International Collaborative Exercises
Drug Analysis
Member States participating in the 2009 round 2 of ICE

International Collaborative Exercise (ICE)

An important element of the UNODC International Quality Assurance Programme (IQAP) is the implementation of the International Collaborative Exercises (ICE). The exercises allow laboratories, from both developing and developed countries, to continuously monitor their performance in drug testing on a truly global scale. The options available for participation in UNODC ICE are analysis of drugs in Seized Materials (SM) and in Biological Specimens (BS, specifically urine). Two rounds are offered in each category (i.e. SM and BS) per year with each round presenting participants with four different test samples for analysis in each category.

The analytical results returned by laboratories participating in ICE are evaluated by UNODC and a confidential report is provided to each laboratory on its own performance. In addition, a summary report is produced that provides information on the performance of all laboratories returning results in the exercises. Codes are used for participating laboratories to maintain confidentiality.

Analytical results are reviewed by the UNODC Standing Panel of Forensic Experts which oversees the implementation of these exercises, and offers guidance and support in addressing relevant quality issues. The exercises provide an overview of performance and capacity of participating laboratories and enable UNODC to tailor technical support in the laboratory sector for greatest impact.

The new ICE web-based portal, developed in 2009 to facilitate return of results and rapid release of their evaluation, was used in the ICE 2009 round 2 (ICE 2009/2) and currently supports 81 laboratories in 40 Member States.

ICE 2009/2

ICE 2009/2 was implemented in the second half of the year. Invitations for participation were sent out to 150 national laboratories. A total of 122 sets of test samples were sent for analysis to 105 national laboratories in 47 countries worldwide, comprising 83 sets of SM and 39 sets of BS samples. Owing to a number of on-going issues encountered by potential participants, particularly with import authorizations for the controlled substances, the active participation rate for 2009/2 was 92%. Results were received for 76 sets of SM (92%) and for 36 (92%) sets of BS samples respectively. There was a 38% increase in active participation in the BS group in 2009/2 compared with 2009/1.

Test samples

Laboratories are invited to analyze four test samples each in the SM group and/or BS group. Using normal laboratory screening and confirmatory tests, laboratories are required to analyze the samples for the substances listed in the ICE menu, which covers the commonly encountered controlled drugs and related compounds, including certain adulterants and metabolites. Laboratories are also encouraged to report the amounts of controlled drugs present. The mean value and the standard deviation of all returned quantitative results for each test sample have been taken to compute the z-scores. However, for distributions
involving obvious outliers, a more robust standard deviation has been computed by excluding these outliers.

Results with z-scores within the range ± 2 are considered satisfactory. Laboratories with results with z-scores between ± 2 and ± 3 should consider the need for corrective action, and those with results with z-scores beyond ± 3 should take corrective action.

Seized Materials (SM)

The controlled substances present in 2009/2 belonged to three drug classes: ecgonine alkaloids, amphetamine-type stimulants and cannabinoids. There were no wrong results returned for the identification of the target drugs in these three classes. In the current exercise, more than 71% of laboratories also quantified the controlled substance(s) in at least one of the test samples. Statistical analyses of the results received from participants are summarised in the tables and charts corresponding to each sample.

SM1

SM1 was prepared from a seizure of cocaine containing 74% w/w cocaine base. All laboratories which analyzed the sample identified the presence of cocaine correctly. One laboratory did not perform the analysis.

Quantitative data were returned by 55 laboratories (73% of participants), 50 (91%) of which provided results within the acceptable z-score range (Figure 1 and 1a). Quantitative data were returned by 46 laboratories (61% of participants), 43 (93%) of which provided results within the acceptable z-score range (Figure 2 and 2a).

SM2

SM2 was prepared from a seizure of ‘Ecstasy’ tablets containing 28% w/w of 3,4-methylenedioxymamphetamine (MDMA) base. All laboratories which analyzed the sample identified the presence of MDMA correctly. One laboratory did not perform the analysis. Quantitative data were returned by 39 laboratories (52% of participants), 38 (97%) of which provided results within the acceptable z-score range (Figure 3 and 3a). In addition, positive identifications were reported for the two related substances encountered in cannabis products, cannabiol and cannabidiol, by 97% and 96% of laboratories respectively.
**SM4**

SM4 was a sample of metamfetamine (81% as base). All laboratories which analyzed the sample identified the presence of metamfetamine correctly. Two laboratories did not perform the analysis. Quantitative data were returned by 46 laboratories (62% of participants), 45 (98%) of which provided results within the acceptable z-score range (Figure 4 and 4a).

**Biological specimens (BS)**

The BS samples contain controlled substances and metabolites in lyophilized (dried) urine. The controlled substances present in 2009/2 belonged to five drug classes: barbiturates, benzodiazepines, ecgonine alkaloids, amphetamine-type stimulants and opioids.

Thirty six laboratories returned results, 19 also performing quantification of at least one of the analytes present in the test samples. Generally, results were within ±20% of the nominal concentration.

The results for the identification and quantification of the BS test samples are provided in Tables 1 and 2 respectively.

**BS1**

Sample BS1 contained phenobarbital at a concentration of 1730 ng/ml. Thirty one (94%) of the 33 laboratories that performed the analysis identified the presence of phenobarbital correctly. Three laboratories did not perform the analysis.

Quantitative data were returned by 15 laboratories (45% of participants), 14 (93%) of which provided results within the acceptable z-score range, given the wide spread of results obtained. The result for the other laboratory had a z-score over +3.

**BS2**

Cocaine is metabolised extensively in man with only 1% excreted unchanged in urine. The major metabolite is benzoylecgonine with ecgonine methyl ester (methylecgonine) and ecgonine present as minor metabolites. Sample BS2 contained both benzoylecgonine and methylecgonine at a concentration of 920 ng/ml each. In addition, the sample contained 7-aminoflunitrazepam at a concentration of 580 ng/ml. For benzoylecgonine, 30 (94%) of the laboratories who performed the analysis reported it to be present while 4 laboratories did not perform the analysis.
For methylecgonine, 29 (94%) of the laboratories who performed the analysis reported it to be present while 5 laboratories did not perform the analysis. For 7-aminoflunitrazepam, 16 (62%) of the laboratories that performed the analysis reported it to be present while 10 laboratories did not perform the analysis.

Quantitative data for benzylecgonine and methylecgonine were returned by 15 laboratories (47% of participants for benzylecgonine and 48% for methylecgonine). All but 2 results for benzylecgonine and 1 for methylecgonine were within the acceptable z-score range; these remaining 3 results fell within the z-score range ± 2 to ± 3. Quantitative data for 7-aminoflunitrazepam were returned by 8 laboratories (31% of participants). Most of the results were below the target value, reflecting the difficulty associated with the analysis of this metabolite.

Individual laboratories were contacted separately to explore possible common difficulties in the identification process.

**BS3**

BS3 was a sample of metamfetamine (1730 ng/ml) and amfetamine (580 ng/ml). All 36 laboratories provided correct results for the presence of metamfetamine. Thirty one (86%) of the laboratories that performed the analysis also identified the presence of amfetamine (580 ng/ml). All 36 laboratories provided BS3 was a sample of metamfetamine (1730 ng/ml) and 33 laboratories provided correct results for the presence of metamfetamine. All but 3 results for metamfetamine were returned by 27 laboratories (75% of participants). Most of the results were below the target value, reflecting the difficulty associated with the analysis of this metabolite.

**BS4**

Sample BS4 contained morphine (580 ng/ml), its metabolite morphine-6-glucuronide (1400 ng/ml) and methadone (2310 ng/ml). Twenty nine (88%) of the laboratories that performed the analysis for morphine and metabolite identified their presence correctly. Three laboratories did not perform this analysis. Thirty four (97%) of the laboratories that performed the analysis for methadone identified its presence correctly. One laboratory did not perform this analysis.

Quantitative data for free morphine were returned by 12 laboratories (36% of participants) and for total morphine (free morphine + morphine-6-glucuronide) by 10 laboratories (30% of participants). Quantitative data for methadone were returned by 16 laboratories (46% of participants). The results for methadone and free morphine were acceptable, but the results for total morphine were lower than expected, reflecting incomplete hydrolysis of the sample.
Comments from the Standing Panel

**SM:**

Overall the results were very satisfactory. All laboratories correctly identified the major drug constituents in each of the four SM samples, although a few false negative results for cannabidiol and cannabinol in sample SM3 and one relating to false positives for lidocaine and ketamine in SM1. Quantitation is not a requirement of the scheme, but the Panel was encouraged by the number of quantitative results returned and endorsed the general recommendation to laboratories participating in ICE to perform quantitative analysis. For each sample in the SM group, most quantitative results were within the acceptable range of ± 2 z-score values and the means were in good agreement with the target purity values. However, the Panel is concerned about the outliers (z-score outside the range of ± 3) that occurred.

**BS:**

Overall, the results for the BS group were good, given the inherently higher level of difficulty in the analysis of biological specimens compared to seized materials. Correct identification results were returned by more than 90% of participating laboratories for all analytes in all four BS samples, a level of performance comparable to that in other schemes. However, the Panel expressed concern about the false negative results for 7-aminoftunitrazepam and morphine because flunitrazepam is used in several countries and morphine is used globally, and encouraged participating laboratories to check their methodologies and the limits of detection for these analytes.

Emerging Drug Trends

Piperazine derivatives including meta-chlorophenylpiperazine (mCPP), often seized as ‘Ecstasy’ tablets, benzylpiperazine (BZP), 3-trifluoromethylphenylpiperazine (TFMPP), parafluorophenylpiperazine and 1,4-dibenzylpiperazine (DBZP) continue to be reported by a number of participating countries. The European laboratories also reported a significant number of new drugs such as mephedrone, methylethylketamine (MDPV) and the naphthoylindoles, including JWH-018 and JWH-013, and CP 47,497, which are cannabinoid receptor agonists. Opium and heroin hydrochloride, with high percentages of caffeine and other adulterants such as phenobarbital, and diazepam, were reported by laboratories in Asia.

Upcoming ICE rounds in 2010 - 2011

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If you have comments on this report please e-mail us at 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.

**Important web-links**

ICE protocols:
www.unodc.org/documents/scientific/IQAP.pdf

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