SAFE DESIGN AND OPERATION OF ON-SITE NITROGEN GENERATORS FOR FOOD USE

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1 Introduction

This publication provides specific guidelines for on-site nitrogen generators for food use.

An on-site nitrogen generator separates nitrogen from air for use directly at the user location delivered by pipeline. In terms of gas flow rates, on-site nitrogen generators range from a few litres per minute up to many tonnes per hour.

In the course of discussions with customers and authorities, requests are often made for compliance certificates related to nitrogen specifications, hazard analysis and critical control point (HACCP) studies, materials of construction and consumable materials for those on-site gas production units: pressure swing adsorption (PSA), membrane air separation system and cryogenic air separation system that are dedicated for food use.

On-site nitrogen generators for food use, for example for modified atmosphere packaging (MAP) or inerting applications, are installed at customer sites by either gas companies or equipment suppliers and can be operated by either gas companies or food companies.

Nitrogen generators which use PSA and membrane technologies have potential hazards that shall be recognized and addressed in view of the applicable food laws.

The nitrogen produced from an on-site generator is used in many food applications as a processing aid (without purity limit*) or additive (E941 *Commission Regulation 2012/231/EC of March 9th 2012 laying down specific purity criteria on food additives other than colours and sweeteners* [1][1]). Typical applications include: atmosphere protection of liquid and solid foodstuffs; tank blanketing; liquids mixing; liquid pressure transfer; modified atmosphere packaging; injection in liquid for container pressurization, decarbonation, deoxygenation, beverage dispensing; aerosol propulsion.

* NOTE No purity criteria are set under European Union (EU) law for the use of gas as a processing aid. However, national legislation can require a purity alignment with those criteria applied to food additives.

2 Scope and purpose

2.1 Scope

The design and operation of on-site nitrogen generators by industrial gas companies for food use.

2.2 Purpose

This publication is intended as a guide for on-site nitrogen generators for food use regarding:

- identification of potential hazards (biological, chemical and physical types) during the equipment design and its operation;
- Hazard Analysis and Critical Control Point (HACCP) procedures;
- description of appropriate design, operation, maintenance and modification;
- recommendations concerning traceability, production lots, registration parameters, product analysis; and
- assurance for operators and authorities of the quality and compliance of the gas produced with an on-site nitrogen generator.

[1] References are shown in bracketed numbers and are listed in order of appearance in the reference section

3 Definitions

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.

3.1.2 Should

Indicates that a procedure is recommended.

3.1.3 May and need not

Indicates that the procedure is optional.

3.1.4 Will

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 Asphyxiation

To become unconscious or die from the lack of oxygen.

3.2.2 Batch

Can be considered as a discrete, defined quantity whose characteristics can be proven.

3.2.3 Critical control point (CCP)

Step where a control must be applied and which is essential to prevent or eliminate a food safety hazard or to reduce it to an acceptable level.

3.2.4 Cryogenic liquid form

Liquid material that is extremely cold at a temperature that is lower than -90°C

3.2.5 Cryogenic air separation generator

Equipment for the production of high purity nitrogen by means of a cryogenic process.

3.2.6 Fail-safe

When a system component fails, the resulting situation does not present a safety concern, e.g. when an isolation valve closes subsequent to generator air or power supply failure.

3.2.7 Food or foodstuff

"Any substance or product, whether processed, partially processed or unprocessed, intended to be, or reasonably expected to be, ingested by humans" (Regulation (EC) No 178/2002 *of the European Parliament and of the Council of 28 January 2002 laying down the general principles and* *requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety* [2]).

3.2.9 Food additive

"Any substance not normally consumed as a food in itself and not normally used as a characteristic ingredient of food, whether or not it has nutritive value, the intentional addition of which to food for a technological purpose in the manufacture, processing, preparation, treatment, packaging, transport or storage of such food results, or may reasonably be expected to result, in it or its by-products becoming directly or indirectly a component of such foods" (Regulation (EC) No 1333/2008 *of the European Parliament and of the Council of 16 December 2008 on food additives* [3]).

3.2.10 Food additive gas

"Any gas, other than air, which is introduced into a container before, during or after the placing of a food in that container" (Regulation 1333/2008/EC [3]). Modified atmosphere packaging (MAP) is the common name for application.

3.2.11 Food grade gases or food gases

Gases intended to be used as a food additive, processing aid or ingredient.

3.2.12 Food grade lubricant

Synthetic lubricant conforming to EN ISO21469: Safety *of machinery. Lubricants with incidental product contact. Hygiene requirements* [4].

3.2.13 Food traceability

Ability to trace and follow a food through all stages of production, processing and distribution. This requires food business operators to know from whom they have received food and to whom they supply it. The principal purpose of the traceability requirement is to enable efficient and rapid withdrawal from the market of any food that may be injurious to the consumer's health.

3.2.14 Good manufacturing practices (GMP)

Safety conditions for operating the supply chain in order not to introduce contamination.

3.2.15 Hazard analysis and critical control points (HACCP)

Method to identify, evaluate, and control hazards which are significant for food safety.

3.2.16 Labelling

Distinctive mark of the on-site nitrogen generator for food use.

3.2.17 Modified atmosphere packaging (MAP)

Application exchanging the air in contact with food with a gas or a gas mixture to increase the shelf-life of food.

3.2.18 Membrane

Polymer material that acts like a filter to separate components such as nitrogen from oxygen in air.

3.2.19 Non-conforming product

Product which does not meet the relevant company specifications nor has other unspecified impurities which are suspected or known to be at levels that might, when used in contact with foods, be injurious to health (Company specifications are assumed to comply with legislative specifications).

3.2.20 On-site nitrogen generator

Equipment for the production of nitrogen from air at the customer site; the equipment usually supplies the nitrogen to a distribution pipework system.

3.2.21 Food business operator

The natural or legal persons responsible for ensuring that the requirements of food law are met within the food business under their control (EC178/2002 [2]).

3.2.22 Oxygen-deficient atmosphere/nitrogen-enriched atmosphere

Air in which the oxygen concentration by volume is less than 19.5%; also known as nitrogen-enriched atmosphere.

3.2.23 Oxygen enriched atmosphere

Air in which the oxygen concentration by volume exceeds 23.5%; also known as an oxygen enriched atmosphere.

3.2.24 Prerequisite programs procedure (PRP)

Basic food safety conditions and activities that are necessary to maintain a hygienic environment throughout the food chain suitable for the production, handling and provision of safe end products and safe food for human consumption.

3.2.25 Operational prerequisite program (OPRP)

PRP identified by the hazard analysis as essential in order to control the likelihood of introducing food safety hazards and/or the contamination or proliferation of food safety hazards in the product(s) or in the processing environment.

3.2.26 Pressure relief device (PRD)

Device designed to protect a vessel or piping from reaching pressures higher or lower (vacuum) than its design limits in order to avoid the failure of the pipe or vessel.

NOTE As these devices can have significant flow when activated, the discharge should be directed towards a safe area.

3.2.27 Pressure swing adsorption (PSA)

Family of generators that separates one gas from another by passing a feed gas over a bed of adsorbent material at one pressure and cleaning the waste product off the adsorbent material at another pressure, hence the term pressure swing.

3.2.28 Processing aid

"Any substance not consumed as a food by itself, intentionally used in the processing of raw materials, foods or their ingredients to fulfil a certain technological purpose during treatment or processing, and which may result in the unintentional but technically unavoidable presence of residues of the substance or its derivatives in the final product, provided that these residues do not present any health risk and do not have any technological effect on the finished product" (Directive 1333/2008/EC [3]).

3.2.29 Safe area

Location where exhaust gases can be discharged safely causing no harm to personnel or property. A safe area is also a place where surrounding materials are compatible with the exhaust gas.

3.2.30 Safety permits (Permit to work)

Procedural documents highlighting specific safety considerations that are issued to allow work to commence in a specific location.

3.2.31 Sanitary standard operating procedure (SSOP)

Common name given to the sanitation procedures in food production plants which are required by the Food Safety and Inspection Service of the United States Department of Agriculture and regulated by 9 CFR part 416 [5] in conjunction with 21 CFR part 178.1010 [5].

NOTE SSOPs are generally documented steps that shall be followed to ensure adequate cleaning of product contact and non-product surfaces. These cleaning procedures shall be detailed enough to make certain that adulteration of product will not occur. All HACCP plans require SSOPs to be documented and reviewed periodically to incorporate changes to the physical plant. This reviewing procedure can take on many forms, from annual formal reviews to random reviews, but any review should be done by “responsible educated management”. As these procedures can make their way into the public record if there are serious failures, they might be looked at as public documents because they are required by the government. SSOPs in conjunction with the Master Sanitation Schedule and Pre-Operational Inspection Program, form the entire Sanitation operational guidelines for food related processing and one of the primary backbones of all food industry HACCP plans.

3.2.32 Standard operating procedure (SOP)

Procedure to manage supply chain system operation and maintenance, in order to supply safe product.

4 Production process

4.1 Nitrogen quality

For any food use, on-site generated nitrogen shall comply with the general principles described in the EIGA Doc 125 *Guide to the Use of Gases in Foods* [6].

In the case where on-site generated nitrogen is used as a food additive, such as in the modified atmosphere packaging (MAP) application, nitrogen should comply with minimum purity criteria for E941 [1] additive described in EIGA Doc 126 [7]:

- nitrogen* ≥ 99% vol
- oxygen ≤ 1%vol
- water ≤ 0.05% vol

*99% including other inert gases such as noble gases (argon mainly)

Impurities:

- carbon monoxide ≤ 10 ppmV
- methane and other hydrocarbons (as methane) ≤ 100 ppmV
- nitrogen monoxide and nitrogen dioxide ≤ 10 ppmV

In the case where on-site generated nitrogen is used as a processing aid, in accordance with the user's specifications, the on-site generator can be designed to produce different nitrogen assay values (typically between 90% and 99% nitrogen, with the balance predominantly noble gases and oxygen).

At least one continuously on-line residual oxygen analyser shall be installed in the generated nitrogen gas stream to ensure the end-user's product quality and traceability requirements. Consideration should be given to the installation of an on-line residual moisture analyser. In any event the process and its environment shall be subject to a food safety risk assessment.

In the event that the on-line instrumentation detects the produced nitrogen stream to be out of specification range for oxygen, this nitrogen stream shall be vented to a safe location, with an automatic switch to another nitrogen source. In case no alternative source is available, an alarm for off-specification should be routed to the operator so that corrective action can be initiated.

An indication of the oxygen concentration shall be present on the front panel of the gas generator and ideally be transmitted to the user.

When commissioned, the on-site generator shall be tested under standard operating conditions to demonstrate that the residual impurities are below the specification limits. A cylinder may be used to take a sample for remote analysis. It should therefore not be necessary to install a dedicated analyser for residual impurities.

4.2 In feed air compression

A typical on-site nitrogen generator will employ one of three different technologies:

- pressure swing adsorption (PSA);
- membrane separation; or
- cryogenic separation.

In all cases the first step in the process is the compression and initial filtration and drying of air. The fundamental requirements for the in feed air compression system are common to all of the downstream separation technologies.

Feed air compression systems for PSA, membrane or cryogenic generators producing nitrogen for use in food applications will typically, though not exclusively, use oil-free screw compressors and, less frequently, oil-free reciprocating or centrifugal compressors. If oil lubricated compressors are used, an oil separation system shall be installed, controlled and maintained in order to prevent oil carry-over and downstream contamination. A food grade lubricant may be used following a review to ensure it is suitable for the application.

Pre-treatment of the in feed air varies depending on the compressor type and separation technology used, typical treatment steps include:

- dust and particulate filtration;
- coalescing-type filters to remove entrained oil and water droplets, entrained mist, and aerosols;
- air dryers (refrigeration or desiccant) to reduce water vapour content and prevent condensation;
- carbon adsorption filters to remove hydrocarbons and other chemicals;
- molecular sieve beds to remove undesirable chemical vapours; and
- air heaters to control the temperature of the feed air supply.

For specific details, reference shall be made to the equipment manufacturer's manuals.

4.3 Pressure swing adsorption

Pressure swing adsorption (PSA) nitrogen generators are used to produce gaseous nitrogen at a specified purity (food grade), flow, and pressure from a compressed air source.

The technology involves the separation of nitrogen from oxygen by passing air through a bed of adsorbent material, typically a carbon molecular sieve (CMS). Under pressure, the CMS material preferentially adsorbs oxygen and system operation moisture while it allows nitrogen to pass through the vessel. During generator operation, the CMS becomes saturated with oxygen. The CMS is systematically regenerated by desorbing the oxygen and moisture at a lower pressure.

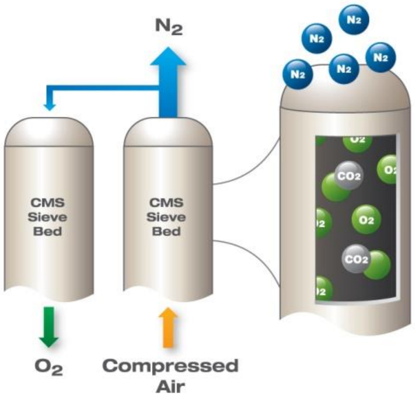

Figure 1 Schematic of a pressure swing adsorber

Major equipment components include a feed air compression system; feed air pre-treatment equipment; vessels containing adsorbent; process piping and valves; a nitrogen compressor (when the customer requires pressures greater than the PSA pressure); process control systems; and other auxiliary components such as coolers, separators, storage receivers, and instrument air systems.

A typical PSA nitrogen generator flow diagram is shown in Figure 2.

Product purity is affected by adjusting the operating pressure, temperature, bed switch frequency and flow through the adsorber vessels.

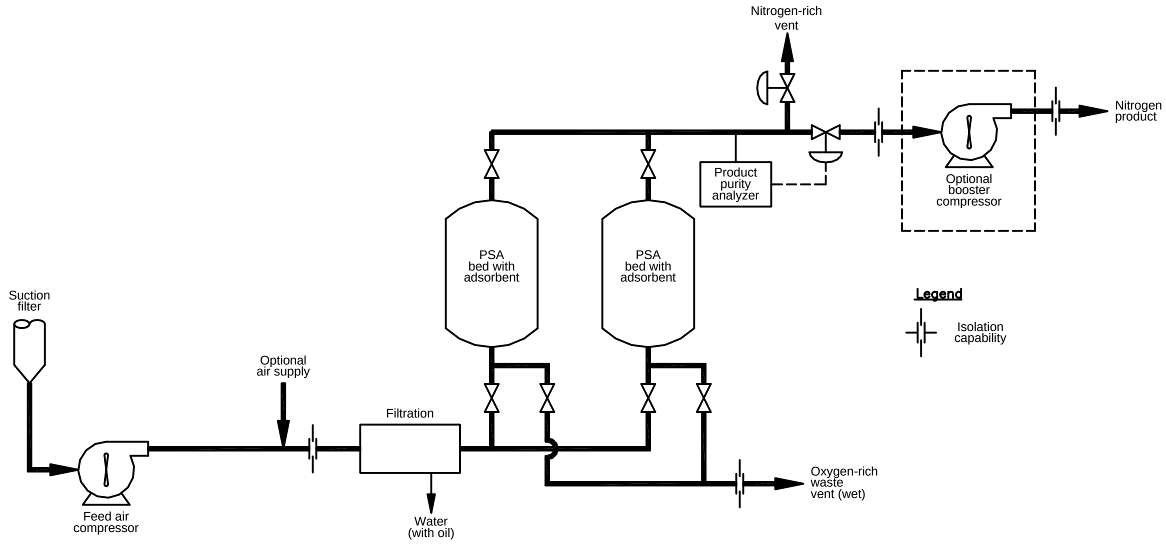

Figure 2 Typical PSA nitrogen generator flow diagram

Figures 3 and 4 are examples of PSA nitrogen generators.

Figure 3 Small enclosed PSA

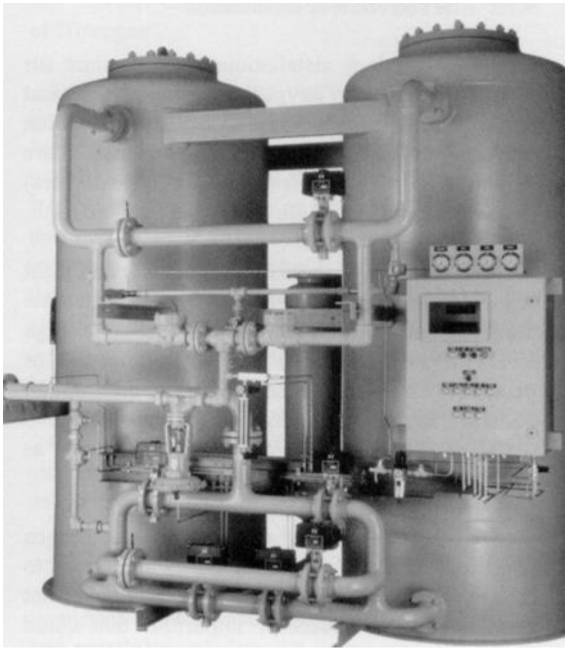
Figure 4 Typical PSA

4.4 Membrane nitrogen generators

Membrane nitrogen generators are used to produce gaseous nitrogen at a specified purity, flow, and pressure from a compressed air source.

At the core of these systems is the membrane module. This module typically consists of thousands of small diameter hollow fibres that are bound together on each end by tube sheaths, formed into bundles, and contained within a protective outer shell. The compressed air feed stream can be introduced either on the shell or bore side of the membrane fibres. Since oxygen permeates more rapidly than nitrogen through the membrane wall, the feed air is separated into two gas streams. The first, the nitrogen product, is produced at a pressure approximately 2 bars lower than the compressed air pressure. The second, the waste stream, is enriched in oxygen and is at a much lower pressure, generally close to atmospheric pressure.

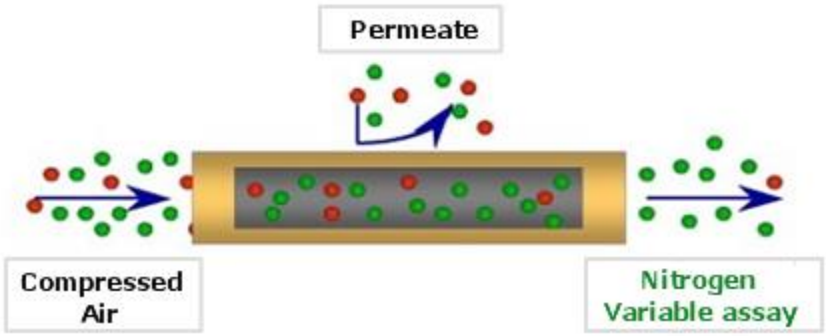

Figure 5 Schematic of membrane nitrogen generator

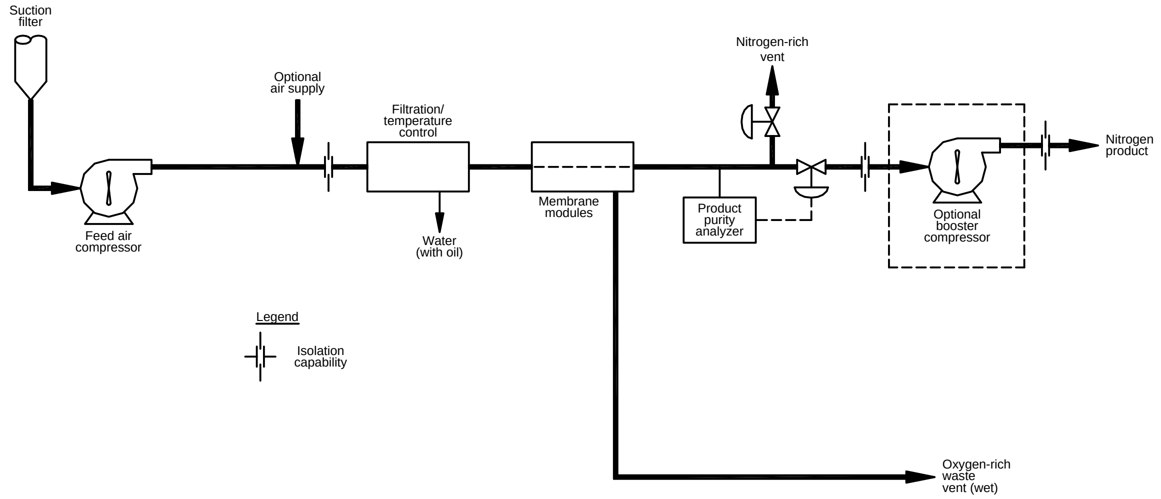

Figure 6 Typical membrane nitrogen generator flow diagram

Major equipment components include the feed air compression system; feed air pre-treatment equipment; membrane modules; process piping and valves; a nitrogen compressor (when the user's process requires higher pressures than the nitrogen membrane output pressure); process control systems and other auxiliary components such as separators, storage receivers, and instrument air systems.

The compressed air is generally treated to remove any condensed liquids, entrained mists, solid particulates, and sometimes vapour-phase contaminants before introduction into the membrane separator. The degree of clean up required depends on the particular contaminants present, the effects those contaminants will have on the performance and lifetime of the membrane, and the final product purity requirements. Pre-treatment steps typically include filtration, and temperature and/or pressure control. After pre-treatment, the clean compressed air is fed to the membrane separator(s), which can be arranged alone or in multiple parallel or series banks.

The nitrogen purity is affected by controlling the operating pressure, temperature, and flow through the membrane module.

Figure 7 Typical membrane nitrogen generator

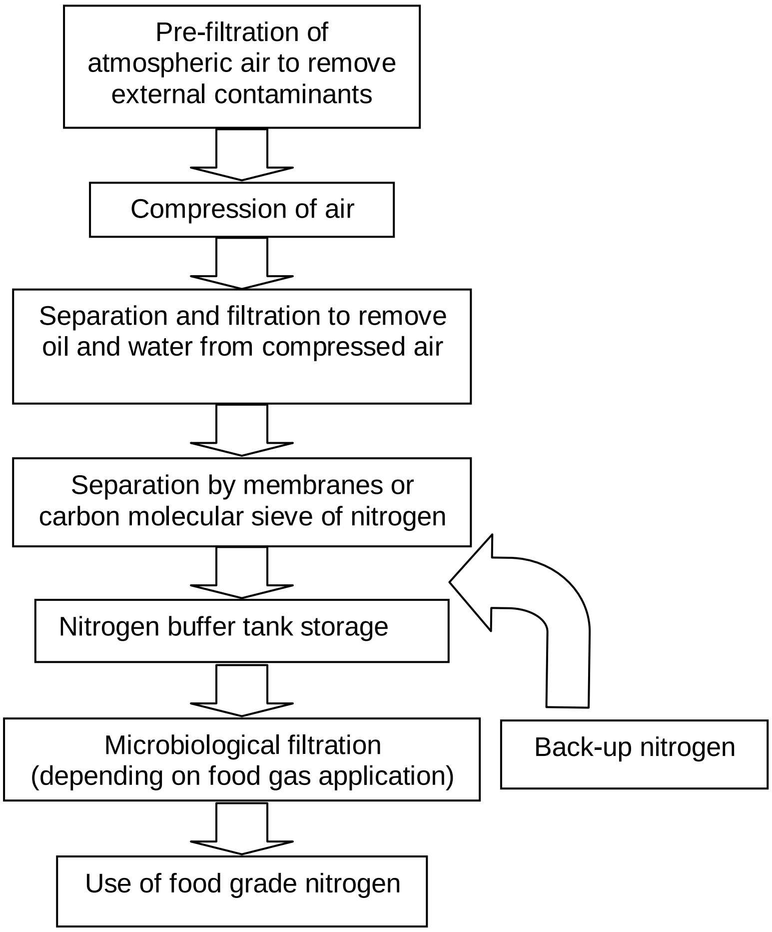

Figure 8 Typical flow chart of an on-site nitrogen generator

4.5 Cryogenic separation

The cryogenic process separates air by means of distillation. This process uses the different evaporation temperatures of the air components. There are inlet filters in the plants which remove external contaminants from the air before it enters the air compressor. Here the air is compressed to the required process pressure. After moving through an oil separator, the air enters one of two carbon molecular sieves (usually composed of CMS and alumina), where impurities such as water and carbon dioxide are removed. Here, one carbon molecular sieve is always effective while the other is being regenerated by residual gas from the separation process.

The processed air is then cooled at a liquefaction temperature in the main heat exchanger and then fed into the bottom of the distillation column. The pure nitrogen fraction is removed from the top of the column and fed into the product line. Cold is supplied in the form of liquid nitrogen (LIN) from the back-up system, which is regenerated with an expansion turbine. The pure nitrogen is stored in cylinders or storage tanks and then distributed.

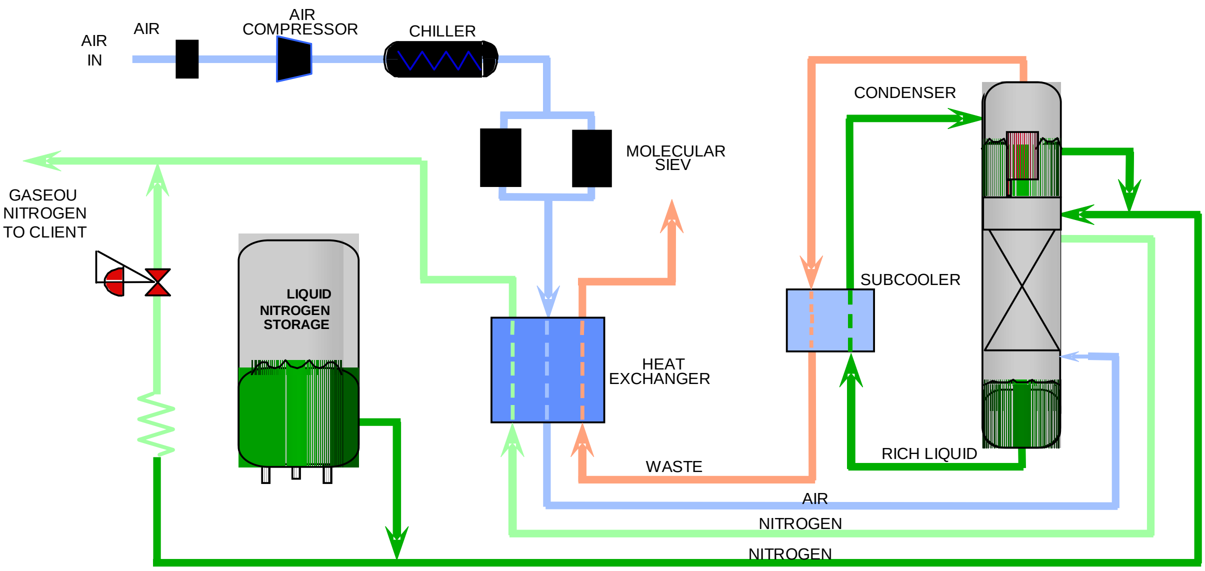

Figure 9 Typical cryogenic nitrogen generator flow diagram

4.6 Food safety risk assessment

On-site generators shall be designed and operated to produce gases suitable for the intended food applications.

This hazard analysis should be structured to systematically examine all relevant parts of the equipment design and operation including both normal and malfunctioning conditions, and interaction of the generator with its surroundings.

When carrying out this risk assessment study, general procedures can be used, which are described in EIGA Doc 149, (Section 5.11) [8].

In order to control the physical, chemical and biological contamination of the nitrogen output, particular attention should be paid to the following process elements:

- filtration of atmospheric air;
- mode of air compression (oil lubricated or non-lubricated);
- removal of oil, water and particles from the compressed air;

- materials for separation of nitrogen from oxygen with membrane modules (plastic polymers) or carbon molecular sieve;
- materials, design and construction of the nitrogen buffering tank;
- microbiological filtration of nitrogen if required by specific HACCP analysis; and
- nitrogen specification control with continuous control of the oxygen concentration.

As air is the raw material for an on-site gas production system, it's quality (type and degree of contamination with pollutants such as dust, allergens, pollens, odours, hydrocarbons, acid gases, carbon monoxide, nitrogen oxide, microbes, bacteria, viruses, spores) are the major considerations in selecting a suitable area of installation.

The physical location of the generator shall be subject to a comprehensive hazard analysis regarding physical, chemical and microbiological contamination of the nitrogen in order to identify potential food contamination hazards and to generate recommendations to reduce the probability of their occurrence and their consequences.

The generator should not be located adjacent to an obvious potential source of pollution (proximity to boiler combustion exhaust, solvents or chemicals venting, cooling air towers, aerosols, open or vented sewage systems, parking areas, main roads, etc.). Any modification of the adjacent environment of the on-site gas production system will require the food business operator to re-assess possible sources of additional pollution of the intake air and could possibly require the end user to stop the generator.

As regards the location of the installation, refer to EIGA Doc 149(Section 5.1) [8].

The use of best practice methods such as Good Manufacturing Practices (GMP), Standard Operating Procedures (SOP), Standard Sanitary Operating Procedures (SSOP), and Critical Control Point – Control Procedures (CCP-CP) within the on-site gas production process will ensure that contamination levels are kept within specification.

Where, as part of the on-site gas production process, water, oil or any consumables (for cleaning, washing or cooling) come in contact with the nitrogen stream their compliance should be controlled as defined in the EC Good Manufacturing Practices (GMP) guide using validated methods for substances approved for contact with foods.

5 Manufacture and installation

Regarding air environment solutions, additional specific filters and also active carbon, zeolite adsorbers or other materials suitable for food use shall be designed to eliminate gas pollutants and all unwanted impurities.

Regarding the inlet air compressor, a so called "oil-free" compressor should be the preferred option as it carries less risk of oil contamination because there is no contact between the oil and the product stream and there is therefore no requirement to maintain an oil/condensate separation and drainage system.

If a lubricated type air compressor is installed, attention shall be paid to the issues mentioned above, and an oil separation system shall be installed, controlled and maintained.

Regarding adequate design of membrane modules for membrane units, the ageing phenomena of modules and their polymer material should be considered: the membrane performance will reduce over time and after micro pollutant capture. Overall a membrane specific life-cycle management program shall be implemented.

Materials and equipment that come in contact with the nitrogen stream shall not contribute an additional food safety hazard. Where available, certificates of food compatibility for components, consumable materials or materials of construction will contribute to the overall food safety validation of the system.

In practice this means that the compatibility of the seals, lubricants, metals and plastics with foodstuffs shall be individually checked.

Consideration should be given to Regulation (EC) No 1935/2004 of the European Parliament and of the Council of 27 October 2004 on materials and articles intended to come into contact with food [9].

See also Commission Regulation (EU) *No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food* [10] regarding plastics.

NOTE There is a positive list, controlled by the European Food Safety Agency (EFSA) of authorized plastics for food use to.

A microbiological filter can be installed downstream of the on-site gas production system, after the gas back-up system or at the point of use.

Cleaning procedures requiring the use of only approved materials or equipment (for example washing/cleaning agents, lubricants, oils, ethylic alcohol, potable water) shall be used during manufacturing and installation.

An air humidity filtration or dryer and condensate trap system shall be installed before the membrane or carbon molecular sieve as applicable.

Labelling: E941 [1], name or formula nitrogen, “for food use” <u>shall</u> be applied to the generator and the buffer tank.

6 Guidelines for food safety hazard analysis

6.1 Potential Hazards

6.1.1 Biological Contamination

TYPE OF CONTAMINANT	POSSIBLE CONTAMINANT	REASON FOR TAKING INTO ACCOUNT OR NOT THESE CONTAMINANTS
Bacteria	Bacillus (sporulated bacteria) Clostridium (sporulated bacteria) Erypsipelothrix rhusiopathiae Corynebacterium Staphylococcus aureus Micrococcus Listeria monocytogenes Legionella Enteric bacteria (Klebsellia) Pseudomonas Brevibacillus	Bacteria which can be found in the environment (air, water or soil), and responsible for human pathology
Moulds	Aspergillus Fusarium	Moulds produce mycotoxins and are present in the atmosphere
Yeasts	Candida albicans	Main yeast presenting risks for humans and present in the atmosphere
Viruses	Not applicable	Does not present a food safety hazard.
Parasites	Not applicable	Cannot survive without host organism.

6.1.2 Physical Contamination

Type of contaminant	Possible contaminant	Reason for taking into account or not these contaminants
Solids > x micron	Carbon molecular sieve or other particulates.	Could present a hazard without correct downstream filtration.
Radioactive elements	Not applicable in normal conditions	No radiation source in the process and control of radioactivity of the air by governmental bodies
Others	Solid (metals, plastics) particles carried over	Will be present in the gas lines of the installation, Could present a hazard without correct downstream filtration.

6.1.3 Chemical Contamination

Type of contaminant	Possible contaminant	Reason for taking into account or not these contaminants
Residues in distribution system	Cleaning agents (i.e. solvents)	Hydrocarbons shall not be present in food products above 1 ppm (0.02 ppm for $C_2H_2F_4$ and $C_2H_2Cl_2$)
Impurities from air gas manufacturing process	Volatile compounds such as VOC, oxygen, water	Applicable for on-site generators depending on their location and malfunction of the on-site generator
Nitrogen product reactions	Products resulting of polymer degradation	Presence of polymer in the process (membrane materials, Teflon)
Metal contaminants and impurities	Arsenic, Lead, Mercury, Cadmium, aluminium, nickel, silver, cobalt.	Specific release limits (SRL) according to the "Metals and alloys practical guide for manufacturers and regulators" EDQM 2013 edition [11]. See EC 1881/2006 [12] and EC 22/2001 [13]
Others	Dioxins and Dioxin-like PCBs	Applicable for on-site generators depending on their location. See EC 1881/2006 [12] and EC 22/2001 [13]

6.2 Hazard evaluation

6.2.1 Example CCPs and PRP determination

Type of hazard	Q1 Is the mitigation procedure specifically designed to eliminate the hazard or reduce it to an acceptable level?	Q2 Is it necessary to establish a critical limit?	Q3 Is an immediate intervention needed?	Output	Suggested monitoring procedures
Bacteria	Y	N	N	PRPO-SOP	Pre-requisite programme.
Carbon molecular sieve	Y	N	N	PRPO-SOP	Pre-requisite programme
High oxygen-reduced shelf life of food.	Y	Y	Y	CCP	Online analysis – Shift to back-up nitrogen.
High moisture – reduced shelf life of food.	Y	Y	Y	CCP	Online analysis – Shift to back-up nitrogen.
Contamination from maintenance activities.	Y	N	N	PRPO-SOP	Pre-requisite programmes.

6.3 Pre-requisite programmes

Food safety risks are primarily managed by pre-requisite programmes applied to periodic maintenance procedures and schedules:

- Inspection of generator condition and operating parameters such as temperature and differential pressures across key components such as filters, if the differential pressure is too low or too high, the filter element shall be changed ;
- Visual inspection and exchange of oil filters, carbon beds, dust filters etc.
- Inspection and exchange of microbiological filter, if installed.
- Instrument calibration; and
- Re-assessment with the food operator of any modification in the adjacent environment that could lead to contamination of the atmospheric air supply to the site generator, for example a new car park.

7 Liquid and gas back-up systems

On-site nitrogen generators are often supplemented by installed liquid or gaseous back-up systems that are intended to maintain a supply of nitrogen to the user's process in the event of:

- generator downtime due to maintenance or failure;
- detection of off-specification generation: typically high oxygen or moisture, and
- high peak demand.

The back-up system shall comply with EIGA Docs 125 [6] and 126 [7].

Where, as part of the manufacturing process, impurities comes in contact with the process gas, the level of the impurities is monitored as defined in the EC Good Manufacturing Practices (GMP) guide using validated methods. This ensures that the introduction of contamination into the on-site nitrogen production is limited.

For cryogenic on-site equipment, refer to EIGA Doc 125 [6].

8 Traceability and batch definition for on-site nitrogen production

Any raw material in the food chain requires traceability in compliance with Regulation (EC) 178/2002 (European Food Act), Article 18 [2]. Since on-site generators are usually supplying only one gas to only one customer, a set of control parameters and quality critical criteria should be mutually defined and recorded in a process trail on both sites. Data may be made available online to the customer or vice versa. The recorded trail provides traceability in the sense of Article 18. Data generation and recording should be subject to a technical agreement defining the responsibilities on both sides.

The production of nitrogen can be divided into batches, according to a "period of production" defined by the operator (for example 1 hour). A batch definition may be not necessary, if a permanent trail recording with defined intervals of analysis is available.

The batch identifier for the nitrogen produced by the on-site generator is the period of time comprising continuous and uninterrupted nitrogen production with a minimum and maximum duration fixed by the operator.

The food business operator shall, for instance, define the parameters that are to be recorded and traced during each production batch:

- date;
- time;
- oxygen concentration / other specified parameters; and
- process online / offline status.

9 Verification

The food business operator shall review the validity of the food safety risk assessment / HACCP for the nitrogen generation system periodically.

10 Equipment maintenance

10.1 General considerations

A rigorous preventive maintenance program is a crucial element of any food safety program designed to maintain the nitrogen food grade compliance. Good prevention is better than a cure. The generator maintenance program shall include periodic functional checks of the plant shut-down system to ensure that it performs as required in an emergency. The operation of pressure and temperature gauges, pressure regulators, automatic valves, and controllers shall be checked, and purity analysers shall be calibrated.

During the maintenance of the nitrogen generator it is possible to use nitrogen from the back-up system where food grade quality or food additive (E941 [1]) nitrogen is present in the cryogenic tank or in the cylinders or bundles.

Particular care shall be taken in the areas where food grade nitrogen is produced in order to avoid contamination of the system during maintenance operations.

Maintenance personnel shall be familiar with all safety regulations and be made aware of all potential hazards.

One of the most significant personal hazards associated with pressure swing adsorption nitrogen generators is exposure to hazardous atmospheres inside the adsorber vessels while loading, removing, or inspecting the sieve material or its supports. Even when the generator is not in service, the sieve material can adsorb or desorb oxygen due to changes in the ambient temperature.

Nitrogen generators are often enclosed in cabinets that can be considered confined spaces. Such spaces can become oxygen deficient, see EIGA Doc 44, *Hazards of inert gases and oxygen depletion* [14]

10.2 Management

With regard to procedures relating to the hazard analysis and critical control points (HACCP), factors affecting food hygiene shall be identified and suitably controlled during the maintenance. These factors will be broadly associated with the people working within the workplace and the materials which can be in contact with the food grade quality nitrogen during the maintenance operation.

The following is a non-exhaustive list of pre-requisite factors that shall be considered and controlled in order to manage the food safety risk:

- For maintenance a check list of control points and operations shall be issued.
- Materials and spare parts in contact with the produced food grade quality nitrogen shall be suitable for use in food applications;

- Washing and cleaning products for the components in contact with the produced food grade quality nitrogen shall be suitable for food (potable water, ethanol, etc.).
- Hygiene of the operator and protection of the equipment in the area where food grade quality nitrogen is produced;
- Specific food safety training for the operator involved in the maintenance; Oil change frequencies and oil control parameters shall comply with the operation manual of the on-site nitrogen generator;
- Periodic calibration of the installed analysis instruments.
- Periodic maintenance of the dust filter(s) of carbon molecular sieves;

NOTE Upon completion of the maintenance, the purity of the nitrogen flow shall be checked to confirm it meets the product specification.

11 References

[1] Commission Regulation 2012/231/EC of March 9th 2012 *laying down specific purity criteria on food additives other than colours and sweeteners*

[2] Commission Regulation (EC) No 178/2002 of the European Parliament and of the Council of 28 January 2002 *laying down the general principles and requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety Directive 1333/2008/EC*

[3] Commission Regulation (EC) No 1333/2008 of the European Parliament and of the Council of 16 December 2008 *on food additives*

[4] EN ISO 21469: *Safety of machinery. Lubricants with incidental product contact. Hygiene Requirements*

[5] United States Code of Federal Regulations; http://www.gpo.gov/

[6] EIGA Doc 125 *Guide to the Use of Gases in Foods*; www.eiga.eu

[7] EIGA Doc 126 *Minimum Specifications for Food Gas Applications*; www.eiga.eu

[8] EIGA Doc 149 *Safe Installation and Operation of PSA and Membrane Oxygen and Nitrogen Generators* www.eiga.eu

[9] Commission Regulation (EC) No 1935/2004 of the European Parliament and of the Council of 27 October 2004 *on materials and articles intended to come into contact with food*

[10] Commission Regulation (EU) No 10/2011 of 14 January 2011 *on plastic materials and articles intended to come into contact with food*

[11] *Metals and alloys practical guide for manufacturers and regulators 2013 edition* www.edqm.eu

[12] Commission Regulation (EC) No 1881/2006 of 19 December 2006 *setting maximum levels for certain contaminants in foodstuffs (Text with EEA relevance)*

[13] Commission Directive 2001/22/EC of 8 March 2001 *laying down the sampling methods and the methods of analysis for the official control of the levels of lead, cadmium, mercury and 3-MCPD in foodstuffs (Text with EEA relevance).*

[14] EIGA Doc 44, *Hazards of inert gases and oxygen depletion*