



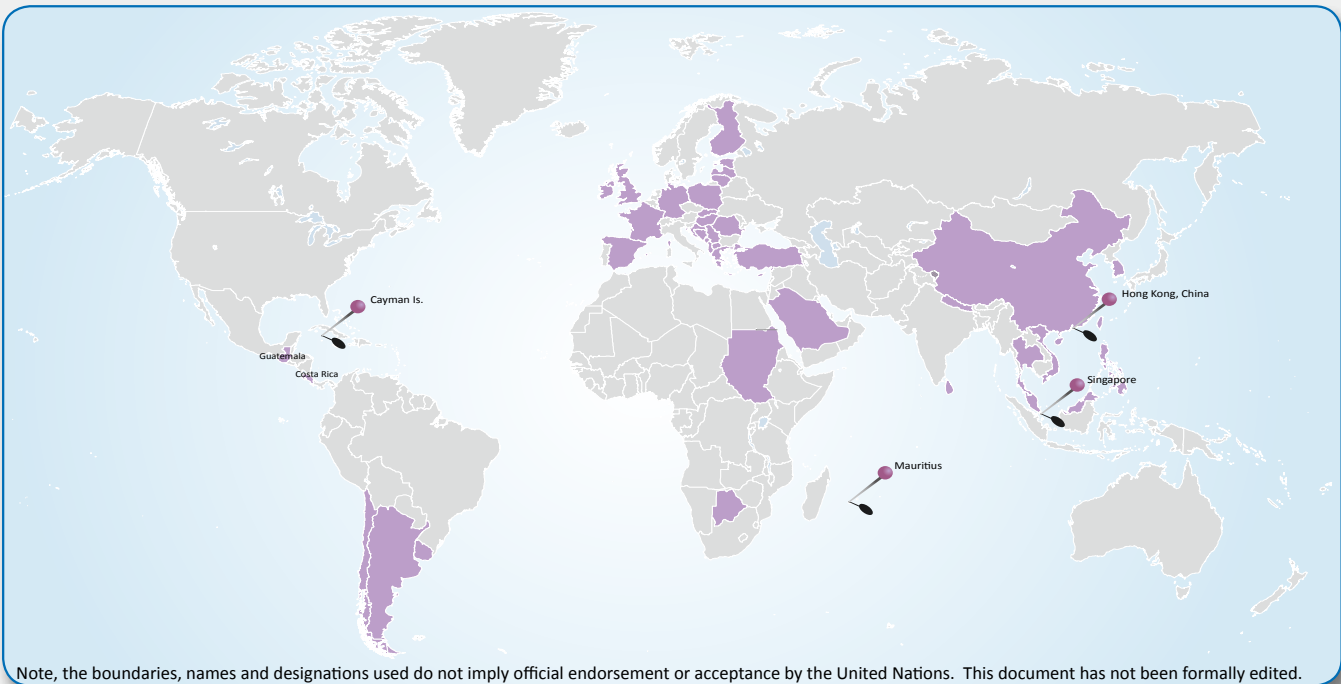
UNODC

United Nations Office on Drugs and Crime

ICE
2010/1



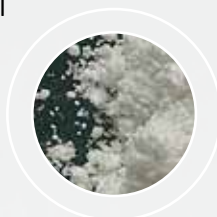
Member States participating in the 2010 round 1 of ICE



International Collaborative Exercise (ICE)

ICE 2010 round 1

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 per year with each round presenting participants with four different test samples for analysis in each category (i.e. SM and BS).



ICE 2010/1 was implemented in the first half of the year. Invitations for participation were sent out to 150 national laboratories.

A total of 134 sets of test samples, comprising 89 sets of SM and 45 sets of BS samples were sent for analysis to 107 national laboratories in 46 countries. One hundred laboratories from 43 countries returned results, amounting to an overall active participation rate of 93% in ICE 2010/1. A number of laboratories continued to experience problems in obtaining import authorization for the controlled substances of the ICE test samples and consequently could not receive the samples and return their results in time.

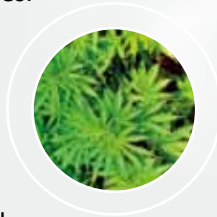
The analytical results returned by laboratories participating in ICE are evaluated by UNODC and a confidential report is provided to each laboratory on its own performance. In addition, a summary report is produced that provides information on the performance of all laboratories returning results in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

Test samples

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 using their normal laboratory screening and confirmatory tests. The menu for the SM group covers controlled drugs and certain adulterants which are commonly-encountered around the world, and the menu for the BS group covers common drugs of abuse, their metabolites and related compounds.

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.



Laboratories are also encouraged to report the purity or concentrations of the controlled drugs present. The mean value and the standard deviation of all returned quantitative results for each test sample are taken to compute z-scores, a statistical measure of the divergence of a result from the mean value. Outliers in each set of results are identified using the Grubbs' Test¹

The ICE portal, which was initially developed in 2009 to facilitate return of results and rapid release of their evaluation to laboratories, was used in the ICE 2010 round 1 (ICE 2010/1) by 93 laboratories (15% increase since ICE 2009 round 2) in 37 Member States.

¹ Glossary of Terms for Quality Assurance and Good Laboratory Practices, UNODC, ST/NAR/26/Rev.1, December 2009.

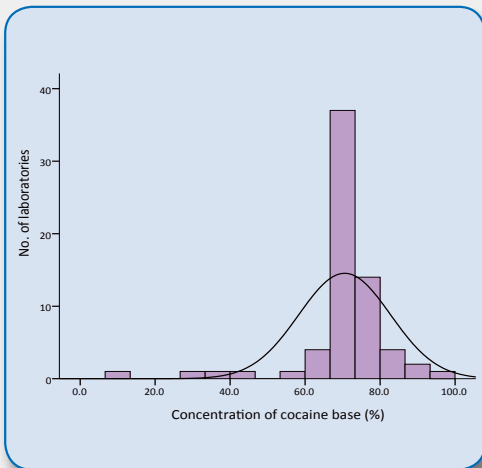


Figure 1. Distribution of results from 67 participating laboratories on content (% by weight) of cocaine base in SM2. Excluding the 8 outliers, the 59 laboratories had a mean of 72% (Std. Dev. = 5).

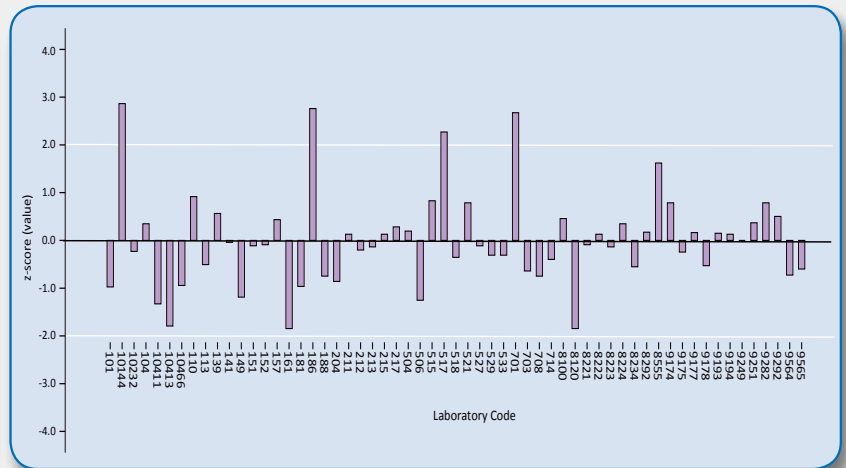


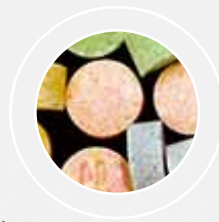
Figure 1a. Z-scores for the 59 individual laboratories submitting results for SM2, excluding 8 outliers.

and for sets of results involving outliers the robust standard deviation is computed by excluding these outliers.

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

Seized Materials (SM)

One sample contained lactose, a common adulterant, while the other three test samples contained controlled substances belonging to three drug classes: ecgonine (cocaine) alkaloids, opioids and cannabinoids, respectively. All laboratories returned correct results for samples containing cocaine and heroin. In the current exercise, more than 78% of laboratories also measured the purity of the controlled substance(s) in at least one of the test samples, which represents a 7% increase compared to ICE 2009/2. Statistical analyses of the results received from participants are summarised in the tables and charts corresponding to each sample.



SM1

SM1 was a “blank” sample containing only lactose. Sixty-three (97%) of the 65 laboratories that performed the analysis correctly identified the presence of lactose. Nineteen laboratories did not perform the analysis for

lactose, following their normal laboratory protocols, but equally they did not mistakenly report the presence of any controlled drugs.

SM2

SM2 was prepared from a seizure of cocaine containing 69% by weight of cocaine base. All laboratories which analyzed the sample correctly identified the presence of cocaine. Quantitative data were returned by 67 laboratories (79% of participants), 59 (88%) of which provided results within the acceptable z-score range (Figure 1 and 1a).

SM3

SM3 was prepared from a seizure of heroin containing 37% by weight of heroin base. The sample also contained two adulterants, caffeine and paracetamol. All laboratories which analyzed the sample correctly identified the presence of heroin. Positive identifications were reported for the two adulterants-caffeine and paracetamol- by 89% and 61% of laboratories respectively. Some laboratories also correctly reported the presence of related substances encountered in heroin seizures but which are not included in the ICE menu e.g. 6-monoacetylmorphine, acetylcodeine, papaverine and noscapine. Quantitative data for heroin were returned by 64 laboratories (75% of participants), 59 (92%) of which provided results within the acceptable z-score range (Figure 2 and 2a).

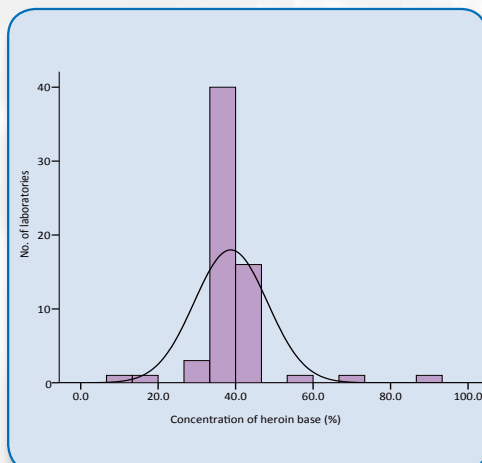


Figure 2. Distribution of all results from participating laboratories on content (% by weight) of heroin base in SM3. Excluding the 5 outliers, the 59 laboratories had a mean of 38% (Std. Dev. = 3).

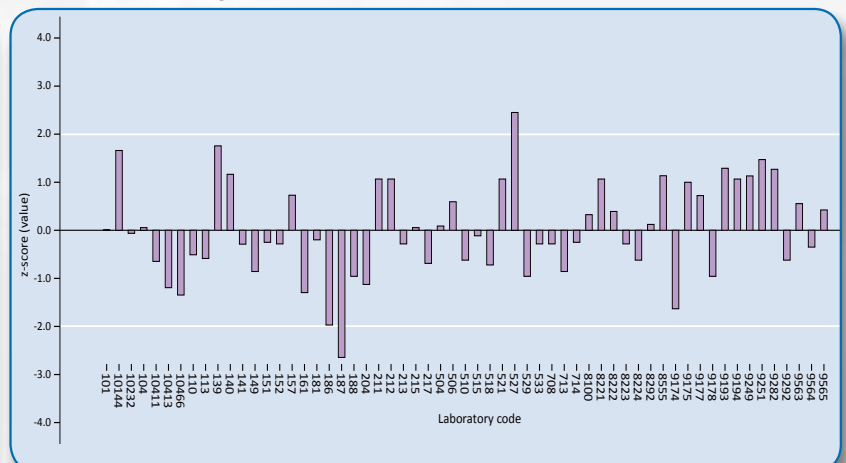


Figure 2a. Z-scores for the 59 individual laboratories submitting results for SM3, excluding 5 outliers.

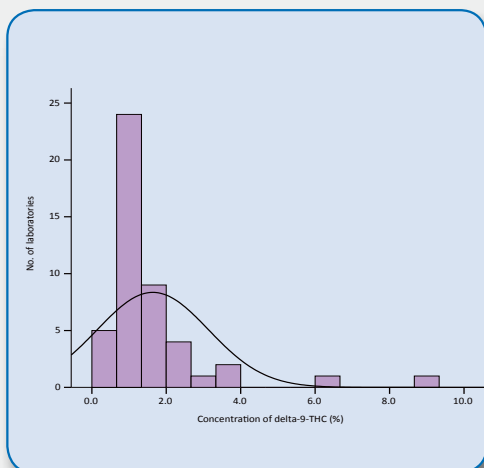


Figure 3. Distribution of all results from participating laboratories on content (% by weight) of *delta*-9-THC in SM4. Excluding the 7 outliers, the 40 laboratories had a mean of 1% (Std. Dev. = 0.3).

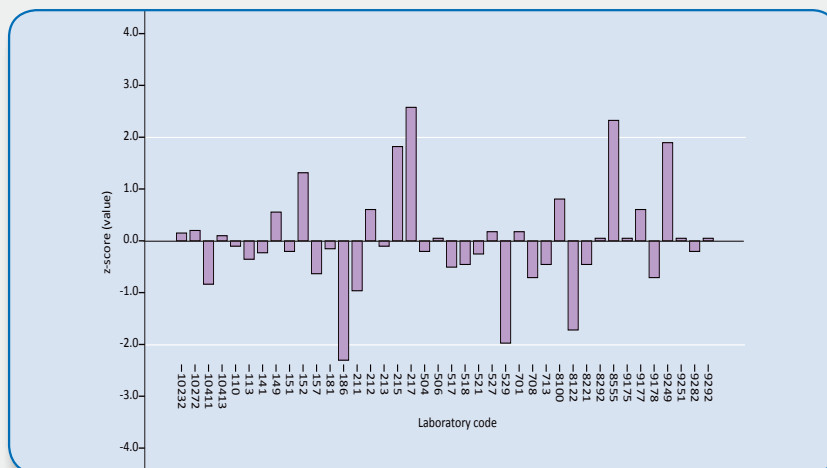


Figure 3a. Z-scores for 40 individual laboratories submitting results for SM4, excluding 7 outliers

SM4

Sample SM4 contained 1.1% by weight of *delta*-9-tetrahydrocannabinol (THC). Seventy-five (95%) of the 79 laboratories which performed the analysis correctly identified the presence of THC. Six laboratories did not perform the analysis. Quantitative data were returned by 47 laboratories (59% of participants), 40 (85%) of which provided results within the acceptable z-score range (Figure 3 and 3a). In addition, the two related substances encountered in cannabis products, cannabidiol and cannabidiol, were identified by 100% and 60% of laboratories respectively.

Biological specimens (BS)

The BS samples contain controlled substances and their metabolites and related compounds in lyophilized (dried) urine. The controlled substances present in 2010/1 belonged to four drug classes: opioids, benzodiazepines, ecgonine (cocaine) alkaloids and amphetamine-type stimulants.

Thirty-six laboratories returned results, of which 21 performed quantification of at least one of the analytes present in the test samples. In all cases, more than 59% of the results were within $\pm 20\%$ 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 codeine (2880 ng/ml) and morphine (290 ng/ml), representing a case involving administration of codeine with morphine as its metabolite. All laboratories that performed the analysis correctly identified the presence of codeine. Only one laboratory did not test for codeine. For morphine, 31 (86%) of the laboratories performing the analysis reported it present while one laboratory did not perform the analysis.

Quantitative data were returned by 19 laboratories for

codeine and/or morphine (54% of participants). All results were within the acceptable z-score range apart from one result for codeine which had a z-score between +3 and +4 and two results for morphine which fell within the z-score range +2 to +3 .

BS2

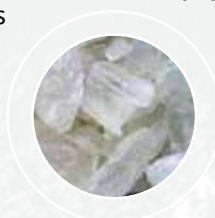
Sample BS2 also contained codeine (230 ng/ml) and morphine (2300 ng/ml) but this time representing a case involving administration of heroin, with morphine as its metabolite, and codeine resulting from the presence of acetylcodeine in the heroin. All 36 laboratories provided correct results for the presence of morphine while 31 (86%) of the laboratories that performed the analysis for codeine reported it to be present. Only one laboratory did not perform the analysis.

Quantitative data for codeine and morphine were returned by 18 and 21 laboratories respectively (51% of participants for codeine and 58% for morphine). All results were within the acceptable z-score range apart from one for codeine and one for morphine which had z-scores over +3.

BS3

BS3 contained 3,4-methylenedioxymetamphetamine (MDMA, 1720 ng/ml), 3,4-methylenedioxyamphetamine (MDA, 350 ng/ml) and temazepam (350 ng/ml). Thirty-two (91%) of the laboratories that performed the analysis for MDMA correctly identified its presence, only one laboratory did not perform the analysis. Twenty-six (76%) of these laboratories also correctly identified the presence of MDA; two laboratories did not perform the analysis. One laboratory did not analyse for either MDMA or MDA. For temazepam, 30 (88%) of the laboratories that performed the analysis reported it to be present while two laboratories did not perform the analysis.

Quantitative data for MDMA were returned by 18 laboratories (51% of participants) and for MDA by 17 laboratories (50% of participants). All results for MDMA were within the acceptable z-score range, apart from



one result with a z-score greater than +3. All quantitative results for MDA were within the acceptable z-score range. Quantitative data for temazepam were returned by 19 laboratories (56% of participants), of which 17 (50 %) were within the acceptable z-score range and two were between ± 2 and ± 3 .

BS4

Sample BS4 contained nordiazepam, a diazepam metabolite, which is also used as a drug on its own (1150 ng/ml), and the cocaine metabolites benzoylecgonine (1150 ng/ml) and methylecgonine (580 ng/ml). Thirty-one (91%) of the laboratories that performed the analysis for nordiazepam correctly identified its presence. Identification results for benzoylecgonine were returned by 30 laboratories (97% of participants) while five laboratories did not perform the analysis. For methylecgonine, 26 (87%) of the laboratories that performed the analysis reported it to be present while six laboratories did not perform the analysis. Four laboratories did not test for either benzoylecgonine or methylecgonine.

Quantitative data for nordiazepam were returned by 20 laboratories (59% of participants). All results were within the acceptable z-score range apart from one which fell within the z-score range -2 to -3. Quantitative data for benzoylecgonine and methylecgonine were returned by 16 laboratories (52% of participants) and 15 laboratories (50% of participants) respectively. All results for benzoylecgonine and methylecgonine were within the acceptable z-score range apart from two (one each for benzoylecgonine and methylecgonine) which had z-scores of ± 2 to ± 3 .

Laboratory code	Sample 1		Sample 2		Sample 3			Sample 4		
	Codeine	Morphine and/or Metabolites	Codeine	Morphine and/or Metabolites	3,4-methylenedioxy metamfetamine (MDMA)	Temazepam	Tenamfetamine (MDA)	Benzoylecgonine	Methylecgonine	Nordiazepam
10412	r	r	r	r	r	nr	r	r	nr	nr
10531	r	w	w	r	w	r	w	w	w	r
110	r	r	r	r	r	r	r	nr	nr	r
113	r	r	r	r	r	r	r	r	r	r
126	r	r	r	r	w	w	w	r	r	w
181	r	r	r	r	r	r	r	r	r	w
313	r	r	r	r	r	r	r	r	r	r
321	r	r	r	r	w	r	w	r	r	r
327	r	r	r	r	r	r	r	r	r	r
330	r	r	r	r	r	r	r	r	r	r
332	r	r	r	r	r	w	w	r	r	nr
337	r	r	r	r	r	r	r	r	r	r
347	r	w	w	r	r	w	r	r	w	w
348	r	r	r	r	r	r	w	r	r	r
349	r	r	w	r	r	r	r	r	w	r
504	r	r	r	r	r	r	w	r	r	r
506	r	r	r	r	r	r	r	r	r	r
510	r	r	r	r	r	r	r	r	r	r
518	r	nr	r	r	r	nr	nr	nr	nr	r
520	r	r	r	r	r	r	w	r	w	r
529	r	r	r	r	r	r	r	r	r	r
533	r	r	r	r	r	r	r	r	r	r
703 BS	r	r	r	r	r	r	r	r	r	r
713	r	r	r	r	r	r	r	r	r	r
751	r	r	r	r	r	r	r	r	r	r
761	r	r	r	r	r	r	w	r	r	r
8329	r	r	r	r	r	r	r	r	r	r
8369	r	r	r	r	r	r	r	r	r	r
8392	r	r	r	r	r	r	r	r	r	r
8396	r	r	r	r	r	r	r	r	r	r
8555	r	r	r	r	r	r	r	r	r	r
9193	r	r	r	r	r	w	r	r	r	r
9366	nr	r	nr	r	r	r	r	r	nr	r
9563	r	w	w	r	nr	r	nr	nr	nr	r
9564	r	w	r	r	r	r	r	nr	nr	r
9565	r	r	r	r	r	r	r	nr	r	r

nr = no report; w = wrong; r = right

Table 1. Results returned by laboratories for identification of substances in the BS category.

Laboratory code	BS1		BS2		BS3			BS4		
	Codeine	Morphine	Codeine	Morphine	MDMA	MDA	Temazepam	Nordiazepam	Benzoylecgonine	Methylecgonine
110	3944	134	276	1269	2399	306	1094	1357	n.r	n.r
321	1330	280	230	1300	n.r	n.r	840	1200	1050	450
330	2133	300	160	2200	1566	375	1250	1050	900	580
332	n.r	1000	n.r	1000	n.r	n.r	n.r	n.r	n.r	n.r
348	714	253	240	2420	4800	n.r	360	250	296	2940
510	2680	310	250	2190	1580	340	1230	1070	1050	530
529	3057	209	134	2732	1890	326	1295	780	1312	751
703	11465	1275	890	10970	1070	320	947	890	1315	551
713	3100	300	210	2200	1800	300	1200	1100	1500	520
751	3000	300	240	2400	2100	280	1100	1100	1400	530
761	2770	341	245	2150	1540	299	989	1040	1040	527
8329	3395	338	262	2215	1685	355	858	942	1409	513
8369	2696	254	218	1974	1452	339	1318	1150	1165	502
8392	2474	266	233	2158	1594	357	1215	1100	1173	585
8396	3127	307	255	2311	1836	334	1166	1275	1197	288
8555	3536	342	305	2951	1740	304	1124	1380	1593	748
9193	3190	250	157	4363	2140	350	n.r	687	1012	2127
9366	n.r	295	n.r	2320	1600	345	1410	990	975	n.r
9564	3767	n.r	346	n.r	1680	330	915	780	n.r	n.r
9565	2530	1480	170	2840	1650	320	470	1560	n.r	n.r
10531	1644	n.r	n.r	2227	n.r	n.r	1990	978	n.r	n.r
Target value	2880	290	230	2300	1720	350	1150	1150	1150	580

n.r = not reported

Table 2. Results returned by laboratories for quantification of substances in the BS category (ng/ml).

Comments from the Standing Panel

Seized Materials (SM)

It is very encouraging to see active participation by 96% of the 89 laboratories receiving SM samples and the increased use of the ICE portal to return results. Overall, the results were very satisfactory, with none of the participants finding any controlled drugs in the lactose (blank) sample SM1, all correctly identifying cocaine in sample SM2 and heroin in sample SM3, and 95% identifying the THC in sample SM4. No laboratory mistakenly identified any other controlled drugs which were not originally present in these samples, reflecting well on their contamination-avoidance procedures. The large proportion of participants also quantifying cocaine (79%), heroin (75%) and THC (59%) shows increasing compliance with the general recommendation to laboratories participating in ICE to perform quantitative analysis, and the large proportion (>60%) also identifying related substances and adulterants not in the ICE menu indicates better information generation for intelligence purposes. 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 four laboratories failing to identify THC in sample SM4, notwithstanding its low concentration, while six did not perform the analysis, and the few outliers in the results for each sample. These concerns should be followed up by the laboratories concerned with assistance from UNODC.

Biological Specimens (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 for the major analytes present in all four BS samples by more than 90% of participating laboratories while metabolites were correctly identified by more than 80% of participants. The detection of metabolites in urine provides support for the correct identification of the administered substance and is particularly important for that reason. Laboratories should seek to include metabolites of target analytes when possible. The Panel is pleased with the number of laboratories undertaking quantitative analysis of urine samples. Quantitative analysis may not always assist in the

interpretation of results in a case but has been found to improve the quality of laboratory analyses.

The Panel considered that these results underlined the value of the ICE programme in revealing weaknesses in analytical methods which need to be addressed. ICE is a training and management tool and participating laboratories should take advantage of the support that LSS can provide to improve performance.

Emerging drug trends

In this round, pink tablets with the Adidas® logo (approx. 8 mm diameter), suspected to be 'Ecstasy' were reported by some East Asian laboratories. However, analysis indicated a mixture of benzylpiperazine (BZP), 3-trifluoromethylphenylpiperazine (TFMPP), 1,4-dibenzylpiperazine (DBZP) and caffeine. Similar tablets with 'O' logo were identified to contain a mixture of piperazines (BZP, TFMPP) and ketamine. Piperazine derivatives continue to be reported by a number of European participating countries. Some piperazines were also reported in Latin America and these have been used as substitutes for amphetamine-type-stimulants (ATS).

As in 2009 round 2, a significant number of ICE participating laboratories worldwide reported drugs such as mephedrone, methylenedioxypyrovalerone (MDPV) and the naphthoylindoles, including JWH-018, JWH-013 and CP 47,497, which are cannabinoid receptor agonists. *N,N*-Dimethyltryptamine (DMT) a naturally occurring hallucinogenic drug of the tryptamine family, has been identified in plant materials in countries both in Europe and in Latin America. Individual cases of liquid LSD, *gamma*-hydroxybutyric acid (GHB), phencyclidine (PCP) and fresh hallucinogenic mushrooms have been reported.

Future timelines

	ICE 2011/1	ICE 2011/2
Invitation for participation	17.01.2011	01.08.2011
Confirmation of participation	28.02.2011	31.08.2011
Receipt of import documents	31.03.2011	17.10.2011
Submission of test results	30.06.2011	16.01.2012

Need additional information

If you have comments or questions related to this report 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.

please e-mail us at Lab@unodc.org. Additional information

Important web-links

ICE protocols:
www.unodc.org/pdf/document_1998-10-01_1.pdf
www.unodc.org/documents/scientific/IQAP.pdf

Acknowledgements

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