01/14/03	767	2	Yes	Regular				Replaced filters, cleaned out chiller
01/15/03	768	4	Yes	Regular				
01/16/03	769	2	Yes	Regular				
01/20/03	769b	2	Yes	Regular				
01/21/03	770	4	Yes	Regular	813.00	0.74	99.91%	January recovery rate sample
01/22/03	770b	2	Yes	+2 hours	1114.00	930.00	16.52%	
01/23/03	771	2	Yes	+2 hours				
01/24/03	772	2	Yes	+2 hours	1920.00	760.00	60.42%	
01/25/03	773	2	Yes	+2 hours	985.00	744.00	24.47%	
01/28/03	774	2	Yes	+2 hours	286.00	2.78	99.03%	
01/30/03	775	2	Yes	+2 hours	311.00	127.00	59.16%	
01/31/03	776	2	Yes	+2 hours	97.60	102.00	-4.51%	
02/03/03	777	2	Yes	+4 hours	923.00	2.36	99.74%	
02/05/03								Replaced 2 heating elements
02/10/03								Replaced carbon in lead filter, swapped filter locations
02/21/03	778	2	Yes	+4 hours	1370.00	2.58	99.81%	
02/28/03								Cleaned pipes
03/08/03								Replaced filters
03/19/03	779	2	Yes	+4 hours				
03/20/03	780	2	Yes	+4 hours	584.00	3.63	99.38%	
03/21/03	781	2	Yes	+4 hours	541.00	27.20	94.97%	
03/24/03	782	2	Yes	+4 hours	480.00	43.00	91.04%	
03/25/03	783	2	Yes	+4 hours	575.00	28.00	95.13%	
03/26/03	784	2	Yes	+6 hours	330.00	17.00	94.85%	
03/27/03	785	2	Yes	+6 hours	1220.00	57.00	95.33%	
03/28/03	786	2	Yes	+6 hours	1030.00	93.00	90.97%	
03/30/03	787	2	Yes	+6 hours	295.00	33.00	88.81%	
04/01/03	789	2	Yes	+6 hours	810.00	21.00	97.41%	
04/02/03								Cleaned chillers
04/07/03	790	2	Yes	+6 hours	597.00	1.90	99.68%	
04/08/03	791	2	Yes	Regular				
04/09/03	792	2	Yes	Regular				
04/11/03	793	2	Yes	Regular				
04/14/03	794	2	Yes	Regular				
04/15/03	795	2	Yes	Regular				Replaced door seal
04/16/03	796	2	Yes	Regular				
04/18/03	797	2	Yes	Regular				
04/21/03	798	2	Yes	Regular				
04/23/03	799	2	Yes	Regular				
04/24/03	800	2	Yes	Regular				
04/28/03	801	2	Yes	Regular				
04/29/03	802	2	Yes	Regular	515.00	3.72	99.28%	April recovery rate sample
05/05/03	803	2	Yes	Regular				
05/07/03								Replaced bearing in vac pump
05/08/03	804	2	Yes	Regular	389.00	4.90	98.74%	
05/09/03	805	2	Yes	Regular				
05/12/03	806	2	Yes	Regular	290.00	0.41	99.86%	
05/13/03	807	2	Yes	Regular				
05/15/03	808	2	Yes	Regular	512.00	7.10	98.61%	
05/22/03								Replaced filters, cleaned chillers and pipes
05/29/03	809	2	Yes	Regular				
06/02/03	810	1	Yes	+2 hours	214.00	0.42	99.80%	
06/04/03								cleaned screens in carbon filters
06/14/03	811	1	Yes	+2 hours	58.30	167.00	-186.45%	
06/15/03	812	1	Yes	+2 hours	197.00	190.00	3.55%	Date in run log lists 6/13/03
06/16/03	813	1	Yes	+2 hours	1.01	165.00	-16236.63%	
06/26/03	814	1	Yes	+2 hours	214.00	0.48	99.78%	Cleaned pipe above DME
07/02/03	815	1	Yes	+2 hours	247.00	7.01	97.16%	
07/08/03	816	1	Yes	+4 hours	109.00	136.00	-24.77%	
07/11/03								Replaced filters
07/15/03	817	1	Yes	+4 hours	184.00	255.00	-38.59%	
07/16/03	818			+4 hours	80.00	142.00	-77.50%	run number not in log
07/17/03	819	1	Yes	+4 hours	217.00	175.00	19.35%	
07/23/03	820	1	Yes	+4 hours	108.00	134.00	-26.42%	
07/28/03	821	1	Yes	+4 hours	108.00	90.40	16.30%	
07/29/03								Replaced heating element, cleaned out DME
08/01/03	822	1	Yes	+6 hours	33.00	127.00	-284.85%	
08/04/03								Replaced bad fuse
08/12/03	823	1	Yes	+6 hours	94.00	9.12	90.30%	
08/14/03	824	1	Yes	+6 hours	105.00	91.20	13.14%	

Date	Run Number	Vac. Reading	Max. Temp	Program	Conc. Before	Conc. After	% Recovery	Maintenance Activities
08/16/03	825	1	Yes	+6 hours	194.00	344.00	-77.32%	
08/18/03	826	1	Yes	+6 hours				
08/25/03								Cleaned chillers and pipes
08/27/03	827	1	Yes	+6 hours	148.00	212.00	-43.24%	
09/03/02								Replaced filters
09/04/03	828	1	Yes	+6 hours	208.00	214.00	-2.88%	
09/12/03	829	1	Yes	Regular				
09/18/03								Replaced bearing in vac pump, cleaned chillers
09/19/03	830	1	Yes	Regular				
09/25/03	831	1	Yes	Regular				