Casting Emission Reduction Program

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US Army Task N256

Emission Measurement Procedure 1

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EPA United States Environmental Protection Agency

CISA

AFS AMERICAN FOUNDRY SOCIETY, INC.

UNITED STATES COUNCIL FOR AUTOMOTIVE RESEARCH
USCAR
DAIMLERCHRYSLER Ford Motor Company General Motors

Emission Measurement Procedure 1

WBS 3.1.1

30 April 2001

Hazardous Air Pollutant (HAP) Method Verification Protocol and Data Quality Requirements

WBS 3.3.1

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A. Background

The Clean Air Act Amendments (CAAA) of 1990 promulgated a list of 188 organic and inorganic compounds and identified them as Hazardous Air Pollutants (HAPs). The CAAA also limited the amount of HAP emissions from a facility. The accurate measurement of HAP emissions from facilities including foundries depends on sampling and analysis methods accepted by the US EPA. A previous work product under this Subtask and Subtask 3.2 identified a list of organic HAPs of interest to the foundry industry and determined that none of the methods had been verified/validated in a foundry environment.

B. Objective

The objective of this work product is to define the verification/validation protocol that will be used to verify (existing method) or validate (new method) the accuracy (bias) and repeatability (precision) of sampling and analysis methods for selected HAPs. The protocol will include an "experimental" section detailing the laboratory process that will be followed. The data quality objectives of the verification/validation protocol will also be defined.

NOTE: The experimental procedures and data quality objectives discussed below are intended to provide guidance for analysts experienced in emissions sample collection and the operation and use of the analytical instrumentation specified.

C. Experimental Procedures

The experimental procedures associated with a method to be verified or validated include those associated with target analyte (HAP) collection, storage of the collected samples, target analyte recovery, target analyte analysis, and the effect of the foundry pouring/cooling/shakeout emissions matrix on the above components of the method.

1. Target Analyte Collection
   The collection efficiency and breakthrough volume of the sample collection media will be determined from the literature or from laboratory work with the target analyte. The collection efficiency will be confirmed by spiking sample collection media with known amounts of the target analyte and then collecting actual foundry emission samples. The breakthrough volume will be determined through the use of sample collection media with "front" and "back" sections that will be analyzed separately. The amount of target analyte added to the sample collection media for matrix spikes will be between 40% and 60% of the amount of target analyte expected to be collected during an emissions test.

2. Collected Sample Storage Stability
   The above samples will be collected in sufficient quantity that samples can be analyzed immediately after collection, and after one (1) two (2) and four (4) weeks. Storage will be evaluated at both room temperature and at freezer temperature.
3. Target Analyte Recovery (Desorption Efficiency)
   Individual sample collection media will be spiked with known amounts of the target analyte, stored at room temperature for at least twelve (12) hours, and desorbed with the appropriate solvent. The desorbate will be analyzed to determine the target analyte recovery. Sample collection media will also be spiked with known amounts of the target analyte and then used to collect emissions from pouring/cooling/shake out.
4. Target Analyte Analysis
   Analysis of the target analyte will be by gas chromatography (GC) with flame ionization detection (FID). A column and instrument conditions will be selected that provides adequate resolution of the target analyte from other analytes so that the amount of target analyte can be accurately determined.

D. Data Quality Objectives

The data quality objectives are designed to meet or exceed the requirements of US EPA Method 18 for the determination of organic compounds in stationary source emission by gas chromatography.

1. Target Analyte Collection
   The sample collection media, sampling flow rate, and duration of the sampling will provide sufficient sample for analysis so that the target analyte response is at least twice to background or blank response. In addition, at least 90% of the total amount of target analyte collected will be retained in the front section of sample collection media.
2. Collected Sample Stability
   The amount of target analyte in the collected samples will be stable for at least two (2) weeks after collection when stored at freezer temperature. Stable means that the amount of target analyte found in the samples stored for two (2) weeks is not statistically different than the amount of target analyte found in the samples analyzed immediately after collection The T Statistic at a 95% confidence interval shall be used to determine if the data are statistically different.
3. Target Analyte Recovery (Desorption Efficiency)
   The desorption efficiency of the target analyte will be at least 75%. Recovery of the target analyte from matrix spikes will no less than 70% and no more than 130%.
4. Target Analyte Analysis
   The initial calibration (IC) of the GC/FID will be over the range of expected target analyte amounts using at least five (5) points. The curve fit shall have a correlation coefficient of at least 0.99. Continuing calibration verification (CCV) will be conducted at a single point using different concentrations within the calibration range for each CCV. The CCV shall be accepted if the percentage difference between the IC response factor and the CCV response factor is less than 10%. The CCV will be run at least once for each sample batch of ten (10) or fewer samples. A solvent blank or sample collection media blank will be run with each sample batch. A sample analysis duplicate will also be run with each sample batch.

Duplicate analyses will be within 5% of the average of the two (2) determinations for single target analytes and 10% for summed parameters.

E. Reporting

The results of a method verification or method development effort will be documented in a final approved report. The report will describe the specific experimental procedures used including the sampling, and analysis protocols used. The report will also compare the results with the data quality objectives specified above.