



St. Joseph, Missouri - Water Protection Division

Handling of Split Sample Results

With Industrial Facilities



Water Protection conducts routine effluent sampling of permitted and unpermitted Industrial Facilities throughout St. Joseph. Some samples are collected solely to determine compliance with permitted effluent limitations and local prohibitive discharge standards and some samples are collected both for compliance purposes and for high strength sewage surcharge billing purposes. Industrial facilities often request to split the samples collected by Water Protection so they can submit them to an independent third party laboratory. Water Protection is happy to oblige these requests when the Industrial Facility is properly prepared to split samples. This fact sheet includes some information that Industrial Facilities should be aware of when splitting samples and describes the process to reconcile large differences between the results of the Water Protection Laboratory and the Industrial Facility's third party laboratory.

Sample Splitting

Water Protection conducts sampling and laboratory analyses according to Federal Regulation 40 CFR Part 136 and Standard Methods for the Examination of Water and Wastewater. These resources specify the analytical methods, sample types, preservation, hold times, chain of custody procedures and Quality Assurance/Quality Control (QA/QC) measures required to ensure that analytical results are accurate. If an Industrial Facility wishes to split samples with Water Protection personnel, they must have all of the correct containers and any applicable preservatives on hand at the time of sample collection.

Water Protection may conduct announced or unannounced sampling. When sampling is announced, the Industrial Facility may ask when they need to have supplies ready and what analytes will be sampled for. When sampling is unannounced, the Industrial Facility still has the opportunity to be prepared. Water Protection personnel will set up the automatic composite sampler on the day of their unannounced visit and will return the following day to collect the composite sample and any required grab samples. If the Industrial Facility is unprepared on day-one, there are third party labs that can ensure that the proper containers and preservatives arrive by day-two sample collection time. To prepare ahead of time, Industrial Facilities can follow these guidelines for analytes that will likely be sampled for:

- **Surcharge Billing Sampling** – Biochemical Oxygen Demand (BOD), Total Suspended Solids (TSS), Ammonia as Nitrogen, and Oil & Grease.
- **Permitted Industrial Facilities** – All analytes listed in your Wastewater Contribution Permit as well as the surcharge billing analytes if not already included in your permit.
- **Unpermitted Industrial Facilities** – All analytes listed in St. Joseph City Code Chapter 29, Section 157.

If split samples submitted to a third party laboratory do not comply with all sample collection, preservation, hold time, and chain of custody requirements, Water Protection will not consider the results valid.

Submission of Split Sample Results

All samples collected by Water Protection may potentially be used for compliance purposes, therefore, U.S. EPA guidance (see below) requires Water Protection to use split sample results in compliance determinations. Permitted Industrial Facilities that split samples with Water Protection are required, by



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permit conditions, to submit the results of all split samples to Water Protection for review. If unpermitted Industrial Facilities split samples with Water Protection they will be required to submit those results when they are available from the third party laboratory.

Water Protection's Handling of Split Sampling Results

In the early 1990s the U.S. EPA wrote a memorandum titled "Determining Industrial User Compliance Using Split Samples" and a letter to the Greater Fort Wayne, Indiana Chamber of Commerce titled "Using Split Samples to Determine Industrial User Compliance", both of which address how Publicly Owned Treatment Works (POTW) such as Water Protection should handle the results of samples split by Industrial Facilities. These documents indicate the following:

1. If an Industrial Facility has split sample results, the POTW must use those results in conjunction with their own results in compliance determinations (this does not apply to surcharge billing).
2. If split sample results are "widely divergent", the POTW should investigate the QA/QC data for each laboratory involved and discard any results that cannot be properly validated.
3. The term "widely divergent" varies from analyte to analyte and the determination of what is considered "widely divergent" is left to the discretion of the POTW.
4. It is the POTW's responsibility to determine the validity of split sample results.
5. The burden of proof is on the Industrial Facility to show that all preservation, chain of custody, and analytical QA/QC methods were followed.

To comply with the requirements of the two cited EPA documents, Water Protection follows the following procedures for Industrial Facility split sample results.

Compliance Determinations – Water Protection works diligently to ensure that any data generated by its laboratory complies with QA/QC requirements and personnel validate results prior to publication. If any non-compliance is found resulting from Water Protection sampling where splits were analyzed by the Industrial Facility, a preliminary compliance determination may be made with the Water Protection Laboratory's results without first obtaining the split sample results. Preliminary actions may include but not be limited to verbal or email notifications or a follow up site visit. No official enforcement actions will be completed until the split sample results have been reviewed. As such, upon preliminary notification by the POTW, the Industrial Facility must submit the split sample results as soon as they are available. Furthermore, if the Industrial Facility receives split sample results that indicate noncompliance but they have not received any notification from the POTW, they are required by St. Joseph City Code § 29-158 to notify the POTW within 24 hours of becoming aware of the noncompliance. Once split sample results have been received by Water Protection the following steps will be followed:

1. Water Protection will evaluate the chain of custody forms, laboratory Sample Condition Upon Receipt (SCUR) documentation, analytical results, and QA/QC information to determine whether or not the split sample results are to be considered valid.
2. If both Water Protection's results and split results are valid and are not "widely divergent", the average of the two results will be used for compliance determination.
3. If either Water Protection's or the split sample results are found to be invalid, they will be discarded and the remaining valid result will be used for compliance determination.



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4. If both results are found to be invalid, both will be discarded.
5. If both results are found to be valid but are also determined to be “widely divergent”, both results will be discarded.
6. “Widely Divergent” will vary from analyte to analyte but will be based on the QC acceptance criteria for analyte standard recovery percentages or relative percent difference (RPD) between duplicate samples that are required by US EPA approved methods and Water Protection Laboratory Standard Operating Procedures. Examples are as follows:
 - a. BOD – RPD above 30.5% is widely divergent
 - b. TSS – RPD above 10% is widely divergent
 - c. Ammonia – RPD above 20% is widely divergent
 - d. Oil & Grease – RPD above 18% is widely divergent

Surcharge Billing – US EPA does not require the POTW to use split sample results for surcharge billing. Typically the Water Protection Laboratory generates final data much sooner than a third party laboratory provides analytical reports to their customers. Because the Water Protection Laboratory takes great effort to thoroughly validate all data prior to sending it to the City of St. Joseph’s Finance Department for billing and because of the time constraints of monthly billing cycles, Water Protection will use their data alone for initial billing purposes. If, after receiving an invoice from the Finance Department, the Industrial Facility believes there are “widely divergent” data between Water Protection’s results and their split results, they must file an appeal per St. Joseph City Code § 29-253 within fifteen work days of the billing date. The appeal must indicate which sample results are in question and must include complete laboratory reports, including chain of custody and SCUR documentation, and all associated QA/QC information for all split samples analyzed during the billing cycle even if not all results are in question. Water Protection will follow steps one through six above upon receipt of a complete billing appeal and will apply those rules to all split sample results for the billing cycle; not just the ones that the Industrial User has questioned.