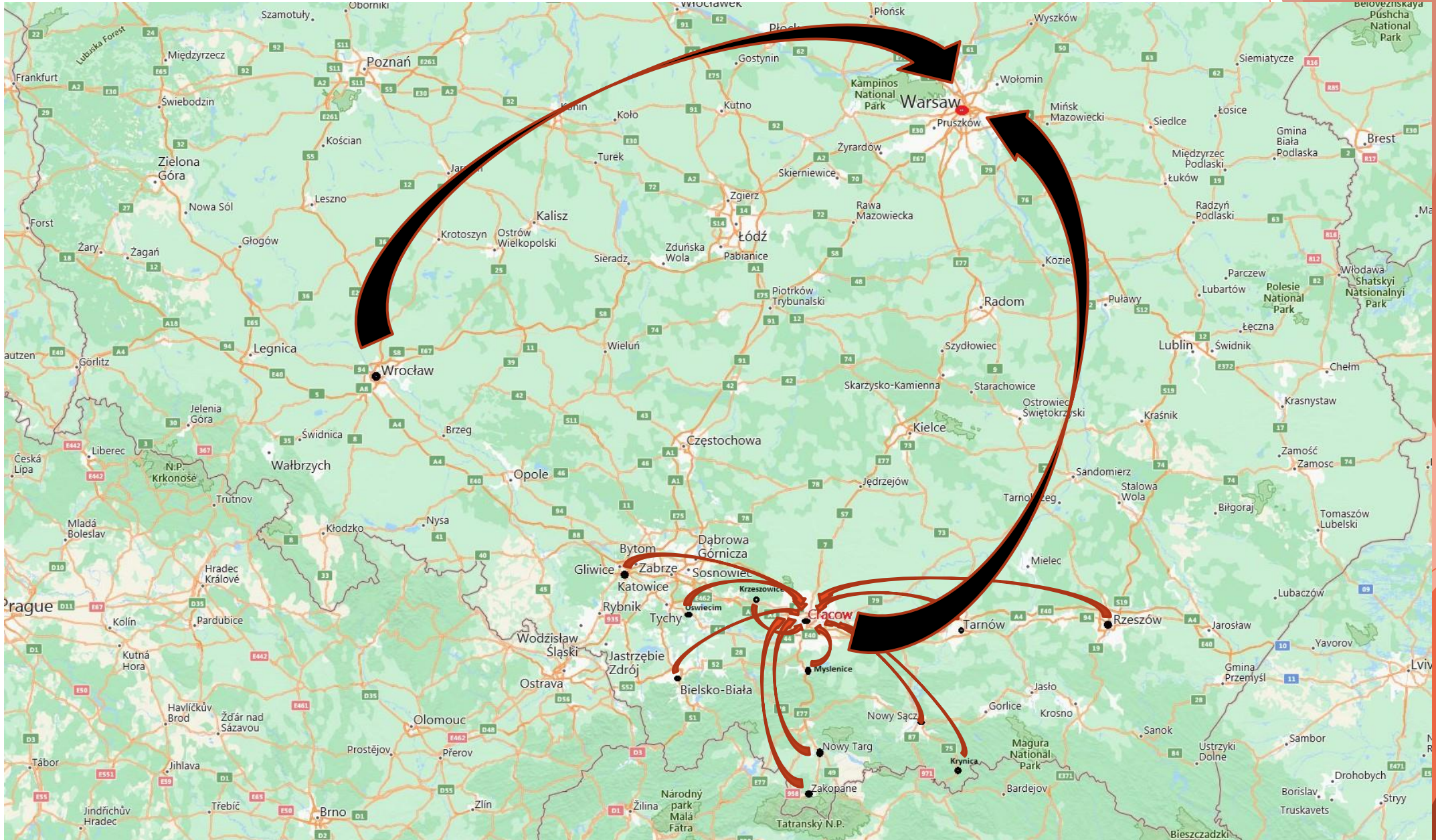




IRMS ANALYSIS DURING THE EUROPEAN GAMES 2023

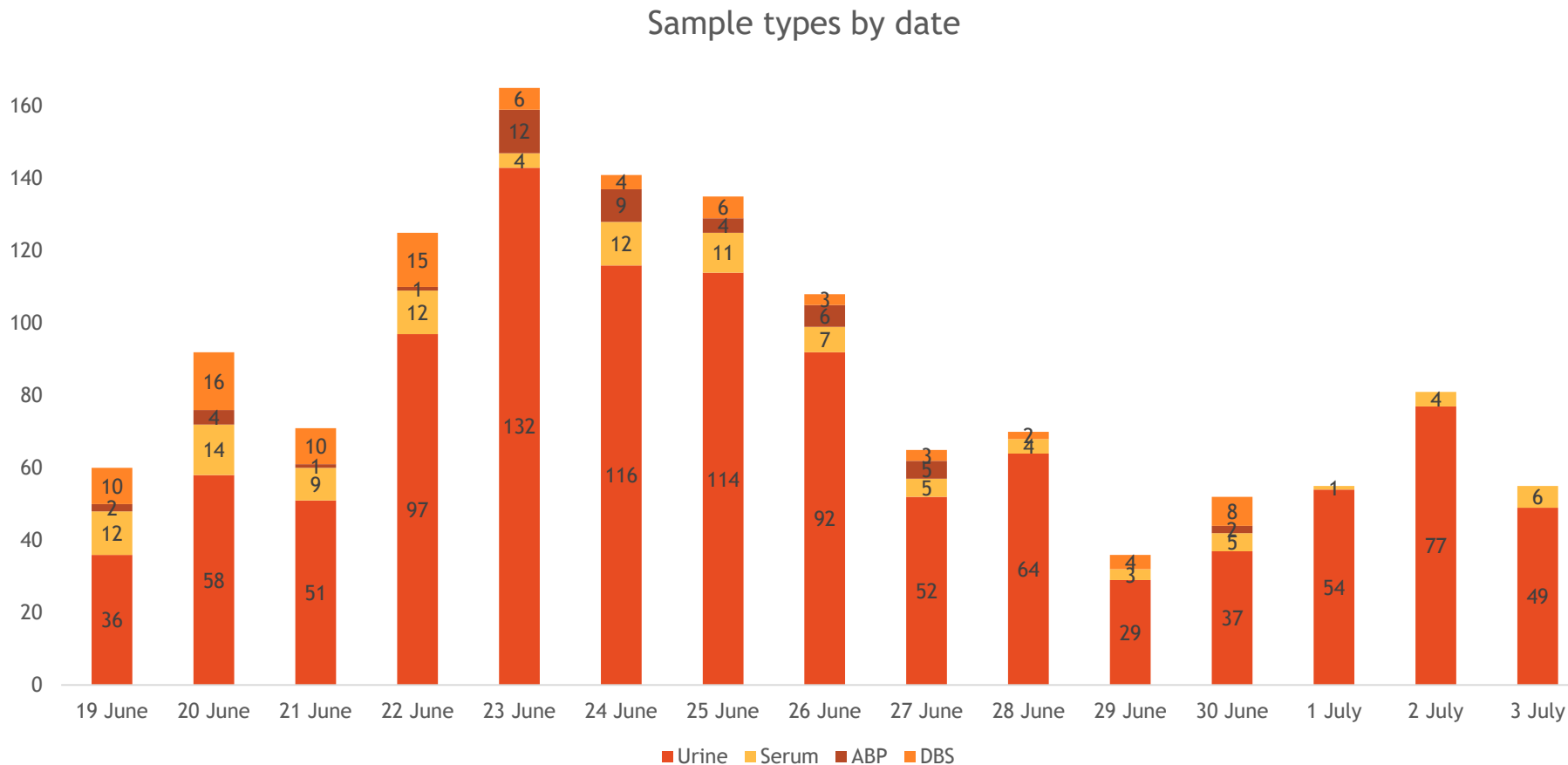
dr Anna Jarek
Polish Anti-Doping Laboratory

EUROPEAN GAMES 21.06 – 02.07. 2023



SAMPLE DISTRIBUTION

Sample types by date				
Date of sample arrival in the Lab.	Urine	Serum	ABP	DBS
19 June	36	11	2	10
20 June	58	18	4	16
21 June	51	7	1	10
22 June	97	7	1	15
23 June	132	20	12	6
24 June	116	19	9	4
25 June	114	14	4	6
26 June	92	18	6	3
27 June	52	7	5	3
28 June	64	5		2
29 June	29	4		4
30 June	37	7	2	8
1 July	54	1		
2 July	77	4		
3 July	49	6		
Total	1058	148	46	87



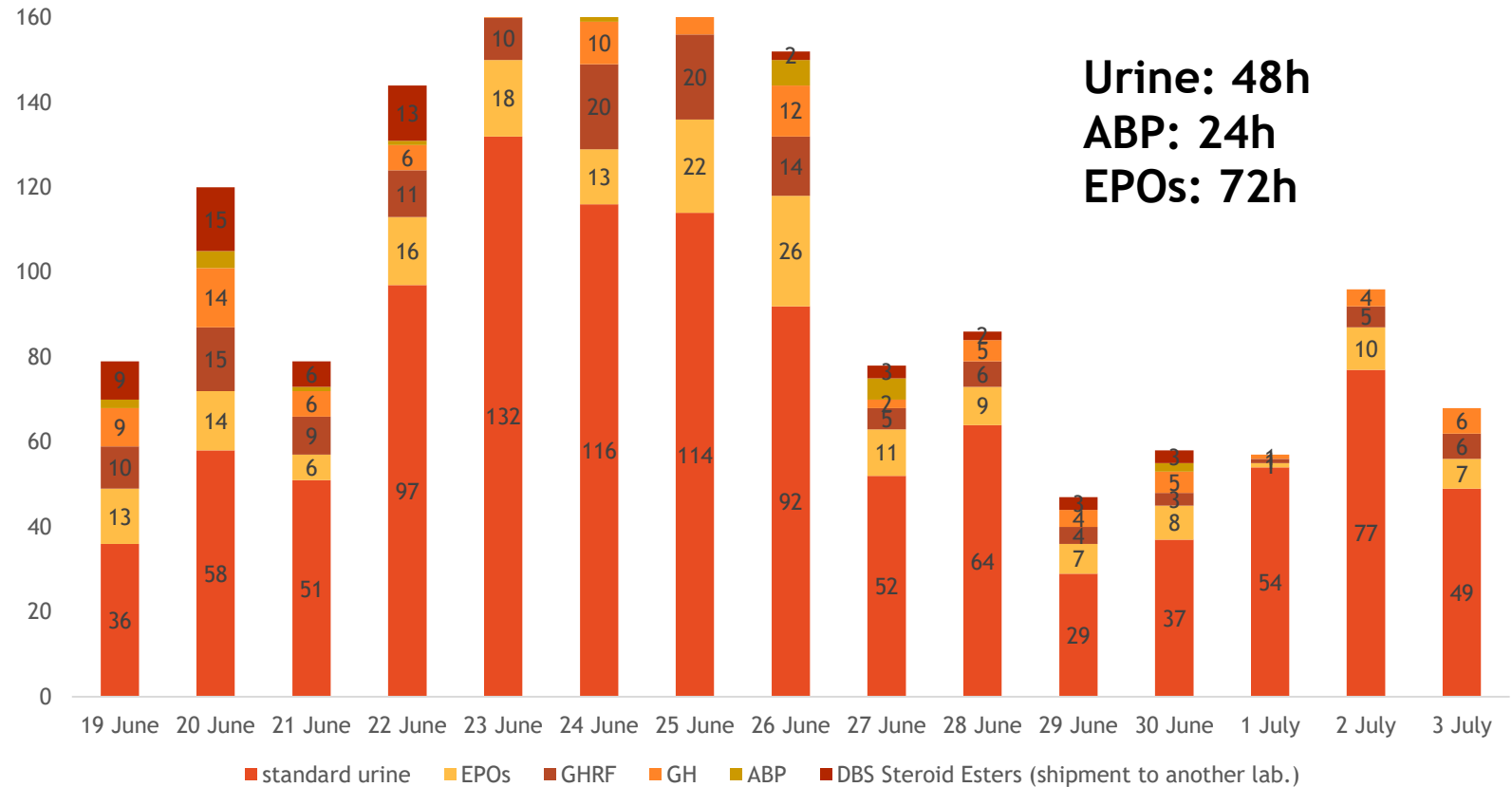
DAILY ANALYSES

daily analyses

Date	standard urine	EPOs	GHRF	GH	ABP	DBS Steroid Esters (shipment to another lab.)
19 June	36	13	10	9	2	9
20 June	58	14	15	14	4	15
21 June	51	6	9	6	1	6
22 June	97	16	11	6	1	13
23 June	132	18	10	8	12	12
24 June	116	13	20	10	9	4
25 June	114	22	20	10	4	3
26 June	92	26	14	12	6	2
27 June	52	11	5	2	5	3
28 June	64	9	6	5		2
29 June	29	7	4	4		3
30 June	37	8	3	5	2	3
1 July	54	1	1	1		
2 July	77	10	5	4		
3 July	49	7	6	6		
Total	1058	181	139	102	46	75

Additionally - 2 blood transfusion

Daily analyses



WORK ORGANIZATION

Work area	Laboratory Staff	temporary staff (students, retirees)	foreign experts (Anti-Doping Laboratory)
Department of quality control (sample distribution area, reception)	4	4	
GC sample preparation	4	4	
GC data analysis	3		2
IRMS and CP sample preparation		1	
IRMS data analysis	2		1
LC sample preparation	2	6	
LC data analysis	5		2
EPO/ABP/GH samples preparation	3		
EPO/ABP/GH data analysis	1		2
ADAMS reporting	2		2
Total	26	15	9

Foreign experts:

Drug Control Centre King's College London

Institute of Doping Analysis and Sports Biochemistry (IDAS) – Dresden, Kreisha

Director Seibersdorf Labor GmbH Doping Control Laboratory, Seibersdorf

Students:

Warsaw University of Technology

University of Warsaw

WORKING HOURS																
Staff	Monday 19.06.2023		Tuesday 20.06.2023		Wednesday 21.06.2023		Thursday 22.06.2023		Friday 23.06.2023		Saturday 24.06.2023		Sunday 25.06.2023		Monday 03.07.2023	Tuesday 04.07.2023
GC Sample preparation																
M. P.	13 - 22/15 - 24				15 - 24 /17 - 2am		15 - 24 /17 - 2am		15 - 24 /17 - 3am		15 - 24 /17 - 3am		15 - 24 /17 - 3am			
M. O.	13 - 22/15 - 24		13 - 22 /15 - 24		15 - 24 /17 - 2am		15 - 24 /17 - 2am		15 - 24 /17 - 2am		15 - 24 /17 - 3am		15 - 24 /17 - 3am			
K. G.	13 - 22/15 - 24		13 - 22 /15 - 24		15 - 24 /17 - 2am		15 - 24 /17 - 2am		15 - 24 /17 - 2am		15 - 24 /17 - 3am		15 - 24 /17 - 3am			
A. S.	13 - 22/15 - 24		13 - 22 /15 - 24		15 - 24 /17 - 2am		15 - 24 /17 - 2am		15 - 24 /17 - 2am		15 - 24 /17 - 3am		15 - 24 /17 - 3am			
Z. Sz.*	9:00 - 18:00															
* CP and IRMS procedures																
GC data analysis																
A. J.	22/24 - 12		22/24 ok 12		22/24 ok 12		22/24 ok 12		22/24 ok 12		22/24 ok 12		22/24 ok 12			
K. Ch.	7:00 - 15:00		9:30 19:00		9:30 19:00		9:00 19:00		9:00 19:00		09:30/11:30 19:00/21		9:00 21:00		9:00 21:00	
M. W-Z.	7:00 - 15:00		9:30 19:00		9:30 19:00		9:00 19:00		9:00 19:00		09:30/11:30 19:00/21		9:00 21:00		9:00 21:00	
A. U-Z.	7:00 - 15:00		9:30 19:00		9:30 19:00		9:00 19:00		9:00 19:00		09:30/11:30 19:00/21		9:00 21:00		9:00 21:00	
M. T.			5:00/6:00 10:00		5:00/6:00 10:00		5:00/6:00 10:00		04:00/6 10:00/12		9:00 21:00		9:00 21:00			
Ekspert 1	7:00 - 15:00		11:00 19:00		11:00 19:00		11:00 19:00		11:00/13:00 19:00/21:00							
Ekspert 2											11:00/13:00 19:00/21:00		11:00/13:00 19:00/21:00			
IRMS analysis																
A. J.	22/24 - 12		22/24 ok 12		22/24 ok 12		22/24 ok 12		21:00 ok 12		21:00 9:00		21:00 ok 12			
A. U-Z.			15:00 0:00		15:00 0:00		15:00 0:00		15:00 0:00							
Ekspert 3																
Service support 8:00-16:00																
Staff	Monday 26.06.2023		Tuesday 27.06.2023		Wednesday 28.06.2023		Thursday 29.06.2023		Friday 30.06.2023		Saturday 01.07.2023		Sunday 02.07.2023		Monday 03.07.2023	Tuesday 04.07.2023
GC Sample preparation																
M. P.	15 - 24 /17 - 3am		13 - 22 /15 - 24		13 - 22 /15 - 24				13 - 22 /15 - 24		13 - 22 /15 - 24				13 - 22 /15 - 24	
M. O.	15 - 24 /17 - 3am		13 - 22 /15 - 24		13 - 22 /15 - 24				13 - 22 /15 - 24		13 - 22 /15 - 24				13 - 22 /15 - 24	
K. G.	13 - 22 /15 - 24		13 - 22 /15 - 24		13 - 22 /15 - 24		13 - 22 /15 - 24				13 - 22 /15 - 24		13 - 22 /15 - 24			
A. S.	15 - 24 /17 - 3am		13 - 22 /15 - 24		13 - 22 /15 - 24*		9:00 15:00						13 - 22 /15 - 24		13 - 22 /15 - 24	
Z. Sz.*	9:00 - 18:00															
* CP and IRMS procedures																
GC data analysis																
A. J.	22/24 ok 12		22/24 ok 12		22/24 ok 12		22/24 ok 12		22/24 ok 12		6:00 6:00(d.n.)		22/24 ok 12		22/24 ok 12	
K. Ch.	9:00 21:00		09:30/11:30 19:00/21		9:30 19:00		9:30 19:00		9:00 21:00				9:00 21:00		9:00 19:00	
M. W-Z.	9:00 21:00		09:30/11:30 19:00/21		9:30 19:00		9:30 19:00		9:00 21:00		9:30 19:00				9:30 19:00	
A. U-Z.	9:00 21:00		9:00 15:00		9:00 15:00		9:00 15:00				9:00 15:00		9:00 15:00		9:00 17:00	
M. T.	9:00 21:00		9:00 15:00		9:00 15:00		9:00 15:00				9:00 15:00		9:00 15:00		9:00 17:00	
Ekspert 1																
Ekspert 2	11:00 19:00		11:00 19:00		11:00 19:00		11:00 19:00									
IRMS analysis																
A. J.	22:00 ok 12		21:00 ok 12		16:00 ok 12		22/24 ok 12		22/24 ok 12		06:00-00:00		00:00-6:00 13:00-00:00		0:00 23:50 00:00 - 18:00	
A. U-Z.			15:00 - 24:00		15:00 - 24:00		15:00 - 24:00		15:00 - 24:00		24-6:00		6:00 15:00		6:00 - 14:00	
Ekspert 3																
Service support 8:00-16:00																

GC/C/IRMS ANALYSIS

Additional analyses not included in the calendar:

IRMS: 57 samples*

Insulin: 16 samples

HBT: 15 samples*

* Does not include potential further analysis requests (e.g., from APMUs).



Who will decide which samples will be subjected to GC/C/IRMS analysis?

How will samples be selected? (discipline?)



Answers

HOW MANY SAMPLES?

27 recommendations for additional analyses

23.06.2023 - 07.07.2023 (25 samples)

19.07.2023 (2 samples)

3 CP

5 SSP - CPR:

1) E > 50 ng/ml

2) T/E > 4

3) A, Etio > 10 000 ng/ml

TA: 17 APMU

After European Games 2023:
2 notifications from APMU

TA

CP

ADAMS

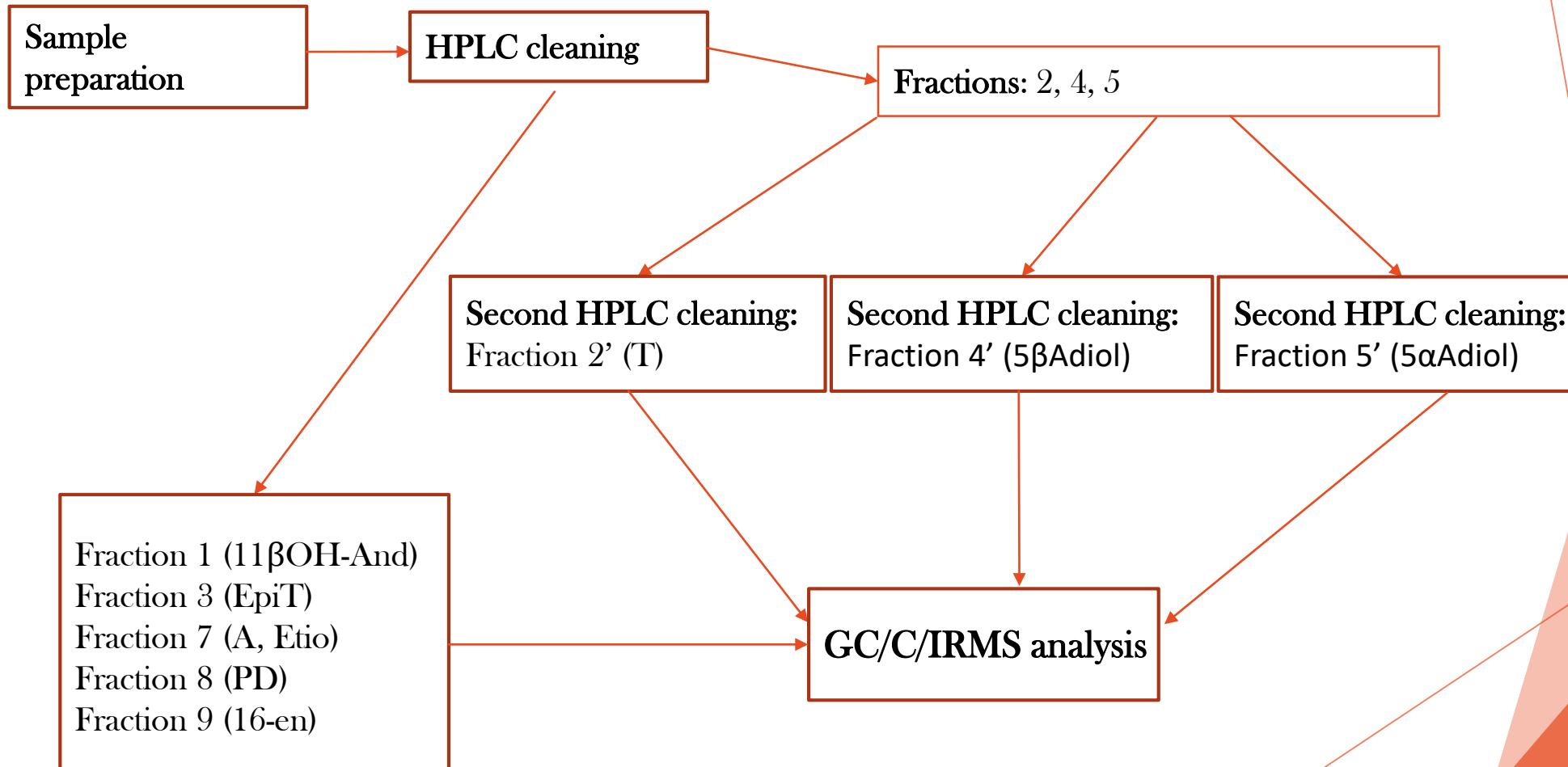
18 IRMS analyses

4 CP do not require further GC/C/IRMS analysis

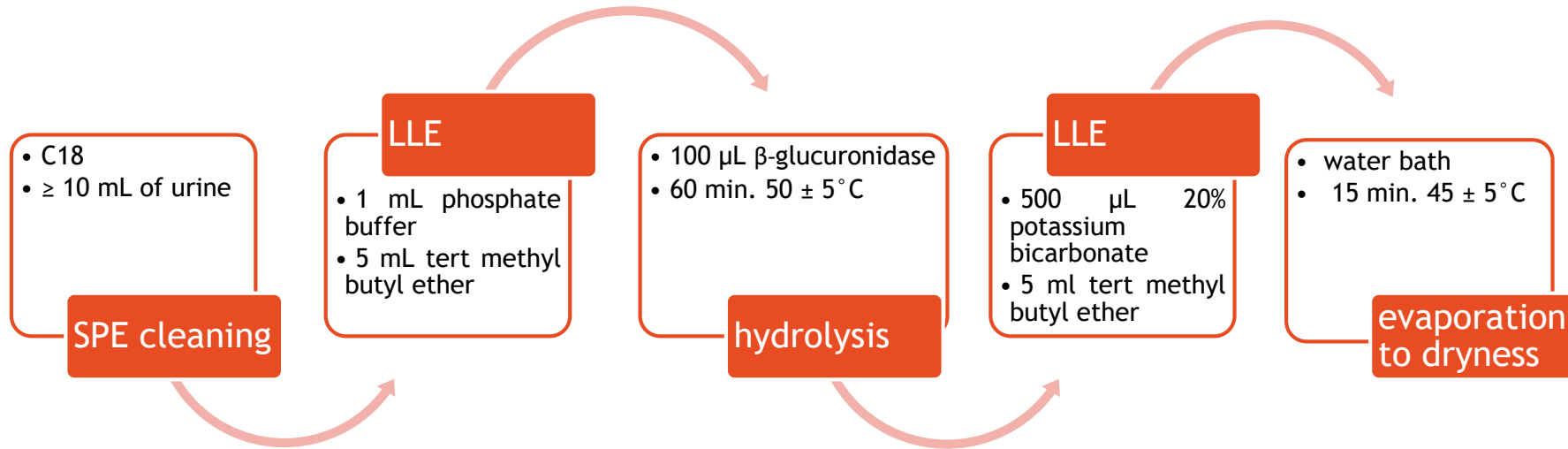
STEROID PROFILE

NR	NAME	GENDER	IC/OOC	DISCIPLINE	urine volume used for IRMS analysis [mL]	Concentration [ng/mL]							PD	11-OH-A	T/E area	A/Etio	A/T	5 α -Adiol/5 β -Adiol	5 α -Adiol/E
						DHEA	Epitestosterone	Testosterone	Androsterone	Etiocolanalone	5 α -Adiol	5 β -Adiol							
1	u23-3059	M	OOC	Canoeing	16	27,3	39,5	4,97	4526	2144	54,5	31,8	397	483	0,15	2,11	911	1,71	1,38
2	u23-3066	M	IC	Athletics	15	21,9	3,79	17,3	2108	949	24,0	41,8	47	496	4,73	2,22	122	0,57	6,34
3	u23-3243	M	IC	Athletics	16	178,8	37,1	80,9	13059	3283	134,7	170,0	1103	2889	1,63	3,98	161	0,79	3,63
4	u23-3317	F	IC	Athletics	20	62,5	14,2	1,11	4937	4117	30,4	30,5	774	1491	0,08	1,20	4446	1,00	2,15
5	u23-2979	M	IC	Canoeing	12	28,5	19,5	20,6	2504	1903	42,7	189,4	201	763	1,15	1,32	122	0,23	2,19
6	u23-3077	M	IC	Canoeing	15	22,3	12,5	14,7	1165	1299	22,5	64,1	255	399	1,29	0,90	80	0,35	1,80
7	u23-3277	M	IC	Box	15	25,1	8,91	30,4	2285	2001	42,0	178,4	199	275	3,79	1,14	75	0,24	4,71
8	u23-3307	M	OOC	Canoeing	15	81,1	59,8	104,8	6677	4260	212,0	401,3	1089	973	1,74	1,57	64	0,53	3,54
9	u23-3390	M	OOC	Badminton	20	14,8	14,1	2,13	841	1324	9,28	18,5	159	187	0,16	0,64	396	0,50	0,66
10	u23-3455	M	IC	Canoeing	15	31,3	15,1	2,23	1967	1562	21,4	26,5	201	989	0,16	1,26	882	0,80	1,41
11	u23-3541	F	IC	Athletics	15	56,8	12,9	4,85	2725	3304	19,9	33,3	508	1023	0,41	0,82	562	0,60	1,54
12	u23-3621	M	IC	Taekwondo	20	6,5	5,20	5,46	217	781	2,90	30,3	44	49	1,12	0,28	40	0,10	0,56
13	u23-3718	M	OOC	Canoeing	20	5,8	7,52	1,17	569	559	5,64	5,64	54	288	0,17	1,02	486	1,00	0,75
14	u23-3814	M	OOC	Teqball	15	33,7	10,5	49,4	3097	2178	54,5	120,2	206	408	4,79	1,42	63	0,45	5,17
15	u23-4033	M	IC	Teqball	12	85,0	43,2	27,1	7766	4971	78,6	121,7	495	1084	0,65	1,56	287	0,65	1,82
16	u23-3931	M	IC	Kickboxing	20	59,0	7,07	11,9	3302	2795	57,6	130,2	921	1047	1,68	1,18	278	0,44	8,15
17	u23-4170	F	IC	Kickboxing	16	54,4	56,2	24,5	6599	3147	41,4	62,5	584	2873	0,47	2,10	269	0,66	0,74
18	u23-4147	F	IC	Box	20	48,4	2,31	12,6	1658	1578	19,1	64,6	562	298	5,00	1,05	131	0,30	8,28

GC/C/IRMS ANALYSIS SCHEME:



SAMPLE PREPARATION



Routine analysis:

- NQC
- PQC
- 10 samples

Single dedicated technician:
duration of sample preparation
of a single batch 6 hours

HPLC CLEANING

First HPLC cleaning: Fraction 1-9; 75 min.

Column: Kinetex EO C18 100 Å, 250 x 4,6 mm 5µm

14 samples batch cleaning 55 h »
2,5 days

Second HPLC cleaning:
Fraction 2 (T); 55 min.

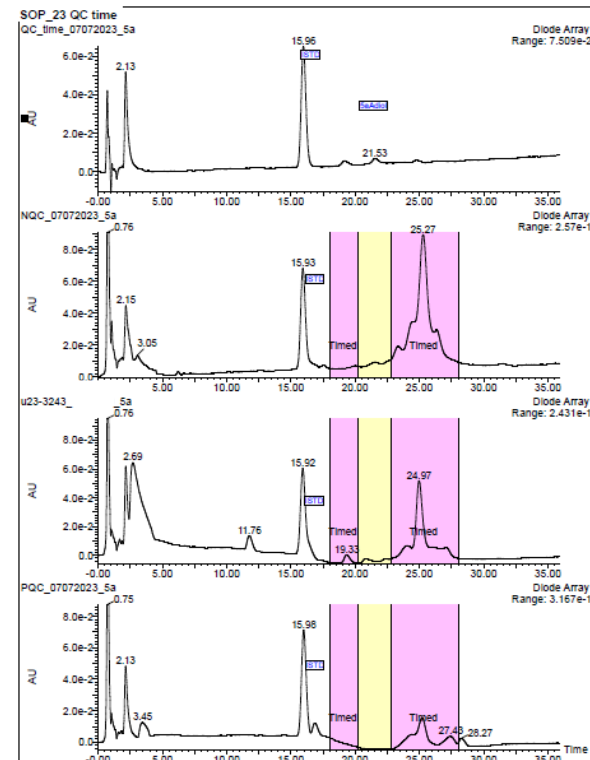
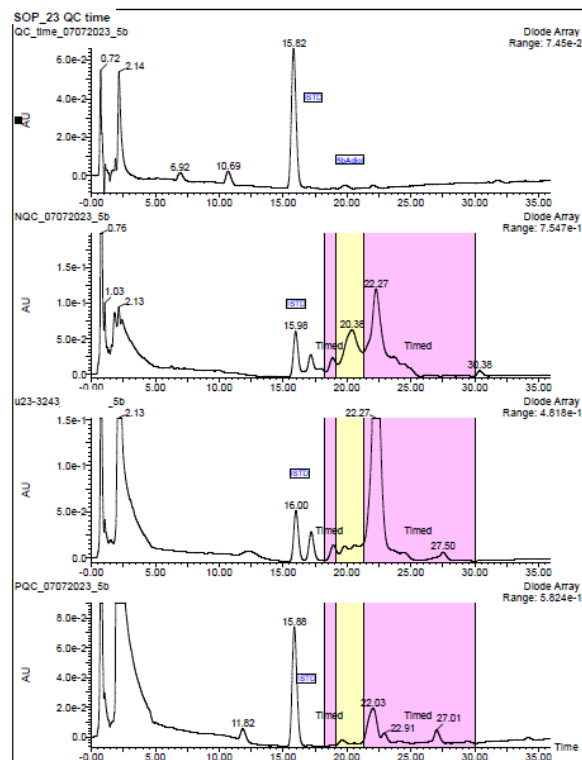
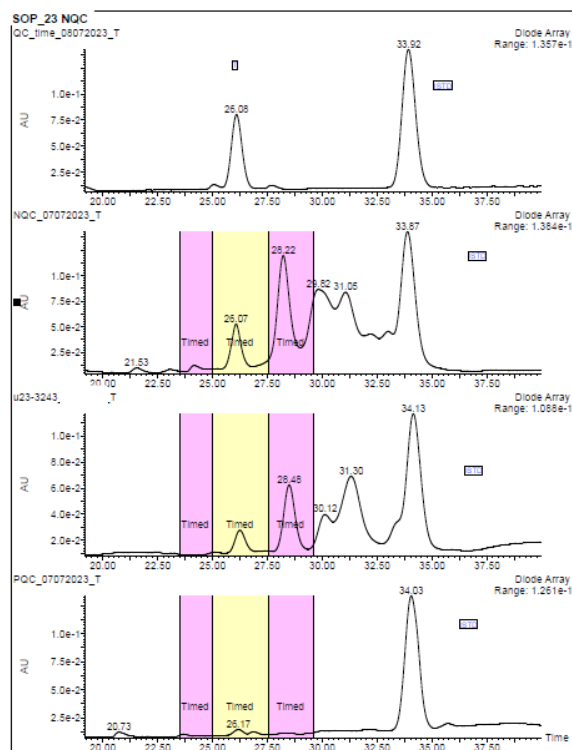
Column: SunFire C18, 150 x 4,6 mm 5µm

Second HPLC cleaning:
Fraction 4' (5βAdiol); 55 min.

Column: Eclipse XDB-C18, 150 x 3,0mm 5µm

Second HPLC cleaning:
Fraction 5' (5αAdiol); 55 min.

Column: Eclipse SDB -C18, 150 x 3,0mm 5µm



THERMO GC/C/IRMS/MS DELTA V PLUS SYSTEM



GC/C/IRMS ANALYSIS PARAMETERS

Thermo GC/C/IRMS system

GC Column: Agilent 19091S-413 UI HP5-MS

(30m x 0.25mm x 0,32 μ m)

Helium constant flow: 1.9 mL/min

Sample injection: 05-2 μ L;

Sample injection mode: splitless

Time of analysis: 15.04 min

GC Temperature programe:

150°C (1 min);

25°C/min \rightarrow 260°C (3.6 min);

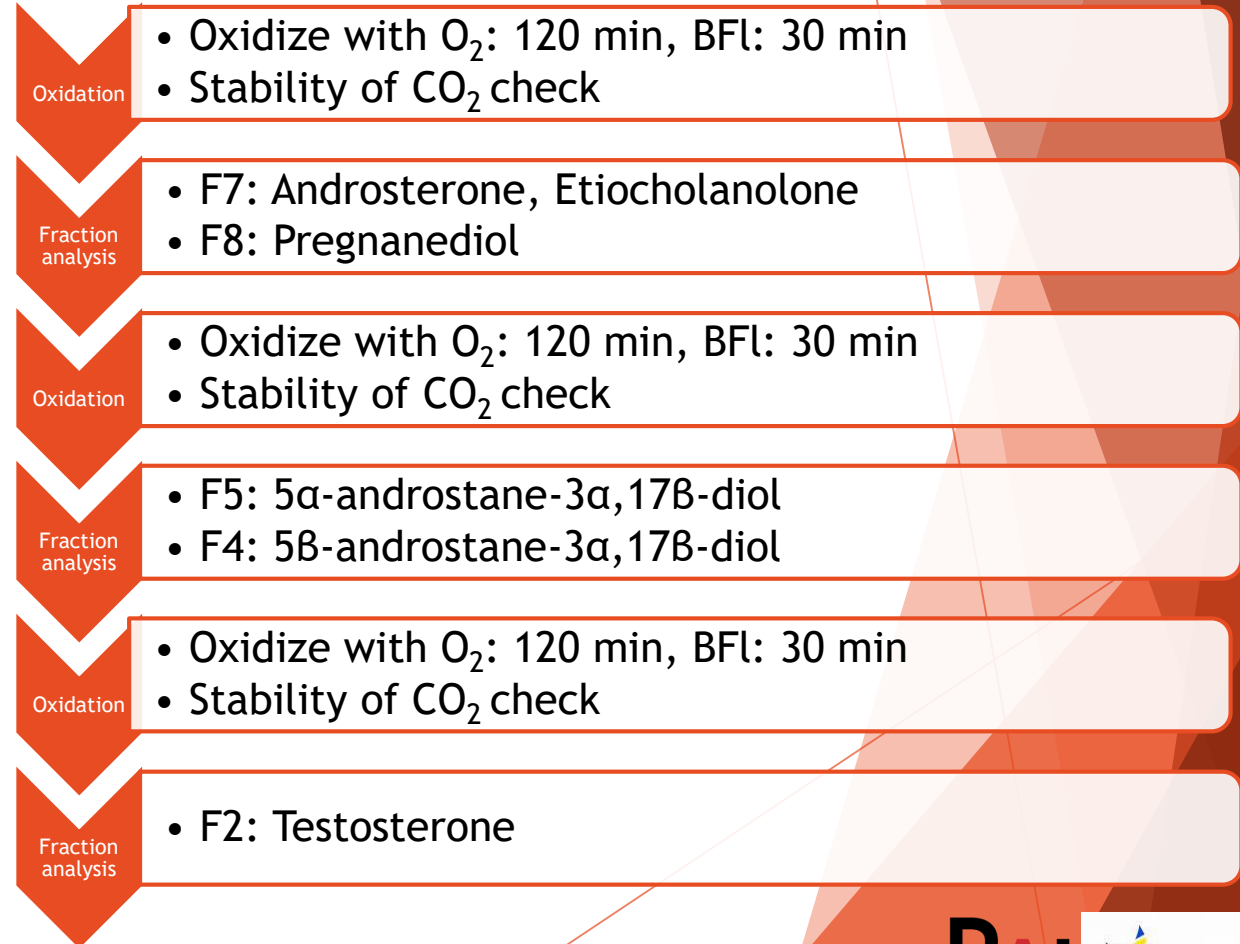
25°C/min \rightarrow 270°C (0.9 min);

35°C/min \rightarrow 290°C (1.2 min);

35°C/min \rightarrow 310°C (1.6 min).

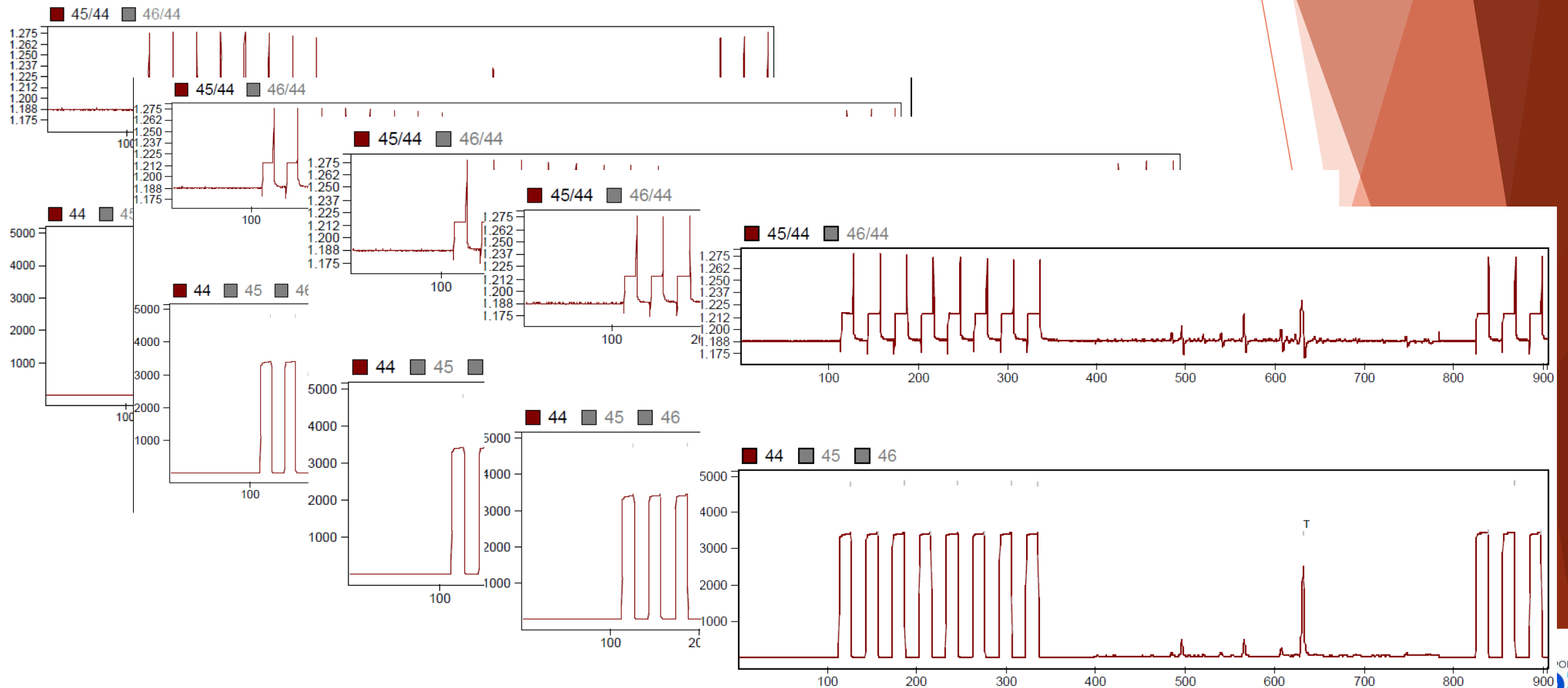
Analysis of 10 samples: 81 h \gg 3,5 days

GC/C/IRMS sequence scheme



GC/C/IRMS ANALYSIS

Sample: 23-3243



GC/C/IRMS RESULTS

Sample: 23-3243

NR	NAME	GENDER	C/OOC	DISCIPLINE	urine volume used for IRMS analysis [mL]	Concentration [ng/mL]									T/E area	A/Etio	A/T	5a-Adiol/5b-Adiol	5a-Adiol/E
						DHEA	Epitestosterone	Testosterone	Androsterone	Etiocolanolone	5a-Adiol	5b-Adiol	PD	11-OH-A					
3	u23-3243	M	C	Athletics	16	178,8	37,1	80,9	13059	3283	134,7	170,0	1103	2889	1,63	3,98	161	0,79	3,63

	QC Chart	TC								ERC1	ERC3
		ERC2	TC						ERC1	ERC3	
		Fraction 1	Fraction 2	Fraction 3	Fraction 4	Fraction 5	Fraction 7		Fraction 8	Fraction 9	
Pass	11-OH-A (ERC2)	Testosterone	Epitestosterone	5βA-diol	5αA-diol	Androsterone	Etiocolanolone	Pregnanediol (ERC1)	16-en (ERC3)		
USADA	Pass	Y / N	Y / N	Y / N	Y / N	Y / N	Y / N	Y / N	Y / N		
MX018-1/MX018-2/ MX018-3*	Y / N	-	-22,802	-	-29,805	-31,075	-28,108	-27,936	-17,000	-	
RM	Y / N	-	-27,625	-	-29,553	-30,265	-33,554	-29,950	-18,781	-	
NQC	Y / N	-	-22,885	-	-23,072	-23,119	-22,631	-23,389	-22,220	-	
PQC	Y / N	-	-28,995	-	-28,160	-29,332	-27,902	-27,950	-21,850	-	
u23-3243		-	-21,619	-	-22,995	-22,584	-21,122	-23,001	-21,867	-	
δ ¹³ C (‰) value difference ERC1 - TC			0,25	-	1,13	0,72	0,75	1,13			
			= PD - T	= PD - EpiT	= PD - 5βA-diol	= PD - 5αA-diol	= PD - A	= PD - Etio			
δ ¹³ C (‰) value difference ERC2 - TC			-	-	-	-	-	-			
			= 11OH-A - T	= 11OH-A - EpiT	= 11OH-A - 5βA-diol	= 11OH-A - 5αA-diol	= 11OH-A - A	= 11OH-A - Etio			
δ ¹³ C (‰) value difference ERC3 - TC			-	-	-	-	-	-			
			= 16-en - T	= 16-en - EpiT	= 16-en - 5βA-diol	= 16-en 5αA-diol	= 16-en - A	= 16-en - Etio			
USADA 34_1		δ ¹³ C (‰) Value Acceptable Range	MX018-1*	δ ¹³ C (‰) Value Acceptable Range *	MX018-2*	δ ¹³ C (‰) Value Acceptable Range*					
Etiocolanolone		-28.90 ± 0.5	T	-27.87 ± 0.5	E	-30.17 ± 0.5					
Androsterone		-27.06 ± 0.5	A	-27.79 ± 0.5	5βAdiol	-29.86 ± 0.5					
Pregnanediol		-31.48 ± 0.5	Etio	-27.94 ± 0.5	5αAdiol	-31.14 ± 0.5					
MX018-3*		δ ¹³ C (‰) Value Acceptable Range *	11BOHetio	-29.51 ± 0.5	PD	-16.79 ± 0.5					
T		-22.52 ± 0.5	11oxoetio	-13.58 ± 0.5	11OH-A	-28.59 ± 0.5					
16-en		-30.96 ± 0.5									
DHEA		-31.63 ± 0.5									

GC/C/IRMS RESULTS

Lp.	nr lab.	11-OH-A (ERC2)*		Testosterone		Epitestosterone		5βA-diol		5αA-diol		Androsterone		Etiocolanalone		Pregnanediol	
		$\delta^{13}\text{C}/^{12}\text{C}$ value [‰]	u_c [‰]	$\delta^{13}\text{C}/^{12}\text{C}$ value [‰]	u_c [‰]	$\delta^{13}\text{C}/^{12}\text{C}$ value [‰]	u_c [‰]	$\delta^{13}\text{C}/^{12}\text{C}$ value [‰]	u_c [‰]	$\delta^{13}\text{C}/^{12}\text{C}$ value [‰]	u_c [‰]	$\delta^{13}\text{C}/^{12}\text{C}$ value [‰]	u_c [‰]	$\delta^{13}\text{C}/^{12}\text{C}$ value [‰]	u_c [‰]	$\delta^{13}\text{C}/^{12}\text{C}$ value [‰]	u_c [‰]
1	u23-2979	-	0,641	-22,45	0,663	-	0,723	-21,886	0,659	-22,121	0,603	-21,826	0,675	-22,523	0,689	-22,34	0,679
2	u23-3059	-	0,641	-22,549	0,663	-	0,723	-23,732	0,659	-23,110	0,588	-21,934	0,675	-23,322	0,689	-20,787	0,679
3	u23-3066	-	0,641	-24,557	0,663	-	0,723	-25,735	0,659	-24,666	0,588	-23,989	0,675	-25,345	0,689	-24,682	0,679
4	u23-3077	-	0,641	-23,183	0,663	-	0,723	-24,352	0,659	-24,092	0,603	-22,974	0,675	-23,62	0,689	-23,148	0,679
5	u23-3243	-	0,641	-21,619	0,663	-	0,723	-22,995	0,659	-22,584	0,603	-21,122	0,675	-23,001	0,689	-21,867	0,679
6	u23-3277	-	0,641	-22,016	0,663	-	0,723	-22,734	0,659	-22,103	0,603	-21,609	0,675	-22,135	0,689	-22,468	0,679
7	u23-3307	-21,825	0,641	-22,742	0,663	-	0,723	-23,829	0,659	-23,789	0,603	-22,471	0,675	-24,002	0,689	*	0,679
8	u23-3317	-	0,641	*	0,663	-	0,723	-24,464	0,659	-23,436	0,603	-22,375	0,675	-23,378	0,689	-22,236	0,679
9	u23-3390	-	0,641	*	0,663	-	0,723	*	0,659	*	0,603	-21,996	0,675	-22,566	0,689	-22,665	0,679
10	u23-3455	-	0,641	*	0,663	-	0,723	-25,327	0,659	-24,708	0,603	-23,249	0,675	-23,94	0,689	-23,277	0,679
11	u23-3541	-	0,641	*	0,663	-	0,723	-23,665	0,659	-23,029	0,603	-22,519	0,675	-23,491	0,689	-22,733	0,679
12	u23-3621	-	0,641	-23,869	0,663	-	0,723	-24,68	0,659	*	0,603	-24,298	0,675	-24,091	0,689	-24,118	0,679
13	u23-3718	-	0,641	*	0,663	-	0,723	*	0,659	*	0,603	-22,315	0,675	-23,338	0,689	-23,479	0,679
14	u23-3814	-	0,641	-23,125	0,663	-	0,723	-23,617	0,659	-23,43	0,603	-22,021	0,675	-23,378	0,689	-23,211	0,679
15	u23-3931	-22,112	0,641	-21,594	0,663	-	0,723	-22,741	0,659	-22,217	0,603	-21,974	0,675	-23,051	0,689	-22,013	0,679
16	u23-4033	-	0,641	-23,048	0,663	-	0,723	-23,931	0,659	-23,351	0,603	-22,319	0,675	-23,985	0,689	-23,348	0,679
17	u23-4170	-21,142	0,641	-21,399	0,663	-22,217	0,723	-22,312	0,659	-21,658	0,603	-21,48	0,675	-22,86	0,689	-21,71	0,679
18	u23-4147	-22,972	0,641	-21,959	0,663	-	0,723	-23,587	0,659	-23,273	0,603	-22,969	0,675	-23,768	0,689	-23,825	0,679

Reporting time: 20 days

GC/C/IRMS ISSUES

- Water in the system (implementation of water traps on Helium and Oxygen line, Nafion exchange)
- Troubles with backflash (capillary exchange)
- System leak (3-port splitter exchange)

SUMMARY:

The laboratory received 1058 urine samples and 194 blood samples over a period of 2 weeks.

The laboratory received 25 recommendations for additional analyses concerning steroid profile.

The laboratory performed 18 GC/C/IRMS analysis.

It is good to have a lot of spare parts for GC/C/IRMS maintenance before a big event.



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- Foreign experts from Anti-Doping Laboratories from London, Kreisha and Seibersdorf.



THANK YOU FOR YOUR ATTENTION