

QUALITY ASSURANCE IN BREATH-ALCOHOL ANALYSIS

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Articolul prezintă aspecte teoretice referitoare la analiza alcoolului din aerul alveolar expirat și la asigurarea trasabilității unității de măsură specifice. Lucrarea prezintă diferite metode fizico-chimice utilizate în analizele legale de alcool etilic din aerul expirat. Asigurarea calității a devenit un instrument indispensabil analizelor judiciare de alcool din aerul expirat. Astfel, pentru a asigura trasabilitatea măsurărilor concentrației de alcool, Laboratorul Concentrație Gaze din cadrul Institutului Național de Metrologie a pregătit un astfel de program de asigurarea calității și utilizează un set de proceduri proiectate pentru identificarea, controlul și monitorizarea tuturor factorilor majori care pot afecta procesul de testare și care să demonstreze în același timp, faptul că rezultatele obținute sunt precise și adevărate. În funcție de diferitele surse de incertitudine este prezentată o estimare a incertitudinii concentrației de alcool generată pentru etalonarea analizoarelor evidențiale de alcool din aerul alveolar expirat.

The article is intended to present theoretical aspects related to breath alcohol analysis and traceability of alcohol concentration. There are presented different physico-chemical methods which are used in legal breath alcohol analysis. Quality assurance has become an indispensable accompaniment to forensic breath-alcohol analysis. Thus, in order to assure traceability of breath alcohol analysers, Gas Concentration Laboratory from National Institute of Metrology have prepared such quality program and uses a set of control procedures designed to identify, control and monitor all major factors that can affect the testing process, and to prove that the results obtained are reliable and true. Depending on different sources of uncertainty is presented an estimation of uncertainty regarding the generated alcohol concentration used for evidential breath analyzers calibration.

Introduction

This article is intended to present details of implementation of quality assurance in Breath-Alcohol Analysis performed by National Institute of Metrology.

Evidential breath analyzers are instruments that automatically measure the mass concentration of alcohol in exhaled breath that originates from the alveoli of the lungs.

The breath-alcohol testing methods have changed over the years from chemical oxidation and calorimetric procedures to physico-chemical techniques

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such as gas-chromatography, electrochemical oxidation and multiple wave length infrared spectrophotometers.

Although the relationship between the breath and blood alcohol concentration is still uncertain, the evidential breath-alcohol instruments are used in different countries for determination alcohol concentration level for forensic purposes.

National authorities may require specific conversion device that converts the measurement result obtained in terms of ethanol content and can approve evidential breath analyzers for law enforcement purposes with threshold limit of breath-alcohol concentration alongside the existing statutory blood-alcohol concentration limits.

QA has become an indispensable accompaniment to forensic breath-alcohol analysis.

Gas Concentration Laboratory from National Institute of Metrology is prepared to provide following control procedures: pattern approval, initial verification of new EBA, periodic verification, performance test and calibrations, and focuses on development, implementation and use of such QA programs for breath-alcohol testing.

Evidential breath analyzer is an instrument which measures accurately the concentration of alcohol in “end-expiratory” air to provide a result which can be used as evidence in drinking and driving offences. End-expiratory air is a breath sample containing air from the end of a forced expiration from lungs.

The concept of quality assurance (QA) is intuitively recognized and accepted by most persons engaged in providing products or services as necessary to assure that the latter meet defined standards. As applied to breath-alcohol analysis, QA is comprehensive program of activities designed and intended systematically to identify, control and monitor all major factors that can affect the testing process, and to prove that the results obtained are reliable and true. The quality assurance concept as applied to measurements and other laboratory activities, of course, evolved and was adapted from its original application to quality control of manufactured products. Quality control and quality assessment are elements of the overall QA program.

Under the impetus of vigorous legal challenges to forensic alcohol analysis and the testing of drivers under influence in particular, a massive body of appellate case law has arisen on both legal and technical issues of alcohol testing. This has also stimulated development and use of increasingly comprehensive and sophisticated QA practices in forensic alcohol testing. An evolving body of appellate case law on QA issues has focused on certain judicial requirements for admissibility of alcohol test result as evidence.

1. Components and elements of a QA program for breath alcohol analysis

In designing an adequate QA program, it is useful to recognize the most common problems currently causing operational difficulties in forensic breath alcohol testing programs and underlying successful legal challenges of breath-alcohol analysis results. Many of these components are encompassed within Good Laboratory Practice standards for laboratories or in current regulations or guidelines for forensic blood testing.

The most common problems and lapses in forensic breath alcohol-analysis are: inadequate rules and regulations, lack of a comprehensive quality assurance program, failure to test replicate breath specimens, lack of periodic personnel retraining.

A comprehensive QA program must address all relevant pre-analytical, analytical and post-analytical factors. It should thus cover – test subject preparations and other preparations, - the analysis process, - test result reporting and records, - performance and proficiency testing, inspections and evaluations, - facilities and personnel aspect.

Quality control constitutes a system, of activities, techniques and procedures to promote, protect and assure the validity and reliability, to a stated level of confidence, of the measurement process and its output – breath alcohol results. The most recognized components of quality control are the necessary scientific safeguard, which have successfully withstood adversarial challenges in the judiciary system.

One important element of quality assurance in forensic breath alcohol analysis is the testing process which comprises: operating procedures, subject preparation (elimination of foreign object or substances from mouth exclusion of eructation, regurgitation), operational safeguard (purging of analyzer, blank analysis before and after each breath specimen, analysis of duplicate breath specimens, printout of test result, control tests), records and reports, inspections and evaluations.

Tests required to assess the performance of an evidential breath analyzers in accordance with the recommendations of the Organisation Internationale de Metrologie Legale (OIML). It outlines the laboratory procedure for assessment of the analytical performance of the instrument in relation to the measurement of vapor samples containing ethanol and specified amounts of possible interfering substances, which may be encountered in the course of breath testing.

2. Adjustment or verification to a standard

Adjusting the instrument is possible to be done using (a) a standard ethanol/air mixture having a relative humidity of at least 90 %, a temperature of

($34 \pm 0,5$) °C and carbon dioxide concentration of (5 ± 1) %, or (b) a dry ethanol/air mixture.

2.1 Verification of correct calibration

Verification of the correct instrument calibration shall be carried out during the measuring cycle before and after supply of breath alcohol specimens for analysis:

- purge and check zero;
- verify calibration by simulation (using an ethanol/air mixture at 35 µg/100ml concentration)
- purge and check zero;
- take an analyze specimen 1;
- purge and check zero;
- take an analyze specimen 2;
- purge and check zero;
- verify calibration by simulation (using an ethanol/air mixture at 35 µg/100ml concentration)
- print out readings and purge.

3.2 Verification of correct operation / calibration of the instrument comprises verification of:

- maximum permissible errors on each indication;
- accuracy/repeatability (standard deviation of a set of readings);
- drift (short term drift, long term drift);
- hysteresis and memory effect;
- effect of delivered volume;
- effect of duration of sample;
- effect of variation in carbon dioxide concentration;
- effect of interrupting sample flow;
- effect of the presence of alcohol in the upper respiratory tract.

3. Breath-alcohol simulators

Control tests in breath-alcohol analysis are performed mainly with breath-alcohol simulators, which are devices for the preparation and delivery of vapor specimens of known alcohol concentration, at a fixed temperature. The resultant vapor effluent has a predictable and controllable alcohol concentration and appropriately simulates alcohol-containing breath for use in calibrating analyzers, control tests and analyst training. Simulators are critically dependent upon

properly prepared and validated alcohol reference solutions for producing vapor-alcohol effluents of specified, known alcohol concentration.

Simulator solutions of known alcohol concentration used for calibrating breath-alcohol analyzers are critically dependent upon the characteristic and functioning of the simulator used, especially the accuracy and stability of the intended equilibration temperature, which is usually 34 °C.

The OIML recommendation R126 for Evidential Breath Analyzers sets high standards for calibrating evidential breath alcohol analyzers. It requires a sample of 3 l with a defined gas temperature of $(34 \pm 0,5)$ °C and a relative humidity of at least 95 %.

Wet bath simulators have been used for more than 4 decades for calibrating breath alcohol analyzers as they deliver a gas sample similar to human breath with regard to temperature and relative humidity. As the alcohol concentration in the liquid is depleted due to evaporation losses the solution has to be changed after a certain number of tests. This restricted their use in automated calibration system. Using a single liquid simulator with a water volume of 500 ml allows the delivery of only 10 to 15 l of test gas, until a 1 % depletion is reached. As this is not acceptable for precision calibration purposes, OIML recommends the use of a “bubble train” of at least two cascaded wash bottles, which are placed in a water bath of defined temperature.

Replacing a certain amount of the simulator solution can also do the compensation of the evaporation losses of ethanol in the liquid phase. The amount of fresh solution supplied after each test is depending on volume of generated gas. Based on this technique Dräger produced a new wet bath simulator system – ALCOCAL which has been approved by the German National Institute of Standards (PTB) in Braunschweig. The Alcocal is equipped with a micro-controller, which checks all functions of the instrument. Tests with the Dräger Alcocal have shown that the calibrator offers good long-term stability and repeatability of generated alcohol concentration. The high temperature stability allows a dynamic calibration of the temperature sensors of the breath analyzers.

Alcohol mixture in inert gases, such as argon or nitrogen, stored under high pressure in disposable cans, is another form of alcohol standard for calibrating and control test purposes.

4. Estimation of uncertainty of measurements related to use of ethylometer calibration

4.1. Uncertainty concerning the concentration of the hydroalcoholic Solution

Principle of measurement

The solution are prepared by ethanol weighing (Merk's absolute ethanol, molar density: 46,07 g/mol; density: 789 kg/m³, purity > 99,8 %) put into a

solution of permuted water obtained by ion exchange in a resin column. The volume is determined by means of a calibrated phial.

The concentration of ethanol in solution corresponds to the following equation:

$$C = \frac{M}{V} \quad (4.1.1)$$

C – ethanol concentration (g/l);

M – mass of ethanol weighed and corrected (g);

V – volume of gauged phial (l).

The propagation law of the uncertainties is expressed as follows:

$$u_c^2(C) = \left(\frac{\partial C}{\partial V} \right)^2 u^2(V) + \left(\frac{\partial C}{\partial M} \right)^2 u^2(M) \quad (4.1.2)$$

Measurement V and M are independent and the terms of covariance are considered as zero.

$$u_c^2(C) = \left(\frac{1}{V} \right)^2 u^2(M) + \left(\frac{M}{V^2} \right)^2 u^2(V) \quad (4.1.3)$$

Therefore, is necessary to evaluate two terms (uncertainty concerning the mass of weighed ethanol and the volume of the phial).

4. 2 Uncertainty concerning the mass of weighed ethanol

To eliminate any defects in balance accuracy, it is calibrated immediately before weighing take place. The purpose of calibration, in this case, is to determine an accuracy error which will be used to estimate than accuracy correction to be applied to the readings of the balance.

The mass of alcohol is therefore:

$$M = M_d + C_p + C_e \quad (4.2.1)$$

M_d – direct reading balance (display in unit of mass) for which the accuracy error is determined at the time of weighing a standard conforming to OIML recommendation 111.

C_p – correction due to purity of ethanol;

C_e – correction due to evaporation of ethanol;

The uncertainty concerning this mass results from three components: uncertainty regarding the weighed mass, uncertainty regarding the purity of the alcohol and uncertainty regarding evaporation.

The corresponding variance is:

$$u^2(M) = u^2(M_d) + u^2(C_p) + u^2(C_e) \quad (4.2.2)$$

$u(C_p)$ – uncertainty of correction due to purity of ethanol; right angle triangle asymmetrical distribution;

$u(C_e)$ – uncertainty of correction due to evaporation of ethanol, right angle triangle asymmetrical distribution;

4.3 Uncertainty regarding the value of calibrated phial volume

The uncertainty regarding the volume of water therefore results from three components (volume of calibrated phial, phial dilatation and dilatation of water). The uncertainty regarding the volume of the calibrated phial is estimated from its class $\pm 0,25$ ml for a 1 liter phial. The phial was verified by the weighing of distilled water.

Considering a rectangular distribution, the variance regarding the knowledge of the phial volume can be estimated as:

$$u^2(V_f) = \frac{(0,25 \cdot 10^{-3})^2}{3} \quad (4.3.1)$$

The corresponding variance regarding dilatation of the phial (which factor is $1 \cdot 10^{-5}$ per $^{\circ}\text{C}$) will be estimated by considering a distribution in the form of a sine arc derivative. This gives a variance of:

$$u^2(V_d) = \frac{(2 \cdot 10^{-5} \cdot V)^2}{2} \quad (4.3.2)$$

The volume of water equals the volume supplied by the phial, except for the uncertainty regarding knowledge of the supplied volume of water to which is added uncertainty regarding the knowledge of the volume related to its dilatation. Water has a volume factor of $2 \cdot 10^{-4} \text{ } ^{\circ}\text{C}^{-1}$.

Considering that the laboratory temperature is $(20 \pm 2) ^{\circ}\text{C}$, the corresponding variance is estimated considering a distribution in the form of a sine arc derivative giving variance of:

$$u^2(V_e) = \frac{(2 \cdot 2 \cdot 10^{-4} \cdot V)^2}{2} \quad (4.3.3)$$

4.4 Uncertainty concerning volume

The corresponding variance regarding the volume of water is:

$$u^2(V) = u^2(V_f) + u^2(V_d) + u^2(V_e) \quad (4.4.1)$$

When the ethanol is put into a solution, alcohol is poured into a large quantity of water so as to stop the evaporation phenomenon. The solution are preserved in hermetically sealed recipients and a mixture of the vapor and liquid phases is carried out before each use. Laboratory tests have not shown any drifting of the concentration during the period of use of the solution.

The propagation law of the uncertainties is expressed as follows:

$$u_c^2(C) = \left(\frac{1}{V}\right)^2 u^2(M) + \left(\frac{M}{V^2}\right)^2 u^2(V) \quad (4.4.2)$$

Tabel 1

Uncertainty in determining C			
Component	Standard uncertainty on parameter	Sensitivity coefficient	$u_i(C)$
Density of weighed alcohol M	$u(M)$	$\left(\frac{1}{V}\right)$	$\left(\frac{1}{V}\right)u(M)$
Volume of water V	$u(V)$	$\left(\frac{M}{V^2}\right)$	$\left(\frac{M}{V^2}\right)u(V)$
Combined standard uncertainty			$u(C) = \sqrt{\sum u_i^2}$
Expanded uncertainty			$U = k \cdot u(C)$

4.5. Uncertainty concerning concentration of bubbling through air

Measurement principle

The Dräger Alcocal used for calibrating evidential breath alcohol analyzers is based on the enrichment of a carrier gas with ethanol vapor when passing through an ethanol-in-water solution. Two cylindrical containers are coaxially arranged and contain the ethanol in water solution. The air coming from the mass flow controller passes firstly the outer, then the inner container and is enriched with ethanol vapor according to the actual concentration and the temperature of the liquid. Depending on the gas volume delivered a certain amount of fresh ethanol in water solution is pumped automatically into the inner container, and a respective amount from the inner to the outer container and then to the solution outlet. This depletion compensation of the ethanol-in-water solution together with the very elaborate temperature control ensures the specific high precision and stability of the ethanol gas concentration delivered by Dräger Alcocal.

The alcohol concentration of the air at the outlet is calculated by the Dubovski formula:

$$C_{air} = C_{water} \cdot K_1 \cdot e^{k_2 \cdot t} \quad (4.5.1)$$

C_{air} – concentration of ethanol in air (mg/l);

C_{water} – concentration in ethanol of solution (g/l);

K_1 – constant = 0,04145;

K_2 – constant = 0,06583;

t – temperature, ($^{\circ}\text{C}$)

This equation established by Professor Kurt Dubowski (Department of Medicine and Toxicology in the University of Oklahoma) stipulate that for a solution of a product A and a product B, we have:

$$p_A = x_A \cdot p_A^0 \quad \text{and} \quad p_B = x_B \cdot p_B^0 \quad (4.5.2)$$

p – partial pressure of component with solution at balance;

x – molar fraction of component;

p^0 – vapor pressure of pure component at the same temperature.

The partial vapor pressure giving the concentration in the air and the molar fraction with the concentration of ethanol in water, we have:

$$\frac{\text{Concentration of ethanol in gas phase}}{\text{Concentration of ethanol in liquid phase}} = \text{const.} \quad (4.5.3)$$

Experimental determination of coefficient of the air/water partition for ethanol led to the following result:

$$\frac{C_{air}(mg/l)}{C_{water}(g/l)} = 0,04145 \cdot e^{0,06583 \cdot t} \quad (4.5.4)$$

4.6 Uncertainty regarding concentration in air

The concentration in air, calculated by Dubowski's equation from the concentration in the water phase and that of temperature is considered as a conventionally true value. Therefore, no estimation of uncertainty will be made for the give factors K_1 and K_2 (0,04145 and 0,06583).

Therefore the expression can be written:

$$C_{air} = C_{water} \cdot 0,04145 \cdot e^{0,06583 \cdot t} \quad (5.6.1)$$

Calculation of variance for the corresponding to the following formula:

$$u_c^2(C_{air}) = \left(\frac{\partial C_{air}}{\partial C_{water}} \right)^2 u^2(C_{water}) + \left(\frac{\partial C_{air}}{\partial t} \right)^2 u^2(t) + 2 \left(\frac{\partial C_{air}}{\partial C_{water}} \right) \left(\frac{\partial C_{air}}{\partial t} \right) u^2(C_{water}, t) \quad (5.6.2)$$

It is accepted that the uncertainty regarding the concentration of the solution and that of the temperature measurement are totally independent. Therefore, the covariance term can be considered equal to 0.

Conclusions

Breath alcohol analyzers are widely accepted as legal measurement instruments used for determination of the mass concentration of alcohol in exhaled breath. Nowadays, Traffic Road Department from Romanian Ministry of Interior is using about one thousand and few hundred of electronic devices for

testing breath alcohol concentration. The specialists from Gas Concentration Laboratory from National Institute of Metrology are prepared to assure traceability of alcohol concentration according to the latest standards and to sustain research activity in this new field.

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