GUIDANCE FOR INSPECTION OF ATMOSPHERIC, REFRIGERATED AMMONIA STORAGE TANKS

2008

Cfma european fertilizer manufacturers association

GUIDANCE FOR INSPECTION OF ATMOSPHERIC, REFRIGERATED AMMONIA STORAGE TANKS

SECOND EDITION

(First Edition 2002)

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EFMA European Fertilizer Manufacturers Association

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Disclaimer

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Users of this Booklet are advised to consult their latest national regulations before carrying out their own inspections, as changes in the regulations may have been made since its publication.

1. SCOPE

This document, produced by EFMA, provides guidance for the periodic in-service inspection of fully refrigerated anhydrous liquid ammonia storage tanks, which operate at or near atmospheric pressure and -33°C and are located in Europe. The Guidance focuses on major periodic inspection, covering its periodic frequency, method of inspection and regular monitoring between major inspections. It does not cover fabrication inspection. In considering the inspection frequency it describes as an option a risk based inspection (RBI) approach requiring the evaluation of the probability and consequences of failure for each individual tank. The underlying intention is to maximise the operational safety and reliability of these tanks.

2. INTRODUCTION

The practice of the inspection of storage tanks, which contain anhydrous liquid ammonia at atmospheric pressure, is not uniform in various countries in Europe. One of the reasons for this is that commonly used regulations relating to pressurised systems do not apply to these storage tanks; because they essentially operate at atmospheric pressure. Whereas in some countries e.g. Austria and Belgium there are regulations specifying the frequency of inspection for these tanks, in some other countries industry codes have been prepared for this purpose e.g. United Kingdom [Ref. 1]. On the other hand, in several other countries e.g. Germany, Greece, Italy and Portugal, there are no specific regulations or codes concerning inspection requirements for these tanks. Some companies have their own internal standards or they supplement the national regulations or industry codes with their own internal standards or codes of practice. Ammonia storage tank systems have to comply with a number of more general safety regulations in most countries. Of particular importance in this regard is the need to comply with specific regulations arising from the SEVESO Directive [Ref. 2], which specifies several safety related requirements relating to process operations including maintenance.

In revising this Guidance, EFMA carried out two types of surveys of tanks operated by its members. The first type, which covered 22 tanks, dealt with the design and construction aspects of the tanks and was the basis for Chapter 3. It showed that virtually all tanks have some form of secondary containment provision to retain liquid in the event of a failure. Of these, more than 80% are of full height concrete or steel wall construction. Most of the tanks have a single roof, whereas some tanks have two independent roofs. In Europe, there are more than 50 refrigerated ammonia storage tanks in operation.

The second more detailed survey, based on 48 tanks, covered factors which affect failure probability and failure consequences. The results of this survey provided the basis for the Risk Based Inspection (RBI) matrix explained in Chapters 4 and 5.

The main purpose of this document is to provide guidance and recommendations for the periodic inspection of fully refrigerated anhydrous liquid ammonia storage tanks. The Guidance is based on experience gained from inspection of ammonia tanks and the knowledge of potential failure mechanisms, which can affect the integrity of the tanks, in particular, stress corrosion cracking (SCC) induced by ammonia under certain conditions.

The Guidance covers the three main stages in the overall process of inspection management viz, determination of periodic frequency by legislation, industry code or a risk based inspection (RBI) approach or other options, methods of major inspections (intrusive and non-intrusive) and monitoring between inspections. Figure 1 summarises this overall approach.

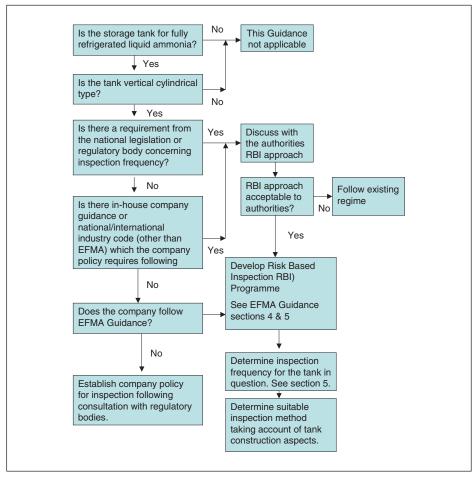


Figure 1 Overall Approach for Inspection

The Guidance describes the RBI approach as a way to optimise the inspection programme between the need for knowledge about the condition of the tank and the negative effects of opening the tank for inspection which could increase the potential for SCC.

Risk based inspection involves the planning of an inspection on the basis of the information obtained from a risk analysis of the equipment. The purpose of the risk analysis is to identify the potential degradation mechanisms and threats to the integrity of the equipment and to assess the consequences and risks of failure. The inspection plan can then target the high risk equipment and be designed to detect potential degradation before fitness for service could be threatened.

The process of risk based inspection should form part of an integrated strategy for managing the integrity of the systems and equipment.

Application of these recommendations requires an appropriate level of competence and experience of ammonia storage tank design and operations.

3. DESCRIPTION OF SPECIFIC AREAS OF CONCERN

3.1 Ammonia Storage Facilities

Liquid ammonia is stored either at ambient temperature under high pressure or at -33°C under atmospheric pressure. (The description *liquefied* is also sometimes used for *liquid*, see Glossary for explanation). In some cases, it is also stored at intermediate temperatures and pressures (semi-refrigerated). For pressure vessels, the inspection requirements in most countries are governed by the respective pressure vessel codes and regulations. The recommendations provided in this Guidance are, therefore, limited to atmospheric pressure storage tanks, which operate at -33°C.

3.2 Types of Ammonia Storage Tanks

Illustrations of different types of storage tanks are shown below. The main types of atmospheric tanks operating at -33°C in Europe are:

- a) Steel tank with full height concrete bund wall close to it with capacity to contain the full contents of the tank and the space between the tank and the bund having an impervious floor and roof covering (see Figure 2).
- b) Steel tank housed within another steel tank to contain the full contents of the tank, with a single roof (cup in tank) or independent roofs (see Figure 3).
- c) Steel tank with a partial height concrete bund wall with impervious floor within the contained area and no roof over the space (see Figure 4).
- d) Steel tank with an embankment of earth to contain the full contents of the tank and no roof over the space between the tank and the embankment (see Figure 5).
- e) Single steel wall tank with no secondary containment (see Figure 6).

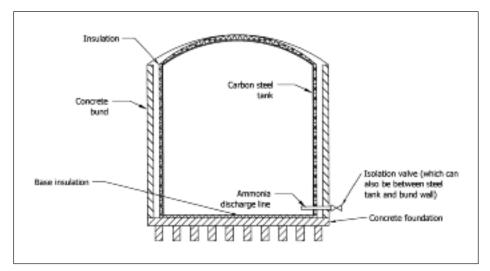


Figure 2 Tank with full height concrete bund



Picture 1 Steel tank surrounded by a concrete wall

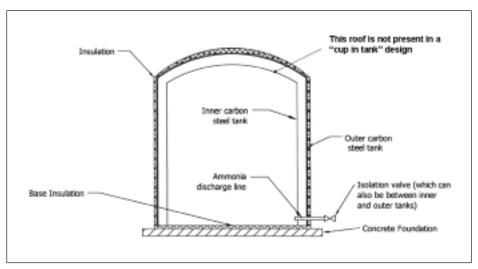


Figure 3 Tank with steel outer and inner walls with separate roofs



Picture 2 Tank with steel outer and inner walls with separate roofs

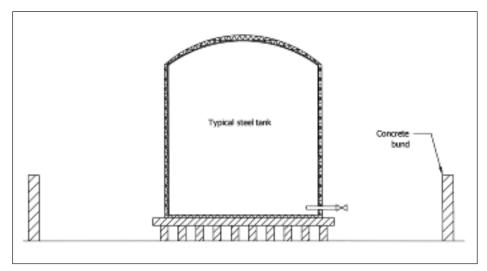


Figure 4 Tank with remote concrete bund

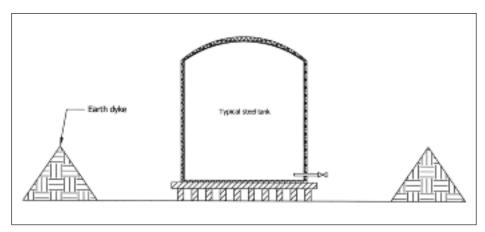


Figure 5 Tank with bund of earth dyke



Picture 3 Single steel wall tank with earth dyke

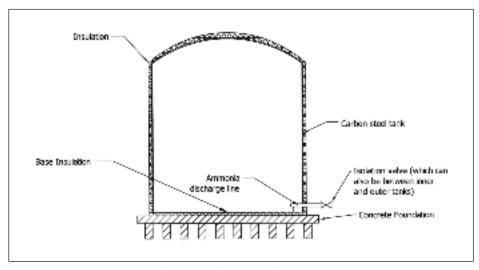


Figure 6 Tank with no secondary containment

As can be seen from the figures above, there are two main types of **foundation**:

- tank resting on concrete plinths such that the ground below is not exposed to freezing conditions due to ammonia; therefore, the heating of the ground below the tank base is not necessary
- the tank sits on a suitable foundation directly on the ground. This arrangement requires heating of the foundation to prevent it freezing.

Bund is a wall of brick, stone, concrete or other suitable material or an embankment of earth, which provides a barrier to retain liquid.

Since the bund is the main part of a spill containment system, the whole system (or bunded area) is generally referred to within industry as the "bund".

Its capacity and strength should be designed so as to be capable of containing liquid ammonia that may be released from the (full) tank in an accidental situation. It should be able to contain spillages and leaks of ammonia stored or processed above-ground and to facilitate clean-up operations.

A bund generally consists of:

- · an impervious bund wall or embankment surrounding the facility or tanks
- a floor (preferably impervious) within the bunded area
- any joints in the floor or the wall or between the floor and the wall
- any associated facilities designed to remove liquids safely from the bunded area without polluting the environment.

3.3 Ancillary Equipment

It is expected that the tank operation is in accordance with best available operating procedures based on HAZOP or similar process risk evaluation tools. The design of individual storage tanks and their associated ancillary equipment can vary between installations. Typical items that require systematic attention during life time of tanks include:

- · Relief valves.
- Nozzles.
- Drainage systems.
- Insulation: at the roof, wall and in the bottom.
- Heating system for foundations (where installed).

The procedure described in this document is not considered to be valid if these items are not effectively operated and maintained. Where appropriate, they should be included as part of a systematic schedule for maintaining the tank and its associated ancillary equipment.

3.4 Design and Materials of Construction

Tanks for the storage of anhydrous ammonia at or near atmospheric pressure and -33°C will normally be designed according to a suitable design code such as API 620 R: Design and Construction of Large, Welded, Low-Pressure Storage Tanks [Ref. 3], EN 14620 [Ref. 4]; or similar codes.

3.4.1 Materials of construction

Materials for atmospheric ammonia tanks are selected to satisfy the requirements specified in the design codes. The standard type of material is low temperature certified carbon manganese steel, impact tested at or near -40°C. The susceptibility to stress corrosion cracking increases with increasing yield strength of the steel. Materials with minimum yield strength between 290 and 360 MPa are often used. For new tanks, the use of material with minimum yield strength in the lower part of the above-mentioned range is recommended.

Various types of welding materials are used in construction, but often with a considerably higher strength level than the base material. Compatibility of yield strength level between weld and base material is an important parameter for resistance against ammonia stress corrosion cracking. Some typical data for welding consumables are shown in Appendix 2.

3.4.2 Pressure relief devices

There are a number of industry codes which specify the design of pressure relieve devices e.g. API 620/API 2000, EN 14620. These requirements should be applied to the construction of new tanks.

Since the inspection frequency for pressure relief devices is higher in most cases than that of the tank, due care shall be given to the inspection and testing requirements of these devices in order to prevent interference with the inspection regime of the tank itself.

3.4.3 Construction documentation

It is important that detailed records are kept of the quality inspection activities during tank construction and fabrication in order to enable an accurate RBI evaluation to be carried out, *in particular material toughness properties*.

3.5 Factors Affecting the Integrity of Ammonia Storage Tanks

As with all other constructions, ammonia tanks can be affected by their internal and/or external environment. Ammonia is not generally corrosive to the materials selected for tank construction. The contaminants normally found are oil and water, but the quantities are normally small. With regard to water, this inhibits SCC and therefore has a positive effect to service life. Oil has no negative effect on service life.

3.5.1 Original weld defects

Ammonia storage tanks are constructed according to appropriate design standards, such as API 620 R, EN 14620 or equivalent. These standards have requirements for the inspection of welds by radiographic (RT) and magnetic testing (MT) to ensure the quality of the welds is of the required standard,. The quality and integrity of the welds prior to first commissioning are vital for the future life of a tank, particularly in the initiation and propagation of SCC under ammonia duty. Residual stresses and local hardness peaks should be minimised by sound welding procedures and the appropriate heat treatment.

3.5.2 Corrosion

External corrosion of the tank due to atmospheric conditions is prevented by appropriate paint and/or by the application of insulation containing a vapour membrane that reduces the ingress of atmospheric moisture. It is worth noting that at the storage temperature of -33°C the corrosion rate is negligible. The roof may be attacked externally by general corrosion, particularly where the insulation is inside the tank and consequently the roof tends to be close to atmospheric temperature. The roof should be regularly inspected and, where possible, repaired without interruption of service. It is important that the condition and integrity of the insulation and vapour membrane on all areas of the tank are considered as part of the overall inspection assessment.

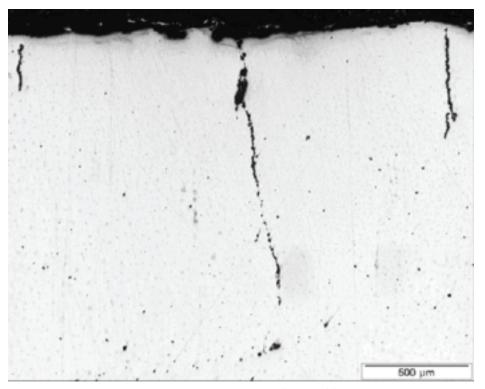
The ingress of oxygen during the emptying of the tank, or caused by leakage in the safety valves can theoretically cause some corrosion in the upper part of the wall. However, in practice, oxygen is effectively removed because of the continuous cooling by compression. No detectable deterioration has therefore been found internally due to general corrosion.

3.5.3 Stress corrosion cracking

Stress corrosion cracking is a phenomenon which can occur in metals exposed to a combination of stress and corrosive environment. The corrosive environment will, under certain circumstances, destabilise the protective oxide layer, without causing general corrosion. This destabilisation is sufficient to prevent the reformation of oxide after a crack, caused by stress.

Liquid ammonia in the presence of oxygen can cause SCC in carbon steels. The probability of SCC increases with increasing yield strength of the plate material, increasing strength of the weld metal and local hardness in the welds.

The stress levels required to initiate such cracking are high and are not experienced during normal operations. However the residual welding stress levels in high and medium strength materials or welds with over matched strength, together with the applied stresses, can be enough to initiate SCC if oxygen is present in sufficient quantities.

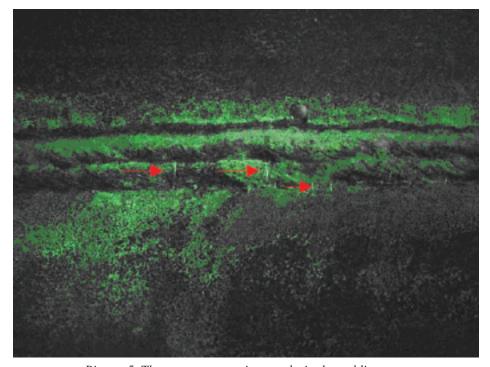


Picture 4 Cross section of crack caused by SCC

Since the late 1980s stress corrosion cracking has been detected in some storage tanks operating at -33°C. Based on experiences from findings and extensive international research work, it appears that the commissioning and to an even greater extent recommissioning are critical phases in the formation of cracks. This is due primarily to the potential for increased oxygen levels inside the tank and temperature variations causing increased stress levels.

Much research work has been carried out to understand the SCC mechanism and the relevant factors [Refs. 5-16]. The main conclusions concerning SCC in ammonia tanks from this work combined with practical experience are:

- SCC is difficult to initiate at -33°C.
- SCC initiation requires applied and/or residual stress levels greater than the yield stress.
- 3) SCC initiation requires the presence of oxygen.
- 4) The presence of water inhibits the formation and growth of SCC.



Picture 5 Three stress corrosion cracks in the welding zone

- 5) Where SCC is found in low temperature tanks, the defects are in general very small (less than 2 mm deep). However, a few exceptions with larger defects have been reported.
- 6) Commissioning and in particular recommissioning is a critical period for the formation and growth of SCC.
- 7) Knowledge and experience of SCC has led to the improved operation of ammonia storage tanks. Due to this, recent experience indicates that the problem occurs less frequently, even in tanks where extensive cracking has been detected earlier.

The phenomenon of SCC is rare in low temperature tanks due to the need for the presence of oxygen to catalyse the process and the low temperature that slows the process.

The dependence of SCC on water content and oxygen concentration in ammonia is shown in Figure 7. Information on the method of analysing oxygen in ammonia is given in Appendix 4.

Experiments at lower temperatures (-33°C) show that SCC can occur in about the same range of oxygen and water content compared to ambient temperatures, although it is much more difficult to initiate stress corrosion cracks at -33°C than at ambient temperature.

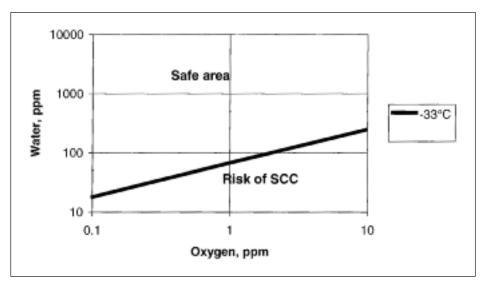


Figure 7 Relationship between water and oxygen content of ammonia and the risk of SCC

Actions to improve service life by shot-peening or cathodic protection are considered to be non proven technology and hence have not been included as beneficial for protection against SCC in ammonia tanks.

Quantification of the probability that a critical crack may develop is based on documented experience with ammonia tanks. A few hundred tanks are estimated to be in operation worldwide, representing about several thousand tank years. Although properly documented inspection results have only been published for relatively few tanks, it is reported that 5 fully refrigerated ammonia storage tanks in Europe had developed ammonia stress corrosion cracks [Refs. 17 and 18].

It should be noted however that critical defect sizes can vary between tanks due to variations in the strength and fracture toughness properties of the actual weld and plate materials, applied stress and residual stress levels.

SCC in fully refrigerated ammonia storage tanks is the main *internal degradation* mechanism which has to be taken into consideration when planning and executing an inspection programme. However, external factors for degradation, such as external corrosion, settling etc., have also to be considered.

3.5.4 Low cycle fatigue

Fatigue has been raised as a possible failure mechanism that may occur because of the long lifetime of an ammonia storage tank. Typical import tanks are filled and emptied every 1-2 weeks. The number of cycles during a lifetime is in the range 50 times/year times 40 years = 2000. Provided there are no significant defects present, this is far below the number of cycles that would be required to cause fatigue under normal operating conditions. Fatigue is therefore not considered to be relevant, unless special conditions may change the number of cycles or the stress levels are far above design specifications.

3.6 Indications from Accidents

A survey of the AIChE Ammonia Safety Symposium proceedings was carried out in order to identify the important relevant factors which affect the integrity of tanks in practice. 18 incidents were found and their types of failures were identified and are listed in table 1.

Table 1 Summary of incidents and basic causes

Type of failure	Basic cause	Can it be discovered / prevented by internal or external inspection of the tank?	Number of occasions
SCC	Corrosion caused by combination of oxygen, ammonia, stress and carbon steel	Yes. However, intrusive inspection can be part of the cause	4
Filled annular space of double wall tank with 'cup in tank' design	Result is the floating of inner tank damaging the construction. a) Leak in the inner cup tank b) Ammonia condensation in annular space between the inner and outer tanks c) Splashing of ammonia over the edge of the inner tank	Yes, only when the root cause is a leaking inner tank. Otherwise, no, because the incident is caused by operational failures or design problems	3
Foundation failure due to frost heave	Freezing up and formation of ice lens under the tank caused by a) Defect of bottom heating tubes (two instances) b) Insulation defect caused by earthquake	By regularly checking the functioning of the bottom heaters this can be prevented. So this check must be part of the regular inspection programme	3

Table 1 Summary of incidents and basic causes (continued)

Type of failure	Basic Cause	Can it be discovered / prevented by internal or external inspection of the tank?	Number of occasions
Overpressure causing complete tank failure	a) Warm ammonia was injected in the tank. b) Sudden mixing of ammonia solution and liquid ammonia when an oil layer between these phases was broken up	No. Incidents were operational mistakes	2
Vacuum causing tank collapse	Failure of the pressure transmitters and a failing vacuum relief valve	The checking of the safety provisions must be part of the regular inspection programme	1
Failure of roof to wall weld	Poorly designed weld with high stress low cycle fatique	Yes	1
Leaking bottom	Improper welding techniques	Yes. In this case the leaks were detected by ammonia vapour around the tank	1
Leaking roof	Poor repair of a construction flaw	Yes	1
Leaking wall	Parent material was used that did not meet the specifications. Fatigue crack developed	Yes	1
Overflow from tank	Misunderstanding of level readings by operators in combination with failure of high level alarm	Operational error in combination with a defective high level alarm. The checking of the safety provisions must be part of the regular inspection programme	1

Historical records indicate that some major tank failures occurred due to a sudden pressure increase. This can happen for various reasons such as:

- exothermic effect of mixing aqueous ammonia or water and anhydrous ammonia during tank commissioning (See Chapter 7, paragraph 3a)
- · unintentional injection of warm ammonia
- flow of syngas due to gas break-through.

The sudden pressure increase caused by the above mentioned failures are such that the pressure relief valves often cannot deal with the amount of gas. This can then result in a failure of the tank. When the weld between the tank bottom and the cylindrical shell is the weakest point, the shell will lift free from the bottom and the ammonia in the tank will be released. For this reason, it is preferable to include in the design of new tanks a weak point at the top to roof weld that fails before the bottom to cylindrical shell weld fails. Such a design change is not applicable to existing tanks. According to API 2000 emergency venting can be accomplished by a gauge hatch that permits the cover to lift under abnormal internal pressure.

When a sudden pressure increase incident occurs that results in the lifting of the cylindrical shell, the liquid outlet piping that is attached to the cylindrical shell can also be damaged. Attention should be given to this possible failure scenario even when a double tank wall is used, since the liquid outlet piping also passes through the outer wall. A damaged outlet pipe can adversely affect the integrity of a double containment tank.

The issue of weak roof to shell attachment is described in the section of API 2000 related to 'Non Refrigerated tanks' as well as API 650. For new tanks consideration should be given to the incorporation of this feature.

4. INSPECTION STRATEGY

The inspection of low temperature ammonia tanks is a compromise between a need for knowledge about the tank condition and the negative effects of opening the tank for inspection, which will cause thermal stress and allow the ingress of oxygen. For ammonia tanks, it is known that decommissioning and recommissioning tends to increase the risk for SCC initiation. The need for inspection and its method, type and scope, therefore, should ideally be evaluated on the basis of the risk and consequence of a failure. Applying RBI means that these factors can be considered and the inspection programme can be established for each individual tank. In practice, however, frequencies of inspection may be 'imposed' by national legislation or industry code. A company may wish to follow a RBI approach and if the result of this is in conflict with the national legislation/code the company may consider taking up the matter with the relevant authorities.

Various steps, which form the main elements of the RBI approach and methodology, are illustrated in Figure 8.

It is essential that the design, construction and operating history of the tank are reviewed with the responsible engineers and operators during the formulation of an inspection strategy.

It is also important to be familiar with and consider any local conditions that may influence the tank inspection programme: e.g. ambient conditions, local soil conditions, etc.

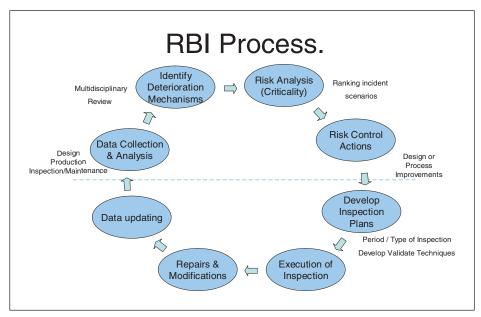


Figure 8 Steps in RBI Process

RBI and the associated structural integrity calculations can help to establish a tank inspection strategy that includes:

- Definition of the most appropriate inspection methods.
- Determination of the most appropriate tank monitoring requirements, including internal and external inspection aspects.
- Establishment of prevention and mitigation steps to reduce the likelihood and consequences of a tank leak or failure.

Different storage tank applications have unique design systems and conditions that must be considered when evaluating the tanks. It is therefore essential that experienced and competent engineers and inspectors are involved in evaluating existing tanks.

The application of RBI to an ammonia tank requires an evaluation of the following factors:

Failure Probability:

- 1. SCC related question
 - 1.1. Oxygen and water content
 - 1.2. Plate and weld material properties
 - 1.3. Pipe connections

- 1.4. Inspection issues
- 1.5. Repair
- 2. Other degradation mechanisms
 - 2.1. External corrosion
 - 2.2. Mechanical damage
 - 2.3. Low cycle fatigue
 - 2.4. Brittle fracture
 - 2.5. Others
- 3. Operational issues
 - 3.1. Pre-commissioning control
 - 3.2. Commissioning procedure (inert purging, cooling rate)
 - 3.3. Operating experience

Failure Consequences:

- 1. Release of ammonia to the atmosphere, extra external safety (tank design, bund)
- 2. Leak before break assessment
- 3. Location of the tank (close to population and watercourse)

5. INSPECTION

5.1 Competence and Independence

A high level of competence and experience is required in order to execute a thorough and effective assessment of the factors which may affect the integrity of tanks and the management of inspections. It is important that reliable data are used for the evaluation and it is essential that those involved have the required knowledge and experience to assess the influence of any uncertainties in the data used on the accuracy of the calculation.

The application of fracture mechanics codes requires a high level of technical expertise and practical experience. Great care is essential in the selection of personnel to carry out such work.

A group of people covering the areas of inspection, engineering/maintenance, operation and process safety should be involved in the evaluation. This team should have the appropriate degree of independence necessary to act impartially in all matters relating to the inspection of the tank.

5.2 Assessment for Inspection Frequency

The purpose of this assessment is to establish the basis for a risk based inspection programme. It covers the relevant parameters that can affect failure probability and failure consequences, as described in Section 3 and various safeguards described in Section 4.

The evaluation will position each tank in an inspection frequency zone in an Inspection Frequency Diagram (Figure 9) as described below.

The Inspection Frequency Diagram has been developed, based on results of surveys carried out within EFMA member companies. The 2007 survey covered 48 ammonia tanks (44 based in Europe, representing more than 80% of European capacity) and consideration of the prevailing legislation/standards.

Whereas national codes are normally based on setting maximum limits for inspection periods, the diagram also provides a means of evaluating tanks that may be more vulnerable, where an improvement programme is required and where the period between inspections may need to be reduced.

The diagram, therefore, provides a means of optimising tank inspections, intrusive or non-intrusive, based on RBI techniques and the practical experience of most European tanks.

The Inspection Frequency Diagram is based on standard RBI processes that have been modified and developed to give a suggested inspection frequency for ammonia tanks based on an evaluation of failure probability and failure consequence factors.

The detailed question list of the RBI assessment is given in Appendix 2. Each question is given a number of points between 0 and 10. The questions have been allocated different weighting factors to take account of the different levels of importance of the various factors considered. The score per question is a multiplication of the points and the weighting factor. The higher the score for each question, the higher is the probability or consequence of failure. The total score on the probability ranking is the sum of the scores of all the probability questions and this total will be between 0 and 100. The same holds for the total score for the consequence ranking. The probability of failure and the probability of consequences are plotted in the RBI matrix diagram in Figure 9.

The matrix shows 5 levels of risk areas identified by different colours and these risk areas are linked to different inspection frequencies.

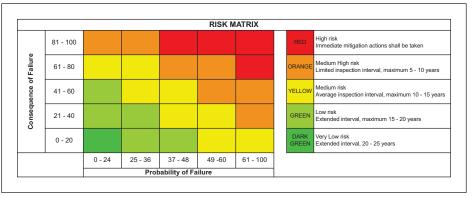


Figure 9 RBI Matrix: Inspection Frequencies as a function of failure probability and failure consequences

20-25 Years

Very low risk. Indicates an ideal situation, where the tank properties are state of the art and the consequences of a failure are at a minimum.

15-20 Years

Low risk. The tank has good properties and the consequence of a failure is well taken into account. Details about properties and/or location determine the given inspection interval.

10-15 Years

Medium risk. The tank has acceptable properties. Details about properties and/or location determine the given inspection interval.

5-10 Years

The tank has some elements in its design or the way it is operated that make an inspection necessary applying a higher frequency compared to the indicative industry average of 10-20 years.

<5 Years

The probability for and/or consequence of a failure is not considered to be in accordance with the indicative industry average. It is recommended that the tank should be subject to an improvement programme

It should be noted that these inspection frequency positions on the diagram provide guidance for the frequency of inspection of the tank. The objective of such inspections is to determine the physical integrity of the tank, for example, to check the effects of general corrosion and SSC. This is only one element of an ongoing programme for ensuring the integrity of the systems and equipment of the storage installation.

5.3 Structural Integrity Calculations

The main purpose of structural integrity calculations is to determine the maximum tolerable defect sizes at relevant locations in the tank wall. These calculations are an integral part of the RBI assessment. Typical assessment locations and defect orientations considered in such calculations are defined in Appendix 3. In practice, these calculations are usually carried out when one or more of the following beneficial reasons apply:

- To justify and support the use of non-intrusive non-destructive testing (NDT)
 inspection methods from the outside of the wall of the tank to check for any
 significant defects including SCC on the internal surface.
- 2) To improve the assessed criticality position of the tank on the RBI diagram and to predict the likely failure mode (Leak Before Break, LBB, or Break Before Leak, BBL), if SCC takes place, and hence the likely consequence.
- To provide additional confidence in the inspection method and inspection coverage selected or in the selection process of the most cost effective inspection plan.
- 4) To provide further confidence in the selected inspection interval.
- 5) To assess the significance of cracks found by either internal magnetic testing (MT) or external non-intrusive NDT methods.

Established fracture mechanics codes such as BS 7910: 1999: Guide on method for assessing the acceptability of flaws in metallic structures, should be used for the calculation of maximum tolerable defect sizes. Ideally, it is useful to calculate tolerable surface breaking as well as fully penetrating defect sizes. The latter is included so that the likely failure mode (LBB or BBL) can be predicted for the defect locations selected.

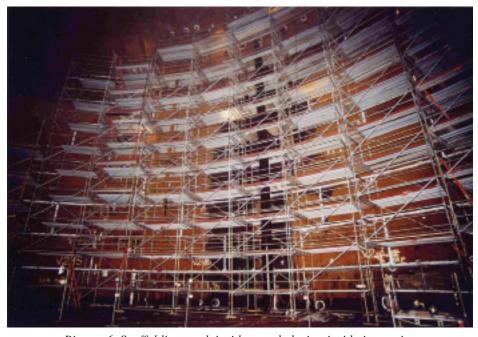
The required data for the fracture mechanics calculations should be established for all the assessment locations and the relevant defect orientations selected. One of the main input data for these calculations is applied stress due to maximum operating height of liquid ammonia and hydro test conditions. It is also useful to calculate the relaxed welding residual stresses resulting from the initial hydro test, so that the beneficial effect of this can be included in the tolerable defect size calculations. The other key input data required are fracture toughness, Charpy impact energy, yield and tensile strengths of the welds and plate materials. In the absence of fracture toughness properties, the derivation of such data from known Charpy values is detailed in various fitness for service national codes e.g. BS 7910, EN 13445-2/A2 and API 579 [Refs. 19-21]. In Level 1 calculations these lower bound properties of BS 7910 can be used as a first estimate. If the calculated tolerable defect sizes are large enough to be easily detected by NDT they can be adopted. If this conservative approach results in tolerable defect sizes too small to measure, it will be necessary to consider obtaining real material properties to facilitate Level 2 calculations.

It is useful to produce the results of the fracture mechanics calculations in a tolerable defect depth *vs.* defect length graphical format for each of the defect orientations and assessment locations considered. Such a presentation will help the interpretation of results and the subsequent decision making process.

5.4 Integrity Inspection from Inside

Most of the ammonia tank inspections that have been carried out at various plants were internal inspections (where the condition of the tank is checked from the inside). Typically wet-fluorescent magnetic particle testing methods are used. In addition, spot checks of the thickness of the bottom and wall plates and 'vacuum box' testing of the tank bottom are recommended. The inspectors should be qualified according to EN 473 [Ref. 22].

The extent of the inspections should depend on the findings and follow a stepwise approach as shown in Table 2 below. If no significant defects are identified in step 1, this should be sufficient to consider the tank free from critical cracks. If defects are found, which cannot be explained as insignificant fabrication defects, it is necessary to move to step 2. As for step 1, if defects are found in step 2, which cannot be explained as insignificant fabrication defects, it is necessary to move to step 3.



Picture 6 Scaffolding work inside a tank during inside inspection

Table 2 Scope of internal inspection

Area	Step 1	Step 2	Step 3
Bottom to shell weld	100%		
Annular ring	100%		
Bottom plates T-welds	50%	100%	
Shell plates, T-welds in courses 1 and 2	40%	100%	
Shell plates, horizontal and vertical welds in courses 1 and 2 courses 1 and 2	10%	100%	
Shell plates, T-welds in course 3 to top	10%	50%	100%
Shell plates, horizontal and vertical welds in course 3 to top		10%	100%
Manholes, pipe connections, pump sink and other special details	100%		
Clamp marks in courses 1 and 2, or temporary fabrication weld marks	10%	100%	
Areas subject to previous repairs	100%		

The scope should be considered as an indicative requirement. It is reasonable to extend the inspection programme in step 1 as decommissioning, recommissioning, cleaning operations and preparation work such as scaffolding etc. are often far more time consuming.

5.5 Non-Intrusive Integrity Inspection (from outside)

5.5.1 General comments

The non-intrusive inspection of the internal condition will be from the external surface of the tank wall, for example using an ultrasonic flaw detection method, to check for any significant SCC that could be present internally at the weld seam areas.

Whichever non-intrusive inspection technique is used, the method has to be suitable for the relevant inspection locations and temperatures. It must be sufficiently reliable and sensitive for the detection of the type, size and shape of cracks that are acceptably below the calculated maximum tolerable defect sizes (Section 5.3). It is also necessary to define a map, which clearly identifies those areas of the tank that are to be inspected by such methods.

Non-intrusive inspection may be carried out more regularly than traditional internal inspection. This method of inspection does not affect the integrity of the tank and is easier and less hazardous to carry out.

Non-intrusive inspection of the tank should be considered if the following conditions are obtained:

- Ideally, at least one internal inspection should have been carried out and no significant SCC or other corrosion should have been detected. This first inspection is aimed at detecting SCC as well as original manufacturing defects. If this precondition is not achieved, some alternatives are available:
- 2) Confirm the integrity of the tank by an extensive inspection from the outside. Such extensive inspection means an inspection with a scope similar to that performed during an internal inspection. (Table 2, excluding the inspection of the bottom).
- 3) Confirm the status of possible original construction defects by having an extensive fully documented quality control record following fabrication. In order to accept this as a valid alternative, 100% of the T-welds in the three lower courses as well as 100% of the shell to bottom weld should have been checked using Magnetic Particle or Radiography.
- 4) RBI assessment (defined in Section 5.2) places the tank in the inspection frequency area of at least 10 years (see Inspection Frequency Diagram).
- 5) Structural integrity calculations (defined in Section 5.3) conclude that the maximum tolerable defect sizes are much higher than detectable defect sizes.
- An inspection programme is available for accessories and connected items, see Section 5.6.

An internal inspection of the tank may be necessary if inspection from the outside highlights/reveals areas of potential problems or the data and measurements taken cannot confirm the integrity of the tank in all areas.

It should be noted that ice could form on the cold exposed surface of the tank (at -33°C) and present difficulties in inspection. Methods to overcome this problem include: provision of an enclosure with a flow of dry air or empty the tank and let it warm up.

Tank bottom

A non-intrusive inspection of the tank bottom is not possible in most cases because most refrigerated ammonia storage tanks are situated on the ground or on a full concrete platform. However, failure of the bottom plates is very unlikely. Possible failure mechanisms for the bottom plates are listed In Table 3 with a remark on the likelihood of occurrence or possible preventive actions.

Table 3 Possible failure mechanisms for the tank bottom plate

Stress Corrosion Cracking	The load bearing part of the tank is the wall. Apart from the part close to the wall, the bottom is mainly subjected to the hydrostatic pressure of the liquid. Therefore the stress level in the bottom is much smaller than in the tank wall. So the non-intrusive inspection findings of the wall inspection are a worst-case representation of the bottom. If no SCC is found in the wall, it is very unlikely that the bottom will be affected. A non-intrusive inspection will prevent the entrance of oxygen, which is one of the pre-requisites for the formation of SCC.
Corrosion from outside	As the temperature in the tank is as low as -33°C and the bottom is insulated, the rate of external corrosion is negligible.
Frost Heave	By using proper insulation and having installed bottom heaters when the tank is installed on the ground, frost heave is prevented.
Fabrication Errors	Some defects and even cracks have been found in ammonia tanks that were caused by fabrication errors (wrong welds or wrong materials used in the bottom plates). Therefore ideally the first tank inspection is an internal inspection, to check the bottom.
Earthquakes, ground movement or foundation movement	Assess the situation and inspect the tank as appropriate.

The consequence of a crack in the bottom plating is also expected to be very limited. A potential crack will only allow a small leakage in the bottom, because the extension of this crack will be limited due to the lack of driving force (stress). Furthermore in the case of a double integrity tank, the outer tank will collect the leaking ammonia. In the case of a single wall tank, the leaking ammonia will evaporate and the leak will be easily detected by the smell of ammonia and the formation of ice.

5.5.2 Non-intrusive in-service inspection methods

Non-intrusive in-service inspection methods applied from outside the tank have not yet been widely used by the industry; but the technology has developed in recent years and has been evaluated on a number of tanks in recent years. The evaluated methods are ultrasonic testing (UT), acoustic emission (AE) and electrical field signature method (FSM). Advantages and disadvantages are given in Table 4 and compared with traditional defect detection methods using magnetic testing (MT).



Picture 7 Inspection of the tank from the outside in the annular space

Table 4 Inspection methods

Method	Advantages	Disadvantages
Magnetic Testing	High sensitivity. Exact length measurement.	Internal access necessary. No depth measurement. Surface preparation needed.
Ultrasonic Testing	Inspection during operation. Possible to indicate length and depth.	Less sensitive than MT. Necessary to remove insulation. Limited areas available.
Acoustic Emission	Inspection during operation.	Less sensitive than UT. Requires mechanical stress to hear noise.
Field Signature Method	Inspection during operation. Measure changes in crack sizes.	Not yet proven technology. Relative method, not possible to detect old cracks.

When applying UT techniques, it should be borne in mind that the SCC cracks are liquid filled and may, therefore, have a much lower reflectivity depending on their width and the frequency of the ultrasound. Secondly, the SCC defects have a complex shape and may occur in clusters. Pulse echo methods in combination with the UT transmission technique using a multiple transducer set-up, have been found to be effective for detecting defects. This can be complemented with the TOFD method.

The NDT technique used must be validated and the NDT operator must be tested on a specimen with either SCC defects or SCC like defects present.

Depending on the minimum required sensitivity, UT is currently the recommended method for in-service internal inspection from the outside of low temperature ammonia tanks.

5.5.3 Number, size and location of areas to be inspected

The number of areas to be inspected depends on a number of factors such as the size of the tank, susceptibility to SCC occurring and its inspection history. It is recommended that inspection areas are chosen which give a representative selection of critical welds and welds where cracks in other parallel tanks on site have been detected.

Two philosophies for inspection are possible: either inspection of a representative percentage of the area, or inspection of selected areas where a specific phenomenon may occur.

Selected areas are those areas where the conditions for initiation of SCC are more favourable than in the rest of the tank or areas where the consequences of SCC are high. From a structural integrity point of view, the last point is the most important. When an area is selected where SCC is most likely to occur, this is chosen as an indicator of the presence of SCC.

Two examples are given below to illustrate the above point.

1) For a single course of the tank, as an example, the selection should cover a number of areas around T-welds, 50 cm in each direction from the T.

This gives the following coverage for one course of a typical tank with 30 m diameter, where 4 areas have been proposed, as illustrated in Table 5.

Table 5	Inspection	coverage	for one	course	of a tank
I abic 5	Inspection	CUVCIAZO	IUI UIIC	course	or a tams

Weld	Area inspected	% of type of weld	Comments
Horizontal weld	4 m	4%	
Vertical weld	2 m	8%	
T-weld	4	40%	No. of welds

It is assumed there are 10 T welds per circumference.

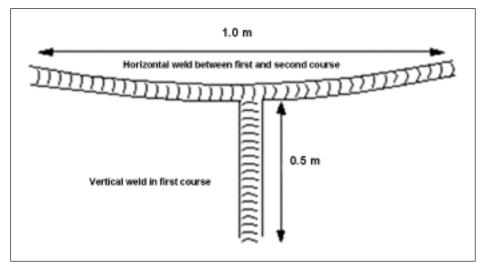


Figure 10 Typical inspection areas around a T-weld

2) In this example the extent of NDT to be carried out from the outside of the tank is given in Figure 11. Sufficient insulation must be removed to expose 150mm each side of each weld within the marked areas.



Picture 8 Weld area to be inspected, with insulation removed

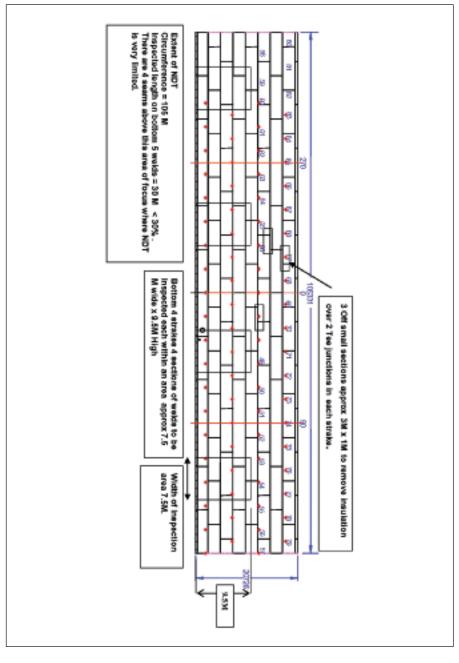


Figure 11 Outer surface of tank showing areas to be inspected by UT inspection method

5.6 Other Inspection Issues

The in-service inspection of the tank from outside or inside is part of an overall programme and other inspection actions, routines and controls will be required in order to ensure that accessories e.g. valves and pipe connections are also in good condition.

These inspections and controls will take place with their own frequency. It could be necessary to use specific methods adapted to each case. They will cover all the critical items and together they will constitute the overall safety inspection system of the storage facility.

The following controls have to be included in this system:

Operational controls

These include:

- 1) Continuous monitoring of operating parameters.
- 2) Regular analyses of water and oxygen contents in the ammonia.
- 3) Regular (e.g. daily, monthly) visits on site with visual checks of the equipment and facilities. These should include visits by the operating technicians and the responsible operating/engineering management personnel e.g. on a less frequent basis.
- 4) Regular (e.g. 6 months, 1 year) operating tests of the safety equipment (tank emergency shut-off valves, automatic valves on lines, etc.).

Preventive maintenance

This includes:

- 1) Ammonia detectors, if installed (6 months).
- 2) Electrical equipment, earth connection, lightning rods (1 year).
- 3) Instrument and control equipment e.g. level, pressure, temperature etc. (1 year / 2 years).
- Pumping and cooling facilities.
- 5) Safety valves.

Specific inspection controls

These include:

- 1) Visual Inspection of tanks, equipment and bund wall (1 year).
- 2) Concrete base inspection and level survey (4-6 year).
- 3) Inspection of insulation e.g. thermographic check (3-5 years).
- 4) Anchor bolts (9 years).
- 5) Visual check of plates and roof on some places after removing the insulation (9 years).

- 6) Inspection of pipes and supports (9 years).
- 7) Inspection of roof and weather protection (1-2 years).
- 8) Inspection of heaters under the base of the tank (3-6 months).

These lists are indicative. They must be adapted for each storage facility according to its design, process and local conditions. Frequencies are also indicative and they have to be defined for each tank.

The external monitoring and inspection of the tank and associated equipment is an important part of the overall inspection programme for ensuring tank integrity.

Operating personnel should routinely monitor the external surfaces of the tank for cold spots, bulges, leaks or any unusual conditions. Changes and unusual occurrences in the tank operation should be recorded and evaluated with respect to the tank inspection programme.

5.7 Leakage Monitoring System

Ammonia detection systems can be installed around ammonia tanks and associated facilities to give early warning on any possible leakages.

Such systems are based on fixed monitors appropriately located.

More advanced detection systems have been developed for specific installations for the continuous detection of ammonia leaks from anywhere in the tank bottom, wall and roof. For example, where there is an annular space, it is possible to purge this space with nitrogen and continuously analyse the off-coming gas for its ammonia content. When such a tank has been confirmed as being in the leak before break mode and where the monitoring system is capable of:

- 1) quantifying the leak rate
- demonstrating that this rate is less than that from the calculated maximum critical sized defect.

The above information may be used in discussions with regulatory authorities to develop the inspection strategy.

5.8 Reporting

All reports from inspections of the tanks should, as a minimum, include the following items:

- 1) Tank identity with information about materials, welds etc.
- 2) Date of inspection and years since the last inspection.
- 3) Areas of the tank inspected (map, drawing, description).
- 4) Map of identified defects from earlier inspections, both repaired and not repaired defects (weld defects from construction etc.).
- 5) Inspection method.

- 6) Inspector qualification data (if relevant).
- 7) Qualification information for the inspection method.
- 8) Reference to the evaluation report and/or inspection programme.
- 9) Findings with a map where defects are identified.
- 10) Reference to further investigations (if relevant).
- 11) Conclusion and recommendations for future inspection requirements.

6. EVALUATION, REPAIRS AND CORRECTIVE ACTIONS

6.1 Evaluation

All findings must be evaluated, identified on a map and assessed for fitness for purpose. Defects found must be well documented for monitoring actions in subsequent inspections. The extent of controls will normally be decided to verify if other defects are present.

More frequent monitoring may be required to check the evolution of the defect.

If a defect is found which is potentially hazardous, an internal inspection must be performed and, if necessary, a repair carried out. Associated decommissioning and recommissioning must be carried out in accordance with Chapter 7.

6.2 Repairs

Repairs involving rewelding introduce local high stress levels. Grinding may be necessary to establish typical and maximum defect depths. It is strongly recommended not to carry out weld repairs if sufficient material thickness is in the area of the defect location. If welding is required, it is vital to use a low strength weld deposit and carry out all necessary actions to avoid local high hardness.

Any repairs should be documented in detail (location, repair method, depth, weld materials and procedures, welder qualifications, thickness tests, etc.) to provide information for later assessments and inspections.

6.3 Corrective Actions

Welding repairs may increase the risk of initiation of cracks. Areas that have been subject to weld repair require adequate follow up. Repairs may move the tank into an area where more extensive inspection is required. Other actions such as the reduced operating level of ammonia liquid to accommodate any apparent weakness should also be considered.

The overall inspection programme should be reviewed and updated accordingly.

7. COMMISSIONING, DECOMMISSIONING AND RE-COMMISSIONING

Commissioning, decommissioning and re-commissioning have to follow procedures which ensure the efficient removal of oxygen and careful and uniform cooling or warming up. This is important in order to keep the thermal stress to a minimum level and to reduce the risk of initiating stress corrosion cracking. The procedure should be well documented and records of actual measurements should be maintained for future reference.

Item 3a of the (re)commissioning procedure, given below, has been introduced to protect the tank bottom from Stress Corrosion Cracking initiation using the inhibiting effect of water on SCC and to ensure a gradual cooling down of the tank bottom, limiting the thermal stresses. However, the use of aqueous ammonia during ammonia storage tank commissioning also introduces an important risk. When there is poor mixing of the anhydrous ammonia being sprayed from the top of the tank with the aqueous ammonia in the bottom of the tank, two separate layers are formed. The risk of poor mixing is especially relevant if the tank has not been cleaned, leaving a certain amount of oil in the tank. This oil film can form a membrane type separation between the aqueous ammonia and the anhydrous ammonia phase. Once two such phases have formed, a subsequent mixing of these two phases causes a significant amount of heat being released as well as a sudden pressure increase in the tank. The amount of vapour being released during this mixing can exceed the capacity of the pressure relief valves and can cause tank failure.

Commissioning and re-commissioning

- 1) Hydrotest at the first commissioning by filling the tank with water, either up to 70 or 100% of the maximum level, depending on the design code.
- Purge with nitrogen until the measured oxygen in the discharge gas is less than 4%.
- 3) a) Before purging with ammonia, leave a certain level of aqueous ammonia (20% or more) in the bottom of the tank. The amount depends on the flatness of the bottom and should, as a minimum, cover the whole tank bottom. The use of aqueous ammonia is only recommended when the tank is clean (free from oil) and good mixing with the liquid ammonia is ensured (e.g. with a recirculation pump). Purge with ammonia gas until the measured oxygen in the discharge gas is less than 0.5%.

OR

- b) Purge with ammonia gas until the measured oxygen in the discharge gas is less than 0.5%.
- 4) Cool the tank down to as low a temperature as possible, at a cooling rate lower than 1°C/hour, preferably using a spray system.

- Measure the temperature in the bulk volume of the tank, away from the gas inlet.
- 6) Within one week after commissioning and when conditions are stable, take samples from the ammonia liquid in the tank and analyse them for water and oxygen.

Decommissioning procedure

- 1) Empty the tank to the absolute minimum liquid level.
- 2) Evaporate the remaining ammonia in a way that ensures uniform and slow heating, not exceeding 1°C/hour.
- 3) Measure the temperature in the bulk volume of the tank, away from the gas inlet. Give careful consideration to temperature measurements at the lower levels of the tank during decommissioning.
- 4) Purge with warm ammonia gas or nitrogen until all liquid ammonia is removed. The bottom area may need to be cleaned before it is possible to get all the ammonia gas out.
- 5) Remove the ammonia gas in the tank by purging with nitrogen (not air, to prevent the formation of an explosive atmosphere). To prevent environmental problems it is best to re-condense the ammonia as much as possible or send this purge stream to an ammonia wash column (with water).
- 6) Remove the nitrogen atmosphere by purging with air until the oxygen content is >19%. If after these steps some ammonia is still measured in the gas phase (for example, due to residual oil from which ammonia is slowly evaporating), breathing equipment must be used when entering the tank.

8. GLOSSARY & EXPLANATION OF TERMS

AE Acoustic Emission

AIChE American Institute of Chemical Engineers

API American Petroleum Institute **AWS** American Welding Society

AWS 60xx AWS (American) coding system for welding consumables

BBL Break Before Leak BS **British Standard**

CIA Chemical Industries Association (UK).

EFMA European Fertilizer Manufacturers Association

EN E 46xx EN (European) coding system for welding consumables

FCAW Flux Cored Arc Welding **FSM** Field Signature Method **GTAW** Gas Tungsten Arc Welding **HAZOP** Hazard and Operability Study

Intrusive Internal inspection by entering the tank

LBB Leak Before Break

Liquefied This term generally describes liquid ammonia which is close to its

> boiling point and is in contact with its vapour. Liquid, on the other hand, can describe sub-cooled as well as near boiling liquid.

MPI Magnetic Particle Inspection

MT Magnetic Testing

NDT Non Destructive Technique

Non-intrusive Inspection of the internal condition of the tank from the outside

RBI Risk Based Inspection SAW Submerged Arc Welding **SMAW** Shielded Metal Arc Welding SCC Stress Corrosion Cracking **TOFD** Time of Flight Diffraction

UT Ultrasonic Testing

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APPENDIX 1: WELDING CONSUMABLES FOR AMMONIA TANK CONSTRUCTION

STRENGTH LEVEL	TYPE OF WELDING CONSUMABLES	STANDARD/ GRADE/ DESIGNATION	TYPICAL YIELD STRENGTH [MPa]	TYPICAL TENSILE STRENGTH [MPa]		
	SMAW	AWS E60xx	min. 331	min. 414		
"LOW"	FCAW	AWS E6xT-x	min. 345	min. 428		
STRENGTH	SAW	AWS F6x-Exxx	min. 330	415-550		
	SMAW, FCAW and SAW	EN E 38 x x x x	min. 380	470-600		
	SMAW	AWS E70xx	min. 390	min. 480		
"MEDIUM"	FCAW	AWS E7xT-x	min. 414	min. 497		
STRENGTH	SAW	AWS F7x-Exxx	min. 400	480-650		
	SMAW, FCAW and SAW	EN E 42 x x x x	min. 420	500-640		
	SMAW	AWS E80xx	min. 460	min. 550		
	SMAW, FCAW and SAW	EN E 46 x x x x	min. 460	530-680		

APPENDIX 2: RISK BASED INSPECTION EVALUATION

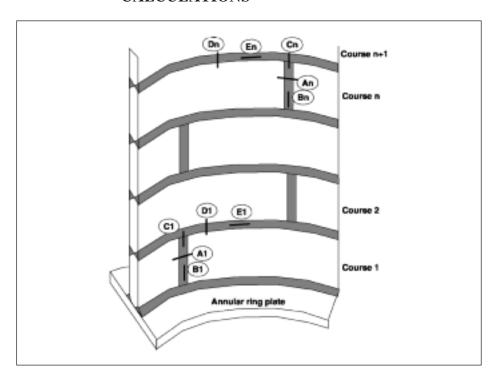
(This appendix is electronically available as an Excel file on EFMA's website www.efma.org)

Probability of Failure

_											_				_				
		2-6			2-5		2-4			2-3				2-2				2-1	
	Have you performed any internal inspections after commissioning? Yes/No	Previous Inspections	Have you observed settling/subsidence pnenomena? No, score is 0 Yes, but additional constructive improvements or simulations made score is 2 Yes, score is 6	External factors like extreme weather conditions leading to stress If no, score is 0 If yes, but additional constructive improvements or simulations made score is 2 If yes, score is 4	External conditions	Do you have material certification, or have you performed a Charpy test at -33°C (or lower) using material with the same charge number that satisfies the requirements in the design code? (Yes / No)	Brittle fracture	Variations in ammonia level to minimum and maximum, frequent (>15 x a year) / non frequent	Flexible pipe connections (Yes / No)	Low Cycle Fatique	Yes, without repairs	Yes, with well-controlled repairs	No No	Mechanical damage	Yes, no coating applied	Yes, corroded location was recoated	No	External corrosion	2 Other Degradation Mechanisms
Total score	0/10		0 / 2 /6	0/2/4		0 / 10		5/0	0/5		10	51	0		10	4	0		Possible Scores
Total score Other degr.		0			0		0			0				0				0	Points
gr.		0.4			0.2		0.8			0.2				0.2				0.2	Weighting Factor
0		0			٥		0			0				0				0	Total
20		0%			0%		0%			0%				0%				0%	% of total

								3 Location of the tank				Assessment benomed	Assessment performed	2 Leak before break assessment								1 Release of Ammonia to the atmosphere / Extra external safety	Consequence Ranking
	Close to population less than 1 km, close to water	Close to population between 1 and 2 km, dose to water	Close to population less than 1 km, not close to water	water	Close to population between 1 and 2 km, not close to	Not close to population and close to water	Not close to external population (2km), not close to water (200m.)	ie tank	No	Yes, conclusion "Break before leak"	Yes, but "Non-conclusive"	Yes, conclusion "Leak before break"		assessment	e) Single steel tank without bundwall (see fig. 6)		 d) Steel tank with an embankment of earth to contain full contents of the tank and no roof over the space between the tank and the embankment (fig.5) 	 Steel tank with a partial height concrete bund wall with impervious floor within the contained area and no roof over the space (see fig.4) 	 b) Sieel tank noused within another steel tank to contain full contents of the tank, with a single roof (cup in tank) or independent roofs (see Figures 3); 		with capacity to contain full contents of the tank and the space between the tank and the bund having impervious floor and roof covering (see Figure 2)	ohere / Extra external safety	king
Total scor	10	7	8	51	_	3 0		10	10	10	7	0		10	10	œ		6	2	2			Possible Scores
e Consequ								0						0								0	Points
Total score Consequence of Fa 0								2.5						2.5								S	Weighting Factor
0								0						0								0	Total
100								0%						0%								0%	% of total

APPENDIX 3: CRACK CONFIGURATIONS THAT SHOULD BE EVALUATED BY STRUCTURAL INTEGRITY CALCULATIONS



The sketch above illustrates the typical defect orientation and locations which should be considered when maximum tolerable defect size calculations are carried out. When necessary, the calculated results for these locations can be interpolated for shell courses 2 to n-1.

- A1 (n): Transverse cracks in vertical welds in course 1 (course n).
- B1 (n): Longitudinal cracks in vertical welds in course 1 (course n).
- C1 (n): Transverse cracks in horizontal welds between courses 1 and 2 (courses n and n+1), located at a T-weld.
- D1 (n): Transverse cracks in horizontal welds between courses 1 and 2 (courses n and n+1).
- E1 (n): Longitudinal cracks in horizontal welds between courses 1 and 2 (courses n and n+1). Course n is at a level higher in the tank wall, e.g. with lower strength material.

APPENDIX 4: ANALYSIS OF OXYGEN IN LIQUID AMMONIA

1. Introduction

In the assessment of the risk for stress corrosion cracking, the oxygen concentration in the liquid ammonia is a key parameter and should therefore be closely monitored. This appendix describes some guidelines for analysing the oxygen content in the range relevant for stress corrosion cracking (0.1-10 ppm).

2. Sampling

The key parameter for stress corrosion cracking is the oxygen content in the liquid ammonia. It is important to realise that the oxygen distribution between the liquid and the vapour phase is such that, in equilibrium at -33° C, the oxygen concentration in the vapour is about 10.000 times higher than the oxygen concentration in the liquid. At temperatures > 0°C this factor decreases to 100-1000.

In cases where equilibrium can be assumed between the liquid and the vapour phases, it is recommended to analyse the vapour phase as the method applied can be less sensitive due to the much higher concentration in the gas phase.

Furthermore it is important to note that when a liquid sample is taken and vaporised, the total sample shall be vaporised and analysed in order to obtain representative results.

3. Analysis methods

When analysing oxygen in the relevant range, it is of vital importance to prevent sample contamination by traces of oxygen in the carrier or purge gases used during the various steps.

Several methods are applied within the industry to analyse oxygen in ammonia in the required concentration range:

Method	Detection limit (ppmv)
Gas chromatograph followed by mass spectrometer	0,1
Gas chromatograph followed by Thermal conductivity detector	1
Gas chromatograph followed by Electron Capture detector	2
Analyser based upon fuel cell, creating an electric current, proportional to the oxygen concentration	1 (Manufacturer claims lower values)

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