

# Application Fibre

## D.7.b. FIBRE THERM Crude Fibre in Animal Feed

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## 1 Principle

When doing a crude fibre analysis, the parts that are not soluble with sulphuric acid and potassium hydroxide such as cellulose, hemicellulose, and lignin are determined. The residual, undigested material is dried, weighed, and then incinerated. The difference between the content of ash and undigested parts is the crude fibre. Use of FibreBags simplifies the process of dissolution and filtration. The most important part in fibre analysis is the exact observation of the respective times for the boiling stages as well as the weighing procedures.

## 2 Method

The chemicals as well as the boiling times correspond to:

- AOAC Official Method 962.09 - Fiber (Crude) in Animal Feed and Pet Food
- Methodenbuch des Verbandes Deutscher Landwirtschaftlicher Untersuchungs- und Forschungsanstalten, Band III, Die chemische Untersuchung von Futtermitteln; Rohfaser 6.1.1
- Amtliche Sammlung von Untersuchungsverfahren nach §64 LFGB, Untersuchung von Futtermitteln, Bestimmung des Rohfasergehaltes in Futtermitteln, F0010EG, September 2010

This application document is intended to be a guide to assist users in the initial use of C. Gerhardt analytical equipment. It is not a definitive method. Users may have to adapt this method to the ambient conditions and to suit their own analytical requirements.

## 3 Chemicals and Accessories

Quality p. a.

1. Water distilled or demineralised
2. Sulphuric acid c ( $\text{H}_2\text{SO}_4$ ) = 0.13 mol/l
3. Potassium hydroxide solution c (KOH) = 0.23 mol/l, alternatively: Sodium hydroxide solution c (NaOH) = 0.313 mol/l
4. Petroleum Ether, Boiling Range 40 to 60 °C
5. Acetone p.a
6. FibreBags RF for crude fibre determination, bag with 100 pcs, cat. no. 10-0128
7. Incineration Module for FibreBags, 12-place, complete with handle and 12 quartz glass crucibles, cat.no. 13-0092
8. Degreasing module/ ADL- module made of PTFE, for the simultaneous degreasing of up to 6 samples, incl. handle and beaker 1000 ml, OrderNo.: 13-0045

## 4 Instruments

- FIBRE THERM FT 12, cat.no. 13-0026, with accessory
- FRITSCH Pulverisette P14, sieve ring 1mm
- Glas Spacer for FibreBags, to open and fix the bags, Package with 6 pieces, Order No.: 10-0124
- Desiccator with a drying agent as silica gel
- Drying chamber, electric driven, temperature 100°C +/- 5°C
- Muffle furnace, temp. 500 °C +/-25°C
- Timer or alarm clock
- Fume cabinet

## 5 Sample Type and Preparation

Approx. 5 - 100 g of the feed are grinded down to a particle size of approx. 1 mm.

- It is important to ensure the mass constancy of the FibreBags, if necessary it is recommended to dry the necessary number of FibreBags in the drying chamber

at 105 +/- 1 °C for 1 hour. The weight of the FibreBags is value M1 for the balance protocol. When storing the FibreBags in the desiccator they only have to be dried once and then, can be weighed directly.

- The glass spacers are put into the FibreBags.
- The weight of the sample to be put into the FibreBag should be around 1 g and should be weighed with 1 mg preciseness; this gives value M2 for the weighing protocol.
- The glass spacers with the samples are inserted into the FIBRETherm.
- The blank value is determined in parallel to the regular analysis.
- The dry matter of the sample should be determined separately and is important for the calculation of the crude fibre content (result related to the dry matter).
- De-fatting of the sample is important especially for samples with a fat content higher than 10 %:  
The carousel is immersed three times in a row into 40/60 petroleum ether. By turning it as well as moving it up and down the sample is defatted. This facilitates the washing and filtration process, which will follow. Furthermore, no crude fibre content is lost. The first petroleum ether fraction is thrown away but the following can be re-used. After a short drying process in the fume cupboard (about 2 minutes) the samples are inserted into the FIBRETherm.

**Table 1: Analysis Parameter FIBRETherm**

**Settings Rinsing**

1.	Water filling	1.3	Liter	
2.	Circulation Time	9	Seconds	
3.	Circulation Pause	4	Seconds	
4.	Suction	2 to 3 min	min / s	The boiling vessel should be empty.
5.	Wash out time	1 min	min / s	

**Settings Preheating**

1.	Circulation Time	10	Seconds	
2.	Circulation Pause	30	Seconds	
3.	Heating delay after start	30	Seconds	
4.	ADF / H <sub>2</sub> SO <sub>4</sub> heat up to	80 to 85	°C	The sample solution should boil.
5.	NDF / KOH heat up to	80 to 85	°C	The sample solution should boil.
6.	H <sub>2</sub> O-Wash heat up to	80 to 85	°C	The sample solution should boil.
7.	ADF / H <sub>2</sub> SO <sub>4</sub> cool down to	85	°C	
8.	NDF / KOH cool down to	85	°C	
9.	H <sub>2</sub> O-Wash cool down to	85	°C	

**Method Settings**

1.	Method Definition	1	No.	
2.	<A> Detergent	H <sub>2</sub> SO <sub>4</sub>	Type	
3.	<A> Add	1.3	Liter	
4.	<A> Heating Power	40 to 45	%	The sample solution should boil gently.
5.	<A> Circulation Time	10	s	
6.	<A> Circulation Pause	30	s	

7.	<A> Boiling Time	0 h 30 min	h / min	
8.	<A> Suction	2 to 3 min	min / s	The boiling vessel should be empty.
9.	<A> Rinsing	2	Cycles	
10.	<B> Detergent	KOH	Type	
11.	<B> Add	1.3	Litre	
12.	<B> Heating Power	40 to 45	%	The sample solution should boil gently.
13.	<B> Circulation Time	10	s	
14.	<B> Circulation Pause	20	s	
15.	<B> Boiling Time	0 h 30 min	h / min	
16.	<B> Suction	2 to 3 min	min / s	The boiling vessel should be empty.
17.	<B> Rinsing	2	Cycles	
18.	<C> Detergent	H <sub>2</sub> O-Wash	Type	
19.	<C> Add	1.3	Liter	
20.	<C> Heating Power	55	%	
21.	<C> Circulation Time	10	s	
22.	<C> Circulation Pause	15	s	
23.	<C> Boiling Time	0 h 5 min	h / min	
24.	<C> Suction	2 to 3 min	min / s	The boiling vessel should be empty.
25.	<C> Rinsing	0	Cycles	
26.	<D> Detergent	-----	Type	
27.	<D> Add		Liter	
28.	<D> Heating Power		%	
29.	<D> Circulation Time		s	
30.	<D> Circulation Pause		s	
31.	<D> Boiling Time		h / min	
32.	<D> Suction		min / s	
33.	<D> Rinsing		Cycles	
34.	Meth. X defined Time :		h / min	

#### Drying of the FibreBags

The FibreBags are taken out of the carousel. The glass spacers are removed from the FibreBags rinsing them carefully with water. Alternatively, the incineration module (s. Appl. D.7.b.1. FibreBag Procedure with Incineration Module 2015\_german) can be used to standardize the method.

The drained FibreBags are put into crucibles, which has been pre-ashed at 500 °C +/- 25°C and are dried in the drying chamber at 105 °C for minimum 4 hours or overnight. Then, the crucibles with FibreBags are put into the desiccator to cool down to ambient temperature and are weighed. The weight of FibreBag and crucible after digestion and drying is value M3.

#### Incineration of Samples



The FibreBags are incinerated at 500 °C +/- 25°C for at least 4 hours or overnight. The FibreBags should be placed in the cold muffle furnace if possible, especially if the FibreBags are ashed together with the glass spacers. This procedure considerably increases the service life of the glass spacers. The vapours produced during the combustion process are harmless.




After incineration, the crucibles are left to cool in the drying chamber at 105°C for 30 minutes and then in the desiccator to cool down to ambient temperature. Then, they are weighed and value M4 (for crucible and ashes) is obtained for the weighing protocol.

## 6 Calculation

The crude fibre is calculated either automatically by use of the Excel calculation macro Crude Fibre or traditionally by the formulas shown in this macro:

**Table 2:** Calculation Macro Crude Fibre

**Calculations Crude Fibre**

**Formula for the calculation**  

$$\% \text{ Crude fibre} = ((M_3 - M_1 - M_4) - (B_3 - B_1 - B_4) \times 100) / M_2$$

$$\text{Blank value} = B_3 - B_1 - B_4$$

**Denominations:**  
M1 = FibreBag [g]  
M2 = Initial sample weight [g] (ev. correct with dry content)  
M3 = Incinerating crucible and dried FibreBag after digestion [g]  
M4 = Incinerating crucible and ash [g]  
B1 = Blank value of the empty fibre bag [g]  
B3 = Incinerating crucible and dried FibreBag Blank value after digestion [g]  
B4 = Incinerating crucible and ash Blank value [g]

**Operator** \_\_\_\_\_

B1 [g]	B3 [g]	B4 [g]	Blank value [g]

Date / Time 25.04.2013 / 15:21

FibreBag number	Sample number	M1 [g]	M2 [g]	M3 [g]	M4 [g]	% Crude fibre

## 7 Reasons for Failures

Result too low:

Reason	Reaction
Cooling time	Insufficient cooling time will lead to minute errors in weighing (weight loss). A strict observance of the weighing procedure is recommended.
Broken FibreBags	Sample is lost from the FibreBag due to leaking or broken bags. Check FibreBag before using it for the analysis

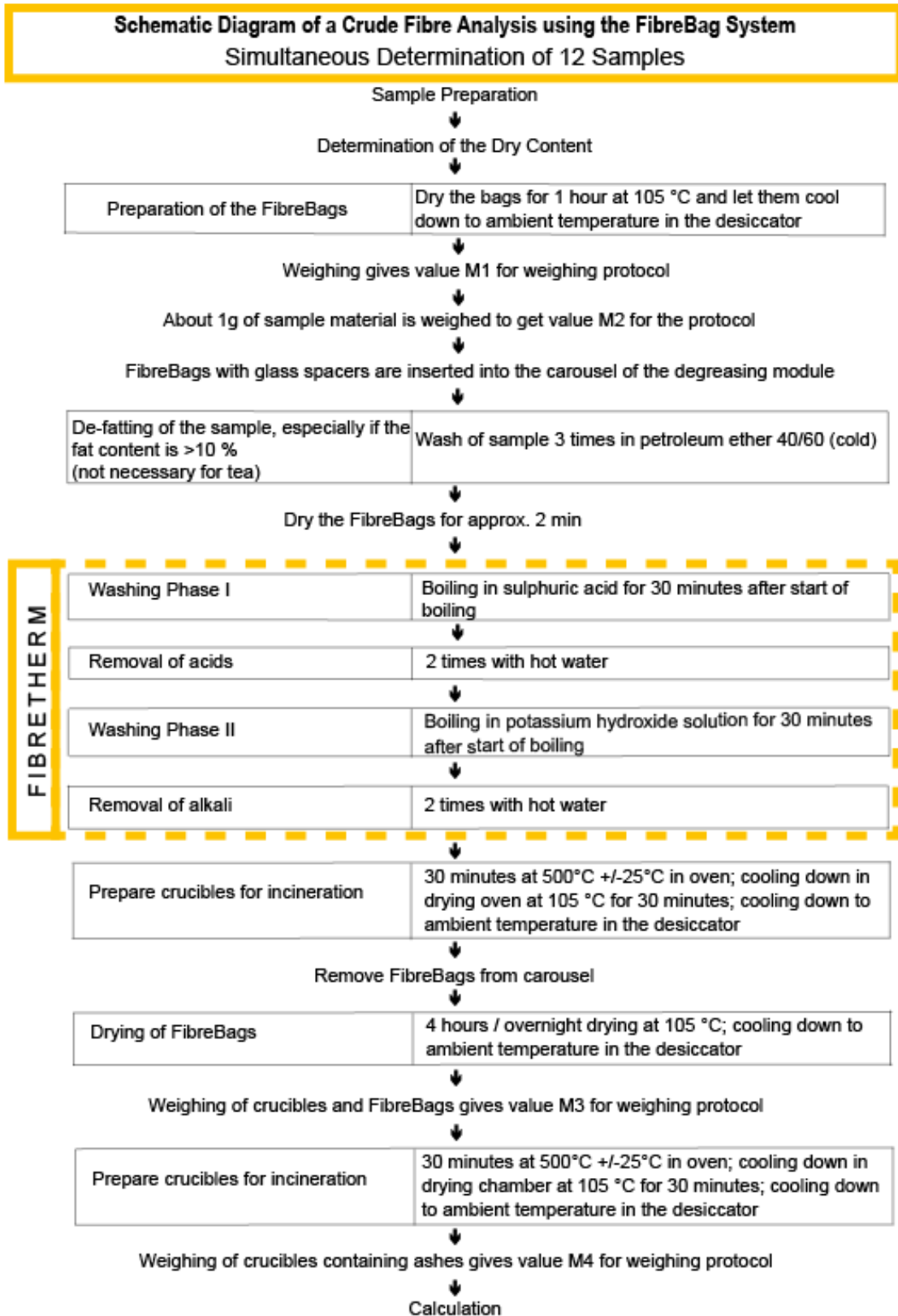
Result too high

Reason	Reaction
Desiccation error	The desiccator might not be filled with a sufficient amount of desiccant or it is wet. Humidity influences sample. Exchange desiccant or regenerate it.
Incomplete desiccation	Check the desiccator and the drying time.

Varying results:

Reason	Reaction
Inhomogeneous sample	Check the sample collection and preparation. No inhomogeneity should be seen during initial weighing.
Poor washing	Ensure that on every wash all bags are completely filled and then are completely emptied so that all solubles are removed.
Poor drying	The samples must always be dried for 4 hours or overnight. Different drying times result in different weights.
Varied analysis conditions or instruments	Changing analysis conditions such as varying characteristics of additives, different apparatus, temperature etc must be avoided. All changes have to be checked thoroughly.









### COMPREHENSIVE APPLICATION DATA BASE

C. Gerhardt offers a wide range of application notes for many methods and procedures. Please contact our application lab team via [application@gerhardt.de](mailto:application@gerhardt.de) for deeper information on:

- Nitrogen in food and feed samples according to Kjeldahl and Dumas
- Crude fibre, ADF and NDF in feed
- Fat in food and feed
- Alcohol determination
- Total cyanide in water
- Trace metal in soil and sludge
- COD determination in water
- Total nitrogen determination in water, soil and plants
- Many more application notes on request.

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