



SAFE PRACTICES FOR THE PRODUCTION OF NITROUS OXIDE FROM AMMONIUM NITRATE

Doc 175/24

Revision of EIGA Doc 175/16

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As part of a programme of harmonisation of industry standards, the European Industrial Gases Association (EIGA) has published EIGA Doc 175, *Safe Practices for the Production of Nitrous Oxide from Ammonium Nitrate*. This publication was jointly produced by members of the International Harmonisation Council.

This publication is intended as an international harmonised publication for the worldwide use and application by all members of the International Harmonisation Council whose members include the Asia Industrial Gases Association (AIGA), Compressed Gas Association (CGA), European Industrial Gases Association (EIGA), and Japan Industrial and Medical Gases Association (JIMGA). Regional editions have the same technical content as the EIGA edition, however, there are editorial changes primarily in formatting, units used and spelling. Regional regulatory requirements are those that apply to Europe.

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Amendments to 175/16

Section	Change
4.1	Reinforced visual detection of iron contamination of ammonium nitrate
4.1.1.1	Ensure cleanliness of ammonium nitrate transport vehicle
4.1.1.2	Address spillage prevention for liquid ammonium nitrate
4.2	Added the possibility of N ₂ O monitoring for long term exposure in improperly ventilated work areas
5.1.1.3	Visual aid to determine iron contamination in the reactor
5.1.1.4	Visual inspection added to LAN delivery vehicle to mitigate contaminated supply
5.3.1	Added an additional layer of overtemperature protection, control of chloride concentration and overpressure caution to LAN transfer in the melter

5.3.2.3	Added text for fail safe thermal restart and protection of electrical heater elements from a known design issue, cooling water system design details and proper return to service after maintenance
5.3.2.5	Added design requirements for reactor pressure relief based on experimental data
5.3.2.6	Added reactor restart thermal runaway and low level risk and mitigation
5.3.6	Moved reference to oxygen compressor document from requirements to a note to address non-applicable mandatory language
6.1	Strengthened maintenance requirements for vessel, control loop and critical safety control system maintenance
6.3	Added a new section for emergency water cooling system testing
8.1	Added new section for abnormal operation procedure requirements
8.2	Rewrite of the emergency procedure section

NOTE Technical changes from the previous edition are underlined

1 Introduction

Nitrous oxide (N₂O) has been produced and distributed by the industrial gases industry for many years. It is mainly used for medical purposes (anesthesia). It is also used in the food and electronic industries.

Severe accidents, such as violent explosion of ammonium nitrate or decomposition of nitrous oxide, have occurred at facilities producing nitrous oxide from ammonium nitrate. In addition, nitrous oxide gas in elevated concentrations can cause health effects in operators that should be prevented.

A major cause of accidents and health effects when producing nitrous oxide from ammonium nitrate has been insufficient attention to the specific properties of these materials when designing equipment and developing operating procedures. For that reason, this publication describes the properties and hazards of ammonium nitrate and nitrous oxide. On this basis, the principles and relevant details of safe production of nitrous oxide from ammonium nitrate are considered.

Regulatory requirements for medical applications shall also be followed, usually specified in the applicable Pharmacopeia for the country of operation. For example, the European Guide Good Manufacturing Practice in Europe, U.S. Food and Drug Administration (FDA) and Health Canada (HC) good manufacturing practices in North America, as well as NFPA 99, *Health Care Facilities Code* in the United States and CSA Z305.1, *Nonflammable Medical Gas Piping Systems* in Canada for medical gas piping systems [1, 2, 3, 4, 5].¹

2 Scope

This publication serves the interest of all who could in any way be associated or concerned with nitrous oxide manufacturing from the thermal decomposition of ammonium nitrate. It also serves to acquaint persons not versed in ammonium nitrate and nitrous oxide technology with those factors considered important to health and safety.

This publication applies to safety in the design, construction, installation, operation, and maintenance of nitrous oxide plants using ammonium nitrate technology. Emphasis is placed on equipment, operational and maintenance features that are particular to nitrous oxide plants.

For details of publications covering the safe practices for storage and handling of nitrous oxide see EIGA Doc 176, *Safe Practices for Storage and Handling of Nitrous Oxide* and CGA G-8.1, *Standard for Nitrous Oxide Systems at Customer Sites* [6, 7].

This publication is not applicable to the process of production of nitrous oxide from other raw materials but may be used as a guideline for the purification, drying, compression, liquefaction, and storage as applicable.

Nitrous oxide emissions during production are not covered by the scope of this publication, for information, see EIGA Doc 112, *Environmental Impacts of Nitrous Oxide Plants* [8]).

3 Definitions

For the purpose of this publication, the following definitions apply.

3.1 Publication terminology

3.1.1 Shall

Indicates that the procedure is mandatory. It is used wherever the criterion for conformance to specific recommendations allows no deviation.

¹ References are shown by bracketed numbers and are listed in order of appearance in the reference section.

3.1.2 Should

Indicates that a procedure is recommended.

3.1.3 May

Indicates that the procedure is optional.

3.1.4 Will

Is used only to indicate the future, not a degree of requirement.

3.1.5 Can

Indicates a possibility or ability.

3.2 Technical definitions

3.2.1 Cylinder

Transportable pressure receptacle of a water capacity not exceeding 150 litres [9].

3.2.2 Decomposition

Separation of a chemical compound into smaller elements.

NOTE Ammonium nitrate and nitrous oxide separates into components in an exothermic reaction that can be accelerated by changes in pressure, temperature, energy inputs, the presence of catalyzer or impurities.

3.2.3 Endothermic

Chemical reaction that absorbs heat.

3.2.4 Exothermic

Chemical reaction that produces heat.

3.2.5 Liquefied gas

Gas which when packaged under pressure for carriage is partially liquid at temperatures above $-50\text{ }^{\circ}\text{C}$ ($-58\text{ }^{\circ}\text{F}$) [9].

3.2.6 Qualified nitrous oxide technician

Person who by reason of education, training and experience is knowledgeable of the properties of nitrous oxide, is familiar with the equipment used to store, transfer, and use nitrous oxide and understands the precautions necessary to safely use nitrous oxide equipment.

3.2.7 Pressure

In this publication, “bar” (“psi”) noted as (psia; bar, abs; and kPa, abs) for absolute pressure or (psid; bar, dif; and kPa, dif) for differential pressure.

3.2.8 Tank

Collective term that includes stationary tanks and transport tanks.

4 Properties and hazards

4.1 Ammonium nitrate

Both forms of the feedstock, solid ammonium nitrate (SAN) and liquid ammonium nitrate (LAN) shall be considered as oxidizing substances with relevant-properties and requiring safety precautions see Table 1.

In both LAN and SAN, the iron concentration can be out of specification if the ammonium nitrate supplier has recently restarted the plant. Since the analysis is typically a batch analysis, high iron concentration at the beginning of the batch can be missed. This may be detected at the nitrous oxide production site by visual inspection for discoloration of the supplied ammonium nitrate and by monitoring the color in the melter and in the reactor (see also 5.1.1.3).

LAN and SAN are both hazardous materials, but LAN has the following safety advantages compared with SAN:

- Cross contamination with anti-caking substances is less likely with LAN;
- It is easier to maintain quality;
- The operation of the plant is safer;
- Consequently, LAN has a lower hazard rating in transport and environmental legislation; and
- Handling is minimised.

Table 1—Ammonium nitrate

	Solid ammonium nitrate (SAN)	Liquid ammonium nitrate (LAN)
<u>Identification</u>		
Chemical formula	NH ₄ NO ₃	80 – 93% NH ₄ NO ₃ in water
CAS No.	6484-52-2	6484-52-2
EC No.	229-347-8	229-347-8
UN-No:	UN 1942	UN 2426
Name according to [9]	ammonium nitrate	ammonium nitrate, liquid
Transport Class according to [9]	5.1	5.1
Classification according to [9]	O2 (oxidizing substance solid)	O1 (oxidizing substance liquid)
Properties	Contact with combustible material can cause fire. Explosive when mixed with combustible material. Decomposes when heated to about 170 °C (338 °F). Fire can cause generation of toxic nitric gases.	LAN has in principle the same properties as solid ammonium nitrate, but due to its water content it is slightly less sensitive.
Safety precautions	Keep away from sources of ignition – no smoking. Avoid contamination by combustible liquids, powdery substances, oxidizing substances, alkalis and acids. Avoid contact with skin and eyes. Clean ammonium nitrate from equipment during maintenance prior to welding or brazing	
Health hazards	The substance can be absorbed into the body by inhalation of its aerosol. Short term exposure: Irritating to the eyes, the skin and the blood respiratory tract.	

4.1.1 Liquid ammonium nitrate (LAN)

4.1.1.1 Quality requirements

Depending on the process 80% to 96% NH_4NO_3 solutions are being used:

NOTE For transportation purposes the concentration is limited to 93% (UN 2426).

- A quality certificate with purchase is required;
- The supplier should be qualified and approved by the user;
- Approved insulated transport container is required (some being equipped with external steam heating coils). Refer to the applicable transport regulations [9];
- Ammonium nitrate transport vehicles should be confirmed cleaned for service prior to use; and
- The product quality shall be analysed by either the supplier or consumer.

Table 2—Typical specification for technical grade liquid ammonium nitrate

NH_4NO_3 concentration range	min. 82.0% – max. 93.0%
NH_3 (free ammonia)	max. 500 ppm (500 mg/kg)
Volatile organic matter (for example, oil)	max. < 20ppm (20 mg/kg [as carbon])
Chloride (Cl)	< 5 ppm (<5 mg/kg)
Iron (Fe)	< 4 ppm (<4 mg/kg)
Phosphate (PO_4)	< 10 ppm (<10 mg/kg)
Calcium (Ca)	< 10 ppm (<10 mg/kg)
Sulphate (SO_4)	< 50 ppm (< 50 mg/kg)
Acidity of 10 % test solution	5 < pH < 6.5
Urea	< 5 ppm
Foreign matter	None visible
Extractable organic matter	< 100 ppm
Anti-caking agent or additive	<u>None</u>

4.1.1.2 Storage requirements

The following storage requirements for ammonium nitrate apply:

- Arrangements for heating (for example, steam, recirculation of LAN) are required. No direct electrical heating is allowed. To avoid localized high temperature, hot points such as exposed electric heating elements shall not be used;
- The temperature shall be controlled to prevent crystallization;
- If necessary the actual ammonium nitrate concentration should be controlled and diluted to the required specification. Additional water should have low chloride and iron content;
- Storage quantity shall be according to local regulations;
- The LAN storage tank area shall be protected against spillage and the tank discharge system shall be protected to prevent inadvertent discharge into drains, for example, by a retention area;
- The LAN unloading system, including hose and fittings, shall be inspected for damage before offloading LAN into the storage tank to prevent leaks;