# Technical Support Document for the Adipic Acid Production Sector: Proposed Rule for Mandatory Reporting of Greenhouse Gases

Office of Air and Radiation U.S. Environmental Protection Agency

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#### 1. Industry Description

Adipic acid is a white crystalline solid used in the manufacture of synthetic fibers, plastics, coatings, urethane foams, elastomers, and synthetic lubricants. Commercially, it is the most important of the aliphatic dicarboxylic acids, which are used to manufacture polyesters. Eighty-four percent of all adipic acid produced in the United States is used in the production of 6,6-nylon, nine percent is used in the production of polyester polyols, four percent is used in the production of plasticizers, and the remaining four percent is accounted for by other uses, including unsaturated polyester resins and food applications (ICIS 2007). Food grade adipic acid is used to provide some foods with a "tangy" flavor (Thiemens and Trogler 1991; EPA 2008).

Worldwide, few adipic acid plants exist. The United States and Europe are the major producers. The United States has three companies in four locations accounting for 34 percent of world production, and eight European producers account for a combined 38 percent of world production (CW 2007).

Adipic acid is produced through a two-stage process during which  $N_2O$  is generated in the second stage. The first stage of manufacturing usually involves the oxidation of cyclohexane to form a cyclohexanone/cyclohexanol mixture. The second stage involves oxidizing this mixture with nitric acid to produce adipic acid. The  $N_2O$  is generated as a by-product of the nitric acid oxidation stage and is emitted in the waste gas stream (Thiemens and Trogler 1991). A representation of the process is shown below.

 $(CH_2)5CO (Cyclohexanone) + (CH_2)5CHOH (Cyclohexanol) + wHNO_3 \rightarrow$ 

 $HOOC(CH_2)4COOH$  (Adipic Acid) +  $xN_2O + yH_2O$ 

Process emissions from the production of adipic acid vary with the types of technologies and level of emission controls employed by a facility. In 1990, two of the three major adipic acid-producing plants had  $N_2O$  abatement technologies in place and, as of 1998, the three major adipic acid production facilities had control systems in place. Only one small plant, representing approximately two percent of production, does not control  $N_2O$  emissions (IPCC 2006; EPA 2008).

# 2. Total Emissions

Process-related  $N_2O$  emissions and stationary combustion emissions from adipic acid production were estimated to be 9,297,866 metric tons  $CO_2e$  in 2006. Process  $N_2O$  emissions alone were estimated at 5,921,434 metric tons  $CO_2e$  (mt $CO_2e$ ). Stationary combustion emissions were estimated at 3,376,432 mt $CO_2e$ . National adipic acid production has increased by approximately 36 percent over the period of 1990 through 2006, to approximately one million metric tons. At the same time, emissions have been reduced by 61 percent due to the widespread installation of pollution control measures in the late 1990s (EPA 2008).

# 2.1 Process Emissions

Process related estimates of  $N_2O$  from adipic acid production were derived for the U.S. Inventory, and are estimated at 5,921,434 metric tons  $CO_2e$  in 2006. Facility-specific data is Confidential Business Information (CBI) for two plants. Consequently, the detailed facilitylevel data are not presented here.

# 2.2 Stationary Combustion

Adipic acid production facilities consume fossil fuels for stationary combustion which contribute to their GHG emission total. GHG (CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O) emissions from on-site fossil fuel combustion were estimated using data collected through title V permitting for two of the four production facilities, as well as CO<sub>2</sub> emission factors, which were derived using heat content and carbon content data contained in the Inventory of U.S. Greenhouse Gas Emissions and Sinks 1990-2006. Emission factors for CO<sub>2</sub> were derived from Table 2.3 of the 2006 IPCC Guidelines for National Greenhouse Gas Inventories (IPCC 2006). These carbon content factors are for Manufacturing Industries and Construction and apply to stationary source combustion CO<sub>2</sub> emissions, not process N<sub>2</sub>O emissions. They are presented in Table 1. For more information on combustion related N2O and CH4 emissions refer to the stationary combustion Technical Support Document in EPA-HQ-OAR-2008-0508-004.

Fuel Category	Fuel Type	Heat Content	Carbon Content
		Value Unit	(Tg Carbon/QBTU)
Solid Fuels	Anthracite Coal	22.57 MMBtu/Short Ton	28.26
	Bituminous Coal	23.89 MMBtu/Short Ton	25.49
	Sub-bituminous Coal	17.14 MMBtu/Short Ton	26.48
	Lignite	12.87 MMBtu/Short Ton	26.30
	Coke	24.80 MMBtu/Short Ton	31.00
	Unspecified	25.97 MMBtu/Short Ton	25.34
Gas Fuels	Natural Gas	1,029 Btu/ft3	14.47
Liquid Fuels	Crude Oil	5.80 MMBtu/Barrel	20.33
-	Nat Gas Liquids and LRGs	3.71 MMBtu/Barrel	16.99
	Other Liquids	5.83 MMBtu/Barrel	20.33
	Motor Gasoline	5.22 MMBtu/Barrel	19.33
	Aviation Gasoline	5.05 MMBtu/Barrel	18.87
	Kerosene	5.67 MMBtu/Barrel	19.72
	Jet Fuel	5.67 MMBtu/Barrel	19.33
	Distillate Fuel	5.83 MMBtu/Barrel	19.95
	Residual Oil	6.29 MMBtu/Barrel	21.49
	Naphtha for Petrofeed	5.25 MMBtu/Barrel	18.14
	Petroleum Coke	6.02 MMBtu/Barrel	27.85
	Other Oil for Petrofeed	5.83 MMBtu/Barrel	19.95
	Special Naphthas	5.25 MMBtu/Barrel	19.86
	Lubricants	6.07 MMBtu/Barrel	20.24
	Waxes	5.54 MMBtu/Barrel	19.81
	Asphalt/Road Oil	6.64 MMBtu/Barrel	20.62
	Still Gas	6.00 MMBtu/Barrel	17.51
	Misc. Products	5.80 MMBtu/Barrel	20.33

# Table 1 - Heat and Carbon Contents Provided by the Inventory of U.S. Greenhouse Gas Emissions and Sinks 1990-2006

Source: Inventory of U.S. Greenhouse Gas Emissions and Sinks 1990-2006

Combustion-related emissions were estimated by averaging the sum of calculated emissions by unit type (e.g. boiler, generator, etc.) from two of the adipic acid plants.. The average combustion emissions were assumed to apply to all four plants. It was also assumed that 100 percent of the emissions from the stationary combustion sources are attributed to adipic acid production, even though there are other processes at the adipic acid plants that may be using steam or electricity from the stationary combustion equipment. The total calculated combustion emissions are shown in Table 2, along with process-related emissions from all four adipic acid plants. Detailed facility information is not provided given the small number of facilities and confidential information.

#### 3. Review of Existing Programs and Methodologies

In evaluating monitoring options for adipic acid production, multiple GHG emissions reporting guidance documents were consulted. These include documents developed by the U.S. Environmental Protection Agency (USGHG, 2008), the Intergovernmental Panel for Climate Change (IPCC, 2006), the Climate Registry (CR, 2007), the World Business Council for Sustainable Development (WBCSD, 2001), and the U.S. Department of Energy (USDOE, 2007). The main monitoring methods from each of these reporting programs are reviewed below.

# 3.1 2006 IPCC Guidelines

The Tier 1 methodology estimates emissions using the total production of adipic acid and the appropriate default emission factor (300 kg N<sub>2</sub>O/metric ton adipic acid). The Tier 1 method should be applied assuming no abatement of N<sub>2</sub>O emissions and the use of the highest default emission factor based on technology type. The Tier 2 methodology estimates emissions using facility-specific information, including the production rate of adipic acid, the default emission factor, the destruction factor for abatement technology, and the utilization factor of the abatement system (if applicable). The equation is shown below. The Tier 3 methodology estimates emission factors that are obtained from direct measurement of emissions. These may be derived from periodic sampling of N<sub>2</sub>O or periodic emissions monitoring of N<sub>2</sub>O over a period that reflects the usual pattern of operation of the plant.

$$E_{N_2O} = \sum \left( EF_i * AAP_i * \left( 1 - DF_j * ASUF_j \right) \right)$$

where:

E <sub>N2O</sub>	=	emissions of N <sub>2</sub> O, kg
EFi	=	$N_2O$ emission factor for technology type i, kg $N_2O/\text{metric}$ ton adipic acid produced
AAP <sub>i</sub>	=	adipic acid production from technology type i, metric tons
$\mathrm{DF}_{\mathrm{j}}$	=	destruction factor for abatement technology type j, fraction
ASUF <sub>j</sub>	=	abatement system utilization factor for abatement technology type j, fraction.

# 3.2 WRI/WBCSD Protocol

Approach 1 involves precise direct monitoring of  $N_2O$  emissions, with measurements at both the exit stream and the uncontrolled stream. Data quality is satisfactory when measurement data are only available for the exit stream. Approach 2 involves site-specific  $N_2O$  emission factors. This approach is based on IPCC Tier 2 methodology. Approach 3 involves the use of default emission factors for  $N_2O$  emissions. This approach is based on IPCC Tier 1 methodology.

#### 3.3 2008 U.S. Inventory of Greenhouse Gas Emissions and Sinks

The Tier 2 and 3 methodologies were used for adipic acid production. For two production plants, 1990 to 2002 emission estimates were obtained directly from the plant engineer and account for reductions due to control systems in place at these plants during the time series (Childs 2002, 2003). These estimates were based on continuous emissions monitoring equipment installed at the two facilities. Reported emission estimates for 2003 to 2006 were unavailable and, thus, were calculated by applying 4.4, 4.2, 0.0, and 0.0 percent national production growth rates, respectively.

For the other two plants,  $N_2O$  emissions were calculated by multiplying adipic acid production by an IPCC 2006 emission factor (i.e.,  $N_2O$  emitted per unit of adipic acid produced) and adjusting for the percentage of  $N_2O$  released as a result of plant-specific emission controls. Emissions are estimated using the following equation:

$$\begin{split} N_2O \ emissions = (production \ of \ adipic \ acid \ [metric \ tons \ of \ adipic \ acid]) \times (0.3 \ metric \ tons \ N_2O \\ / \ metric \ ton \ adipic \ acid) \times (1 - [N_2O \ destruction \ factor \times \ abatement \ system \ utility \ factor]) \end{split}$$

# 3.4 The Climate Registry

The Tier A2 methodology is a mass balance approach based on plant-specific factors for destruction and utilization factors for an abatement technology and  $N_2O$  emission factor based on direct measurements. The Tier B methodology is a mass balance approach based on default  $N_2O$  emission factors by technology type. This methodology is consistent with Tier 2 methodology from IPCC.

#### **3.5** Technical Guidelines Voluntary Reporting of Greenhouse Gases (1605(b)) Program

The "A" rated approach involves continuous emission monitoring from confined and uncontrolled streams. If pollutant information is not available for uncontrolled streams, monitoring of confined streams only is acceptable. If continuous monitoring is not possible, emissions can be estimated using an emission factor based on direct, periodic measurements of plant emissions. Emission factors must account for emission rates and abatement system efficacy and frequency of use of abatement technologies. The "B" rated approach involves the use of default IPCC emission factors and production if plant-level emission information is not available. The "C" rated approach involves the use of estimates based on "other published default values."

# **3.6 Environment Canada's** *Technical Guidance on Reporting Greenhouse Gas Emissions*

The guidance for mandatory reporting in Canada primarily references the IPCC guidelines. There is no specific guidance on adipic acid production.

#### 4. Options Considered for Reporting Threshold

#### 4.1 Emissions Thresholds

Four reporting threshold levels were considered for the adipic acid manufacturing sector. The emission thresholds, 100,000, 25,000, 10,000, and 1,000 mtCO<sub>2</sub>e per year, were analyzed based on production data.

Table 2 provides the emission threshold analysis which estimated total emissions of 9,297,866 mtCO<sub>2</sub>e. This total was the sum of process emissions (5,921,434 mtCO<sub>2</sub>e) and combustion emissions (3,376,432 mtCO<sub>2</sub>e). All plants would be covered at all production-based thresholds. These results are summarized in Table 2.

Threshold	Process Emissions (Tons CO <sub>2</sub> e/yr)	CO₂ Emissions (Tons/yr)	Total National Emissions (Tons CO <sub>2</sub> e)	Number of Entities	Emissions Covered		Entities Covered	
Level (Metric Tons)					Tons CO₂e/yr	Percent	Number	Percent
100,000	5,921,434	3,376,432	9,297,866	4	9,297,866	100%	4	100%
25,000	5,921,434	3,376,432	9,297,866	4	9,297,866	100%	4	100%
10,000	5,921,434	3,376,432	9,297,866	4	9,297,866	100%	4	100%
1,000	5,921,434	3,376,432	9,297,866	4	9,297,866	100%	4	100%

Table 2. Threshold Analysis for Adipic Acid Production

# 4.2 Capacity Thresholds

Capacity-based thresholds are not presented here because each of the four plants exceeds highest emissions-based thresholds.

# 4.3 No Emissions Threshold

The no emissions threshold includes all adipic acid production facilities regardless of their emissions or capacity.

The option of requiring all adipic acid production facilities regardless of their emissions profile is similar to the emissions threshold option because at each emission threshold level all adipic acid facilities would be reporting.

# 5. Options for Monitoring Methods

# 5.1 Option 1: Simplified Emissions Calculation

A simplified emissions calculation option would use the default emission factors established by the Intergovernmental Panel on Climate Change (IPCC, 2006).

Two different approaches could be used.

<u>Approach 1.</u> Use the default emission factors and apply to the total facility production of adipic acid. When applying this approach, no abatement of  $N_2O$  emissions is considered.

N<sub>2</sub>O Emissions from Adipic Acid Production - IPCC (2006) TIER 1 Approach:

$$E_{N2O} = EF \ge AAP$$

Where

E <sub>N2O</sub>	=	N <sub>2</sub> O emissions, kg
EF	=	$N_2O$ emission factor (default), kg $N_2O/\text{metric}$ ton adipic acid produced
AAP	=	adipic acid production, metric tons

<u>Approach 2.</u> Use the default emission factors on a site-specific basis using the Tier 2 approach established by the IPCC. These emission factors are dependent on the type of abatement technology used. The amount of  $N_2O$  emissions are determined by multiplying the emission factor by the production level of adipic acid.

This is consistent with the Tier 2 methodology from IPCC, the Tier B methodology from The Climate Registry, and the "B" rated approach from USDOE.

# 5.2 Option 2: Stack Testing

Follow the Tier 3 approach established by IPCC using non-continuous monitoring. Use noncontinuous direct monitoring of  $N_2O$  emissions and determine the relationship between adipic acid production and the amount of  $N_2O$  emissions; i.e., develop a site-specific emissions factor. The site-specific emissions factor and production rate (activity level) is used to calculate the emissions. Annual testing of  $N_2O$  emissions would also be required to verify the emission factor over time. Testing should be conducted without using any  $NO_X$  or  $N_2O$  abatement technologies. Testing would also be required whenever significant process changes are made. This approach is consistent with the Tier 3 methodology from IPCC, the Tier A1 methodology from the Climate Registry, and Approach 1 from WBCSD.

Emissions would be calculated according to the following equations.

The average site-specific emission factor for the process would be calculated according to the following equation:

$$EF_{N2O} = \frac{\sum_{1}^{n} \frac{C_{N2O} * 1.14 \times 10^{-7} * Q}{P}}{n}$$

Where:

EF <sub>N2O</sub>	= acid pr	Average site-specific N <sub>2</sub> O emissions factor (lb N <sub>2</sub> O/ton adipic roduced, 100 percent acid basis)
C <sub>N2O</sub>	=	$N_2O$ concentration during performance test (ppm $N_2O$ )
$1.14 \times 10^{-7}$	=	Conversion factor (lb/dscf-ppm N <sub>2</sub> O)
Q	=	Volumetric flow rate of effluent gas (dscf/hr)
Р	= produc	Production rate during performance test (tons adipic acid ced per hour (100 percent acid basis))
n	=	Number of test runs

The  $N_2O$  emissions for the process are then calculated by multiplying the emission factor by the total production, according to following equation:

$$E_{N_2O} = \frac{EF_{N20} * P_a * (1 - DF_N) * AF_N}{2205}$$

Where:

E <sub>N2O</sub>	=	N <sub>2</sub> O mass emissions per year (metric tons of N <sub>2</sub> O)
EF <sub>N20</sub>	=	Site-specific N <sub>2</sub> O emission factor (lb N <sub>2</sub> O/ton acid produced, 100 percent acid basis)
Pa	=	Total production for the year (ton acid produced, 100 percent acid basis)
DF <sub>N</sub>	=	Destruction factor of $N_2O$ abatement technology (percent of $N_2O$ removed from air stream)
AF <sub>N</sub>	=	Abatement factor of $N_2O$ abatement technology (percent of year that abatement technology was used)
2205	=	Conversion factor (lb/metric ton).

For direct measurement using stack testing, sampling equipment would be periodically brought to the site and installed temporarily in the stack to withdraw a sample of the stack gas and measure the flow rate of the stack gas. Similar to CEMS, for stack testing the emissions are calculated from the concentration of GHGs in the stack gas and the flow rate of the stack gas. The difference between stack testing and continuous monitoring is that the CEMS data provide a continuous measurement of the emissions, while a stack test provides a periodic measurement of the emissions.

#### 5.3 Option 3: Direct Measurement

For industrial source categories for which the process emissions and/or combustion GHG emissions are contained within a stack or vent, direct measurement constitutes either measurements of the GHG concentration in the stack gas and the flow rate of the stack gas using a CEMS, or periodic measurement of the GHG concentration in the stack gas and the flow rate of the stack gas using periodic stack testing. Under either a CEMS approach or a stack testing approach, the emissions measurement data would be reported annually.

Elements of a CEMS include a platform and sample probe within the stack to withdraw a sample of the stack gas, an analyzer to measure the concentration of the GHG (e.g.,  $CO_2$ ) in the stack gas, and a flow meter within the stack to measure the flow rate of the stack gas. The emissions are calculated from the concentration of GHGs in the stack gas and the flow rate of the stack gas. A CEMS continuously withdraws and analyzes a sample of the stack gas and continuously measures the GHG concentration and flow rate of the stack gas.

Follow the Tier 3 approach established by IPCC using continuous monitoring. Use CEMS to directly measure  $N_2O$  concentration and flow rate to directly determine  $N_2O$  emissions. This option is available but is not currently being used in the adipic acid production sector. This is consistent with the Tier 3 approach established by IPCC, the Tier A1 methodology from the Climate Registry, Approach 1 from WBCSD, and the "A" rated approach from USDOE.

#### 6. Options for Estimating Missing Data

Options and considerations for missing data vary will vary depending on the proposed monitoring method. Each option would require a complete record of all measured parameters as well as parameters determined from company records that are used in the GHG emissions calculations (e.g., carbon contents, monthly fuel consumption, etc.).

#### 6.1 Procedures for Option 1: Simplified Emissions Calculation

If facility-specific production data is missing for one year, an average value using the production data from the year prior and the year after the missing year may be calculated. Default emission factors are readily available through IPCC guidelines (IPCC 2006).

In general, adipic acid default emission factors are fairly certain because they are derived from the stoichiometry of an intended chemical reaction (nitric acid oxidation) and N<sub>2</sub>O-specific abatement systems. The uncertainty in the emission factor for adipic acid represents variability in N<sub>2</sub>O generation due to differences in the composition of the cyclohexanone and cyclohexanol feedstock (i.e. ketone and alcohol) that are used by different manufacturers. Higher ketone content results in increased N<sub>2</sub>O generation, whereas higher alcohol content results in less N<sub>2</sub>O generation (Reimer 1999). An individual plant should be able to determine the production of N<sub>2</sub>O (based on HNO<sub>3</sub> consumption) within 1%. The uncertainty associated with adipic acid production may be more significant when converted into N<sub>2</sub>O emissions. A properly maintained and calibrated monitoring system can determine emissions to within ±5% at the 95% confidence level (IPCC 2006).

## 6.2 **Procedures for Option 2: Stack Testing**

For process sources that use the stack testing approach, the following data are needed on a monthly basis: adipic acid production rate, adipic acid production capacity, number of operating hours, emission rate factor, and the type of abatement technology used and its utilization factor. In general, the substitute data value would be the arithmetic average of the quality-assured values of that same parameter immediately preceding and immediately following the missing data incident. If no quality-assured data are available prior to the missing data incident, the substitute data value would be the first quality-assured value obtained after the missing data period. For missing oil or gas flow rates, the rule would require using the standard missing data procedures in section 2.4.2 of appendix D to part 75.

#### 6.3 Procedures for Option 3: Direct Measurement

# 6.3.1 Continuous Emission Monitoring Data CEMS

CEMS for monitoring  $N_2O$  emissions are not currently in use in the industry and there is no existing EPA method for certifying  $N_2O$  CEMS. In general, the missing data procedures for  $CO_2$  CEMS, listed below would be adequate.

For options involving direct measurement of  $CO_2$  flow rates or direct measurement of  $CO_2$  emissions using CEMS, Part 75 establishes procedures for management of missing data. Procedures for management of missing data are described in Part 75.35(a), (b), and (d). These procedures for managing missing data for  $CO_2$  CEMS would also apply to missing data for  $N_2O$  CEMS. In general, missing data from operation of the CEMS may be replaced with substitute data to determine the  $N_2O$  flow rates or  $N_2O$  emissions during the period in which CEMS data are missing.

Under Part 75.35(a), the owner or operator of a unit with a  $CO_2$  continuous emission monitoring system for determining  $CO_2$  mass emissions in accordance with Part 75.10 (or an  $O_2$  monitor that is used to determine  $CO_2$  concentration in accordance with appendix F to this part) shall substitute for missing  $CO_2$  pollutant concentration data using the procedures of paragraphs (b) and (d) of this section. Subpart (b) covers operation of the system during the first 720 quality-assured operation hours for the CEMS. Subpart (d) covers operation of the system after the first 720 quality-assured operating hours are completed. These procedures would generally apply to N<sub>2</sub>O CEMS as well as  $CO_2$  CEMS.

Under Part 75.35(b), during the first 720 quality assured monitor operating hours following initial certification at a particular unit or stack location (i.e., the date and time at which quality assured data begins to be recorded by a CEMS at that location), or (when implementing these procedures for a previously certified  $CO_2$  monitoring system) during the 720 quality assured monitor operating hours preceding implementation of the standard missing data procedures in paragraph (d) of this section, the owner or operator shall provide substitute  $CO_2$  pollutant concentration data or substitute  $CO_2$  data for heat input determination, as applicable, according to the procedures in Part 75.31(b). [Note that for CEMS that are measuring process  $N_2O$  emissions the term "heat input determination" may be replaced by the term "raw material input determination."]

Under Part 75.35(d), upon completion of 720 quality assured monitor operating hours using the initial missing data procedures of Part 75.31(b), the owner or operator shall provide substitute data for  $CO_2$  concentration or substitute  $CO_2$  data for heat input determination, as applicable, in accordance with the procedures in Part 75.33(b) except that the term "  $CO_2$  concentration" shall apply rather than "SO<sub>2</sub> concentration," the term "  $CO_2$  pollutant concentration monitor" or "  $CO_2$  diluent monitor" shall apply rather than "SO<sub>2</sub> concentration, as defined in section 2.1.3.1 of appendix A to this part" shall apply, rather than "maximum potential SO<sub>2</sub> concentration." [Note that for CEMS that are measuring process N<sub>2</sub>O emissions the term "heat input determination" may be replaced by the term "raw material input determination."]

CEMS can also be used to measure the stationary combustion emissions of  $CO_2$  from boilers at the adipic acid plants. Note that the CEMS procedures used for the N<sub>2</sub>O CEMS system for process emissions described above would be the same as the CEMS procedures used for the  $CO_2$  CEMS for stationary combustion emissions.

# 6.3.2 Stack Testing Data

For options involving direct measurement of flow rates or emissions using stack testing, "missing data" is not generally anticipated. Stack testing conducted for the purposes of compliance determination is subject to quality assurance guidelines and data quality objectives established by the U.S. EPA, including the Clean Air Act National Stack Testing Guidance published in 2005 (USEPA 2005). The 2005 EPA Guidance Document indicates that stack tests should be conducted in accordance with a pre-approved site-specific test plan to ensure that a complete and representative test is conducted. Results of stack tests that do not meet pre-established quality assurance guidelines and data quality objectives would generally not be acceptable for use in emissions reporting, and any such stack test would need to be reconducted to obtain acceptable data.

The U.S. EPA regulations for performance testing under 40 CFR § 63.7(c)(2)(i) state that before conducting a required performance test, the owner/operator is required to develop a sitespecific test plan and, if required, submit the test plan for approval. The test plan is required to include "a test program summary, the test schedule, data quality objectives, and both an internal and external quality assurance (QA) program" to be applied to the stack test. Data quality objectives are defined under 40 CFR § 63.7(c)(2)(i) as "the pre-test expectations of precision, accuracy, and completeness of data." Under 40 CFR § 63.7(c)(2)(ii), the internal QA program is required to include, "at a minimum, the activities planned by routine operators and analysts to provide an assessment of test data precision; an example of internal QA is the sampling and analysis of replicate samples." Under 40 CFR § 63.7(c)(2)(iii) the external QA program is required to include, "at a minimum, application of plans for a test method performance audit (PA) during the performance test." In addition, according to the 2005 Guidance Document, a site-specific test plan should generally include chain of custody documentation from sample collection through laboratory analysis including transport, and should recognize special sample transport, handling, and analysis instructions necessary for each set of field samples (USEPA 2005).

The U.S. EPA anticipates that test plans for stack tests that are expected to be used to obtain data for the purposes of emissions reporting would be made available to EPA prior to the stack test and that the results of the stack test would be reviewed against the test plan prior to the data being deemed acceptable for the purposes of emissions reporting.

# 7. QA/QC Requirements

Facilities should conduct quality assurance and quality control of the production and consumption data, and emission estimates reported. Facilities are encouraged to prepare an indepth quality assurance and quality control plan which would include checks on production data, the carbon content information received from the lab analysis, and calculations performed to estimate GHG emissions.

#### 7.1 Stationary Emissions

Facilities should follow the guidelines given for stationary combustion in EPA-HQ-OAR-2008-0508-004.

#### 7.2 Process Emissions

Options and considerations for QA/QC will vary depending on the proposed monitoring method. Each option would require unique QA/QC measures appropriate to the particular methodology employed to ensure proper emission monitoring and reporting.

#### 7.2.1 Continuous Emission Monitoring System (CEMS)

For units using CEMS to measure  $N_2O$  flow rates or  $N_2O$  emissions, the equipment should be tested for accuracy and calibrated as necessary by a certified third party vendor. These procedures should be consistent in stringency and data reporting and documentation adequacy with the QA/QC procedures for CEMS described in Part 75 of the Acid Rain Program.

# 7.2.2 Stack Test Data

U.S. EPA regulations for performance testing under 40 CFR § 63.7(c)(2)(i) state that before conducting a required performance test, the owner/operator is required to develop a sitespecific test plan and, if required, submit the test plan for approval. The test plan is required to include "a test program summary, the test schedule, data quality objectives, and both an internal and external quality assurance (QA) program" to be applied to the stack test. Data quality objectives are defined under 40 CFR § 63.7(c)(2)(i) as "the pre-test expectations of precision, accuracy, and completeness of data." Under 40 CFR § 63.7(c)(2)(ii), the internal QA program is required to include, "at a minimum, the activities planned by routine operators and analysts to provide an assessment of test data precision; an example of internal QA is the sampling and analysis of replicate samples." Under 40 CFR § 63.7(c)(2)(iii) the external QA program is required to include, "at a minimum, application of plans for a test method performance audit (PA) during the performance test." In addition, according to the 2005 Guidance Document, a site-specific test plan should generally include chain of custody documentation from sample collection through laboratory analysis including transport, and should recognize special sample transport, handling, and analysis instructions necessary for each set of field samples (US EPA 2005).

#### 7.3 Data Management

Data management procedures should be included in the QA/QC Plan. Elements of the data management procedures plan are as follows:

- Check for temporal consistency in production data and emission estimates. If outliers exist, they should be explained by changes in the facility's operations or other factors. A monitoring error is probable if differences cannot be explained by changes in activity levels, changes concerning fuels or input material, or changes concerning the emitting process (e.g. energy efficiency improvements) (European Commission 2007).
- Determine the "reasonableness" of the emission estimate by comparing it to previous year's estimates and relative to national emission estimate for the industry:
  - Comparison of data on fuel or input material consumed by specific sources with fuel or input material purchasing data and data on stock changes,
  - Comparison of fuel or input material consumption data with fuel or input material purchasing data and data on stock changes,
  - Comparison of emission factors that have been calculated or obtained from the fuel or input material supplier, to national or international reference emission factors of comparable fuels or input materials
  - Comparison of emission factors based on fuel analyses to national or international reference emission factors of comparable fuels, or input materials,
  - o Comparison of measured and calculated emissions (European Commission 2007).
- Maintain data documentation, including comprehensive documentation of data received through personal communication.
- Check that changes in data or methodology are documented.

#### 8. Types of Emission Information to be Reported

#### 8.1 Types of Emissions to be Reported

Adipic acid facilities should report both process ( $N_2O$ ) and combustion related ( $CO_2$ ,  $CH_4$ , and  $N_2O$ ) greenhouse gas emissions. The data to be reported may very depending on monitoring options selected. However, an adipic acid facility should report the number of adipic acid production lines, annual adipic acid production, annual adipic acid production capacity, electricity usage (kilowatt-hours), emission factor(s) used, abatement technology used (if applicable), abatement utilization factor (percent of time that abatement system is operating), abatement technology efficiency, and annual operating hours. For reporting options for stationary combustion refer to EPA-HQ-OAR-2008-0508-004.

# 8.2 Other Information to be Reported

Other information to be reported will vary depending on selected option. For verification and checks of reasonableness facilities should also report their annual adipic acid production, abatement system uptime and percent of total operational hours/year (i.e. the fraction of operational hours where the abatement system is in use at the plant); abatement system downtime and percent of total operational hours per year (i.e. the fraction of operational hours where the abatement system is NOT in use and the abatement system is bypassed); and, the destruction factor for each relevant abatement system.

# 8.3 Additional Data to be Retained Onsite

Facilities should be required to retain data concerning monitoring of GHG emissions onsite for a period of at least five years from the reporting year. For CEMS these data would include CEMS monitoring system data including continuous-monitored GHG concentrations and stack gas flow rates, calibration and quality assurance records. For stack testing these data would include stack test reports and associated sampling and chemical analytical data for the stack test. Process data including process raw material and product feed rates and carbon contents should also be retained on site for a period of at least five years from the reporting year. EPA could use such data to conduct trend analyses and potentially to develop process or activity-specific emission factors for the process.

#### 9. References

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