INFARM
NH4+ acidification system
Biogas yield of fibres from manure treated with sulphuric acid

Verification Report

J.no.1002
Test no.1 Swine manure

23. 04. 2010 version 3

1. Table of Contents

2. Introduction

Environmental technology verification (ETV) is an independent (third party) assessment of the performance of a technology or a product for a specified application, under defined conditions and quality assurance.

DANETV is a Danish centre for verification of environmental technology.

The verification is conducted under the verification protocol for the specific technology tested in accordance with the test plan.

2.1. Name of product

The product is the "INFARM NH4+ acidification".

2.2. Name and contact of vendor

Grundfos New Business A/S, Poul Due Jensens vej 7, DK-8850 Bjerringbro.
Contact: Jesper Ravn Lorenzen, phone: +45 47501400, e-mail: jlorenzen@grundfos.com.

2.3. Name of centre/verification responsible

Danish Technological Institute, Verification Centre, Life Science Division, Kongsvang Allé 29, DK-8000 Aarhus C, Denmark.
Verification responsible: Arne Grønkjær Hansen, phone: +4572202142, e-mail: agha@teknologisk.dk.
Internal reviewer: Nils H. Nilsson (NHN), phone: +45 72201825, e-mail: nhn@teknologisk.dk.

2.4. Verification and test organization

The verification was conducted by Danish Technological Institute.

The test organization is shown in
Figure 1.

The verification was planned and conducted to satisfy the requirements of the ETV scheme currently being established by the European Union (EU ETV).

Verification and tests was performed by Danish Technological Institute under DANETV under contract with Grundfos New Business A/S.

The day to day operations of the verification and tests was coordinated and supervised by TI personnel, with the participation of the vendor, Grundfos New business A/S and the company INFARM A/S.

The samples of acidified fibres were taken from a plant with a well operating INFARM acidification plant as chosen by INFARM/Grundfos after inspection of logged key performance data.

The Subbody at TI test centre performed all samplings during the verification.

INFARM and/or farmer personnel operated the acidification plant and assisted with all tasks described as necessary for verification according to the contract.

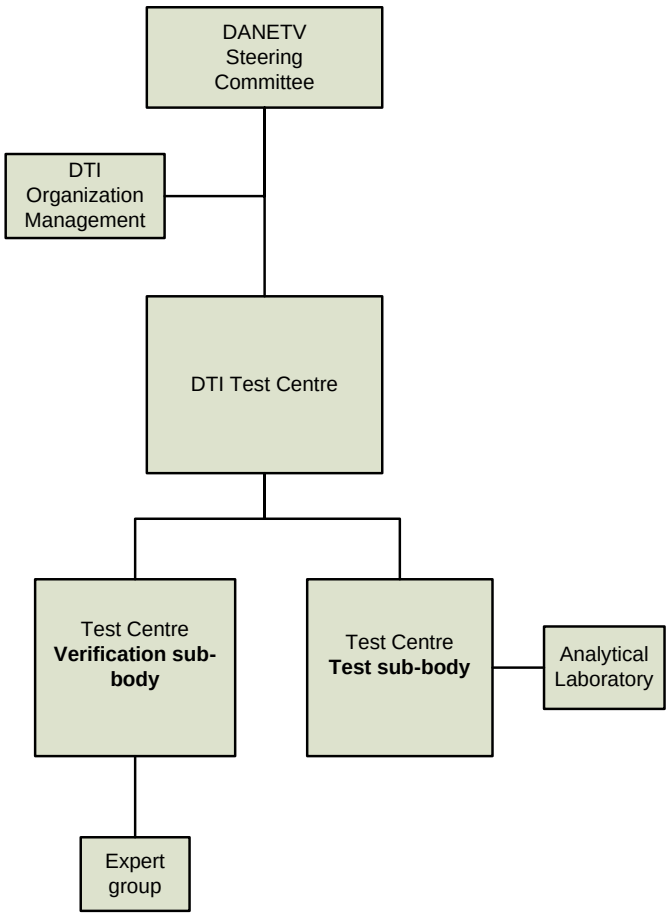

Figure 1 Verification organization

Table 1 Responsible personnel in test organization

Unit in test organization	**Responsible**
DTI Dan ETV steering committee member	Lars Jøker
DTI organization management Life science division	Bo Frølund
DTI Life science division Test Centre, Verification subbody	Arne Grønkjær Hansen
DTI Life science division Test Centre, Test subbody	Bjørn Malmgren-Hansen

2.5. Expert group

The expert group assigned to this verification and responsible for review of the verification plan and report documents includes:

Thorkild Qvist Frandsen (TQF), Agrotech, phone: +45 87438468, email tqf@agrptech.dk.

Lars Ditlev Mørck Ottosen (LDMO), Mikrobiologi, Biologisk Institut, Aarhus Universitet, Bygning 1540, Ny Munkegade 114, 8000 Århus C, phone: +45 89423306, e-mail: lars.ottosen@biology.au.dk.

2.6. Verification process

Verification and tests were conducted in two separate steps, by the Verification sub body and the Test sub-body respectively.

The verification sub-body is responsible for preparation and compilation of the Verification protocol and the Verification report.

The Test sub-body is responsible for the test plan and the test report.

A DANETV verification is issued after completion of the verification report.

The steps in the verification are shown in Figure 2.

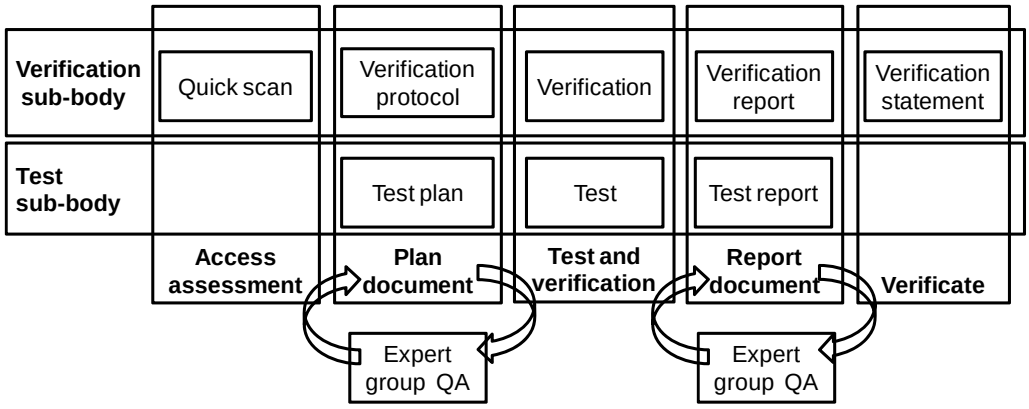

Figure 2 Verification steps

3. Description of the technology

The process is a chemical process for pre-treatment of biomass to reduce loss of ammonia

This is accomplished by adjusting the pH of manure in a tank using sulphuric acid. The target is a pH value at 5.5.

4. Description of the product

The manure acidification technology developed by INFARM A/S is a technology to reduce ammonia emissions from cattle or pig farms by adding sulphuric acid to the slurry. Aeration and homogenization is part of the patented application method.

This verification of the NH4+ acidification system is not a verification of the acidification process on farms but rather a verification of the product from the process, acidified manure fibre as substrate for biogas production.

5. Application and performance parameter definitions

5.1. Matrix/matrices

The matrix is the type of material the product is intended for
The matrix of the verification is chemical pre-treatment of biomass to be used for anaerobic digestion.

Matrix: Biomass for anaerobic digestion
Application: Chemical pre-treatment of biomass

5.2. Target(s)

A target is defined as the property affected by the product
The target of the product is:

- Methane yield of separated treated (acidified) fibres in manure.

5.3. Effects

The effects are described as the way the target is affected
The effects of this application are:

- Change in methane yield for treated biomass compared to untreated biomass in the treatment period of interest in biogas plants (15-30 days).

5.4. Performance parameters for verification

The ranges of performance relevant for the application, as derived in Appendix 3, are presented in Table 2. These ranges are used for planning the verification and testing only.

For added biomass and treated biomass the following parameters were measured:

- Methane potential
- Dry matter
- Volatile solids
- Sulphur

Other performance parameters measured includes:

- pH of acidified manure

5.5. Additional parameters

Besides the performance parameters that are part of test result, compilation of parameters describing, and occupational health & safety issues of the product and user manual were evaluated as part of the verification.

6. Existing data

6.1. Summary of existing data

Data from Aarhus University, (Henrik B. Møller, Ref.6) with varying concentrations of acidified manure mixed with non acidified manure shows an inhibition from the increased sulphur content at higher concentrations. In this study the accumulated biogas production from swine manure after 34 days was approximately:

Table 2 Data for biogas production from acidified manure

% acidified manure	ml Biogas/g VS
25	360
13	575
6	620

From this experiment it is expected that there is no significant inhibition of the anaerobic fermentation when the proportion of manure acidified with sulphuric acid is less than approximately 10% of the total biomass. It is assumed that the same will be the case for biogas production from fibres. Test of this hypothesis is the purpose for this verification.

6.2. Quality of existing data

The data is not obtained for the separated fibres selected for verification, but gives an indication of the inhibition effects.

6.3. Accepted existing data

The existing data is not accepted. Tests with separated fibres from a well performing NH4+ acidification system is needed to verify that inhibition of the biogas process does not occur when separated fibres are used as substrate.

7. Test plan requirements

7.1. Test design

The test design is based on a comparison of methane potential of untreated biomass fibres and biomass fibres treated with the NH4+ process. The fibres are obtained from farms with comparable operation followed by a laboratory measurement of the methane yield.

The effects of the process are tested by:

- Change in methane yield in tests with acidified fibres compared to non acidified reference fibres.

The non acidified reference fibres were retrieved from a farm with comparable operation (type of production, feeding, stable design etc.) to the fibres retrieved from a farm with acidification.

Inhibition caused by the increased content of sulphur was tested by using different concentration levels of acidified fibres in the inoculum.

A suitable method is to use different concentrations of acidified fibres mixed with reference fibres with the same total concentration of volatile solids in all tests. The amount of total volatile solids added in bio gasification tests should be of the typical amount used in bio gasification plants which is in the range 25gVS/l or more for fibres from separated swine manure. Concentrations of acidified fibres added should be around the value stated in vendor claims. At least 3 concentrations of acidified fibres should be used in tests and in addition a test with reference fibres without acidified fibres should be performed.

The detailed test design is given in the test plan.

Measuring methane potential
Methane potential was measured on reference biomass and treated biomass from the farms which have comparable conditions in the stables (same age of pigs, feeding, use of straw, construction etc.).

The methane potential is measured according to the method for measuring methane potential described in ref. 5.

The result is a calculation of (l CH4 /VS of added biomass) for treated and non treated biomass as function of time for mesophilic bio gasification.

The dry matter (DM) and volatile solids (VS) content of the samples to be tested was analyzed before performing biogas tests.

Sampling
To obtain homogeneous, representative samples the following sampling plan has been made.

Sampling of acidified manure
Samples are taken by etc. 15 litre buckets lowered into the stirring tank of the NH4+ plant
The content is transferred to 50 litre storage containers.

The sample is taken when the plant has pumped in manure and acidified it to the set point.

The manure must be stirred during sampling.

A subsample is taken for measurement of pH within one day.

Sampling of untreated manure
Untreated manure may either be retrieved from a stirred tank like acidified manure above or from a separator separating fibres of interest.

Treatment of samples
As the test focus on the fibre content, the fibres of treated and non-treated fibres shall be separated using an appropriate and comparable method. This means that fibres of comparable size should be separated using etc. sieving techniques.

The selected method will be described in the test plan.

Test of plant operation
During the test period (14 days-1 month before the test) the operational stability and deviations from normal operational functioning shall be observed and registered, through inspection of the acidification plants data logger and the observations reported in the test report. pH is measured on the acidified manure samples immediately after sampling and compared with internal pH measurement of the plant.

7.2. Reference analysis

For batch tests of methane potential a reference component in the medium will be tested as described in ref. 5.

7.3. Data management

Data storage, transfer and control must be done in accordance with the requirements of ISO 9001 (or compatible systems) enabling full control and retrieval of documents and records.

7.4. Quality assurance

The quality assurance of the tests includes control of the reference system, control of the test system and control of the data quality and integrity.
The test plan, test and verification protocol and the test report has been reviewed by the expert group see Figure 2.

7.5. Test report

The test report follows the template of the TI verification centre quality manual /2/ with data and records from the tests presented.

8. Evaluation

8.1. Performance parameter summary

The average figures of performance parameters are shown in Table 3.

Table 3 Target and measured values of tested parameters.

Parameters	Target	Measured value	Method
Overall performance			
pH regulation	5.5	5.68	Calibrated pH meter
Chemicals			
Sulphuric acid liter/stable/day		64	Calculated from log file weighing cells
Fibre quality			
Sulphur content mg/kg DM		32333	Analysis
DM %		12.2	Analysis
Inhibition in Biogasification at 5 and 10% mixture with nonacidified fibers		No inhibition	Methane potential (mesophilic 35˚C)
Inhibition in Biogasification at 20% mixture with nonacidified fibers		Beginning inhibition	Methane potential (mesophilic 35˚C)
Operation			
User manual		Checked	Checked according to manual

The pH regulation in the INFARM acidification system seems to be well operating since pH of separated manure was very close to set point of 5.5. Inspection of the datalog for 4 stables and a period of one month showed that >95% of all measurements where within +/-0.1 pH unit of the set point.

The addition of sulphuric acid was controlled by pH and not a fixed amount per stable.

The purpose of the present verification was not a complete verification of an INFARM NH4+ system but a verification of the fibre quality regarding use as substrate in biogas production.

Biogas tests
Biogas tests with addition of different amounts of acidified fibres to reference fibres showed the same amount of produced methane at 5 and 10% addition of acidified fibres as the reference fibres after 30 days of biogasification at mesophilic conditions.

Main results from biogas test are depicted in Figure a7.7 in test report (repeated below).

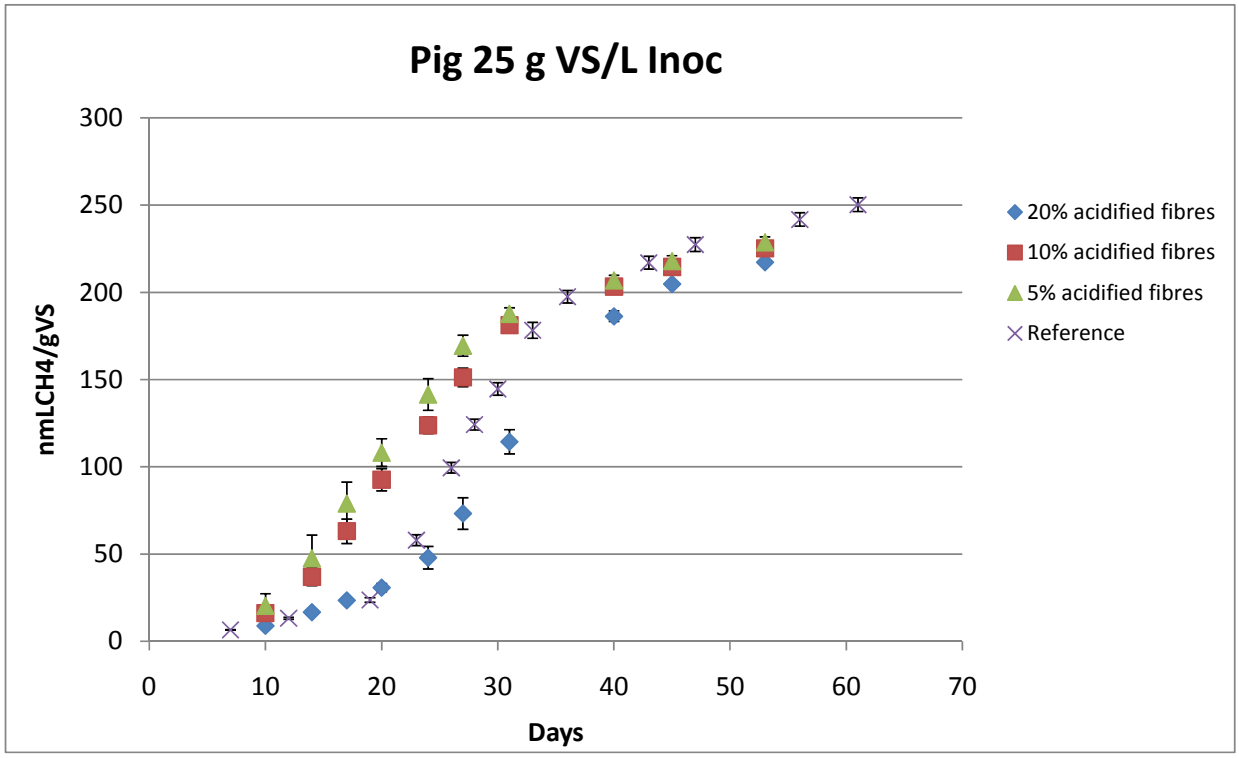

Fig a7.7 Accumulated methane production for 25 g VS/l (average of a triple analysis) 5, 10, 20% was measured from 20/11-2009 until 12/1-2010 whereas the reference (0%) was measured from 4/11-2009 to 4/1-2010. Standard deviations for each triple measurement are shown in the figure.

It is observed that there is no inhibition of the mixed methane production in the bio gasification tests at 10% addition of acidified fibres. There is a inhibition (delay) at 20% addition of acidified fibres although the same accumulated methane production is obtained after 30-35 days of active growth (when correcting for 10 days initial lag phase corresponding to data at day 40-45).

It is concluded for the INFARM NH4+ system that:

- there is no significant inhibition in biogas production with addition of 10% acidified separated fibres with a dry matter content(DM) of 12% to non acidified fibres
- there may be inhibition by addition of 20% acidified fibres (DM=12%) to non acidified fibres
- From the results of tests with 100 % acidified fibres there is a significant risk of inhibition using acidified fibres only and a high H2S concentration in the gas phase must be expected
- The hydrogen sulphide concentration in the gas phase is increased by approximately a factor of four with addition of 10% acidified fibres (DM=12%) to reference fibres
- When comparing conditions in test incubators and full scale plants and results from N_2 flushing of incubators it is likely that full scale biogasification plants will have less inhibition than the incubators used in this test (where pressure builds op between samplings and no stirring exist).

8.2. Evaluation of test data quality

The data quality is evaluated to be at scientific level and standard deviations of repeated measuring were all very low, which is a good indicator that repeated tests will show same results and therefore the data can be used as a good indicator. However the method being af batch laboratory test set up does not simulate the real conditions in biogas plants.

8.2.1. Control data

The test results were checked using parallel tests in two laboratories of the following parameters:

- Dry matter (DM), Volatile solids (VS), Selected tests of Methane Potential.

8.2.2. Audits

No audits were performed.

8.2.3. Deviations

Standard deviation of all relevant parameters are calculated and depicted as bars on the graphs in the testreport.

The test plan has been followed - No deviations from test plan are registered.

8.3. Additional parameters summary

8.3.1. User manual

The manuals for use and technical manuals for the INFARM NH4+ acidification plant (version 2007) were read thoroughly and the description of the following topics was found sufficient:

- Operation of the system
- Prevention of and dealing with incidents
- Occupational health and safety measures
- Service and maintenance
- Surveillance of the installation

8.3.2. Occupational health and environment

The safety instructions in the user manual regarding handling of chemicals (sulphuric acid) are regarded as sufficient.

Recommendations for verification statement

It is recommended to issue a verification statement exclusively on acidified fibres as substrate for biogasproduction and emphasize that this typical product from the INFARM NH4+ process had no negative effects on biogasproduction under laboratory conditions given that concentrations of the product did not exceed 10 %. In this case it is can only be verified that fibres derived from acidified manure with a specific pH and sulpur content has no negative impact on methane production in laboratory test set up when diluted to a concentration of maximum 10 %. It is assumed that inhibition occurs at a higher concentration in full scale biogas, but no guarantees can be given. Therefore the following liability exclusions should be included in the verifications statement.

9. Liability exclusion

ETV verifications are based on an evaluation of technology performance under specific, predetermined operational conditions and parameters and the appropriate quality assurance procedures. DTI makes no expressed or implied warranties as to the performance of the technology and do not certify that the technology will always operate as verified.
The end user is solely responsible for complying with any applicable regulatory requirements.

10. Quality assurance

The test protocol, test plan, test report and verification report was reviewed by internal and external experts according to the Quality plan for the verification, see Table 4.

Table 4 QA plan for the verification

Reviewers	TI	Experts
Plan document with application definition, verification protocol and test plan	NHN	TQF, LDMO
Report document with test report and verification report	NHN	TQF, LDMO

Reviews were done using the TI review report template.

Appendix 1 Terms and definitions used in the verification protocol

Terms and definitions used in the protocol are explained in Table 1.

Table 1 Terms and definitions used by the DANETV test centres.

Word	DANETV	Comments on the DANETV approach
Analytical laboratory	Independent analytical laboratory used to analyse test samples	The test centre may use an analytical laboratory as subcontractor
Application	The use of a product specified with respect to matrix, target, effect and limitations	The application must be defined with a precision that allows the user of a product verification to judge whether his needs are comparable to the verification conditions
DANETV	Danish centre for verification of environmental technologies	
(DANETV) test centre	Preliminary name for the verification bodies in DANETV with a verification and a test sub-body	Name will be changed, when the final nomenclature in the EU ETV has been set.
Effect	The way the target is affected	The effect could be concentration reduction, decrease in treatment period, pH increase etc
(Environmental) product	Ready to market or prototype stage product, process, system or service based upon an environmental technology	The product is the item produced and sold and thus the item that a vendor submit for verification
Environmental technology	The practical application of knowledge in the environmental area	The term technology is covering a variety of products, processes, systems and services.
Evaluation	Evaluation of test data for a technology product for performance and data quality	None

Word	DANETV	Comments on the DANETV approach
Experts	Independent persons qualified on a technology in verification	These experts may be technical experts, QA experts for other ETV systems or regulatory experts
Matrix	The type of material that the product is intended for	Matrices could be soil, drinking water, ground water etc.
Method	Generic document that provides rules, guidelines or characteristics for tests or analysis	An in-house method may be used in the absence of a standard, if prepared in compliance with the format and contents required for standards.
Performance claim	The effects foreseen by the vendor on the target (s) in the matrix of intended use	None
Performance parameters	Parameters that can be documented quantitatively in tests and that provide the relevant information on the performance of an environmental technology product	The performance parameters must be established considering the application(s) of the product, the requirements of society (regulations), customers (needs) and vendor claims
Procedure	Detailed description of the use of a standard or a method within one body	The procedure specifies implementing a standard or a method in terms of e.g.: equipment used
Producer	The party producing the product	None
Standard	Generic document established by consensus and approved by a recognized standardization body that provides rules, guidelines or characteristics for tests or analysis	None
Target	The property that is affected by the product	Targets could be *e.g.*. contaminant concentration
Test centre, test sub-body	Sub-body of the test centre that plans and performs test	None

Word	DANETV	Comments on the DANETV approach
Test centre, verification sub-body	Sub-body of the test centre that plans and performs the verification	None
Test/testing	Determination of the performance of a product for parameters defined for the application	None
Vendor	The party delivering the product to the customer	Can be the producer
Verification	Evaluation of product performance parameters for a specified application under defined conditions and adequate quality assurance	None

Appendix 2 References (verification protocols, requirement documents, standards, methods)

1. DANETV. Centre Quality Manual, 2008
2. European Parliament and Council. Directive 2006/42/EC of the 17th May 2006 on machinery and amending Directive 95/16/EC (recast).
3. European Council: Directive 89/655/EEC of 30 November 1989 concerning the minimum safety and health requirements for the use of work equipment by workers at work (amended 2007/30/EC).
4. ISO 12100-2:2003: Safety of machinery - Basic concepts, general principles for design - Part 2: Technical principles
5. Measurement protocol for methane potential measurements for ETV tests at DANETV
6. Henrik B. Møller, Status og udfordringer for forsuringsteknologien I relation til biogasproduktion, Aarhus University, Power point presentation.

Appendix 3 Application and performance parameter definitions

This appendix defines the application and the relevant performance parameters application as input for verification and test of an environmental technology following the DANETV method.

A3.1 Applications

A3.1.1 Matrix/matrices

A3.1.2 Target(s)

A3.1.3 Effects

The effects claimed by the vendor are presented in Table 2.

Table 2 Performance parameters and vendor claims

Performance parameter	Vendor claim of performance
Methane potential	No significant inhibition in biogasification of acidified fibres from separated swine manure when added in concentrations up to 10% of the total amount of added separated fibres