Introduction

An important part of the UNODC International Quality Assurance Programme (IQAP) is the implementation of the International Collaborative Exercises (ICE). Participation in such exercises, inter-laboratory comparisons or proficiency tests is one of the essential elements for the implementation of a laboratory quality management system and ultimately accreditation. This is recognised by the International Organization for Standardization in ISO/IEC 17025-2005: “General requirements for the competence of testing and calibration laboratories” as contributing to assuring the quality of test results.

The UNODC ICE programme allows drug testing laboratories from both developing and developed countries to continuously monitor their performance on a global scale. The options available for participation are in the analysis of drugs in Seized Materials (SM) and in Biological Specimens (BS, specifically urine). Two rounds are offered per year with each round presenting participants with four different test samples for analysis in each test group.

Laboratories participating in the ICE programme can use an online portal for direct submission of results to UNODC. This enables participants to receive immediate confidential feedback from UNODC on their performance and greatly facilitates the implementation of the programme.

Upon completion of each ICE round, the analytical results are evaluated by UNODC and an International Panel of Forensic Science Experts which oversees the implementation of ICE and offers guidance and support in addressing relevant quality issues.

Following evaluation, summary reports of the performance of participating laboratories in both the SM and BS test groups are made available to participants through the ICE portal and the UNODC website. These summary reports allow participants to evaluate their performance while maintaining confidentiality.

Participation

The number of laboratories worldwide who participate in the ICE programme has continued to increase in recent years and there are now 183 laboratories from 59 Member States actively participating in the programme, representing an 17% increase in year on year participation. Figure 2 shows the participation of laboratories in the SM and BS test groups for all ICE rounds from 2012/1 up to the most recent 2014/1 round of the programme.

The continued increase in participation during 2014 is a result of the greater recognition globally of the importance of quality assurance and the benefits of participation in the ICE programme. Focused technical assistance was also provided to more laboratories in collaboration with regional forensic networks such as AFSN, AICEF and ENFSI and with support from UNODC staff in regional and country programmes.

It is recognised that some participants continue to have difficulties with obtaining import authorization for the SM test samples and the reference samples for the SM and BS groups and this caused some delays in sending test samples and in the submission of results from a small number of laboratories.
Test samples

Participating laboratories are requested to analyse four test samples in the SM group and/or four test samples in the BS group for the substances listed in the ICE menu. This menu covers the controlled substances, certain new psychoactive substances (NPS) and adulterants most commonly encountered in drug seizures. The ICE menu for the BS test group covers selected drugs of abuse, their metabolites and related compounds.

The composition of test samples within the ICE programme is designed to simulate actual casework encountered by forensic laboratories. In the SM group, the test samples are prepared in the Laboratory and Scientific Section of UNODC using donations of seized materials from Member States, while the BS test samples are prepared using controlled substances, their metabolites and related compounds in urine.

Laboratories are asked to analyse the test samples using the screening and confirmatory tests they routinely employ in casework. These may range from simple techniques such as colour tests and Thin Layer Chromatography (TLC) to more advanced methods such as Gas Chromatography-Mass Spectrometry (GC-MS). By recording the techniques they use, the laboratories are able to assess their performance against that of other laboratories of similar capabilities and to identify any limitations of their performance compared with that of differently equipped laboratories. Indeed, the ICE programme is specifically designed as such to enable participation of laboratories with differing capacities. Participants are requested to identify the substances in the test samples and in addition, are encouraged to report the purity or concentrations of the controlled drugs present in the test samples.

ICE results

Qualitative analysis

The data in Table 1 shows the composition of the SM test samples in rounds 2013/2 and 2014/1 and gives the percentage of laboratories that correctly identified each test sample and the numbers of false positive and false negative results reported for controlled substances. Overall, the results for qualitative analysis within the SM test group in both rounds were excellent, with 93% and above of laboratories correctly identifying the controlled substances in each round.

While the number of false positive and false negative results reported through 2013/2 and 2014/1 was low in most test samples, laboratories who do report false positive or negative results should investigate the reasons for this and corrective actions should be taken in order to continuously improve performance.

The qualitative performance of laboratories in the BS test group in 2013/2 and 2014/1 are shown in Table 2. Within the BS test group, the results were good in both rounds for the majority of test samples, given the inherently higher level of difficulty in the analysis of the low concentrations of drugs in biological specimens and the complexity of the matrix.

2-(4-Bromo-2,5-dimethoxyphenyl)ethanamine (2C-B) was introduced to the ICE menu for the BS test group in 2014 and 50% of participants identified its presence in 2013/2, while 49% identified it in 2014/1. It is notable that in these two ICE rounds, 37% and 42% of participants respectively, did not carry out analyses for 2C-B. GHB, present as a test sample in ICE 2013/2 for the second consecutive time was correctly identified by 29% of participants and 47% of labs did not carry out analysis for this substance which, in March 2013, was transferred from schedule IV to schedule II of the United Nations Convention on Psychotropic Substances, 1971.

Quantitative analysis

Quantification of test samples within the ICE programme is not compulsory, however, laboratories who do so are encouraged to quantify all test samples (depending on jurisdictional requirements) in order to get a better measure of their performance over time. z-scores are a statistical parameter used in proficiency tests and collaborative exercises as a measure of performance in quantitative analysis.

Table 1: Composition of SM test samples, performance of participants and number of false positive and false negative results in ICE 2013/2 and 2014/1.

Table 2: Composition of BS test samples, performance of participants and number of false positive and false negative results in ICE 2013/2 and 2014/1.
Following a recommendation from the International Panel of Forensic Science Experts and in order to allow ICE participants to interpret their z-scores in line with recommendations in ISO 13528:2005 and ISO/IEC guide 43-1:1997(E), UNODC have introduced an revision to the method used to calculate the z-scores (z = (x - μ) / σ), where x = result of participant, X = assigned value of component being quantified and σ is the standard deviation.

One of the methods recommended in ISO 13528:2005 for determining the assigned value X is to use the consensus value from participants. The standard deviation of the results of participants is also used.

Initial estimates of X and σ are considered to give an action signal and a score plot for ICE 2014/1 SM-1 (33.5%)

Using these estimates, an iterative calculation (for details see ISO13528:2005, Annex C) is carried out to determine the final values of the robust average (x̄) and robust standard deviation (s*) and the z-scores are calculated as

\[ z = \frac{x - x^*}{s^*} \]

Consequently z-scores can be interpreted by participants in line with ISO 13528:2005, section 7.4.2 and as follows:

<table>
<thead>
<tr>
<th>z</th>
<th>Interpretation</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 2</td>
<td>satisfactory</td>
</tr>
<tr>
<td>2 ≤</td>
<td>questionable</td>
</tr>
<tr>
<td>&gt; 3</td>
<td>unsatisfactory</td>
</tr>
</tbody>
</table>

According to the recommendations in ISO 13528:2005, an unsatisfactory z-score is considered to give an action signal and a questionable z-score is considered to give a warning signal. A single action signal or warning signal in two successive rounds shall be taken that an anomaly has occurred that requires investigation.

Table 3: Complete list of z-scores for participating laboratories (with their lab code) within the SM test group in 2013/2 and 2014/1. Asterisks indicate that quantification was not performed or that the laboratory did not participate in that particular round of ICE.

![Figure 3: z-score plot for ICE 2014/1 SM-1 (33.5% mCPP). Each Bar represents the z-score of a laboratory who performed quantitation and the lines indicates the level below which z-scores are considered satisfactory, questionable and unsatisfactory.](image-url)
A comprehensive list of the performance of all laboratories that performed quantitative analysis during ICE 2013/2 and 2014/1 is given in Tables 3 and 4 using the revised method of calculation of the z-scores for the results of the 2014-1 round.

This information enables laboratories to compare their individual quantitative performance with all other participants. Laboratories whose results are classified as outliers should investigate the cause and take appropriate corrective action, with support from UNODC, if required.

z-score plots are also provided to all participants in the summary report after each ICE round. Typical plots for ICE 2014/1 SM-1 (33.5% mCPP) and 2014/1 BS-1 (7651ng/ml benzylecgonine) are shown in Figures 3 and 4 respectively. Participants who obtained questionable or unsatisfactory z-scores are highlighted in amber and red respectively.

New Psychoactive Substances (NPS)

During the 2013/2 and 2014/1 rounds of ICE, participants provided 456 reports of the identification of 195 different NPS in their laboratories. As illustrated in figure 5, synthetic cannabinoids corresponded to 35.5% of all reports followed by phenethylamines (21.9%) and synthetic cathinones (19.5%).

The most commonly reported substance was 25I-NBOMe followed by 25C-NBOMe and Methylone. UNODC would like to thank ICE participants for providing this information and encourage them to use the UNODC early warning advisory on NPS accessible through their ICE portal accounts to submit reports of NPS that they detect as this information enables UNODC to more effectively target the assistance it provides to forensic laboratories.

**Table 4:** Complete list of z-scores for participating laboratories (with their lab code) within the BS test group in 2013/2 and 2014/1. Asterisks indicate that quantification was not performed or that the laboratory did not participate in that particular round of ICE.

**Figure 4:** z-score plot for 2014/1 BS-1 (7651ng/ml benzylecgonine). Each Bar represents the z-score of a laboratory who performed quantitation and the lines indicates the level below which z-scores are considered satisfactory, questionable and unsatisfactory.

**Figure 5:** New psychoactive substances reported by ICE participants during the 2013/2 and 2014/1 rounds of the ICE programme.
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Additional information
If you have comments or questions related to this report, please e-mail us at UNODC-ICE@unodc.org or Lab@unodc.org. Additional information on the ICE programme and other UNODC Laboratory and Scientific Section programmes can be found via the internet at www.unodc.org, or by writing to UNODC at the Vienna International Centre, P.O. Box 500, A-1400 Vienna, Austria. Tel.: (+43-1) 26060-0, Fax: (+43-1) 26060-5866. February 2014