

Determination of readily liberated sulfide

Distillation of water, sludge and soil samples with MicroDist and determination of sulfide with LCK 653 cuvette test

APP-PHM-0012 application

EN

General

The presence of hydrogen sulfide (H₂S) in the wastewater leads to problems in the wastewater treatment plant with occupational safety, purification problems (filamentous bacteria), and unpleasant odors, as well as through the formation of sulfuric acid, which corrodes building and raw materials.

The sulfides either come from inorganic compounds from industry (lubricants, pharmaceutical products) or are created through the development of bacterial sulfides from organic sulfur compounds (meat industry) or sulfur oxides (households, industry).

The wastewater threshold value for sulfide is 2 mg/L H₂S.

This application describes the distillation of samples which contain sulfide and their subsequent determination with the LCK653 sulfide cuvette test. It is an alternative to the standard DIN 38405 – D27: „Determination of readily liberated sulfide“.

Material

MDI001	MicroDist Thermoblock, complete starter set plus Cuvette Test for sulfide
LCK653	
or	
LTV082.99.51002	Thermostat LT 200, 2 black blocks
LZT144	8 Adapter for 20-mm bores
A17117	MicroDist Tubes (to be filled by the user)
A17070	MicroDist starter set
LCK653	Cuvette Test for sulfide

Chemicals

Releasing solution:
9 M H₂SO₄

Preparation:

1. First, pour **150 mL** distilled water into a 500-mL beaker.
2. Next, add **276 g** concentrated sulfuric acid (H₂SO₄) **slowly** in small portions.
Caution: solution becomes hot! Only add the sulfuric acid in small portions. Work in a fume hood throughout this task!
3. After it has cooled to room temperature, decant solution into a storage bottle.

Trapping solution:
1 M NaOH

DOC042.52.00832

Determination process

General:

Caution: Perform these steps in a fume hood, as H_2S can volatilize.

Either a 6 mL liquid sample (up to 10 mg/L acid-soluble sulfide) or a 0.5–1.0 g solid sample (sludge or soil) is added to the sample tubes. For solid samples, the tubes should contain 5 mL of distilled water additionally.

In order to complete the determination process with the LCK653 cuvette test, samples with a high content (up to 10 mg/L) must be diluted after distillation to bring them into the measurement range for the cuvette test (max 2.0 mg/L).

MicroDist work process

Prior to distillation

1. Switch on the thermostat and preheat to 120°C.

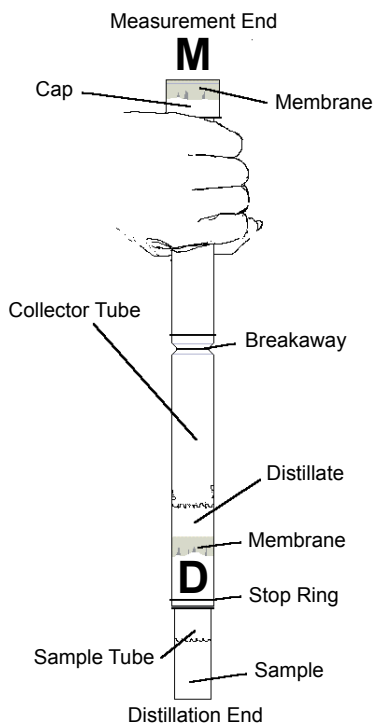


Figure 1

2. Place a MicroDist (MD) collector tube (Figure 1) in a suitable holder with the D end pointed downward and pour in 1.5 mL trapping solution (1 M NaOH).

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



Figure 2

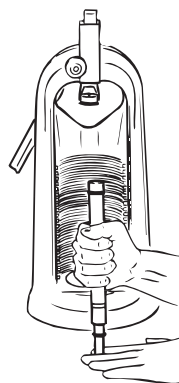


Figure 3

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 0.45 mL releasing solution (9 M H_2SO_4) to the sample.

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

6. Insert the MD tube in the pre-heated thermostats (120 °C) (caution: wear heat-resistant protective gloves) and distill for 40 minutes.

After distillation (40 minutes)

7. Remove the MD tube from the thermostat after 40 minutes (wear protective gloves) and immediately separate the sample tube from the collector tube.

8. Discard the sample tube and dispose of the content correctly (the solution is acid!).

9. Place the collector tube in a suitable holder with the M end facing downward and allow to cool (10 minutes).

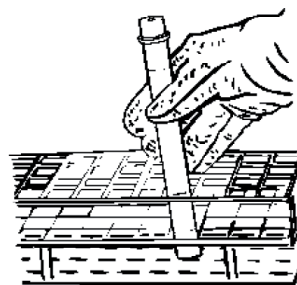


Figure 4

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

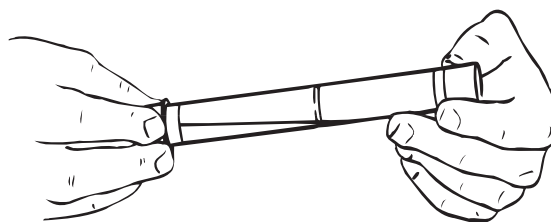


Figure 5

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

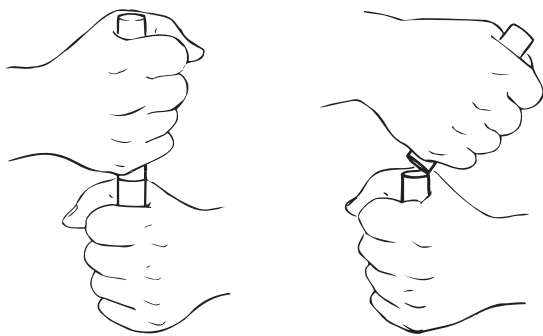


Figure 6

- 12.** Top up the distillate to **6 mL** with distilled water.
13. Insert the distillate for measurement with LCK653 (refer to the working procedure for the LCK653 for the work process).
14. The test results must be assessed with a plausibility check (dilution and/or concentration).

Refer to the MicroDist manual for details.

Troubleshooting – membrane caking

If solid samples or sludges are distilled, foam may come up through the membrane, or scum may cake and block the underside of the membrane and cause it to be pushed up. This occurs when the sample has a lot of organic substances in it such as grease or oils. The scum or foam is an organic surfactant that wets the hydrophobic membrane from underneath. This causes the membrane to lose its hydrophobicity and therefore not function properly.

The placement of the membrane in the collector tube is slightly raised so that the foam does not normally come into contact with the membrane.

Be careful of scummy organic material that can wet the membrane or ooze through the membrane, as this causes pressure to build up in the sample tube. The pressure is not very high, but it is sufficient to cause spattering of the hot sample when the sample tube is separated from the collection tube. In a small number of cases, the distillation membrane may completely come away from the ring.

Distillation of soil samples with MicroDist

Micro Dist can be used to process a wide range of soil samples, from sands to sludges. As a general guideline, if the sample is high in organic content, make sure that only 0.5 g or less of the sample is weighed out. If the sample has a lower organic content, up to 1.0 g of sample can be used.

The ideal sample weight depends on the sample type and must be optimized on the spot if necessary. The soil samples are topped up with distilled water (5 to 6 mL).

Calculating the sample content in mg/kg after analysis:

Multiply the determined concentration in mg/L by the volume in the tube (in mL, normally 6 mL) then divide by 1000 (L/mL conversion), to obtain the amount of analyte in mg.

Divide the content of analyte in mg by the weight of original sample (in g) then multiply by 1000 (g/kg conversion) to obtain the result as mg analyte/kg sample.

If the membrane continues to foam or cake and remains a problem even with reduced sample weights of < 0.5 g, try the following:

- Add activated charcoal to the sample tube so that it covers the surface of the sample. Fill the remaining empty spaces with glass wool. With this method, it is only necessary to add 4 to 5 mL of water (rather than 6 mL).
- For soil or organic samples containing cyanide, Biobeads™, manufactured by BIO-RAD (item number SM-2), have proven effective.
- Test a known standard and a spiked sample with one of these methods to make sure that the method will work.