Application HYDROTHERM

B.6.1.HT. Determination of acid-insoluble ash in feedstuff

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1 Introduction

This application is used for the determination of hydrochloric acid-insoluble mineral components in animal feed. HYDROTHERM is used to digest the ash with hydrochloric acid and to filter out the insoluble residue. This filtration is sometimes very time-consuming and can be significantly shortened and automated by using HYDROTHERM.

Figute 1: Feed pellets, Laboratory number.: 8504

2 Principle

The sample is first completely incinerated at 550°C in the muffle furnace in the incineration crucible. This takes approx. 2-4 hours, the ash must be light grey to white and free of carbon particles. The incinerated sample is then weighed. The raw ash content is calculated from this. The ash is digested in the HYDROTHERM with 15% hydrochloric acid at boiling heat and the insoluble residue is filtered out. After drying, the residue is incinerated at 550°C, cooled out and then weighed in. From this, the content of hydrochloric acid-insoluble ash can be calculated.

3 Methods

This application note is meant to be a guideline for the operation of your C. Gerhardt analysis system and has to be adapted to your sample matrix and the local peculiarities in your laboratory.

This document is based on the following official methods:

- Official methods for the analysis of food and feed according to § 64 LFGB (formerly § 35 LMBG) F 0015 (EC) Determination of ash insoluble in hydrochloric acid in animal feed of September 2010.
- VDLUFA Method Book Volume III The chemical analysis of animal feed: Method 8.2. Determination of acid insoluble ash

4 Chemicals and material

Quality p. a.

1. Water: demineralised or distilled
2. Hydrochloric acid HCl 15 %
3. pH indicator paper
4. MN folded filter Ø240mm white belt, ash-free, order number: 203524
5. Incineration crucible

5 Instruments

- Analytical balance (Accuracy 0,1 mg)
- Desiccator, with a desiccation, e.g.: Silica gel orange / Sorbsil C 2.5
- HYDROTHERM hydrolysis system HT 6
- Drying cabinet, electrically heated, with natural ventilation and automatic temperature control
- Electric muffle furnace with thermostat

6 Procedure

6.1 Sample preparation

Approximately 5g of sample is weighed into the previously annealed and tared incineration crucible. The sample is either preincinerated on a heating plate or incinerated directly in the muffle furnace by slow heating up to 550°C and subsequent incineration at 550°C until the ash turns white (2-4 h). Subsequently, the crucibles with the ash are cooled down in the desiccator and then weighed in (calculation of the crude ash).

Weighed in beet pellets

Weighed in beet pellets

Ash of beet pellets

Ash of beet pellets

6.2 Hydrolysis

The ash is placed in the HYDROTHERM hydrolysis beaker. The beaker is inserted into the HYDROTHERM and locked.

HYDROTHERM is taken into operation according to the operating instructions.
A dry folded filter (4.4.) is inserted into the respective position. The apparatus is closed and the programme can be started.
The hydrochloric acid (4.2.) is now added automatically.
The liquid is slowly heated to boiling temperature and kept at low boiling for approx. 15 min. with reduced heating power. When hydrolysis is complete, the digestion mixture is diluted to 1.5 times its original volume with hot water and immediately filtered through a folded filter which has been automatically moisturised by the apparatus. The hydrolysis beaker, condenser and filtration device are rinsed several times with hot water. The filter is washed with hot water until the rinsing water reacts neutrally. HYDROTHERM carries out all these steps automatically.

Recommended method parameters and settings: HYDROTHERM **acid insoluble ash**

Parameter	Setting	Scale unit	Note
Filling levels:			
Amount HCl	100	ml	
Amount H_2O / dilution	50	ml	
Heating / cooling phases:			
Heat-up phase	**12**	min	
Boiling phase I	**15**	min	Power 50 %
Boiling phase II	1	min	Power 70 %
Cooling down phase - duration	**5**	min	
Filter moisturisation:			
Number of moisture cycles	3		
Moisture amount per cycle	30	ml	
Filter phase:			
Filter wait time	5	s	
Rinsing cycles	20		
Pipe opening time	200	ms	
Sample rinse time	10	s	
Sample shower - amount	30	ml	
Condenser shower - amount	30	ml	
Filter shower - amount	50	ml	

At the end of the programme, the filters are placed on a watch glass and dried for approx. 1.5 h at 103 ± 2°C in the drying cabinet.

6.3 Incineration

The dried folded filters are placed in the respective incineration crucibles and incinerated at 550°C. After cooling, the incineration crucibles are weighed and the results calculated.

Acid-insoluble ash of beet pellets

7 Calculation

The acid-insoluble ash content w in g/100 g (equivalent to %) of the sample is calculated according to the following equation:

$$w = \frac{(m_2 - m_1) \times 100}{m_0}$$

m_1: Mass of the empty incineration crucible in g
m_2: Mass of the ashing crucible with the ash in g
m_0: sample weight [g] of the sample

The result is rounded to one decimal place.

8 Sample details

Sample	Laboratory no.	Sample weight g	Ash %	Acid-insoluble ash %
Pellets gemahlen	8504	5,0016	8,89	3,36
Pellets gemahlen	8504	5,0927	8,88	3,40
Pellets gemahlen	8504	5,0014	8,72	3,33
Pellets gemahlen	8504	5,0168	8,71	3,38

To check that the folded filters were free of ash, they were also incinerated when they were dry.

9 Troubleshooting Hydrolysis

Cause	Solution
Sample with hydrochloric acid does not boil properly, sample is not is not hydrolysed properly → inferior results	Increase the heating plate power; ensure that the digestion tube is placed evenly on the heating plate.
No emerging boiling bubbles at the end of the lift-off tube, no homogeneous boiling → fluctuating results	The screw joint of the digestion tube is not properly sealed; screw tight, place the tube flat on the heating plate.
Digestion tube dirty → fluctuating results	Clean digestion tube before hydrolysis, check that digestion tube is clean.

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 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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