



SB Engineering SB 250 Slurry separator

Test Report



Version 2-0





Document information

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2 INTRODUCTION

This test report is made for the verification of SB Engineering slurry separator model SB 250 following the AgroTech Test Centre Quality Manual.

2.1 Verification protocol reference

This test report is made to meet the requirements defined in the verification protocol for SB Engineering slurry separator model SB 250.

2.2 Name and contact of vendor

SB 250 slurry separator is developed and produced by SB Engineering Aps, Bjergegaardsvej 10, DK-9620 Aalestrup, Denmark. Contact person is Søren Brams. Phone: +45 9864 3178. E-mail: mail@sbrams.dk.

SB 250 is marketed and sold by AL-2 Agro, Kroegebaekvej 25, DK-6682 Hovborg, Denmark. Contact person is Preben Nissen. E-mail: <u>pbn@al-2.dk</u>. Phone: +45 3169 6501.

2.3 Name of centre and verification responsible

The test was performed by DANETV Verification Centre AgroTech, Udkaersvej 15, DK-8200 Aarhus N, Denmark.

Test responsible: Hans Jørgen Tellerup, AgroTech, Udkaersvej 15, 8200 Aarhus N. Phone: +45 8743 8406. E-mail: <u>hjt@agrotech.dk</u>.

2.4 Expert group

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

Internal expert: Bjørn Hjortshøj Andersen, AgroTech, Udkærsvej 15, DK-8200 Århus N, phone: +45 8743 8420, e-mail: <u>bha@agrotech.dk</u>.

External expert: Maibritt Hjorth, Aarhus University, Faculty of Agricultural Sciences. E-mail: Maibritt.Hjorth@agrsci.dk. Phone: +45 8999 1932.

3 TEST DESIGN

The SB 250 slurry separator was tested in full-scale on a commercial farm. The test was carried out under normal operational conditions that reflect how the separator is used by a farmer at farm level.

The test was designed so that mass balances of total solids (TS), volatile solids (VS), total nitrogen, ammonium -nitrogen, phosphorus and potassium could be calculated.





3.1 Test site

3.1.1 Characterization of the test site

The test took place at a commercial cattle farm in Himmerland region of Denmark. At the test farm there is a livestock production of 400 dairy cows. All cows are in gathered in one big cow house, which is naturally ventilated. The cattle farm is operated as a conventional (non-organic farm). The cows are kept inside the cattle house throughout the year. The cows are milked using a manual milking system. As bedding material for the cows in the cattle house pulverised straw is used.

3.1.2 Addresses

The test was undertaken at Risgaard I/S, Svinget 10, GI. Hvam, DK-9620 Aalestrup. Contact person of Risgaard I/S is Jørgen Risgaard. E-mail: <u>is-risgaard@mail.dk</u>.

3.1.3 Descriptions

SB 250 is a slurry separator of the screw press type. This separator is functioning mechanically and no additives are used in the process. The SB 250 separator used for the test is installed in a container. However, SB 250 can also be delivered as a mobile unit if the separator is intended for use at more than one farm location.

Slurry is led to the screw press from a small slurry collection tank or directly from the slurry channel in the animal house.

The slurry is transported into a cylindrical screen with a screw. The diameter of the openings in the screen of the separator in the test is $250 \ \mu\text{m}$ (this is reflected in the name of the model, SB 250). However, the screen can easily be replaced with screens with other diameters. Normally screens with selected diameters within the range of 250 $\ \mu\text{m}$ and 1000 $\ \mu\text{m}$ are used when slurry is separated.

The liquid passes through the screen and is collected in a container surrounding the screen. To remove even more liquid the solid fraction is pressed against a plate at the end of the axle. The solid fraction drops out from the opening between the plate and the opening of the cylindrical mesh.

From the screw press the solid fraction is transported to a closed container. At the test site the container has a capacity of approximately 8 tonnes. When the container is full it is transported to an anaerobic digestion plant where the solid fraction is used as sub-strate for biogas production.

The liquid fraction is pumped from the separator to a big storage slurry tank with a cover. Here the liquid fraction is stored and later applied to land as liquid manure. Figure 1 shows how the SB 250 separator is installed in a container.





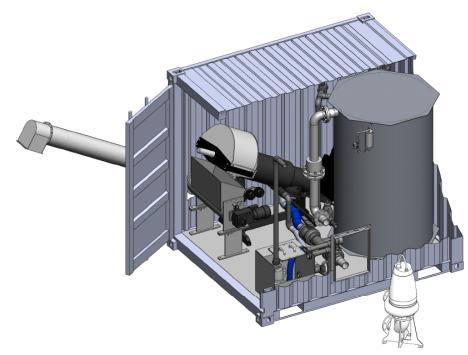


Figure 1. SB 250 separator installed in a container.

3.2 Tests

3.2.1 Test methods

To determine the separation efficiency of the SB 250 slurry separator a mass balance is calculated for total solids (TS), volatile solids (VS), total nitrogen (N_{Tot}), ammonium nitrogen (N_{ammo}), phosphorus (P) and potassium (K).

This is done by taking representative samples of all inputs to and all outputs from the slurry separator during operation. Since the separation process is mechanical and no additives are used the only input is the cow slurry led to the separator from the collection tank at the cattle house.

There are two outputs from the separator:

- A liquid fraction with a lower content of dry matter than the input to the separator.
- A solid fraction rich in dry matter.

Since the separation process takes place in a closed system (inside the separator) and since the separation process is relatively fast, it is assumed that the amounts of nitrogen and other nutrients lost as gaseous emissions during processing are very low. Thus, it is judged not to be relevant to measure any potential gaseous outputs from the separator as part of this test.

For the input flow and each of the two output flows the <u>weight</u> and the <u>concentrations</u> of the relevant components were measured as part of the test in order to calculate the mass balance.

Figure 2 below illustrates schematically the input to and the two output flows from the SB 250 slurry separator.





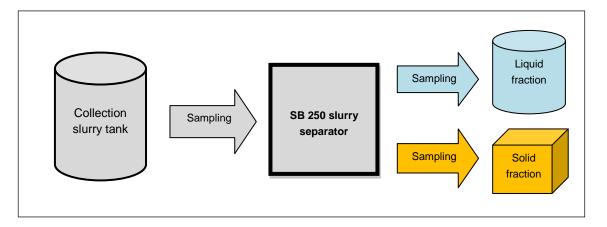


Figure 2. Illustration of the input stream and the two output streams from the SB 250 slurry separator.

3.2.2 Test staff

The test staffs involved in the test of SB 250 are:

Marie Louise Nielsen, AgroTech, Udkaersvej 15, 8200 Aarhus N. E-mail: <u>mln@agrotech.dk</u>.

Hans Jørgen Tellerup, AgroTech, Udkaersvej 15, 8200 Aarhus N. Phone: +45 8743 8406. E-mail: <u>hjt@agrotech.dk</u>.

3.2.3 Test schedule

The test schedule is presented in the table below.

Task	2009				2010						
Month	6	7	8	9	10	11	12	1	2	3	4
Test plan	Х	Х									
Practical planning		Х	Х								
Test start (26.08.2009)			Х								
Period reserved for sampling			Х	Х							
Chemical analyses					Х						
Methane yield test					Х	Х	Х				
Test report draft							Х	Х			
Test report quality assurance										Х	Х
Test report final version											Х

Table 1. Test schedule for SB 250 slurry separator.

3.2.4 Test equipment

The test included 5 batches. Each batch lasted minimum 2 hours at normal operation of the separator.

At the beginning of each batch:

• The settings of the separator was checked:





- Pressure into the separator: 200-250 mbar
- o Counter pressure: 22,5 bar
- Collection of solid fraction in an empty container was prepared.
- A flowmeter of the liquid output fraction was read and the figure registered
- The time when the batch was started was registered
- An electricity meter was read and the figure registered

During the batch:

- The time when the sub-samples are taken
- The settings of the separator was checked and the figures registered
 - Pressure into the separator: 200-250 mbar
 - Counter pressure: 22,5 bar
- In case of any irregularities these were registered.

At the end of the batch:

- The time when the batch stopped was registered (minimum duration is 2 hours)
- The flowmeter of the liquid output fraction was read and the figure registered
- The collection of solid output fraction in container was stopped.
- The electricity meter was read and the figure registered.

After the batch has ended the container was weighed using the weight at DLG, Vest-Himmerland, Aars. Then the solid fraction was unloaded from the container and the now empty and clean container was weighed again. The result is the net weight of the produced solid fraction during the specific batch.

3.2.5 Type and number of samples

During each batch at least 3 sub-samples were taken from the input and each of the two output flows from the slurry separator. In each set of sub-samples the input flow is sampled first. After 10 - 20 seconds (corresponding to the treatment time of the slurry inside the separator) sub-samples of the two output streams were taken. The sub-samples of the two output streams were taken simultaneously by two persons. The intention was to have a set of sub-samples where the two output sub-samples match a sub-sample of what is led into the separator at the same time.

The first set of sub-samples was taken after approximately 30 minutes of the batch. The other sets of sub-samples were distributed evenly over the rest of the batch.

When the batch was completed the sub-samples were mixed and 4 pooled samples were taken:

3 pooled samples for chemical analyses:

- 0,8 L of input slurry
- 0,8 L of liquid output fraction
- 1 L of solid output fraction

1 pooled sample for methane yield determination:

• 1 L of solid output fraction

The 4 pooled samples were cooled down immediately (<5 degree C) and frozen within 6 hours.





3.2.6 Operation conditions

During the batch a number of operational parameters were registered from the relevant meters of the slurry separator:

- Flowmeter of the liquid output fraction (m³/hour)
- Pressure into the separator (mbar)
- Counter pressure (bar)

Each parameter was read and registered minimum 3 times during a batch using the data reporting form included in Appendix 6 of the test plan.

In case of any irregularities or stops of the separator during the batch these were registered in the data reporting form, too.

3.2.7 Operation measurements

The electricity consumption of the separator during the batch was registered as described in section 3.2.4 above.

3.2.8 Product maintenance

The need for product maintenance was not evaluated as part of this test.

3.2.9 Health, safety and wastes

Issues related to health, safety and wastes were not evaluated as part of this test.

4 REFERENCE ANALYSIS

4.1 Analytical laboratory

Application of other methods according to both international standard methods or inhouse methods that are validated as required for accredited methods.

For chemical analyses the 5 sets of pooled samples were sent to Eurofins Steins laboratory, Hjaltesvej 8, DK-7500 Holstebro. Website: <u>www.eurofins.dk</u>. E-mail: <u>agro@eurofins.dk</u>.

Eurofins Steins laboratory maintains an ISO 17025 accreditation with the quality management system required herein. Eurofins Steins applies accredited analytical methods, where available.

Measurement of methane yield of the solid fraction was done by the AgroTech internal laboratory, Udkaersvej 15, 8200 Aarhus N. Responsible for the methane yield test was laboratory technician Merete Maahn.

4.2 Analytical parameters

In table 2 the analytical parameters included in the test are presented.





4.3 Analytical methods

In table 2 the analytical methods of the analytical parameters are presented.

Parameter	Unit	Measured in	Analytical method
Total nitrogen	Kg/ton	Input biomass Liquid output fraction Solid output fraction	Kjeldahl / Dumas
Ammonium nitrogen	Kg/ton	Input biomass Liquid output fraction Solid output fraction	71/393/EØF
Total phosphorous	Kg/ton	Input biomass Liquid output fraction Solid output fraction	ICP/OES
Total potassium	Kg/ton	Input biomass Liquid output fraction Solid output fraction	ICP/OES
Total solids, TS	%	Input biomass Liquid output fraction Solid output fraction	EØF 103°C, 20 hours
Ash content	%	Input biomass Liquid output fraction Solid output fraction	DS204
рН	pH unit	Input biomass Liquid output fraction Solid output fraction	Radiometer, GLP
Methane yield	Nm ³ /ton	Solid output fraction	Modified version of ISO 11734 (see test method description in Appendix 4)

Table 2. Analytical parameters and corresponding analytical methods.

From the results of these analyses the following parameters will be calculated:

- Organic nitrogen (kg/ton) as the difference between total-N and ammonium-N
- Volatile solids, VS (%) as the difference between total solids and ash content.

For all parameters except pH and methane yield a mass balance will be calculated. The mass of each parameter pumped into and out of the separator was calculated as the weight of the input and the two output fractions respectively multiplied by the average concentration in the three flows determined from chemical analyses of the samples.

The test method used to determine the methane yield of the solid output fraction was a modified version of the ISO 11734-standard. The test method is described in Appendix 4. For the present test it was judged by the test staff, that N-inhibition would take place and consequently no pre-test was performed.

The solid output fraction was characterised by a homogenous structure allowing for taking representative samples. Consequently, no conditioning of the test material (solid output fraction) was done.





The methane yield test was carried out at 48 degree Celcius using inoculum from Baanlev Biogas Plant. The test period of the methane yield test was 90 days.

4.4 Analytical performance requirements

The mass of each parameter entering the SB 250 slurry separator should equal the mass of that parameter that leaves the separator in both the solid and liquid fraction.

To evaluate the validity of the mass balance it was calculated to what extent the mass led into the separator was recovered in the solid and liquid output fractions (expressed as a percent).

$$R_{i} = \frac{M_{i}^{I} - (M_{i}^{L} + M_{i}^{S})}{M_{i}^{I}} \times 100$$

Where:

 $R_i =$ Percent of component *i* not recovered in liquid or solid output fraction

 $M_i^{I,L,S}$ = Mass of component *i* in input slurry (I), liquid (L) or solid output fraction (S)

In each batch for every parameter:

• R_i (per batch) has to be less than +/- 25%

In cases where R_i (per batch) is larger than +/- 25% the result has to be omitted from the calculation of separation efficiency. If possible the batch is repeated.

All batches together for every parameter:

• R_i (all batches) has to be less than +/- 15%.

In cases where R_i (all batches) is larger than +/- 15% the result has to be omitted from the calculation of separation efficiency. If possible the test is repeated.

4.5 Preservation and storage of samples

The 4 pooled samples were cooled down immediately and frozen within 6 hours. When all 5 batches were completed (and 35 days after the first samples were taken) the samples for chemical analyses were unfrozen and sent to Eurofins Steins laboratory together with written requisitions specifying what types of analyses to be performed.

5 DATA MANAGEMENT

Data management including filing and archiving procedures are described in the Agro-Tech Test Centre Quality Manual [5].

5.1 Data storage, transfer and control

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





Results from external laboratories were sent to AgroTech test staff electronically by email or in paper version by mail.

Table 3. 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 followed the AgroTech Test Centre Quality Manual, which is ISO 9001 compliant, but not certified.

6.1 Test plan review

The test plan was subject to internal review by the verification responsible from Agro-Tech Test Centre.

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

6.2 *Performance control – reference analysis*

6.2.1 Mass balances

The mass of each parameter led into the SB 250 slurry separator should equal the mass of that parameter that leaves the system in the solid and liquid fraction.

To evaluate the validity of the mass balance it was calculated to what extent the mass led into the separator was recovered in the solid and liquid output fractions (expressed as a percent).

$$R_{i} = \frac{M_{i}^{I} - (M_{i}^{L} + M_{i}^{S})}{M_{i}^{I}} \times 100$$

Where:

 R_i = Percent of component *i* not recovered in liquid or solid output fraction

 $M_i^{I,L,S}$ = Mass of component *i* in input slurry (I), liquid (L) or solid output fraction (S)

In each batch for every parameter:





• R_i (per batch) has to less than +/- 25%

In cases where R_i is larger than +/- 25% the result for the specific batch has to be omitted from the calculation of separation efficiency for that specific parameter. If it is not possible to repeat the test the test responsible will assess whether some of the pooled samples should be re-analysed.

All batches together for every parameter:

• R_i (all batches) has to less than +/- 15%.

In cases where R_i is larger than +/- 15% seen over 5 batches the separation efficiency cannot be calculated for that respective parameter. If possible the test including all 5 batches is repeated. If it is not possible to repeat the whole test the test responsible assesses whether some of the pooled samples should be re-analysed.

6.2.2 Methane yield tests

As part of the quality control a reference compound, cellulose powder, has been tested parallel with the samples of solid fraction. The reference compound was tested like the samples of solid fraction (triplicate samples using same inoculum). This control is used to show both the variation between the individual samples and the variation between average values compared to average values from previous test series.

6.3 Test system control

The stability of the test equipment (e.g. flowmeters, weighing machines, etc.) is 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 have been 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 are done following the procedure described in the Agro-Tech Test Centre Quality Manual [5].

6.6 Test report review

The test report was subject to internal review by the verification responsible from Agro-Tech Test Centre.

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





7 TEST REPORT

The test report follows 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 has been made since it is not judged relevant to make as part of this test. At the test site data were collected and registered in the data reporting forms found in Appendix 6 of the test plan.

7.2 Test data report

No specific test data report was made since it was not judged relevant to make this as part of this test. All data recorded during the test including results from external analytical laboratories have been gathered and archived according to the AgroTech Test Centre Quality Manual.

7.3 Amendment report

In this test report there is a section (section 8.4) on amendments to and deviations from the test plan. This section compiles changes of the test plan occurring <u>before</u> 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 compiles all changes of the test plan that occurred <u>during</u> testing with justification of deviations and evaluation of any consequences for the test data quality.

8 TEST RESULTS

8.1 Test performance summary

The test started on the 26th of August 2009 with completion of the first batch out of 5 batches included in the test. The last batch (number 5) on the farm was completed on the 22nd of September 2009. The table below presents dates for all 5 batches and the duration of each batch.

Batch	Date	Length of batch	Amount of treated	Amount of solid	Amount of liquid
number		(hours:minutes)	slurry (m ³)	fraction (kg)	fraction (kg)
1	26.08.2009	4:00	26,58	4.580	22.000
2	02.09.2009	4:00	25,35	4.220	21.130
3	08.09.2009	4:00	21,34	3.760	17.580
4	22.09.2009	3:00	19,69	3.720	15.970

Table 4. Overview of the 5 batches in the test.





5	22.09.2009	2:24	16,15	3.120	13.030
Average		3:29	21,82	3.880	17.942
batch 1-5					

In average over the 5 batches the SB 250 separator was running with a capacity of 6,3 tonnes of slurry treated per hour.

8.2 Test measurement summary

The table below presents the average content of total solids, ashes, volatile solids and pH of input slurry, liquid output fraction and solid output fraction respectively.

Table 6. Average content of total solids, ashes, volatile solid and pH over 5 batches.

Fraction	Total solids (%)	Ash content (%)	Volatile Solids* (%)	рН (pH units)
Input slurry	7,88	1,60	6,28	6,85
Liquid output fraction	4,95	1,50	3,45	6,86
Solid output fraction	22,39	2,06	20,33	7,58

*Note: Values for volatile solids are calculated as the difference between total solids and ash content, not measured.

In table 6 below are shown the average concentrations of nutrients in the input slurry, the liquid output fraction and the solid output fraction respectively.

Fraction	Total Nitrogen (Kg/ton)	Ammonium Nitrogen (Kg/ton)	Organic Nitrogen* (Kg/ton)	Total phosphorous (Kg/ton)	Total potassium (Kg/ton)
Input slurry	3,7	1,72	1,98	0,50	3,30
Liquid output fraction	3,48	1,73	1,75	0,47	3,40
Solid output fraction	4,59	1,45	3,14	0,65	3,08

*Note: Values for organic nitrogen are calculated (difference between total-N and ammonium-N), not measured.

For each batch a mass balance on the nutrients is calculated. The mass balances are based on the measured concentrations of the nutrients and the masses of input and the output fractions. In table 7 the results are presented.

The mass of the input slurry is calculated as the mass of liquid output fraction plus mass of solid output fraction.

In converting the measured volume of liquid output fraction to mass, the density of the treated biomass is approximated to 1 ton/m³.

The share of the nutrients recovered in the solid fraction is added to the share of the nutrient recovered in the liquid fraction as control. Due to uncertainty in sampling and analysing the samples the control does not sum up to 100%.





Batch no.	Fraction	Total Nitro-	Ammonium-	Organic Ni-	Total phos-	Total potas-
		gen %	Nitrogen %	trogen %	phorous %	sium %
	O all'id autout					
	Solid output	21	11	31	22	16
1	Liquid output	77	81	74	73	83
	Control	99	92	105	95	99
	Solid output	21	14	27	22	16
2	Liquid output	77	85	71	78	89
	Control	98	99	98	100	105
	Solid output	22	16	27	22	16
3	Liquid output	78	81	76	79	85
	Control	100	96	103	101	101
	Solid output	24	18	28	24	17
4	Liquid output	76	82	71	75	81
	Control	100	100	99	98	98
	Solid output	24	18	28	28	18
5	Liquid output	77	82	72	81	86
	Control	100	100	100	108	104
	Solid output	22	15	28	23	17
Average	Liquid output	77	82	73	77	85
	Control	99	97	101	101	101

Table 7. Mass balances for nutrients.

It is seen in table 7 that in average for 5 batches the mass balances of total nitrogen, ammonium nitrogen, organic nitrogen, phosphorous and potassium sum up to 99 %, 97 %, 101 %, 101 %, and 101 % respectively. In other words, the nutrients in the input biomass are well recovered in the liquid and solid output fractions. In table 8 the mass balances for total solids, ashes, volatile solids, and treated biomass are presented.

Batch no.	Fraction	Total solids	Volatile Solids*	Amount of treated input slurry
		%	%	%
	Solid output	49	56	17
1	Liquid output	52	46	83
	Control	101	101	100
	Solid output	49	56	17
2	Liquid output	51	44	83
	Control	100	100	100
	Solid output	51	59	18
3	Liquid output	53	47	82
	Control	104	105	100
	Solid output	53	60	19
4	Liquid output	51	44	81
	Control	104	104	100
	Solid output	53	60	19
5	Liquid output	51	44	81
	Control	103	104	100
	Solid output	51	58	18
Average	Liquid output	52	45	82
	Control	102	103	100

Table 8. Mass balances for total solids, volatile solids, and treated biomass.

*Note: Mass balances of volatile solids are based on calculated concentrations, not measures concentrations.





The mass balances presented in table 7 and table 8 have been adjusted so that they sum up to 100 %. The adjusted mass balances are presented in table 9 and table 10 respectively as the best estimates for the performance of the SB 250 slurry separator.

Batch no. 1 - 5	Fraction	Total Nitro- gen %	Ammonium- Nitrogen %	Organic Ni- trogen %	Total phos- phorous %	Total potas- sium %
	Solid output	22	16	28	23	17
Average	Liquid output	78	84	72	77	83
	Control	100	100	100	100	100

Table 9. Adjusted mass balances of nutrients to make masses of the nutrients sum up to 100 %.

Table 10. Adjusted mass balances of solids to make masses of the solids sum up to 100 %.

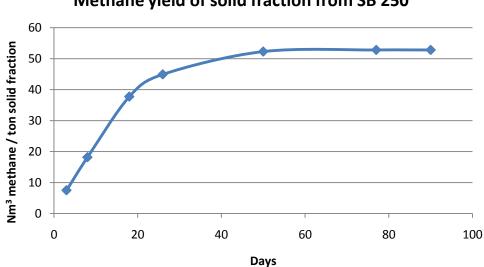
Batch no.	Fraction	Total solids	Volatile Solids	Treated input slurry
1 - 5		%	%	%
	Solid output	50	56	18
Average	Liquid output	50	44	82
	Control	100	100	100

In table 11 the results of the methane yield measurements are summarised. The detailed data are found in Appendix 6.

Table 11. Methane yields (90 days) of solid fraction from separation of cattle slurry using SB 250.

Substrate	Methane yield Nm ³ / ton VS	Methane yield Nm ³ / ton solid fraction
Solid fraction from SB 250 slurry separator	263	52,8

In figure 3 the results of the methane yield test are presented in a graph showing the accumulated methane production as a function of time.



Methane yield of solid fraction from SB 250

Figure 3. Accumulated methane yield per ton solid fraction (fresh weight)





In table 12 the measured capacity of the SB 250 separator and the results of the electricity consumption measurements are presented.

Batch no.	Capacity	Electricity consumption
	Tons of input slurry treated per hour	kWh / ton input slurry
1	6,65	0,83
2	6,34	0,83
3	5,33	0,89
4	6,56	0,71
5	6,73	0,74
Average	6,3	0,80

Table 12. Capacity and electricity consumption.

8.3 Test quality assurance

Calculation of mass balances showed that the analytical performance requirements have been met. Consequently, no analytical results from the samples have been omitted in the calculation of the separation efficiencies. The R_i (per batch) values and R_i (all batches) are presented in the table below (see definition of R_i values in section 6.2.1).

Batch	Total	Ammonium-	Organic	Total phos-	Total	Total	Volatile
no.	Nitrogen	Nitrogen	Nitrogen	phorous	potassium	solids	Solids
1	1,37%	8,02%	-4,73%	4,58%	1,01%	-0,96	-1,42
2	1,57%	1,19%	1,89%	0,01%	-4,69%	0,36	0,19
3	0,18%	3,59%	-2,75%	-0,93%	-0,87%	-4,40	-5,44
4	0,50%	-0,06%	1,00%	1,58%	1,72%	-3,60	-4,01
5	-0,09%	0,07%	-0,23%	-8,40%	-3,84%	-3,35	-4,26
1-5	0,71%	2,56%	-0,97%	-0,63%	-1,33%	-2,39	-2,99

Table 13. Percent of components not recovered in liquid or solid output fraction (R_i values).

The methane yield test had to be repeated because the test on reference material (Avicel, cellulose material) did not meet the analytical performance requirements during the first test. When the test was carried out the second time the methane yields on the reference material were within the accepted range (See methane yields of reference material in Appendix 6).

In table 14 the results of the total solids and volatile solids analyses of Eurofins Steins laboratory are compared with the results from AgroTech's internal laboratory.

Table 14. Comparison of average values for total solids and volatile solids (5 bate	ches).
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Laboratory	Total solids (%)	Volatile solids (%)	Volatile solids / Total solids (%)
Eurofins Steins	22,39	20,33	90,80
AgroTech laboratory	22,07	20,10	91,07





8.4 Amendments to and deviations from test plan

According to the test plan Marie Louise Nielsen was responsible for the test and responsible for part of the sampling and measurement activities. However, since Marie Louise Nielsen moved to a new job during the test period, Hans Jørgen Tellerup has taken over the responsibility for the test. This delayed the analysing and reporting phase of the test.

As mentioned in section 8.3 the methane yield test had to be repeated because the test on reference material (Avicel, cellulose material) did not meet the analytical performance requirements during the first test.





Terms and definitions used in the test plan





Word	DANETV	
Analytical labo- ratory	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	
Liquid fraction	Liquid or thin fraction derived from the separation of slurry.	
Performance claim	The effects foreseen by the vendor on the target (s) in the matrix of in- tended use	
Performance parameters	Parameters that can be documented quantitatively in tests and that pro- vide 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	
Recovery factor	Expresses the proportion of a given component in the input slurry that end up in a specific output stream. The recovery factor is expressed as a percent.	
Separation effi- ciency	In this verification separation efficiency is defined as the recovery factor for the solid fraction. Thus separation efficiency is a measure of the pro- portion of a given component in the input slurry that ends up in the solid	





Word	DANETV
	fraction (expressed as a percent).
Slurry	Faeces and urine produced by housed livestock, usually mixed with some bedding material and some water during management to give a liquid manure with a dry matter content in the range from about $1 - 10\%$. A slurry is a mixture of liquid and solid materials, where typically the solid materials are not dissolved in the liquid phase, and will precipitate out of the slurry under a prolonged period of storage.
Slurry additive	Manufactured or naturally occurring products or substances that are added to manures to modify their biological, chemical or physical proper- ties. Many additives are commercially available but most have not been subjected to independent testing so their effectiveness has not been as- sessed.
Slurry separator	Slurry separators (separation technologies) are here defined as technolo- gies that divide liquid livestock manure (slurry) into one or more solid frac- tions and one or more liquid fractions.
Solid fraction	A fraction from separation with a higher content of solids (e.g. dry matter or phosphorus) than the input material. Normally the solid fraction is stackable.
Standard	Generic document established by consensus and approved by a recog- nized standardization body that provides rules, guidelines or characteris- tics 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 defined conditions and adequate quality assurance





References





- [1] Hjorth, M., Christensen, K.V., Christensen, M.L. & Sommer, S.G. (2009): Solid-liquid separation of animal slurry in theory and practice. A review. Agronomy for Sustainable Development.
- [2] Angelidaki, I., Alves, M., Bolzonella, D., Borzacconi, L., Campos, J.L., Guwy, A.J., Kalyuzhnyi, P., Jenicek, P. & van Lier, J.B. (2009): Defining the biomethane potential (BMP) of solid organic wastes and energy crops: a proposed protocol for batch assays. Water Science & Technology – WST. 59.5.
- [3] Malmgren-Hansen, B. & Ottosen, L.D.M. (2009): Measurement protocol for biogas potential measurements for verification tests (ETV, CBMI).
- [4] SB Engineering (2009): Separation SB 250. Product description. Not published.
- [5] AgroTech (2009): AgroTech Test Centre Quality Manual. Not published.





References methods





In the table below the analytical methods used in this test are listed.

Parameter	Unit	Measured in	Analytical method
Total nitrogen	Kg/ton	Input biomass Liquid output fraction Solid output fraction	Kjeldahl / Dumas
Ammonium nitrogen	Kg/ton	Input biomass Liquid output fraction Solid output fraction	71/393/EØF
Total phosphorous	Kg/ton	Input biomass Liquid output fraction Solid output fraction	ICP/OES
Total potassium	Kg/ton	Input biomass Liquid output fraction Solid output fraction	ICP/OES
Total solids, TS	%	Input biomass Liquid output fraction Solid output fraction	EØF 103°C, 20 hours
Ash content	%	Input biomass Liquid output fraction Solid output fraction	DS204
рН	pH unit	Input biomass Liquid output fraction Solid output fraction	Radiometer, GLP
Methane yield Nm ³ /ton		Solid output fraction	Modified version of ISO 11734 (see test method description in Appendix 4)

Table A1. Analytical methods used for analysing the relevant parameters for the test.





In-house test methods





AgroTech's protocol for batch test measurement of methane yields

Introduction to test method

This test method is a modified version of the method described in Angelidaki et. Al (2009) [2]. The modifications have been discussed and coordinated with Bjørn Malmgren-Hansen and Lars D. M. Ottosen, Danish Technological Institute.

Thus, the test method follows the same principles of the biogas measuring protocol produced by Malmgren-Hansen and Ottosen [3] but there are a few exemptions.

Purpose

The purpose of the test is to measure the methane yield of a given substrate used for biogas production.

The test is based on performing batch biogasification with degassed inoculum from a biogas plant and added media with recording of produced biogas amounts and content of methane.

The biogasification is performed for:

- Test material
- Inoculum (blank test)
- Reference material

Furthermore, for unknown test materials it can be relevant to carry out a pre-test to determine whether inhibition is taking place. In the inhibition test varying concentrations of test material are used.

Test material, blanks and reference material are tested as triplicate tests.

Conditioning of test material

Samples must be representative of the biomass to be tested and with a homogeneous structure allowing for taking representative subsamples. Procedures for correct conditioning of test material and sub-sampling must be described elsewhere as it will depend on the structure of the specific biomass.

Handling and storing of samples

Samples of test material are taken in e.g. 1 litre PE bottles, filled only 80% allowing for freezing.

If testing cannot be performed immediately, the samples are frozen.

Materials

- Infusion bottles which can withstand a pressure of 2 bar (volume ¹/₂-1 litre)
- Brown butyl rubber stoppers + Al Crimps
- Measurement device for measuring volume of produced gas. Volume are measured directly with a volume collection tube (water filled gas collecting cylinder).
- Reference material.





Conditions

Incubation takes place at temperatures within the mesophilic interval or thermophilic interval. The temperature used during the test should match the temperature of the biogas reactor from which the inoculums for the test is collected.

The incubation temperature must be verified in the thermostating equipment within at least +/- 1°C using calibrated temperature measurement devices. When infusion bottles are removed for gas volume measurement, the period of storage outside the incubation chamber should be minimized (<20 minutes).

Inoculum

Biomass from biogas plant degassed 2-3 days at temperature of interest (mesophilic or thermophilic).

pH of inoculums is measured and must be between 6,5 and 8,5. If it is relevant the NH_4 -N content is measured and the concentration should be below 4 g/l unless a special test condition is chosen.

Test period

If the purpose of the test is to determine the methane potential the test period is minimum 90 days. However, the test period may be shortened depending on the purpose of the test.

Sufficient measurements (10-15) should be made so that a curve can be made showing the development of methane production over time.

When running comparisons of biomasses, process treatments etc. inoculum from the same batch of biomass shall be used to decrease uncertainty from blank subtraction.

Methane potential test in infusion bottles

Inoculum of known volume/weight and test material are added to the infusion bottles.

There must be 40-60% free space in the bottles allowing for accumulation of gas.

Addition of inoculum:

Preferred conditions:

500 ml infusion bottles: 200 ml inoculum (measured with 0,1% accuracy) 1000 ml infusion bottles: 400 ml inoculum (measured with 0,1% accuracy)

Addition of test material (biomass):

TS and VS of test material shall be known before addition. Test material is added within a range that gives sufficient sensitivity and no inhibition. For unknown test materials it can be relevant to determine the exact concentration in an inhibition pre-test.

Typical concentrations of test material are expected to be in the range 10 - 30 g VS per liter inoculum. The added amount is measured with 0,1% accuracy.

The infusion bottles are flushed with N_2 for 2 minutes before testing.

Blanks

Triplicate tests are performed on inoculum for each new batch of inoculum.

The infusion bottles for blanks are flushed with N₂ for 2 minutes before testing.





Reference

A reference material (like sodium benzoate, cellulose powder) shall be run in inoculum (triplicate tests) for each new batch of inoculum.

The reference samples are flushed with N₂ for 2 minutes before testing.

Amount of produced biogas

Volume can be calculated as pressure increase (ISO 11734) in headspace or measured directly with a volume collection tube (syringe or water filled gas collecting cylinder). Efforts must be made to ensure no loss of process gas (ensuring gastight connections by pressure test).

CH₄ and CO₂ contents

The CH₄ and CO₂ content of the produced biogas shall be measured by appropriate measuring equipment, for instance gas chromatography (method description in Biomass and Energy v26, 2004, p.487). CH₄ and CO₂ contents should be measured for each measurement point during test. GC must be calibrated using reference gas each day.

pН

pH is measured on inoculum batch before test. pH is measured in infusion bottles after finished biogasification as control of inhibitory acidification. The measurement may be reduced to 1 pH measurement of triplicates showing same biogas production curves.

Result

For each measurement point, the volume of produced methane is calculated and expressed in Nml methane/ g VS (0 degrees C and 101325 Pa) . Volume of produced methane (Nml / g VS) in blanks is subtracted.

A sum curve of produced (net) Nml methane / g VS as function of time is calculated and plotted.

All raw data on produced biogas volume and methane shall be available upon request.





In-house analytical methods





In the table below the analyses carried out by AgroTech laboratory are mentioned and the method used is described.

Table A2. In house analytical methods used during the test.

Analysis	Method used
Total solids measurement for setting up the me-	EØF 103 degrees Celcius, 20 hours.
thane yield measuring test	
Volatile solids measurement for setting up the me-	DS204
thane yield measuring test	





Test data report





Analytical results from Eurofins Steins Laboratory covering all samples are presented in the table below.

				Results from analyses of samples								
Batch	Date	Code	Fraction	Total-N	NH4-N	Org N	Р	к	TS	Ashes	VS	рН
No.				kg/t	kg/t	kg/t	kg/t	kg/t	%	%	%	Unit
1	26.08.2009	A1	Slurry input	3,78	1,81	1,97	0,52	3,40	7,93	1,60	6,33	6,86
1	26.08.2009	B1	Liquid output	3,53	1,77	1,76	0,46	3,40	4,98	1,50	3,48	6,78
1	26.08.2009	C1	Solid output	4,68	1,16	3,52	0,67	3,20	22,54	2,00	20,54	7,66
2	02.09.2009	A2	Slurry input	3,72	1,67	2,05	0,50	3,20	7,92	1,60	6,32	6,94
2	02.09.2009	B2	Liquid output	3,45	1,71	1,74	0,47	3,40	4,86	1,50	3,36	6,96
2	02.09.2009	C2	Solid output	4,72	1,35	3,37	0,65	3,10	23,07	2,00	21,07	7,86
3	08.09.2009	A3	Slurry input	3,72	1,72	2,00	0,50	3,40	7,82	1,60	6,22	6,88
3	08.09.2009	B3	Liquid output	3,53	1,69	1,84	0,48	3,50	5,04	1,50	3,54	6,90
3	08.09.2009	C3	Solid output	4,57	1,51	3,06	0,62	3,10	22,77	2,10	20,67	7,67
4	22.09.2009	A4	Slurry input	3,67	1,71	1,96	0,50	3,30	7,86	1,60	6,26	6,80
4	22.09.2009	B4	Liquid output	3,44	1,73	1,71	0,46	3,30	4,92	1,50	3,42	6,86
4	22.09.2009	C4	Solid output	4,56	1,63	2,93	0,63	3,00	21,98	2,20	19,78	7,49
5	22.09.2009	A5	Slurry input	3,63	1,71	1,92	0,46	3,20	7,89	1,60	6,29	6,79
5	22.09.2009	B5	Liquid output	3,44	1,73	1,71	0,46	3,40	4,94	1,50	3,44	6,81
5	22.09.2009	C5	Solid output	4,44	1,62	2,82	0,66	3,00	21,58	2,00	19,58	7,21

Table A3. Concentrations of nutrients in input slurry, liquid output fraction and solid output fraction.

Results from methane yield test are presented in the 2 tables below.

Table A4. Methane yields expressed in <u>Nm³/ton VS</u>.

code no.	Substrate	Days after start of methane yield test						
		3	8	18	26	50	77	90
3	Solid fraction, bottle 1-3	35,9	96,7	200,7	237,2	265,9	268,7	268,7
7	Solid fraction, bottle 4 - 6	38,9	84,0	174,8	209,6	254,2	256,6	256,6
Average	Solid fraction, bottle 1 – 6	37,4	90,3	187,7	223,4	260,1	262,6	262,6

Table A5. Methane yields expressed in Nm³/ton solid fraction – fresh weight.

Code no.	Substrate	Days after start of methane yield test						
		3	8	18	26	50	77	90
3	Solid fraction, bottle 1-3	7,2	19,4	40,3	47,7	53,5	54,0	54,0
7	Solid fraction, bottle 4 - 6	7,8	16,9	35,1	42,1	51,1	51,6	51,6
Average	Solid fraction, bottle 1 – 6	7,5	18,2	37,7	44,9	52,3	52,8	52,8

Table A6. Biogas composition.

Biogas composition			Days after start of methane yield test								
Component	Unit	3	3 8 18 26 50 77 90								
Methane	%	34,7	44,6	55,3	65,6	54,5	53,3	55,80			
CO2	%	22,4	28,6	27,5	33,3	41,6	32,4	45,10			
O ₂	%	0,6	4,8	3,4	0,4	1,1	1,9	0,45			
H₂S	ppm	363	434	392	579	823	629	917			





In figure A1 and A2 below is presented the results of the reference material. In this test 2 reference tests have been undertaken: One reference test using Avicel, old deliverance and one reference test using Avicel, a new deliverance.

The measured methane shall be within the range shown in the figures as a two grey curves. It is seen that for both the old and the new deliverance of the reference material the measured methane yields lie within the accepted range.

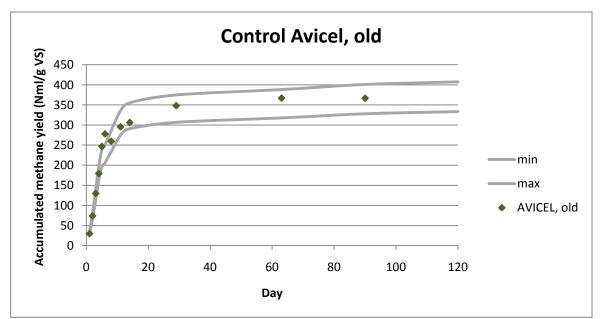


Figure A1. Measured methane yields of reference material Avicel, old (adjusted for yields of blind test).

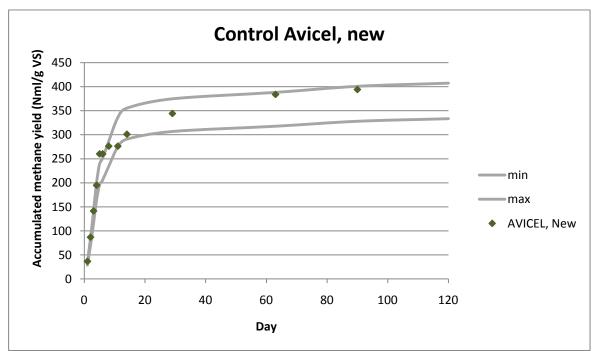


Figure A2. Measured methane yields of reference material Avicel, new (adjusted for yields of blind test).





Amendment and deviation reports for test





During the test the following deviations from the test report were registered:

The methane yield test had to be repeated because the test on reference material (Avicel, cellulose material) did not meet the analytical performance requirements during the first test.

During the test one of the persons involved, Marie Louise Nielsen moved to a new job. Consequently, a new person, Hans Jørgen Tellerup, was involved in the test.