Joint verification in practice: An example



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Head of DANETV



WATER • ENVIRONMENT • HEALTH



HACH-LANGE is supplier for water analysis:

- Wastewater, drinking water, and process water
- On-site and laboratory analysis, process analysis, and samplers
- Municipal and industrial applications

HACH-LANGE got their ETV interest by getting the following message:

There is a good opportunity to sell X amount of a portable luminescent bacteria testing system in country Y. The customer is a national authority. Within the specifications required they asked for a system according to ISO 11348 and USEPA ETV verification statement.

# The principle of luminescent bacteria test





# Bioluminescence as indicator of toxicity

Luminescent bacteria, e.g. Vibrio fisheri NRRL B-11177, naturally emit light, called bioluminescence.

Light can easily be measured by a luminometer.

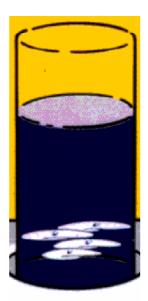
Results from tests are % inhibition or EC-values (EC<sub>20</sub>, EC<sub>50</sub>, EC<sub>80</sub>)



#### When toxins attack

Bioluminescence is directly linked to the physiology of the bacteria.

A toxic compound will change or damage this system so the bacteria will rapidly lose their ability to emit light.





## LUMIStox, lab system in accordance with ISO11348



The national authority put ETV verification statement as a requirement in the tender.

Competitive systems were part of a US driven ETV program regarding rapid toxicity testing done in 2003 and 2006.

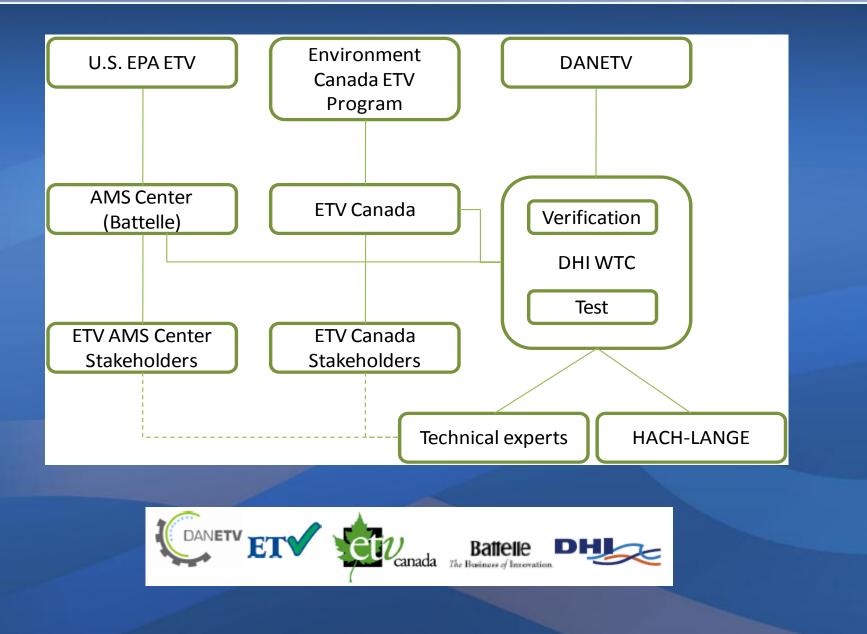
Unknown why LUMIStox was not included. ECLOX is recently developed.



ECLOX, portable system

## Organisation of joint verification







- Quick scan (ETV screening)
- Contract
- Process document for joint verification
- Verification protocol
- Test plan

#### TESTING

- Test report
- Verification report
- Verification statements

Performance parameters
Test design



What would a potential customer like to know?

- Minimum level of detection (criterion of detection)
- Range of application
- Precision
- Repeatability Reproducibility Trueness (agreement with accepted values) Robustness

Relevant ranges were identified for all performance parameters



### Example of tests

- TEST A: Test of dilution series for 6 compounds. 3 test replicates (include 2 measurement replicates each)..

Range, Repeatability, and Agreement with trusted values

 TEST D: Test of dilution series for 1 compound. 3 test replicates (include 2 measurement replicates each). Repeated on 3 different days with 3 different bacteria batches.

#### Reproducibility

TEST G: One concentration (~ EC<sub>20</sub>) for 1 compound. For pH 6.0, 7.0 and 8.5, 3 test replicates (include 2 measurement replicates each).
 *Robustness (pH)*



# Parameters evaluated and compounds used:

Temperature in thermoblock	pH in sample	Color correction	Cuvette material	Water types	Compounds
14.0 – 16.1ºC	6.0 - 8.5	Colored samples Turbid samples	Plastic Glass	2% NaCl MilliQ water Domestic wastewater Industrial wastewater	Metal ions Organic pesticide Inorganic pollutant Surfactants (6 compounds)



LUMIStox	Criterion of detection	Range of application	Precision		Agreement with accepted values	Robustness		
Compound			Repeat- ability	Reproduc- ibility		pH, color, turbidity, laboratory temperature <sup>1)</sup>	Cuvette material	Waste- water matrix <sup>1)</sup>
	% inhibition	mg/L	%	%	%	%	%	%
General	5.8							
Zn <sup>2+</sup>		8.3 -130	5.0	30	186		106 -117	43 -123
Cr <sub>2</sub> O <sub>7</sub> <sup>2-</sup>		35 - 560	29		91			0 -22
Triclosan		1.1 - 17	5.5		189			96 -141
Cyanide		48 - 780	24					
SDS		2.0 - 32	33			71 - 114	90 - 101	28 - 96
CTAB		1.9 - 31	2.4		100			68 - 78

<sup>1)</sup>For colored samples, robustness results are presented after the use of color correction. For the BaSO<sub>4</sub>-turbid samples, robustness results are presented <u>without</u> the use of color correction. For domestic wastewater, adjustment was made to account for the negative inhibition from the wastewater; if color correction was used the robustness was -20% to 70%. The values listed in table are the best achievable robustness.

# Quality assurance



	DHI	Battelle AMS Center	U.S. EPA ETV	ETV Canada	Environment Canada	Expert Group
Verification protocol and test plan	Review	Review	Review	Review	-	Review
Test system	Audit	Audit	-	-	-	-
Test report	Review	-	-	Review	-	Review
Verification report	Review	Audit/Review	Review	Review	Review	Review

## Verification statement



## The statement contains:

- Technology and product description
- Verification and test description, incl. QA
- Verification results
  - Performance parameters
  - Evaluation of user manual
  - Health and safety issues
  - Purchase price
  - Operation and maintenance cost



#### **ETV** Joint Verification Statement

TECHNOLOGY TYPE: Bench top luminometer				
APPLICATION:	Toxicity testing of wastewater effluent.			
PRODUCT NAME:	LUMIStox 300 Bench Top Luminometer with LUMIStherm Thermoblock and LUMISsoft4 Software			
COMPANY:	HACH-LANGE GmbH			
ADDRESS:	Willstätterstrasse 11 D-40549 Düsseldorf, Germany	PHONE: +49 211 5288 0		
WEB SITE:	www.hach-lange.de			
E-MAIL:	elmar.grabert@hach-lange.de			

Testing and verification of a bench top luminometer for detecting toxicity of wastewater effluent was conducted as a joint verification project with the Danish Centre for Verification of Climate and Environmental Technologies (DANETV), ETV Canada, and the United States Environmental Protection Agency (U.S. EPA) Environmental Technology Verification Program (ETV). The testing and verification satisfied the requirements of the Canadian ETV program, U.S. ETV program and the ETV scheme currently being established by the European Union (EU ETV).

Environment Canada established the Canadian ETV program to provide credible information for promoting the commercialization and market deployment of new environmental technologies, thus helping to address environmental challenges efficiently, effectively and economically. Information on the Canadian ETV program is available at www.etvcanada.ca. luent 1ate and al e testing n and

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- Meets tender requirement of U.S. EPA ETV
- Intensive third-party test of their products, new knowledge
- Easy for customers to compare the two products
- Has opportunity to get EU ETV (when programme starts in operation)
- If one of the products is redesigned or replaced, ETV can be reevaluated under EU ETV

# DANETV lessons learned

# Joint

#### Second DHI joint verification

- Good cooperation and knowledge sharing between ETV-operators
- Input to joint and coverification roadmaps
- US EPA ETV require both review and approval => extended time schedule
- DHI working methods evaluated by US EPA and Environment Canada ETV programmes => extended trust for next joint or coverification

# ETV

Third DHI DANETV verification

- Improved DHI know-how on performance of verifications
  - Reference system important to establish and verify early. If not possible, leave reference system out and decide for alternative QA
  - Test design must ensure statistical significance of data sets (something might have to be left out during testing)
- Practical experience has been used for update of and input to
  - DANETV QA Manual
  - EU ETV General Verification
     Protocol





# DHJ

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