



CATALYST TEST REPORT

GAINESVILLE RENEWABLE ENERGY CENTER

**Presented to:
GREC / NAES**

**Prepared by:
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1.0 TEST RESULTS SUMMARY

Catalyst activity and characterization testing is an important part of an effective catalyst management program. A honeycomb catalyst test element manufactured by CERAM was removed from the Gainesville Renewable Energy Center (GREC) for catalyst testing. The objectives of the catalyst testing were to determine the current catalyst activity and assess the deactivation mechanisms. This report documents the catalyst test results and provides an analysis of the cause for catalyst deactivation.

One (1) catalyst test element was removed from the GREC SCR reactor in May 2017 and sent to CERAM. GREC has one initial catalyst layer and one empty spare layer (e.g., 1 + 1 reactor). The test element was removed from the catalyst layer from module L1-D4 (Reference Appendix A). The test element was received by CERAM at their shop in Olathe, Kansas, processed and then sent to the CERAM laboratories in Frauental, Austria for activity and chemical analysis. Table 1-1 summarizes the results of the activity testing. Activity testing was performed in our semi-bench test reactor and compared to the original activity (K_o) from the QA/QC testing that was performed during production.

Table 1-1. NAES GREC Activity Test Results				
Catalyst Element Location / Serial No.	Activity (K) (semi-bench at 230 C) (m/hr)	K/K_o	K/K_o at 16,000 hrs	Operating Hours from GREC⁽¹⁾
Module L1-D4 ⁽²⁾ #0351493-01747	27.2	0.965	0.966	17,156
Fresh (K _o) #0351493	28.2	1.0	1.0	0
Notes:				
1. Operating hours provided by Mr. Tommy Gardner from NAES.				
2. Previous test report noted sample removed from L1-D4; production documentation confirms that the sample removed in 2017 is from a separate module than the sample removed in 2015.				

It should be noted that test sample #0351493-01721 (tested in 2015) had been previously indicated as being removed from module L1-D4, similar to test sample #0351493-01747 as noted in Table 1-1. Review of the module production documentation confirms that sample #0351493-

01721 was installed into the module marked #42, and sample 0351493-01747 was installed into the module marked #38. CERAM recommends that NAES review the marked module numbers indicated on the stainless-steel tag located near the inside corner of the inlet side of the modules and document the as-installed locations for both modules #38 and #42 as being sampled to ensure that future test sampling does not inadvertently remove a replacement sample. Reference Appendix A for additional information.

Graphical results of the activity testing are depicted in Figure 1-1. Also shown on Figure 1-1 are the historical activity testing results and current catalyst deactivation trends compared to the design/expected deactivation curve. The relative activity (K/Ko) values compare the aged catalyst activity (K) to the original catalyst activity (Ko). The measured relative activity of the test sample was 0.96. Using CERAM's Manage CATLife[®] Model the test results were normalized to a K/Ko at 16,000 hour basis ($K/Ko|_{16K}$), which allows for a direct comparison to the design deactivation rate. This analysis calculated a $K/Ko|_{16K}$ of 0.97 from the most recent activity testing.

Consistent with the previous activity test results, the measured activity was within the error of the test accuracy of $\pm 1K$ of the original production results, suggesting that there has been little deactivation after 17,156 hours of flue gas exposure. The anticipated design deactivation rate corresponds to a relative activity of 0.75 at 16,000 operating hours ($K/Ko|_{16K} = 0.75$). The current results indicate that the catalyst tested is aging well within an acceptable range given the existing flue gas conditions.

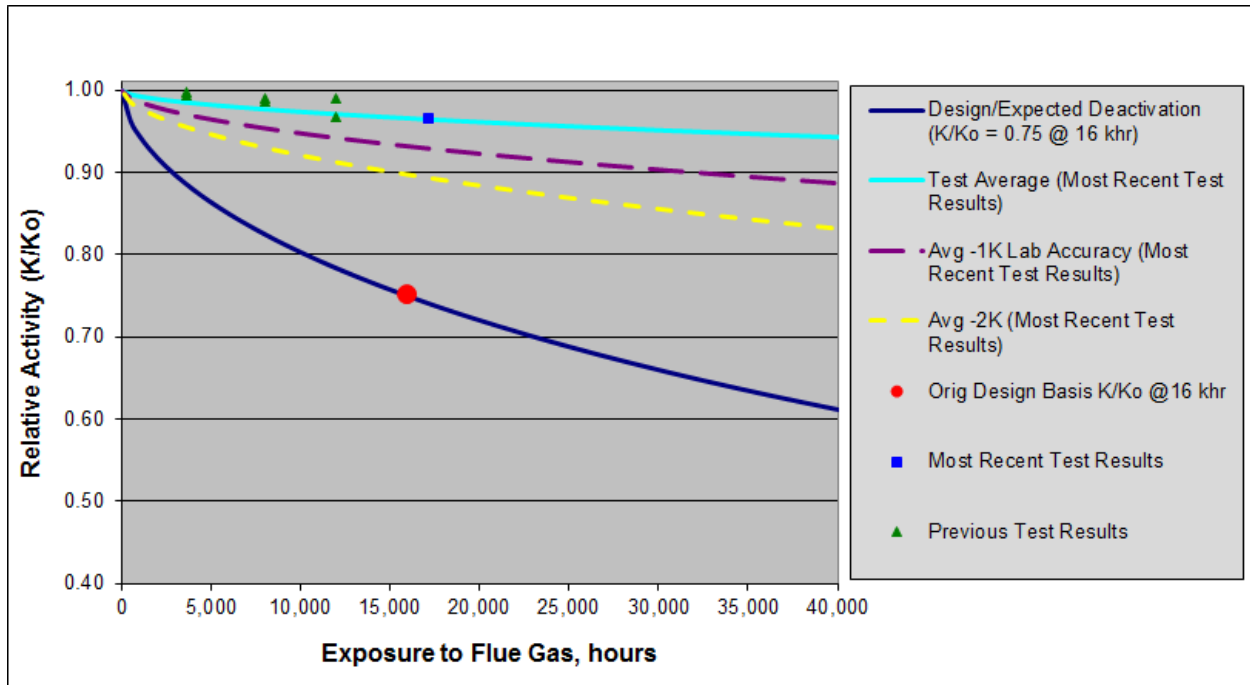


Figure 1-1. GREC catalyst activity trends.

The accumulation of certain chemical compounds on the catalyst in excessive amounts can prove to be detrimental to the catalyst performance and remaining catalyst life. Catalyst poisons will destroy active pore sites resulting in reduced catalyst activity. Catalyst deactivation can also occur due to fouling agents that block active catalyst sites thereby reducing catalyst activity. Table 1-2 summarizes the results of the bulk and surface chemical analysis.

Table 1-2. GREC Chemical Analysis		
Chemical Compound	Module L1-D4 #0351493-01747	
	Bulk (wt%)	Surface (wt%)
SiO ₂	7.96	7.49
Al ₂ O ₃	1.49	1.44
Fe ₂ O ₃	0.24	0.24
TiO ₂	78.69	79.29
CaO	2.11	1.66
MgO	0.24	0.22
BaO	0.09	ND
Na ₂ O	0.01	ND
K ₂ O	0.09	0.08
SO ₃	0.49	0.65
P ₂ O ₅	0.04	0.05
V ₂ O ₅	2.97	2.93
WO ₃	5.43	5.42
MoO ₃	0.03	ND
As ₂ O ₃	ND	ND
Note: ND = Non-detectable		

Analysis of the bulk and surface chemical test results correlate to the low deactivation rate that was measured in the test sample. Sulfur trioxide (SO₃) deposition was noted on the surface of the catalyst sample. This could be an indication of calcium sulfate (CaSO₄) masking resulting from normal operation. Typically, the accumulation of calcium sulfate is observed as an increase in both calcium oxide (CaO) and SO₃. In this instance, a corresponding accumulation in calcium oxide was not noted on the catalyst sample; however, minor accumulations of calcium sulfate could still be a cause of deactivation. Calcium sulfate products can mask active catalyst sites thereby reducing catalyst activity. SO₃ deposition can also result from operation below the minimum ammonia injection temperature which creates ammonium bisulphate (ABS) or from sustained exposure to flue gas below the acid dew point temperature.

Catalyst deactivation can also occur due to catalyst poisoning from high concentrations of phosphorus pentoxide (P₂O₅), potassium oxide (K₂O), sodium oxide (Na₂O), etc., however the measured quantities of these catalyst poisons were well below levels of concern. Based on the measured activities and chemical test results, there are no action items at this time regarding the catalyst and/or plant operations affecting catalyst life. Future catalyst testing after three to four years of operation is recommended to identify any trends that may occur in catalyst activity. If operating practices should change or there is a noted change in catalyst performance, testing is recommended prior to this time frame.

2.0 TESTING PERFORMED

Table 2-1 lists the tests that were performed to evaluate the activity and chemical properties of the catalyst. The catalyst test procedures are described in Appendix B.

Table 2-1. NAES GREC Testing Performed	
	Module L1-D4 #0351493-01747
Semi-Bench Activity	X
Bulk Chemical	X
Surface Chemical	X

Measuring the activity and testing the physical and chemical properties of the aged catalyst requires accurate and established test procedures. Accurate testing aids in predicting catalyst performance and diagnosing any performance related problems. At CERAM, catalyst tests are performed in an ISO 9001 certified laboratory and manufacturing facility located in Frauental, Austria. It should be noted that catalyst testing is performed on a very small statistical sampling of the entire reactor. Variations in the catalyst activity, physical properties and chemical properties may be expected within the reactor. This variability may be attributed to several factors, including: fuels fired, flue gas distribution and unit operating practice. The test results should be considered representative, but does not necessarily depict the overall actual activity or chemical properties of the catalyst as a whole. Additional catalyst sample testing would improve the statistical representation.

APPENDIX A – GREC TEST ELEMENT REMOVAL DIAGRAM

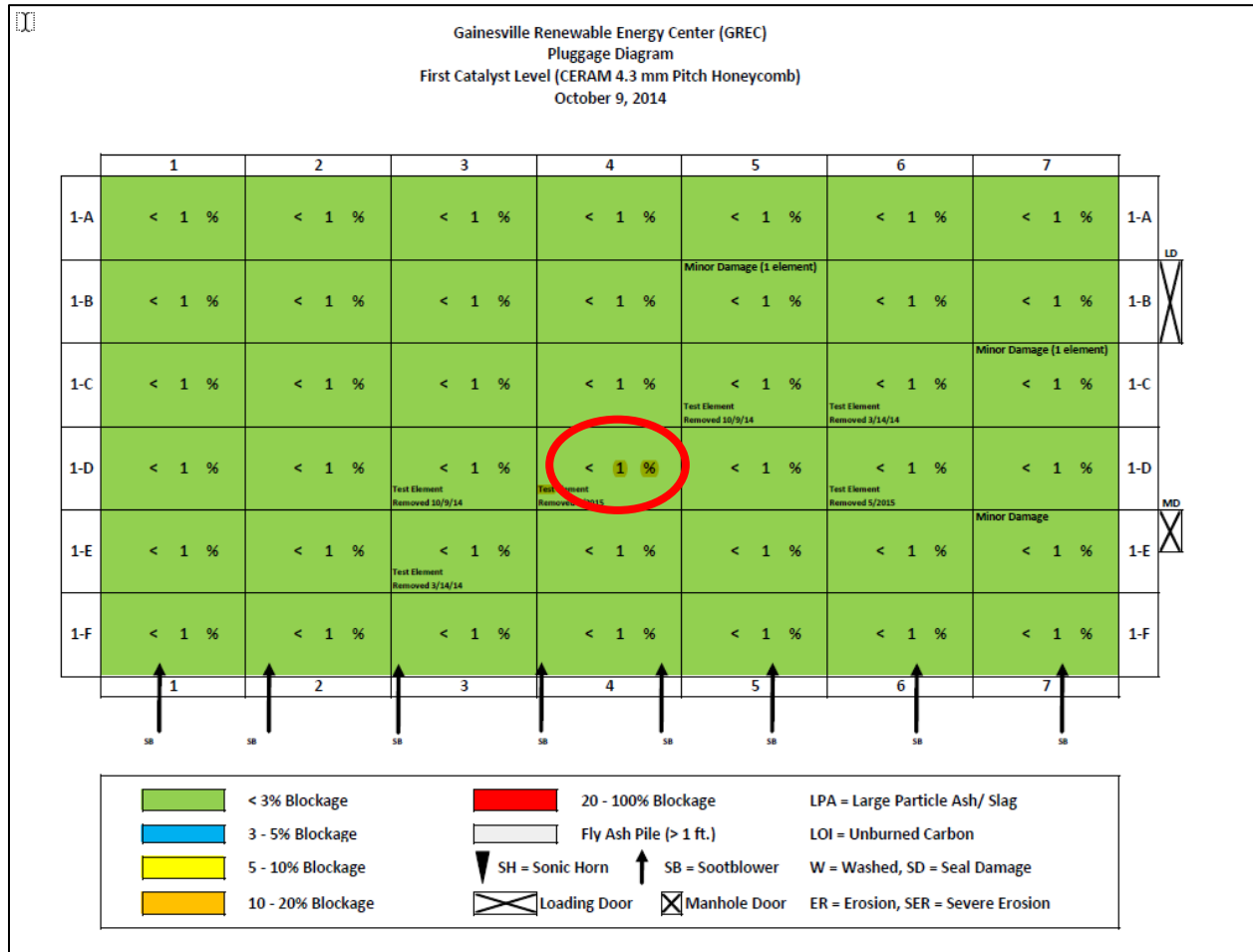
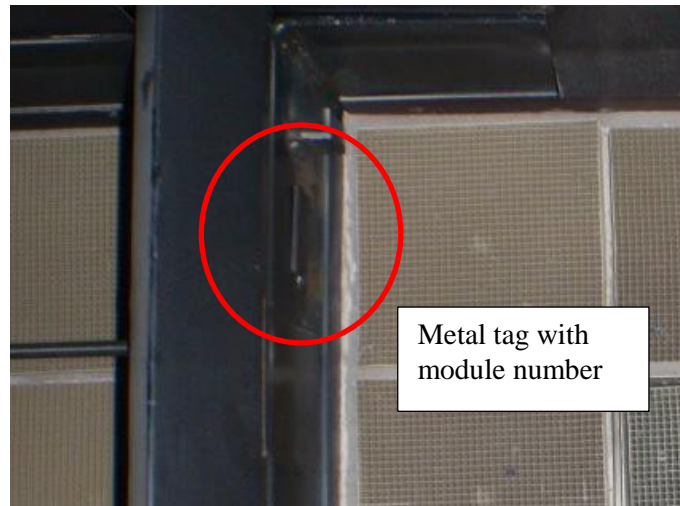


Figure A-1. GREC module grid with test element removal locations from May 2017 (note pluggage results from October 9, 2014).

Please note that the samples removed in 2017 and 2015 refer to the same test module (L1-D4). Review of module production documents confirms that these sample elements originated from separate modules. It is requested that GREC confirm the module numbers adjacent to the module sample locations. Figure A-2 provides an example of the module tag location.



(a) Location of metal tag with module number.



(b) Example of module tag number (note, not from GREC).

Figure A-2. Example of module tag location.

APPENDIX B – CATALYST TEST PROCEDURES

A brief description of the catalyst testing methods and procedures is outlined herein to explain the different testing performed on the catalyst samples that were used to evaluate the catalyst activity and overall catalyst performance.

B.1 Activity Test

The catalyst activity tests were performed in a semi-bench scale reactor which is used for all our quality control / quality assurance measurements during production. The semi-bench reactor tests a section of catalyst that is cut to an appropriate size and is exposed to flue gas consisting of nitric oxide (NO), sulfur dioxide (SO₂), nitrogen (N₂), water vapor (H₂O) and oxygen (O₂). The temperature is maintained at 230 C (446 F) and the area velocity (AV) is maintained at 25 Nm/h. Ammonia is then added to the flue gas and after equilibrium is achieved the NO inlet and outlet concentrations are measured in parts per million (ppm). The catalyst activity tests were performed at the test conditions shown in Table A-1. The NO_x removal efficiency is calculated, using equation [1]:

$$\eta = (\text{NO}_{\text{inlet}} - \text{NO}_{\text{outlet}}) / \text{NO}_{\text{inlet}} \quad [1]$$

From the NO_x removal efficiency, the activity (K) is calculated as shown in equation [2], where AV is still defined as the area velocity expressed in units of Nm/h.

$$K = -AV * \ln(1 - \eta) \quad [2]$$

Table B-1. Semi-Bench Scale Reactor Test Conditions		
Parameter	Unit	Test Value
Temperature	F	446 (230 C)
Area Velocity	Nm ³ /m ² h	25
H ₂ O	Vol %	10
NO	ppmvd	200
Molar Ratio	NH ₃ /NO _x	1.2
SO ₂	ppmvd	0
O ₂	Vol %, d	2
N ₂	Vol %, d	Balance

B.2 Bulk and Surface Chemical Analysis

The chemical composition of the catalyst sample was tested using X-Ray Fluorescence (XRF). This test identifies potential catalyst poisons and determines the presence of undesirable constituents in the flue gas stream that may lead to catalyst deactivation. This test also identifies the major catalyst constituents as weight distribution percentages.