

**Continuous Leak-Monitoring
in Stamicarbon designed high pressure
Urea Equipment with Loose Liners**

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All technical and other information contained herein is based on general Stamicarbon/DSM experience and within this limit is accurate to the best of our knowledge. However, no liability is accepted therefor and no warranty or guarantee is to be inferred.

1. Preface

Established in 1947, Stamicarbon is the licensing subsidiary of DSM, a leading producer of life science products, performance materials and industrial chemicals. Stamicarbon licenses proprietary processes, know-how and expertise developed and commercially proven by its parent company.

Royal DSM NV is a private corporation headquartered in the Netherlands. DSM is active world-wide in a number of branches of the chemical process industry and has a workforce of 18.500.

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2. Introduction

Pure urea solutions are not very corrosive, but ammonium carbamate, an intermediate in urea synthesis, is highly corrosive. For instance, carbamate solutions in contact with carbon steel will cause corrosion proceeding at rates of more than 900 mm per year.

To avoid corrosion by the synthesis solution Stamicarbon has developed strict specifications as to the quality of the materials in contact with the process fluids.

For this reason a corrosion-resistant layer is used to protect the carbon steel pressure vessel wall. This layer which is made of a special type of stainless steel, can be applied as a loose liner, as an overlay welding or as an explosion cladding.

With a loose liner a hazardous situation may arise if a leak occurs and carbamate containing fluids enters the space between liner and carbon steel. This leads to corrosion in the carbon steel and will weaken the pressure vessel wall.

For this reason Stamicarbon designed a leak detection system with continuous monitoring to allow safe operation of the equipment. Stamicarbon strongly advises to use a continuous leak monitoring system, which enables the plant operating staff to take adequate action.

3. System outline

The leak detection system consists of four parts:

3.1 Gaps between liner and pressure vessel wall

It is known, that the liner welds are the most critical parts as regards to leakage due to weld defects or due to the application of the liner.

In case of a leak detection should be possible by means of passageways or by the gap between liner and pressure vessel wall and by two leak detection holes per liner compartment in the pressure vessel wall.

In recently fabricated HP equipment, Stamicarbon has advised a network of small longitudinal and lateral grooves along the liner welds attached to the carbon steel, this to ensure the passage of gases.

Note: A liner compartment is defined as that part of a lining which is comprised between two welds connecting the lining material to the carbon steel of the pressure vessel wall.

3.2 Piping

The piping between the leak detection holes and the leak detector should be provided rather under the equipment insulation or on the outside, but heated in order to avoid blockage due to crystallization of fluid leaking out of the equipment.

3.3 Monitor

An electrochemical sensor that allows a continuous ammonia gas detection.

3.4 Leak tracer

Ammonia is used as an indicator for a leak. Ammonia is present throughout the whole synthesis section of the plant and is easy to detect and emerges through moisture.

4. Features

The features of the Stamicarbon leak detection system are:

1. Accurate detection.
2. Detection of a gas leak of $1 \cdot 10^{-7}$ STD cc/sec is possible.
3. The response time (i.e. the time elapsing before a leak is detected) is less than 60 minutes.
4. The location of the leak can be traced.
5. The leakage rate can be calculated.
6. More than one piece of equipment can be connected to one gas monitor.

Note: Leakage rates are normally indicated in STD cc/sec. (standard cubic centimeters per second).

It should be taken into account that the product composition varies throughout the HP synthesis. Therefore the composition of fluid leaking behind the liner will also differ from one place to another and thus the ammonia concentrations will differ. This will influence the ammonia concentrations measured.

A fast detection method is needed because, when liquid-phase product is leaking away, also urea will enter the space between liner and carbon steel. This fluid will partly dissociate and fill the space. Finally it will crystallize as biuret or triuret, with the risk that detection becomes impossible due to blockage of the passageways.

5. Operating principles

The gap between the carbon steel pressure vessel wall and the liner, together with the leak detection holes, form a closed circuit with the piping used. The air present in this system is brought to the monitor with help of a pump. This monitor provides a continuous indication of gaseous ammonia concentrations. The monitor operates with help of an electrochemical sensor, allowing to measure ammonia concentrations up to 1,000 ppm in a mixture of gases.

Stamicarbon has two systems for leak detection available.

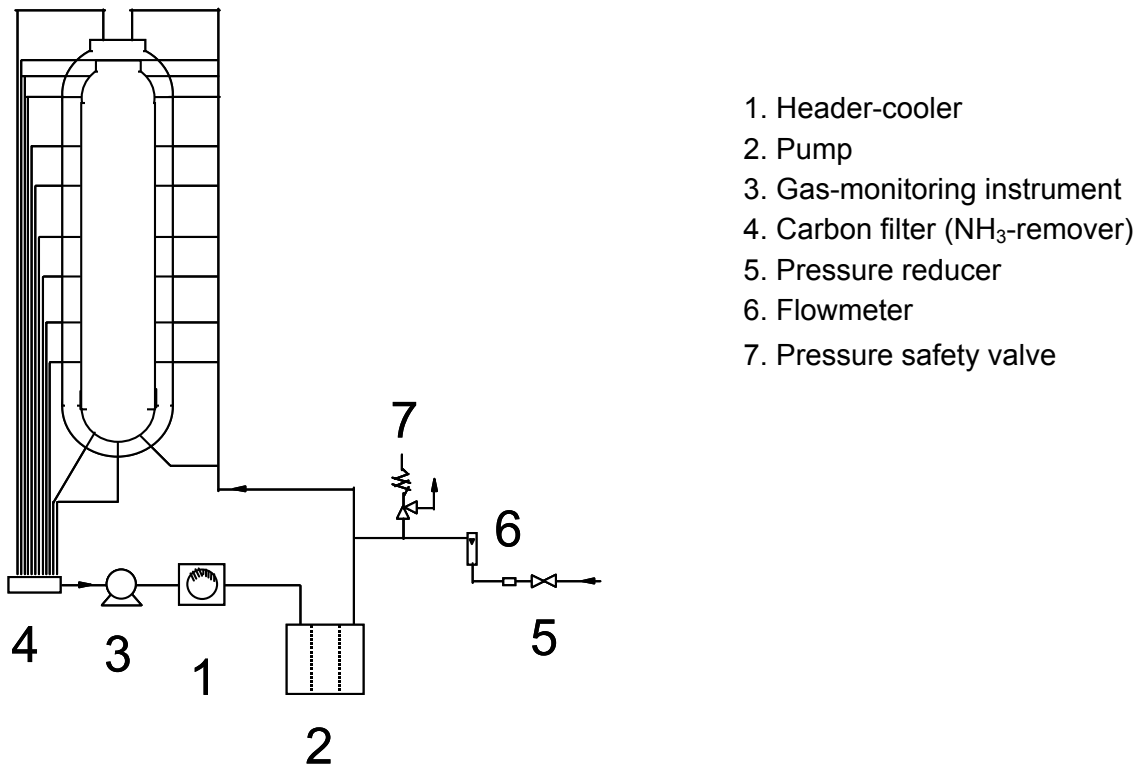
- A flow type leak detection system.
- A vacuum type leak detection system.

The selection whether a flow type or a vacuum type leak detection system should be utilized, depends on the possibility to circulate gas through the liner compartments.

5.1 Flow type leak detection system

The flow type leak detection system can be used in all new equipment since they are equipped with grooves passaways systems.

Figure 1: Flow type leak-detection system



In the gap between liner and pressure vessel wall air is circulated by a small air pump via piping.

The temperature in the system should be kept at minimum 150°C to avoid crystallization of leaking fluid. During this circulation, the resistance is not the same in each liner compartment. To equalize this, a flow meter and a control valve is required in the piping to the header to optimize the flow in each liner compartment. The piping of each liner compartment ends in a header/cooler in which the gas cools down.

A small part of the gas is fed into the monitor in which ammonia is analyzed continuously. The other gases circulate back into the leak detection holes via a carbon filter to prevent that ammonia accumulates in the system.

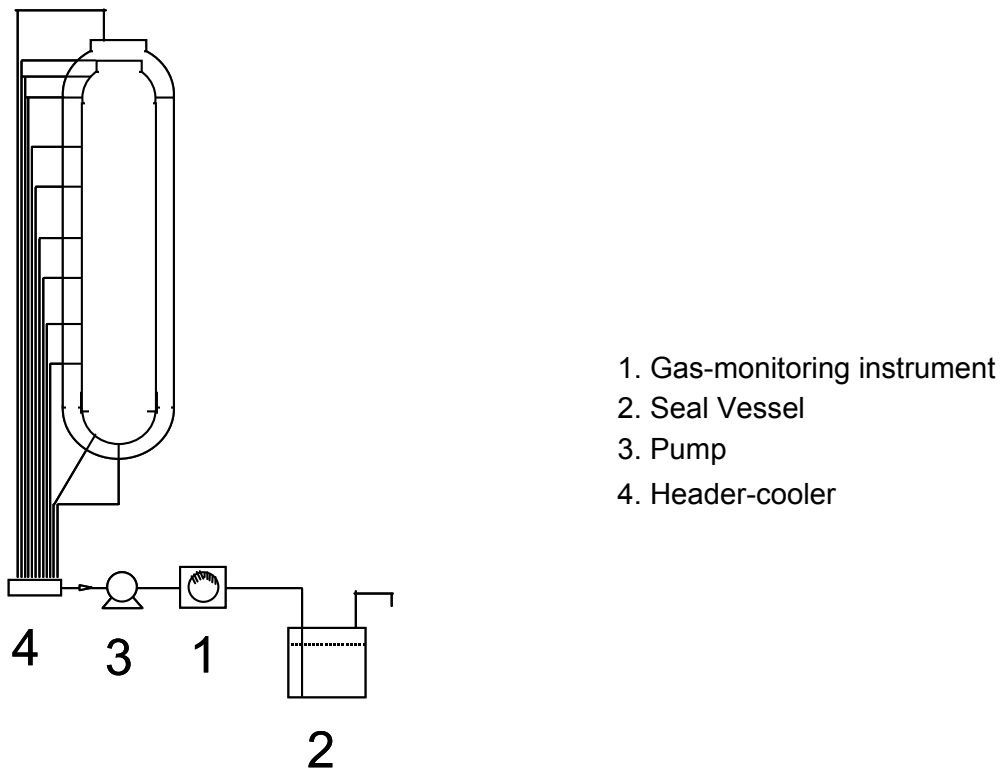
A reducing valve permits supply of instrument air into the system. If the pressure drops as a result of a leak developing in the connecting tubing or as a result of a shut-down, air will be supplied too. A flow meter is used to signalize such a pressure drop.

A safety valve prevents that the pressure in the system becomes too high.

5.2 Vacuum type leak detection system

If there is no passaway system present between lining and pressure vessel wall, it is advisable to use the vacuum system.

Figure 2: Vacuum Type Leak Detection System



Fluids leaking from a liner are continuously sucked to a monitor by means of a vacuum pump.

The temperature in the system should be kept at minimum 150°C to avoid crystallization of leaking product. The piping of each liner compartments ends in a header/cooler, in which the gas is collected and cooled down.

Then the gas is sucked into a gas monitor in which the ammonia concentration is continuously measured. Gases leaving are discharged through the flow indicator and a seal vessel. The seal vessel avoids that the environment contaminated with ammonia may enter the leak detection system.

6. Locating leaks

The liner compartment, in which a leak occurs can be traced by searching each compartment with help of the valves on the header. When the leaking compartment and the ammonia content is known, the leak rate can be calculated. This rate gives an indication about which method to use for locating the leak.

There are three methods for locating a leak:

- Bubble test.
- Ammonia leakage test
- Pressure leakage test.

These methods have been described in a previous paper, "Leak-detection in Stamicarbon's HP Urea equipment with loose liners".