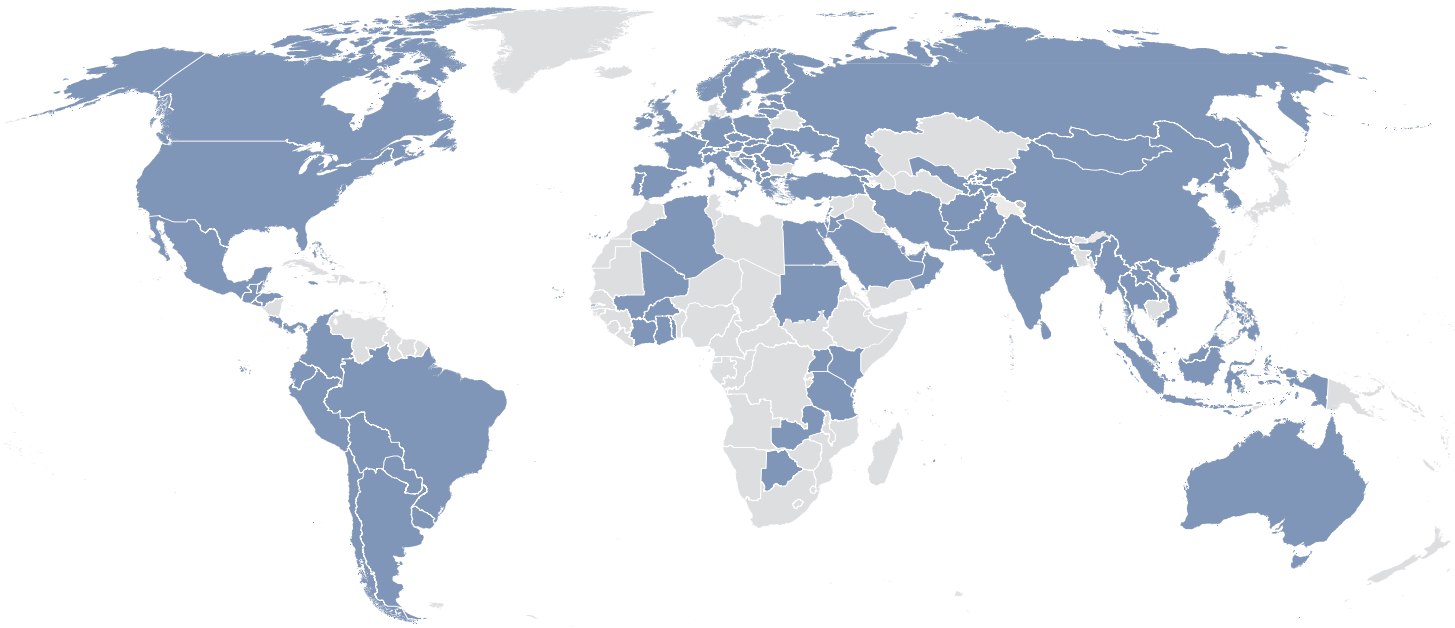
UNODC
United Nations Office on Drugs and Crime

INTERNATIONAL COLLABORATIVE EXERCISES

Drug Analysis 2019

ICE Rounds 2018/2 and 2019/1

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



The boundaries shown 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 Falksland Islands (Malvinas).

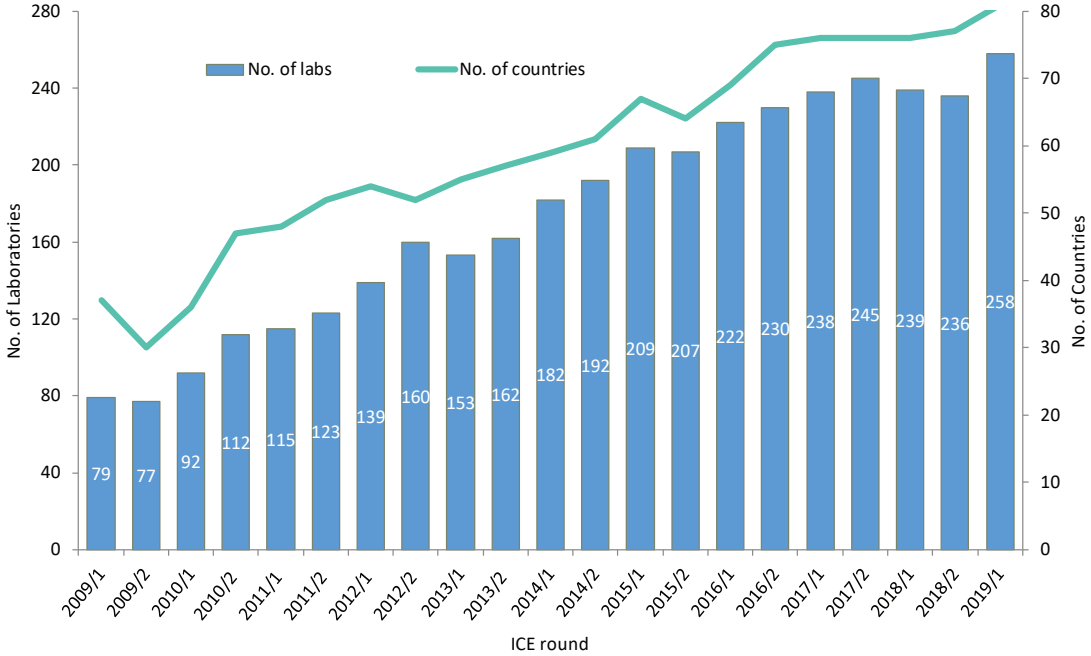
Introduction

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.

Figure 2: Growth of the ICE 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 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

Figure 3: Number of laboratories that have participated in the ICE programme since 2017/1.

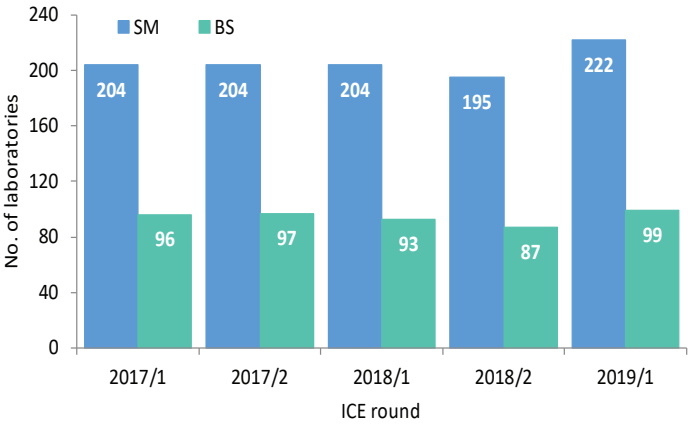
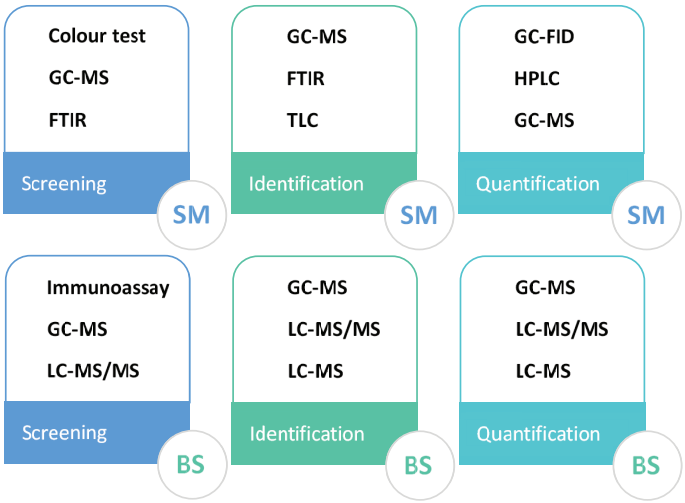


Figure 4: Top 3 analytical techniques used by laboratories in the SM and BS test groups for screening, identification and quantification of the test samples.



the UNODC website. These summary reports allow participants to evaluate their performance while maintaining confidentiality.

Participation

Since its inception in 1995, the number of laboratories worldwide who participate in the ICE programme has continued to increase. There are now 258 laboratories from 81 Member States actively participating in the programme, representing a 9% increase in year on year participation. Figure 2 shows the growth of 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 2017/1 up to the most recent 2019/1 round of the programme.

The continued increase in participation during 2019 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 science networks and with support from UNODC regional and country programmes.

It is recognised that some participants continue to have difficulties with obtaining import authorization for the test samples and the reference samples that contained internationally controlled substances. This has caused some delays in sending test samples and in the submission of results from a small number of laboratories.

There were also some cases where test samples were returned to UNODC as they could not be delivered. Hence, it would be helpful for participants to nominate a dedicated focal point in the country for obtaining import authorisation as well as to facilitate receipt of the test samples. It reinforces the importance for laboratories to dedicate specific resources, human and financial, for the participation of proficiency tests and quality assurance management.

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 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 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. The BS test samples are prepared by spiking controlled substances, their metabolites and related compounds into urine blanks.

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 used by laboratories in the SM and BS test groups for screening, identification and quantification of the test samples.

By comparing analytical techniques used, 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.

Results from ICE rounds 2018/2 and 2019/1
Qualitative analysis

In general, the performance of participants in both SM and BS groups improved from round 2018/2 to 2019/1. For BS round 2018/2 which included mephedrone, a relatively new substance in the ICE menu, 41% of BS participating laboratories had correctly

identified all 4 test samples. In round 2019/1, 79% of BS participating laboratories were able to correctly identify all 4 test samples. For the SM group, correct identification of all test samples by laboratories increased from 89% in round 2018/2 to 96% in round 2019/1.

For the SM group, the detailed performance indicators of participants for qualitative analysis are shown in figure 5. 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 test sample.

While the number of false positive and false negative results reported through 2018/2 and 2019/1 was low in most test samples, it is notable that there were 13 false negative results for 2018/2 SM-4 (amfetamine) and 11 false positive results for 2019/1 SM-2 (morphine). Some examples of false positive results for the amphetamine test sample include metamfetamine, cocaine and ephedrine; and as for the morphine test sample, heroin, hydromorphone and butabarbital were among the false positive substances reported.

Within the BS group, the detailed performance indicators of participants for qualitative analysis are shown in figure 6. 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 possibly multiple drugs in biological specimens and the complexity of the matrices. However, the number of false positives reported has increased slightly from the previous year. In particular, there were 10 false positive results for 2018/2 BS-2 (MDA) and of these 6 laboratories had reported amfetamine.

The number of false negative results for buprenorphine, mephedrone and morphine in 2018/2 BS-1, 2018/2 BS-3 and 2018/2 BS-4 respectively are concerning. Laboratories that reported false positive or false negative results should investigate the reasons for this and corrective actions should be taken as soon as possible in order to continuously improve performance.

Figure 5: Seized Materials group: Performance of participants in qualitative analysis in ICE 2018/2 and 2019/1.

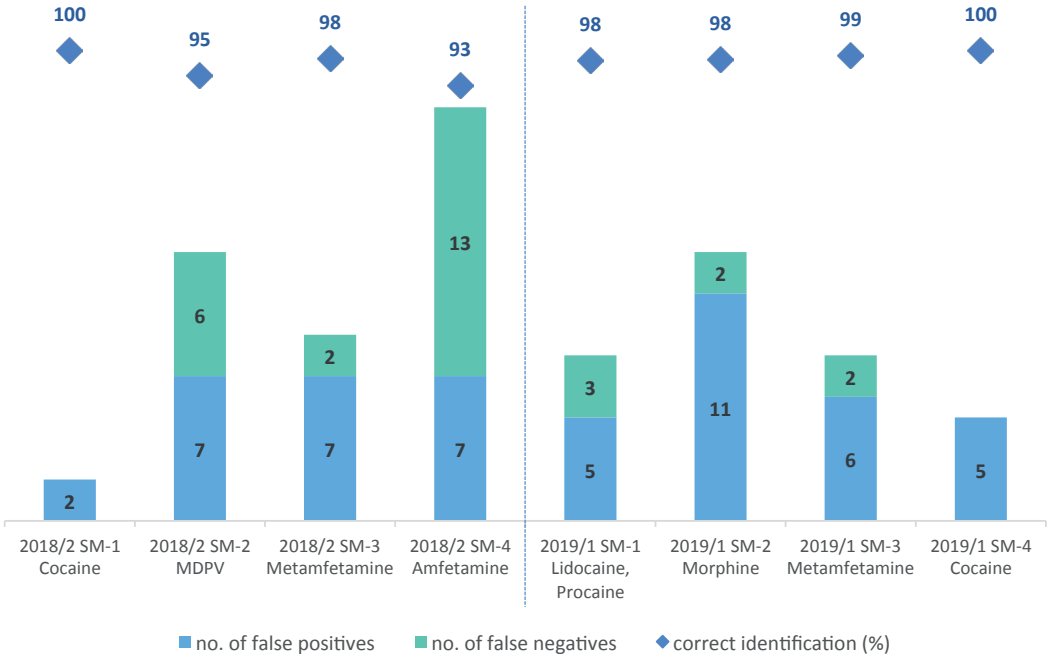
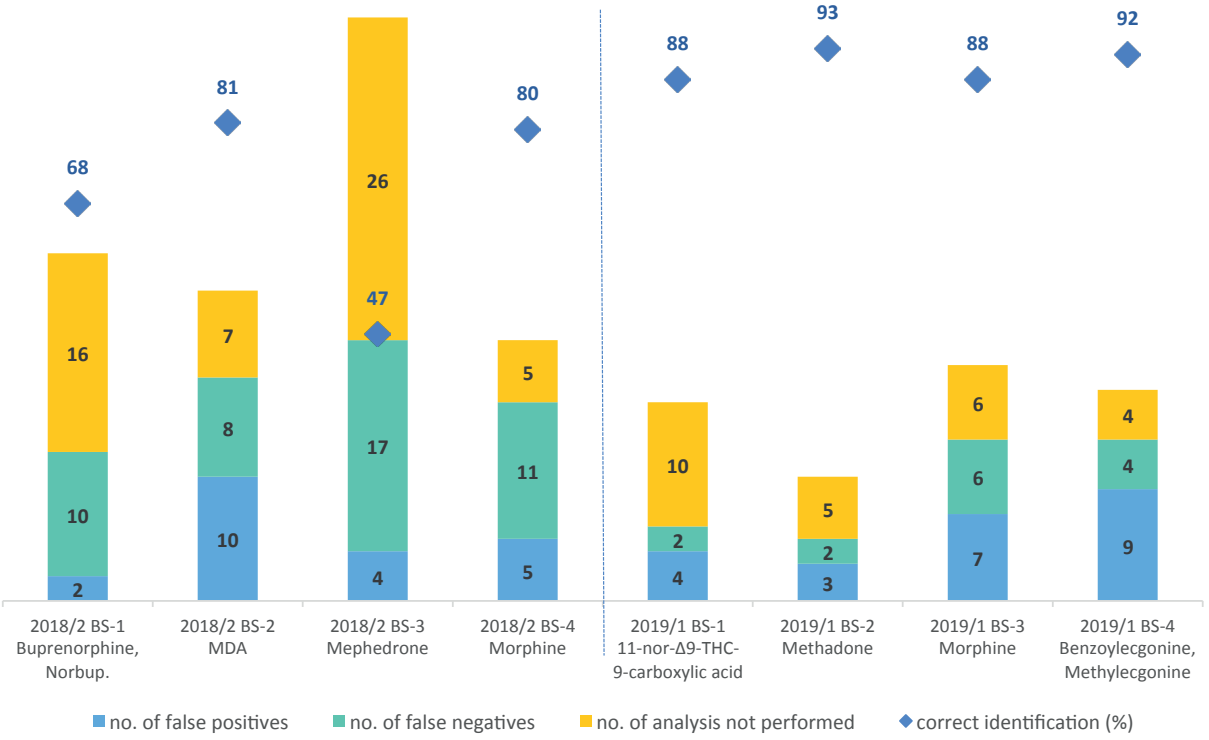


Figure 6: Biological Specimens group: Performance of participants in qualitative analysis in ICE 2018/2 and 2019/1.



A considerable number of laboratories did not perform any analysis on test samples 2018/2 BS-1 (16 laboratories) and 2018/2 BS-3 (26 laboratories). Laboratories are reminded that test samples can contain any of the substances in the ICE menu and are encouraged to analyse them.

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 SM and BS group participants quantitating at least 1 drug in both rounds are 74% and 45% 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 samples in the ICE rounds 2018/2 and 2019/1. Graphical plots of *z*-scores are also included in the summary reports after each ICE round and a typical plot for ICE 2019/1 BS-3 (Morphine) is shown in figure 7. Participants who obtained questionable or unacceptable *z*-scores are highlighted in amber and red respectively.

Table 1: Seized Materials group: Performance of participants in quantitative analysis in ICE 2018/2 and 2019/1.

Test sample	Purity (%)	Robust average reported (%)	$\|z\| \leq 2$ Acceptable (no. of labs)	$2 < \|z\| < 3$ Questionable (no. of labs)	$\|z\| \geq 3$ Unacceptable (no. of labs)
2018/2 SM-1 Cocaine	22.7	22.4	109 (77%)	14 (10%)	18 (13%)
2018/2 SM-2 MDPV	9.8	9.8	45 (82%)	4 (7%)	6 (11%)
2018/2 SM-3 Metamfetamine	19.6	19.3	89 (80%)	6 (5%)	16 (15%)
2018/2 SM-4 Amfetamine	7.1	6.6	79 (83%)	7 (7%)	9 (10%)
2019/1 SM-1 Lidocaine, Procaine	N/A	N/A	N/A	N/A	N/A
2019/1 SM-2 Morphine	14.2	13.6	70 (84%)	7 (9%)	6 (7%)
2019/1 SM-3 Metamfetamine	9.4	9.1	90 (77%)	10 (9%)	17 (14%)
2019/1 SM-4 Cocaine	14.4	14.2	123 (82%)	8 (5%)	19 (13%)

Table 2: Biological Specimens group: Performance of participants in quantitative analysis in ICE 2018/2 and 2019/1.

Test sample	Concentration (ng/ml)	Robust average reported (ng/ml)	$\|z\| \leq 2$ Acceptable (no. of labs)	$2 < \|z\| < 3$ Questionable (no. of labs)	$\|z\| \geq 3$ Unacceptable (no. of labs)
2018/2 BS-1 Buprenorphine	570	626	21 (87%)	0 (0%)	3 (13%)
Norbuprenorphine	580	553	14 (87%)	2 (13%)	0 (0%)
2018/2 BS-2 Tenamfetamine (MDA)	1150	1080	27 (87%)	2 (6.5%)	2 (6.5%)
2018/2 BS-3 Mephedrone	479	329	17 (85%)	0 (0%)	3 (15%)
2018/2 BS-4 Morphine	580	494	30 (84%)	3 (8%)	3 (8%)
2019/1 BS-1 11-nor-Δ9-THC-9-carboxylic acid	690	626	29 (78%)	5 (14%)	3 (8%)
2019/1 BS-2 Methadone	1380	1299	30 (88%)	2 (6%)	2 (6%)
2019/1 BS-3 Morphine	580	527	34 (87%)	2 (5%)	3 (8%)
2019/1 BS-4 Benzoylecgonine	1150	1151	30 (81%)	5 (14%)	2 (5%)
Methylecgonine	690	582	20 (84%)	2 (8%)	2 (8%)

Figure 7: *Z*-score plot for 2019/1 BS-3 (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.

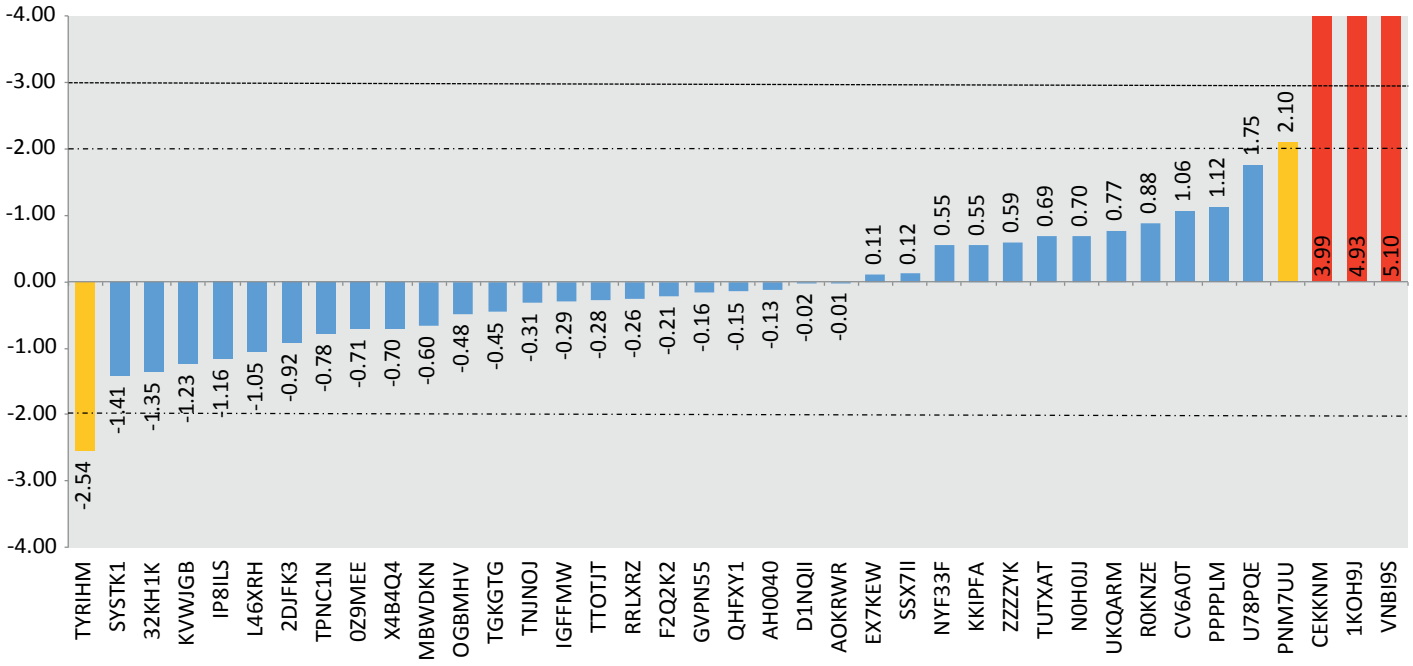


Table 3: Seized Materials group: Participants in quantitative analysis who obtained at least an action or a warning signal in both ICE 2018/2 and 2019/1 rounds. Note: acceptable results (blue), questionable results (amber), unacceptable results (red).

Seized materials group	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	Lab 9	Lab 10	Lab 11
2018/2 round code	V4HVM5	07RNYQ	7JGGMW	T0PFVQ	14YQYB	QMQ1QP	CPBBXT	555HC5	PUTLIT	Y3XX1B	HATVYH
2019/1 round code	0E49T9	2MPUPL	2XGWKJ	2XXLPG	54CQ2S	5HWYZN	6666WM	9YCKGZ	BIA0EZ	BMPXHL	BWSSKK
2018/2 SM-1 Cocaine	●	●	●	●	●	●	●	●	●	●	●
2018/2 SM-2 MDPV	●	●	–	–	●	●	–	●	●	●	●
2018/2 SM-3 Metamfetamine	●	●	–	–	●	●	●	●	●	●	●
2018/2 SM-4 Amfetamine	●	●	–	–	●	●	–	●	●	●	●
2019/1 SM-2 Morphine	●	–	–	–	–	●	●	●	●	●	●
2019/1 SM-3 Metamfetamine	●	●	–	–	●	●	●	●	●	●	●
2019/1 SM-4 Cocaine	●	●	●	●	●	●	●	●	●	●	●

Seized materials group	Lab 12	Lab 13	Lab 14	Lab 15	Lab 16	Lab 17	Lab 18	Lab 19	Lab 20	Lab 21	Lab 22
2018/2 round code	2P2PU2	8XQEGQ	7Z33IM	HDYYZY	AAPSTZ	Y68SYY	YSSSDR	A8R888	GU6GGK	ANAAAW	CELGEE
2019/1 round code	E44WTB	FFUFOA	GZFJAG	HP2FIF	I3H5RP	KAPAAS	KII7PP	LRPCVG	OIMJHC	PIRYEY	XPYAOU
2018/2 SM-1 Cocaine	●	●	●	●	●	●	●	●	●	●	●
2018/2 SM-2 MDPV	●	●	–	●	–	–	●	●	–	–	–
2018/2 SM-3 Metamfetamine	●	●	●	●	●	●	●	●	●	●	–
2018/2 SM-4 Amfetamine	–	●	●	●	●	●	●	●	–	–	–
2019/1 SM-2 Morphine	●	●	●	●	–	–	●	●	–	●	–
2019/1 SM-3 Metamfetamine	●	●	●	●	●	●	●	–	●	●	–
2019/1 SM-4 Cocaine	●	●	●	●	●	●	●	●	●	●	●

Tables 3 and 4 show quantitative performance of laboratories who obtained at least an action or a warning signal in both ICE 2018/2 and 2019/1 rounds. These laboratories whose results are classified as unacceptable or questionable in two successive rounds should investigate the cause and take appropriate corrective action, with support from UNODC, if required.

New Psychoactive Substances (NPS)

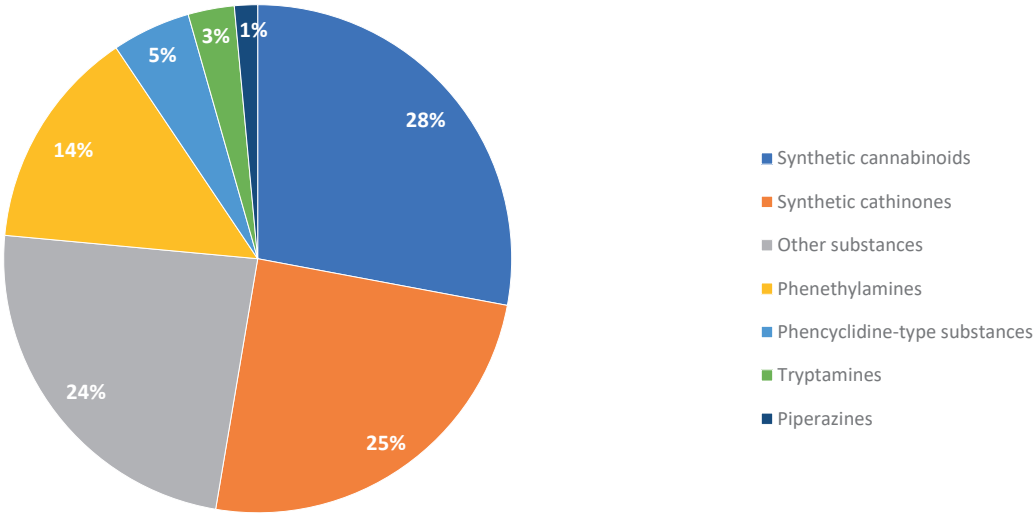
During the 2018/2 and 2019/1 rounds of ICE, participants provided 350 reports of the identification of 179 different NPS in their laboratories. As illustrated in Figure 8, synthetic cannabinoids corresponded to 28% of all reports followed by synthetic cathinones with 25%. 24% of the substances reported belong to a group that could not be classified structurally, known as "other substances".

The most commonly reported substances during 2019 were the synthetic cannabinoids, 5F-MDMB-PINACA and 5F-MDMB-PICA, followed by the synthetic cathinones, ephylone and *N*-ethylhexedrone. 5F-MDMB-PINACA and ephylone are currently controlled under Schedule II of the Convention on Psychotropic Substances, 1971. The other two substances were also placed under international

Table 4: Biological Specimens group: Participants in quantitative analysis who obtained at least an action or a warning signal in both ICE 2018/2 and 2019/1 rounds. Note: acceptable results (blue), questionable results (amber), unacceptable results (red).

Seized materials group	Lab 1	Lab 2	Lab 3	Lab 4
2018/2 round code	MAH4QP	YCH0DH	DDY2DD	A0OXAA
2019/1 round code	N0H0JJ	PNM7UU	SYSTK1	U78PQE
2018/2 BS-1 Buprenorphine	●	●	●	●
2018/2 BS-1 Norbuprenorphine	●	–	●	–
2018/2 BS-2 MDA	●	–	●	●
2018/2 BS-3 Mephedrone	●	–	●	●
2018/2 BS-4 Morphine	●	–	●	●
2019/1 BS-1 11-nor-Δ9-THC-9-carboxylic acid	●	●	●	●
2019/1 BS-2 Methadone	●	●	●	●
2019/1 BS-3 Morphine	●	●	●	●
2019/1 BS-4 Benzoylecgonine	●	●	●	●
2019/1 BS-4 Methylecgonine	●	–	●	–

Figure 8: NPS reported by ICE laboratories during the 2018/2 and 2019/1 rounds of ICE



control under Schedule II of the Convention on Psychotropic Substances, 1971, at the 63rd session of the Commission on Narcotic Drugs in March 2020. These decisions will come into force later this year.

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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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 Section (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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