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Micro Dist™ Operation and Applications

USER MANUAL

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Section 1 Specifications

Specifications are subject to change without notice.

Installation Environment	
Bench Space Needed	1.5 m (4 ft)
Total Height, tubes and block	33 cm (13 inches)
Ambient Operating Temperature Range	5–40 °C
Altitude	Up to 2 Km (6800 ft) above sea level
Relative Humidity	Up to 92.5%, non-condensing
Shelf Weight	20 kg (45 lb)
Ventilation Needed	Ordinary air-conditioned lab. Provide at least 6 inches distance between the Micro Dist block and all other vertical surfaces.
Safety	Laboratory 61010-1, 61010-2-010 (UL, CSA, EN Safety Standards)
EN61010 Pollution Degree	II
EN61010 Installation Category	II
Micro Dist Heating Block	
AC Input Frequency	100–115 V, 50/60 Hz 230 V, 50/60 Hz
Maximum Active Current (A) Rating	100 V, 9 A 115 V, 11 A 230 V, 5 A
Fuse Rating	100–115 VAC Model: 15 A slow-blow, 125 VAC 230 VAC Model: 6.3 A slow-blow, 250 VAC
Block Operating Temperature Range	120 °C typical; 30–135 °C, L-type thermocouple, PID variable cycle controller
Controller Setpoint Error	<1% of setpoint temperature
Controller Precision	0.5 °C
Hole-to-Hole Temperature Variation	<0.5 °C
Sample Capacity	21 Micro Dist™ Tubes
Micro Dist Tubes	
Sample Size	6.0 mL capacity
Tube Material	Analytical-grade pure polypropylene
Compliance	
Certifications	100–115 V model: approved to UL and CSA safety standards (cETLus mark); 230 V model: CE certified by Hach

Section 2 General Information

2.1 Safety Information

Please read this entire manual before unpacking, setting up, or operating this equipment. Pay attention to all danger and caution statements. Failure to do so could result in serious injury to the operator or damage to the equipment.

To ensure that the protection provided by this equipment is not impaired, do not use or install this equipment in any manner other than that specified in this manual.

2.1.1 Use of Hazard Information

DANGER
Indicates a potentially or imminently hazardous situation which, if not avoided, will result in death or serious injury.

DANGER
Indique une situation de danger potentiel ou imminent qui, si elle n'est pas évitée, peut entraîner la mort ou des blessures graves.

WARNING
Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

AVERTISSEMENT
Indique une situation potentiellement ou immédiatement dangereuse qui, si elle n'est pas évitée, peut entraîner des blessures graves, voire mortelles.

CAUTION
Indicates a potentially hazardous situation that may result in minor or moderate injury.

ATTENTION
Indique une situation potentiellement dangereuse qui peut entraîner des blessures mineures ou modérées.

Notice: *Indicates a situation that is not related to personal injury.*

Remarque *: Indique une situation qui n'est pas liée à des blessures aux personnes.*

Important Note: *Indicates a situation which, if not avoided, may cause damage to the instrument. Information that requires special emphasis.*

Remarque importante *: Indique une situation qui, si elle n'est pas évitée, peut endommager l'instrument. Informations nécessitant une mise en avant particulière.Information that supplements points in the main text.*

Note: *Information that supplements points in the main text.*

Remarque *: Information complétant certains points dans le texte principal.*

2.1.2 Precautionary Labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed.

	This is the safety alert symbol. Obey all safety messages that follow this symbol to avoid potential injury. If on the instrument, refer to the instruction manual for operation or safety information. Ceci est le symbole d'alerte de sécurité. Se conformer à tous les messages de sécurité qui suivent ce symbole afin d'éviter des blessures potentielles. Si apposés sur l'instrument, se référer au manuel d'utilisation pour le fonctionnement ou les informations de sécurité.
	Electrical equipment marked with this symbol may not be disposed of in European public disposal systems after 12 August of 2005. In conformity with European local and national regulations (EU Directive 2002/96/EC), European electrical equipment users must now return old or end-of life equipment to the Producer for disposal at no charge to the user. ***Note:*** *For return for recycling, please contact the equipment producer or supplier for instructions on how to return end-of-life equipment, producer-supplied electrical accessories, and all auxiliary items for proper disposal.* L'équipement électrique portant ce symbole ne peut être mis au rebut dans les systèmes de mise au rebut publics européens à compter du 12 août 2005. Conformément aux règlements nationaux et européens (Directive 2002/96/EC), les utilisateurs européens d'appareils électriques obsolètes ou hors d'usage doivent désormais les renvoyer au fabricant, qui se chargera de les mettre au rebut à ses frais. ***Remarque*** *: Pour le retour à des fins de recyclage, veuillez contactez le fabricant ou le fournisseur d'équipement pour obtenir les instructions sur la façon de renvoyer l'équipement usé, les accessoires électriques fournis par le fabricant, et tous les articles auxiliaires pour mise au rebut appropriée.*
	This symbol identifies a risk of chemical harm and indicates that only individuals qualified and trained to work with chemicals should handle chemicals or perform maintenance on chemical delivery systems associated with the equipment. Ce symbole identifie un risque chimique et indique que seules les personnes qualifiées et formées pour travailler avec des produits chimiques sont autorisées à les manipuler ou à réaliser des opérations de maintenance sur les systèmes associés à l'équipement et utilisant des produits chimiques.
	This symbol identifies the presence of a strong corrosive or other hazardous substance and a risk of chemical harm. Only individuals qualified and trained to work with chemicals should handle chemicals or perform maintenance on chemical delivery systems associated with the equipment. Ce symbole identifie la présence d'une substance fortement corrosive ou autre substance dangereuse et donc, un risque de blessure chimique. Seuls les individus qualifiés et formés pour travailler avec des produits chimiques doivent manipuler des produits chimiques ou procéder à des travaux de maintenance sur les systèmes de distribution chimique associés à l'équipement.
	This symbol indicates that a risk of electrical shock and/or electrocution exists. Ce symbole indique qu'il existe un risque de choc électrique et/ou d'électrocution.
	This symbol indicates a hot surface and the risk of a burn. Ce symbole indique une surface à haute température et un risque de brûlure.
	This symbol, when noted on the product, identifies the location of a fuse or current limiting device. Lorsque ce symbole se trouve sur un produit, il indique l'emplacement d'un fusible ou d'une protection contre les surcharges.

2.2 Principle of Operation

The purpose of any analytical distillation is sample clean-up. In a sample distillation, the idea is to remove the analyte from a possibly interfering or otherwise undesirable original sample matrix and to place it into a clean, reproducible distillate solution. During some distillations, such as the method Cyanide–1, a digestion may take place as well.

The Micro Dist™ system was developed for the analytical distillation of samples which are largely aqueous. In such samples an *in situ* steam distillation can be done simply by boiling the

sample. The analyte must have some vapor pressure at the boiling point of water (100 °C). When an aqueous sample is boiled, the steam and analyte distill over together. When the steam and analyte condense, a clean aqueous distillate solution is collected which can be determined free of matrix effects or interferences present in the original sample matrix.

For at least the last 200 years, analytical distillations have been performed in large-scale distillation glassware which was designed more for preparative distillations, particularly of organic compounds and solvents. The large volume and numerous joints in this type of apparatus made it difficult to recover very small amounts of analyte. Because of this, large volumes of sample were necessary to distill and transfer a sufficiently precise amount of analyte into the receiving flask.

The Micro Dist system strips analytical distillation down to its bare essentials. The sample is pipetted into a sample tube and sealed to a collector tube. This assembly is placed into a special heating block which fits it very closely, allowing the poor-conducting polypropylene to be heated rapidly yet locally. As the sample boils, its vapors rise within the tube and pass through a hydrophobic, porous membrane. The vapors condense above the membrane to form droplets, which pool over the membrane, but cannot pass back through it. The short vertical path of distillation is possible because of the physical separation of distilling sample and distillate by the hydrophobic membrane. As little as 10 ng of analyte can be precisely distilled.

The heating block temperature is set well above the boiling point of water. This keeps the headspace above the boiling sample sufficiently hot to prevent the condensation of the distillate below the lower membrane. The portion of the Micro Dist collector tube above the block cools off rapidly because of the very slow thermal conductivity of polypropylene. This allows the tube to be efficiently air-cooled as there is little heat transferred to the cool portion of the collector tube.

When the distillation is complete, the hot assembly is pulled from the heating block. Because the cooling of the air in the sample tube will soon cause a vacuum which will aspirate back the distillate through the membrane, the sample tube is pulled off immediately. This is easy to do when the plastic tube is hot. The collector tube is set aside to cool for about 10 minutes.

The distillate and droplets are then easily coaxed from the non-wettable walls to the bottom of the collector tube. The top is then broken off and the distillate sample is diluted to the original volume with deionized water. After capping and mixing, the distillate sample is ready to be determined by a suitable method, automated or manual.

2.3 Distilling Calibration Standards with the Micro Dist System

Analytically speaking, calibration standards should be distilled in the same manner as samples. Such sample preparation steps are a vital and inseparable part of the whole analytical method used to determine the analyte in the original sample. Examples of such sample preparations are a Kjeldahl digestion, an extraction of a

sample, and a distillation such as those enabled by the Micro Dist system.

When large-scale glassware is used for such distillations, one generally does not distill a set of calibration standards because this reduces the already limited sample throughput of the distillation step. At best, one occasionally distills a check standard. This is not an ideal method of calibration.

With the Micro Dist system, the manufacturer recommends distilling a set of standards at least once a day, or once every batch of samples. The Micro Distilled standards should then be used as the calibration standards for the method used to determine the samples.

This investment in data quality will give the best possible data as both standards and samples will be carried all the way through the distillation. For example, doing this will correct for any distillation reagent blank.

2.4 Trapping Solutions in the Collector Tube

When working with pre-filled tubes, the collector tube comes with a trapping solution suitable to the Micro Dist Method. Some tubes such as the phenolics method tube do not contain trapping solution. Be sure to use the Micro Dist tubes which match the Micro Dist Method. The Micro Dist Method will define the trapping solution; see Table 3 on page 67.

When using the User-Fill option, there will be no trapping solution in the collector tube; it is added as part of the method.

2.5 Shelf Life of the Pre-filled 21 tube Kits

Open the tube kit package and write the date next to “Opened on:”. Use all 21 tubes within two weeks, if possible. If all 21 tubes are not used, re-seal the tube kit package with tape to prevent the trapping solution from evaporating from the collector tubes. Unopened packages of tubes are best when used within 24 months.

For the User-Fill option, there will be no trapping solution in the collector tube and, therefore, no need to keep track of shelf life as there is no trapping solution to evaporate. User-Filled tubes should be used the same day as filling.

DANGER
Burn hazard. Only qualified personnel should conduct the tasks described in this section.

DANGER
Risque de brûlure. Les opérations décrites dans cette section ne doivent être exécutées que par un personnel qualifié.

3.1 Unpacking the Instrument

Keep the original shipping cartons. Returns for service must be transported in the original packaging.

3.1.1 Micro Dist Setup

CAUTION
Personal injury hazard. The Micro Dist is heavy. To prevent possible lifting related injuries, do not attempt to carry or move the product without assistance from a second person.

ATTENTION
Risque de blessure personnelle. Le Micro Dist est lourd. Pour empêcher les blessures potentielles que sa manutention pourrait occasionner, n'essayez pas de porter ou de déplacer le produit sans l'aide d'une deuxième personne.

- To move the Micro Dist, position a person on each side of the instrument. With both hands under the side, lift the analyzer simultaneously.
- Place the instrument on a flat surface that it is not exposed to direct sunlight.
- To avoid overheating or damage to the surrounding area during operation, allow six inches of clearance around the Micro Dist.

3.1.2 Connecting the Power Supply

DANGER
Electric shock hazard. A protective earth ground is required for safe operation of this equipment. Always use a power cord and plug that has a protective earth ground terminal connection.

DANGER
Risque de décharge électrique. Une prise de terre protectrice est requise pour pouvoir utiliser cet équipement sans danger. Utilisez toujours un câble d'alimentation et une prise ayant des fiches pouvant être branchées sur une prise de terre.

DANGER
Electric shock and fire hazard. Secure the power cord position to prevent contact with the heater block.

DANGER
Risque de décharge électrique et danger d'incendie. Placez le câble d'alimentation de telle manière qu'il ne puisse pas entrer en contact avec le bloc de chauffage.

Note: *For wet location installation, use a ground fault interrupt circuit (GFIC).*

Section 4 Operation

4.1 Setting the Temperature

4.1.1 Heater Block

CAUTION
Burn hazard. The Micro Dist heater block top surface is a burn hazard at operating temperature. Wear heat-resistant gloves when putting tubes in or taking them out of the heating block.

If a burn occurs, immediately put the burned area under cold running water for five minutes. Seek appropriate medical attention if the area is blistered or white.

ATTENTION
Risque de brûlure. La surface supérieure du Micro Dist présente un risque de brûlure à la température d'utilisation. Veuillez porter des gants résistants à la chaleur lorsque vous mettrez des tubes à l'intérieur ou quand vous les enlèverez du bloc de chauffage.

En cas de brûlures, passer immédiatement les zones de peau brûlées sous l'eau froide pendant cinq minutes. Si la zone touchée est couverte d'ampoules ou présente une coloration blanche, demander l'aide d'un médecin.

If a large amount of liquid is spilled on the heating block, unplug it immediately. Do not turn off at the power switch. The Micro Dist enclosure and block are grounded, but it is best to allow the spill to dry before using the block again. Rinse the holes with water and drain through the drain-hole at the bottom.

To set the temperature, locate the heater control on the top right. See Table 1.

Figure 1 Heater Control

Table 1 Heater Control Keys

	**ADVANCE**
	**UP** and **DOWN**
	**INFINITY**

Note: *The lower display indicates the temperature set point.*

1. Press the **UP** and **DOWN** keys to increase or decrease the set point. Once the temperature has been set, it will be saved automatically after three seconds. The display will show the current temperature in °C.
2. Press **INFINITY** once to clear alarms. The display will show the current temperature in °C. The red light will be lit when the heater is ON.

Note: *The maximum temperature set point of this equipment is 135 °C.*

4.2 Using Micro Dist™ Tubes

DANGER
Fire hazard. Do not use the Micro Dist with flammable substances.

DANGER
Risque d'incendie. Ne pas utiliser le Micro Dist avec des substances inflammables.

CAUTION
Explosion and burn hazards. To reduce the risk of pressure build-up and potential explosion, do not tape over or clog the pinhole in the tube caps.

ATTENTION
Risques d'explosion et de brûlure. Pour réduire le risque d'accumulation de la pression et une explosion potentielle, ne recouvrez pas le trou d'aiguille des capuchons des tubes et ne les bouchez pas.

CAUTION
Burn hazard. When inserting and pulling the tubes in and out of the heating block, wear safety glasses and the supplied heat-resistant gloves. Be careful when removing the hot sample tubes from the collector tube. The solution in the sample tube will be boiling or near boiling temperature.

ATTENTION
Risque de brûlure. Lorsque vous placez des tubes à l'intérieur du bloc de chauffage ou lorsque vous les enlevez, veuillez porter des lunettes de sécurité et les gants résistants à la chaleur livrés avec l'appareil. Faites attention lorsque vous enlèverez les tubes d'échantillon chauds du tube collecteur. La

solution dans le tube d'échantillon sera bouillante ou sur le point de bouillir.

The tubes are not closed systems and cannot explode. The pressure inside the tubes is very close to that of the atmosphere at all times.

Note: *Do not refrigerate Micro Dist tubes. This will cause the membrane ring to shrink and the trapping solution to leak out.*

4.2.1 Filling and Assembling the User-Fill Collector Tubes

The User-Fill collector tube option is designed for the user who:

- wishes to fill the collector tube just before use, thus avoiding any unwanted evaporation after the foil pack is opened.
- wishes to reduce shipping costs by receiving a dry product.
- wants to design a custom trapping solution.
- wants to keep tubes in stock indefinitely.

Lachat Instruments takes great care in filling the pre-filled collector tubes accurately and precisely. The tube's performance depends greatly on adding the correct, precise aliquot of trapping solution.

Trapping solution recipes for each of the Micro Dist™ tubes available from Lachat Instruments can be found in Table 3 on page 67.

When measuring the trapping solution aliquot by volume, the manufacturer recommends that a good quality re-pipettor be used to dispense the trapping solutions into the collector tubes. A hand-held automatic pipet can also be used, but this will be less convenient. Always calibrate any automatic pipet before using it by dispensing 1.0 mL water onto a top-loading balance. 1.0 mL water should weigh 1.0 g.

4.2.2 Filling and Assembling the Collector Tube

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See on page 17.

1. Place as many collector tubes as will be used in one day in a rack, with the **D** end down and the **M** end up. For very low-concentration work, such as ammonia, rinse each tube with deionized water first.
2. By Volume: With the re-pipettor or automatic pipet, dispense the volume of trapping solution specified in Table 3 on page 67, into the **M** end of the collector tube.
3. By Weight: Place the rack of collector tubes onto a 0.01 g top-loading balance. Zero or tare the balance. With a disposable pipet or wash bottle, dispense the weight of trapping solution specified in Table 3 on page 67, into the **M** end of the collector tube.
4. Repeat step 2 or 3 for each collector tube in the rack.
5. Pick up a filled collector tube and set the **D** end down on the bench.

6. Rest one of the supplied 30 mm User Fill Option membranes (Cat. No. 17031) on the **M** end. Center the membrane as much as possible on the collector tube's **M** end.
7. Take one of the supplied caps and push it over the membrane resting on the **M** end of the collector tube until it cannot be pushed any further onto the collector tube.
8. The 'skirt' around the sides of the mounted membrane may be wrinkled and somewhat uneven; this is OK. However, the mounted membrane should not be torn, especially at its upper edge. The skirt should not rise to within less than 3 mm from the end of the tube. If it is torn or very unevenly mounted, remove the cap and membrane and repeat from step 6, discarding the old membrane. There is one extra membrane supplied for every 10 tubes in case this happens.
9. The collector tube is now ready to use with Micro Dist methods.

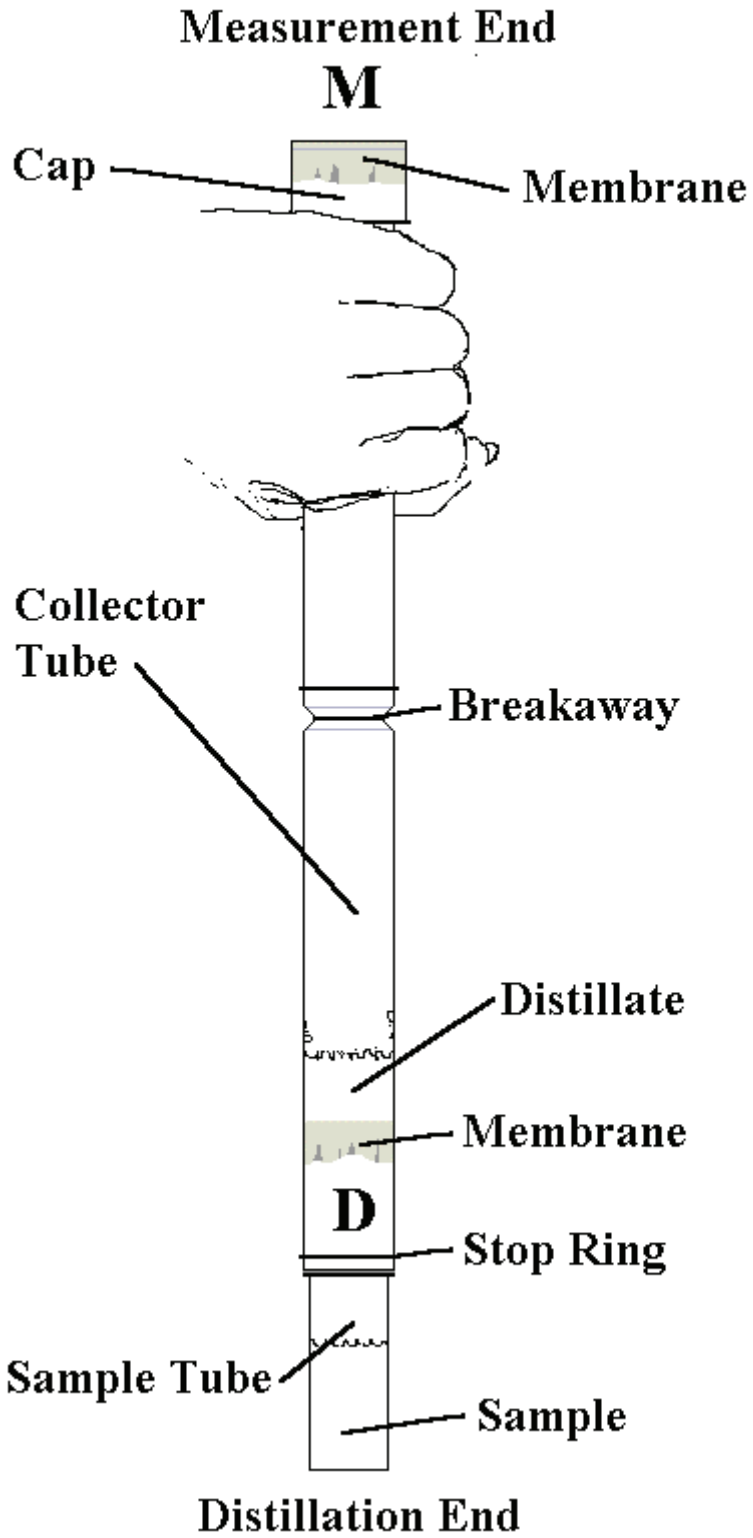

Figure 2 Collector Tube

DANGER
Fire and electric shock hazard. Secure the power cord position to prevent contact with the heater block.

DANGER
Risque d'incendie et de décharge électrique. Placez le câble d'alimentation de telle manière qu'il ne puisse pas entrer en contact avec le bloc de chauffage.

DANGER
Chemical hazard. To avoid potential exposure to toxic gas, the manufacturer recommends operating the Micro Dist under a fume hood.

DANGER
Risque chimique. Pour éviter le contact potentiel avec des gaz toxiques, le fabricant recommande d'utiliser le Micro Dist sous une hotte aspirante.

CAUTION
Personal injury hazard. Always use two people to lift or move the instrument.

ATTENTION
Risque de blessure personnelle. L'instrument doit toujours être soulevé ou déplacé par deux personnes.

1. Place the Micro Dist on a level surface, preferably under a fume hood. Leave at least six inches of clearance on all sides of the instrument to allow for ventilation and heat dissipation.
2. Connect the Micro Dist to power. Switch the instrument on.
3. Set the temperature on the heater controller (section 4.1 on page 13).
4. While the heater block is coming to operating temperature for the selected test, prepare the sample tubes (section 4.2 on page 14).
5. When the Micro Dist heater block reaches operating temperature, use protective gloves to insert the tubes into the block.

DANGER
Fire, burn, electric shock and chemical hazards. Only qualified personnel should conduct the maintenance tasks described in this section of the manual.

DANGER
Risques d'incendie, de brûlure, de décharge électrique, et risque chimique. Seul le personnel qualifié doit effectuer les tâches de maintenance détaillées dans cette section du manuel.

DANGER
Fire and electric shock hazard. Secure the power cord position to prevent contact with the heater block.

DANGER
Risque d'incendie et de décharge électrique. Placez le câble d'alimentation de telle manière qu'il ne puisse pas entrer en contact avec le bloc de chauffage.

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques.

6.1 Cleaning the Micro Dist

CAUTION
Burn hazard. Before attempting any cleaning operation, allow sufficient time for the heater to cool to a temperature that is safe to touch without risk of burn. A temperature of less than 50 °C (122 °F) for metal parts is generally considered safe to touch without risk of burn.

ATTENTION
Risque de brûlure. Avant d'entreprendre toute opération de nettoyage, laissez au bloc de chauffage assez de temps pour se refroidir à une température qui ne présente pas de danger de brûlure au toucher. Une température de moins de 50 °C (122 °F) pour les parties métalliques est généralement considérée comme sans danger de brûlure au toucher.

Clean the Micro Dist exterior by wiping down with water.

Note: *Before using any cleaning or decontamination method except those recommended by the manufacturer, contact the manufacturer to confirm that the proposed method will not damage the equipment.*

In the event of a spill in the heater block, immediately disconnect the equipment from the power source and allow the equipment to cool. When sufficiently cool, carefully flush the affected block test tube holes with water. If necessary, use a test tube brush to clean the block test tube holes.

6.2 Calibrating the Automatic Re-pipettor

1. Use the supplied instruction sheet to assemble the re-pipettor.
2. Fill the bottle with deionized water.
3. Prime the re-pipettor several times, then place next to a top-loading balance. Put a weighing boat on the balance pan and tare the balance.
4. Set the re-pipettor to 0.75 mL using the lock screw.
5. Dispense water into the weighing boat.
6. The weight in grams is the volume of water dispensed in mL. If the weight is less than 0.75 g, set the screw up higher and try again. If the weight is more than 0.75 g, set the screw down lower and try again.
7. Repeat until the weight is 0.75 g for three consecutive measurements. Check the calibration of the re-pipettor at least monthly.

6.3  Fuse Replacement

DANGER
Fire hazard. Always replace fuses with fuses of the same type and rating.

DANGER
Risque d'incendie. Remplacez toujours les fusibles avec des fusibles du même type et du même calibre.

Important Note: *100–115 V models: Although there are positions for 2 fuses in the 100–115 V Micro Dist model, only the upper fuse in the fuse drawer is connected in the circuit. A spare fuse may be present in the lower slot, but it is not required.*

Note: *Fuse failure is generally an indication of a problem with the equipment. If fuses continue to fail, contact the service center for return authorization and repair.*

1. Switch the Micro Dist heater power switch to the off position (O). Disconnect the power cord from the appliance inlet.
2. Locate the fuse drawer. Pull the fuse drawer from the appliance inlet module.
3. Remove the fuse(s) from the fuse drawer and install the new fuse(s) of the same type and rating. Use only T, 15A, 125 V (for 100–115 V operation) or T, 6.3A, 250V (for 230 V operation) fuses.
4. Replace the fuse drawer and push it all the way in. Reconnect to power.

6.4 Parts

Other than the fuses, there are no user-replaceable or user-serviceable parts. Contact the factory for repair/service information.

Section 7 General Procedure

7.1 General Procedure

DANGER
Chemical hazard. To avoid potential exposure to toxic gas, the manufacturer recommends operating the Micro Dist under a fume hood.

DANGER
Danger chimique. Pour éviter le contact potentiel avec des gaz toxiques, le fabricant recommande d'utiliser le Micro Dist sous une hotte aspirante.

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Review all material safety data sheets (MSDS) for safety information specific to chemicals used in Micro Dist procedures.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez revoir toutes les feuilles de données de sécurité sur les matériaux concernant les informations en matière de sécurité spécifiques aux produits chimiques utilisés dans les procédures Micro Dist.

Though the Micro Dist methods normally release no hazardous gases, there exists the potential for a hazardous situation to occur. Table 2 describes the occupational threshold limits for gases that may potentially be released from the Micro Dist in a fault condition.

Table 2 Hazardous gas information for specific methods

Method name	Gas	Manual section	Occupational Threshold Limit[1,2,3]	Concentration (per m^3)
Cyanide-1	Cyanide (HCN)	8.1 on page 31	10 ppm per 8 hours	11 mg
Cyanide-2	Cyanide (HCN)	9.1 on page 37	10 ppm per 8 hours	11 mg
Cyanide-3 (WAD)	Cyanide (HCN)	10.1 on page 39	10 ppm per 8 hours	11 mg
Cyanide-5	Cyanide (HCN)	11.1 on page 43	10 ppm per 8 hours	11 mg
Phenolics-1	Phenol (C_6H_5OH)	12.1 on page 45	5 ppm per 8 hours	19 mg
Sulfide-1	Hydrogen Sulfide (H_2S)	13.1 on page 47	20 ppm	15 mg
Sulfide-2	Hydrogen Sulfide (H_2S)	14.1 on page 51	20 ppm	15 mg
Ammonia-1	Ammonia (NH_3)	15.1 on page 55	50 ppm per 8 hours	35 mg
Ammonia-2	Ammonia (NH_3)	16.1 on page 59	50 ppm per 8 hours	35 mg
Tritium-1	Tritium (^3H)	17.1 on page 61	5000 mrem per year	N/A

[1] http://www.cdc.gov/niosh/pdfs/0337.pdf (H_2S)
[2] http://www.cdc.gov/NIOSH/pdfs/0333.pdf (HCN)
[3] http://www.cdc.gov/niosh/pdfs/74-136a.pdf (Ammonia)

Important Note: *Use this procedure as a template for all Micro Dist™ Methods. The specific methods refer to figures in this section.*

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to temperature specified in method. Allow the heater block to warm up. This will take about 40 minutes.
2. With the **M** end up, put collector tubes for the samples into the collector tube rack. When using the Pre-filled collector tubes, re-seal the foil pack after removing the tubes if there are any left. When using the User-Fill collector tubes, fill and cap the tubes before using; see section 3.1 on page 11.
3. Put the sample tubes into the sample tube rack; up to 21 for one block. Pipet 6.0 mL of aqueous sample into each sample tube with an automatic pipet or weigh 6.0 grams of sample on a balance (Figure 3).
    a. When distilling solids, contaminated soils or sludge, place 0.5 g of sample, then 5.5 mL of deionized water into the sample tube.

Figure 3 Placing Sample in Tubes

4. In order to release the analyte from the matrix, it may be necessary to add a releasing solution. The releasing solution is added via a repipetor at a volume that is specified in the Micro Dist method (Figure 4).

Figure 4 Adding the Releasing Solution

5. Immediately push the **D** end of a collector tube over the open end of each sample tube to start the seal (Figure 5).
6. Place the assembly in the press (Figure 5), putting the sample tube through the hole in the white base. Before pressing, the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.

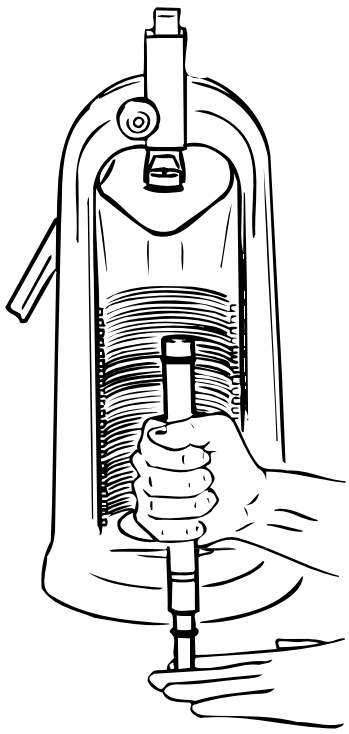

Figure 5 Starting the Seal

7. The pressing motion should be a smooth constant pressure which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.
8. Put on the heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.
9. Set timer for specified time in method.
10. When time is up, put on the heat-resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion (Figure 6) as opposed to a sideways motion. Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur, which may cause low recoveries. Dispose of the sample tube and the hot solution left in it by dropping it into the sink or waste bucket.

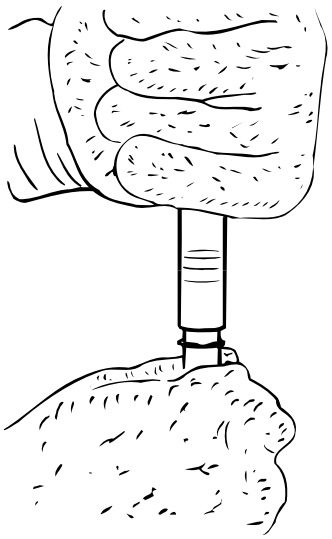

Figure 6 Removing the Sample Tube

11. Place each collector tub into the collector tube rack, with the **M** end up (Figure 7). It should take less than two minutes to pull and separate all 21 tubes. Parafilm the **D** end of the tube and allow the tubes to cool for at least 10 minutes.

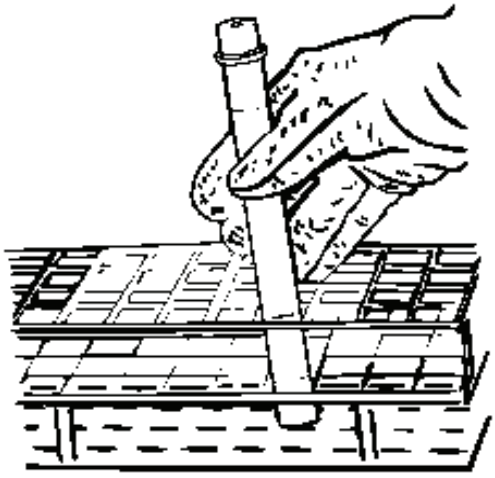

Figure 7 Placing the Collector Tube in the Rack

12. For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Holding the tube up to the light aids in seeing the condensate inside the tube. Then, slowly return the collector tube to an upright position so that the **D** end is up (Figure 8). Stubborn drops will often fall into the **M** end when the tube is flicked with a finger.

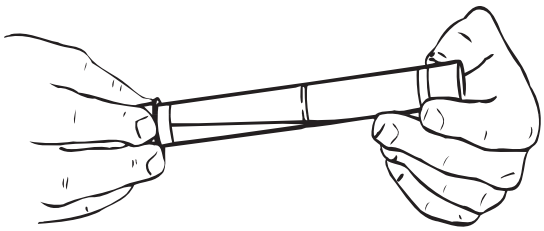

Figure 8 Rinsing the Collector Tube Walls

13. With the **D** end still up, break the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end (Figure 9). Discard the **D** end (Figure 10). Prevent lost droplets by holding the **M** end on a firm surface and cracking off the **D** end.

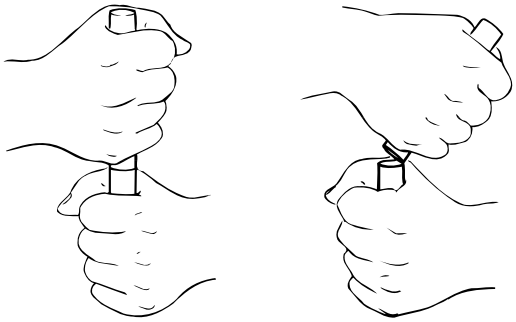

Figure 9 Breaking the ‘D’ End of the Collector Tube

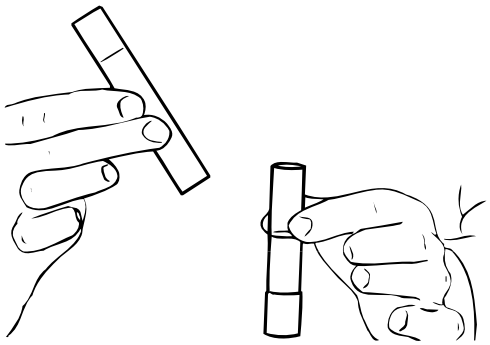

Figure 10 Discarding the ‘D' End

14. In the remaining **M** end of the collector tube, dilute to the 6.0 mL mark with deionized water, or dilute to 10 mL. This results in the original sample volume.
15. Shake the tube with a gentle whipping motion to mix in diluent water (Figure 11). Do not invert it. With the **M** end down, place the tube into the collector tube rack (Figure 7 on page 27).
16. Determine the solution in the tube with a Lachat QuikChem Method. There is enough distillate for two 25 second sample periods. Seal both ends of the tube with Parafilm if the sample will not be determined immediately..

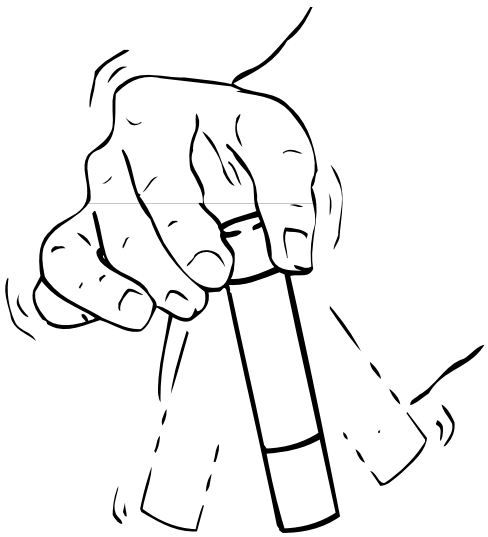

Figure 11 Shaking the Tube

Section 8 Cyanide–1

8.1 Micro Dist™ Method (Method Number 10-204-00-1-X)

DANGER
Chemical inhalation hazard. To avoid potential exposure to cyanide gas (HCN), the manufacturer recommends operating the Micro Dist under a fume hood.

DANGER
Risque d'inhalation de produits chimiques. Pour éviter le contact potentiel avec du gaz de Cyanure d'hydrogène (HCN), le fabricant recommande d'utiliser le Micro Dist sous une hotte aspirante.

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Always wear personal protective equipment appropriate for the chemicals you are handling.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez toujours porter l'équipement de protection personnel approprié pour les produits chimiques que vous manipulez.

This is a method for distilling samples containing 0.002 to 100 mg Free and Combined Cyanide/L with the Micro Dist distillation apparatus.

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to 120 °C. Allow the heater block to warm up. This will take about 40 minutes.
2. With the **M** end up, put the required number of collector tubes into the collector tube rack. When using the Pre-filled collector tubes, re-seal the foil pack after removing the tubes if there are any left. When using the User-Fill collector tubes, fill and cap the tubes before using.
3. Put the required number of sample tubes into the sample tube rack; up to 21 for one block. Place 6.0 mL of sample (or standard) into each sample tube with an automatic pipet.
4. Add between 0.5 to 1.0 g of soil or sludge to 5 mL of DI water. The amount of cyanide in the soil or sludge in 6 mL should be between about 0.012 and 600 µg. If the sample is high in organics, a smaller sample size or less DI water may be required. Foaming or caking of the membrane is often eliminated by the above measures. If not, see Running Solid Samples with Micro Dist on page 70.
5. In this method, it is imperative that the standards be distilled with the samples.
6. In order to both release the free cyanide as HCN and to digest complexed cyanides during the digestion/distillation, first prime the re-pipettor several times into a waste container. Then, add 0.75 mL of 7.11 M sulfuric acid / 0.79 M magnesium chloride solution to the sample tube using the supplied automatic pipet.
7. Immediately push the **D** end of a Cyanide–1 collector tube over the open end of each sample tube to start the seal.
8. Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
9. The pressing motion should be a smooth constant pressure which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.

10. Put on the heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.
11. Set the timer for 30 minutes.
12. When 30 minutes is up, put on the heat-resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion as opposed to a sideways motion. Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution left in it by dropping it into the sink or waste bucket.
13. Invert each collector tube and place it into the collector tube rack, with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.
14. Allow tubes to cool for at least 10 minutes.
15. For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Then, slowly return the collector tube to an upright position so that the **D** end is up. Stubborn drops will often fall into the **M** end when the tube is flicked with a finger.
16. With the **D** end still up, break the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end. Discard the **D** end.
17. In the remaining **M** end of the collector tube, dilute to the 6.0 mL mark with deionized water. This results in the original sample volume, but now in 0.25 M NaOH.
18. Shake the tube with a gentle whipping motion to mix in diluent water. Do not invert the sample. With the **M** end down, place the tube into the collector tube rack.
19. Determine the solution in the tube with Lachat's QuikChem Method 10–204–00–1–X or equivalent. There is enough distillate for two 25 second sample periods. Seal both ends of the tube with Parafilm if the sample will not be determined immediately.

8.2 Releasing Agent

CAUTION
Chemical and burn hazard. Upon the addition of sulfuric acid to water, the solution will become very hot. Never add water to concentrated acid. When preparing this reagent, follow these steps very carefully.

ATTENTION
Risque chimique et risque de brûlure. Lorsque de l'acide sulfurique (H_2SO_4) est ajouté à de l'eau, la température de la solution augmente rapidement. N'ajoutez jamais de l'eau dans de l'acide concentré. Lors de la préparation de ce réactif, suivez ces étapes avec beaucoup de précautions.

200 mL of 7.11 M sulfuric acid / 0.79 M magnesium chloride recipe:

1. In the hood, place a 500 mL beaker on a top-loading balance with a 400 g capacity or greater. It is best to do this in the hood as HCl fumes will be released. Tare the balance.
2. Into the tared 500 mL beaker place 110.8 g deionized water. Then add and dissolve completely 32.2 g magnesium chloride hexahydrate ($MgCl_2 \cdot 6\ H_2O$) in this water. Use 99% A.C.S. reagent as any trace cyanide in this reagent will contribute to a distillation blank.
3. Slowly add 139 g concentrated sulfuric acid in increments of 40 g at a time, swirling and allowing to cool. HCl fumes will be released. Use the purest grade possible, as any trace cyanide in this reagent will contribute to a distillation blank.
4. Transfer the solution to the automatic pipet container. Place the assembled and calibrated pipet cap on loosely and allow the solution to cool to room temperature in the hood.
5. When the solution is at room temperature, screw the cap on tightly. Prime the pipet and the solution is ready to use in the Cyanide–1 method.

8.3 Sources

- U.S. Environmental Protection Agency, Methods for Chemical Analysis of Waters and Wastes, Method 335.2. EPA–600, 4–79–020, Revised March 1983.
- APHA–AWWA–WEF Standard Methods for the Examination of Water and Wastewater, 20th ed. (1998), Method 4500–CN–N., pp 4–0 to 4–34.
- U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical / Chemical Methods (SW–846), Method 9010B, Revision 2, December 1996, “Total and Amenable Cyanide: Distillation”.
- U.S. Environmental Protection Agency, Methods for the Determination of Inorganic Substances in Environmental Samples, Method 335.4. EPA/600/R–93, August 1993.
- U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical / Chemical Methods (SW–846), Method 9213, Revision 0, December 1996, “Potentiometric determination of cyanide in aqueous samples and distillates with ion-selective electrodes”

8.4 USEPA Method 335.2 Scaled-Down

For 6.0 mL of Micro Distilled Sample, the scale-down factor for this batch colorimetric method is 6/50 or 0.120. Here, the changes we suggest in apparatus or technique are [added in brackets]. The scale-down gives the same concentrations of reagents, sample, and analyte from Section 8.7, p 335.2–4 (see section 8.3 on page 33):

8.7 Withdraw 6.0 mL of the solution from the flask [collector tube] and transfer to a 20 mL erlenmeyer flask. Add 1.80 mL of sodium phosphate solution (7.6) and mix.

8.7.1 Pyridine-barbituric acid method: Add 0.24 mL of chloramine T (7.12) and mix. After one to two minutes, add 0.60 mL of pyridine-barbituric acid solution (7.13.1) and mix. [Now add 3.36 mL of deionized water] and mix again. Allow eight minutes for color development, then read absorbance at 578 nm in a 1 cm cell within 15 minutes.

8.7.2 Pyridine-pyrazolone method: Add 0.060 mL of chloramine T (7.12) and mix. After one to two minutes add 0.60 mL of pyridine-pyrazolone solution (7.13.1) and mix. [Now add 3.54 mL deionized water] and mix again. After 40 minutes read absorbance at 620 nm in a 1 cm cell.

Note: *Some distillates may contain compounds that have a chlorine demand. One minute after the addition of chloramine T, test for residual chlorine with KI-starch paper. If the test is negative, add an additional 0.060 mL of chloramine T. After one minute, recheck the sample.*

Note: *More than 0.060 mL of chloramine T will prevent the color from developing with pyridine-pyrazolone.*

8.5 Calculations Showing Equivalency of Concentrations of Releasing Solutions

Equivalency of releasing solutions in Micro Dist Method Cyanide–1 and in USEPA Method 335.2 (see section 8.3 on page 33).

8.5.1 Micro Dist Method Cyanide–1

To 6.0 mL of sample is added 0.75 mL of 7.11 M H_2SO_4. This gives a sample concentration in H_2SO_4:

$$7.11\ \text{M}\ H_2SO_4 \times 0.75\ \text{mL} / (6.0\ \text{mL} + 0.75\ \text{mL}) = 0.79\ \text{M}\ H_2SO_4$$

This same solution is also 0.79 M in $MgCl_2 \cdot 6\ H_2O$. This gives a concentration of:

$$0.79\ \text{M}\ MgCl_2 \cdot 6\ H_2O \times 0.75\ \text{mL} / (6.75\ \text{mL total})$$
$$= (0.088\ \text{M}\ MgCl_2 \cdot 6\ H_2O)$$

8.5.2 USEPA Method 335.2

To 500 mL of sample is added 50 mL of 9 M (18 N) H_2SO_4. This gives a sample concentration in H_2SO_4:

$$0.9\ \text{M}\ H_2SO_4 \times 50\ \text{mL} / (500\ \text{mL} + 50\ \text{mL} + 20\ \text{mL}) = 0.79\ \text{M}\ H_2SO_4$$

To 500 mL of sample is also added 20 mL of a 510 g $MgCl_2 \cdot 6\ H_2O$/L (2.5 M) solution. This gives a concentration of:

$$2.5\ \text{M}\ MgCl_2 \cdot 6\ H_2O \times 20\ \text{mL} / (570\ \text{mL total})$$
$$= (0.088\ \text{M}\ MgCl_2 \cdot 6\ H_2O)$$

8.5.3 USEPA Method 335.4

To 50 mL of sample is added 5 mL of 9 M (18 N) H_2SO_4. This gives a sample concentration in H_2SO_4:

$$9\ \text{M}\ H_2SO_4 \times 5\ \text{mL} / (50\ \text{mL} + 5\ \text{mL} + 2\ \text{mL}) = 0.79\ \text{M}\ H_2SO_4$$

To 50 mL of sample is also added 2 mL of a 510 g $MgCl_2 \cdot 6\ H_2O$/L (2.5 M) solution. This gives a concentration of:

$$2.5\ \text{M}\ MgCl_2 \cdot 6\ H_2O \times 2\ \text{mL} / (57\ \text{mL total})$$

$$= (0.088\ \text{M}\ MgCl_2 \cdot 6\ H_2O)$$

8.6 Calculations Showing Equivalency of Concentrations of Trapping Solution

Equivalency of Micro Dist Method Cyanide–1 and in USEPA Method 335.2.

8.6.1 Micro Dist Method Cyanide–1

In the Micro Dist Method, 1.5 mL of 1.0 M NaOH becomes 6.0 mL solution after distillation and dilution to the mark:

1.0 M NaOH x (1.5 mL / 6.0 mL) = 0.25 M NaOH

8.6.2 USEPA Method 335.2

In the macro distillation, 50 mL of 1.25 M NaOH becomes 250 mL solution after distillation and dilution to the mark:

1.25 M NaOH x (50 mL / 250 mL) = 0.25 M NaOH

USEPA Method 335.4

In the macro distillation, 50 mL of 0.25 M NaOH remains undiluted.

9.1 Micro Dist™ Method

DANGER
Chemical inhalation hazard. To avoid potential exposure to cyanide gas (HCN), the manufacturer recommends operating the Micro Dist under a fume hood.

DANGER
Risque d'inhalation de produit chimique. Pour éviter le contact potentiel avec du gaz de Cyanure d'hydrogène (HCN), le fabricant recommande d'utiliser le Micro Dist sous une hotte aspirante.

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Always wear personal protective equipment appropriate for the chemicals you are handling.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez toujours porter l'équipement de protection personnel approprié pour les produits chimiques que vous manipulez.

This is a method for preparing and distilling caustic extract solution samples (1.25 M NaOH) containing 0.5 to 50 mg Free and Combined Cyanide/L with the Micro Dist distillation apparatus.

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to 120 °C. Allow the heater block to warm up. This will take about 40 minutes.
2. With the **M** end up, put the required number of collector tubes into the collector tube rack. When using the Pre-filled collector tubes, re-seal the foil pack after removing the tubes if there are any left. When using the User-Fill collector tubes, fill and cap the tubes before using.
3. Put the required number of sample tubes into the sample tube rack; up to 21 for one block. Place 5.75 mL of sample (or standard) into each sample tube with an automatic pipet.
4. Add between 0.5 to 1.0 g of soil or sludge to 5 mL of DI water. If the sample is high in organics, a smaller sample size or less DI water may be required. Foaming or caking of the membrane is often eliminated by the above measures. If not, see Running Solid Samples with Micro Dist on page 70.
5. In this method, it is imperative that the standards be distilled with the samples.
6. Prior to releasing the free cyanide as HCN the sample must first be neutralized. Using a pipette add 200 µL concentrated H_2SO_4.
7. In order to both release the free cyanide as HCN and to digest complexed cyanides during the digestion/distillation, first prime the re-pipettor several times into a waste container. Then, add 0.75 mL of 7.11 M sulfuric acid / 0.79 M magnesium chloride solution to the sample tube using the supplied automatic pipet.

8. Immediately push the **D** end of a Cyanide–1 collector tube over the open end of each sample tube to start the seal.
9. Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
10. The pressing motion should be a smooth constant pressure which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.
11. Put on the heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.
12. Set the timer for 30 minutes.
13. When 30 minutes is up, put on the heat-resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion as opposed to a sideways motion. Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution left in it by dropping it into the sink or waste bucket.
14. Invert each collector tube and place it into the collector tube rack, with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.
15. Parafilm the **M** end and allow tubes to cool for at least 10 minutes.
16. For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Then, slowly return the collector tube to an upright position so that the **D** end is up. Stubborn drops will often fall into the **M** end when the tube is flicked with a finger.
17. With the **D** end still up, break the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end. Discard the **D** end.
18. In the remaining **M** end of the collector tube, dilute to the 6.0 mL mark with deionized water. This results in the original sample volume, but now in 0.25 M NaOH.
19. Shake the tube with a gentle whipping motion to mix in diluent water. Do not invert the sample. With the **M** end down, place the tube into the collector tube rack.
20. Determine the solution in the tube with Lachat's QuikChem Method 10–204–00–1–X or equivalent. There is enough distillate for two 25 second sample periods. Seal both ends of the tube with Parafilm if the sample will not be determined immediately.

Section 10 Cyanide–3 (WAD)

10.1 Micro Dist™ Method

DANGER
Chemical inhalation hazard. To avoid potential exposure to cyanide gas (HCN), the manufacturer recommends operating the Micro Dist under a fume hood.

DANGER
Risque d'inhalation de produit chimique. Pour éviter le contact potentiel avec du gaz de Cyanure d'hydrogène (HCN), le fabricant recommande d'utiliser le Micro Dist sous une hotte aspirante.

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Always wear personal protective equipment appropriate for the chemicals you are handling.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez toujours porter l'équipement de protection personnel approprié pour les produits chimiques que vous manipulez.

This is a method for preparing and distilling samples containing 0.002 to 0.50 mg Free and Weak Acid Dissociable (WAD) Cyanide/L with the Micro Dist distillation apparatus.

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to 120 °C. Allow the heater block to warm up. This will take about 40 minutes.
2. With the **M** end up, put the required number of collector tubes into the collector tube rack. When using the Pre-filled collector tubes, re-seal the foil pack after removing the tubes if there are any left. When using the User-Fill collector tubes, fill and cap the tubes before using.
3. Put the required number of sample tubes into the sample tube rack; up to 21 for one block. Place 6.0 mL of sample into each sample tube with an automatic pipet.
4. Add between 0.5 to 1.0 g of soil or sludge to 5 mL of DI water. If the sample is high in organics, a smaller sample size or less DI water may be required. Foaming or caking of the membrane is often eliminated by the above measures. If not, see Running Solid Samples with Micro Dist on page 70.
5. In this method, it is imperative that the standards be distilled with the samples.
6. In order to release the cyanide as HCN, add 0.75 mL of 0.50 M zinc acetate/0.52 M sodium acetate/0.87 M acetic acid releasing solution to the sample tube using the supplied pipet. See section 10.2 on page 41 for a recipe for this.

7. Immediately push the **D** end of a Cyanide–3 collector tube over the open end of each sample tube to start the seal.
8. Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
9. The pressing motion should be a smooth constant pressure which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.
10. Put on the heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.
11. Set the timer for 30 minutes.
12. When 30 minutes is up, put on the heat-resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion as opposed to a sideways motion. Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution left in it by dropping it into the sink or waste bucket.
13. Invert each collector tube and place it into the collector tube rack, with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.
14. Parafilm the **M** end and allow tubes to cool for at least 10 minutes.
15. For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Then, slowly return the collector tube to an upright position so that the **D** end is up. Stubborn drops will often fall into the **M** end when the tube is flicked with a finger.
16. With the **D** end still up, break the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end. Discard the **D** end.
17. In the remaining **M** end of the collector tube, dilute to the 6.0 mL mark with deionized water. This results in the original sample volume, but now in 0.25 M NaOH.
18. Shake the tube with a gentle whipping motion to mix in diluent water. Do not invert the sample. With the **M** end down, place the tube into the collector tube rack.
19. Determine the solution in the tube with Lachat's QuikChem Method 10–204–00–1–X or equivalent. There is enough distillate for two 25 second sample periods. Seal both ends of the tube with Parafilm if the sample will not be determined immediately.

10.2 Releasing Agent

1 L of 0.87 M acetic acid / 0.50 M zinc acetate / 0.52 M sodium acetate

Make this releasing solution in a 1 L container and then place it directly in the automatic re-pipettor supplied with the starter kit. This is enough to treat 1300 samples.

1. Place a 1 L container onto a top-loading balance with a 1000 g capacity or greater. We recommend this be prepared in the hood as acetic acid fumes will be released. Tare the balance.
2. Into the tared 1 L container place 950 g deionized water. Then dissolve 70.97 g sodium acetate trihydrate ($NaC_2H_3O_2 \cdot 3\ H_2O$) in this water. Dissolve 100 g zinc acetate monohydrate ($ZnC_2H_3O_2 \cdot H_2O$) in this solution. Then add 52.5 g glacial acetic acid ($C_2H_3O_2$).
3. Transfer this solution to the re-pipettor, screw the cap on tight and prime the pipet. The solution is ready to use in method Cyanide–3.

10.3 Sources

- APHA–AWWA–WPCF Standard Methods for the Examination of Water and Wastewater, 20th ed., Method 4500–CN–N., pp 4–0 to 4–34.

Section 11 Cyanide–5

11.1 Micro Dist™ Method

DANGER
Chemical inhalation hazard. To avoid potential exposure to cyanide gas (HCN), the manufacturer recommends operating the Micro Dist under a fume hood.

DANGER
Risque d'inhalation de produit chimique. Pour éviter le contact potentiel avec du gaz de Cyanure d'hydrogène (HCN), le fabricant recommande d'utiliser le Micro Dist sous une hotte aspirante.

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Always wear personal protective equipment appropriate for the chemicals you are handling.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez toujours porter l'équipement de protection personnel approprié pour les produits chimiques que vous manipulez.

This is a method for preparing and distilling samples containing 0.05 to 50 mg Free and Combined Cyanide/L in the presence of sulfide with the Micro Dist distillation apparatus.

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to 120 °C. Allow the heater block to warm up. This will take about 40 minutes.
2. With the **M** end up, put the required number of collector tubes into the collector tube rack. When using the Pre-filled collector tubes, re-seal the foil pack after removing the tubes if there are any left. When using the User-Fill collector tubes, fill and cap the tubes before using.
3. Put the required number of collector tubes into the sample tube rack; up to 21 for one block. Place 6.0 mL of sample (or standard) into each sample tube with an automatic pipet.
4. Add between 0.5 to 1.0 g of soil or sludge to 5 mL of DI water. If the sample is high in organics, a smaller sample size or less DI water may be required. Foaming or caking of the membrane is often eliminated by the above measures. If not, see Running Solid Samples with Micro Dist on page 70.
5. In this method, it is imperative that the standards be distilled with the samples.
6. In order to both release the free cyanide as HCN and to digest complexed cyanides during the digestion/distillation, first prime the re-pipettor several times into a waste container. Add 0.75 mL of 7.11 M sulfuric acid / 0.79 M magnesium chloride solution

to the sample tube using the supplied automatic pipet. See section 8.2 on page 32 for this recipe.

7. Immediately push the **D** end of a Cyanide–5 collector tube over the open end of each sample tube to start the seal.

Section 12 Phenolics–1

12.1 Micro Dist™ Method

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Always wear personal protective equipment appropriate for the chemicals you are handling.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez toujours porter l'équipement de protection personnel approprié pour les produits chimiques que vous manipulez.

This is a method for distilling samples containing 0.002 to 50.0 mg phenolics/L with the Micro Dist distillation apparatus.

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to 130 °C. Allow the heater block to warm up. This will take about 40 minutes.
2. The pH of the sample should be adjusted to about pH 4 with 1 M NaOH or 10% H_2SO_4 before distillation.
3. With the **M** end up, put the required number of collector tubes into the collector tube rack.
4. Place 6.0 mL of sample into each sample tube with an automatic pipet.
5. Add between 0.5 to 1.0 g of soil or sludge to 5 mL of DI water. If the sample is high in organics, a smaller sample size or less DI water may be required. Foaming or caking of the membrane is often eliminated by the above measures. If not, see Running Solid Samples with Micro Dist on page 70.
6. Immediately push the **D** end of a Phenolics–1 collector tube over the open end of each sample tube to start the seal.
7. Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
8. The pressing motion should be a smooth constant pressure which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.
9. Put on the heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.
10. Set the timer for 90 minutes.
11. When 90 minutes is up, put on the heat-resistant gloves. Remove the first tube from the block and immediately pull off its

sample tube using a downward, twisting motion as opposed to a sideways motion. Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution left in it by dropping it into the sink or waste bucket.

12. Invert each collector tube and place it into the collector tube rack, with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.
13. Parafilm the **M** end and allow tubes to cool for at least 10 minutes.
14. For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Then, slowly return the collector tube to an upright position so that the **D** end is up. Stubborn drops will often fall into the **M** end when the tube is flicked with a finger.
15. With the **D** end still up, break the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end. Discard the **D** end.
16. In the remaining **M** end of the collector tube, dilute to the 6.0 mL mark with deionized water.
17. Shake the tube with a gentle whipping motion to mix in diluent water. Do not invert the sample. With the **M** end down, place the tube into the collector tube rack.
18. Determine the solution in the tube with Lachat's QuikChem Method 10–210–00–1–A, B, D, Y or equivalent. There is enough distillate for two 25 second sample periods. Seal both ends with Parafilm if the sample will not be determined immediately.

12.2 Sources

- U.S. Environmental Protection Agency, Methods for the Determination of Inorganic Substances in Environmental Samples, Method 420.4. EPA/600/R–93/100, Revised August 1993.
- U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical / Chemical Methods (SW–846), Method 9065, Revision 0, September 1986, “Phenolics.”

Section 13 Sulfide–1

13.1 Micro Dist™ Method

CAUTION
Chemical inhalation hazard. To avoid potential exposure to hydrogen sulfide gas (H_2S), the manufacturer recommends operating the Micro Dist under a fume hood.

ATTENTION
Risque d'inhalation de produit chimique. Pour éviter le contact potentiel avec du gaz de sulfure d'hydrogène (H2S), le fabricant recommande d'utiliser le Micro Dist sous une hotte aspirante.

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Always wear personal protective equipment appropriate for the chemicals you are handling.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez toujours porter l'équipement de protection personnel approprié pour les produits chimiques que vous manipulez.

This is a method for distilling solid and liquid samples containing 0.2 to 50 mg/kg acid-soluble sulfide, for iodometric determination.

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to 120 °C. Allow the heater block to warm up. This will take about 45 minutes.
2. With the **M** end up, put the required number of collector tubes into the collector tube rack. When using the Pre-filled collector tubes, re-seal the foil pack after removing the tubes if there are any left. When using the User-Fill collector tubes, fill and cap the tubes before using.
3. Put the required number of sample tubes into the sample tube rack; up to 21 for one block. Place 1.0 g of solid sample or 5.5 mL of standard solution into each sample tube. For solid samples, add about 5.0 mL deionized water.
4. Add between 0.5 to 1.0 g of soil or sludge to 5 mL of DI water. If the sample is high in organics, a smaller sample size or less DI water may be required. Foaming or caking of the membrane is often eliminated by the above measures. If not, see Running Solid Samples with Micro Dist on page 70.
5. In order to both release the free sulfide as H_2S (g) and to digest complexed sulfides during the digestion/ distillation, add 0.45 mL of 9.0 M sulfuric acid solution to each sample tube using the supplied automatic pipet.

   Note: *When running an unknown matrix for the first time it is advisable to determine the approximate amount of concentrated sulfuric required to reduce the pH of the sample to less than or equal to 1. The volume*

required may be more than 0.45 mL. See section 13.3 on page 49 for further details.

6. Immediately push the **D** end of a Sulfide–1 collector tube over the open end of each sample tube to start the seal.
7. Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
8. The pressing motion should be a smooth constant pressure which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.
9. Put on the heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.
10. Set the timer for 30 minutes.
11. When 30 minutes is up, put on the heat-resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion as opposed to a sideways motion. Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution left in it by dropping it into the sink or waste bucket.
12. Invert each collector tube and place it into the collector tube rack, with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.
13. Parafilm the **M** end and allow tubes to cool for at least 10 minutes.
14. For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Then, slowly return the collector tube to an upright position so that the **D** end is up. Stubborn drops will often fall into the **M** end when the tube is flicked with a finger.
15. With the **D** end still up, break the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end. Discard the **D** end.
16. In the remaining **M** end of the collector tube, dilute to the 6.0 mL mark with deionized water.
17. Shake the tube with a gentle whipping motion to mix in diluent water. Do not invert the sample. With the **M** end down, place the tube into the collector tube rack.
18. The solution in the remaining **M** end of the collector tube contains the sample's sulfide in the form of ZnS (s). This solid can then be transferred and titrated iodometrically with an appropriately scaled-down method such as EPA's SW–846 Method 9030, Revision 1 (Dec. 1987) or EPA's Method 376.1.

13.2 Releasing Agent

300 mL of 9.0 M sulfuric acid releasing agent for the Sulfide–1 Method:

Make this releasing solution up in a beaker under the hood and transfer to the automatic re-pipettor supplied with the starter kit after cooling. This is enough to treat 250 samples.

CAUTION
Chemical burn hazard. Upon the addition of sulfuric acid to water, the solution will become very hot. Never add water to concentrated acid. When preparing this reagent, follow these steps very carefully.

ATTENTION
Risque de brûlure chimique. Lorsque de l'acide sulfurique (H_2SO_4) est ajouté à de l'eau, la température de la solution augmente rapidement. N'ajoutez jamais d'eau dans de l'acide concentré. Lorsque vous préparez ce réactif, suivez ces étapes avec précaution.

1. In the hood, place a 500 mL beaker onto a top loading balance with a 800 g capacity or greater. Tare the balance.
2. Place 150 g deionized water into the tared 500 mL beaker.
3. Slowly add 276 g concentrated sulfuric acid in increments of 40 g at a time, swirling and allowing to cool after each increment.
4. Allow the solution to cool to room temperature in the hood.
5. When the solution is at room temperature, transfer to the automatic re-pipettor and screw the cap on tightly. Prime the pipet, and the solution is ready to use in method Sulfide–1.
6. Periodically calibrate the re-pipettor for 1.0 mL as in Method Cyanide–1.

13.3 Sources

- U.S. Environmental Protection Agency, Methods for Chemical Analysis of Waters and Wastes, Method 376.1 (Titrimetric, Iodine), EPA–600, 4–79–020, Revised March 1983.
- U.S. Environmental Protection Agency, Methods for Chemical Analysis of Waters and Wastes, Method 376.2 (Colorimetric, Methylene Blue), EPA–600, 4–79–020, Revised March 1983.
- U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Wastes (SW–846) Method 9030B, Revision 2, December 1996, “Acid-soluble and acid-insoluble sulfides: distillation”.
- U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Wastes (SW–846) Method 9215, Revision 0, December 1996, “Potentiometric determination of sulfide in aqueous samples and distillates with ion-selective electrode”

13.4 Comparison of this method and SW–846 Method 9030B, Revision 2

Both methods use a trapping solution which is 0.043 M in zinc acetate and which contains 1.60% formaldehyde. The stock 37% formaldehyde solution contains 10–15% methanol.

2.0 mL of this solution is in each Sulfide–1 collector tube.

Section 14 Sulfide–2

14.1 Micro Dist™ Method

CAUTION
Chemical inhalation hazard. To avoid potential exposure to hydrogen sulfide gas (H_2S), the manufacturer recommends operating the Micro Dist under a fume hood.

ATTENTION
Risque d'inhalation de produit chimique. Pour éviter le contact potentiel avec du gaz de sulfure d'hydrogène (H2S), le fabricant recommande d'utiliser le Micro Dist sous une hotte aspirante.

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Always wear personal protective equipment appropriate for the chemicals you are handling.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez toujours porter l'équipement de protection personnel approprié pour les produits chimiques que vous manipulez.

This is a method for distilling samples containing 0.01-10.0 mg acid soluble S/L with the Micro Dist distillation apparatus, for colorimetric determination.

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to 120 °C. Allow the heater block to warm up. This will take about 40 minutes.
2. With the **M** end up, put the required number of collector tubes into the collector tube rack. When using the Pre-filled collector tubes, re-seal the foil pack after removing the tubes if there are any left. When using the User-Fill collector tubes, fill and cap the tubes before using.
3. Put the required number of sample tubes into the sample tube rack; up to 21 for one block. Place 6.0 mL of sample into each sample tube with an automatic pipet.
4. Add between 0.5 to 1.0 g of soil or sludge to 5 mL of DI water. If the sample is high in organics, a smaller sample size or less DI water may be required. Foaming or caking of the membrane is often eliminated by the above measures. If not, see Running Solid Samples with Micro Dist on page 70.
5. In order to both release the free sulfide as H_2S (g) and to digest complexed sulfides during the digestion/ distillation, add 0.45 mL of 9.0 M sulfuric acid solution to each sample tube using the supplied automatic pipet.

   Note: *When running an unknown matrix for the first time it is advisable to determine the approximate amount of concentrated sulfuric required to reduce the pH of the sample to less than or equal to 1. The volume*

required may be more than 0.45 mL. See section 14.2 on page 53 for further details.

6. Immediately push the **D** end of a Sulfide–2 collector tube over the open end of each sample tube to start the seal.
7. Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
8. The pressing motion should be a smooth constant pressure which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.
9. Put on the heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.
10. Set the timer for 30 minutes.
11. When 30 minutes is up, put on the heat-resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion as opposed to a sideways motion. Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution left in it by dropping it into the sink or waste bucket.
12. Invert each collector tube and place it into the collector tube rack, with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.
13. Parafilm the **M** end and allow tubes to cool for at least 10 minutes.
14. For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Then, slowly return the collector tube to an upright position so that the **D** end is up. Stubborn drops will often fall into the **M** end when the tube is flicked with a finger.
15. With the **D** end still up, break the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end. Discard the **D** end.
16. In the remaining **M** end of the collector tube, dilute to the 6.0 mL mark with deionized water. This results in the original sample volume, but now in 0.25 M NaOH.
17. Shake the tube with a gentle whipping motion to mix in diluent water. Do not invert the sample. With the **M** end down, place the tube into the collector tube rack.
18. Determine the solution in the remaining **M** end of the collector tube with Lachat's QuikChem Method 10–116–29–1–A equivalent. There is enough distillate for two 25 second

sample period. If the sample is not analyzed immediately, seal both ends of the tube with Parafilm.

14.2 Sources

- U.S. Environmental Protection Agency, Methods for Chemical Analysis of Waters and Wastes, Method 376.1 (Titrimetric, Iodine), EPA–600, 4–79–020, Revised March 1983.
- U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Wastes (SW–846) Method 9030B, Revision 2, December 1996, “Acid-soluble and acid-insoluble sulfides: distillation”.
- U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Wastes (SW–846) Method 9215, Revision 0, December 1996, “Potentiometric determination of sulfide in aqueous samples and distillates with ion-selective electrode”

14.3 Releasing Agent

300 mL of 9.0 M sulfuric acid releasing agent for the Sulfide–2 Micro Dist Method:

Make this releasing solution up in a beaker under the hood and transfer to the automatic re-pipettor supplied with the starter kit after cooling. This is enough to treat 600 samples.

CAUTION
Chemical burn hazard. Upon the addition of sulfuric acid to water, the solution will become very hot. Never add water to concentrated acid. When preparing this reagent, follow these steps very carefully.

ATTENTION
Risque de brûlure chimique. Lorsque de l'acide sulfurique (H_2SO_4) est ajouté à de l'eau, la température de la solution augmente rapidement. N'ajoutez jamais d'eau dans de l'acide concentré. Lorsque vous préparez ce réactif, suivez ces étapes avec précaution.

1. In the hood, place a 500 mL beaker onto a top loading balance with a 800 g capacity or greater. Tare the balance.
2. Place 150.0 g deionized water into the tared 500 mL beaker.
3. Add slowly 276 g concentrated sulfuric acid in increments of 40 g sulfuric acid while swirling. Allow to cool.
4. Allow the solution to cool to room temperature in the hood.
5. When the solution is at room temperature, transfer to the automatic re-pipettor and screw the cap on tightly. Prime the pipet, and the solution is ready to use in method Sulfide–2.
6. Monthly, calibrate the re-pipettor to 0.45 mL.

Section 15 Ammonia–1: Phenate Method/ISE

15.1 Micro Dist™ Method

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Always wear personal protective equipment appropriate for the chemicals you are handling.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez toujours porter l'équipement de protection personnel approprié pour les produits chimiques que vous manipulez.

This is a method for distilling samples containing 0.010 to 2.0 mg N/L with the Micro Dist distillation apparatus.

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to 120 °C. Allow the heater block to warm up. This will take about 40 minutes.
2. With the **M** end up, put the required number of collector tubes into the collector tube rack. Fill the tubes with the trapping solution and cap the tubes.
3. Place 6.0 mL of sample into each sample tube with an automatic pipet.
4. Add between 0.5 to 1.0 g of soil or sludge to 5 mL of DI water. If the sample is high in organics, a smaller sample size or less DI water may be required. Foaming or caking of the membrane is often eliminated by the above measures. If not, see Running Solid Samples with Micro Dist on page 70.
5. To decrease the hydrolysis of cyanates and organic nitrogen compounds, the sample is buffered at pH 9.5 by adding 1.0 mL of 0.55 M borate buffer ($Na_2B_4O_7$). for samples preserved with 2 mL concentrated sulfuric acid per liter or 0.75 mL of a 0.011 M borate buffer ($Na_2B_4O_7$) for samples that are not preserved with acid.
6. Immediately push the **D** end of a Ammonia–1 collector tube over the open end of each sample tube to start the seal.
7. Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
8. The pressing motion should be a smooth constant pressure which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.
9. Put on the heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that

the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.

10. Set the timer for 30 minutes.
11. When 30 minutes is up, put on the heat-resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion as opposed to a sideways motion. Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution left in it by dropping it into the sink or waste bucket.
12. Invert each collector tube and place it into the collector tube rack, now with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.
13. Parafilm the **M** end and allow tubes to cool for at least 10 minutes.
14. For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Then, slowly return the collector tube to an upright position so that the **D** end is up. Stubborn drops will often fall into the **M** end when the tube is flicked with a finger.
15. With the **D** end still up, break the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end. Discard the **D** end.
16. In the remaining **M** end of the collector tube, dilute to the 6.0 mL mark with deionized water. This results in the original sample volume, but now in 0.003 M sulfuric acid.
17. Shake the tube with a gentle whipping motion to mix in diluent water. Do not invert the sample. With the **M** end down, place the tube into the collector tube rack.
18. Determine the solution in the tube with Lachat QuikChem Method 10–107–06–1–X. or equivalent. There is enough distillate for two 25 second sample periods. It is best to do this the same day if possible. Seal both ends with Parafilm if the sample will not be determined immediately.
19. Typically, the Ammonia–1 collector tubes will contribute an ammonia blank of 15 mg N/L, or about 1 mM in the 6.0 mL of distillate. Therefore, it is important to distill both calibration standards and all samples using the Ammonia–1 tubes.

15.2 Borate Buffer

Make this buffer solution up in a 1 L container and then place directly in the automatic re-pipettor supplied with the starter kit. This will make 1 L of 0.011 M $Na_2B_4O_7$. This is enough to treat 1300 samples.

For samples not preserved with acid:

1. Place a 1 L container onto a top-loading balance with a 1000 g capacity or greater. Tare the balance.
2. Dissolve 2.17 g sodium borate ($Na_2B_4O_7$) in 964 g deionized water into the tared 1 L container. Add 36 g 0.1 M sodium hydroxide (NaOH). Mix well.
3. Transfer this solution to the re-pipettor, screw the cap on tight and prime the pipet. The solution is ready to use in method Ammonia–1. Recalibrate the re-pipettor once a month with deionized water.

For acid preserved samples (2 mL concentrated sulfuric acid per liter):

1. Place a 1 L container onto a top-loading balance with a 1000 g capacity or greater. Tare the balance.
2. Dissolve 5 g sodium borate ($Na_2B_4O_7$) in 964 g deionized water into a 1 L volumetric flask. Add 22 g sodium hydroxide (NaOH). Mix well.
3. Transfer this solution to the re-pipettor, screw the cap on tight and prime the pipet. The solution is ready to use in method Ammonia-1. Recalibrate the re-pipettor once a month with deionized water.

15.3 Sources

- APHA–AWWA–WEF Standard Methods for the Examination of Water and Wastewater, 20th ed. (1998), Method 4500–NH3 D. to H., pp. 4–106 to 4–112.

Section 16 Ammonia–2: Nesslerization Method

16.1 Micro Dist™ Method

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Always wear personal protective equipment appropriate for the chemicals you are handling.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez toujours porter l'équipement de protection personnel approprié pour les produits chimiques que vous manipulez.

This is a method for distilling samples containing 0.010 to 2.0 mg N/L with the Micro Dist distillation apparatus.

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to 120 °C. Allow the heater block to warm up. This will take about 40 minutes.
2. With the **M** end up, put the required number of collector tubes into the collector tube rack. Fill the tubes with the trapping solution and cap the tubes.
3. Place 6.0 mL of sample into each sample tube with an automatic pipet.
4. Add between 0.5 to 1.0 g of soil or sludge to 5 mL of DI water. If the sample is high in organics, a smaller sample size or less DI water may be required. Foaming or caking of the membrane is often eliminated by the above measures. If not, see Running Solid Samples with Micro Dist on page 70.
5. To decrease the hydrolysis of cyanates and organic nitrogen compounds, the sample is buffered at pH 9.5 by adding 0.75 mL of 0.011 M borate buffer ($Na_2B_4O_7$).
6. Immediately push the **D** end of a Ammonia–2 collector tube over the open end of each sample tube to start the seal.
7. Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
8. The pressing motion should be a smooth constant pressure which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.
9. Put on the heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.
10. Set the timer for 30 minutes.

11. When 30 minutes is up, put on the heat-resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion as opposed to a sideways motion. Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution left in it by dropping it into the sink or waste bucket.
12. Invert each collector tube and place it into the collector tube rack, now with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.
13. Parafilm the **M** end and allow tubes to cool for at least 10 minutes.
14. For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Then, slowly return the collector tube to an upright position so that the **D** end is up. Stubborn drops will often fall into the **M** end when the tube is flicked with the finger.
15. With the **D** end still up, break the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end. Discard the **D** end.
16. In the remaining **M** end of the collector tube, dilute to the 6.0 mL mark with deionized water. This results in the original sample volume, but now in 0.043 M boric acid solution.
17. Shake the tube with a gentle whipping motion to mix in diluent water. Do not invert the sample. With the **M** end down, place the tube into the collector tube rack.
18. Determine the solution in the tube following Standard Methods. Seal both ends with Parafilm if the sample will not be determined immediately.

16.2 Borate Buffer

Make this buffer solution up in a 1 L container and then place directly in the automatic re-pipettor supplied with the starter kit. This will make 1 L of 0.011 M $Na_2B_4O_7$, enough to treat 1300 samples.

1. Place a 1 L container onto a top-loading balance with a 1000 g capacity or greater. Tare the balance.
2. Dissolve 2.17 g sodium borate ($Na_2B_4O_7$) in 964 g deionized water into the tared 1 L container. Add 36 g 0.1 N sodium hydroxide (NaOH). Mix well.
3. Transfer this solution to the re-pipettor, screw the cap on tight and prime the pipet. The solution is ready to use in method Ammonia–2.

17.1 Micro Dist™ Method

CAUTION
Chemical hazard. Always follow appropriate laboratory safety procedures when handling chemicals. Always wear personal protective equipment appropriate for the chemicals you are handling.

ATTENTION
Danger chimique. Suivez toujours les procédures de laboratoire de sécurité lors de l'utilisation de produits chimiques. Veuillez toujours porter l'équipement de protection personnel approprié pour les produits chimiques que vous manipulez.

As part of routine nuclear power station monitoring, process water samples not directly from condensates, such as from release tanks, and ion-exchange streams and resins, are distilled to collect the volatile tritiated compounds, mainly tritiated water. The distillate is then counted in a scintillation instrument and this activity is used to determined the degree of contamination of the water sample. The distillation discriminates against any non-volatile source of contamination such as particles.

In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** means 'distillation' or 'discarded' end and **M** means 'measuring' end. See Figure 2 on page 13.

1. Set the controller to 120 °C. Allow the heater block to warm up. This will take about 40 minutes.
2. With the **M** end up, put the required number of collector tubes into the collector tube rack.
3. Put the required number of sample tubes into the sample tube rack; up to 21 for one block. Place 6.0 mL of water sample (or standard) into each sample tube with an automatic pipet. If the sample appears to be primarily oil, do not distill, but count directly with the scintillation counter.
4. Add 1 or 2 drops of 10 M NaOH to each sample tube.
5. Push the **D** end of a Tritium–1 collector tube over the open end of each sample tube to start the seal.
6. Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
7. The pressing motion should be a smooth constant pressure which is just enough to slide the sample tube inside the collector tube. A jerky, forced motion may cause added strain to the tube and could potentially crack it. Press down on the handle until the stop ring on the sample tube hits the **D** end of the collector tube.
8. Put on the heat-resistant gloves. Push the sample tube and **D** end of each tube all the way into the preheated block so that the collector tube stop ring touches the block. Placing 21 tubes should take less than one minute.
9. Set the timer for 90 minutes.

10. When 90 minutes is up, put on the heat-resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion as opposed to a sideways motion. Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur. Dispose of the sample tube and the hot solution left in it by dropping it into the sink or waste bucket.
11. Invert each collector tube and place it into the collector tube rack, now with the **D** end up. It should take less than two minutes to pull and separate all 21 tubes.
12. Allow tubes to cool for at least 10 minutes.
13. For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Then, slowly return the collector tube to an upright position so that the **D** end is up. Stubborn drops will often fall into the **M** end when the tube is flicked with a finger.
14. With the **D** end still up, break the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end. Discard the **D** end.
15. From the **M** end distillate, pipet sufficient sample and, if needed, dilution water into the scintillation vials. Then add scintillation cocktail and count, following the scintillation counter manufacturer's instructions. Seal both ends of the tube with Parafilm if the sample will not be determined immediately.

17.2 Sources

- DOE Method RP580 “Water distillation from soil and aqueous matrices using a microdistillation system for tritium determination”, from “DOE Methods for evaluating environmental and waste management samples”, DOE/EM–0089T, Rev 2, April 1995, U.S. Dept. of Energy.
- S. Stieg, “A Miniature Membrane Tube for Rapid Parallel Distillation of Cyanide, Phenolics, Ammonia, Sulfide, Methylmercury, and Tritium from Waters and Solids”, American Environmental Laboratory, v9, no. 10, Nov–Dec 1997, pp10–11.

Section 18 Replacement Parts and Accessories

Description	Cat. No.
Accessories Kit	17907
Fuse, T, 15A, 125V (for 100–115 V operation)	17085
Fuse, T, 6.3A, 250V (for 230 V operation)	20210
Automatic Pipettor	17024
Collector Tube Rack, 24 tube capacity	17012
Gloves, Neoprene, Large	17013S
Gloves, Neoprene, Small	17013L
Micro Dist™ Heating Block, Assembly	A17102/202
Pipettor Stand (for 17024)	17025
Sample Tube Rack, 60 tube capacity[1]	17011
Step-Up Transformer (110 V to 220 V), non-CE models only	17020
Timer	17021
Tube Press Assembly	17023

[1] The starter kit includes one set of 21 Micro Dist tubes with the purchase of the Micro Dist block.

Section 19 Contact Information

U.S.A. Customers

By Telephone:
6:30 a.m. to 5:00 p.m. MST
Monday through Friday
(800) 247-7613

By Fax:
(970) 461-3915

By Mail:
Hach Company
P.O. Box 608
Loveland, Colorado 80539-0608 U.S.A.
Ordering information by e-mail: sales@LachatInstruments.com

Information Required

- Hach account number (if available)
- Your name and phone number
- Purchase order number
- Brief description or model number
- Billing address
- Shipping address
- Catalog number
- Quantity

International Customers

Hach maintains a worldwide network of dealers and distributors. To locate the representative nearest you, send an e-mail to: intl@hach.com or contact:

Hach Company World Headquarters; Loveland, Colorado, U.S.A.
Telephone: (970) 669-3050; Fax: (970) 461-3915

Technical and Customer Service (U.S.A. only)

Lachat Instruments–Hach Company Technical and Customer Service Department personnel are eager to answer questions about our products and their use. Specialists in analytical methods, they are happy to put their talents to work for you.

Call 1-800-247-7613 or e-mail support@LachatInstruments.com

Section 20 Limited Warranty

Lachat Instruments, a Hach Company Brand, warrants its products to the original purchaser against any defects that are due to faulty material or workmanship for a period of *one year* from date of shipment unless otherwise noted in the product manual.

In the event that a defect is discovered during the warranty period, Lachat Instruments–Hach Company agrees that, at its option, it will repair or replace the defective product or refund the purchase price, excluding original shipping and handling charges. Any product repaired or replaced under this warranty will be warranted only for the remainder of the original product warranty period.

This warranty does not apply to consumable products such as chemical reagents; or consumable components of a product, such as, but not limited to, lamps and tubing.

Contact Lachat Instruments–Hach Company or your distributor to initiate warranty support. Products may not be returned without authorization from Lachat Instruments–Hach Company.

Limitations

This warranty does not cover:

- Damage caused by acts of God, natural disaster, labor unrest, acts of war (declared or undeclared), terrorism, civil strife, or acts of any governmental jurisdiction
- Damage caused by misuse, neglect, accident or improper application or installation
- Damage caused by any repair or attempted repair not authorized by Lachat Instruments–Hach Company
- Any product not used in accordance with the instructions furnished by Lachat Instruments–Hach Company
- Computer, Printer, or any other product which is covered by a warranty of the Original Manufacturer.
- Freight charges to return merchandise to Lachat Instruments–Hach Company
- Freight charges on expedited or express shipment of warranted parts or product
- Travel fees associated with on-site warranty repair

This warranty contains the sole express warranty made by Lachat Instruments–Hach Company in connection with its products. All implied warranties, including without limitation, the warranties of merchantability and fitness for a particular purpose, are expressly disclaimed.

Some states within the United States do not allow the disclaimer of implied warranties and if this is true in your state the above limitation may not apply to you. This warranty gives you specific rights, and you may also have other rights that vary from state to state.

This warranty constitutes the final, complete, and exclusive statement of warranty terms and no person is authorized to make any other warranties or representations on behalf of Lachat Instruments–Hach Company.

Limitation of Remedies

The remedies of repair, replacement or refund of purchase price as stated above are the exclusive remedies for the breach of this warranty. On the basis of strict liability or under any other legal theory, in no event shall Lachat Instruments–Hach Company be liable for any incidental or consequential damages of any kind for breach of warranty or negligence.

Appendix A Micro Dist Methods

A.1 Methods List

See Table 3 for descriptions of the Micro Dist methods. See Table 4 for a list of related QuikChem methods.

Table 3 Micro Dist Methods Summary

Micro Dist Method No.	Matrix and Chemistry	Collector Tube Trapping Solution Recipe	Assembled Pkg/21	User-Fill Pkg/10	User-Fill Pkg/50	User-Fill Pkg/100
Cyanide–1	waters, solids, strong acid dissociable (SAD), total cyanide	Add 1.5 mL or 1.55 g of 1.00 M standardized NaOH solution to each tube. When diluted to 6.0 mL, this gives 0.25 M NaOH. Other standardized concentrations can be used by adjusting the amount added to each tube.	A17001	A17017	A17517	A17117
Cyanide–2	caustic extracts, SAD, total cyanide	Add 1.5 mL or 1.55 g of 1.00 M standardized NaOH solution. When diluted to 6.0 mL, this gives 0.25 M NaOH. Other standardized concentrations can be used by adjusting the amount added to each tube.	A17001	A17017	A17517	A17117
Cyanide–3	waters, solids, weak acid dissociable (WAD)	Add 1.5 mL or 1.55 g of 0.1.00 M standardized NaOH solution. When diluted to 6.0 mL, this gives 0.25 M NaOH. Other standardized concentrations can be used by adjusting the amount added to each tube.	A17001	A17017	A17517	A17117
Cyanide–5	waters, solids, SAD (total cyanide) in the presence of sulfide	Dissolve 0.80 g PbCO3 in 1 L of 1.00 M standardized NaOH solution. Then, add 1.5 mL or 1.55 g of this solution to each tube. When diluted to 6.0 mL, this gives 0.25 M NaOH and 0.8 mM Pb. Other standardized NaOH concentrations can be used by adjusting the amount added to each tube.	A17011	A17017	A17517	A17117
Phenolics–1	waters, solids, 4–AATP	No trapping solution.	A17002	A17017	A17517	A17117
Sulfide–1 Acid Soluble Sulfides	waters, iodometric determination	Prepare a 0.043 M zinc acetate solution by dissolving the following: 8.78 g zinc acetate dihydrate, 0.10 g conc. HCl, 880.0 g deionized water, and 43.2 g of a 37% formaldehyde solution. Mix well until dissolved.Add 2.0 mL or 2.0 g of the zinc acetate solution to each tube.	A17003	A17017	A17517	A17117
Sulfide–2	waters/MTB colorimetric determination	Add 1.5 mL or 1.55 g of 1.00 M standardized NaOH solution. When diluted to 6.0 mL, this gives 0.25 M NaOH. Other standardized concentrations can be used by adjusting the amount.	A17001	A17017	A17517	A17117
Ammonia–1	waters, phenate colorimetric or ISE determination	Add 1.0 mL or 1.0 g of standardized 0.016 M sulfuric acid solution. When diluted to 6.0 mL, this gives 0.003 M sulfuric acid. Other standardized concentrations can be used by adjusting the amount.	Not Available	A17017A	A17517A	A17117A
Ammonia–2	waters, solids, nesslerization	Prepare a 0.13 M boric acid solution by dissolving 8.0 g boric acid in 995.0 g deionized water. Add 2.0 mL or 2.0 g of this boric acid solution to each tube.	Not Available	A17017A	A17517A	A17117A

Table 3 Micro Dist Methods Summary

Micro Dist Method No.	Matrix and Chemistry	Collector Tube Trapping Solution Recipe	Assembled Pkg/21	User-Fill Pkg/10	User-Fill Pkg/50	User-Fill Pkg/100
Tritium–1	contaminated waters, solids	No trapping solution.	A17014 (pkg of 10)	Not Available	Not Available	Not Available

Table 4 Related QuikChem Methods

Micro Dist Method Number	Related QuikChem Method
Cyanide–1	10–204–00–1–X; 10-204-00-1-X2
Cyanide–2	10–204–00–1–W
Cyanide–3	10–204–00–1–W
Cyanide–5	10–204–00–1–X
Phenolics–1	10–210–00–1–X
Sulfide–1 (Acid Soluble Sulfides)	none
Sulfide–2	10–116–29–1–X
Ammonia–1	10–107–06–1–X
Ammonia–2	none
Tritium–1	none

Appendix B Troubleshooting

B.1 Common Problems, Likely Causes, and Possible Solutions

Very low or erratic recoveries
The most common cause of very low or erratic recoveries is that the tubes are not being pushed all the way into the block. It is not enough to simply let them drop into the holes. The tubes must be pushed in until the stop ring on the collector tube hits the block surface!

This problem can be also caused by over-preserved samples. For Cyanide–1, samples are supposed to either be distilled within 24 h and thus preserved by Micro Distillation or raised to a pH greater than 12 with very little NaOH. The resulting concentration of NaOH is about 0.05 M. Addition of too much NaOH to a sample will prevent the releasing acid from acidifying the sample enough to release HCN (g). Solution: Either do not preserve samples and distill them within 24 h of collection to preserve the HCN in them, or lower the pH with sulfuric acid before running. The second one is hard to do if the samples are over-preserved as one loses HCN this way. Over-preservation really ruins the sample.

This problem could also be caused by not pulling off the sample tubes immediately after removing the collector/sample tube assembly from the block. “Immediately” is pulling the assembly from the hot block within 2–3 seconds. If one lets the assembly cool for much longer, a dramatic suck-back will be seen. A partial suck-back is also possible and will cause low recoveries. A firm rocking/twisting and pulling down action along with a little practice does it. It's all in the wrist!

The sample volume must be 6.0 mL. A pipet must be used and it should be calibrated. If the sample volume is much higher, there will not be enough air left in the sample tube during distillation and suck-back can occur unnoticed.

For method Cyanide–1, the 0.75 mL of releasing acid must be added from the re-pipettor which should have been calibrated by dispensing water and weighing it per the instructions on page 7. If the re-pipettor is set by the marks on the top, it will probably be very inaccurate. If it is much greater than 0.75 mL, unnoticed suck-back can occur. This is due to lack of make-up air in the sample tube.

For method Cyanide–1, the method of determination which follows the distillation must anticipate both 6.0 mL samples and 0.25 M NaOH. For example, when using USEPA Method 335.2 and scaling down the reagent quantities, but the solution is diluted to 100 mL as in Section 8.7.1, color development will not occur. See the scaled-down version of this method on page 23 for an example.

Some “auto-analyzer” methods anticipate a different concentration of NaOH. If the undistilled standards used in these methods are not made up in 0.25 M NaOH, then there will be a matrix effect when the Micro Distilled samples, which are always in 0.25 M NaOH, are determined. If the method anticipates a lower concentration, the samples will give low recoveries; if the method anticipates a higher NaOH concentration, the samples will give high recoveries.

Large blanks in cyanide
Large blanks in cyanide are probably caused by contaminated $MgCl_2$. Always use 99.9999% pure or other ultra-pure grade. A good grade (better than A.C.S.) of sulfuric acid also helps.

Bumping (violent boiling) in the sample tube
This may sometimes actually push the membrane and ring up in the collector tube.

Add one Hengar granule or other small (ca. 1 mm diameter) boiling stone to the sample tube.

Results from Micro Dist™ do not agree with other methods
Try calibrating the analytical method with Micro Distilled standards. If the recoveries improve for reference samples, then use Micro Distilled standards. Remember that analytical methods which follow the distillation must be scaled down in proportion to the smaller Micro Distilled sample.

Block takes hours to come up to temperature
The red light goes out on the block when it's at temperature. If it takes more than 30–40 minutes, contact the Lachat Technical Support Department.

Can't break the collector tube or solution spills all over when they break
Purchase a pair of pruning shears or poultry scissors and cut the collector tube at the break point.

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 Micro Dist manual.

Calculating the amount of sample in mg/kg after analysis:

Multiply the determined concentration in mg/L by (1 L / l000 mL) * dilution vol. in tube = mg analyte in sample.

Divide by g of original sample * 1000 g / kg to give 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.