

[Note: with the publication of the Fifth Edition of AP-42, the Chapter and Section number for Nitric Acid was changed to 8.8.]

BACKGROUND REPORT

AP-42 SECTION 5.9

NITRIC ACID

Prepared for

**U.S. Environmental Protection Agency
OAQPS/TSD/EIB
Research Triangle Park, NC 27711**

1-96

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919/941-0333

1-96
AP-42 Background Report

TECHNICAL SUPPORT DIVISION

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Air Quality Planning and Standards
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1.0 INTRODUCTION

The document "Compilation of Air Pollutant Emission Factors" (AP-42) has been published by the U.S. Environmental Protection Agency (EPA) since 1972. Supplements to AP-42 have been routinely published to add new emission source categories and to update existing emission factors. AP-42 is routinely updated by the EPA to respond to new emission factor needs of the EPA, State and local air pollution control programs, and industry.

An emission factor relates the quantity (weight) of pollutants emitted to a unit of activity of the source. The uses for the emission factors reported in AP-42 include:

1. Estimates of area-wide emissions;
2. Emission estimates for a specific facility; and
3. Evaluation of emissions relative to ambient air quality.

The purpose of this report is to provide background information from process information obtained from industry comment and 14 test reports to support revision of the process description and/or emission factors for nitric acid plants.

Including the introduction (Chapter 1), this report contains four chapters. Chapter 2 gives a description of the nitric acid industry. It includes a characterization of the industry, an overview of the different process types, a description of emissions, and a description of the technology used to control emissions resulting from the nitric acid process. A review of specific data sets which contributed any additional information for the revised AP-42 section is also presented.

Chapter 3 is a review of emissions data collection and analysis procedures. It describes the literature search, the screening of emission data reports, and the quality rating system for both emission data and emission factors. Chapter 4 details criteria and noncriteria pollutant emission factor development. It includes the review of specific data sets and the results of data analysis. A data gap analysis for the pollutant emission factor development process is also presented. Appendix A presents a copy of the revised AP-42 Section 5.9.

2.0 INDUSTRY DESCRIPTION

2.1 GENERAL^{1,2}

In 1991, there were approximately 65 nitric acid (HNO_3) manufacturing plants in the U.S. with a total capacity of 10 million megagrams (11 million tons) of acid per year.² The plants range in size from 5,400 to 635,000 megagrams (6,000 to 700,000 tons) per year. About 70 percent of the nitric acid produced is consumed as an intermediate in the manufacture of ammonium nitrate (NH_4NO_3), which is primarily used in fertilizers.¹ The majority of the nitric acid plants are located in agricultural regions such as the Midwest, South Central, and Gulf States in order to accommodate the high volume of fertilizer use. Another 5 to 10 percent of the nitric acid produced is used for organic oxidation in adipic acid manufacturing. Nitric acid is also used in organic oxidation to manufacture terephthalic acid and other organic compounds. Explosive manufacturing utilizes nitric acid for organic nitrations. Nitric acid nitrations are used in producing nitrobenzene, dinitrotoluenes, and other chemical intermediates.¹ Other end uses of nitric acid are gold and silver separation, military munitions, steel and brass pickling, photoengraving, and acidulation of phosphate rock.

2.2 PROCESS DESCRIPTION^{1,3,4}

Nitric acid is produced by two methods. The first method utilizes oxidation, condensation, and absorption to produce a weak nitric acid. Weak nitric acid can have concentrations ranging from 30 to 70 percent nitric acid.³ The second method combines dehydrating, bleaching, condensing, and absorption to produce high strength nitric acid from weak nitric acid. High strength nitric acid generally contains more than 90 percent nitric acid.³ The following text provides more specific details for each of these processes.

Weak Nitric Acid Production

Nearly all the nitric acid produced in the U.S. is manufactured by the high temperature catalytic oxidation of ammonia as shown schematically in Figure 2.2-1.

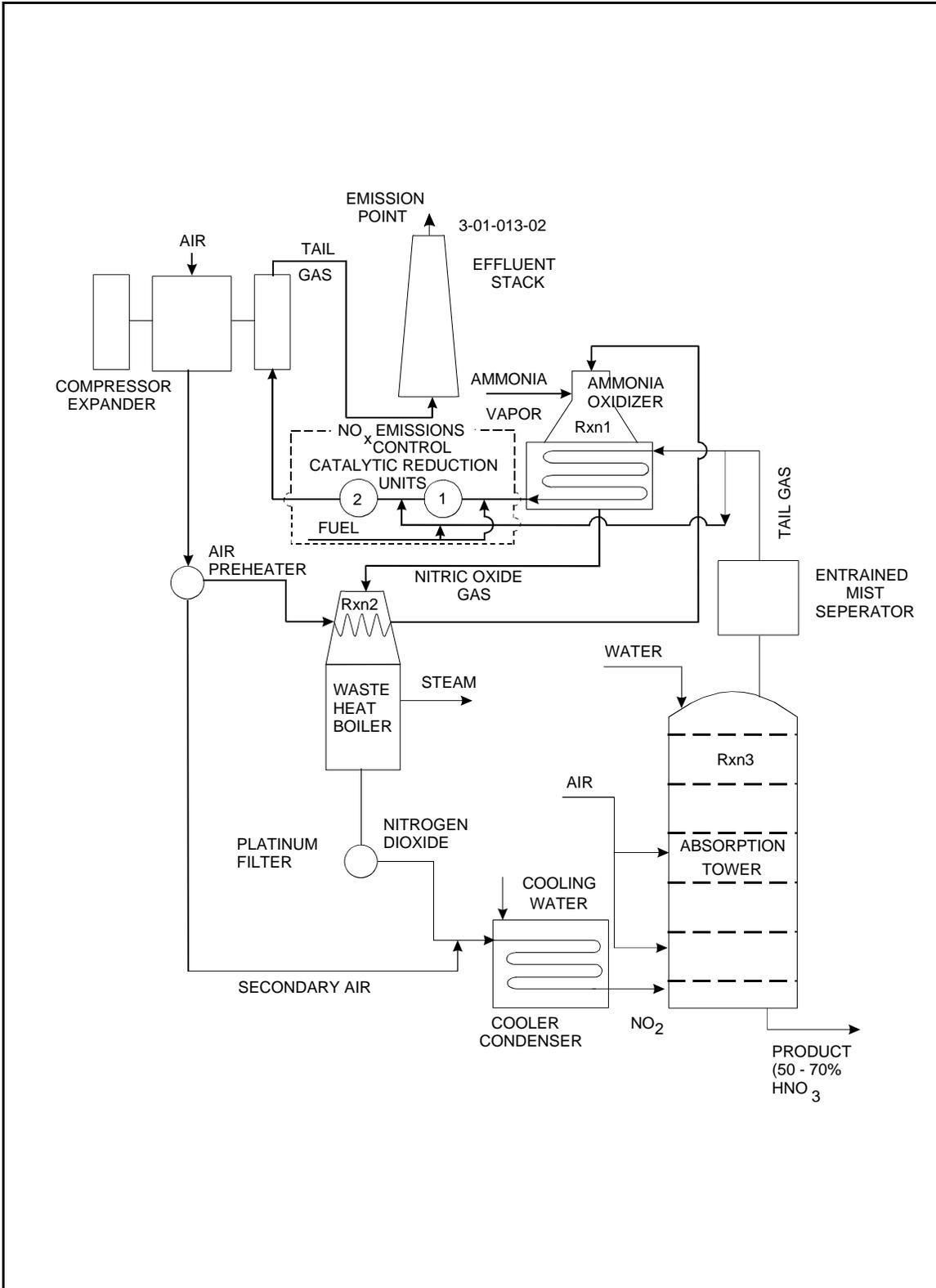


Figure 2.2-1 Flow diagram of typical nitric acid plant using single-pressure process (high-strength acid unit not shown).

This process typically consists of three steps: 1. ammonia oxidation, 2. nitric oxide oxidation, and 3. absorption. Each step corresponds to a distinct chemical reaction.

Ammonia Oxidation. First, a 1:9 ammonia/air mixture is oxidized at a temperature of 750 to 800°C (1380 to 1470°F) as it passes through a catalytic converter, according to the following reaction:⁴



The most commonly used catalyst is made of 90 percent platinum and 10 percent rhodium gauze constructed from squares of fine wire. Under these conditions the oxidation of ammonia to nitric oxide proceeds in an exothermic reaction with a range of 93 to 98 percent yield.¹ Higher catalyst temperatures increase reaction selectivity toward nitric oxide (NO) production. Lower catalyst temperatures tend to be more selective toward nitrogen (N₂) and nitrous oxide (N₂O).¹ Nitric oxide is considered to be a criteria pollutant and nitrous oxide is known to be a global warming gas. The nitrogen dioxide/dimer mixture then passes through a waste heat boiler and a platinum filter.

Nitric Oxide Oxidation. The nitric oxide formed during the ammonia oxidation is oxidized in another operation. The process stream is passed through a cooler/condenser and cooled to 38°C (100°F) or less at pressures up to 800 kPa (116 psia). The nitric oxide reacts noncatalytically with residual oxygen to form nitrogen dioxide and its liquid dimer, nitrogen tetroxide:¹



This slow, homogeneous reaction is temperature and pressure dependent. Operating at low temperatures and high pressures promotes maximum production of NO₂ within a minimum reaction time.

Absorption. The final step introduces the nitrogen dioxide/dimer mixture into an absorption process after being cooled. The mixture is pumped into the bottom of the absorption tower, while liquid dinitrogen tetroxide is added at a higher point. Deionized water enters the top of the column. Both liquids flow countercurrent to the dioxide/dimer gas mixture. Oxidation takes place in the free space between the trays, while absorption occurs on the trays. The absorption trays are usually sieve or bubble cap trays. The exothermic reaction occurs as follows:



A secondary air stream is introduced into the column to re-oxidize the NO which is formed in Reaction 3. This secondary air also removes NO₂ from the product acid. An aqueous solution of 55 to 65 percent (typically) nitric acid is withdrawn from the bottom of the tower.¹ The acid concentration can vary from 30 to 70 percent nitric acid.³ The acid concentration depends upon the temperature, pressure, number of absorption stages, and concentration of nitrogen oxides entering the absorber.

There are two basic types of systems used to produce weak nitric acid: 1) single-stage pressure process, and 2) dual-stage pressure process. In the past, nitric acid plants have been operated at a single pressure, ranging from atmospheric pressure to 1400 kPa (14.7 to 203 psia).¹ However, since Reaction 1 is favored by low pressures and Reactions 2 and 3 are favored by higher pressures, newer plants tend to operate a dual-stage pressure system, incorporating a compressor between the ammonia oxidizer and the condenser. The oxidation reaction is carried out at pressures from slightly negative to about 400 kPa (58 psia), and the absorption reactions are carried out at 800 to 1,400 kPa (116 to 203 psia).¹

In the dual-stage pressure system, the nitric acid formed in the absorber (bottoms) is usually sent to an external bleacher where air is used to remove (bleach) any dissolved oxides of nitrogen. The bleacher gases are then compressed and passed through the absorber. The absorber tail gas (distillate) is sent to an entrainment separator for acid mist removal. Next, the tail gas is reheated in the ammonia oxidation heat exchanger to approximately 200°C (392°F). The final step expands the gas in the power-recovery turbine. The thermal energy produced in this turbine can be used to drive the compressor.¹

High Strength Nitric Acid Production

High strength nitric acid (98 to 99 percent concentration) can be obtained by concentrating weak nitric acid (30 to 70 percent concentration) using extractive distillation.¹ The weak nitric acid cannot be concentrated by simple fractional distillation. The distillation must be carried out in the presence of a dehydrating agent. Concentrated sulfuric acid (typically 60 percent sulfuric acid) is most commonly used for this purpose. The nitric acid concentration process consists of feeding strong sulfuric acid and 55 to 65 percent nitric acid into the top of a packed dehydrating column at approximately atmospheric pressure. The acid mixture flows downward,

countercurrent to ascending vapors. Concentrated nitric acid leaves the top of the column as 99 percent vapor, containing a small amount of NO_2 and O_2 resulting from dissociation of nitric acid.¹ The concentrated acid vapor leaves the column and goes to a bleacher and a countercurrent condenser system to effect the condensation of strong nitric acid and the separation of oxygen and nitrogen oxide by-products. These by-products then flow to an absorption column where the nitric oxide mixes with auxiliary air to form NO_2 , which is recovered as weak nitric acid. Inert and unreacted gases are vented to the atmosphere from the top of the absorption column. Emissions from this process are relatively small. A small absorber can be used to recover NO_2 . Figure 2.2-2 presents a flow diagram of high strength nitric acid production from weak nitric acid.

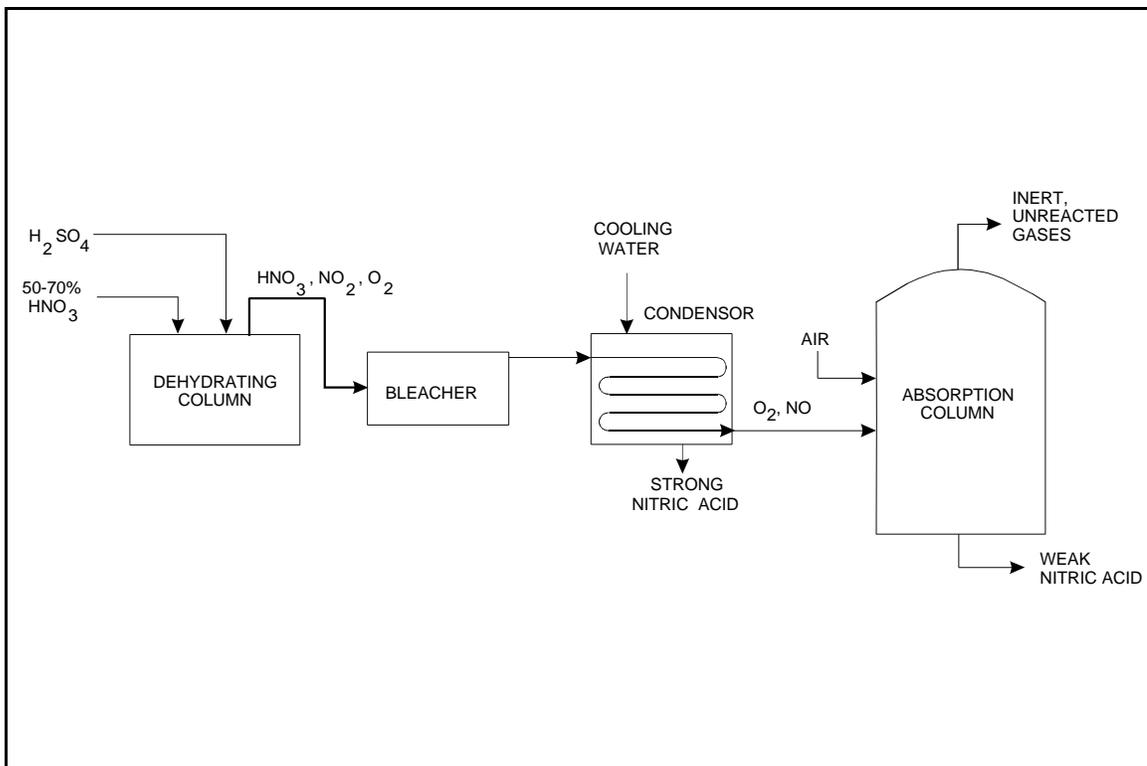


Figure 2.2-2. Flow diagram of high-strength nitric acid production from weak nitric acid.

2.3 EMISSIONS AND CONTROLS^{3,4,5}

Emissions from nitric acid manufacture consist primarily of NO and NO_2 (which account for visible emissions), and trace amounts of HNO_3 mist and NH_3 . By far, the major source of nitrogen oxides is the tail gas from the acid absorption tower. In general, the quantity of NO_x

emissions are directly related to the kinetics of the nitric acid formation reaction and absorption tower design. NO_x emissions can increase when there is (1) insufficient air supply to the oxidizer and absorber, (2) low pressure, especially in the absorber, (3) high temperatures in the cooler/condenser and absorber, (4) production of an excessively high-strength product acid, (5) operation at high throughput rates, and (6) faulty equipment such as compressors or pumps which lead to lower pressures, leaks, and reduced plant efficiency.⁴

The two most common techniques used to control absorption tower tail gas emissions are extended absorption and catalytic reduction. Extended absorption reduces nitrogen oxide emissions by increasing the efficiency of the existing process absorption tower or incorporating an additional absorption tower. An efficiency increase is achieved by increasing the number of absorber trays, operating the absorber at higher pressures, or cooling the weak acid liquid in the absorber. The existing tower can also be replaced with a single tower of a larger diameter and/or additional trays (see reference 5 for the relevant equations).

In the catalytic reduction process (often termed catalytic oxidation or incineration), tail gases from the absorption tower are heated to ignition temperature, mixed with fuel (natural gas, hydrogen, propane, butane, naphtha, carbon monoxide, or ammonia) and passed over a catalyst bed. In the presence of the catalyst, the fuels are oxidized and the nitrogen oxides are reduced to N_2 . The extent of reduction of NO_2 and NO to N_2 is a function of plant design, fuel type operating temperature and pressure, space velocity through the reduction catalytic reactor, type of catalyst, and reactant concentration. Catalytic reduction can be used in conjunction with other NO_x emission controls. Other advantages include the capability to operate at any pressure and the option of heat recovery to provide energy for process compression as well as extra steam. Catalytic reduction can achieve greater NO_x reduction than extended absorption. However, high fuel costs have caused a decline in its use.

Two seldom used alternative control devices for absorber tail gas are molecular sieves and wet scrubbers. In the molecular sieve technique, tail gas is contacted with an active molecular sieve which catalytically oxidizes NO to NO_2 and selectively adsorbs the NO_2 . The NO_2 is then thermally stripped from the molecular sieve and returned to the absorber. The molecular sieve technique has successfully controlled NO_x emissions in existing plants. However, many new plants do not install this method of control. Its implementation incurs high capital and energy costs. The molecular sieve technique is a cyclic system, whereas most new nitric acid plants are continuous systems.

Wet scrubbers use an aqueous solution of alkali hydroxides or carbonates, ammonia, urea, potassium permanganate, or caustic chemicals to "scrub" NO_x from the absorber tail gas. The NO and NO_2 are absorbed and recovered as nitrate or nitrate salts. When caustic chemicals are used, the wet scrubber is referred to as a caustic scrubber. Some of the caustic chemicals used are solutions of sodium hydroxide, sodium carbonate, or other strong bases that will absorb NO_x in the form of nitrate or nitrate salts. Although caustic scrubbing can be an effective control device, it is often not used due to its incurred high costs and the necessity to treat its spent scrubbing solution.

Comparatively small amounts of nitrogen oxides are also lost from acid concentrating plants. These losses (mostly NO_2) are from the condenser system, but the emissions are small enough to be controlled easily by inexpensive absorbers.

Acid mist emissions do not occur from the tail gas of a properly operated plant. The small amounts that may be present in the absorber exit gas streams are removed by a separator or collector prior to entering the catalytic reduction unit or expander.

The acid production system and storage tanks are the only significant sources of visible emissions at most nitric acid plants. Emissions from acid storage tanks may occur during tank filling.

The emission factors vary considerably with the type of control employed and with process conditions. For comparison purposes, the EPA New Source Performance Standard on nitrogen emissions expressed as NO_2 for both new and modified plants is 1.5 kilograms of NO_2 emitted per megagram (3.0 lb/ton) of 100 percent nitric acid produced.³

2.4 REVIEW OF SPECIFIC REFERENCES

Pacific Environmental Services (PES) contacted the following sources to obtain the most up-to-date information on process descriptions and emissions for this industry.

- 1) Alabama Air Division, ADEM, Montgomery, Alabama.
- 2) Arkansas Department of Pollution Control and Ecology Division of Air Pollution, Little Rock, Arkansas.
- 3) Chemical Manufacturers Association.
- 4) Florida Department of Environmental Regulation - Bureau of Air Quality Management, Tallahassee, Florida.

- 5) Georgia Department of Natural Resources - Environmental Protection Division, Atlanta, Georgia.
- 6) J.R. Simplot Company, Pocatello, Idaho.
- 7) Kansas Department of Health and Environment - Bureau of Air Quality, Topeka, Kansas.
- 8) Michigan Department of Natural Resources - Air Pollution Control Division, Lansing, Michigan.
- 9) Missouri Department of Natural Resources - Division of Environmental Quality, Jefferson City, Missouri.
- 10) Monsanto Company, Pensacola, Florida.
- 11) Ohio Environmental Protection Agency.
- 12) Pennsylvania Department of Environmental Resources, Harrisburg, Pennsylvania.

Responses were received from the J.R. Simplot Company and the Monsanto Company. No responses were received from the remaining sources. PES was unable to incorporate the information received from these two sources into the AP-42 section revision because the data contained in their reports are not complete. Additional details on the exclusion of these reports can be found in Section 4.1 of this report.

Reference 1: Alternative Control Techniques Document: Nitric and Adipic Acid Manufacturing Plants.

This reference provided the nitric acid process description. It details ammonia oxidation, nitric oxide oxidation, and absorption. Percent yields, temperatures, and pressures are a few of the details given by this reference. The dual-stage pressure system is also detailed.

Reference 2: North American Fertilizer Capacity Data.

This reference provided production capacities for the nitric acid plants located in the United States as of 1991. There were approximately 65 nitric acid manufacturing plants with a total capacity of 10 million megagrams (11 million tons) of acid per year.

Reference 3: Code of Federal Regulations.

This reference provided information on the industrial manufacture of nitric acid. High strength nitric acid (95 to 99 percent concentration) can be obtained by concentrating weak nitric acid (30 to 70 percent concentration). The New Source Performance Standard on nitric acid

plant emissions was also located in the CFR. For both new and modified plants, approximately 1.5 kilograms of NO₂ is emitted per megagram (3.0 pounds/ton) of 100 percent nitric acid produced.

Reference 4: A Review of Standards of Performance for New Stationary Sources—Nitric Acid Plants.

This reference provided details on the control equipment used in nitric acid plants. This control equipment consists of extended absorption and catalytic reduction. This reference also provided information for weak nitric acid production; specifically for the first step of ammonia oxidation. There were several small differences between References 1 and 4; the values presented in this report are taken from the most current reference, Reference 1.

Reference 5: Unit Operations of Chemical Engineering.

This reference provides the equations necessary to evaluate the efficiency of increasing the number of absorber trays in an absorption tower, operating the absorber at higher pressures, or cooling the weak acid liquid in the absorber. This reference also gives equations to determine whether an existing tower should be replaced with a single tower of a larger diameter or if adding additional trays would be sufficient or whether utilizing a combination of these options would be more efficient.

2.5 REFERENCES FOR CHAPTER 2

1. Alternative Control Techniques Document: Nitric and Adipic Acid Manufacturing Plants. EPA-450/3-91-026. U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards, Research Triangle Park, NC., December 1991.
2. North American Fertilizer Capacity Data, Tennessee Valley Authority, Muscle Shoals, AL, December 1991.
3. Code of Federal Regulations. "Standards of Performance for Nitric Acid Plants," 40 CFR 60., Subpart G, July 1, 1989.
4. Marvin Drabkin, A Review of Standards of Performance for New Stationary Sources—Nitric Acid Plants, EPA-450/3-79-013, U.S. Environmental Protection Agency, Research Triangle Park, NC, March 1979.
5. McCabe and Smith, Unit Operations of Chemical Engineering, 3rd Edition, McGraw-Hill, Inc. 1976, p. 701.

3.0 EMISSION DATA REVIEW AND ANALYSIS PROCEDURES

3.1 LITERATURE SEARCH AND SCREENING SOURCE TESTS

The first step in the investigative process involved a search of available literature relating to criteria and noncriteria pollutant emissions associated with nitric acid plants. This search included, but was not limited to, the following references:

- 1) AP-42 background files maintained by the Emission Factor and Methodologies Section provided the three references from the 1980 AP-42 and several source tests.
- 2) "Locating and Estimating" (L&E) reports (as applicable) published by the Emission Factor and Methodologies Section. Nitrogen oxides are the primary emission from nitric acid production, and there are no L&E reports based on nitrogen oxides (NO_x).
- 3) Handbook of Emission Factors, Parts I and II, Ministry of Housing, Physical Planning and Environment, The Netherlands, 1980/1983 provided NO_x emission factors for uncontrolled processes, catalytic reduction, and extended absorption of the tail gas from nitric acid processes. For additional details, see Chapter 4.1 of this report under the discussion for Reference 15.

To reduce the amount of literature collected to a final group of references pertinent to this report, the following general criteria were used:

1. Emissions data must be from a primary reference; i.e., the document must constitute the original source of test data. For example, a technical paper was not included if the original study was contained in the previous document.
2. The referenced study must contain test results based on more than one test run.
3. The report must contain sufficient data to evaluate the testing procedures and source operating conditions (e.g., one-page reports were generally rejected).

If no primary data were found and the previous update utilized secondary data, secondary data were still used and the Emission Factor Rating lowered, if needed. A final set of reference materials was compiled after a thorough review of the pertinent reports, documents, and information according to these criteria. The final set of reference materials is given in Chapter 4.5.

3.2 EMISSION DATA QUALITY RATING SYSTEM

As part of Pacific Environmental Services' analysis of the emission data, the quantity and quality of the information contained in the final set of reference documents were evaluated. The following data were always excluded from consideration:

1. Test series averages reported in units that cannot be converted to the selected reporting units;
2. Test series representing incompatible test methods (e.g., comparison of the EPA Method 5 front-half with the EPA Method 5 front- and back-half);
3. Test series of controlled emissions for which the control device is not specified;
4. Test series in which the source process is not clearly identified and described; and
5. Test series in which it is not clear whether the emissions were measured before or after the control device.

Data sets that were not excluded were assigned a quality rating. The rating system used was that specified by the OAQPS for the preparation of AP-42 sections. The data were rated as follows:

A Rating

Multiple tests performed on the same source using sound methodology and reported in enough detail for adequate validation. These tests do not necessarily conform to the methodology specified in either the inhalable particulate (IP) protocol documents or the EPA reference test methods, although these documents and methods were certainly used as a guide for the methodology actually used.

B Rating

Tests that were performed by a generally sound methodology but lack enough detail for adequate validation.

C Rating

Tests that were based on an untested or new methodology or that lacked a significant amount of background data.

D Rating

Tests that were based on a generally unacceptable method but may provide an order-of-magnitude value for the source.

The following criteria were used to evaluate source test reports for sound methodology and adequate detail:

1. Source operation. The manner in which the source was operated is well documented in the report. The source was operating within typical parameters during the test.
2. Sampling procedures. The sampling procedures conformed to a generally acceptable methodology. If actual procedures deviated from accepted methods, the deviations are well documented. When this occurred, an evaluation was made of the extent such alternative procedures could influence the test results.
3. Sampling and process data. Adequate sampling and process data are documented in the report. Many variations can occur unnoticed and without warning during testing. Such variations can induce wide deviations in sampling results. If a large spread between test results cannot be explained by information contained in the test report, the data are suspect and were given a lower rating.
4. Analysis and calculations. The test reports contain original raw data sheets. The nomenclature and equations used were compared to those (if any) specified by the EPA to establish equivalency. The depth of review of the calculations was dictated by the reviewer's confidence in the ability and conscientiousness of the tester, which in turn was based on factors such as consistency of results and completeness of other areas of the test report.

3.3 EMISSION FACTOR QUALITY RATING SYSTEM

The quality of the emission factors developed from analysis of the test data was rated utilizing the following general criteria:

A (Excellent)

Developed only from A-rated test data taken from many randomly chosen facilities in the industry population. The source category is specific enough so that variability within the source category population may be minimized.

B (Above average)

Developed only from A-rated test data from a reasonable number of facilities. Although no specific bias is evident, it is not clear if the facilities tested represent a random sample of the industries. As in the A-rating, the source category is specific enough so that variability within the source category population may be minimized.

C (Average)

Developed only from A and B-rated test data from a reasonable number of facilities.

Although no specific bias is evident, it is not clear if the facilities tested represent a random sample of the industry. As in the A-rating, the source category is specific enough so that variability within the source category population may be minimized.

D (Below average)

The emission factor was developed only from A- and B-rated test data from a small number of facilities, and there is reason to suspect that these facilities do not represent a random sample of the industry. There also may be evidence of variability within the source category population. Limitations on the use of the emission factor are noted in the emission factor table.

E (Poor)

The emission factor was developed from C and D-rated test data, and there is reason to suspect that the facilities tested do not represent a random sample of the industry. There also may be evidence of variability within the source category population. Limitations on the use of these factors are always noted.

The use of these criteria is somewhat subjective and depends to an extent on the individual reviewer.

3.4 REFERENCES FOR CHAPTER 3

1. Technical Procedures for Developing AP-42 Emission Factors and Preparing AP-42 Sections. U.S. Environmental Protection Agency, Air Management Technology Branch, Office of Air Quality Planning and Standards, Research Triangle Park, NC, April 1992.
2. AP-42, Supplement A, Appendix C.2, "Generalized Particle Size Distributions." U.S. Environmental Protection Agency, October 1986.

4.0 POLLUTANT EMISSION FACTOR DEVELOPMENT

4.1 REVIEW OF SPECIFIC DATA SETS

Reference 1: La Roche Industries, Inc. Oxides of Nitrogen Emissions Test Report.

This test was performed in accordance with the EPA Reference Methods 1 through 4 and 7E. All of the required information as outlined in Chapter 3.2 of this report is provided and the results are consistent; therefore, this test is rated "A". No control device was used in this process. Production data are given in terms of short tons of 100 percent HNO₃ produced per day. Emission factors are given as pounds of NO_x emitted per short ton of 100 percent HNO₃ produced. Emission rates were calculated by multiplying the emission factor by the production rate and any relevant conversion factors. Equivalent metric units were also calculated for the emissions data. CO₂ and O₂ emissions were also tested. The data state that zero percent CO₂ was emitted. Since the measuring device is not specified, the accuracy of these readings is not known.

Reference 2: Bison Nitrogen Products Co. Source Test Report. October 1978.

This test was performed in accordance with the EPA Reference Methods 1 through 4 and 7. All of the required information as outlined in Chapter 3.2 of this report is provided and the results are consistent; therefore, this test is rated "A". Extended absorption was used as a control device in this process. Production data are given in terms of short tons of 100 percent HNO₃ produced per day. Emission factors are given as kilograms of NO_x emitted per metric ton of 100 percent HNO₃ produced. These emission factors were converted into the desired units (pounds per short ton) using the appropriate conversion factors.

CO₂ emissions were also tested using an ORSAT. The CO₂ emissions were calculated by using the percent of CO₂ contained in the stack gas on a dry basis and dividing it by 100 to obtain a fractional value of CO₂. This fraction was then multiplied by the stack gas volumetric flow rate and any corresponding conversion factors to obtain the emission rate of CO₂ in terms of pounds of CO₂ emitted per hour. The production rates and emission factors were obtained as described in the preceding paragraph.

Reference 3: Agrico Chemical Company Source Test Report. May 1980.

This test was performed in accordance with the EPA Reference Methods 1 through 4 and 7. All of the required information as outlined in Chapter 3.2 of this report is provided and the results are consistent; therefore, this test is rated "A". Extended absorption with a caustic scrubber were used as control devices in this process. Production data are given in terms of short tons of 100 percent HNO₃ produced per day. Emission factors are given as pounds of NO_x emitted per short ton of 100 percent HNO₃ produced. PES calculated emission rates by multiplying the emission factor by the production rate. Equivalent metric units were also calculated for the emissions data. CO₂ emissions were also tested using gas chromatography. No emissions were detected.

Reference 4: Agrico Chemical Company Source Test Report. November 1976.

This test was performed in accordance with the EPA Reference Methods 1 through 4 and 7. All of the required information as outlined in Chapter 3.2 of this report is provided and the results are consistent; therefore, this test is rated "A". Extended absorption with a caustic scrubber were used as control devices in this process. Production data are given in terms of short tons of 100 percent HNO₃ produced per day. Emission factors are given as pounds of NO_x emitted per short ton of 100 percent HNO₃ produced. PES calculated emission rates by multiplying the emission factor by the production rate. Equivalent metric units were also calculated for the emissions data. CO₂ emissions were also tested using gas chromatography. No emissions were detected.

Reference 5: Agrico Chemical Company Source Test Report. June 1976.

This test was performed in accordance with the EPA Reference Methods 1 through 4 and 7. All of the required information as outlined in Chapter 3.2 of this report is provided and the results are consistent; therefore, this test is rated "A". A caustic scrubber was used as the control device in this process. Production data are given in terms of short tons of 100 percent HNO₃ produced per day. Emission factors are given as pounds of NO_x emitted per short ton of 100 percent HNO₃ produced. PES calculated emission rates by multiplying the emission factor by the production rate. Equivalent metric units were also calculated for the emissions data. CO₂ emissions were also tested using gas chromatography. No emissions were detected.

Reference 6: Bison Nitrogen Products Co. Source Test Report. November 1978.

This source test was classified as unusable to update the existing AP-42 section for the following reasons; no production data, no documentation of the EPA Reference Methods 1 through 4, and no documentation of the process tested.

Reference 7: J.R. Simplot Company Source Test Report. July 1987.

This source test was classified as unusable to update the existing AP-42 section for the following reasons; no documentation of the EPA Reference Methods 1 through 4 or the NO_x emissions sampling method, no documentation of the process tested, no equipment prep documentation, and no field data sheets.

Reference 8: J.R. Simplot Company Source Test Report. April 1990.

This source test was classified as unusable to update the existing AP-42 section for the following reasons; no production rate, no documentation of the EPA Reference Methods 1 through 4, no documentation of the process tested, no equipment prep documentation, and no field data sheets.

Reference 9: Air Products & Chemicals, Inc. Source Test Summary. April 1992.

This source test was classified as unusable to update the existing AP-42 section for the following reasons; no documentation of the EPA Reference Methods 1 through 4 or the NO_x emissions sampling method, no documentation of the process tested, no equipment prep documentation, and no field data sheets.

Reference 10: Air Products & Chemicals, Inc. Source Test Summary. April 1992.

This source test was classified as unusable to update the existing AP-42 section for the following reasons; no documentation of the EPA Reference Methods 1 through 4 or the NO_x emissions sampling method, no documentation of the process tested, no equipment prep documentation, and no field data sheets.

Reference 11: Monsanto Company Source Test Summary. April 1992.

This source test was classified as unusable to update the existing AP-42 section for the following reasons; no documentation of the EPA Reference Methods 1 through 4 or the NO_x

emissions sampling method, no documentation of the process tested, no equipment prep documentation, and no field data sheets.

Reference 12: J.R. Simplot Co. Emissions Summary. February 1990.

This source test was classified as unusable to update the existing AP-42 section for the following reasons; no documentation of the EPA Reference Methods 1 through 4 or the NO_x emissions sampling method, no documentation of the process tested, no documentation of control equipment and its efficiency, no equipment prep documentation, and no field data sheets.

Reference 13: J.R. Simplot Co. Emissions Summary. May 1990.

This source test was classified as unusable to update the existing AP-42 section for the following reasons; no documentation of the EPA Reference Methods 1 through 4 or the NO_x emissions sampling method, no documentation of the process tested, no documentation of control equipment and its efficiency, no equipment prep documentation, and no field data sheets.

Reference 14: J.R. Simplot Co. Emissions Summary. July 1990.

This source test was classified as unusable to update the existing AP-42 section for the following reasons; no documentation of the EPA Reference Methods 1 through 4 or the NO_x emissions sampling method, no documentation of the process tested, no documentation of control equipment and its efficiency, no equipment prep documentation, and no field data sheets.

Reference 15: Handbook of Emission Factors, Parts I and II, Ministry of Housing, Physical Planning and Environment.

This reference provides NO_x and NH₃ emission factor ranges for uncontrolled processes, catalytic reduction, and extended absorption of tail gas for the nitric acid process. The reducing agent used in the catalytic reduction is not specified. The units in the emissions table are in kilograms of emission per ton of 100 percent nitric acid produced. For comparison purposes, PES has converted these factors into units similar to those presented in the AP-42. Uncontrolled emissions from the tail gas are given as a range from 20 to 40 pounds of NO_x emitted per ton of 100 percent HNO₃ produced and 0.02 to 0.2 pounds of NH₃ emitted per ton of 100 percent HNO₃ produced. The revised uncontrolled emission factor in the revised AP-42 is 57 pounds of NO_x emitted per ton of 100 percent HNO₃ produced; NH₃ emissions are not given. The uncontrolled

emissions in this reference (15) are lower than those reported in the revised AP-42. Since Reference 15 does not provide the raw test data used in developing these emission factors, the uncontrolled NO_x emission factor in the revised AP-42 will remain unchanged. For this same reason, none of the NH₃ emission factors will be added to the revised AP-42.

The catalytic reduction and extended absorption NO_x emission factors in the revised AP-42 fall within the ranges specified by Reference 15. However, since no raw test data are provided to support the emission ranges, the revised AP-42 will remain unchanged. Reference 15 did not specify whether the emissions resulted from a single-stage or dual-stage pressure process.

Reference 16: Control of Air Pollution from Nitric Acid Plants.

The 1980 AP-42 has this reference noted to be the source of the emission factor for hydrogen fueled catalytic reduction, natural gas/hydrogen fuel catalytic reduction, and a high strength acid plant. EPA performance tests on four nitric acid plants are used to provide results of NO_x emissions testing. It is unclear from this reference whether the reported emissions occur before or after a control device. It is also unclear what type of control device may be present. The emissions are given in parts per million by volume (ppmv). Since no stack gas flow rate is provided, there is no way of converting ppmv to pounds per ton (lb/ton) or kilogram per megagram (kg/Mg). Emissions in the 1980 AP-42 are given in these later units. It cannot be verified whether these emission values are the same as those appearing in the 1980 AP-42.

Reference 17: Atmospheric Emissions from Nitric Acid Manufacturing Process.

The 1980 AP-42 has this reference noted to be the source of the emission factors for uncontrolled systems, hydrogen fueled catalytic reduction, natural gas/hydrogen fuel catalytic reduction, and a high strength nitric acid plant.

Uncontrolled. Twelve plants were tested to provide uncontrolled NO_x emissions data from a single-stage pressure process. The average emission factor calculated the results of these tests is 57 pounds of NO_x emitted per ton of 100 percent HNO₃ produced with a range from 33 to 110 pounds per ton. The average production rate is 229 tons of HNO₃

produced per day, with a range from 55 to 750 tons per day at an average rated capacity of 97 percent with a range from 72 to 100 percent. This uncontrolled emission factor will replace the 1980 AP-42 uncontrolled emission factor. However, the emission factor rating will be lowered to an "E" rating.

Hydrogen fueled Catalytic Reduction. Three plants were tested for NO_x emissions from hydrogen fueled catalytic incinerators in a single-stage pressure process. One of the plant's results was not included in the average emission factor calculation. This plant's emissions were extremely high when compared to the other facilities which were tested on catalytic incineration. The 1980 AP-42 also excluded this plant's results. The revised AP-42 will leave this emission factor unchanged. However, the emission factor rating will be lowered to an "E" since there are no raw source test data to support the emission factor. The emission factor is based on data from two plants with an average production rate of 160 tons of 100 percent HNO₃ produced per day with a range of 120 to 209 tons per day at an average rated capacity of 98 percent (range of 95 to 100). The average absorber exit temperature is 85°F (29°C) with a range from 78 to 90°F and the average exit pressure is 85 psig (range of 80 to 94).

Natural gas/hydrogen fueled Catalytic Reduction. This emissions data was not included in the 1980 AP-42. Six plants were tested for NO_x emissions from natural gas/hydrogen fueled catalytic reduction in a single-stage pressure process. These tests were performed prior to the initiation of New Source Performance Standards (NSPS). Reference 18 provides NO_x emissions data for both single-stage and dual-stage pressure processes that were gathered after the initiation of NSPS. Testing performed after NSPS implementation will provide more accurate and detailed data. For this reason, the emissions from this reference will not be used to revise the AP-42 factors.

High Strength Acid Plant. A single unit was tested at a high strength acid plant for NO₂ emissions. The production rate was 3000 pounds of HNO₃ produced per hour (1.5 tons per hour) at a 100 percent rated capacity, of 98 percent nitric acid. The NO₂ emission factor reported is 5 pounds of NO₂ emitted per 1000 pounds of 100% HNO₃ produced. This factor has been converted to units of pounds per ton and kilograms per megagram in AP-

42. The AP-42 emission factor will remain unchanged. However, since there are no raw test data given to support this factor, the emission factor rating will be lowered to an "E".

This reference also provides emissions information for natural gas fueled catalytic reduction. However, the authors of the 1980 AP-42 chose not to include these emissions in developing the 1980 emission factors; instead, Reference 18, taken from the 1980 AP-42, provided the necessary emissions. It is uncertain why the emission factors contained in Reference 17 were not included. An assumption can be made based upon the fact that since the testing was performed prior to NSPS, the authors assumed that testing data gathered after NSPS was initiated would provide more valid information to develop emission factors. Reference 18, taken from the 1980 AP-42, provides testing data completed after the initiation of NSPS. Emissions occurring after the NSPS should be better controlled than those occurring prior to the NSPS. For these reasons, the natural gas fueled catalytic reduction emission factor was left unchanged in the 1992 AP-42.

Reference 18: A Review of Standards of Performance for New Stationary Sources-Nitric Acid Plants.

The 1980 AP-42 lists this reference as providing emissions information for uncontrolled processes as well as natural gas fueled catalytic reduction, extended absorption, and chilled absorption with a caustic scrubber for nitric acid manufacturing processes. The following is a detailed description of the information available from this reference.

Uncontrolled. The uncontrolled emission factor given in the 1980 AP-42 is noted to originate from this reference. However, after reviewing this reference, PES could not verify that the emissions in the 1980 AP-42 originated from information contained in Reference 18. The only discussion on uncontrolled emissions provides a sentence stating a range of NO_x emissions from 20 to 28 kilograms of NO_x emitted per megagram of 100 percent HNO₃ produced. No further information is provided. For this reason, the uncontrolled emission factor presented in the revised AP-42 will be taken from Reference 17 of this report.

Natural Gas fueled Catalytic Reduction. The fuel for the catalytic incinerator is assumed to be natural gas as implied by this reference. Seven plants were tested for NO_x emissions. The average production rate was 341 tons of 100 percent HNO₃ produced per day with a range of 55 to 1077 tons per day. The emission factor in the 1992 AP-42 will remain the same; however, the emission factor rating will be lowered to an "E" since there are no raw test data provided for evaluation.

Extended Absorption. The extended absorption emission factor in the 1980 AP-42 is noted to originate from this reference. Five single-stage pressure process plants and three dual-stage pressure process plants were tested for NO_x emissions. The 1980 AP-42 did not differentiate between the two processes. The number of plants tested were noted incorrectly in the 1980 AP-42. The emission factor in the revised AP-42 will be corrected to reflect the two different processes. The single-stage pressure process plants had an average production rate of 542 tons of 100 percent HNO₃ produced per day with a range from 209 to 1049 tons per day. The dual-stage pressure process plants had an average production rate of 586 tons of 100 percent HNO₃ per day with a range from 315 to 937 tons per day. Since no raw test data is provided, the emission factors will be given a rating of "E".

Chilled Absorption with Caustic Scrubber. One plant was tested for NO_x emissions from a chilled absorption system with a caustic scrubber. This single-stage pressure process had a production rate of 692 tons of 100 percent HNO₃ produced per day. The emission factor will be added to the revised AP-42, and it will be given an emission factor rating of "E" since there are no raw test data available for review.

4.2 CRITERIA POLLUTANT EMISSIONS DATA

No data on emissions of volatile organic compounds, lead, sulfur dioxide, carbon monoxide, or particulate matter were found nor expected for the nitric acid process. The remaining criteria pollutant, nitrogen oxide, is discussed below.

Nitrogen oxides.

Fourteen source tests were received to update the AP-42 section. Only five tests contained sufficient information to qualify as valid reports. The other nine were omitted due to various reasons; no production data, no EPA sampling method specified or no process description. One of the valid source tests which had no control device contained lower emission factors than any of the other tests with control devices. After careful review, PES could not conclude why these factors were lower without any additional information. Emission data for the five source tests can be seen in Table 4.2-1, but were not used to revise existing emission factors since these tests may not be representative of the entire industry. Further details on all of these tests can be found in Chapter 4.1 of this report.

After careful evaluation of the existing emission factors for NO_x in the 1980 AP-42, PES concluded that several emission factors needed to be corrected. The uncontrolled emission factor, the natural gas/hydrogen combination fuel for catalytic reduction emissions, and the extended absorption emission factor were corrected. The emission factor footnotes were corrected for each of the emission sources. For additional details on how all of these corrections were made, please see Chapter 4.1 of this report under the discussion for References 16, 17, and 18.

**TABLE 4.2-1 (METRIC UNITS)
NITROGEN OXIDES**

Source Test #	Test Rating	Test Method	Run #	Production Rate ^a	Emission Rate ^b	Emission Factor ^c
Control device: None						
1	A	7E	1	490	254	0.518
			2	490	202	0.432
			3	490	185	0.379
			Average	490	212	0.434
Control device: Extended Absorber						
2	A	7	1	192	209	1.089
			2	192	105	0.545
			3	192	31.3	0.163
			Average	192	113	0.590
Control device: Extended Absorber with Caustic Scrubber						
3	A	7	1	679	645	0.950
			2	679	605	0.890
			3	679	629	0.925
			Average	679	625	0.920
Control device: Extended Absorber with Caustic Scrubber						
4	A	7	1	632	512	0.810
			2	632	531	0.840
			3	632	762	1.210
			Average	632	594	0.940
Control device: Caustic Scrubber						
5	A	7	1	538	1921	3.57
			2	538	2055	3.82
			3	538	2351	4.37
			Average	538	2109	3.92

^aUnits in Mg (100% HNO₃)/day.

^bUnits in kg (NO_x)/day.

^cUnits in kg (NO_x)/Mg (100% HNO₃).

**TABLE 4.2-1 (ENGLISH UNITS)
NITROGEN OXIDES**

Source Test #	Test Rating	Test Method	Run #	Production Rate ^a	Emission Rate ^b	Emission Factor ^c
Control device: None						
1	A	7E	1	540	558.9	1.035
			2	540	446.0	0.863
			3	540	408.8	0.757
			Average	540	468.2	0.867
Control device: Extended Absorber						
2	A	7	1	212	461.5	2.177
			2	212	230.9	1.089
			3	212	69.1	0.326
			Average	212	249.9	1.179
Control device: Extended Absorber with Caustic Scrubber						
3	A	7	1	749	1423	1.90
			2	749	1333	1.78
			3	749	1386	1.85
			Average	749	1378	1.84
Control device: Extended Absorber with Caustic Scrubber						
4	A	7	1	697	1129	1.62
			2	697	1171	1.68
			3	697	1680	2.41
			Average	697	1310	1.88
Control device: Caustic Scrubber						
5	A	7	1	593	4234	7.14
			2	593	4530	7.64
			3	593	5183	8.74
			Average	593	4649	7.84

^aUnits in tons (100% HNO₃)/day.

^bUnits in lb (NO_x)/day.

^cUnits in lb (NO_x)/ton (100% HNO₃).

4.3 NONCRITERIA POLLUTANT EMISSIONS DATA

Hazardous Air Pollutants.

Hazardous Air Pollutants (HAPs) are defined in the 1990 Clean Air Act Amendments. No data on emissions of these pollutants were found for the nitric acid process.

Global Warming Gases.

Pollutants such as methane, carbon dioxide, and NO have been found to contribute to overall global warming. Nitrogen oxide can be classified within the NO_x category; however, no information was provided to distinguish the percent of NO contained in the NO_x data. Fourteen source tests were received for review to update AP-42 emission factors. These tests provided NO_x emissions data. Five of these tests contained enough data to use as valid source tests. The results of these tests can be seen in Table 4.2-1 in Chapter 4.2 under the NO_x section.

One of the valid source tests, Reference 2, also provided CO₂ emissions data. The other four tests stated that no CO₂ emissions were present. For details on how CO₂ emissions were calculated, see Chapter 4.1 under the Reference 2 discussion. The CO₂ emissions can be found in Table 4.3-1. One test does not provide sufficient data to establish emission values which are characteristic of the nitric acid industry.

**TABLE 4.3-1 (METRIC UNITS)
CARBON DIOXIDE**

Source Test #	Test Rating	Test Method	Run #	Production Rate ^a	Emission Rate ^b	Emission Factor ^c
Control device: Extended Absorption						
2	A	7	1	192	16264	84.7
			2	192	19574	102
			3	192	22415	117
			Average	192	19418	101

^aUnits in Mg (100% HNO₃)/day.

^bUnits in kg (CO₂)/day.

^cUnits in kg (CO₂)/Mg (100% HNO₃).

**TABLE 4.3-1 (ENGLISH UNITS)
CARBON DIOXIDE**

Source Test #	Test Rating	Test Method	Run #	Production Rate ^a	Emission Rate ^b	Emission Factor ^c
Control device: None						
2	A	7	1	212	35856	169
			2	212	43152	203
			3	212	49416	233
			Average	212	42808	202

^aUnits in tons (100% HNO₃)/day.

^bUnits in lb (CO₂)/day.

^cUnits in lb (CO₂)/ton (100% HNO₃).

Ozone Depletion Gases.

Chlorofluorocarbons have been found to contribute to ozone depletion. No data on emissions of these pollutants were found for the nitric acid process.

4.4 DATA GAP ANALYSIS

None of the emission factors in the AP-42 is based on primary source test data. Consequently, the emission factor ratings have been lowered to an "E". After review of the references used to create the AP-42 emissions, the emission factors have been redefined to reflect the nitric acid process more effectively. Although more definition has been given to the factors, more testing is necessary to determine if the values are indeed correct.

Three tests received to update the AP-42 utilized extended absorption for the control device. The emission factors calculated from these tests are below the emission factors specified in the revised AP-42. It seems that the AP-42 emission factors are valid for the extended absorption process. One test received used a caustic scrubber to control emissions from the tail gas. There is no emission factor in the AP-42 to compare its results. Only one test was received which contained no control device on the tail gas stream. This test provides surprising results in that its emission factor has the same magnitude of those facilities utilizing control devices. Without further information, it cannot be determined why these results occurred.

4.5 REFERENCES FOR CHAPTER 4

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14. Emissions Summary. J.R. Simplot Company, Don Siding, ID. July 1990.
15. Handbook of Emission Factors, Parts I and II, Ministry of Housing, Physical Planning and Environment, The Netherlands, 1980/1983.
16. Control of Air Pollution from Nitric Acid Plants, Office of Air Quality Planning and Standards, U.S. Environmental Protection Agency, Research Triangle Park, NC. August 1971. Unpublished.

17. Atmospheric Emissions from Nitric Acid Manufacturing Processes, 999-AP-27, U.S. Department of Health, Education, and Welfare, Cincinnati, OH. 1966.
18. Marvin Drabkin, A Review of Standards of Performance for New Stationary Sources - Nitric Acid Plants, EPA-450/3-79-013, U.S. Environmental Protection Agency, Research Triangle Park, NC. March 1979.

**TABLE 4-4.
LIST OF CONVERSION FACTORS**

Multiply:	by:	To obtain:
mg/dscm	4.37 x 10 ⁻⁴	gr/dscf
m ²	10.764	ft ²
acm/min	35.31	acfm
m/s	3.281	ft/s
kg/hr	2.205	lb/hr
kPa	1.45 x 10 ⁻¹	psia
kg/Mg	2.0	lb/ton
Mg	1.1023	ton
l	1000	m ³
mg	1000	g
metric ton	0.001	kg

Temperature conversion equations:

Fahrenheit to Celsius:

$$^{\circ}\text{C} = \frac{(^{\circ}\text{F} - 32)}{1.8}$$

Celsius to Fahrenheit:

$$^{\circ}\text{F} = 1.8(^{\circ}\text{C}) + 32$$

APPENDIX A.

AP-42 SECTION 5.9.

[Not presented here. See instead current AP-42 Section 8.8.]