



Missouri Department of dnr.mo.gov

# NATURAL RESOURCES

Eric R. Greitens, Governor

Carol S. Comer, Director

**MAR 13 2018**

Mr. Terry Miner  
Manager - EHS  
Zoltek  
27 Guenther Boulevard  
St. Peters, MO 63376

RE: New Source Review Permit Amendment - Permit Number: 112015-003A  
Project Number: 2017-07-056; Installation Number: 183-0261

Dear Mr. Miner:

On 7/24/2017, the Department of Natural Resources received Zoltek's request to amend Construction Permit No. 112015-003 in order for the facility to use a new epoxy resin on its eighteen (18) pultrusion lines. A second application was received on 9/14/2017 in order to further amend 112015-003 in response to the Missouri Department of Natural Resources and Zoltek's Administrative Order on Consent (AOC) executed on 8/15/2017 (APCP-2017-013) and to reflect the recent addition of a back-up power generator.

### Permit Corrections/Clarifications

The primary concern of the AOC was to address how the fluidized bed concentrator and thermal oxidizer should operate during malfunctions. This has been addressed by amending Special Condition 1.B of the original permit. Specifically, if the thermal oxidizer malfunctions, the facility can operate the production lines up to 70 minutes provided the fluidized bed concentrator operates according to manufacturer's specifications during the 70-minute period. Operation beyond the 70-minute period will trigger compliance with the rules for start-up, shutdown, and malfunction conditions found at 10 CSR 10-6.050. All recordkeeping and monitoring of these malfunctions shall be documented.

Zoltek's pultrusion processes were originally permitted with the intent that New Source Performance Standards (NSPS) Subpart VVV applied to the facility. However, according to the document entitled, "USEPA, NSPS Subpart VVV Applicability to Pultrusion Facilities, 68 Fed. Reg. 40,655, 40,662" (July 8, 2003), Zoltek is not subject to the NSPS. Specifically, the USEPA states that Subpart VVV is intended to apply to those "polymeric coating processes where solvents are intentionally volatilized out of the coating as a necessary part of the process." This differs from pultrusion processes where "the volatile organic compound (styrene) is a reactant, not a solvent. The styrene predominantly becomes an integral part of the final product." Originally Zoltek was permitted to use styrene resins which the USEPA document specifically mentions. The new epoxy resin, while not containing styrene, is used in exactly the same way as



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the original styrene resin. Therefore, Subpart VVV does not apply to Zoltek's pultrusion machines operating with the styrene resin or epoxy resin coatings.

Zoltek will also be installing a KOHLER Power Systems Model 60REOZK diesel backup generator rated at 96 bhp. Diesel fuel will be held within a "belly" tank attached directly to the generator. Since the new epoxy resin and the emergency generator are being permitted simultaneously, emissions from these sources were aggregated in this project. The emergency generator was evaluated at 500 hours of annual operation (rather than 8760 hours) per the EPA memo "Calculating Potential to Emit (PTE) for Emergency Generators" (September, 1995). 10 CSR 10-6.070 *New Source Performance Standards* and 40 CFR 60 Part III, *Standards of Performance for Stationary Compression Ignition Internal Combustion Engines* as well as 10 CSR 10-6.075 *Maximum Achievable Control Technology Regulations* and 40 CFR 63 Part ZZZZ (only 40 CFR 63.6590(c)), *National Emission Standards for Stationary Reciprocating Internal Combustion Engines*, apply to the backup emergency generator.

#### Epoxy Resin Addition

Zoltek was originally permitted using a styrene based resin (VEX pultrusion) and was required to operate a fluidized concentrator and regenerative thermal oxidizer (FBC-RTO) at all times when production lines are in operation. Zoltek has proposed to use a new epoxy resin (EPON™ Resin 862) which will emit VOC and HAPs in the form of formaldehyde (CAS 50-00-0), phenol (CAS 108-95-2), and phthalic anhydride (CAS 85-44-9). Since the FBC-RTO was previously permitted it will control formaldehyde, phenol, and phthalic anhydride similar to styrene because they are all organic compounds that may readily oxidize. Therefore, the control and capture efficiency of the FBC-RTO will be applied to the new epoxy resin emissions without an additional permit.

On 5/3/2017, Civil & Environmental Consultants, Inc. (CEC) performed engineering testing on the outlet of the FBC-RTO system to determine emissions from a pultrusion machine using only the epoxy resin. The sampling was performed using USEPA Method 316 for formaldehyde and USEPA Method 0010 for phenol and phthalic anhydride. Only the three HAPs were tested because the epoxy resin is entirely (100%) composed of phenol-formaldehyde polymer glycidyl ether (CAS 28064-14-4) which decomposes into the constituent HAPs. No Method 18 pre-test was performed since the pre-test would not be useful in this situation. Specifically, Section 1.2.2 of Method 18 states, "This method will not determine compounds that (1) are polymeric (high molecular weight), (2) can polymerize before analysis, or (3) have very low vapor pressures at stack or instrument conditions." Essentially, all of these conditions apply to the epoxy operation.

The pultrusion machines can output carbon fiber at 30 inches/minute (length) with dimension of either 150x5 mm (width x height) or 200x2 mm (width x height). While the 150x5 mm carbon fiber will occupy a larger volume than the 200x2 mm carbon fiber, the epoxy resin emissions testing was performed with the 200x2 mm carbon fiber and proportionally scaled up by a factor

of 1.875 for 150x5 mm carbon fiber throughput (since epoxy resin usage and carbon fiber usage are directly proportional). Additionally, since the tests were performed with the pultrusion machine producing 16 inches/minute (length) of carbon fiber, the emissions were proportionally scaled up by a factor of 1.875 in order to account for a maximum extrusion rate of 30 inches/minute (length) of carbon fiber. Table 1 below summarizes the before scaling results and after scaling results for maximum emission rates of a single pultrusion machine.

Table 1: May 2017 Stack Test Results

HAP	Average Bay 2 Inlet Concentration (ppb) <sup>1</sup>	Average Bay 2 Inlet Volumetric Flowrate (dscfm)	Average Outlet Volumetric Flowrate (dscfm)	Ratio Of 150x5 mm to 200x2 mm	Ratio Of 30 in/min to 16 in/min	Maximum Hourly Emission Rate (Uncontrolled) (lb/hr) <sup>2</sup>	Maximum Hourly Emission Rate (Controlled) (lb/hr) <sup>3</sup>
Formaldehyde	16.3	2833.0	17359.7	1.875	1.875	8.944E-04	4.472E-05
Phenol <sup>4</sup>	3.1					4.389E-04	2.194E-05
Phthalic Anhydride <sup>5</sup>	N/Q					4.389E-04	2.194E-05

<sup>1</sup>Average Bay 2 Inlet Concentrations represent concentration of pollutants before the thermal oxidizer (uncontrolled). Average Outlet concentration represents outlet of the thermal oxidizer (controlled) calculated by applying the previously permitted 100% capture efficiency and 95% control efficiency.

<sup>2</sup>Uncontrolled lb/hr emission rates represent the Bay 2 inlet concentrations (no thermal oxidizer).

<sup>3</sup>Controlled lb/hr emission rates represent the thermal oxidizer outlet concentration. Emissions represent a single pultrusion line and a single stream (all 18 pultrusion lines are composed of two parallel carbon fiber production streams).

<sup>4</sup>Phenol emissions were detected neither at the Bay 2 epoxy inlet nor at the RTO outlet. The reporting limit from the laboratory was applied to calculate the concentrations and mass emission rates.

<sup>5</sup>N/Q represents Non-Quantifiable. Results for phthalic anhydride were inconclusive. Quality control samples at the analytical laboratory revealed unacceptable recovery efficiency from the sample matrix spike. Therefore the results for phthalic anhydride could not be quantified. At the laboratory, phthalic anhydride was assumed to have decomposed to phthalic acid on contact with water in the sampling train. Therefore phthalic acid was the analyte measured at the laboratory. The concentration of phthalic acid was below quantifiable thresholds and therefore no reporting limit could be determined by the laboratory. After both the Air Pollution Control Program and Zoltek were unable to establish a reporting limit for phthalic anhydride following diligent inquiry of authoritative sources, in order to conservatively account for phthalic anhydride for the project emissions, the reporting limit for phenol was used to calculate emissions for phthalic anhydride for the purpose of this permit amendment. However, if future testing is conducted to measure phenol emissions under this permit, it will not be assumed that phthalic anhydride emissions are equal to the phenol emissions as demonstrated through testing. Zoltek will continue to rely on the reporting limit for phenol established in the May 2017 test when calculating emissions of phthalic anhydride.

Resin is mixed with various additives before being added to the pultrusion machines. Mixing Emissions for the epoxy resin were found to be small based on results from the Emission Master batching calculations for VEX resin (the styrene resin currently used at the facility). The

uncontrolled VEX Resin pultrusion mixing evaporation for total VOC is 2.80E-03 tpy (this emission rate relates to the VEX resin that was previously permitted). This VOC values constitutes both styrene and MIBK (methyl isobutyl ketone) emissions from mixing. For the new epoxy resin, VOC in the form of formaldehyde, phenol, and phthalic anhydride will primarily evolve from the evaporation and degradation of the only epoxy resin compound, Phenol-Formaldehyde Polymer Glycidyl Ether (aka Poly[(phenyl glycidyl ether)-*co*-formaldehyde]) with an SDS provided vapor pressure of 0.62 mmHg (82 Pa) at standard temperature and pressure (STP). Phenol is a solid at STP while Formaldehyde is a vapor at STP, however both VOCs were observed during testing of the pultrusion lines which operate at STP/ambient conditions. Additionally, the SDS states that the Phenol-Formaldehyde polymer constitutes 100% of the epoxy resin (i.e. little to no free formaldehyde or phenol). This suggests that formaldehyde and phenol emissions result entirely from the evaporation and degradation of the epoxy resin polymer. According to the National Institute for Occupational Safety and Health (NIOSH), the vapor pressure for styrene and MIBK is 5 mmHg (667 Pa STP) and 16 mmHg (2133 Pa STP), respectively. Therefore, it was assumed that since Phenol-Formaldehyde Polymer Glycidyl Ether has magnitudes smaller vapor pressure, the constituent HAP emissions would not exceed the 2.80E-03 tpy emission rate for styrene and MIBK.

#### Revised Special Conditions

As a part of this permitting action, all special conditions from the previously issued Construction Permit No. 112015-003 are being superseded and revised consistent with the terms of the August 15, 2017, AOC, which among other issues, allowed Zoltek a 70 minute time frame to cease operation of all 18 of the carbon fiber pultrusion lines in the event of a regenerative thermal oxidizer (RTO) malfunction.

Currently, the pultrusion lines are controlled by a fluidized bed concentrator (FBC) which feeds an RTO. Emissions from the pultrusion lines are captured by an exhaust system built to have a 100% capture efficiency in compliance with Method 204 for enclosures. The exhaust system then feeds the fluidized bed concentrator (FBC). Essentially the FBC concentrates VOCs, such as styrene, by adsorbing them with activated carbon. The inlet stream to the FBC from the pultrusion lines has a high air flow rate which is brought into contact with activated carbon which pulls the vast majority of VOCs out of the air. The high volume of air is then vented outside of the facility. The activated carbon, which is carrying the VOCs, is then transferred within the FBC to a desorption chamber. The activated carbon then desorbs the VOCs into a new stream of air with a much smaller volumetric flowrate than the FBC inlet air. In this way volatiles are concentrated before being destroyed within the RTO, allowing for much greater destruction efficiency. The RTO itself is rated at 0.867 MMBtu/hr (867 scfh of natural gas rated at 1,000 btu/ft<sup>3</sup>). Essentially, the FBC allows the RTO to have a lower natural gas fueling capacity than similar systems in which there is no initial concentration and all air from the pultrusion lines are fed directly into the RTO.

Between 8/31/2016 and 9/1/2016, Civil & Environmental Consultants, Inc. (CEC) performed stack tests on the FBC-RTO in order to determine the overall control efficiency for the entire system. It was determined that the combined system has an overall control efficiency of 98.97%. To be conservative, Zoltek has requested a slightly lower overall 95% control efficiency. In the event of an RTO failure, the FBC has the capacity to stop desorbing material. Essentially, the FBC can act as temporary storage for volatiles. The CEC report was performed while 9 of the pultrusion lines were operating at maximum capacity and found that the FBC could capture 95% of pollutants for over 2 hours. Thus, it was extrapolated that all 18 lines could have pollutants effectively adsorbed at 95% capture over half as much time (70 minutes).

The FBC has a sophisticated software system that can measure various parameters of the FBC such as temperatures. The software is also specifically set to shut down desorption in the FBC if the RTO begins to fall below 1,500 °F. As stated earlier, the FBC will continue to adsorb pollutants from all pultrusion lines at 95% efficiency for 70 minutes (i.e. the activated carbon in the adsorption chamber continues to adsorb even though the FBC is no longer transferring carbon between the adsorber and the desorber). In order to monitor that the FBC is operating properly, the special conditions of this amendment require Zoltek to monitor the pressure in the adsorption chamber and the desorption chamber. Zoltek will also need to use sight glasses on the FBC to visually inspect and verify activated carbon is flowing between the two sides of the FBC.

#### Existing Potential to Emit Calculations

As a part of this amendment, Zoltek submitted additional usage rate information for the styrene resin which changes the original permit's potential to emit slightly. The original permit used AERSCREEN to show that while styrene emissions are above the SMAL of 1.0, emissions do not exceed the 24-hour and annual RAL limits. Styrene emissions calculated for this amendment are lower than those of the original permit and therefore additional modeling was not required. Currently, Zoltek can produce 390.0 board feet per hour (where 1 board feet [bf] = 0.08333 ft<sup>3</sup>) of carbon fiber product from all 18 pultrusion machines. Based upon observed usage rates, styrene resin usage is 0.197 gallons per bf of product. Therefore maximum hourly styrene resin usage is 77 gallons/hour. Zoltek follows a specific recipe for mixing and producing the resin in house which contains 44.13% styrene, 0.17% MIBK (methyl isobutyl ketone), and 46.82% VOC by weight and has a maximum density of 10.8 lb/gal. Hourly emissions for styrene, MIBK, and VOC were then calculated by multiplying the maximum hourly styrene resin usage rates by the weight percent of each pollutant. Styrene emissions are smaller for this project since guidance from the South Coast AQMD document entitled, "Guidelines for Calculating Emissions from Polyester Resin Operations" (December, 2016), states that only between 4-7% of styrene throughput will become styrene emissions (7% was conservatively used for calculations). Therefore styrene emissions calculated via mass balance were then multiplied by an additional 7% in accordance with the AQMD document.

Emissions for mixing the styrene resin were calculated using Emission Master. Emission Master software utilizes a chemical properties database to estimate volatile emissions, batch density, and batch volume. Typical batches weigh around 467.28 lbs (54.1768 gallons) and take about 100 minutes to properly mix all of the ingredients. The final batch volume was then divided by the total yearly styrene resin usage rates which determined that 12,450 batches per year would be needed for the pultrusion machines operating at full capacity. The batch emissions from Emission Master software were then multiplied by the total yearly batches to calculate yearly styrene, MIBK, and VOC emissions from resin mixing. When comparing epoxy resin emissions calculated in the previous section with those emissions produced from the styrene resin, it was observed that the styrene resin is by far the worst case emitter except for formaldehyde and phenol. This is true for both pultrusion emissions and mixing emissions.

Particulate emission rates from cutting carbon fiber were not recalculated for this amendment since PM, PM<sub>10</sub>, and PM<sub>2.5</sub> emission rates were correctly calculated by the original permit to be 0.003 tons/year. This was based upon the volume of carbon fiber that is cut, estimated at 1.01E-4 cubic feet per cut. It was conservatively assumed the total volume cut would result entirely in particulate less than 2.5 microns in diameter since PM<sub>2.5</sub> has a smaller de minimis limit than PM or PM<sub>10</sub>.

Natural gas and diesel fuel combustion emission factors were derived from EPA document AP-42, *Compilation of Air Pollutant Emission Factors, Fifth Edition, Section 1.4 Natural Gas Combustion* (July, 1998) and Section 3.3, *Gasoline and Diesel Industrial Engines* (October, 1996). The RTO's maximum hourly fuel throughput is 867 standard cubic feet (scf) of natural gas and the diesel backup generator is rated with a maximum fuel consumption of 5.4 gallons per hour. AP-42 designates the heat content and density of diesel fuel to be 19,300 btu/lb and 6.94 lb/gal, respectively. The backup generator was evaluated over 500 hours since it is used for emergency purposes only.

For this amendment's New Installation Conditioned Potential, the emissions from the new 96 bhp diesel backup generator were added to the emissions from the pultrusion lines and thermal oxidizer operating with styrene resin. Existing Potential Emissions were recalculated using the updated information provided by Zoltek for this amendment. The below Table 2 summarizes the controlled emissions of using the new epoxy resin using a thermal oxidizer evaluated over an 8760 hour period. Formaldehyde and phenol emissions from the epoxy resin were listed separately since styrene resin is the worst case emitter. The maximum hourly emission rates summarized in Table 1 were multiplied by 36 in order to account for each of the two lines of all 18 pultrusion devices simultaneously utilizing the new epoxy resin.

Table 2: Potential To Emit (tpy)

Pollutant	Regulatory <i>De Minimis</i> Levels or <i>SMAL</i>	Existing Potential Emissions <sup>1</sup>	Existing Actual Emissions (2015 EIQ) <sup>2</sup>	Controlled Potential Emissions from Epoxy-Resin Pultrusion	New Installation Conditioned Potential <sup>3,4</sup>
PM	25.0	0.03	N/A	N/A	0.09
PM <sub>10</sub>	15.0	0.03	0.03	N/A	0.09
PM <sub>2.5</sub>	10.0	0.03	0.03	N/A	0.09
SO <sub>2</sub>	40.0	2.30E-03	N/A	N/A	0.06
NOx	40.0	0.38	N/A	N/A	1.18
VOC	40.0	6.47	20.71	5.00E-03	6.53
CO	100.0	0.32	N/A	N/A	0.49
Total HAPs	10.0/25.0	4.75	20.45	5.00E-03	4.75
Styrene	1.0	4.49	20.38	N/A	4.49
MIBK	10.0	0.25	0.07	N/A	0.25
Formaldehyde	2.0	N/A	N/A	4.00E-03	4.00E-03
Phenol	0.1	N/A	N/A	1.70E-03	1.70E-03
Phthalic Anhydride	5.0	N/A	N/A	1.70E-03	1.70E-03

N/A = Not Applicable

<sup>1</sup>Includes only controlled vex (styrene) resin pultrusion emissions, oxidizer combustion emissions, and cutting emissions.

<sup>2</sup>Existing potential emissions were recalculated for this project. Existing actual emissions are larger than existing potential emissions because they use the original emission factors which were larger.

<sup>3</sup>Styrene emissions were originally modeled using AERSCREEN for construction permit 112015-003 at 7.57 tons per year which satisfied the 24-hour and annual RAL. Styrene emissions have now been calculated to still be greater than the SMAL of 1 tpy but less than the previously modeled 7.57 tons per year and therefore re-modeling was not required. Styrene emissions are smaller for this project since guidance from the South Coast AQMD document entitled, "Guidelines for Calculating Emissions from Polyester Resin Operations" (December, 2016), states that only between 4-7% of styrene throughput will become styrene emissions (7% was used for calculations).

<sup>4</sup>Includes the existing potential emissions, the emissions of the new backup generator, and adds additional entries for formaldehyde, phenol and phthalic anhydride emissions from the new epoxy resin.

If you were adversely affected by this permit decision, you may be entitled to pursue an appeal before the administrative hearing commission pursuant to Sections 621.250 and 643.075.6 RSMo. To appeal, you must file a petition with the administrative hearing commission within thirty days after the date this decision was mailed or the date it was delivered, whichever date was earlier. If any such petition is sent by registered mail or certified mail, it will be deemed filed on the date it is mailed; if it is sent by any method other than registered mail or certified mail, it will be deemed filed on the date it is received by the administrative hearing commission,

Mr. Terry Miner  
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whose contact information is: Administrative Hearing Commission, United States Post Office Building, 131 West High Street, Third Floor, P.O. Box 1557, Jefferson City, Missouri 65102, phone: 573-751-2422, fax: 573-751-5018, website: [www.oa.mo.gov/ahc](http://www.oa.mo.gov/ahc).

If you have any questions regarding this amendment, please do not hesitate to contact Hans Robinson, at the department's Air Pollution Control Program, P.O. Box 176, Jefferson City, MO 65102 or at (573) 751-4817. Thank you for your attention to this matter.

Sincerely,

AIR POLLUTION CONTROL PROGRAM



Kendall B. Hale  
Permits Section Chief

KBH:hrj

Enclosures

cc: St. Louis Regional Office  
PAMS File: 2017-07-056

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**SPECIAL CONDITIONS:**

The permittee is authorized to construct and operate subject to the following special conditions:

*The special conditions listed in this permit were included based on the authority granted the Missouri Air Pollution Control Program by the Missouri Air Conservation Law (specifically 643.075) and by the Missouri Rules listed in Title 10, Division 10 of the Code of State Regulations (specifically 10 CSR 10-6.060). For specific details regarding conditions, see 10 CSR 10-6.060 paragraph (12)(A)10. "Conditions required by permitting authority."*

Zoltek

St. Charles County, Landgrant 00731, T47N, R3E

1. The Special Conditions of this permit supersede all special conditions found in Construction Permit 112015-003 previously issued by the air Pollution Control Program.
2. Control Device Requirement – Thermal Oxidizer and Fluidized Bed Concentrator
  - A. Zoltek shall control emissions from the eighteen (18) pultrusion lines (EP-1 – EP-18) and resin mixing/ handling (EP-19) using a fluidized bed concentrator (FBC) which feeds a regenerative thermal oxidizer (RTO), as specified in the revised permit application.
  - B. The fluidized bed concentrator and thermal oxidizer shall be operated and maintained in accordance with the manufacturer's specifications. The RTO shall be in use at all times when EP-1 – EP-18 and/or EP-19 are in operation. If the RTO malfunctions, production lines may operate for a period not to exceed 70 minutes from the start of the RTO malfunction, as long as Zoltek operates the FBC in accordance with manufacturer's specifications during the 70-minute period. If the RTO has not resumed normal operating condition at the end of the 70-minute period, Zoltek shall assume that excess emissions are occurring and shall operate and comply under the Start-Up, Shutdown, and Malfunction Conditions of 10 CSR 10-6.050 if such excess emissions exceed one (1) hour as set forth in 10 CSR 10-6.050(3)(A).
  - C. Zoltek shall continuously monitor and record the combustion zone temperature of the RTO while the plant is operating. This includes all periods of startup, shutdown, and malfunction (SSM). Zoltek shall maintain the temperature of the RTO at a minimum of 1,500 °F (i.e. any 15 minute period during which the RTO's temperature falls below 1,500 °F must be recorded as a SSM period if after a period of 70 minutes the thermal oxidizer has not returned to normal operation per Special Condition 2.B).

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**SPECIAL CONDITIONS:**

The permittee is authorized to construct and operate subject to the following special conditions:

- D. If the temperature of the RTO falls below 1,500 °F, Zoltek shall ensure the FBC ceases to desorb VOC and HAPs (i.e. VOC and HAPs are no longer flowing from the FBC to the RTO). Documentation of shut-downs of the FBC generated by the FBC software shall be kept available as electronic copies per Special Condition 3. These logs are required to show the date and time of the FBC shut-down.
  - E. Zoltek shall monitor the differential pressures of the middle and lower chambers of the FBC adsorber to ensure the pressure differential between the middle chamber and atmospheric pressure, and the pressure differential between the lower chamber and the middle chamber, are maintained above baseline levels established during functional acceptance testing. Zoltek shall monitor activated carbon flow within the FBC by visually observing activated carbon flowing via sight glasses.
  - F. Zoltek shall maintain a copy of the RTO's and FBC's manufacturers' performance warranties on site.
  - G. Zoltek shall maintain an operation and maintenance log for the RTO and FBC, which shall include the following:
    - 1) Incidents of malfunction, with impact on emissions, duration of event, probable cause, and corrective actions; and
    - 2) Maintenance activities, with inspection schedule, repair actions, and replacements, etc.
3. Record Keeping and Reporting Requirements
- A. Zoltek shall maintain all records required by this permit for not less than five years and shall make them available immediately to any Missouri Department of Natural Resources' personnel upon request. These records shall include SDS for all materials used.
  - B. Zoltek shall report to the Air Pollution Control Program's Compliance/Enforcement Section, P.O. Box 176, Jefferson City, MO 65102 or [AirComplianceReporting@dnr.mo.gov](mailto:AirComplianceReporting@dnr.mo.gov) no later than 10 days after the end of the month during which any record required by this permit shows an exceedance of a limitation imposed by this permit, including all time periods in which operation under the malfunction provisions of Special Condition 2.B of this permit occur.