# Determination of the Organic Acids in fermenter samples

## Distillation of fermenter samples using Micro Dist and measurement of the organic acids using LCK365 cuvette tests or ion chromatography

Application APP-PHM-0006

EN

**General**

Determination of the organic acids plays an important role with in-process fermenter monitoring in fermentation plants. Fermentation is a four-stage process. Steps two and three produce low-molecular fatty acids, in particular: acetic acid, propanoic acid, and butanoic acid.
If the fermentation process is sound, the values for these compounds, which are collectively referred to as an acetic acid equivalent, are between 500 mg/l and 3000 mg/l. Concentrations of acetic acid equivalent in excess of 3000 mg/l can cause a change in the pH value, which would lead to a serious malfunction of the fermentation plant. The acetic acid equivalent must therefore be checked on a regular basis.

The results of determination of the organic acids using the cuvette test LCK365 and the Nordmann titration method show the total of the individual acids as an acetic acid equivalent. Selective indication of the individual substances originally present is possible using ion chromatography (IC).

Fermenter samples are often a yellowish color by nature (lignin) or are opalescent (turbid) in appearance. When photometric methods are used, this results in inconclusive findings. The fermenter samples must be distilled or prepared in advance by means of dilution and centrifugation before their organic acid content can be determined using the cuvette test LCK365. Distillation in accordance with DIN 38414-19 is costly and time-consuming. This application describes the distillation of fermenter samples using Lachat Micro Dist equipment prior to measurement using the cuvette test LCK365.

**Material**

| | |
|---|---|
| • MDI001 | Micro Dist Thermoblock, complete starter set plus |
| • LCK365 | Cuvette test for organic acids |
| or | |
| • LTV082.99.51002 | Thermostat LT200, 2 black blocks |
| • LZT144 | 8 adapters for 20-mm bores |
| • A17117 | Micro Dist tubes (to be filled by the user) |
| • 17023L | Press |
| • 17013L | Protective gloves, large |
| • 17013S | Protective gloves, small |
| • 17012 | Stands, collection tubes |
| • 17011 | Stands, sample tubes |
| • LCK365 | Cuvette test for organic acids |

**Chemicals:**

Digestion solution: 85% $H_3PO_4$
Collected solution: Distilled water

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## *Determination process*

**Sample preparation**

- The samples are diluted with distilled water using a ratio of **1:10** (top up a 5-g fermenter sample to 50 ml with distilled water) and mixed.
- Course solids are filtered out during this process.

**Micro Dist work process**

**Prior to distillation**

**1.** Switch on the thermostat and preheat to **130°C**.

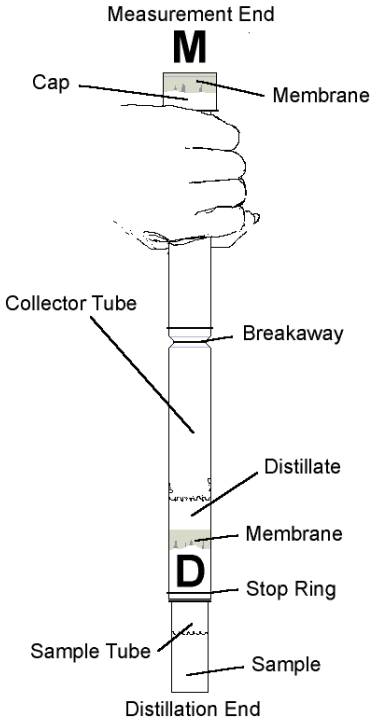



*Figure 1*

**2.** Place a **Micro Dist** (MD) collection tube (Figure 1) in a suitable holder with the **D** end facing downward and add **1,0 ml** of distilled water.

**3.** Seal the **measurement end** (**M** end) of the MD tube using a Teflon membrane and a cap.



*Figure 2*

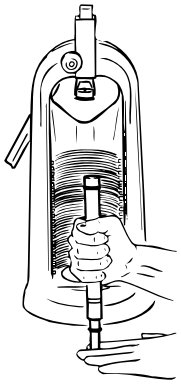


*Figure 3*

**4.** Add a **6,0-ml** sample (dilution) to the sample tube (Figure 2). Add a suitable standard to a tube as a sample and process it simultaneously.

**5.** Add **0,4 ml** of digestion solution to the sample.

**6.** Immediately afterward, insert the sample container into the collection container and seal with the press (Figure 3).

**7.** Insert the MD tube in the pre-heated thermostat (130°C) (warning: wear heat-resistant protective gloves) and distill for **2 hours**.

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**After distillation (2 hours)**

**1.** Remove the MD tube from the thermostat after 2 hours (wear protective gloves) and ***immediately*** separate the sample tube from the collection tube.
**2.** Discard the sample tube and dispose of the content correctly (the content is acid).
**3.** Place the collection tube in a suitable holder (17012) with the **M** end facing downward and allow to cool.

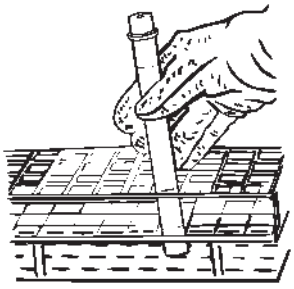


*Figure 4*

**4.** Collect the distillate by tilting and rotating the tube.

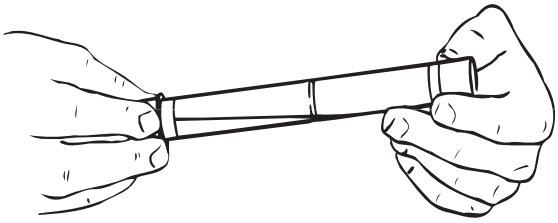


*Figure 5*

**5.** Position the collection tube with the **M** end facing downward and break off the **D** end.

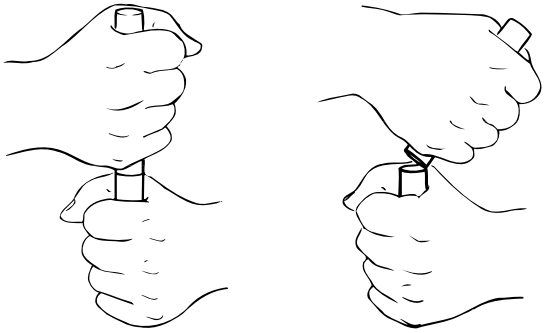


*Figure 6*

**6.** Top up the distillate to **6 ml** with distilled water (if recommended).
**7.** Insert **0,4 ml** of distillate for the measurement using LCK365 (refer to the work instructions for the LCK365 for the work process).
**8.** Determine extraction using the measured standard (approximately 70%). Factor in the extraction rate for subsequent measurements.

**Calculation:**

$$\text{Sample result} = \frac{\text{result read off} \times \text{standard concentration (target)}}{\text{measured standard concentration}}$$

The Micro Dist manual provides further details.

## *Additional measurement options*

The organic acid content of the distillates received can also be checked using ion chromatography or titration.
Please note that the distillates only exhibit approximately 70% of the original organic acid concentration.
We recommend distilling a suitable standard with Micro Dist as a control sample and processing it simultaneously.

If the acids are to be considered separately when using ion chromatography, the results must be calculated as follows:

- Acetic acid: indicated result × 1,6
- Propanoic acid: indicated result × 1,3
- Butanoic acid: indicated result × 1,1

Alternatively, a standard can be introduced into the distillation process and used as a reference point for the ion chromatography.

## *Disposal information*

Waste disposal must be carried out in compliance with regional and national regulations.

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