Determination of the Phenol Index

Distillation of water, sludge, and soil samples using Micro Dist and determination of phenol using LCK345 cuvette tests

Application APP-PHM-0009

EN

General
The application below describes the distillation of samples (water, waste water, soil) containing phenol using the MicroDist system and subsequent measurement of the phenol content using the cuvette test LCK345.

Distillation is suitable for samples containing up to 50 mg of phenol.
The application is an alternative to the method outlined in DIN 38409 H16.

Material

- MDI001 — Micro Dist Thermoblock, complete starter set plus
- LCK345 — Cuvette test for Phenol

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)
- A17070 — MicroDist Accessory Kit incl. Cap press
- LCK345 — Phenol cuvette test

Chemicals:
pH adjustment: 1 M NaOH
10% H_2SO_4

Disposal information

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

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Determination process

General

- 6-ml liquid sample or a solid sample of between 0,5 g and 1,0 g (sludge, soil) is added to the sample tubes. For solid samples, the tubes should contain 5 mL of distilled water. The sample may contain up to 50 mg of phenol.
- In order to complete the determination process using cuvette test LCK345, samples with a high content must be diluted after distillation to bring them into the measurement range for the cuvette test (max 5 mg/l).

Sample preparation

Adjust the sample pH to approximately pH 4 with 1 M NaOH or 10% H_2SO_4.

Micro Dist work process

Prior to distillation

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

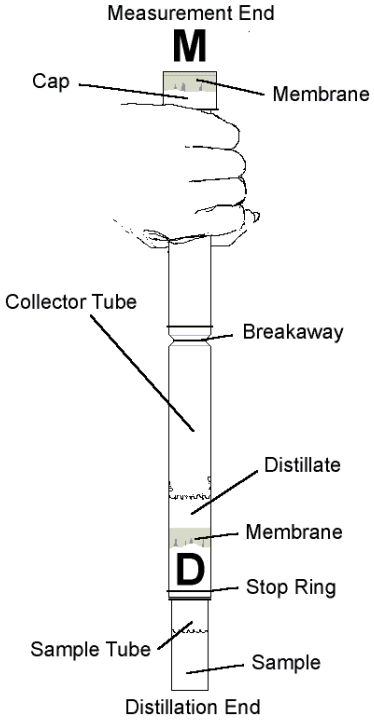

Figure 1

2. Place a **Micro Dist** (MD) collector tube (Figure 1) in a suitable holder with the **D** end facing downward.

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

Figure 2

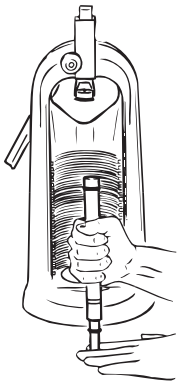

Figure 3

4. Add a **6,0-ml** sample/**0,5 – 1,0-g** soil sample plus **5 ml** of distilled water to the sample tube (Figure 2).
Add a suitable standard to a tube as a sample and process it simultaneously.

5. Immediately afterward, insert the sample container into the collector container and seal with the press (Figure 3).

6. Insert the MD tube in the pre-heated thermostat (130°C) (warning: wear heat-resistant protective gloves) and distill for **90 minutes**.

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After distillation (90 minutes)

1. Remove the MD tube from the thermostat after 90 minutes (wear protective gloves) and ***immediately*** separate the sample tube from the collector tube.
2. Discard the sample tube and dispose of the content correctly (the content is acid).
3. Place the collector tube in a suitable holder with the **M** end facing downward and allow to cool (10 minutes).

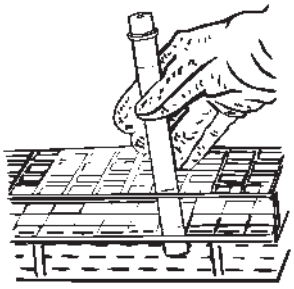

Figure 4

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

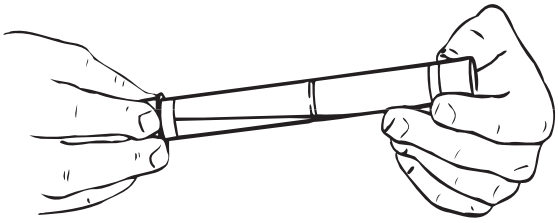

Figure 5

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

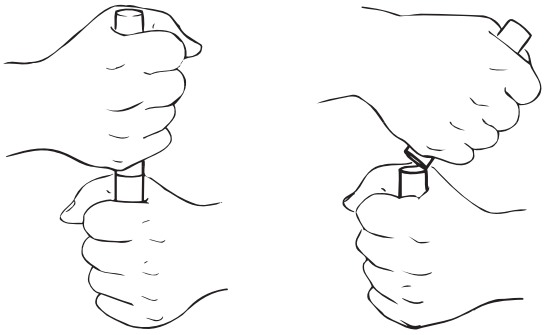

Figure 6

6. Top up the distillate to **6 ml** with distilled water (if necessary).
7. Insert the distillate for measurement using LCK345 (refer to the work instructions for the LCK345 for the work process).
8. Determine extraction using the measured standard (should be > 80%). Factor in the extraction rate for subsequent measurements.

Calculation:

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

The measurement results must be subjected to plausibility checks (dilute and/or spike the sample).

The Micro Dist manual provides further details.

Trouble shooting – Membrane caking

When solid samples or sludges are distilled, foam comes up through the membrane, or scum cakes over the underside of the membrane causing it to be pushed up. This occurs when the sample has a lot of organics in it such as grease or oils. The scum or foam is organic surfactants which wet the hydrophobic membrane. This causes it to lose its hydrophobicity and thus not function properly. The placement of the membranes on all collector tubes is elevated such that the matrix foam normally will not come into contact with the membrane. Be careful of scummy organic material caking the membrane or actually oozing through the membrane as this causes pressure to build up in the sample tube. The pressure is not large but it is sufficient to cause spattering of the hot sample when the sample tube is removed. In some cases the distillation membrane may pop out of the ring.

Running Solid Samples with Micro Dist

The Micro Dist is capable of handling many different kinds of solid samples from sands to sludges. As a general guideline, if the sample is high in organic content use only 0,5 g or less sample. If the sample is low in organic content, one can use up to 1 g of sample. Experiment with samples to determine the best weight of sample to add for each matrix type. The sample will be diluted with DI water (5 to 6 ml) per the MicroDist manual.

Calculating the amount of sample in mg/kg after analysis:
Multiply the determined concentration in mg/l by the volume in the tube (in ml, normally 6 ml) and divide then with 1000 (conversion l/ml) to get the amount of analyte in mg.
Divide the content of analyte in mg by the weight of original sample (in g) and multiply then with 1000 (conversion g/kg) to get the result as mg analyte / kg sample.

If foaming or caking of the membrane continues to be a problem even with reduced sample weights of 0,5 g, try the following:

- Add activated charcoal so it covers the surface of the solid and then fill the remaining void space with glass wool. When trying this procedure it would be recommended that 4-5 ml of water be used versus 6 ml.
- For soil or organic samples containing cyanide, Biobeads™, manufactured by BIO-RAD, part number SM-2, have proven effective in laboratories.
- Test a known standard with one of these procedures and a spiked sample of the foaming or caking matrix to conclude whether these solutions will work.

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