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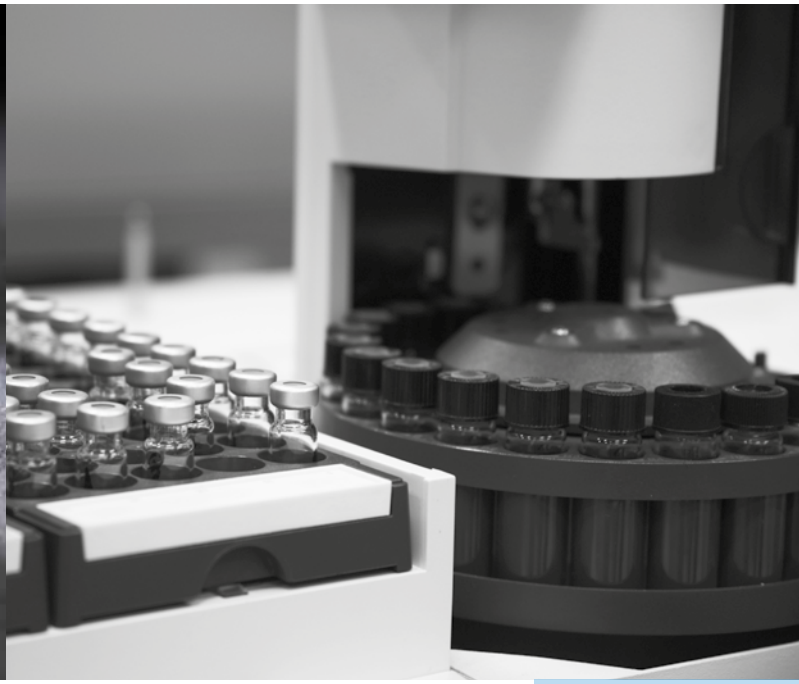


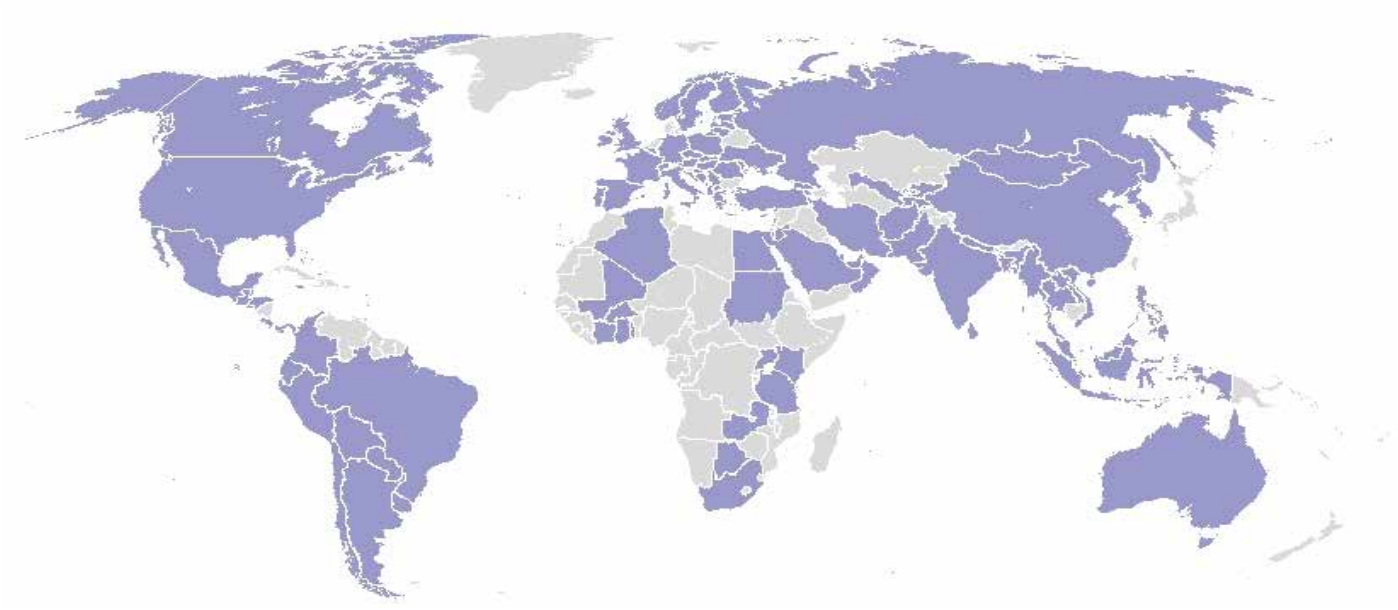
INTERNATIONAL COLLABORATIVE EXERCISES

Drug Analysis

2020

ICE Rounds 2019/2 and 2020/1

Figure 1: Member States who have participated in the International Collaborative Exercises programme since 2009.



The boundaries and names shown and the designations used on this map do not imply official endorsement or acceptance by the United Nations. Dashed lines represent undetermined boundaries. The dotted line represents approximately the Line of Control in Jammu and Kashmir agreed upon by India and Pakistan. The final status of Jammu and Kashmir has not yet been agreed upon by the parties. The final boundary between the Republic of Sudan and the Republic of South Sudan has not yet been determined. A dispute exists between the Governments of Argentina and the United Kingdom of Great Britain and Northern Ireland concerning sovereignty over the Falkland Islands (Malvinas).

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 17025:2017, "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. The immediate feedback allows the laboratories to quickly review their procedure and take corrective actions whenever necessary.

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 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

Since its inception in 1995, the number of participants in the ICE programme has grown significantly. In the ICE rounds of 2019/2 and 2020/1, a total of 304 laboratories from 90 Member States participated* in the programme. The number of laboratories taking part in round 2020/1 decreased 5% in year on year participation. This is mainly due to shipping restrictions to a number of countries experiencing the on-going COVID-19 pandemic. Nonetheless, a total of 164 laboratories participated consecutively in the last four rounds (2018/2 - 2021/1) of the ICE programme representing an increase of 6% compared to the previous four rounds (2016/2 - 2018/1). Figure 2 shows the growth in laboratory participation in the ICE programme over the last 10 years and figure 3 shows the participation of laboratories in the SM and BS test groups for all ICE rounds from 2018/1 up to the most recent 2020/1 round of the programme.

Due to the on-going pandemic, participants faced several challenges including lack of human resources, delays in obtaining import permits and shipping restrictions of postal services. To overcome these challenges, some competent national authorities started issuing electronic import permits, while some laboratories took the initiative to arrange shipment of the test samples at their own expense. Although these measures helped, there were still considerable delays in sending out test samples and in the submission of results from a number of laboratories.

* laboratories who submitted results in rounds 2019/2 and/or 2020/1

However, despite these challenges, most participants continued to enrol in 2020 which demonstrates recognition among the laboratories of the importance of quality assurance and the benefits of participation in the ICE programme. Focused technical assistance was also provided to laboratories in collaboration with regional forensic science networks and with support from UNODC regional and country programmes.

Figure 2: Growth in laboratory participation in the ICE programme

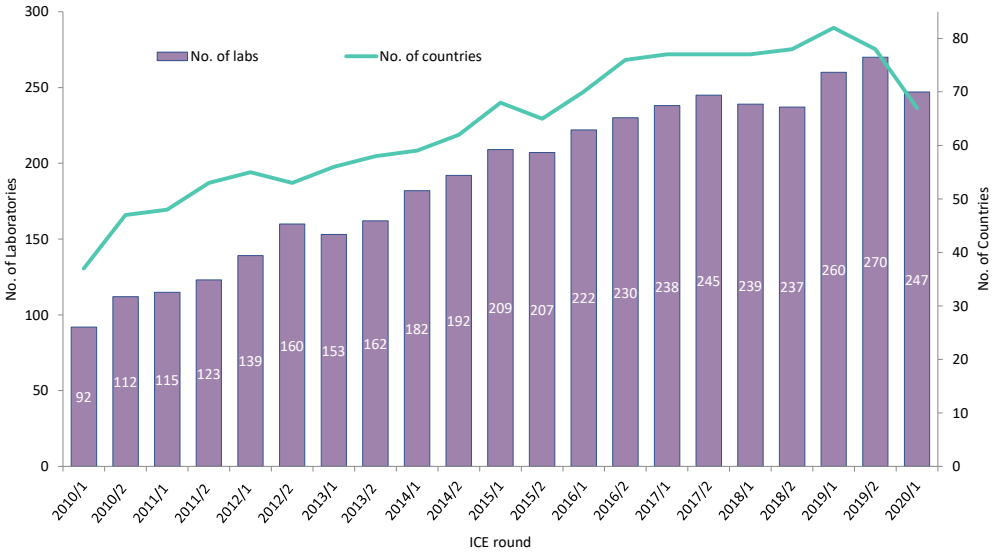


Figure 3: Number of laboratories that have participated in the ICE programme since 2018.

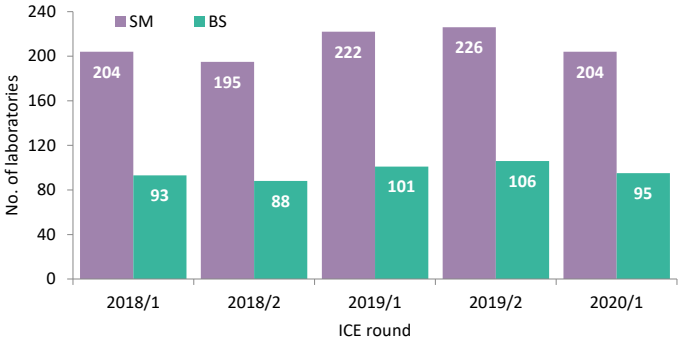
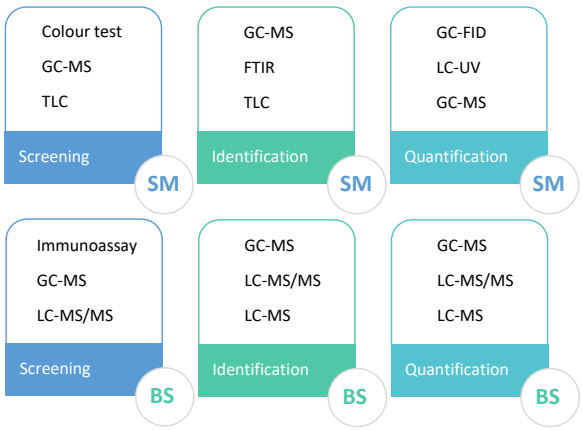


Figure 4: Top 3 analytical techniques most frequently used by laboratories in the SM and BS test groups, in descending order, for screening, identification and quantification of the test samples.



Analysis of ICE test samples

Laboratories participating in the ICE programme are required to analyse four test samples in the seized materials (SM) group and/or four test samples in the biological specimens (BS) group for the substances listed in the ICE menu. This menu covers controlled substances, new psychoactive substances (NPS) and adulterants/diluents that are 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 are designed to mimic actual casework encountered by forensic laboratories. In the SM group, the test samples are prepared in the Laboratory and Scientific Service (LSS) of UNODC using donations of seized materials from Member States. The BS test samples are prepared by spiking controlled substances, their metabolites and related compounds into blank 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). Figure 4 shows the top 3 analytical techniques most frequently used by laboratories in the SM and BS test groups, in descending order, for screening, identification and quantification of the test samples.

By comparing analytical techniques used, laboratories can 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 strongly encouraged to report the purity or concentrations of the controlled drugs present in the test samples.

Results from ICE rounds 2019/2 and 2020/1

Qualitative analysis

Within the SM group, correct identification of all four test samples by laboratories decreased from 94% in round 2019/2 to 83% in round 2020/1. This was mainly due to difficulties encountered by a number of participants with test sample 2020/1 SM-3 which was a plant material containing caffeine. 23 laboratories failed to identify caffeine while 8 laboratories did not analyse this test sample.

Aside from this test sample, the number of false positive and false negative results reported in rounds 2019/2 and 2020/1

were generally low. The test sample 2020/1 SM-2 containing cocaine had the highest number of false positive results reported (9 laboratories). Some examples of false positive results reported in this sample were aminorex, caffeine, lidocaine and procaine. Detailed performance indicators of participants for qualitative analysis in the SM group are shown in figure 5.

Within the BS group, correct identification of all four test samples by laboratories increased slightly from 70% in round 2019/2 to 74% in round 2020/1. The results were good in both rounds for most test samples, given the inherently higher level of difficulty in the analysis of the low concentrations of possibly multiple drugs in biological specimens and the complexity of the matrices.

It is encouraging to note that a significant reduction in the total number of false positives and false negatives reported was also observed in round 2020/1 from the previous round. The test sample 2019/2 BS-4 containing temazepam had the highest number of false positive results reported (14 laboratories). Some examples of false positive results reported in this sample were benzoylecgonine, diazepam, metamfetamine, nordazepam, oxazepam and zolpidem.

A considerable number of laboratories did not perform any analysis on test samples 2019/2 BS-3 (11 laboratories) and 2019/2 BS-4 (13 laboratories). Laboratories are reminded that test samples can contain any of the substances in the ICE menu and should analyse for their presence. Detailed performance indicators of participants for qualitative analysis in the BS group are shown in figure 6.

Laboratories reporting false positive or false negative results are reminded to investigate the reasons for this and take corrective actions as soon as possible in order to continuously improve performance.

Figure 5: Seized Materials group: Performance of participants in qualitative analysis in ICE 2019/2 and 2020/1.

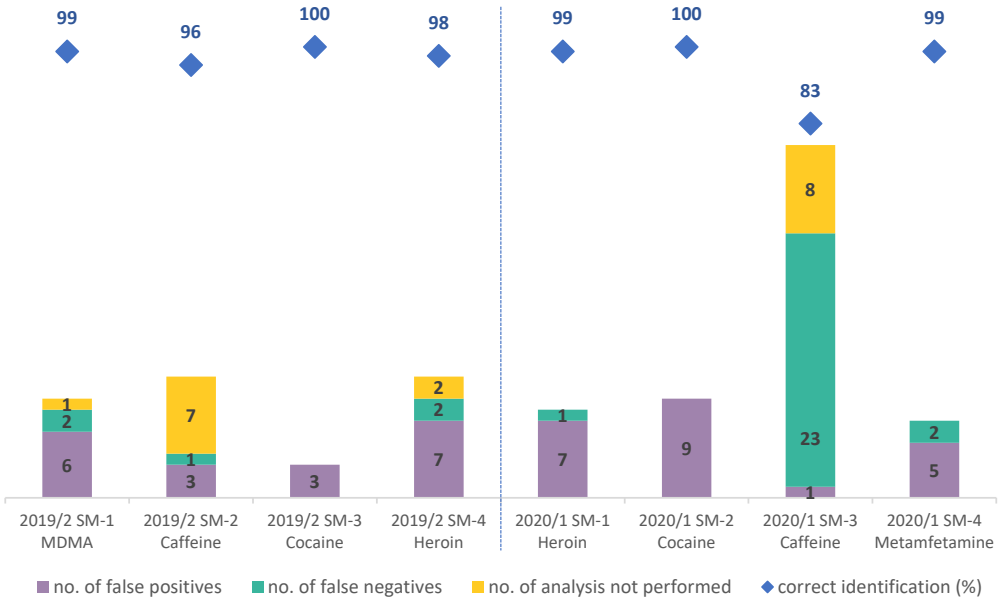
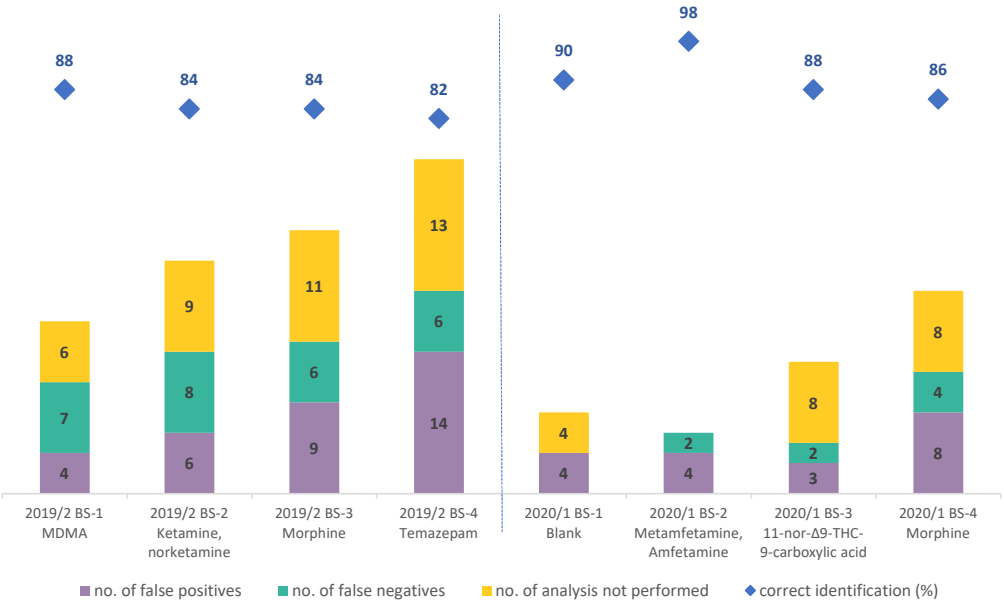


Figure 6: Biological Specimens group: Performance of participants in qualitative analysis in ICE 2019/2 and 2020/1.



Quantitative analysis

Quantification of test samples within the ICE programme is not compulsory, although laboratories are encouraged to quantify all test samples (depending on jurisdictional requirements) in order to get a better measure of their performance over time. On average, the percentages of participants in the SM and BS group quantitating at least 1 drug in both rounds are 70% and 42% respectively.

Z-scores are a statistical parameter used in proficiency tests and collaborative exercises as a measure of performance in quantitative analysis and can be interpreted by ICE participants in line with ISO 13528:2015 (section 9.4.2) as follows:

$\|z\| \leq 2.00$	is considered to be acceptable
$2.00 < \|z\| < 3.00$	is considered to be questionable (or warning signal)
$\|z\| \geq 3.00$	is considered to be unacceptable (or action signal)

According to the recommendations in ISO 13528:2015, an unacceptable *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. Tables 1 and 2 provide information on the number of laboratories who obtained acceptable, questionable and unacceptable *z*-scores for all test

Table 1: Seized Materials group: Performance of participants in quantitative analysis in ICE 2019/2 and 2020/1.

Test sample	Purity (%)	Robust average reported (%)	$\|z\| \leq 2$ Acceptable (%)	$2 < \|z\| < 3$ Questionable (%)	$\|z\| \geq 3$ Unacceptable (%)
2019/2 SM-1 MDMA	13.6	13.7	86	7	7
2019/2 SM-2 Caffeine	N/A	N/A	N/A	N/A	N/A
2019/2 SM-3 Cocaine	38.4	37.4	85	5	10
2019/2 SM-4 Heroin	21.1	18.9	88	6	6
2020/1 SM-1 Heroin	42.9	41.8	84	6	10
2020/1 SM-2 Cocaine	48.3	48.1	86	6	8
2020/1 SM-3 Caffeine	N/A	N/A	N/A	N/A	N/A
2020/1 SM-4 Metamfetamine	19.6	19.6	82	5	13

Table 2: Biological Specimens group: Performance of participants in quantitative analysis in ICE 2019/2 and 2020/1.

Test sample	Concentration (ng/ml)	Robust average reported (ng/ml)	$\|z\| \leq 2$ Acceptable (%)	$2 < \|z\| < 3$ Questionable (%)	$\|z\| \geq 3$ Unacceptable (%)
2019/2 BS-1 MDMA	920	785	95	0	5
2019/2 BS-2 Ketamine	920	955	83	7	10
Norketamine	1150	885	100	0	0
2019/2 BS-3 Morphine	690	634	86	3	11
2019/2 BS-4 Temazepam	920	895	84	6	10
2020/1 BS-1 Blank	N/A	N/A	N/A	N/A	N/A
2020/1 BS-2 Metamfetamine	920	876	93	7	0
Amfetamine	345	346	83	7	10
2020/1 BS-3 11-nor-Δ9-THC-9-carboxylic acid	345	270	92	4	4
2020/1 BS-4 Morphine	920	788	81	13	6

samples in the ICE rounds 2019/2 and 2020/1. Graphical plots of *z*-scores are also included in the summary reports after each ICE round and a typical plot for ICE 2020/1 BS-4 (Morphine) is shown in figure 7. Participants who obtained questionable or unacceptable *z*-scores are highlighted in amber and red respectively.

Tables 3 and 4 show quantitative performance of laboratories who obtained at least an action or a warning signal in both ICE 2019/2 and 2020/1 rounds. These laboratories whose results are classified as unacceptable or questionable in two successive rounds should investigate the cause and take appropriate corrective action as soon as possible.

Table 3: Seized Materials group: Participants in quantitative analysis who obtained at least an action or a warning signal in both ICE 2019/2 and 2020/1 rounds.

Seized materials group	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8
2019/2 round code	ICNCMG	DSEBBS	IWWWZH	AK3UYT	MOGXMB	BXCKYU	ZNRYRN	48PXX4
2020/1 round code	3CAJ33	EQNEIU	FTFGF7	GIG3NC	JYYOJY	NT6XKR	PKIAAH	T6MTE2
2019/2 SM-1 MDMA	●	●	●	●	●	●	●	●
2019/2 SM-3 Cocaine	●	●	●	●	●	●	●	●
2019/2 SM-4 Heroin	●	●	●	●	-	●	●	●
2020/1 SM-1 Heroin	●	●	●	●	-	●	●	●
2020/1 SM-2 Cocaine	●	●	●	●	●	●	●	●
2020/1 SM-4 Metafetamine	●	●	●	●	●	●	●	●

Note: acceptable results (blue), questionable results (amber), unacceptable results (red), quantitation not performed (-).

Figure 7: *Z*-score plot for 2020/1 BS-4 (Morphine). Each bar represents the *z*-score of a laboratory who performed quantitation and the lines indicates the levels below and above, where *z*-scores are considered acceptable, questionable and unacceptable.

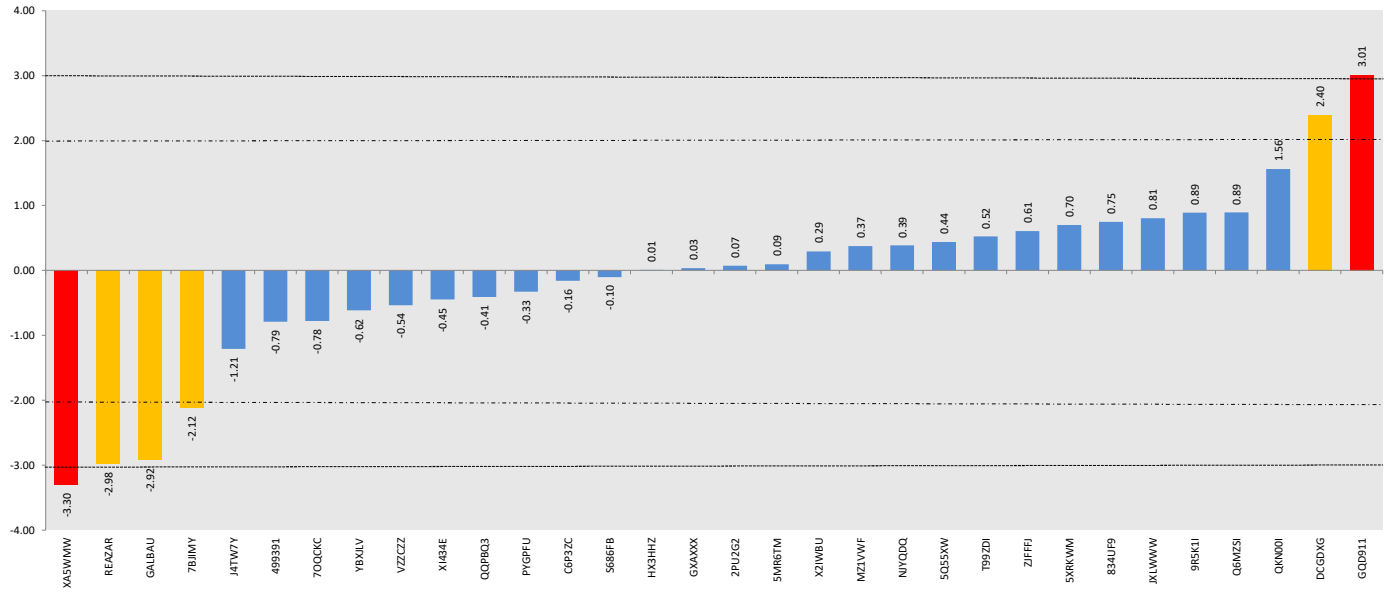


Table 4: Biological Specimens group: Participants in quantitative analysis who obtained at least an action or a warning signal in both ICE 2019/2 and 2020/1 rounds.

Seized materials group	Lab 1
2019/2 round code	1WXG1M
2020/1 round code	PYGPFU
2019/2 BS-1 MDMA	●
2019/2 BS-2 Ketamine	●
2019/2 BS-2 Norketamine	●
2019/2 BS-3 Morphine	●
2019/2 BS-4 Temazepam	●
2020/1 BS-2 Metamfetamine	●
2020/1 BS-2 Amfetamine	●
2020/1 BS-3 11-nor-Δ9-THC-9-carboxylic acid	●
2020/1 BS-4 Morphine	●

Note: acceptable results (blue), questionable results (amber), unacceptable results (red).

Technical support from UNODC

Through the ICE programme, participating laboratories have the possibility to identify areas where improvements of their work can be made to enhance their capacities in analysis and identification of controlled drugs and new psychoactive substances as well as in the generation of quality results. Should further support from UNODC be required, participants are welcome to contact the UNODC ICE team at unodc-ice@un.org.

New Psychoactive Substances (NPS)

During the 2019/2 and 2020/1 rounds of ICE, participants provided 489 reports of the identification of 246 different NPS in their laboratories. As illustrated in figure 8, synthetic cannabinoids corresponded to 27% of all reports followed by synthetic cathinones with 25%. 19% of the substances reported belong to a group, known as "other substances". These substances do not fall under the main categories listed in figure 8.

The most commonly reported substances during 2020 were ketamine and the synthetic cannabinoids, 5F-MDMB-PICA, 4F-MDMB-BINACA and MDMB-4en-PINACA. While ketamine is currently not under international control, 5F-MDMB-PICA and 4F-MDMB-BINACA are currently controlled under Schedule II of the Convention on Psychotropic Substances, 1971. MDMB-4en-PINACA was also placed under international control under Schedule II of the Convention on Psychotropic Substances, 1971, at the 64th session of the Commission on Narcotic Drugs in April 2021. This decision will come into force on 7 December 2021.

In order to identify the NPS with the greatest potential for harm, it is necessary to collect and analyse toxicology data from adverse events due to the use of NPS. UNODC, in collaboration with the International Association of Forensic Toxicologists (TIAFT), has developed an online tool for the collection and sharing of such toxicology data. This online portal (www.unodc.org/tox) allows the forensic community to identify and anticipate threats due to NPS; and to formulate measures needed to address gaps in analytical preparedness, where necessary.

ICE participants are encouraged to use the UNODC early warning advisory on NPS, accessible through their ICE portal accounts, to submit reports of NPS that they detect. This information enables UNODC to more effectively tailor the assistance it provides to forensic laboratories.

Acknowledgements

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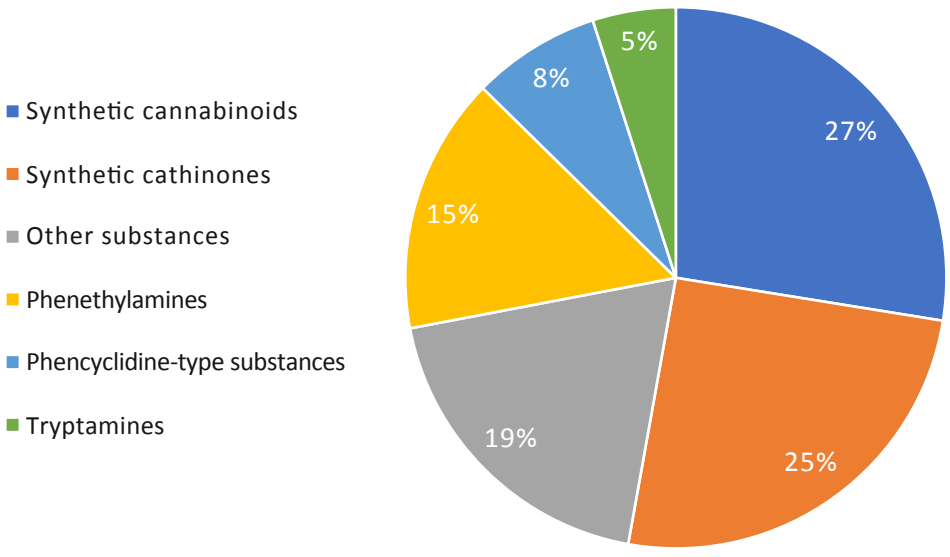
Figure 8: NPS reported by ICE laboratories during the 2019/2 and 2020/1 rounds of ICE



International Panel of Forensic Experts (Mr. Benoit Archambault, Mr. Elvio Dias Botelho, Prof. Heesun Chung, Prof. Niamh Nic Daéid, Mr. Scott Oulton, Ms. Catherine Quinn, Prof. Franco Tagliaro and Dr. Angeline Tiong Whei Yap), and other UNODC LSS staff members (Dr. Conor Crean and Ms. Romana Luger) are gratefully acknowledged.

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If you have comments or questions related to this report, please e-mail us at unodc-ice@un.org. Additional information on the ICE programme and other UNODC Laboratory and Scientific Service (LSS) programmes can be found via the internet at www.unodc.org/lab, or by writing to UNODC LSS at the Vienna International Centre, P.O. Box 500, A-1400 Vienna, Austria.

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Registration in the UNODC ICE programme can be requested via email to unodc-ice@un.org
If required, in-person training workshops can be arranged subject to the availability of funds.
Requests for UNODC training services on identification of drugs and precursors and development of quality assurance systems should be channelled through the office of UNODC's Country/Regional Representative. In countries/regions where no such office exists, please contact UNODC Laboratory and Scientific Service at:

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