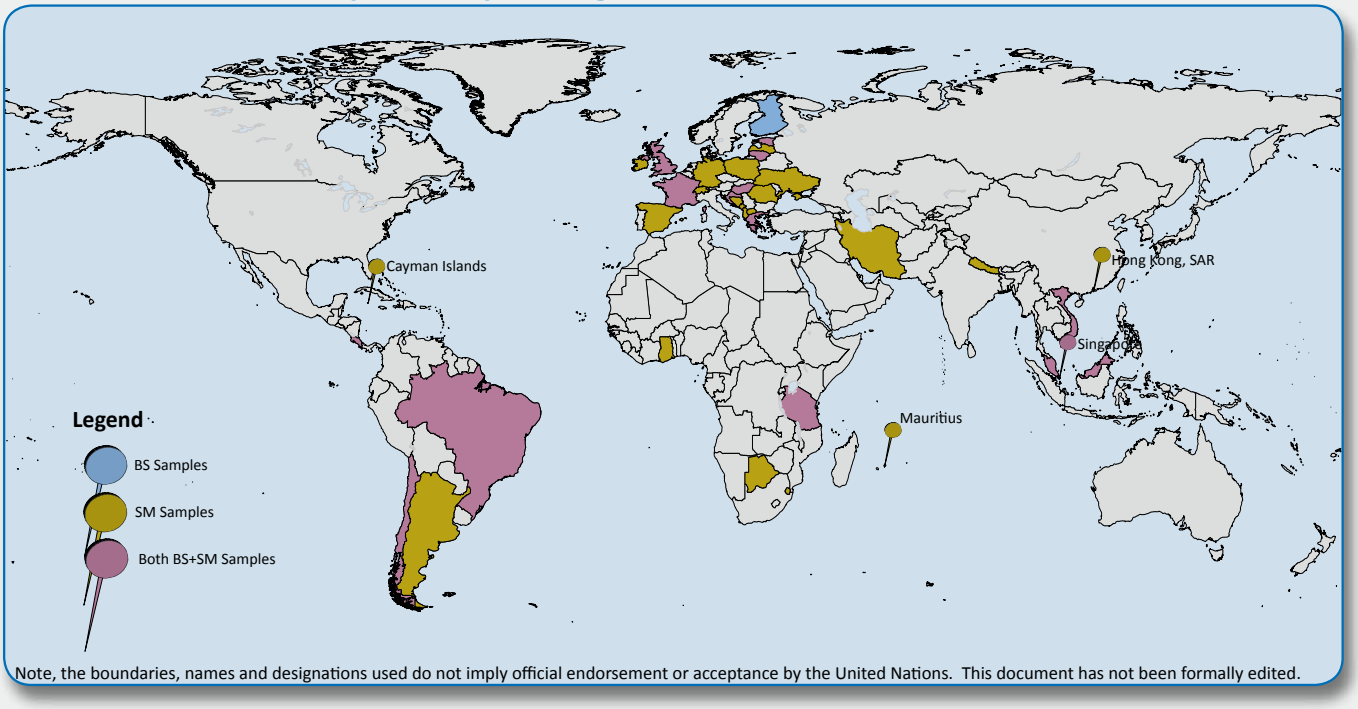
UNODC
United Nations Office on Drugs and Crime

ICE 2009

International Collaborative Exercises

Drug Analysis

Member States participating in the 2009 Round 1 of ICE



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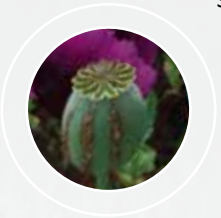
International Collaborative Exercise (ICE)

An important element of the UNODC International Quality Assurance Programme (IQAP) is the implementation of the International Collaborative Exercises (ICE). The exercise allows 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 drug analysis in Seized Materials (SM) and in Biological Specimen (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. The summary report provides information on the performance of all laboratories participating in the exercises. Codes are used for participating laboratories to maintain confidentiality.

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

Further to CND resolution 52/7, UNODC launched an on-line version of the ICE. The portal allows laboratories to register for participation and also submit their analytical results online. The user-friendly process is conducted in a secure environment and provides laboratories near real time, personalised and confidential evaluation of the submitted results. The new electronic system has been used in the evaluation of results in the ICE 2009 Round 1 and currently supports 60 laboratories in 34 Member States.

ICE 2009 Round 1

The ICE 2009 Round 1 was implemented in the first half of the year. Invitations for participation were sent out to 150 laboratories. Due to a number of issues encountered by potential participants particularly with import authorizations for the controlled substances, the active participation rate for 2009 Round 1 was at 93%.

A total of 108 sets of test samples were sent for analysis to national laboratories in 39 countries worldwide, comprising 71 sets of seized materials and 37 sets of biological specimens. Results were received for 65 sets of SM (92%) and for 24 (65%) sets of BS samples respectively. For laboratories who obtained import authorization for the controlled substances, the failure to return results has been attributed to problems with delivery of samples and technical difficulties in analysis. A separate national ICE round was implemented for the first time in Brazil with the participation of 34 laboratories. The summary report of the implementation of this exercise is presented separately. Comparative statistics for 2008 and 2009 Round 1 showed an 8% decrease in the number of sample sets distributed.

Test samples

Participating laboratories receive for analysis four test samples each in the SM group and BS group. Using normal laboratory screening and confirmatory tests, laboratories are required to analyse the samples guided by the substances listed in the ICE menu which covers the commonly encountered controlled drugs and related compounds, including certain adulterants or metabolites. Laboratories are also encouraged to report the amounts of controlled drugs present using the standard form (Analytical Result Form, ARF) provided by UNODC.

Seized Materials (SM)

The four SM test samples for the ICE 2009 Round 1 were representative of seizures of heroin, cocaine, cannabis and amphetamine. Although quantification of substances is currently not mandatory, participating laboratories are encouraged to return quantitative data on the samples. In the current exercise, more than 70% of laboratories determined the content of 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 the sample. For quantitative data, the acceptable result to the reference value is considered to be within a *z*-score range from -2 to +2.

SM1

SM1 was prepared from a seizure of heroin with a diacetylmorphine content of 61%. All participating laboratories identified correctly the presence of diacetylmorphine in SM1. In addition, two laboratories reported the presence of heroin-related substances such as acetylcodeine, monoacetylmorphine, papaverine, narcotine and meconine in SM1 although these were not indicated in the ICE menu. Quantitative data (Figure 1) on the content of diacetylmorphine were returned by 45 laboratories (69% of participants) with 89% of laboratories providing results within the acceptable range (Figure 2).

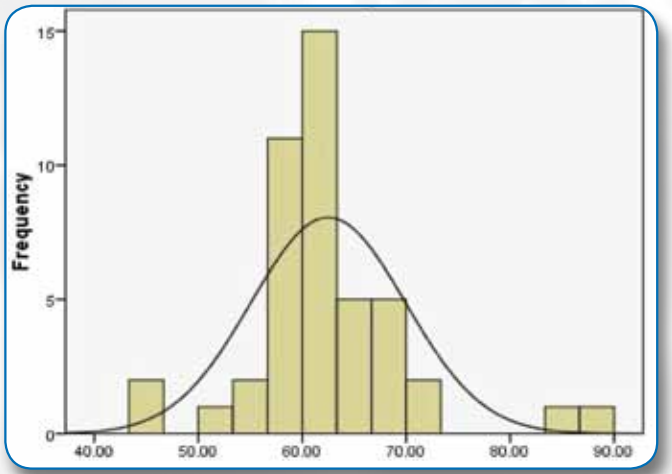


Figure 1. Distribution of results from participating laboratories on content (%) of diacetylmorphine in SM1

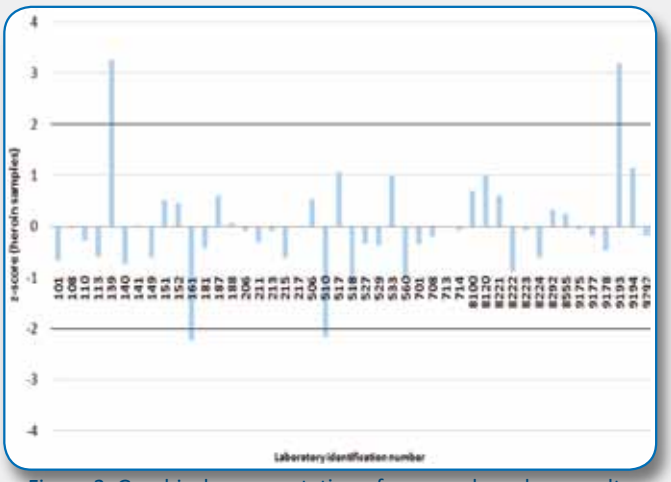


Figure 2. Graphical representation of z-scores based on results submitted for SM1

SM2

SM2 was representative of a cocaine seizure containing 67% cocaine base. Similar to the results reported for SM1, all participating laboratories correctly identified cocaine in SM2. Quantification of SM2 was performed by 72% of the laboratories (47 laboratories, Figure 3) with 94% of laboratories providing results within the acceptable range (Figure 4).

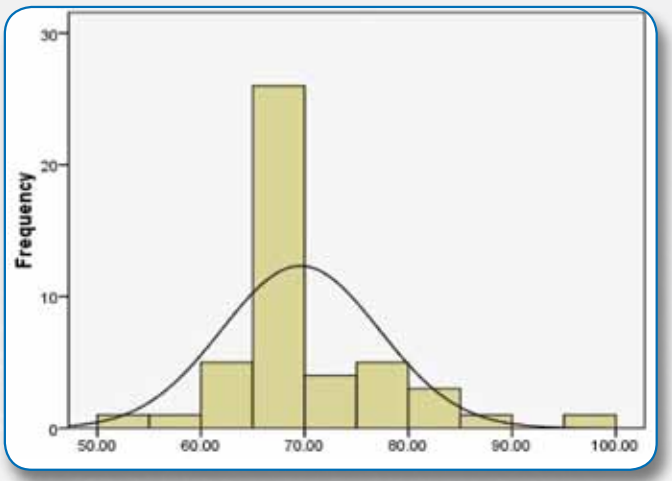


Figure 3. Distribution of results from participating laboratories on content (%) of cocaine in SM2

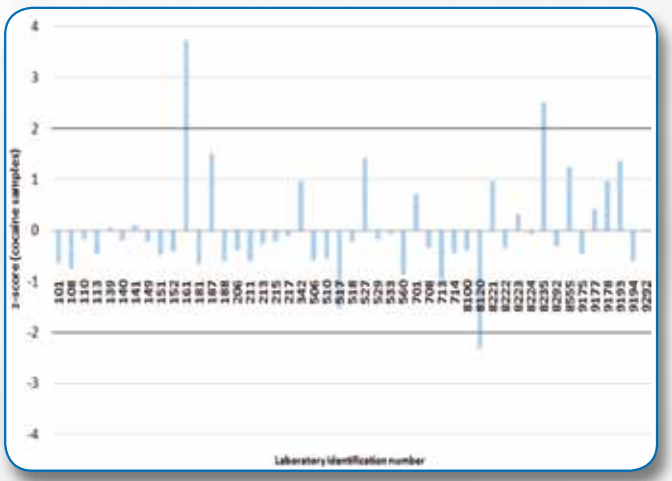


Figure 4. Graphical representation of z-scores based on results submitted for SM2

SM3

The sample was cannabis resin containing 4% Δ-9-Tetrahydrocannabinol (THC). Except for one false negative result for THC, all the other laboratories correctly identified the presence of THC (Figure 5). In addition, positive results were reported for the two related substances encountered in cannabis products, cannabinol and cannabidiol, by 95% and 94% of laboratories respectively. Only 35 laboratories (54%) quantified THC with 97% of laboratories reporting within the ±2 *z*-score range (one value was considered an outliner, see Figure 6). However, it should be noted that the majority of laboratories provided results below the reference value for SM3. In some cases, it was reported that quantification of cannabis samples was not performed routinely in the laboratories or that the amount of sample was not sufficient for quantitative purposes. Laboratories are referred to the UNODC manual on "Recommended methods for the identification and analysis of cannabis and cannabis products" (see *Book Review* section).

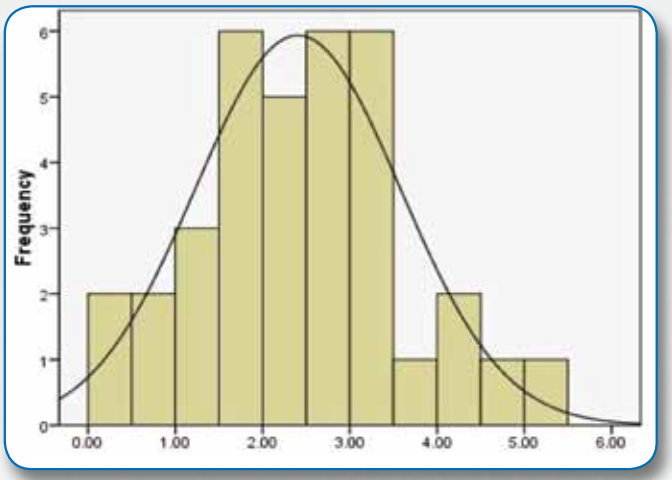


Figure 5. Distribution of results from participating laboratories on content (%) of THC in SM3

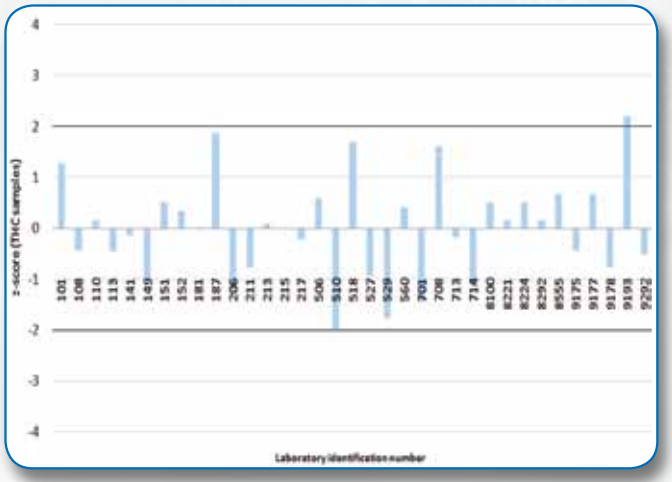


Figure 6. Graphical representation of z-scores based on results submitted for SM3

SM4

SM4 was a sample of amphetamine (42% as base) containing the adulterant caffeine. Amphetamine was correctly identified in the sample by 98% (64) of the participating laboratories while a high incidence of erroneous results was noticed for caffeine, 23 laboratories reporting false negative results while only 15 correctly identified caffeine in this sample. In addition, 26 laboratories did not analyse at all the presence of the adulterant. Quantification of SM4 was performed by 38 laboratories (58%) with 89% of laboratories providing results within the acceptable range (Figure 8).

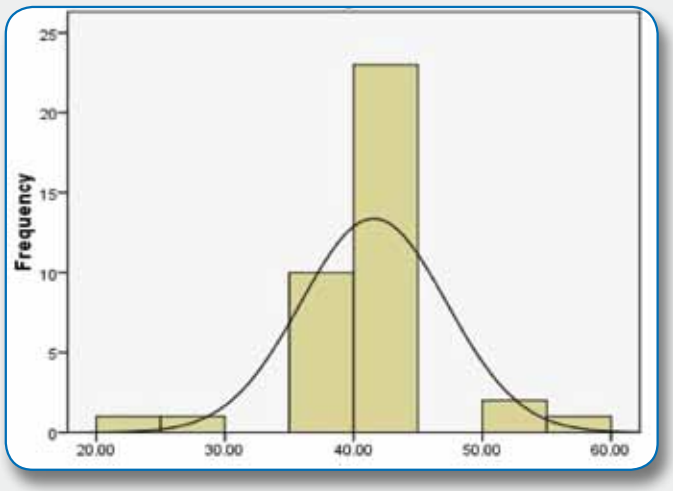


Figure 7. Distribution of results from participating laboratories on content (%) of amphetamine in SM4

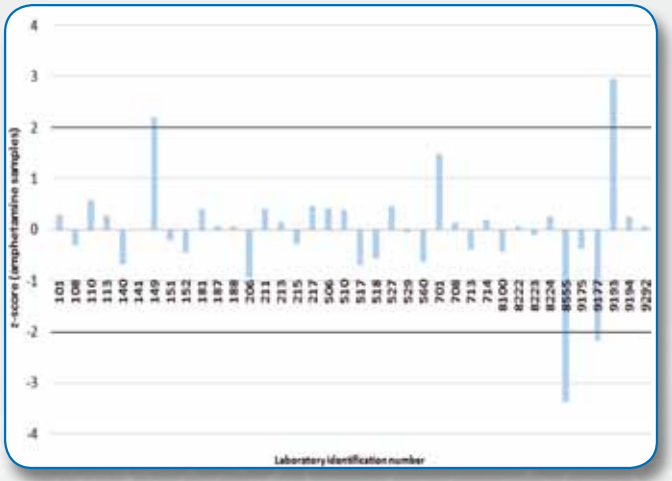


Figure 8. Graphical representation of z-scores based on results submitted for SM4

General comments

Most of the quantitative results for each sample were within an acceptable spread around the mean but a small number of outliers were also present. Satisfactory results were submitted by most laboratories in the SM group; all laboratories correctly identified heroin and cocaine, and approximately 98% correctly identified THC (in the cannabis sample) and amphetamine. Regional trends were not noted with respect to wrong results. Incorrect results were randomly distributed amongst the returns made by various laboratories and were not due to one or two particular laboratories.

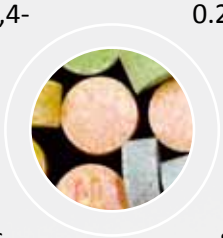
Biological specimens (BS)

The BS samples contain controlled substances and metabolites in lyophilized (dried) urine which mimics human urine from persons who have used controlled substances listed in the ICE menu. A total of 24 laboratories participated in BS category for ICE 2009 Round 1.

Four samples were prepared from urine which was free from all major groups of abused drugs. The urine was then spiked by addition of weighed-in quantities of the analytes specified in the samples. Analytes except codeine, the benzodiazepines and the cannabis metabolite 11-nor-Δ-9-tetrahydrocarboxylic acid were dissolved in water for addition to urine. Codeine and the benzodiazepines were added as solutions in ethanol and the cannabis metabolite in methanol. The urine was dispensed in 50 ml aliquots and lyophilised. No preservative was added to the urine.

BS1

Sample BS1 simulates urine from a human subject after taken ecstasy, and contained MDMA (3,4-methylenedioxymetamfetamine hydrochloride) and MDA (tenamfetamine; 3,4-methylenedioxyamfetamine hydrochloride) at concentrations of 2.73 mg/L (2.30 with respect to the base) and 0.28 mg/L (0.23 with respect to the base) respectively. Positive results for the identification of MDMA were returned by 21 laboratories (91%) and 18 for MDA (86%). Some laboratories reported incorrect results for MDMA and MDA using immunoassays. These results were attributed to the low sensitivity and/ or cross reactivity of the tests.



BS2

Sample BS2 contained morphine (0.58 mg/L), the morphine metabolite morphine-6-glucuronide (1.40mg/L) and codeine (0.12mg/L). With the exception of one laboratory, all other participants correctly identified morphine. Positive results for codeine were reported by 19 laboratories (79%).

BS3

Cocaine is metabolised extensively in man with only 1% excreted unchanged in urine. The major metabolite is benzoylecgonine with ecgonine methyl ester and ecgonine present as minor metabolites. The sample contained benzoylecgonine (1.38 mg/L) and ecgonine methyl ester (0.69 mg/L) and simulated the profile of urine from a cocaine user. For benzoyl ecgonine, 19 laboratories (83%) reported a positive identification while 15 laboratories (65%) detected methyl ecgonine ester.

BS4

The most active component in cannabis, Δ-9-tetrahydrocannabinol (THC) is metabolized in man to 11-nor-Δ-9-tetrahydrocarboxylic acid which can be identified in urine. Sample BS4 contained 11-nor-Δ-9-tetrahydrocarboxylic acid at a concentration of 0.23mg/L. In addition, the sample contained the major metabolites of diazepam; nordiazepam (2.30 mg/L), temazepam (0.58 mg/L) and oxazepam (1.38 mg/L) thus simulating a urine sample after concomitant use of cannabis and diazepam. Out of the 24 participating laboratories, correct identification of substances was reported by 20 laboratories for 11-nor-Δ-9-tetrahydrocarboxylic acid, 21 laboratories for nordiazepam, 18 laboratories for oxazepam and 18 laboratories for temazepam.

The results returned by the laboratories for identification of substances in the SM category are summarised in Table 1.

Only 14 of the 24 laboratories participating in the BS category attempted quantification of the analytes. Generally, results were within ±20% of the nominal concentration. However, in view of the small sample size, summary statistics have not been computed for the quantitative data in the BS category. The results for the quantification of the BS test samples are provided in Table 2.

	Sample 1		Sample 2		Sample 3		Sample 4			
Laboratory Code	3,4-methylenedioxymetamfetamine (MDMA)	Tenamfetamine (MDA)	Codeine	Morphine and/or Metabolites	Benzoylecgonine	Methylecgonine	11-nor-Δ9-THC-9-carboxylic acid	Nordiazepam	Oxazepam	Temazepam
110	r	r	r	r	r	w	r	r	r	w
157	r	0	0	0	0	0	0	r	r	0
181	r	r	r	r	w	r	r	r	r	r
321	r	r	r	r	r	r	r	r	r	r
327	r	r	r	r	r	r	r	r	r	r
328	r	r	r	r	r	0	r	r	r	r
330	r	r	w	r	r	r	r	r	r	r
332	r	w	r	r	r	r	r	w	r	w
337	w	0	r	r	r	w	0	r	w	r
349	r	r	r	r	r	w	r	r	w	r
506	r	r	r	r	r	r	r	r	r	r
510	r	r	r	r	r	r	r	r	r	r
517	r	r	r	r	r	r	r	r	r	r
518	w	w	r	r	w	w	w	w	w	w
520	r	w	r	r	r	r	r	r	w	r
533	r	r	r	r	r	r	r	r	r	r
560	0	r	r	r	w	r	r	r	w	w
751	r	r	w	r	r	r	r	r	r	r
761	r	w	w	r	r	r	r	r	r	r
8329	r	r	r	r	w	w	w	w	w	w
8392	r	r	r	r	r	w	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
9366	r	r	0	r	r	0	r	r	r	r

r = right result; w = wrong result; 0 = not reported

Table 1. Laboratory results for the identification of substances in the BS category

	Sample 1		Sample 2			Sample 3		Sample 4			
Laboratory Code	BS-1/ MDMA	BS-1/ MDA	BS-2/ Total Morphine	BS-2/ Free Morphine	BS-2/ Codeine	BS-3/ Benzyl Ecgonine	BS-3/ Methyl Ecgonine	BS-4/ THC-COOH	BS-4/ Nor-Diazepam	BS-4/ Temazepam	BS-4/ Oxazepam
110	2151	251	n.r.	732	n.r.	629	n.r.	240	n.r.	n.r.	n.r.
321	1700	249	n.r.	580	n.r.	n.r.	n.r.	199	2000	610	1400
328	2850	308	760	540	120	1260	n.r.	190	1915	582	1811
330	2603	232	832	625	n.r.	1008	561	198	1863	596	965
332	n.r.	n.r.	n.r.	1143	n.r.	n.r.	n.r.	204	n.r.	n.r.	n.r.
510	2821	301	777	548	115	1222	350	182	1880	575	1811
517	2201	209	382	338	471	2100	662	122	2463	710	2435
751	2998	260	880	470	170	1400	480	350	7500	660	1600
761	n.r.	n.r.	1286	n.r.	n.r.	1362	702	200	2112	454	1042
8329	2660	261	n.r.	631	143	wrong	wrong	wrong	wrong	wrong	wrong
8392	2560	260	n.r.	260	80	1215	n.r.	260	680	540	600
8396	2201	224	n.r.	577	134	1510	650	225	2300	570	1450
8555	2343	230	548	503	149	1338	628	178	2493	553	1523
9366	2233	280	n.r.	592	n.r.	1223	n.r.	168	2000	510	1750

n.r = not reported

Table 2. Laboratory results for the quantification of substances in the BS category

Book Review

Guidance on Validation

The validation of analytical methods and the calibration of equipment are important aspects of quality assurance in the laboratory. This manual provides an introduction and practical guidance to national authorities and analysts in the implementation of method validation, and also the performance verification of laboratory equipment within their existing internal quality assurance programmes. The procedures described represent a synthesis of the experience of scientists from several reputable laboratories around the world. While there is diversity with respect to detail in existing method validation protocols according to their context, there is also a common thread of principle underlying all systems. The manual focuses specifically on the issue of quality assurance and good laboratory practices in drug testing laboratories, it can serve as an educational document and as a means of encouraging laboratories to consider quality assurance matters.

Identification and analysis of cannabis

The manual on "Recommended methods for the identification and analysis of cannabis and cannabis products" has been designed to provide practical guidance to drug analysts on the identification of cannabis and cannabis products. It is an updated and significantly revised version of the earlier manual (ST/NAR/8), and has been prepared taking into account both developments in analytical technology and advances in the science of cannabis, and with a view to providing the analytical basis for an objective discussion about changes in THC content over time, and differences between regions and products.

Multilingual Dictionary on Precursor Chemicals

Similar to controlled Narcotic Drugs and Psychotropic Substances, Precursors and Chemicals appear under a wide variety of names. This complicates the task of national and international drug control authorities. UNODC has responded to this problem by developing a Multilingual Dictionary of Precursors and Chemicals Frequently Used in the Illicit Manufacture of Narcotic Drugs and Psychotropic Substances under International Control, covering all Precursors and Chemicals used in the illicit manufacture of Narcotic Drugs and Psychotropic Substances under international control with information on chemical names and variants, synonyms and trade names. It is structured in four parts: Part One - Individual monographs on all 23 Precursors and Chemicals under International Control; Part Two - Alphabetical cross-index of all names and synonyms included in the monographs; Part Three - Bilingual lists of all listed substances and common salts in Arabic, Chinese, French, Spanish and Russian each translated from and to English; Part Four - Information on the international regime of control, listing history and control status of the precursors and chemicals under international control.

UNODC reference manuals, guidelines and publications can be found on-line at: www.unodc.org/unodc/en/scientists/publications.html?ref=menuside

Upcoming ICE rounds in 2010

	ICE 2010/1	ICE 2010/2
Invitation for participation	16.01.2010	01.08.2010
Confirmation of participation	28.02.2010	31.08.2010
Receipt of import documents	31.03.2010	15.10.2010
Submission of test results	30.06.2010	15.01.2011

Need additional information

Contact us

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/pdf/document_1998-10-01_1.pdf

www.unodc.org/documents/scientific/IQAP.pdf

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