

On-Site Sample Analyses with Portable Lab

Working procedure

Introduction:

This working procedure has been written in order to evaluate the 7 parameters: total nitrogen (TN), ammonia (NH₄), nitrate (NO₃), nitrite (NO₂), total phosphate (TP), ortho-phosphate (PO₄), COD. It had been used to evaluate 3 type of wastewater in Egypt: septage, sewage, animal liquid manure.

This report gathers the different working procedure for the analysis of these parameters. In the procedure the “*step + number*” refer to the procedure on test box.

General remarks:

- Maximum for 6 samples and only 4 for LCK138 test
- Some parameters have to be evaluated on site: NH₄, NO₃, PO₄
- TP, TN, NO₃ and COD can be evaluated later, ideally the same day. Samples have to be cooled down to 4°C and left to warm up at room temperature before analysis
- Nitrate and nitrite can't be evaluated for manure. The samples are too turbid and the concentration is too low to allow dilution while staying in the range.

Dissolved Oxygen, pH and conductivity have also been analysed. But, because of the easiness of the used we didn't wrote the procedure.

Devices used:

- Photometer: DR2800 from HACH-LANGE
- Thermostat: LC200 from HACH-LANGE
- Homogenizer

Test used (all from HACH-LANGE):

- LCKtest: NH₄(LCK304), TN(LCK138), TP(LCK349) and COD(LCK504)
- Powder pillows: NO₂(NitriVer3, method 8507), NO₃(NitroVer5, method 8171) and PO₄(PosphVer3, method 8048)

Material needed:

- 4x400ml Becker: for the sample
- 2x250ml Becker: used as bin
- 4x100ml Becker: for the First filtration using 0.7µm or 0.8µm filter paper.
- 4x50ml Becker: for the second filtration using 0.45µm filter paper.
- 12x50ml graduated cylinder: for dilutions
- 4x25ml graduated cylinder: for dilutions
- 6 test tubes: for powder pillow test
- 2 cuvettes: for powder pillow test
- Other: Pipette 1 and 5ml, pipette tips, pens, alcohol, gloves, disinfectant, filter holder and filter paper (at least 0.45µm), distilled water
- Cool/ice box to preserve the samples at 4 degrees.

Basics remarks:

- Always note the number of the sample, the filtration, and the dilution on the vial, Becker etc.
- When taking the solution for a non-filtrated, always mix the sample.

PREPARATION																																		
	Time	Activity	Material																															
Preparation	0 - 40min	<ul style="list-style-type: none"> - Install the homogenizer and the DR. - Install and heat the thermostat to 148 degrees in the right side and 100 degrees in the left side (just for second evaluation) - Put each sample in a 400ml becker - Homogenize all samples during 1min - Make the Quicktest of NH4, PO4, NO3 and NO2 - Compute the dilution and the volume needed. <ul style="list-style-type: none"> o LCK: 5ml needed o Powder pillow: 20ml needed 	2*250ml Becker x 400ml Becker x 50ml Becker (x 100ml Becker) 50ml graduated cylinder 25ml graduated cylinder																															
		<p style="text-align: center;">EXPECTED DILUTION</p> <table border="1" style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th></th> <th>NH4</th> <th>PO4</th> <th>NO3</th> <th>TP</th> <th>TN</th> <th>COD</th> <th>NO2</th> </tr> </thead> <tbody> <tr> <td>Sewage</td> <td>100</td> <td>50</td> <td>1</td> <td>10</td> <td>50</td> <td>1</td> <td>1</td> </tr> <tr> <td>Septage</td> <td>50-500</td> <td>50</td> <td>1</td> <td>20 -100</td> <td>100</td> <td>2-10</td> <td>1</td> </tr> <tr> <td>Manure</td> <td>2500</td> <td>50</td> <td>lab</td> <td>50</td> <td>500</td> <td>10-20</td> <td>lab</td> </tr> </tbody> </table> <ul style="list-style-type: none"> - Filtrate the sample by 0.45µm. <i>Filtration is needed for NH4, PO4, NO3 and NO2. For manure and septage it is recommended to pre-filter with 0.7µm or 0.8µm micron filter paper.</i> - Make the appropriate dilution for filtrated and non-filtrated solution 		NH4	PO4	NO3	TP	TN	COD	NO2	Sewage	100	50	1	10	50	1	1	Septage	50-500	50	1	20 -100	100	2-10	1	Manure	2500	50	lab	50	500	10-20	lab
	NH4	PO4	NO3	TP	TN	COD	NO2																											
Sewage	100	50	1	10	50	1	1																											
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ON-SITE EVALUATION			
	Time	Activity	Material
NH4	0min	Put the LCK304 on the support <ol style="list-style-type: none"> 1) LCK304: Step 1-5, <i>step2:keep the stopper on the same side</i> <i>step3-4: be quick</i> 2) Alarm 1: 15min 	x LCK304 vial
	20min	Alarm 1 ring: Measure LCK304	
PO4	15min	Phosphorus: <ol style="list-style-type: none"> 1) Step 1-8 (need to wait 2min) 2) Rinse the two cells 3) Repeat for each sample 	2 sample cells x PhosVer3
NO3	30min	Nitrate: <ol style="list-style-type: none"> 1) Step 1-10 (need to wait 5min) 2) Rinse the two cells 3) Repeat for each sample 	2 sample cells x NitraVer5

SECOND EVALUATION			
	Time	Activity	Material
COD	0min	Put the LCK514 vials on the support. 1) LCK 514: step 1-3, then 2h at 148°C on the left side of thermostat	x LCK514 vial
TP, TN	15min	Put the LCK349 vials and x dry test tubes on the support. 2) LCK349: step 1-4 3) LCK138: step1 <i>step 1: close immediately, do not invert.</i> 4) LCK349 & LCK138 during 1h at 100°C on the right side of thermostat Be quick	x LCK349 vial x LCK138 dry test tube LCK138 prod. A and B
NO2	40min	Nitrite: 1) Step 1-8 (need to wait 20min) 2) Rinse the two cells 3) Set alarm Note: if more than 1 sample to analyze, do the first sample in the 2 cells, and during the break time begin another sample every 5 min in test tube. Transfer the solution from the test tube to the cells to evaluate.	2 sample cells x NitriVer3 x 10ml test tube (with stopper)
	1h30	Stop the right side of thermostat 1) Take out the vials (LCK138, LCK349) 2) Wait until they are cold.	
TN	1h50	1) LCK138: step 3-8 <i>step4: mix until no streaks can be seen</i> <i>step5-6: pipette slowly, stopper quickly</i> <i>step 7: mix until no streaks can be seen</i> 2) Alarm 1: 15 minutes	x LCK138 LCK138 prod. C and D LCK349 prod. B and C
TP	2h00	1) LCK349: mix it and then step 6-8, <i>step 6,7 :close with C stopper quickly</i> 2) Alarm 2: 10 minutes	
	2h10	Alarm 1 ring: LCK138, clean the outside and evaluate Alarm 2 ring: LCK349, invert a few time, clean the outside and evaluate	
COD	2h10	Stop the left side of thermostat 1) LCK 514: Mix the vials 2) LCK 514: Wait until it's cold 3) LCK 514: Clean the outside and evaluate	
		Clean and rinse all the lab material	