

Jørgen Hyldgård Staldservice A/S

JH-FORSURING NH4+

Test plan



Version 1-3



Document information

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|-----------------------|---------------------------------------|
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2 INTRODUCTION

This test plan is developed to be used for verification of JH-FORSURING NH4+ following the AgroTech DANETV Test Centre Quality Manual.

2.1 Verification protocol reference

This test plan is prepared to meet the requirements defined in the VERA Test Protocol for Livestock Housing and Management Systems, version 1 2009-12-09

2.2 Name and contact of vendor

The JH-FORSURING NH4+ is developed and produced by Jørgen Hyldgaard Staldservice A/S, Nørgårdsvej 18 , 7500 Holstebro. Contact person of Jørgen Hyldgaard Staldservice is Ken Hyldgård. Phone: +45 (0) 97 42 81 89. E-mail: info@jhstaldservice.dk

The JH-FORSURING NH4+ is marketed and sold in Denmark by Jørgen Hyldgaard Staldservice A/S

The test is undertaken by DANETV Test Centre AgroTech, Agro Food Park 15, DK-8200 Aarhus N, Denmark.

Test responsible: Peter Hansen, AgroTech, Agro Food Park 15, Skejby, DK-8200 Aarhus N. Phone: +45 2172 7942. E-mail: pth@agrotech.dk

2.3 Technical experts

The technical experts assigned to this test and responsible for review of test plan and test report includes:

Internal experts:

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External expert:

Arne Grønkjær Hansen, Danish Technological Institute, Kongsvang Allé 29, DK-8000 Aarhus C., Phone: +45 7220 2142.

E-mail: arne-gronkjaer.hansen@teknologisk.dk.

3 TEST DESIGN

The JH-FORSURING NH4+ is tested in full-scale on 4 commercial dairy farms during a 12 months period covering both summer and winter temperatures.

3.1 Test site

3.1.1 Characterization of the test site

4 commercial dairy farms are used for the test site. The capacity of the housing system is between 150 – 550 cows. 3 of the test sites have Holstein-Friesian cattle and 1 has Jersey cattle.

The flooring system of the all the dairy farms are slatted floor. The manure from the cattle is collected in a circular pit under the slats. The floor is either scraped with a line scraper or by cleaning robots.

The ventilation system is natural ventilation with regulated curtains or lamella openings in the sides. The number of cattle's indicated in table 1-4 are the present number before test start.

The bedding material is either straw or Sawdust. The cows are all fed with a mix of corn silage and grass silage balanced with soybean meal and wheat.

Table 1 - 4 gives an overview of key characteristics of the dairy farms used for the test.

Table 1. Key characteristics of the dairy farm no. 1 used for test.

| Parameter | Test site characteristics |
|---------------------------------|---|
| Farm owner | Milthers Lodahl |
| Address | Herningvej 66, Trandum 7800 Skive |
| Contact Info | Phone: 9754 4152 mob.: 2013 41 52 |
| CHR no. | 66571 |
| Grazing cows in summer | No. |
| Number of cows | 140 |
| weight range (kg) | 600-650 |
| Milk production l/year/cow | 10.500 |
| Bedding material | Straw pellets (Easy Strø) |
| Space provided per animal | 1,25 m wide 2,8 m long |
| Number of Heifers | 60 |
| Number of cows in dry period | 15 |
| Number of calves | 0 |
| Floor design | Slats |
| Manure removal system | Recirculated manure in circular pit |
| Scraper systems on top of slats | Robot Scraper |
| Cooling of manure | No. |
| Feed composition | ½ corn silage ½ grass silage, soybean meal, weate |
| Feeding system | Dry food feeding |
| Feed analysis | 3 x per year |
| Ventilation | Natural ventilation with automatically regulated curtains |

Table 2. Key characteristics of the dairy farm no. 2 used for test.

| Parameter | Test site characteristics |
|---------------------------------|--|
| Farm owner | Søren Hansen |
| Address | Sevelvej 85 7830 Vinderup |
| Contact Info | mob.: 4057 6877 |
| CHR no. | 57194 |
| Grazing cows in summer | No. |
| Number of cows | 190 |
| weight range (kg) | 600-650 |
| Milk production l/year/cow (km) | 10.300 |
| Bedding material | Sawdust |
| Space provided per animal | 1,25 m wide 2,8 m long |
| Number of Heifers | 0 |
| Number of cows in dry period | 0 |
| Number of calves | 0 |
| Floor design | Slats |
| Manure removal system | Recirculated manure in circular pit |
| Scraper systems on top of slats | line scraper |
| Cooling of manure | No. |
| Feed composition | 2/3 corn silage 1/3 grass silage, wheat, soybean meal |
| Feed analysis | Once per month |
| Feeding system | Dry food feeding |
| Ventilation | Natural ventilation with lamella openings in both sides, not adjustable. Can be supplemented with opening of windows |

Table 3. Key characteristics of the dairy farm no. 3 used for test.

| Parameter | Test site characteristics |
|------------------------------|----------------------------|
| Farm owner | Jens Erik Damtoft |
| Address | Struervej 1, 7830 Vinderup |
| Contact Info | mob.: 6175 2767 |
| CHR no. | 57133 |
| Grazing cows in summer | No. |
| Number of cows | 282 |
| weight range (kg) | 600-650 |
| Milk production l/year/cow | 10.700 |
| Bedding material | Sawdust |
| Space provided per animal | 1,25 m wide 2,8 m long |
| Number of Heifers | 90 |
| Number of cows in dry period | 18 |
| Number of calves | 0 |
| Floor design | Slats |

| | |
|---------------------------------|--|
| Manure removal system | Recirculated manure in circular pit |
| Scraper systems on top of slats | line scraper |
| Cooling of manure | No. |
| Feed composition | 2/3 corn silage 1/3 grass silage |
| Feeding analysis | Once per month |
| Feeding system | Dry food feeding |
| Ventilation | Natural ventilation with manually regulated curtains |

Table 4. Key characteristics of the dairy farm no. 4 used for test.

| Parameter | Test site characteristics |
|---------------------------------|---|
| Farm owner | Knud Erling Birch |
| Address | Ejsingholmvej 35, 7830 Vinderup |
| Contact Info | Phone: 9744 6395 mob.: 2091 6395 |
| CHR no. | 54645 |
| Grazing cows in summer | No. |
| Number of cows | 520 |
| weight range (kg) | 450-500 |
| Milk production | 8.270 |
| Bedding material | Sawdust |
| Space provided per animal | 1,25 m wide 2,8 m long |
| Number of Heifers | 0 |
| Number of cows in dry period | 0 |
| Number of calves | 0 |
| Floor design | Slats |
| Manure removal system | Recirculated manure in circular pit |
| Scraper systems on top of slats | Line scraper |
| Cooling of manure | No. |
| Feed composition | ½ corn silage ½ grass silage, soybean meal, weate |
| Feeding analysis | Once per month |
| Feeding system | Dry food feeding |
| Ventilation | Natural ventilation with automatically regulated curtains |

Figure 1 - 4 gives an overview of the construction of the 4 dairy farms used for the test.

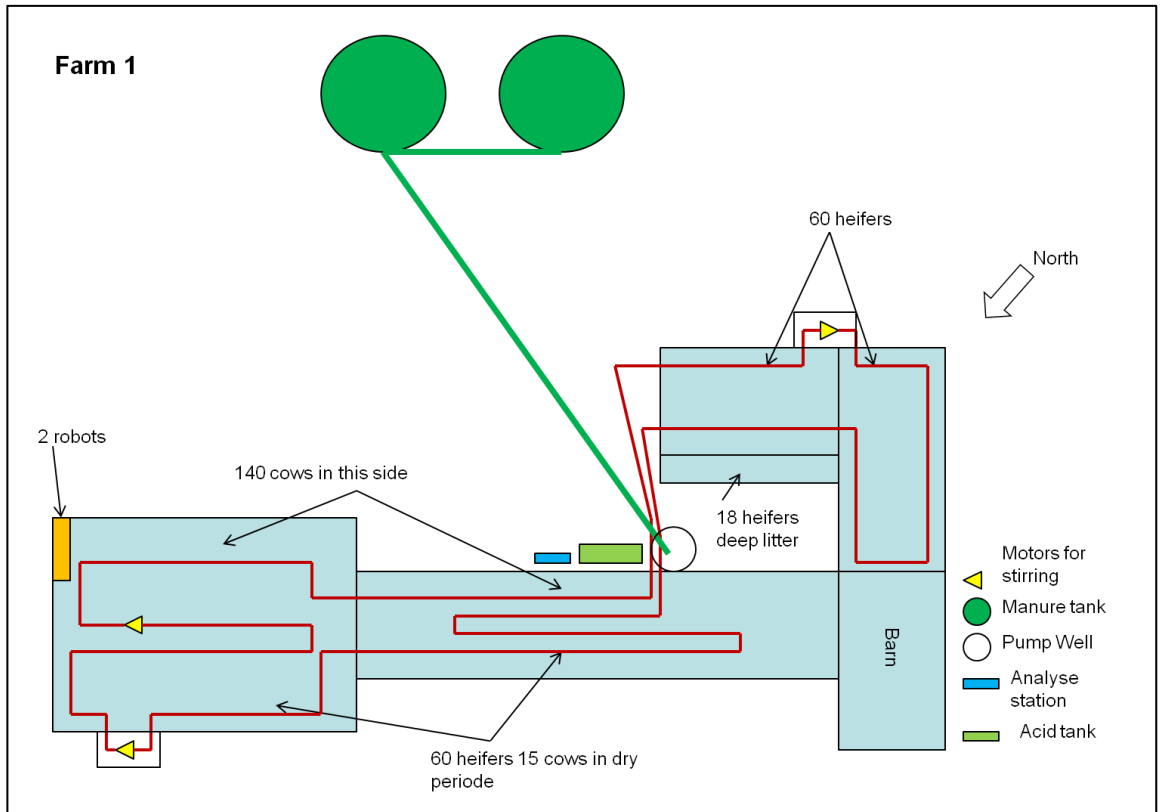


Figure 1 shows a diagram of the building design at farm 1.

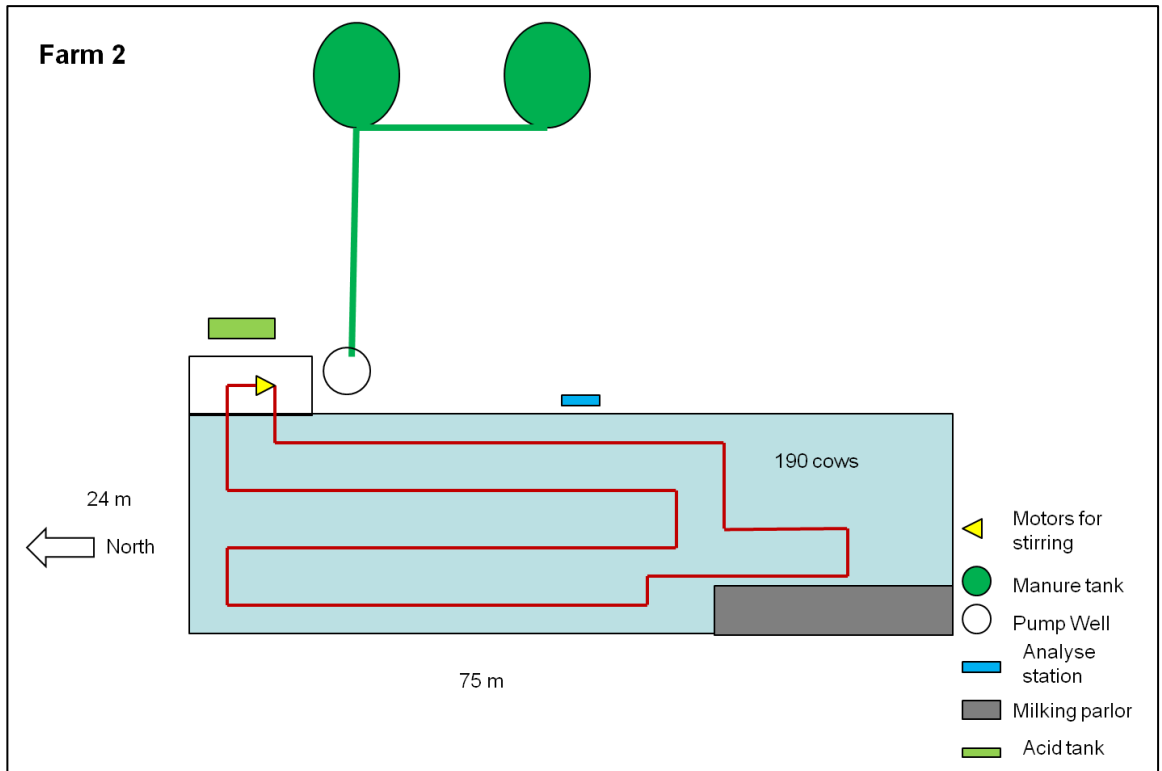


Figure 2 shows a diagram of the building design at farm 2.

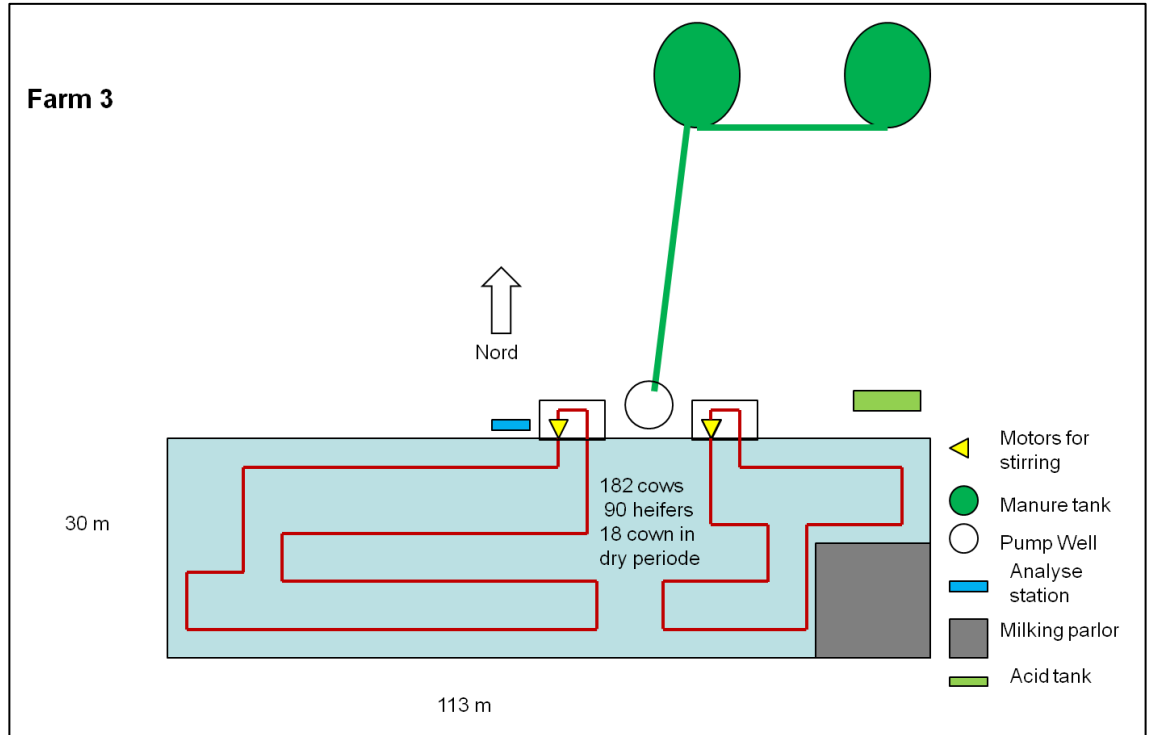


Figure 3 shows a diagram of the building design at farm 3.

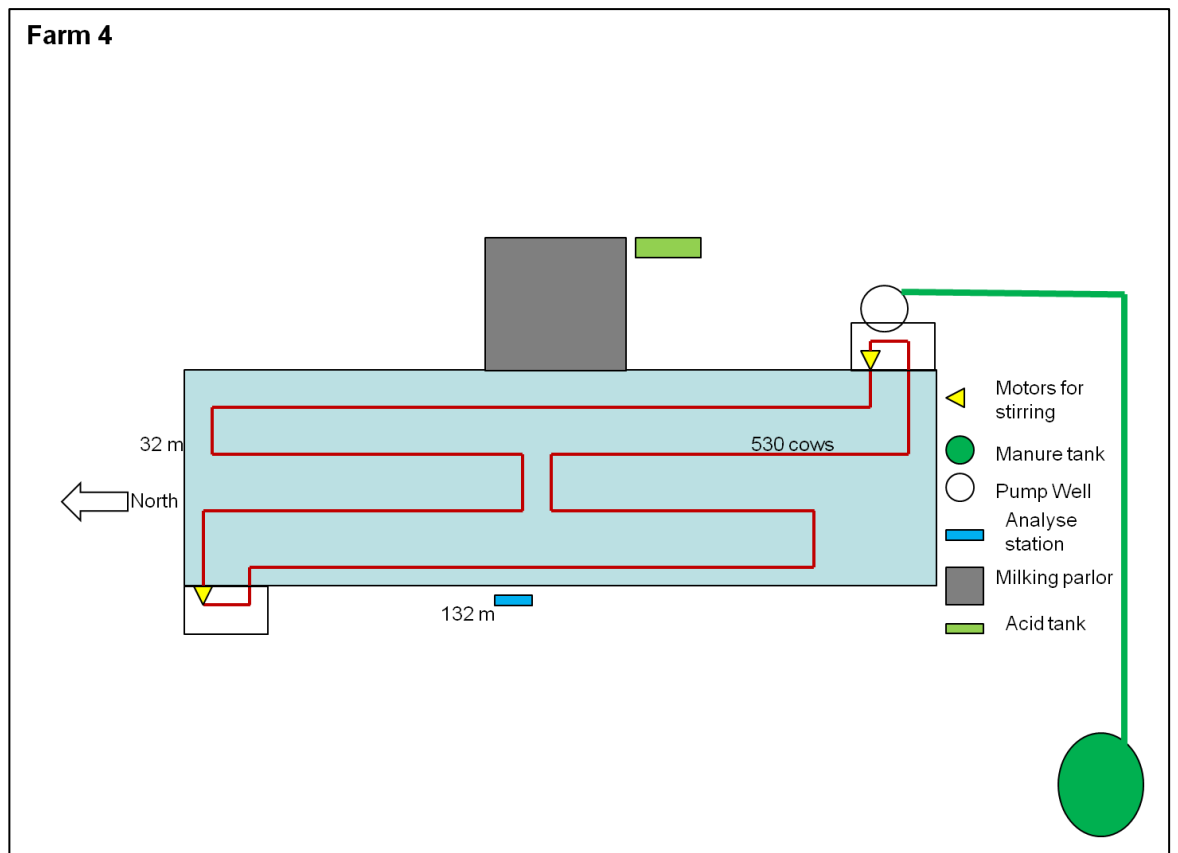


Figure 4 shows a diagram of the building design at farm 4.

3.1.2 Addresses

The test will take at the following farms:

Farm no. 1

Milther Lodahl
Herningvej 66, Trandum
7800 Skive

Farm no. 2

Søren Hansen
Sevelvej 85
7830 Vinderup

Farm no. 3

Jens Erik Damtoft
Struervej 1
7830 Vinderup

Farm no. 4

Knud Birch
Ejsingholmvej 35
7830 Vinderup

3.1.3 Descriptions

Functional description of JH-acidification

Jørgen Hyldgaard, Housing Service A/S, has developed a new technique called JH-acidification NH₄⁺, which is an acidification system for both cattle and pig manure. The current acidification system has been developed for a dairy production with a circular pit system.

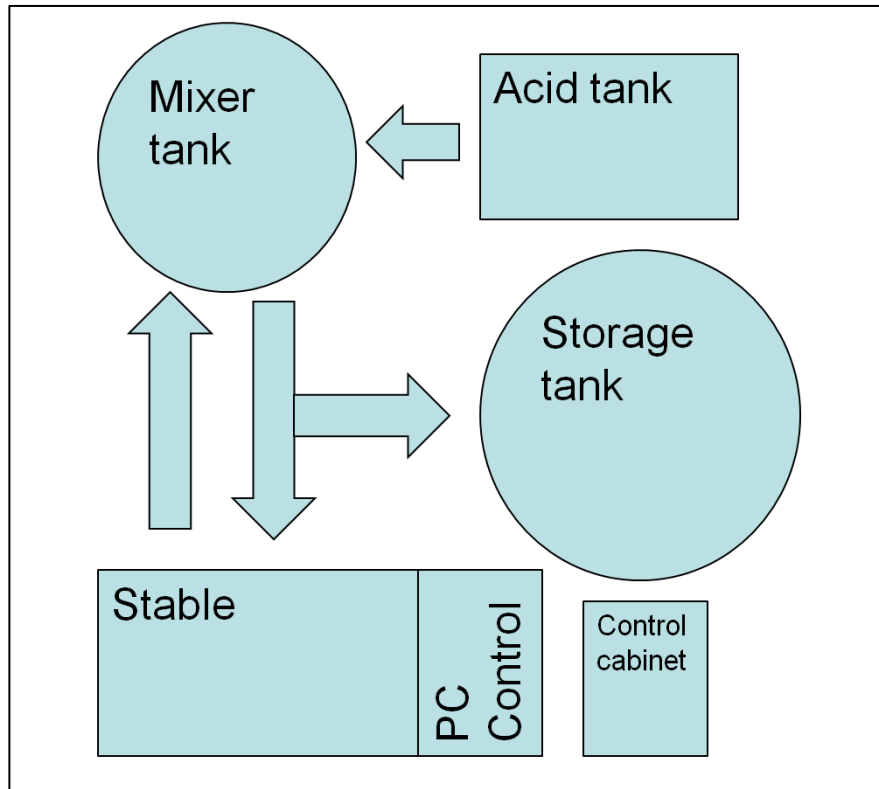


Figure 5. A diagram of the JH-acidification system for a cattle stable.

The manure acidification system for cattle farms includes the following key elements:

- Acid tank, where the sulphuric acid (96 %) is stored until it is added to the manure.
- Mixing tank in which stirring, acid addition and pumping take place. The mixing tank is generally an existing tank.
- Newly produced manure is mixed with acidified manure and returned to the stable in the circular pit below the floor.
- Storage tank, where acidified manure is stored after the pumping process from the mixing tank.
- Control box and PC controller, which are used for configuration, data logging and alarms.

The acidification system works as follows:

At a preset time every day of the year acidification takes place in the following chronological order:

1. The two pH electrodes which are placed in the mixer tank are flushed with water.
2. Stirring of the manure in the mixing tank begins and manure from the mixing tank is pumped into the circular pit in the stable.
3. After 10-20 minutes of stirring, sulfuric acid (96 %) is added from the acid tank to the manure in the mixing tank. A metering pump is used for this purpose.
4. The stirring stops after 30-60 minutes. At this time the pH value has reached its set point at pH 5.5.
5. After 10 minutes break the pH is measured in the mixing tank.

Dependent on the time when the daily acidification occurs, a preset amount of manure is pumped into the storage tank. This occurs when manure in the mixing tank exceeds a preset level.

All processes such as stirring, pumping, addition of sulfuric acid and pumping to the storage tank are controlled automatically. The control cabinet manages the acidification. Logging of all measured pH values are uploaded to a web server, which can be accessed from everywhere. This gives an opportunity to continuously monitor and verify that the installation works properly [8].

The acid used for acidification of the manure is a 96 % sulfuric acid technical grade.

3.2 Tests

3.2.1 Test methods

The overall principle for testing the performance of the JH-FORSURING NH₄⁺ is to measure the emission of ammonia from dairy farms with the acidification technology installed and compare it to normative emission factors for similar housing systems. As we are dealing with naturally ventilated dairy farms with different building design and capacity case-control studies are not be suitable. In this case we need to monitor 4 different test farms equipped with the acidification system. The required test period is one year and the emission factors are calculated accordingly.

For reference and comparison the norm emission factors will be used for the respective housing systems.

Emission measurements require the measurement of ventilation rates. In naturally ventilated building, ventilation rates cannot be measured by fans and have to be estimated by using tracer gas methods.

The tracer gas used in this test is CO₂ produced from the animals. The production of CO₂ can be estimated from the size of the animals and the milk production. The tracer gas method assumes even distribution of gases and that the dilution rate of CO₂ is equal to the one of NH₃.

The effect is measured over the basis of 12 months covering measurements during summer period and measurements during winter period. The technology is tested at 4 different dairy farms. The primary performance parameter is ammonia.

In addition to the primary performance parameters a number of operational parameters are measured throughout the test periode. A list of the operational parameters is found in section 4.3.

3.2.2 Test staff

The test staffs involved in the test of JH-FORSURING NH4+ are:

Amparo Gomez Cortina, AgroTech, Agro Food Park 15, Skejby, DK-8200 Aarhus N.
Phone: +45 3091 0324. E-mail: aco@agrotech.dk

Peter Hansen, AgroTech, Agro Food Park 15, Skejby, DK-8200 Aarhus N.
Phone: +45 2172 7942. E-mail: pth@agrotech.dk

Linda Veggebros, AgroTech, Agro Food Park 15, Skejby, DK-8200 Aarhus N.
Phone: +45 3092 1795. E-mail: liv@agrotech.dk

3.2.3 Test schedule

The test schedule is presented in table 5.

Table 5. Test schedule.

| Task/md-year | mar-11 | apr-11 | maj-11 | jun-11 | jul-11 | aug-11 | sep-11 | okt-11 | nov-11 | dec-11 | jan-12 | feb-12 | mar-12 | apr-12 |
|----------------------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| Test plan | x | | | | | | | | | | | | | |
| Installation and pre-testing | x | | | | | | | | | | | | | |
| Start test period (4.april.2011) | | x | | | | | | | | | | | | |
| Sampling period | | x | | x | | x | | x | | x | | x | | |
| End of test period (20.feb.2012) | | | | | | | | | | | | x | | |
| Test report draft | | | | | | | x | | | | | | | |
| Test report quality assurance | | | | | | | | | | | | | x | |
| Test report final version | | | | | | | | | | | | | | x |

Here is a preliminary list of measuring dates:

| | Farm 1 | Farm 2 | Farm 3 | Farm 4 |
|--------------------------|------------------|------------------|------------------|------------------|
| Period 1_April | | | | |
| Start measurement | 04 April 2011 | 07 April 2011 | 11 April 2011 | 14 April 2011 |
| Stop measurement | 07 April 2011 | 11 April 2011 | 14 April 2011 | 18 April 2011 |
| Period 2_June | | | | |
| Start measurement | 06 June 2011 | 09 June 2011 | 14 June 2011 | 17 June 2011 |
| Stop measurement | 09 June 2011 | 14 June 2011 | 17 June 2011 | 21 June 2011 |
| Period 3_August | | | | |
| Start measurement | 08 August 2011 | 11 August 2011 | 15 August 2011 | 18 August 2011 |
| Stop measurement | 11 August 2011 | 15 August 2011 | 18 August 2011 | 22 August 2011 |
| Period 4_October | | | | |
| Start measurement | 03 October 2011 | 06 October 2011 | 13 October 2011 | 17 October 2011 |
| Stop measurement | 06 October 2011 | 10 October 2011 | 17 October 2011 | 20 October 2011 |
| Period 5_December | | | | |
| Start measurement | 05 December 2011 | 08 December 2011 | 12 December 2011 | 15 December 2011 |
| Stop measurement | 08 December 2011 | 12 December 2011 | 15 December 2011 | 19 December 2011 |
| Period 6_February | | | | |
| Start measurement | 06 February 2012 | 09 February 2012 | 13 February 2012 | 16 February 2012 |
| Stop measurement | 09 February 2012 | 13 February 2012 | 16 February 2012 | 20 February 2012 |

3.2.4 Test equipment

Equipment used for the test is described in section 4.3 Analytical methods and in Appendix 4 In-house test methods.

3.2.5 Type and number of samples

The sample types and the number of samples to be taken are described in section 4.3.

3.2.6 Operation conditions

Operational parameters like temperature, air humidity, electrical and acid consumption are recorded during the test. A description of the measurement of operational parameters is found in section 4.3.

3.2.7 Operation measurements

The measurement of operational parameters is described in section 4.3.

3.2.8 Product maintenance

Maintenance of the JH-FORSURING NH₄⁺ during the test period is the responsibility of the farm owner. If the farm owner identifies a problem with the JH-FORSURING NH₄⁺ that the farmer cannot solve himself he shall contact Jørgen Hyldgård Staldservice A/S and also inform AgroTech's test staff. As the company marketing the JH-FORSURING NH₄⁺ in Denmark Jørgen Hyldgård Staldservice A/S has the responsibility of repairing the JH-FORSURING NH₄⁺ in case of break down during the test.

Irregularities and break downs during the test period are recorded by AgroTech's test staff.

3.2.9 Health, safety and wastes

Laboratory work during the test will be done according to the Danish rules for safe occupational health and the European regulations regarding work with chemicals. Field work will be done according to Danish rules for safe field work.

Chemicals used for the test are discarded according to Danish regulations for chemical waste by collection and destruction.

It is judged by the AgroTech test staff that the use of the JH-FORSURING NH₄⁺ does not imply any special health, safety or waste issues.

4 REFERENCE ANALYSIS

4.1 Analytical laboratory

Manure samples from the dairy farms where JH-FORSURING NH₄⁺ is installed is analysed by Eurofins Danmark. Address: Smedeskovvej 38, DK-8464 Galten, Denmark. Phone: +45 7022 4266. E-mail: info@eurofins.dk.

Reference analysis is either analysed by AgroTechs or by Analytech Miljølaboratorium A/S, Bøgildsmindevej 21, 9400 Nørresundby. 1 out of 6 manure samples are controlled by the reference laboratorie.

As a reference method for ammonia concentration the Impinger method is used. In this method a fraction of the air is continuously drawn through a pair of impingers (0.5 L each) containing a strong acid solution (sulphuric acid, 0.005 to 0.2 M) and connected in series. An air stream is drawn into the impingers at a fixed flow rate, which is controlled by a critical orifice (usually 1 L min⁻¹).

NH₃ is trapped by the acid and accumulates in the bottles until they are replaced depending on the measurement time. Fluctuations in the NH₃ concentration of the sampled air are thus time-averaged. The values of the sampling flow rate and sulphuric acid concentration are chosen so that the second impinger, which serves as a control, does not contain more than 5% of the amount of NH₃ trapped in the first impinger.

All sampling tubes are made of Teflon, insulated, and heated with a coil of resistance wire to approximately 20°C higher than ambient to prevent condensation of water and subsequent adsorption of NH₃.

Finally, the NH₃ concentration of the air is calculated from the nitrogen content of the acid solution in the bottles, which is determined using a spectrophotometer or a Hach Lange test kit, and the given air sampling flow rate. The impinger method is mainly used in environments with high humidity, as it can deal more easily with moisturized air [2].

Detection limit of the impinge method is 0,000003 mg/m³, and uncertainty is around 15% RSD.

4.2 Analytical parameters

In table 6 the primary analytical parameters are presented. Table 7 presents the operational parameters (conditional measurement parameters).

4.3 Analytical methods

In table 6 the analytical methods of the primary parameter are presented. In table 7 the analytical methods of the operational parameters are presented.

Table 6 shows the primary measurement parameters consisting of the primary environmental pollutant emitted from the livestock housing unit which is the primary target of the environmental technologies for the dairy farms. As seen in Table 6 the primary measurement parameter is ammonia. This technology does not have any effect on odour nor dust. Therefore those parameters are set to zero by default and will not be measured in this test.

Table 7 shows the operational parameters, which include parameters that may influence the emission level of the primary environmental pollutant or which are relevant reference values. In addition the table includes other secondary environmental pollutants.

All analytic parameters listed in those 2 tables are measured at each of the 4 different dairy farms.

Table 6. Primary analytical parameters and corresponding analytical methods.

| Parameter | Analytical method | Number of samples | Sampling time |
|-----------|--|--|---------------|
| Ammonia | ISO 7150/2, NIOSH6015, VDI 2461/1 Innova 1412 | 6 measuring periods evenly distributed during the test over one year | Min 72 hours |

Table 7. Operational and secondary parameters and corresponding analytical methods.

| Parameter | Analytical method | Number of samples | Sampling time |
|--|---|---------------------------------|---|
| CO ₂ | Photoacoustic multigas analyzer/Kitagawa Innova 1412 | 6 | Minimum 72 hours for multigas analyzer. |
| H ₂ S | Jerome 631-X TM | 6 | 30 minutes |
| CH ₄ | Photoacoustic multigas analyzer, Innova 1412 | 6 | Minimum 72 hours |
| N ₂ O | Photoacoustic multigas analyzer, Innova 1412 | 6 | Minimum 72 hours |
| Ventilation rate | Tracer gas method with CO ₂ -balance | 6 | Minimum 72 hours |
| Temperature | VE10 - Temperature sensor | Continuous measurements in situ | |
| Relative humidity | VE14 universal input from VENG system combined with a humidity sensor. | Continuous measurements in situ | |
| Noise | Brüel and Kjær modular precision sound analyzer type 2260. ISO 9001:2000 | 6 | 30 minutes |
| Electricity consumption | VE14 universal input from VENG system combined with a power meter | Continuous measurements in situ | |
| Acid consumption | VE14 universal input from VENG system combined with a power meter | Continuous measurements in situ | |
| pH in manure | Alpha pH 2000W pH meter | Continuous measurements in situ | |
| Manure parameters (M) <ul style="list-style-type: none"> • Amount [kg] [m³] • pH • DM [%] • Organic DM [%] • N [%] [g/kg] • TAN [%] [g/kg] • C:N • P, K • Additives/residues | Acredited laboratorie, se chapter 4.2 | 6 | |
| Wind <ul style="list-style-type: none"> • direction [°] • - speed [m/s] | VE14 universal input from Rotor weather station (Cup anemometer) Placed in kip | Continuous measurements in situ | |

Ammonia analysis

The ammonia concentration is measured with INNOVA 1412, photoacoustic gas detector. This method is used when more frequent continuous measurements, i.e., on a 1 to 5 min sampling basis, are required for the sample air.

In the photoacoustic gas analyzer the exhaust air is continuously sampled at a known flow rate and the concentration of NH₃ in the sample air is determined with the photoacoustic gas analyzer. The NH₃ measurements are corrected for temperature and interference with H₂O and CO₂ [2].

Detection limit and uncertainty

The accuracies of the two techniques described above, as expressed by the standard error under repeatability conditions, show levels that are within the 1% to 3% range (Mosquera et al., 2002; Ogink, 2005) [3].

Detection limit: 0.2 mg/m^3

Ventilation rate

Ventilation rates are required to estimate the amount of gases emitted from dairy buildings. The rate of production (P in m^3h^{-1}) of a specific gas in a dairy building is estimated as:

$$P = q_v (C_g - C_{out}) = q_v \Delta C \quad (1)$$

where q_v (m^3h^{-1}) is the ventilation rate, and C_g (m^3/m^3) and C_{out} (m^3/m^3) are the concentrations of the gas inside and outside the dairy structure respectively. For the above mentioned gas production rate to be valid, the air in the dairy building must be ideally mixed i.e. C_g must be the same all over the building and must not change with time. This is often not the case in real situations. Therefore it is necessary to sample at different locations to get a representative sample of the gas concentration in a dairy buildings.

Sample location for air

Below is a diagram of the sampling procedure. Sampling tubes are installed longitudinal in 3 parallel lines through the stable. Each line has several inlets. All the sample tubes are connected to the photoacoustic gas analyzer in the mobile analyse station. If ΔC is higher than 109 ppm the sample is chosen to represent the inside concentration. The line which is far away from the luv side will normally always be chosen, because the sample inlet on this line has the highest concentration.

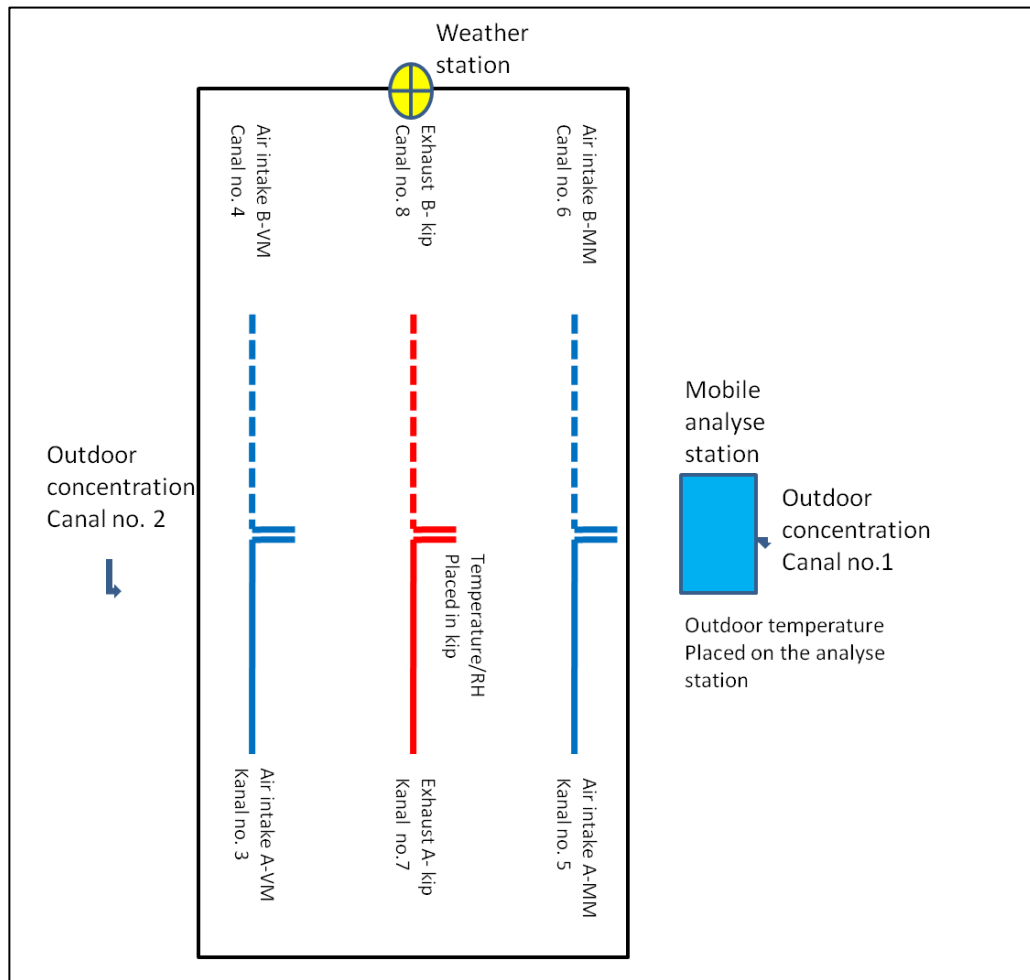


Figure 6. shows a diagram over a dairy building seen from the top and the position of the sampling tubes.

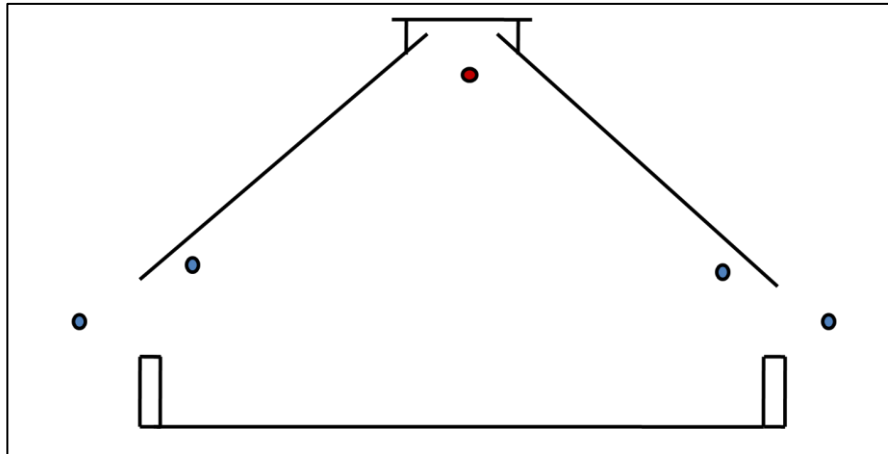


Figure 7 shows a cross section of the stable and the position of the lines of sampling tubes.

Sample location for manure

Manure samples can be obtained from the mixing tank near the acidification system. Sampling is preferably done shortly after mixing. A 10 litre bucket is used to obtain a representative sample. The manure is then homogenized and poured into 2 one liter bottles, see figure 1-5.

4.4 Analytical performance requirements

In table 8 the limits of detection and in some cases the uncertainty of the analytical methods are presented.

Table 8. Limits of detection for the analytical methods used.

| Parameter | Analytical method | Limit of detection | Uncertainty |
|-------------------------|--|----------------------------|-------------|
| Ammonia | ISO 7150/2 | 0,000003 mg/m ³ | 15 % RSD |
| CO ₂ | Photoacoustic multigas analyzer | 1,5 mg/m ³ | --- |
| H ₂ S | Jerome 631-X™ | 0,003 ppm | 5 % RSD |
| CH ₄ | Photoacoustic multigas analyzer | 0,4 mg/m ³ | --- |
| N ₂ O | Photoacoustic multigas analyzer | 0,03 mg/m ³ | --- |
| Ventilation rate | Fan wheel anemometer | --- | --- |
| Temperature | VE10 - Temperature sensor | 0,05 °C | 0,2 °C |
| Relative humidity | VE14 universal input from VENG system combined with a humidity sensor. | --- | --- |
| Noise | Brüel and Kjær modular precision sound analyzer type 2260. ISO 9001:2000 | --- | --- |
| Electricity consumption | VE14 universal input from VENG system combined with a power meter | --- | --- |
| Acid consumption | VE universal input from VENG system combined with a water meter | --- | --- |
| pH in manure | Alpha pH 2000W pH meter | --- | --- |

Note: RSD: Relative standard deviation.

4.5 Preservation and storage of samples

Ammonia

When the impinger method is used the sample must be analyzed as soon as possible after sampling, preferably within 3 hours. The sample must be less than 4 °C until analysis. A representative part of the sample must be kept as a backup in the refrigerator minimum one year.

Manure samples

Immediately after sampling the samples are stored in a cooled box and within 5 hours the samples are placed in a freezer.

5 DATA MANAGEMENT

Data management including filing and archiving procedures are described in the AgroTech Test Centre Quality Manual.

5.1 Data storage, transfer and control

Some data are collected and written down at the test site. Appendix 6 includes data recording sheets to be used for registration of data at the test site.

Some data are collected by electronic means at the test site and send via internet to a PC in the AgroTech main office.

Results from external laboratories are sent electronically by email or in paper version by mail.

Table 9. Data compilation and storage summary.

| Data type | Data media | Data recorder | Data record timing | Data storage |
|-------------------------------------|----------------------|----------------------------|--------------------|--------------------------------|
| Test plan and test report | Protected pdf-files. | Test responsible | When approved | Files and archives at AgroTech |
| Data manually recorded at test site | Data recording forms | Test staff at test site | During collection | Files and archives at AgroTech |
| Calculations | Excel files | Test responsible, AgroTech | During calculation | Files and archives at AgroTech |
| Analytical reports | Paper / pdf-files | Test responsible, AgroTech | When received | Files and archives at AgroTech |

6 QUALITY ASSURANCE

The test will be follow the AgroTech Test Centre Quality Manual, which is ISO 9001 compliant, but not certified.

6.1 Test plan review

The test plan will be subject to internal review by the verification responsible from AgroTech Test Centre.

External review of the test plan will be done by the technical expert assigned to this verification task.

6.2 Performance control – reference analysis

To verify the performance with respect to ammonia a mass balance on nitrogen is made. The purpose is to compare the amount of nitrogen removed from the exhaust air with the amount of nitrogen found in the manure.

6.3 Test system control

The stability of the test equipment will be controlled continuously by supervision and recording of data. Procedures for ensuring that test facilities and equipment are calibrated and fit for the purposes are described in the Quality Manual for the Laboratories of AgroTech. These procedures are subject to internal audits from the AgroTech Management.

6.4 Data integrity check procedures

All transfers of data from printed media to digital form and between digital media are checked by spot check undertaken by test responsible. If errors are found in a spot check, all data transfers from the specific data collection are checked.

6.5 Test system audits

Internal audits from AgroTech will be done following the procedure described in the AgroTech Test Centre Quality Manual.

6.6 Test report review

The test report will be subject to internal review by the verification responsible from AgroTech Test Centre.

External review of the test report will be done by the technical expert assigned to this verification task as part of the review of the verification report. The verification report includes the full test report as an appendix.

7 TEST REPORT

The test report will follow the template of the AgroTech Test Centre Quality Manual and will be included as an appendix in the verification report.

7.1 Test site report

No specific test site report will be made unless it is judged necessary to make this report later. At the test site data are collected and registered on data reporting forms. Templates for data reporting forms are included in this test plan in Appendix 6.

7.2 Test data report

No specific test data report will be made unless it is judged necessary to make this report later. All data recorded during the test including results from external analytical laboratories will be gathered and archived according to the AgroTech Test Centre Quality Manual.

7.3 Amendment report

In the test report there is a section on amendments to and deviations from the test plan. This section will compile all changes of the test plan occurring before testing with justification of deviations and evaluation of any consequences for the test data quality.

7.4 Deviations report

In the test report there is a section on amendments to and deviations from the test plan. This section will compile all changes of the test plan occurring during testing with justification of deviations and evaluation of any consequences for the test data quality.



A P P E N D I X 1

Terms and definitions used in the test plan

| Word | DANETV |
|------------------------------------|--|
| Analytical laboratory | Independent analytical laboratory used to analyse test samples |
| Application | The use of a product specified with respect to matrix, target, effect and limitations |
| DANETV | Danish center for verification of environmental technologies |
| (DANETV) test center | Preliminary name for the verification bodies in DANETV with a verification and a test sub-body |
| Effect | The way the target is affected |
| (Environmental) product | Ready to market or prototype stage product, process, system or service based upon an environmental technology |
| Environmental technology | The practical application of knowledge in the environmental area |
| Evaluation | Evaluation of test data for a technology product for performance and data quality |
| Experts | Independent persons qualified on a technology in verification |
| Matrix | The type of material that the product is intended for |
| Method | Generic document that provides rules, guidelines or characteristics for tests or analysis |
| NOWATECH | Nordic Water Technology Verification Centers |
| Performance claim | The effects foreseen by the vendor on the target (s) in the matrix of intended use |
| Performance parameters | Parameters that can be documented quantitatively in tests and that provide the relevant information on the performance of an environmental technology product |
| Procedure | Detailed description of the use of a standard or a method within one body |
| Producer | The party producing the product |
| Standard | Generic document established by consensus and approved by a recognized standardization body that provides rules, guidelines or characteristics for tests or analysis |
| Target | The property that is affected by the product |
| Test center, test sub-body | Sub-body of the test center that plans and performs test |
| Test center, verification sub-body | Sub-body of the test center that plans and performs the verification |
| Test/testing | Determination of the performance of a product for parameters defined for the application |
| Vendor | The party delivering the product to the customer |
| Verification | Evaluation of product performance parameters for a specified application under de- |



| Word | DANETV |
|------|---|
| | fined conditions and adequate quality assurance |

A P P E N D I X 2

References

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A P P E N D I X 3

References methods



The reference methods are presented in section 4 Reference analysis.



A P P E N D I X 4

In-house test methods



The tests are described in section 3.2 Test.



A P P E N D I X 5

In-house analytical methods

In the following detailed specifications (work instructions) are given for the in-house analytical methods relevant for the JH-FORSURING NH4+.

**Method for measuring ammonium in manure
with Hach Lange test kit**

This method of analysis follows the same chemical principles as ISO 7150-1, 1984, and can be applied to both wash water as sewage. Because the assay reaction is dependent on Ph, may be necessary to neutralize the sample before analysis, especially for the washing of water with low pH.

Principle: Ammonium ions react at pH 12.6 with hypochlorite ions and salicylate ions in the presence of sodium nitro preside as a catalyst, thereby forming indophenol blue

Apparatus: Spectrophotometer: Hach Lange DR 3800 sc VIS

Equipment: Hach Lange Test Kit for analysis of ammonium
Finnpipette 5000 μ l
Stopwatch
Preparation glass with stopper
Div glassware

Neutralization: There may be necessary to neutralize acidic samples, because color is only accurate at pH 4-9. This will typically be the case if samples from detergent bottles. These will be highly acidic and can be neutralized with a weak alkaline solution.

Dilution: If the concentration in samples exceeding the assay measuring range may be necessary to dilute samples before analysis.

Interferences: A very high levels of ammonia could lead to results which are within the range. Is there a suspicion of this should be a dilution of the sample. I doubt should always be made plausibility's control of the analytical result in terms of dilution and / or recovery control.

Please note that the presence of certain ions in high concentrations can have an interference effect on the analytical result. For detailed list of examined interferences, see the accompanying data sheet.

Analysis: Before a Test Kit into use, it is necessary to check the expiration date is stamped on the box is not exceeded.

- Protective film over the lyophilized reagent in DosiCap lid removed, after which the lid screwed by.
- Take 5000 μ l neutralized and diluted sample to the test tube.
- Then turn DosiCap lid and tightened so that the lyophilized reagent is now turning into the test tube.
- Test tube is shaken vigorously to ensure that all freeze-dried reagent is dissolved.
- The reaction should then proceed for 15 min, color remains stable for another 15 min.
- After reaction provides content to NH4-N directly in the spectrophotometer using the predictive method.

Note that the analysis be performed at a temperature in samples of approx. 20 ° C, the temperature differs greatly from this it may mean incorrect analysis.

Data: Did the sample be diluted or neutralized this must be included in the final calculation of the result.

Storage: Before use, Hach Lange Test Kit refrigerates. The sample must be analyzed as soon as possible after sampling, preferably within 3 hours. The sample must be storage un-

der 4 degrees Celsius until analysis. In case an analysis within 3 hours was not possible the samples must be frozen.

Safety: When working with Hach Lange Test Kit used gloves and coat. Work in fume hood or with adequate suction point.

Used tubes assembled in the original package and sent back to Hach Lange in appropriate labeled boxes. Hazard label for transport is included when purchasing the test kit.

Xi



Lokalirriterende

Cuvette contains: Sodium hydroxide

DosiCap contains: Sodium nitro preside, Troclosen Sodium

Xn



Sundhedsskadelig

R-phrases:

- R22 Harmful if swallowed
- R31 liberates toxic gas on contact with acid
- R36/37/38 Irritating to eyes, respiratory system and skin
- R51/53 Toxic to organisms living in water, can cause long term adverse effects on the aquatic environment.

N



Miljefarlig

S-phrases:

- S7 Keep container tightly closed
- S16 Keep away from sources of ignition - No smoking
- S26 of contact with eyes, rinse immediately with water and seek medical advice
- S37/39 Wear suitable gloves and eye / face work
- S45 In case of accident or illness are urgently needed medical care show the label where possible.



A P P E N D I X 6

User manual

2010

Jørgen Hyldgård Staldservice A/S



Egenkontrol af JH-gylleforsøringsanlæg NH4+ fra Jørgen Hyldgård Staldservice A/S.

Mindske risiko for påkørsel og syre udslip i arundvand/fjord.



Rindende vand til rådighed under påfyldning, samt nødbruise system og øjenskylling-hane, som sikrer rindende vand i frostvejr.

Stigeglas holdes under opsyn for at forhindre overfyldning af syretank.

Opstart af anlægget samt indkøring.

Anlægget opstartes med gradvis og tæt tilrettelse af syre under opsyn af montør.

APPENDIX 7A – DATAREGISTRERING

DATO: _____

Besætning: _____

Prøveudtager: _____

| | Ammoniak koncentra- tion | Forsuring Start/stop | Temperatur UD/IND | Fugtighed UD/IND | Hydrogen- sulfid | Støj | Antal Dyr | Strømfor- brug | Syreforbrug |
|------------------|---|---------------------------------|---------------------------------|--------------------------------|-----------------------------|-------------|------------------|---------------------------|--------------------|
| Tidspunkt | | | | | | | | | |
| Enheder | ppm | | ° C | % | ppb | dbe | | KWt/d | l/d |

APPENDIX 7B - LOGBOG

| | April | June | August | October | December | February |
|---|--------------|-------------|---------------|----------------|-----------------|-----------------|
| Amount of feed up-take in the test period (kg, feed units). | | | | | | |
| Feed composition (N, P, K, dry matter) | | | | | | |
| Product composition (g N, P, K) | | | | | | |
| Amount of milk in the test period | | | | | | |
| Digestibility coefficients of the feed ingredients (N, P, K, dry matter). | | | | | | |
| Number of lactating cows | | | | | | |