



Routine Analytical Chemistry Sub-Group

Technical Report

2011 Collaborative Study of CORESTA Monitors #6 (CM6) and #7 (CM7) for the Determination of Test Piece Weight, TPM, Water, Nicotine, NFDPM, Carbon Monoxide and Puff Count Obtained Under Mainstream 'ISO' and 'Intense' Smoking Regimes

Co-ordinator: Linda A. Crumpler, Cerulean, USA

Study Co-ordinator: Linda A. Crumpler, Cerulean, USA

Author: Thomas Verron, SEITA Imperial Tobacco Group, France

February 2012

Contents

1. INTRODUCTION	3
2. ORGANISATION	3
2.1 Participants	3
2.2 Protocol	4
3. RAW - DATA	5
4. STATISTICAL ANALYSIS	13
4.1 Exclusion of Outliers	13
4.1.1 Mandel graphical outlier technique:	13
4.1.2 Numerical outlier technique: Cochran & Grubbs test	13
4.1.3 Outlier results	14
4.2 Repeatability and Reproducibility estimation	15
5. DATA REPRESENTATION	16
5.1 ISO smoking regime	17
5.1.1 CM6	17
5.1.2 CM7	20
5.2 Intense smoking regime	24
5.2.1 CM6	24
5.2.2 CM7	27
6. COMPARISON	30
6.1 Comparisons of Linear with Rotary smoking machines for test piece results of CM6 and CM7	30
6.2 Comparison with previous studies	30
7. CONCLUSION	32
APPENDIX A - List of Participating Laboratories	33
APPENDIX B – Experimental Protocol	34
APPENDIX C – Departures from Experimental Protocol	38
APPENDIX D – RAW DATA SET	39
APPENDIX E – MANDEL’S GRAPH	44
APPENDIX F – IUPAC 1994 HARMONIZED STATISTICAL PROCEDURE	51
APPENDIX G - LINEAR – ROTARY COMPARISONS	52
APPENDIX H - ROTARY SMOKING MACHINES	58
APPENDIX I - LINEAR SMOKING MACHINES	73

1. INTRODUCTION

The CORESTA Routine Analytical Chemistry Sub Group has been given the responsibility to organize the annual testing of the CORESTA Monitor test piece.

The 2011 study was designed:

- to measure mainstream ISO smoke yields of nicotine-free dry particulate matter (NFDPM), nicotine and carbon monoxide (CO) to verify the current monitor test piece CM6
- to establish ISO mean values of NFDPM, nicotine and CO for the new test piece CM7
- to determine intra- and inter-laboratory variability for the measured ISO smoke yields for the CM7 and CM6
- to establish the conditioned weight of CM7 prior to smoking and to verify the conditioned weight for the CM6
- to qualify CM7 for use as a Monitor test piece
- to determine smoke deliveries under an intense smoking regime for the test pieces CM6 and CM7
- to determine intra- and inter-laboratory variability for the measured intense smoke yields for the CM7 and CM6

2. ORGANISATION

2.1 Participants

In total 33 laboratories participated in the 2011 study. Appendix A lists the participating laboratories in alphabetical order. Note: the mix of labs between CM6, CM7, ISO and Intense differed.

Thirty two laboratories delivered data for ISO mainstream smoke for the CM6 test piece using 50 smoking machines (21 linear smoking machines and 29 rotary smoking machines). CM7 ISO mainstream smoke data have been delivered by 32 laboratories with 51 smoking machines (21 linear smoking machines and 30 rotary smoking machines).

Only 30 laboratories provided data for the intense smoking regime on CM6 and CM7. 41 smoking machines were used (20 rotary and 21 linear) for both test pieces.

Due to technical reasons some laboratories provided incomplete data sets. Table1 summarises the number and type of smoking machines used and data sets provided by the parameters reported.

Table 1: Number of smoking machines and data sets obtained

	CM6		CM7	
	ISO	Intense	ISO	Intense
Rotary smoking machines	29	20	30	20
Linear smoking machines	21	21	21	21
Total	50	41	51	41
Individual data				
WEIGHT	250		254	
TPM	250	205	254	205
WATER	250	205	254	205
NICOTINE	250	205	254	205
NFPDM	250	205	254	205
CO	245	205	249	205
Puff Count	250	205	254	205
Total	1495	1230	1519	1230

A code number has been assigned in confidence by the study co-ordinator to each of the individual smoking machines used in the study.

2.2 Protocol

Participants were requested to follow the protocol “Annual study of the CORESTA monitor test piece CM6 and CM7 – February 28, 2011” (provided in Appendix B) to analyse the two products CM6 and CM7 and to report the seven parameters listed in Table 2.

Table 2: Parameters to be reported

Parameter	Number of replicates	Unit	Code
nicotine free dry particulate matter	5	mg/t.p.	NFPDM
smoke nicotine	5	mg/t.p.	NIC
carbon monoxide	5	mg/t.p.	CO
total particulate matter	5	mg/t.p.	TPM
water content (smoke)	5	mg/t.p.	WATER
conditioned weight	5	mg/t.p.	WEIGHT
puff number	5	/t.p.	PUFF

Both test pieces should have been smoked under two different mainstream smoking regimes with a fixed butt length of 32 mm as defined in the following table:

Table 3: Mainstream ISO and Intense smoking regime parameter

Smoking Regime	Puff Volume (mL)	Puff Frequency* (s)	Puff Duration (s)	Vent Blocking (%)
ISO	35	60	2	0
Intense	55	30	2	100

The CM6 test piece was purchased by the participants individually via Borgwaldt or Cerulean.

The CM7 test piece was produced on June 23rd to 24th 2010 in Berlin Germany. A one point in time sampling according to ISO 8243:2006 was performed during the production. 35 bundles of 10 packs (200 test pieces) were drawn over the entire production. Packs have been randomised and composite laboratory samples each containing 20 packs were produced. Laboratory samples were distributed by Borgwaldt KC to the participating labs for analysis.

3. RAW - DATA

Tables 4 to 10 list the results for the seven reported parameters by smoking regime and test piece. Basic statistics (mean and standard deviation) were applied to summarize the raw data for each smoking machine used and parameters reported for both test pieces. At this stage no outlier statistics were applied. Appendix D contains the raw data (single values) for every measured parameter reported by smoking machine included in the study.

Table 4: WEIGHT mean and standard deviation per laboratory code

Labs	WEIGHT (mg/t.p.)			
	CM6		CM7	
	Mean	Sd	Mean	Sd
1	973.8	5.1	966.5	2.5
2A	971.2	5.0	965.1	6.3
2B	970.0	3.1	957.7	3.9
3A	969.4	2.4	959.7	7.4
3B	969.3	1.7	960.5	6.5
3C	968.6	3.8	957.2	6.5
4	969.4	6.1	955.3	4.6
5	962.4	1.2	948.6	8.0
6	966.2	4.7	964.9	1.8
8A	975.0	4.0	952.5	6.0
8B	971.7	4.1	957.1	8.4
8C	973.5	4.2	952.4	3.2
9A	975.1	3.0	958.7	2.3
9B	975.2	2.4	961.4	6.1
10A	974.0	3.5	958.3	3.5
10B	975.0	3.4	956.1	2.7
11A	975.4	2.6	960.3	3.1
11B	974.9	4.7	961.3	4.1
11C	974.4	3.3	956.9	3.0
12A	977.8	5.8	960.9	4.7
12B	974.1	3.7	962.7	4.4
14	974.8	6.4	957.9	3.8
16	968.3	1.7	948.7	3.0
18A	975.8	2.8	959.9	4.4
18B	974.2	2.7	960.2	5.8
19	984.6	6.7	966.5	2.7
21	974.2	3.0	956.9	5.5
25	976.8	3.8	961.6	2.9
26A	976.9	3.1	955.6	3.2
27A	965.9	5.8	951.8	2.3
27B	960.9	4.6	946.9	2.6
28A	970.9	3.0	955.5	5.8
28B	972.9	7.7	959.0	8.9
29A	974.9	4.1	960.1	2.1
29B	972.6	1.3	956.9	1.6
30A	970.6	4.8	958.6	5.7
30B	972.5	6.2	955.1	2.7
31A	977.1	6.4	965.1	4.4
31B	972.7	3.2	966.2	3.0
32	966.9	4.2	951.3	4.7
33A	970.3	2.3	951.2	1.3
33B	972.0	4.3	952.5	3.0
35	960.7	3.6	946.3	3.7
39	974.7	4.4	952.9	5.4
40	970.3	5.5	958.9	3.6
41	963.2	1.9	951.8	3.7
42A	974.4	4.4	960.7	1.5
42B	979.2	3.7	959.5	6.1
43			963.0	3.0
44	956.3	6.4	937.2	6.9
45	970.9	4.2	958.7	1.8

Table 5: TPM mean and standard deviation per laboratory code

Labs	TPM (mg/t.p.)							
	ISO				Intense			
	CM6		CM7		CM6		CM7	
	Mean	Sd	Mean	Sd	Mean	Sd	Mean	Sd
1	17.50	0.58	17.90	0.44	45.66	1.31	48.52	2.18
2A	16.81	0.44	15.64	0.31	39.45	0.46	37.88	0.52
2B	17.83	0.34	16.54	0.16	43.44	0.31	42.95	0.45
3A	17.53	0.18	16.66	0.17	40.34	0.39	40.31	0.44
3B	17.05	0.29	16.25	0.23	39.93	0.41	39.49	0.39
3C	16.99	0.16	16.70	0.15	44.36	0.17	44.27	1.08
4	18.35	0.25	17.04	0.46	47.55	0.93	46.92	1.11
5	18.55	0.50	17.09	0.53	48.79	1.26	46.40	0.60
6	17.22	0.37	15.84	0.14	39.42	0.58	38.33	0.35
8A	17.92	0.09	16.93	0.33	41.96	0.55	40.72	0.64
8B	17.76	0.20	16.20	0.25				
8C	17.78	0.21	16.71	0.14	41.20	0.56	40.04	0.62
9A	18.09	0.46	16.88	0.53	44.26	1.39	43.91	1.18
9B	17.07	0.14	15.94	0.33	40.03	0.27	39.45	0.51
10A	17.36	0.12	16.20	0.22				
10B	16.92	0.19	15.95	0.12	37.82	0.67	38.09	0.54
11A	17.55	0.26	16.87	0.43	44.47	0.98	43.45	0.98
11B	16.74	0.31	16.01	0.30	38.25	0.80	38.38	0.38
11C	17.19	0.24	16.22	0.23	39.30	0.46	38.88	0.51
12A	17.86	0.29	17.11	0.33	47.20	0.80	47.20	0.80
12B	17.21	0.26	16.05	0.22	41.08	0.76	41.17	0.52
14	17.19	0.18	16.15	0.15	39.26	0.35	38.27	0.24
16	16.62	0.42	15.78	0.19	39.60	0.53	38.43	0.64
18A	16.90	0.46	16.24	0.44	43.34	0.83	42.51	0.71
18B	17.32	0.15	16.40	0.29				
19	18.40	0.33	17.35	0.25	46.37	1.12	45.53	1.31
21	17.07	0.44	15.84	0.65	44.69	1.37	44.69	1.37
25	18.50	0.61	17.47	0.61	44.17	0.86	43.99	1.98
26A	16.67	0.14	15.58	0.36	39.92	0.43	38.26	0.45
27A	17.06	0.36	15.85	0.22	38.56	0.70	37.58	0.58
27B	18.30	0.47	16.48	0.42	46.08	1.10	44.94	1.17
28A	17.00	0.30	16.15	0.25				
28B	17.05	0.37	16.13	0.15				
29A	17.72	0.15	17.13	0.41	40.72	0.32	40.87	0.37
29B	17.34	0.21	16.44	0.32				
30A	17.51	0.33	16.06	0.32	44.69	0.69	42.82	0.49
30B	16.10	0.81	15.04	1.16				
31A	17.82	0.33	16.04	0.21	40.28	1.38	38.62	0.58
31B	17.74	0.48	16.61	0.43	44.16	0.76	43.03	0.58
32	17.25	0.25	16.02	0.30	40.94	0.27	40.96	0.53
33A	17.31	0.13	15.97	0.21				
33B	17.53	0.26	15.95	0.23	38.26	0.11	37.17	0.57
35	17.73	0.37	17.06	0.42	45.63	0.72	44.11	0.08
39	17.83	0.37	16.96	0.47	49.61	0.83	48.97	0.81
40	17.27	0.38	16.41	0.25	45.89	0.76	46.16	0.83
41	18.08	0.35	16.47	0.36	47.41	0.82	45.12	1.27
42A	17.22	0.25	16.34	0.19	39.94	0.37	39.95	0.63
42B	16.85	0.13	16.08	0.12	39.30	0.38	38.92	0.32
43			15.96	0.10				
44	17.35	0.52	15.45	0.47	41.90	0.93	40.91	2.49
45	16.08	0.37	15.19	0.19				

Table 6: WATER mean and standard deviation per laboratory code

Labs	WATER (mg/t.p.)							
	ISO				Intense			
	CM6		CM7		CM6		CM7	
	Mean	Sd	Mean	Sd	Mean	Sd	Mean	Sd
1	1.67	0.11	2.23	0.51	13.29	0.54	15.67	1.06
2A	2.09	0.22	1.89	0.16	9.59	0.20	9.03	0.15
2B	1.46	0.10	1.35	0.07	11.45	0.35	11.71	0.24
3A	2.18	0.12	2.13	0.04	9.63	0.14	9.87	0.09
3B	1.90	0.18	1.94	0.12	9.11	0.33	9.34	0.19
3C	1.36	0.08	1.39	0.08	11.64	0.41	12.03	0.54
4	1.86	0.19	1.79	0.20	13.27	0.48	13.17	0.42
5	1.38	0.13	1.34	0.24	13.86	0.71	13.14	0.40
6	1.97	0.18	1.78	0.09	8.84	0.27	8.63	0.35
8A	2.19	0.11	2.20	0.10	11.45	0.51	10.99	0.45
8B	2.12	0.09	2.00	0.08				
8C	2.25	0.13	2.23	0.07	10.45	0.48	10.28	0.44
9A	1.85	0.13	1.78	0.14	12.65	0.86	13.09	0.53
9B	2.06	0.05	2.08	0.05	9.69	0.43	9.79	0.43
10A	1.89	0.03	1.86	0.11				
10B	1.57	0.26	1.58	0.07	7.39	0.41	7.80	0.45
11A	1.52	0.12	1.52	0.10	11.81	0.43	11.37	0.45
11B	1.92	0.06	1.92	0.05	8.59	0.27	9.16	0.30
11C	2.22	0.06	2.14	0.09	9.30	0.44	9.40	0.35
12A	1.57	0.23	1.50	0.20	13.28	0.30	13.28	0.30
12B	2.22	0.12	2.11	0.10	10.84	0.57	11.23	0.32
14	2.24	0.29	1.94	0.10	9.71	0.23	9.75	0.79
16	1.86	0.21	1.82	0.15	8.82	0.55	8.90	0.58
18A	1.46	0.15	1.50	0.09	11.40	0.60	11.36	0.50
18B	2.33	0.07	2.29	0.08				
19	1.88	0.18	1.91	0.28	13.16	1.01	12.85	0.73
21	1.11	0.16	1.04	0.21	11.46	0.64	11.46	0.64
25	1.53	0.16	1.42	0.07	8.88	0.84	8.65	0.66
26A	1.77	0.04	1.76	0.09	9.28	0.27	9.26	0.30
27A	1.99	0.13	1.84	0.12	8.77	0.24	8.88	0.37
27B	1.63	0.28	1.44	0.25	12.28	1.24	11.98	1.06
28A	2.04	0.19	2.01	0.15				
28B	2.07	0.19	2.01	0.14				
29A	2.24	0.06	2.29	0.15	10.55	0.34	10.50	0.31
29B	2.15	0.18	2.08	0.24				
30A	1.75	0.37	1.41	0.22	11.59	1.81	10.64	1.40
30B	1.24	0.15	1.32	0.21				
31A	1.38	0.05	1.20	0.07	9.54	0.73	9.20	0.31
31B	1.69	0.10	1.59	0.11	12.24	0.28	12.16	0.41
32	2.29	0.20	2.18	0.10	10.16	0.14	10.41	0.50
33A	2.19	0.05	2.06	0.05				
33B	2.12	0.11	2.11	0.15	8.22	0.12	8.13	0.09
35	1.71	0.35	1.63	0.20	11.37	0.51	10.65	0.29
39	1.34	0.16	1.28	0.18	15.03	0.70	15.02	0.66
40	1.52	0.16	1.52	0.15	12.10	0.61	12.08	0.41
41	1.78	0.10	1.61	0.13	13.87	0.60	13.19	0.79
42A	1.95	0.14	1.98	0.12	9.69	0.29	10.17	0.32
42B	1.83	0.13	1.88	0.11	9.51	0.17	9.98	0.25
43			1.74	0.18				
44	1.93	0.43	1.70	0.48	12.19	2.41	12.30	2.38
45	1.75	0.21	1.68	0.23				

Table 7: Nicotine mean and standard deviation per laboratory code

Labs	Nicotine (mg/t.p.)							
	ISO				Intense			
	CM6		CM7		CM6		CM7	
	Mean	Sd	Mean	Sd	Mean	Sd	Mean	Sd
1	1.313	0.057	1.326	0.023	2.688	0.050	2.660	0.022
2A	1.305	0.021	1.203	0.022	2.595	0.030	2.420	0.048
2B	1.408	0.022	1.273	0.018	2.753	0.048	2.572	0.030
3A	1.329	0.019	1.206	0.014	2.580	0.031	2.424	0.043
3B	1.329	0.018	1.220	0.019	2.612	0.021	2.455	0.034
3C	1.345	0.018	1.274	0.017	2.726	0.017	2.564	0.055
4	1.415	0.025	1.261	0.044	2.783	0.054	2.590	0.055
5	1.423	0.037	1.257	0.055	2.890	0.055	2.590	0.046
6	1.310	0.040	1.184	0.019	2.645	0.039	2.539	0.041
8A	1.374	0.010	1.231	0.028	2.698	0.043	2.476	0.030
8B	1.363	0.027	1.187	0.016				
8C	1.307	0.008	1.167	0.015	2.608	0.026	2.440	0.026
9A	1.424	0.028	1.271	0.035	2.768	0.033	2.578	0.060
9B	1.380	0.041	1.220	0.032	2.692	0.023	2.511	0.047
10A	1.308	0.010	1.166	0.009				
10B	1.285	0.013	1.162	0.020	2.482	0.030	2.334	0.038
11A	1.385	0.025	1.275	0.028	2.737	0.060	2.552	0.043
11B	1.358	0.031	1.263	0.029	2.629	0.065	2.507	0.078
11C	1.369	0.028	1.252	0.014	2.705	0.039	2.503	0.038
12A	1.360	0.022	1.242	0.012	2.626	0.043	2.626	0.043
12B	1.328	0.028	1.169	0.028	2.551	0.056	2.446	0.077
14	1.300	0.045	1.201	0.013	2.521	0.024	2.371	0.047
16	1.366	0.046	1.270	0.025	2.764	0.039	2.560	0.043
18A	1.399	0.033	1.283	0.035	2.799	0.061	2.622	0.036
18B	1.354	0.009	1.238	0.017				
19	1.328	0.031	1.202	0.040	2.650	0.048	2.452	0.055
21	1.380	0.046	1.265	0.032	2.707	0.065	2.707	0.065
25	1.254	0.046	1.131	0.035	2.450	0.051	2.290	0.062
26A	1.324	0.015	1.220	0.018	2.846	0.037	2.608	0.026
27A	1.408	0.046	1.254	0.041	2.693	0.060	2.504	0.067
27B	1.444	0.052	1.265	0.025	2.852	0.082	2.630	0.094
28A	1.305	0.021	1.205	0.020				
28B	1.311	0.027	1.197	0.011				
29A	1.328	0.026	1.251	0.025	2.575	0.096	2.433	0.064
29B	1.348	0.027	1.233	0.034				
30A	1.471	0.030	1.306	0.037	2.904	0.053	2.628	0.054
30B	1.382	0.111	1.222	0.073				
31A	1.426	0.014	1.261	0.020	2.679	0.038	2.490	0.019
31B	1.391	0.025	1.274	0.026	2.709	0.028	2.556	0.021
32	1.288	0.034	1.150	0.020	2.559	0.082	2.427	0.072
33A	1.378	0.020	1.242	0.023				
33B	1.398	0.010	1.207	0.023	2.676	0.034	2.464	0.055
35	1.378	0.030	1.274	0.034	2.726	0.029	2.522	0.036
39	1.423	0.034	1.313	0.029	2.753	0.028	2.632	0.044
40	1.444	0.020	1.334	0.034	2.884	0.037	2.720	0.080
41	1.448	0.021	1.263	0.024	2.862	0.022	2.574	0.025
42A	1.351	0.015	1.222	0.018	2.682	0.041	2.490	0.020
42B	1.315	0.011	1.215	0.012	2.644	0.024	2.448	0.029
43			1.270	0.007				
44	1.496	0.037	1.299	0.029	2.972	0.051	2.762	0.035
45	1.258	0.031	1.156	0.030				

Table 8: NFDPM mean and standard deviation per laboratory code

Labs	NFDPM (mg/t.p.)							
	ISO				Intense			
	CM6		CM7		CM6		CM7	
	Mean	Sd	Mean	Sd	Mean	Sd	Mean	Sd
1	14.51	0.51	14.37	0.14	29.68	0.85	30.31	1.56
2A	13.42	0.23	12.55	0.15	27.27	0.33	26.43	0.36
2B	14.96	0.25	13.91	0.09	29.23	0.28	28.66	0.23
3A	14.03	0.15	13.33	0.18	28.14	0.28	28.02	0.40
3B	13.82	0.12	13.09	0.25	28.21	0.22	27.69	0.45
3C	14.28	0.19	14.04	0.08	30.00	0.39	29.68	0.65
4	15.07	0.23	13.99	0.37	31.50	0.44	31.16	0.65
5	15.75	0.45	14.50	0.33	32.05	0.80	30.67	0.26
6	13.94	0.26	12.88	0.11	27.93	0.42	27.16	0.22
8A	14.36	0.19	13.49	0.28	27.81	0.35	27.27	0.33
8B	14.27	0.19	13.01	0.19				
8C	14.22	0.11	13.32	0.07	28.14	0.35	27.32	0.27
9A	14.82	0.35	13.84	0.40	28.84	0.61	28.25	0.67
9B	13.63	0.12	12.65	0.28	27.65	0.27	27.15	0.52
10A	14.16	0.12	13.19	0.15				
10B	14.06	0.17	13.21	0.07	27.95	0.41	27.95	0.25
11A	14.64	0.25	14.07	0.35	29.92	0.64	29.53	0.60
11B	13.46	0.28	12.83	0.24	27.03	0.55	26.72	0.42
11C	13.60	0.24	12.83	0.15	27.30	0.16	26.97	0.54
12A	14.93	0.33	14.37	0.20	31.30	0.51	31.30	0.51
12B	13.67	0.18	12.77	0.29	27.70	0.54	27.49	0.42
14	13.65	0.20	13.00	0.07	27.03	0.39	26.15	0.61
16	13.39	0.22	12.69	0.24	28.01	0.56	26.98	0.46
18A	14.04	0.34	13.46	0.34	29.14	0.76	28.53	0.73
18B	13.64	0.10	12.88	0.20				
19	15.19	0.39	14.24	0.22	30.56	0.59	30.23	1.07
21	14.58	0.39	13.54	0.48	30.52	0.86	30.52	0.86
25	15.72	0.47	14.92	0.53	32.84	0.87	33.05	1.28
26A	13.57	0.15	12.60	0.32	27.79	0.24	26.39	0.36
27A	13.66	0.33	12.75	0.19	27.09	0.63	26.20	0.46
27B	15.22	0.34	13.78	0.48	30.95	0.38	30.34	0.80
28A	13.65	0.25	12.93	0.14				
28B	13.67	0.20	12.93	0.13				
29A	14.16	0.20	13.59	0.27	27.60	0.22	27.94	0.33
29B	13.84	0.15	13.13	0.17				
30A	14.29	0.56	13.34	0.28	30.19	2.14	29.56	1.39
30B	13.10	0.70	12.49	0.90				
31A	15.01	0.28	13.58	0.13	28.06	0.61	26.94	0.28
31B	14.66	0.36	13.75	0.31	29.22	0.52	28.31	0.26
32	13.66	0.17	12.69	0.22	28.22	0.33	28.12	0.54
33A	13.74	0.17	12.66	0.16				
33B	14.02	0.21	12.63	0.29	27.37	0.16	26.57	0.48
35	14.64	0.25	14.16	0.50	31.53	0.71	30.94	0.33
39	15.07	0.27	14.36	0.30	31.83	0.96	31.32	0.71
40	14.30	0.34	13.56	0.20	30.90	0.59	31.33	1.15
41	14.85	0.24	13.60	0.24	30.68	1.06	29.35	1.46
42A	13.91	0.12	13.13	0.28	27.57	0.31	27.30	0.41
42B	13.70	0.10	12.99	0.12	27.15	0.30	26.48	0.24
43			12.95	0.26				
44	13.93	0.57	12.45	0.51	26.74	1.54	25.85	1.39
45	13.07	0.34	12.35	0.26				

Table 9: CO mean and standard deviation per laboratory code

Labs	CO (mg/t.p.)							
	ISO				Intense			
	CM6		CM7		CM6		CM7	
	Mean	Sd	Mean	Sd	Mean	Sd	Mean	Sd
1	13.72	0.49	13.30	0.55	25.17	0.46	25.19	0.87
2A	13.76	0.38	12.54	0.29	25.78	0.28	23.63	0.31
2B	13.69	0.29	12.27	0.26	25.59	0.27	23.62	0.51
3A	14.94	0.23	13.99	0.34	27.66	0.23	26.04	0.26
3B	14.79	0.34	13.59	0.39	27.77	0.16	26.30	0.29
3C	13.73	0.26	13.20	0.26	26.73	0.58	24.89	0.41
4	15.11	0.25	13.30	0.23	27.55	0.27	25.61	0.32
5	15.14	0.35	13.47	0.48	27.37	0.42	25.12	0.28
6	14.54	0.66	12.31	0.18	26.58	0.19	24.69	0.23
8A	15.68	0.16	13.33	0.28	27.69	0.37	24.94	0.55
8B	14.80	0.30	12.06	0.21				
8C	15.28	0.27	12.81	0.25	27.26	0.33	24.32	0.34
9A	14.50	0.23	13.04	0.40	26.50	0.55	24.67	0.47
9B	14.56	0.21	13.10	0.22	27.17	0.12	25.24	0.40
10A	14.84	0.13	13.52	0.14				
10B	14.93	0.07	13.77	0.13	26.38	0.18	25.52	0.27
11A	14.02	0.48	12.84	0.56	26.10	0.56	24.03	0.74
11B	14.48	0.21	13.41	0.24	26.20	0.49	25.41	0.10
11C	14.84	0.29	13.69	0.09	27.48	0.34	25.76	0.35
12A	14.59	0.26	13.20	0.39	26.64	0.58	26.64	0.58
12B	15.76	0.32	14.12	0.21	28.21	0.58	26.43	0.30
14	14.71	0.14	13.48	0.12	27.39	0.44	25.57	0.09
16	14.28	0.37	12.66	0.17	27.25	0.23	25.07	0.36
18A	13.56	0.12	12.32	0.08	26.08	0.29	23.56	0.37
18B	14.12	0.33	12.54	0.23				
19	13.96	0.19	12.48	0.18	25.91	0.20	23.74	0.49
21	13.23	0.38	11.63	0.75	25.51	1.06	25.51	1.06
25	13.92	0.53	12.42	0.48	25.35	0.55	23.64	0.89
26A	15.30	0.36	13.11	0.17	28.07	0.22	25.12	0.25
27A	14.78	0.49	13.36	0.26	27.14	0.48	25.14	0.57
27B	14.50	0.35	12.90	0.52	26.84	0.64	24.68	0.88
28A	14.60	0.45	13.40	0.31				
28B	14.24	0.38	13.16	0.48				
29A	14.39	0.23	12.96	0.23	26.95	0.84	25.58	0.70
29B	14.21	0.24	12.88	0.31				
30A	14.18	0.27	12.37	0.35	26.64	0.41	23.85	0.39
30B	13.98	0.57	12.25	0.87				
31A	14.07	0.28	12.36	0.35	25.49	0.59	22.96	0.26
31B	14.34	0.37	12.79	0.41	26.94	0.38	24.31	0.28
32	14.42	0.32	13.00	0.32	27.22	0.13	25.36	0.27
33A	14.62	0.29	12.40	0.32				
33B	14.53	0.35	12.16	0.23	26.01	0.46	23.27	0.35
35	13.70	0.47	12.61	0.53	26.78	0.53	24.24	1.04
39	15.25	0.36	14.18	0.33	27.94	0.49	26.30	0.61
40	13.44	0.25	12.14	0.21	25.82	0.34	24.37	0.27
41	13.77	0.26	12.30	0.51	26.70	0.37	24.38	0.32
42A	14.45	0.28	13.30	0.17	26.73	0.36	25.17	0.33
42B	14.70	0.18	13.90	0.23	27.12	0.20	25.65	0.17
43			11.92	0.13				
44	13.78	0.38	12.08	0.41	26.37	0.43	24.12	0.97
45								

Table 10: Puff Count mean and standard deviation per laboratory code

Labs	Puff Count							
	ISO				Intense			
	CM6		CM7		CM6		CM7	
	Mean	Sd	Mean	Sd	Mean	Sd	Mean	Sd
1	9.12	0.08	8.81	0.09	12.90	0.23	12.19	0.29
2A	8.91	0.04	8.35	0.16	12.93	0.11	12.11	0.17
2B	9.01	0.07	8.49	0.05	12.80	0.04	11.75	0.22
3A	8.81	0.16	8.29	0.16	12.70	0.21	11.75	0.22
3B	8.88	0.05	8.28	0.08	12.79	0.14	11.78	0.08
3C	9.29	0.05	8.77	0.18	13.21	0.09	12.27	0.19
4	9.25	0.11	8.63	0.22	12.97	0.11	11.90	0.15
5	9.21	0.06	8.44	0.22	12.65	0.10	11.47	0.23
6	9.06	0.07	8.40	0.07	13.08	0.13	12.25	0.30
8A	8.94	0.12	8.04	0.05	12.90	0.18	11.41	0.16
8B	8.94	0.07	8.14	0.10				
8C	8.69	0.07	7.90	0.13	12.51	0.20	11.23	0.08
9A	9.10	0.16	8.43	0.25	12.68	0.32	11.64	0.15
9B	9.02	0.11	8.38	0.05	13.02	0.20	11.99	0.14
10A	9.05	0.08	8.40	0.10				
10B	8.65	0.11	7.92	0.12	12.31	0.09	11.17	0.19
11A	9.35	0.11	8.72	0.16	13.32	0.30	12.28	0.29
11B	8.93	0.08	8.24	0.12	12.84	0.17	11.63	0.09
11C	8.93	0.06	8.24	0.07	12.91	0.26	11.77	0.19
12A	9.39	0.08	8.73	0.07	13.16	0.08	13.16	0.08
12B	8.90	0.10	8.16	0.04	12.69	0.18	11.71	0.14
14	8.67	0.10	8.01	0.04	12.55	0.18	11.55	0.07
16	8.77	0.08	8.11	0.07	12.78	0.11	11.64	0.18
18A	9.14	0.09	8.53	0.20	13.04	0.10	12.00	0.14
18B	9.04	0.11	8.26	0.09				
19	9.76	0.31	8.97	0.09	13.59	0.21	12.63	0.04
21	9.13	0.26	8.43	0.25	12.52	0.26	12.52	0.26
25	9.10	0.21	8.47	0.26	12.58	0.13	11.68	0.25
26A	8.60	0.09	7.87	0.13	12.36	0.15	11.41	0.13
27A	8.72	0.13	8.03	0.08	12.34	0.33	11.34	0.15
27B	9.24	0.18	8.53	0.10	12.90	0.16	12.01	0.18
28A	8.80	0.08	8.26	0.15				
28B	8.82	0.12	8.25	0.19				
29A	9.28	0.07	8.70	0.11	13.51	0.12	12.61	0.18
29B	9.06	0.07	8.33	0.07				
30A	9.13	0.13	8.35	0.17	12.89	0.18	11.89	0.06
30B	9.17	0.11	8.37	0.17				
31A	10.03	0.08	9.09	0.09	13.71	0.16	12.63	0.08
31B	9.39	0.16	8.75	0.21	13.22	0.19	12.54	0.08
32	8.98	0.07	8.31	0.12	12.99	0.23	11.95	0.14
33A	8.90	0.11	8.17	0.08				
33B	8.78	0.04	8.08	0.09	12.57	0.16	11.57	0.29
35	8.85	0.11	8.18	0.11	12.50	0.13	11.50	0.06
39	9.51	0.15	8.71	0.07	13.41	0.15	12.55	0.24
40	9.15	0.11	8.55	0.07	12.89	0.14	12.09	0.07
41	9.27	0.10	8.49	0.13	13.00	0.09	11.85	0.08
42A	8.93	0.13	8.24	0.03	12.86	0.15	11.88	0.13
42B	8.74	0.09	7.97	0.03	12.45	0.18	11.24	0.11
43			8.19	0.04				
44	8.95	0.24	8.19	0.13	12.50	0.13	11.61	0.28
45	8.60	0.08	7.97	0.14				

4. STATISTICAL ANALYSIS

4.1 Exclusion of Outliers

4.1.1 Mandel graphical outlier technique:

The graphical representation of the Mandel's tests was examined first.

ISO 5725-2 Tests	Consistency
Mandel's k	Within-laboratory variability.
Mandel's h	Between-laboratory variability.

Examination of h and k plots indicated the presence of any suspicious result regarding the within-laboratory standard deviation or any extreme laboratory mean value compared to others.

In the simple bar plots, lines were drawn indicating 0.95 stragglings and 0.99 outlying limits. Please note that at this stage definite decisions were delayed until completion of numerical outlier techniques mentioned in 5.1.2. Mandel h and k plots are available in the Appendix E additional folder.

4.1.2 Numerical outlier technique: Cochran & Grubbs test

The statistical evaluation of data for this collaborative study followed the methods provided by ISO 5725-2¹. For outlier testing, the Grubbs and Cochran methods were used.

Note: ISO 5725-2 only provides critical values of the Cochran C and Grubbs statistics up to n=40. Since there were up to n=54 machines included in this study, the critical values applied for n>40 were determined by the software "R" for the Cochran test, and as described in the article² "*Extension of Sample Sizes and Percentage Points for Significance Tests of Outlying Observations*" for the Grubbs' test.

ISO 5725-2 Tests	Consistency
Cochran's – test	Within-laboratory variability: Suitable for detecting whether the highest value in a set of laboratory standard deviations is an outlier or not.
Grubbs' – test	Between-laboratory variability: Suitable for detecting whether the highest (or lowest) laboratories averages are outliers or not.

The protocol "Harmonized statistical procedure" defined by IUPAC (International Union of Pure and Applied Chemistry) has been applied as well. It consists of sequential applications of the Cochran and Grubbs tests until no further outliers are detected or until a drop of more than 22.2% (for this study =2/9) in the original number of laboratories would occur (see flowchart in Appendix F).

First apply the Cochran outlier test, and if an outlying laboratory is identified, then a single values Grubbs test is performed on the individual values of this outlying laboratory (Individual Grubbs). If no individual value is identified as outlier, the

¹ "ISO 5725-2:1994 : Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

² Frank E. Grubbs And Glenn Beck, *Extension of Sample Sizes and Percentage Points for Significance Tests of Outlying Observations*, TECHNOMETRICS, vol.14(4) (1972).

outlying laboratory is removed. When an individual value is identified as an outlier, only this value is removed.

Afterwards single-value Grubbs test is applied and outlying laboratories are removed. If no laboratory is identified as outlier, the pair-value test is applied (two values at the same end). Remove any laboratory(ies) flagged by these tests, but stop removal if more than 22.2% (2 of 9 laboratories) would be removed.

Note:

- The paired value Grubbs' test is an extension of the single value Grubbs' test for pairs of outliers. The use is advised in ISO 5725 to detect pairs of outlier undetectable sequentially by using single value Grubbs' test.

4.1.3 Outlier results

Table 11 lists the laboratories which were rejected (by analyte and smoking regime), and states the outlier exclusion technique used. A total of 9 smoking machines have been rejected at least once for either one product, one smoking regime or one parameter (1, 19, 21, 25, 27B, 30A, 30B, 31A, 44) respectively. A large number of outliers (96%) relate to within-laboratory variability. The outliers detected by Cochran's test are mainly attributable to two laboratories (30B and 44).

Table 11: List of Outliers

Coresta monitor	Smoking Regimes	Analyte	All smoking machines		
			Cochran's Outliers	Grubbs' Outliers	Grubbs for individual Outliers
CM6		WEIGHT			
	ISO	TPM	30B		30B
		WATER	44		
		Nicotine	30B		
		NFDPM	30B		
		CO			
PUFF	19	31A			
Intense	TPM		27B, 30A, 44		
	WATER				
	Nicotine		30A, 44		
	NFDPM		21		
	CO				
PUFF					
CM7		WEIGHT			
	ISO	TPM	30B		1
		WATER	1, 44		
		Nicotine	30B		
		NFDPM	30B		
		CO	30B		
PUFF					
Intense	TPM		1, 25, 44		1
	WATER		30A, 44		
	Nicotine				
	NFDPM				
	CO				
PUFF					

4.2 Repeatability and Reproducibility estimation

Repeatability and reproducibility limits are calculated for both test pieces, both smoking regimes, each parameter and for both smoking machine types. The results are summarized in tables 12, 13 and 14. The corresponding graphs named “individual values” are given in Part 6 of this report.

Table 12: r&R estimation of test piece weight

Parameter	Coresta Monitor	No of Labs	Mean	Repeatability		Reproducibility		Outlying Labs			Stragglng Labs	No data
				sr	r	sR	R	C	G/G2	Gi		
WEIGHT	CM6	50	971.8	4.3	12.1	6.4	18.0				35, 44	
	CM7	51	957.2	4.6	13.0	7.1	20.0				35, 44	

Table 13: r&R estimation for CM6 and CM7 under ISO smoking regime

Parameter	Coresta Monitor	Machine class	No of Labs	Mean	Repeatability		Reproducibility		Outlying Labs			Stragglng Labs	No data
					sr	r	sR	R	C	G/G2	Gi		
TPM	CM6	ALL	49	17.43	0.33	0.94	0.61	1.72	30B				43
		LINEAR	21	17.77	0.41	1.16	0.61	1.73					
		ROTARY	28	17.17	0.26	0.73	0.46	1.30	30B				43
	CM7	ALL	50	16.37	0.33	0.93	0.63	1.77	30B				
		LINEAR	21	16.69	0.42	1.18	0.69	1.95					
		ROTARY	29	16.13	0.24	0.69	0.45	1.28	30B				
WATER	CM6	ALL	49	1.84	0.17	0.47	0.35	0.98	44		30B	30A	43
		LINEAR	21	1.59	0.20	0.58	0.28	0.80		30B	44	43	
		ROTARY	28	2.05	0.15	0.43	0.23	0.66					
	CM7	ALL	49	1.78	0.15	0.41	0.34	0.97	1, 44				
		LINEAR	19	1.48	0.17	0.48	0.26	0.73	1, 44				
		ROTARY	30	1.96	0.13	0.36	0.24	0.69				10B, 30B	
Nicotine	CM6	ALL	49	1.362	0.030	0.084	0.061	0.173	30B				43
		LINEAR	21	1.398	0.033	0.094	0.063	0.179					
		ROTARY	28	1.335	0.027	0.076	0.044	0.124	30B				43
	CM7	ALL	50	1.236	0.026	0.075	0.053	0.150	30B				
		LINEAR	20	1.276	0.031	0.089	0.041	0.116		25			
		ROTARY	29	1.212	0.022	0.062	0.040	0.114	30B				
NFDPM	CM6	ALL	49	14.21	0.28	0.80	0.68	1.92	30B				43
		LINEAR	21	14.78	0.37	1.03	0.58	1.64					
		ROTARY	28	13.78	0.20	0.56	0.35	0.98	30B				43
	CM7	ALL	50	13.34	0.27	0.77	0.67	1.88	30B		1		
		LINEAR	21	13.89	0.34	0.96	0.60	1.70			1	44	
		ROTARY	29	12.94	0.21	0.59	0.35	0.98	30B				
CO	CM6	ALL	49	14.42	0.34	0.95	0.65	1.83					43, 45
		LINEAR	21	14.10	0.34	0.97	0.64	1.82					
		ROTARY	28	14.66	0.33	0.94	0.55	1.54				8A, 12B	43, 45
	CM7	ALL	49	12.93	0.34	0.95	0.68	1.94	30B			21	45
		LINEAR	21	12.72	0.42	1.19	0.70	1.97					
		ROTARY	28	13.09	0.25	0.71	0.64	1.81	30B				45
Puff Count	CM6	ALL	48	9.00	0.12	0.33	0.25	0.69	19	31A		21	43
		LINEAR	19	9.19	0.14	0.40	0.20	0.58		19, 31A		19, 31A	
		ROTARY	29	8.88	0.09	0.26	0.19	0.53					43
	CM7	ALL	51	8.36	0.13	0.38	0.30	0.86					
		LINEAR	21	8.58	0.16	0.46	0.27	0.77					
		ROTARY	30	8.19	0.11	0.30	0.21	0.58					

Table 14: r&R estimation for CM6 and CM7 under intense smoking regime

Parameter	Coresta Monitor	Machine class	No of Labs	Mean	Repeatability		Reproducibility		Outlying Labs			Stragglng Labs	No data
					sr	r	sR	R	C	G/G2	Gi		
TPM	CM6	ALL	41	42.57	0.78	2.20	3.33	9.43					
		LINEAR	21	45.23	0.97	2.75	2.36	6.67					
		ROTARY	20	39.76	0.50	1.42	1.17	3.30					
	CM7	ALL	38	41.70	0.73	2.07	3.32	9.38	1, 25, 44			44	
		LINEAR	21	44.52	1.20	3.39	2.65	7.51					
		ROTARY	20	39.16	0.50	1.42	1.30	3.68					
WATER	CM6	ALL	38	10.79	0.50	1.40	1.91	5.41	27B, 30A, 44				
		LINEAR	19	12.24	0.67	1.89	1.59	4.50	30A, 44				
		ROTARY	20	9.48	0.35	0.99	0.99	2.80					
	CM7	ALL	39	10.86	0.50	1.41	1.93	5.47	30A, 44			27B	
		LINEAR	20	12.13	0.66	1.86	1.78	5.04	44			30A	
		ROTARY	20	9.58	0.39	1.10	0.96	2.71				14	
Nicotine	CM6	ALL	41	2.699	0.047	0.133	0.125	0.353					
		LINEAR	21	2.758	0.048	0.136	0.123	0.347					
		ROTARY	20	2.638	0.046	0.131	0.095	0.269				29A	
	CM7	ALL	41	2.529	0.050	0.141	0.112	0.316					
		LINEAR	21	2.587	0.050	0.142	0.110	0.310				19, 25	
		ROTARY	20	2.468	0.049	0.139	0.077	0.218					
NFDPM	CM6	ALL	39	29.02	0.55	1.55	1.76	4.97	30A, 44			44	
		LINEAR	20	30.27	0.75	2.13	1.61	4.57	30A				
		ROTARY	20	27.65	0.38	1.06	0.53	1.49					
	CM7	ALL	41	28.48	0.70	1.99	1.95	5.50			1		
		LINEAR	21	29.80	0.90	2.53	1.84	5.20			1		
		ROTARY	20	27.12	0.42	1.18	0.73	2.06					
CO	CM6	ALL	40	26.76	0.42	1.19	0.86	2.43	21				
		LINEAR	21	26.38	0.51	1.44	0.88	2.48					
		ROTARY	19	27.11	0.33	0.94	0.74	2.09	29A				
	CM7	ALL	41	24.87	0.52	1.46	1.03	2.90				21	
		LINEAR	21	24.54	0.63	1.79	1.09	3.08					
		ROTARY	20	25.21	0.36	1.00	0.85	2.39				2A, 29A, 33B	
Puff Count	CM6	ALL	41	12.87	0.18	0.50	0.37	1.06					
		LINEAR	21	12.97	0.17	0.49	0.38	1.08					
		ROTARY	20	12.75	0.18	0.52	0.34	0.95					
	CM7	ALL	41	11.91	0.18	0.50	0.48	1.36					
		LINEAR	21	12.10	0.18	0.52	0.48	1.35					
		ROTARY	20	11.70	0.17	0.48	0.40	1.12					

5. DATA REPRESENTATION

The following figures show the raw data for CM6 and CM7 for all parameter. The plots indicate mean values with 95% confidence interval (CI) highlighted in green (for valid participants) or in red (for participants detected as outlier) for each smoking machine. Laboratories excluded by outlier statistics are denoted by the name of the test rejecting the laboratory (G for Grubbs, G2 for double Grubbs and C for Cochran). The confidence limits at 95% and 99% for the difference between the reference value (average of all laboratories) and the average of one laboratory are plotted in green and red respectively.

Lab codes of participants that did not provide data are marked with an asterisk.

5.1 ISO smoking regime

5.1.1 CM6

Figure 1: CM6 Individual observations of conditioned test piece WEIGHT

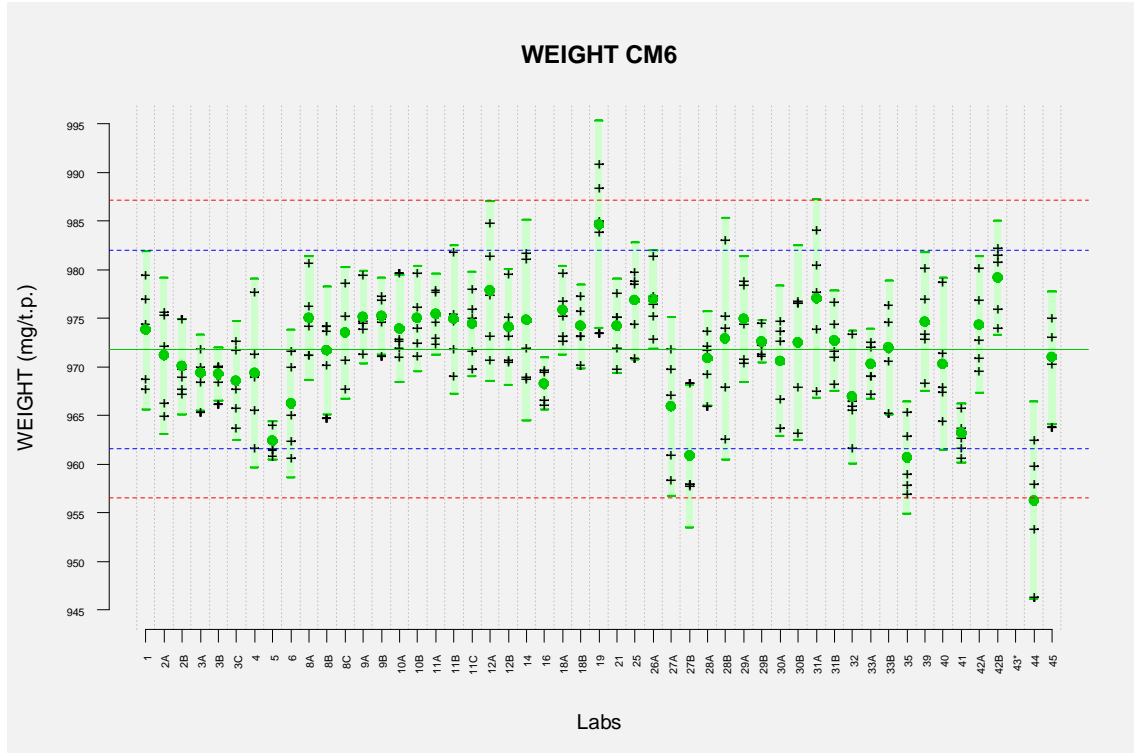


Figure 2: CM6 Individual observations of mainstream "ISO" TPM

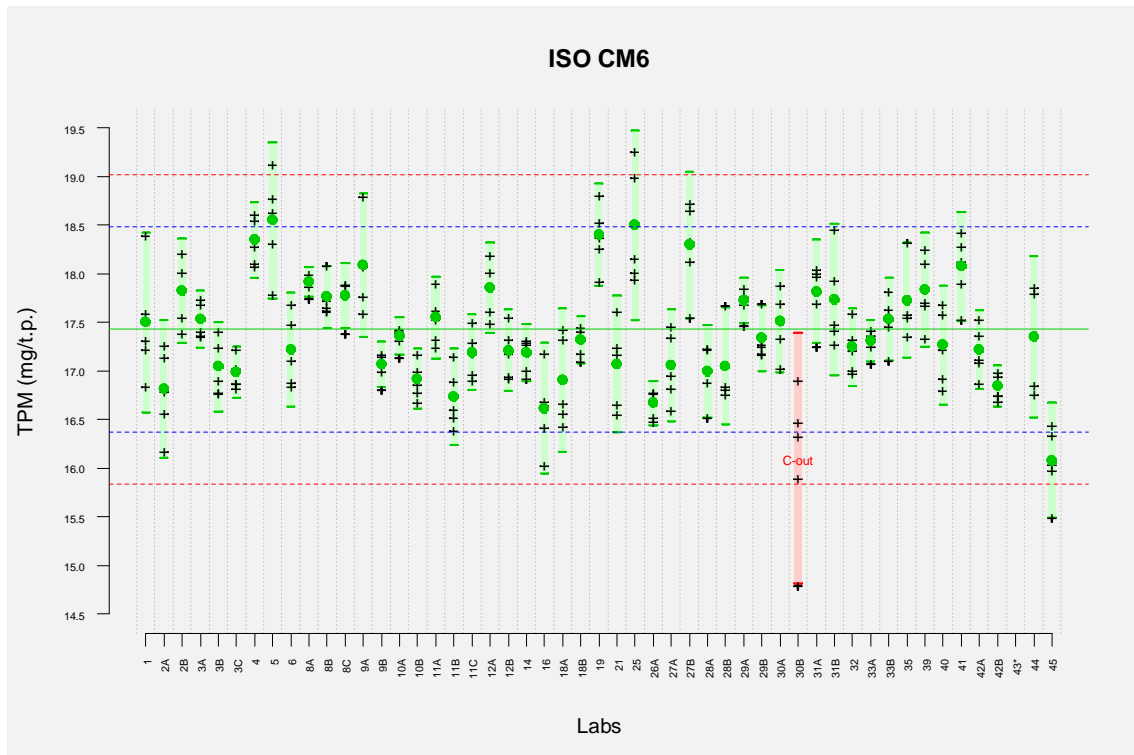


Figure 3: CM6 Individual observations of mainstream “ISO” WATER

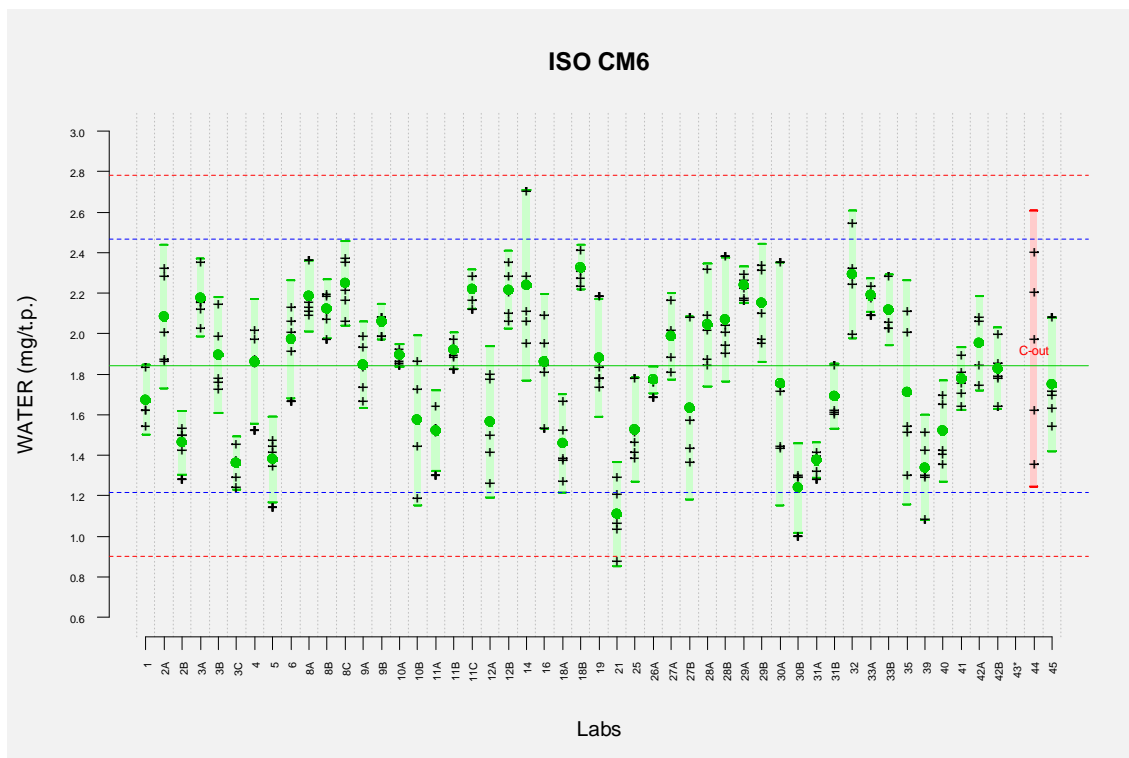


Figure 4: CM6 Individual observations of mainstream “ISO” Nicotine

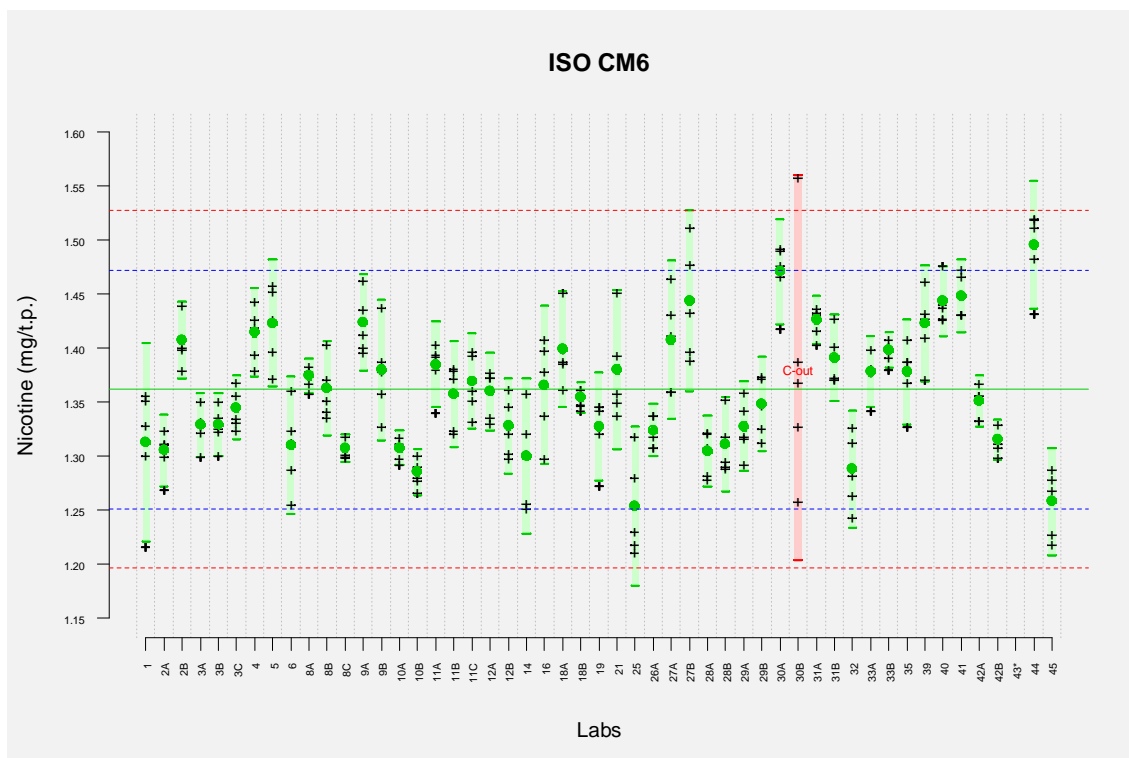


Figure 5: CM6 Individual observations of mainstream “ISO” NFDPM

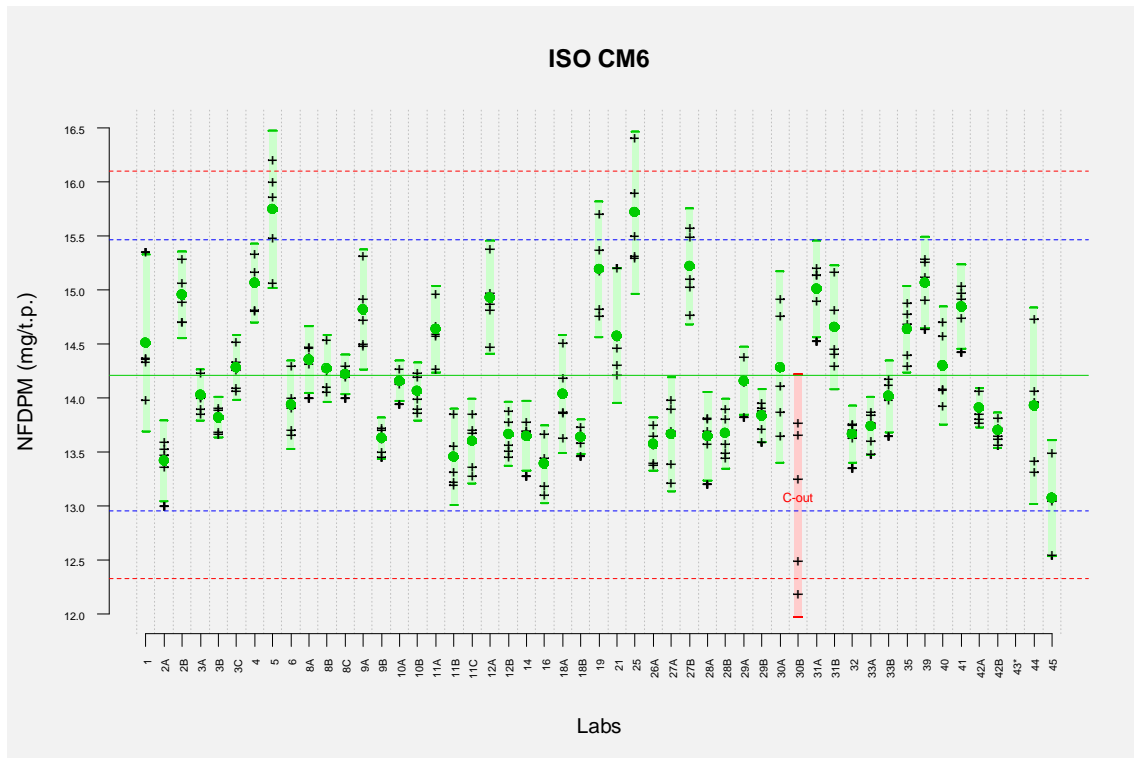


Figure 6: CM6 Individual observations of mainstream “ISO” CO

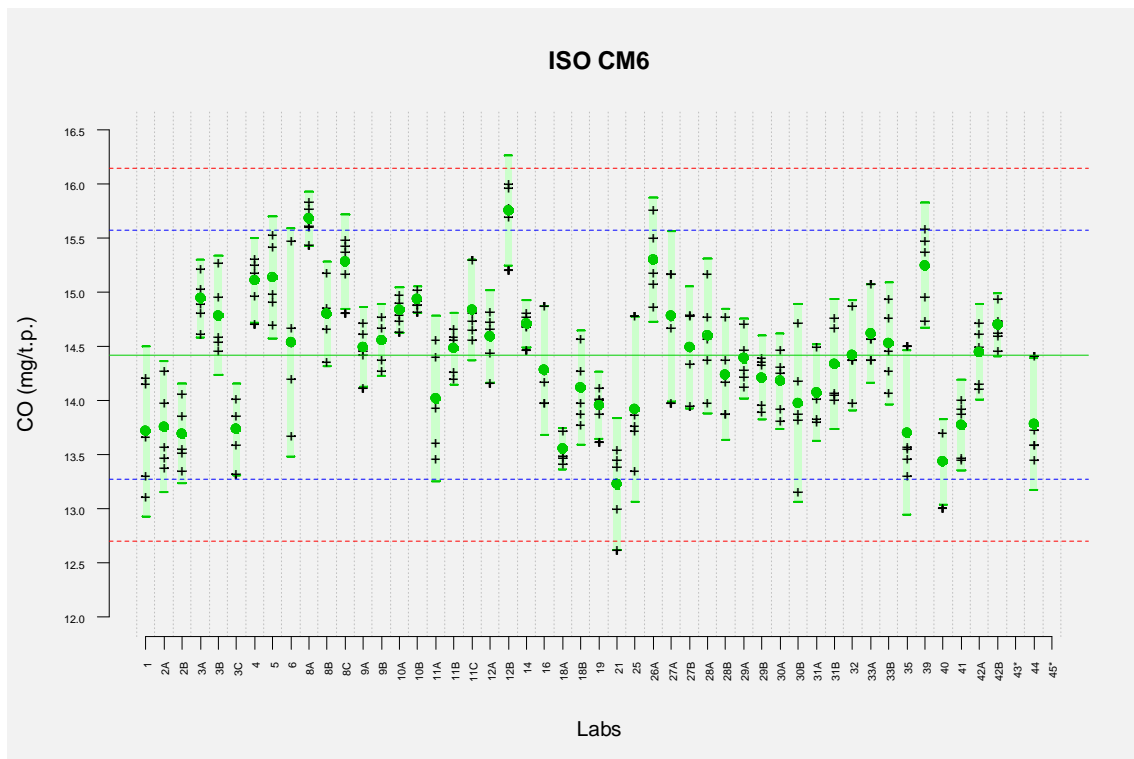
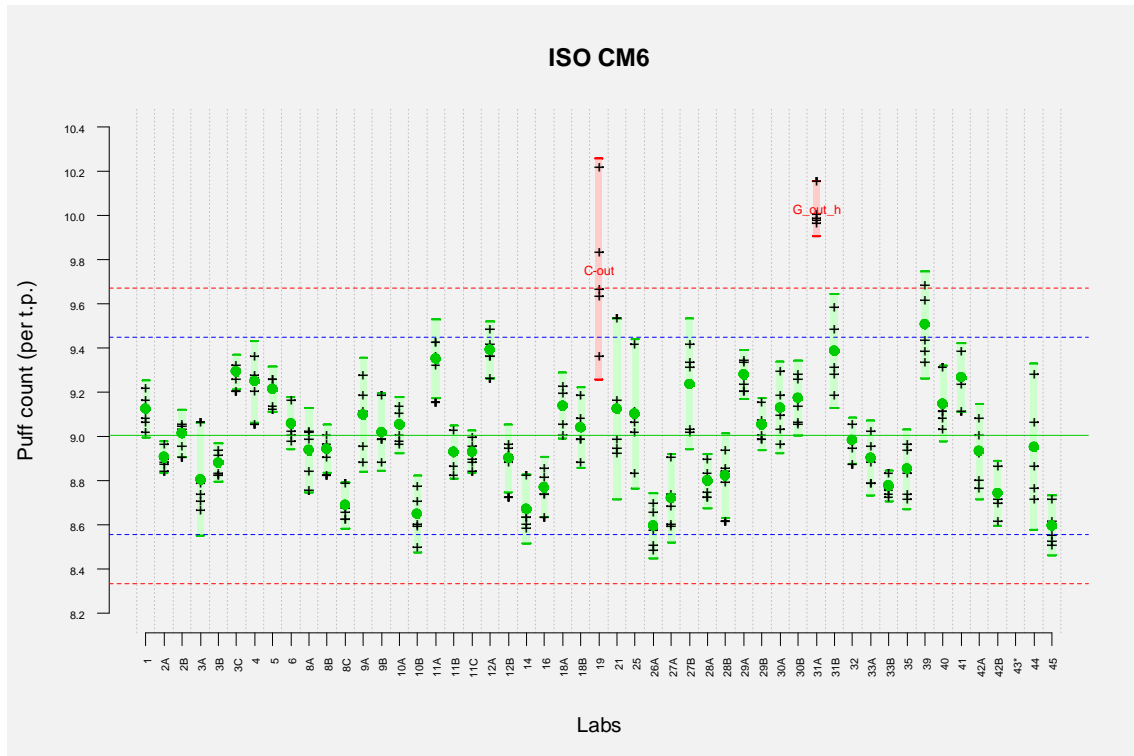


Figure 7: CM6 Individual observations of mainstream “ISO” PUFF Count



5.1.2 CM7

Figure 8: CM7 Individual observations of conditioned test piece WEIGHT

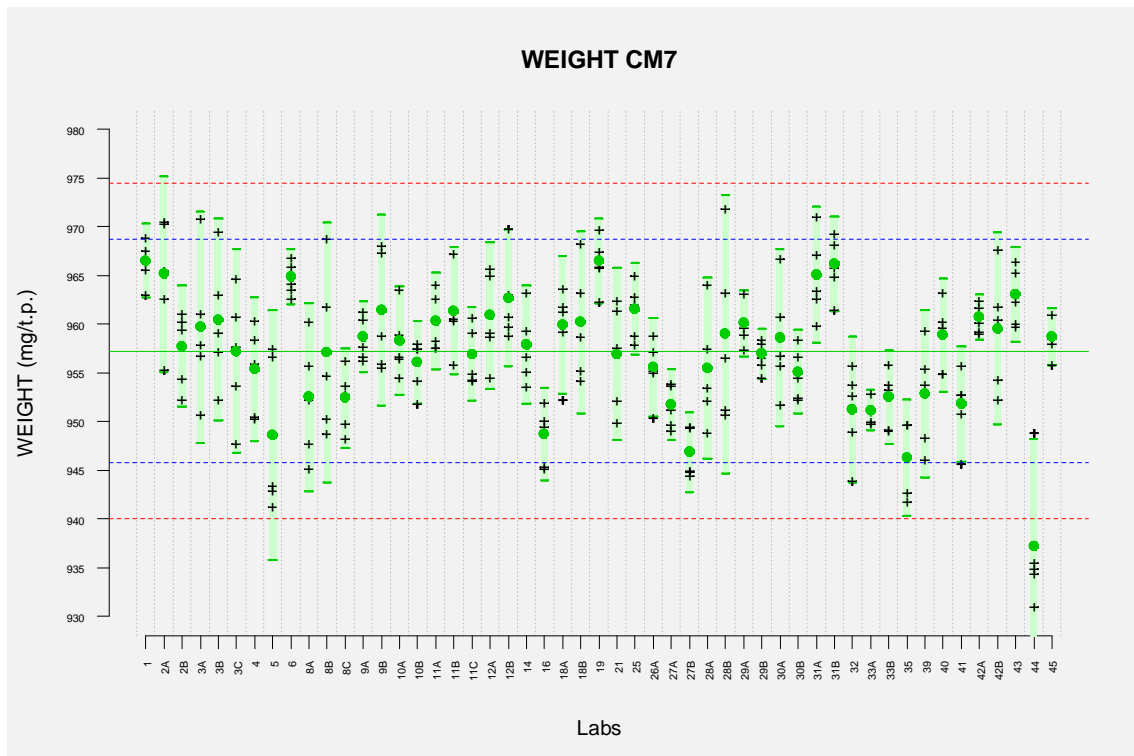


Figure 9: CM7 Individual observations of mainstream "ISO" TPM

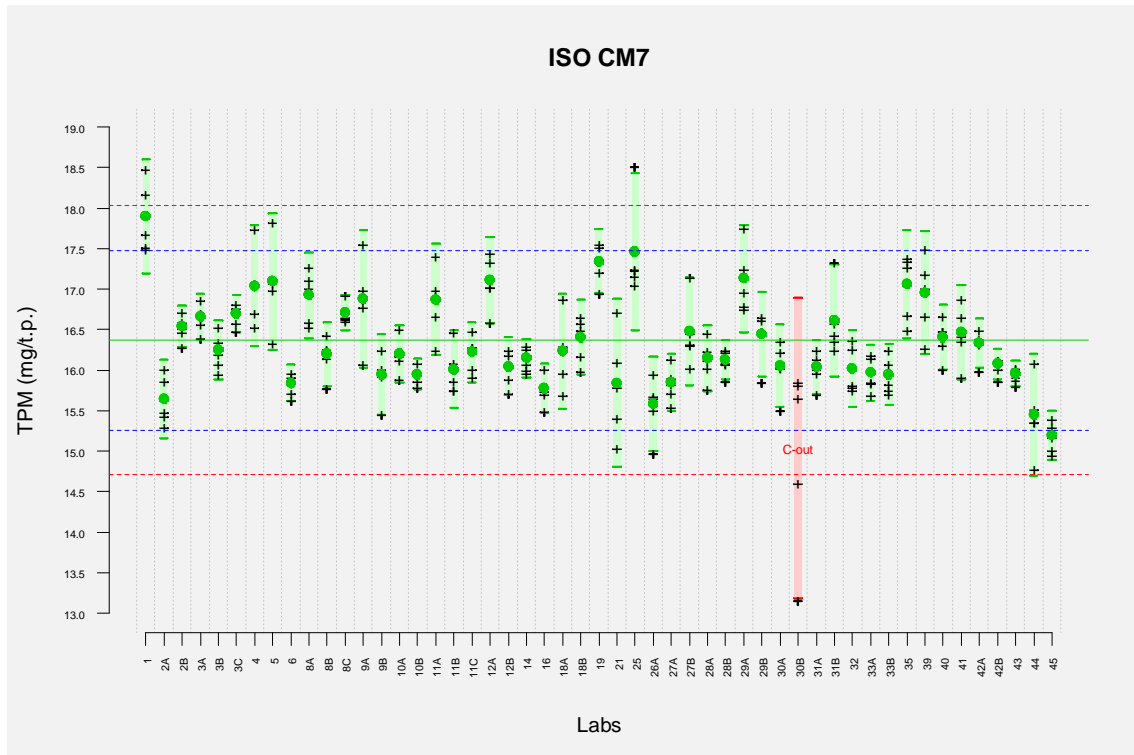


Figure 10: CM7 Individual observations of mainstream "ISO" WATER

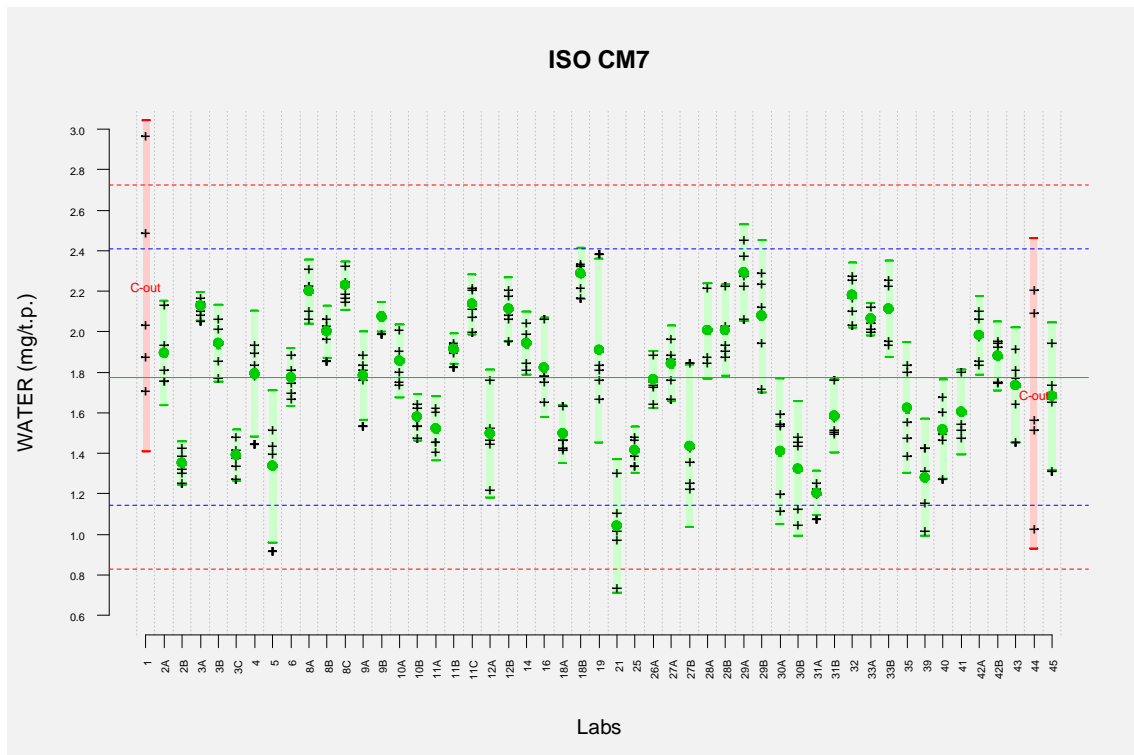


Figure 11: CM7 Individual observations of mainstream "ISO" Nicotine

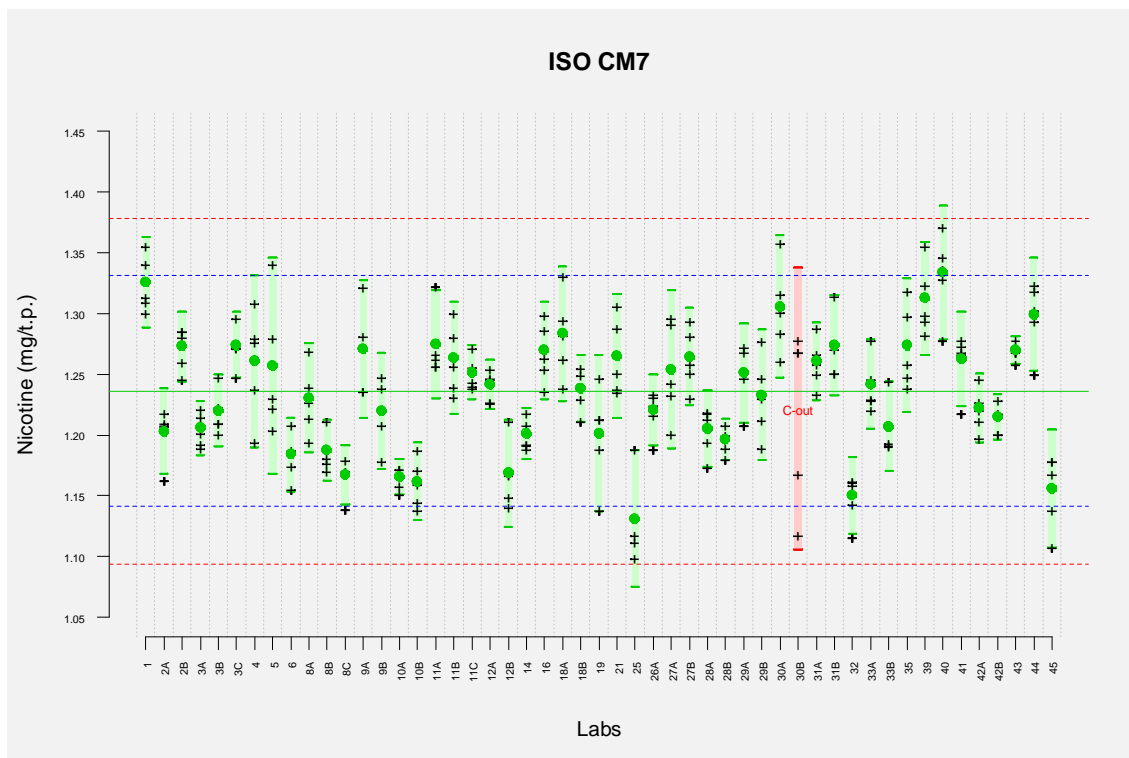


Figure 12: CM7 Individual observations of mainstream "ISO" NFDPM

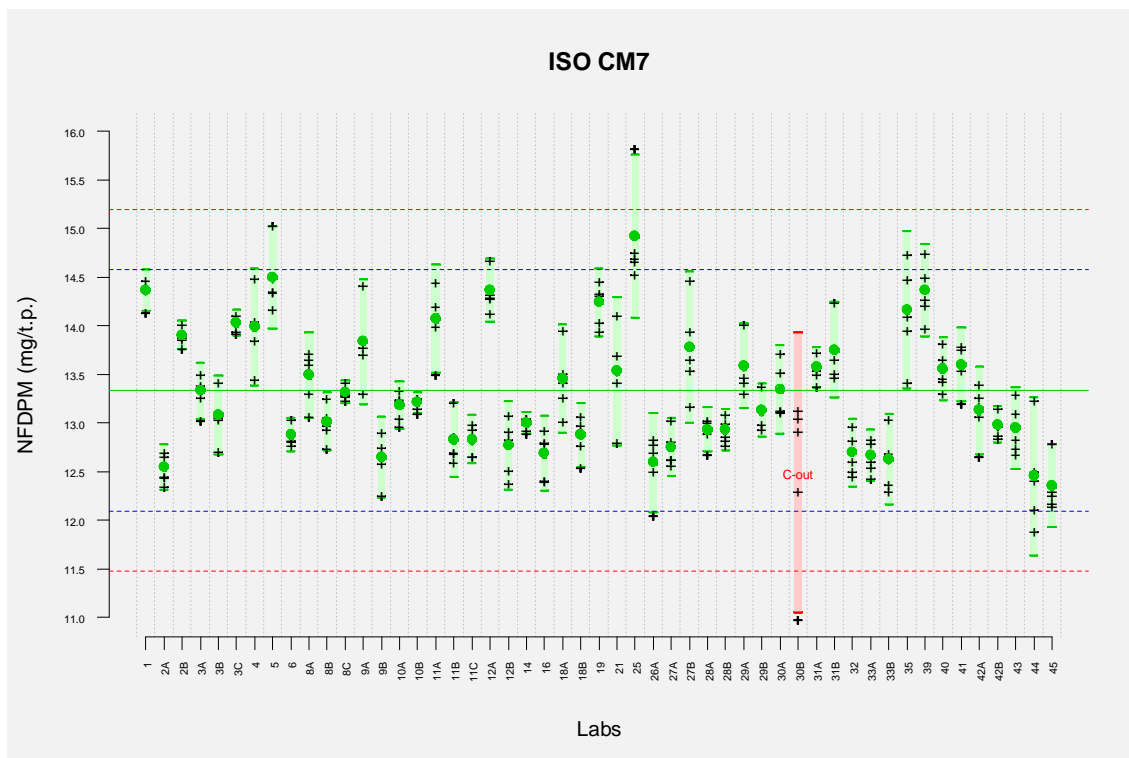


Figure 13: CM7 Individual observations of mainstream "ISO" CO

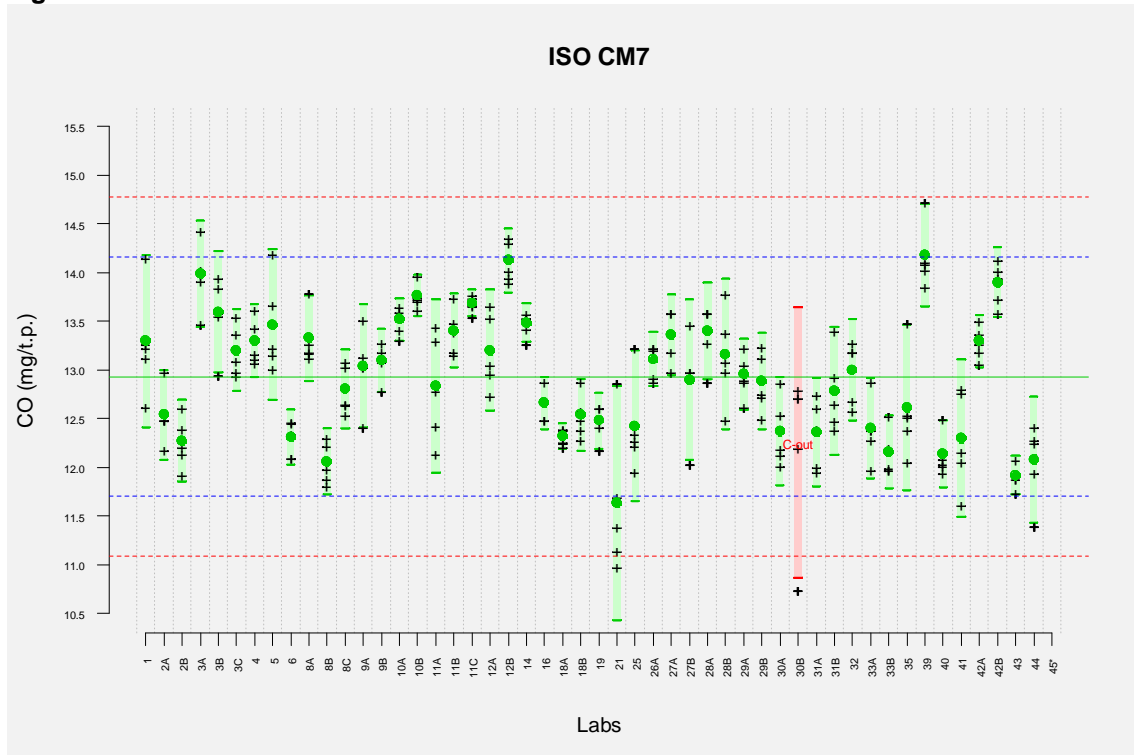
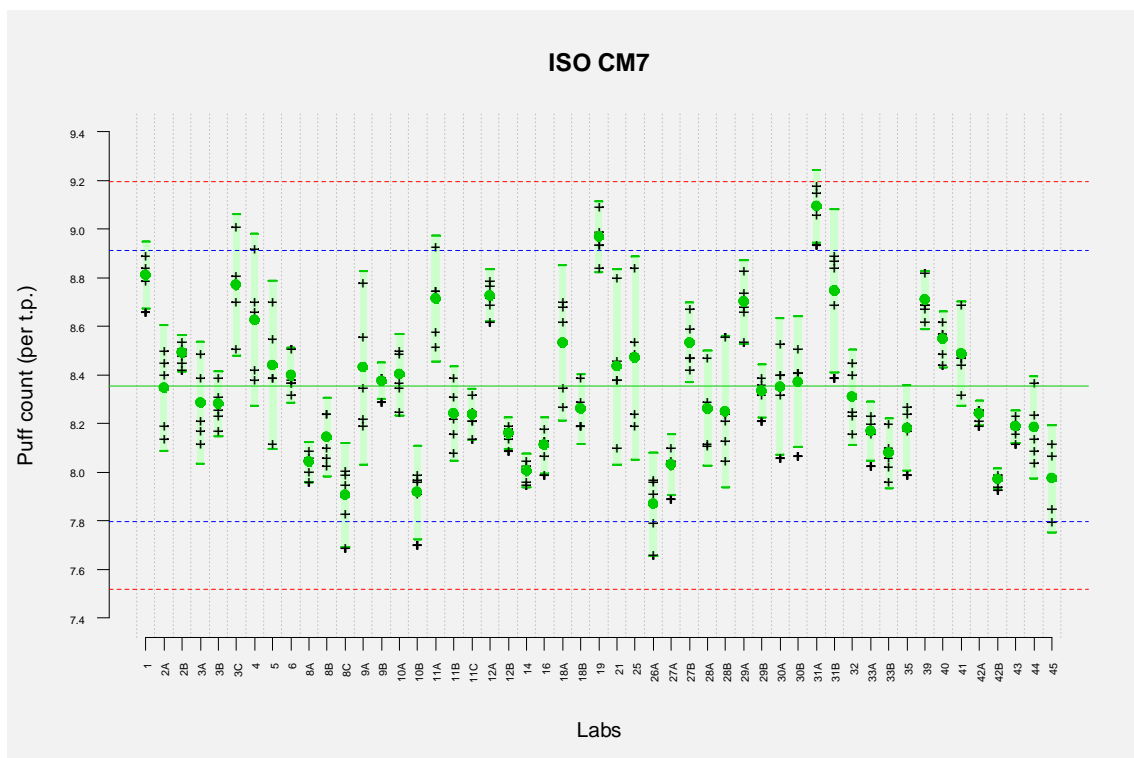


Figure 14: CM7 Individual observations of mainstream "ISO" PUFF Count



5.2 Intense smoking regime

5.2.1 CM6

Figure 15: CM6 Individual observations of mainstream “Intense” TPM

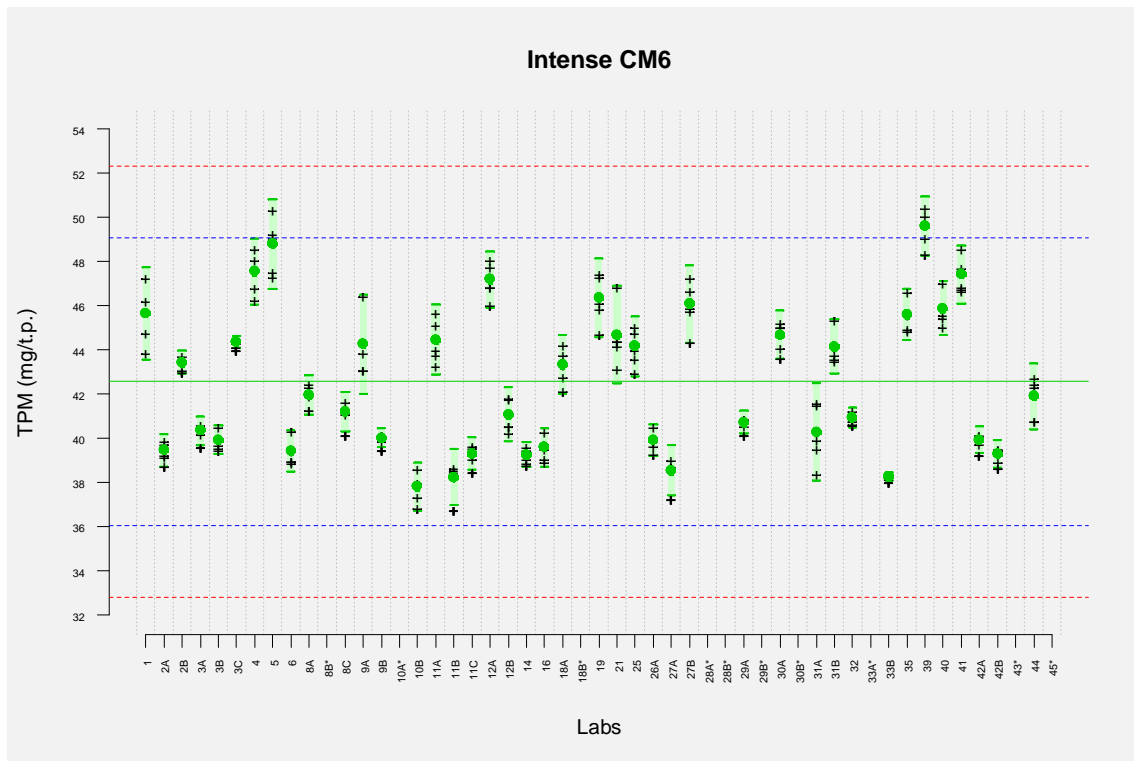


Figure 16: CM6 Individual observations of mainstream “Intense” WATER

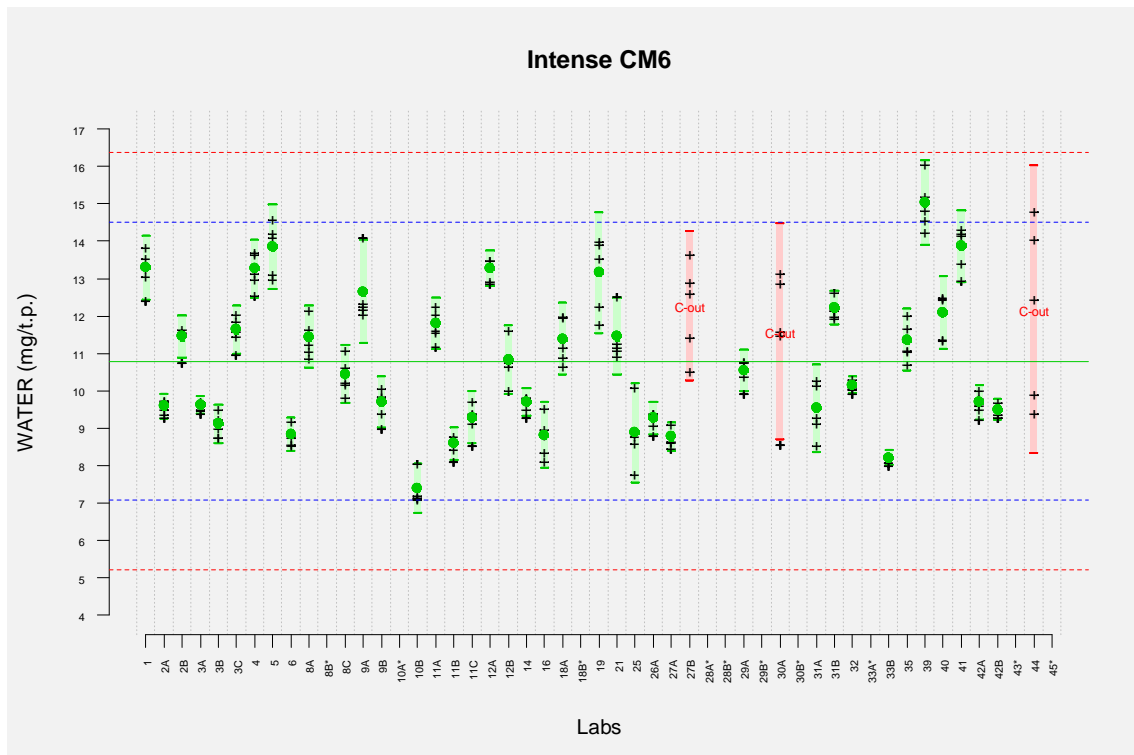


Figure 17: CM6 Individual observations of mainstream “Intense” Nicotine

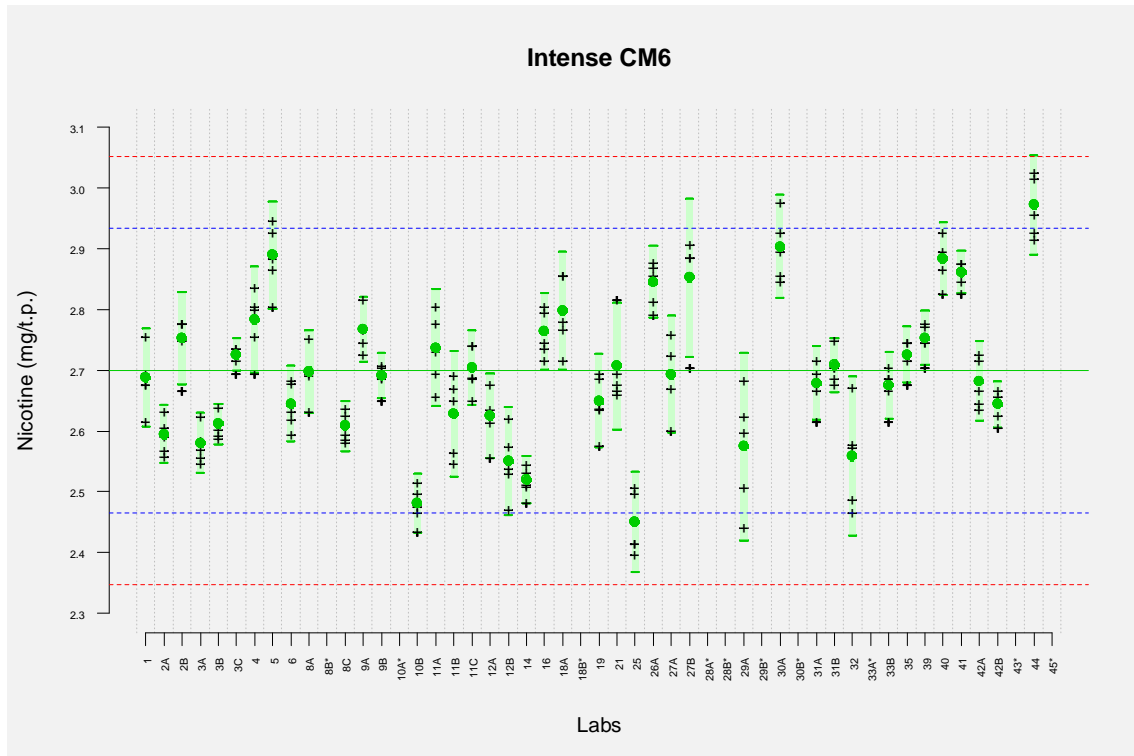


Figure 18: CM6 Individual observations of mainstream “Intense” NFDPM

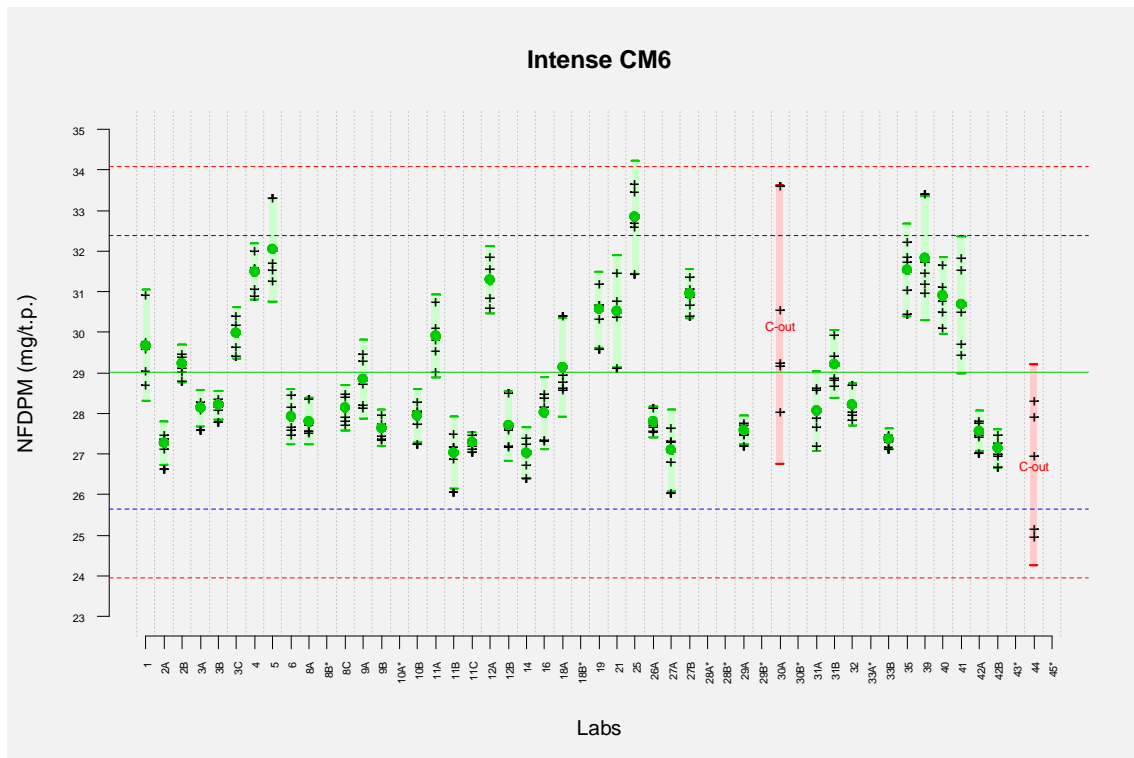


Figure 19: CM6 Individual observations of mainstream "Intense" CO

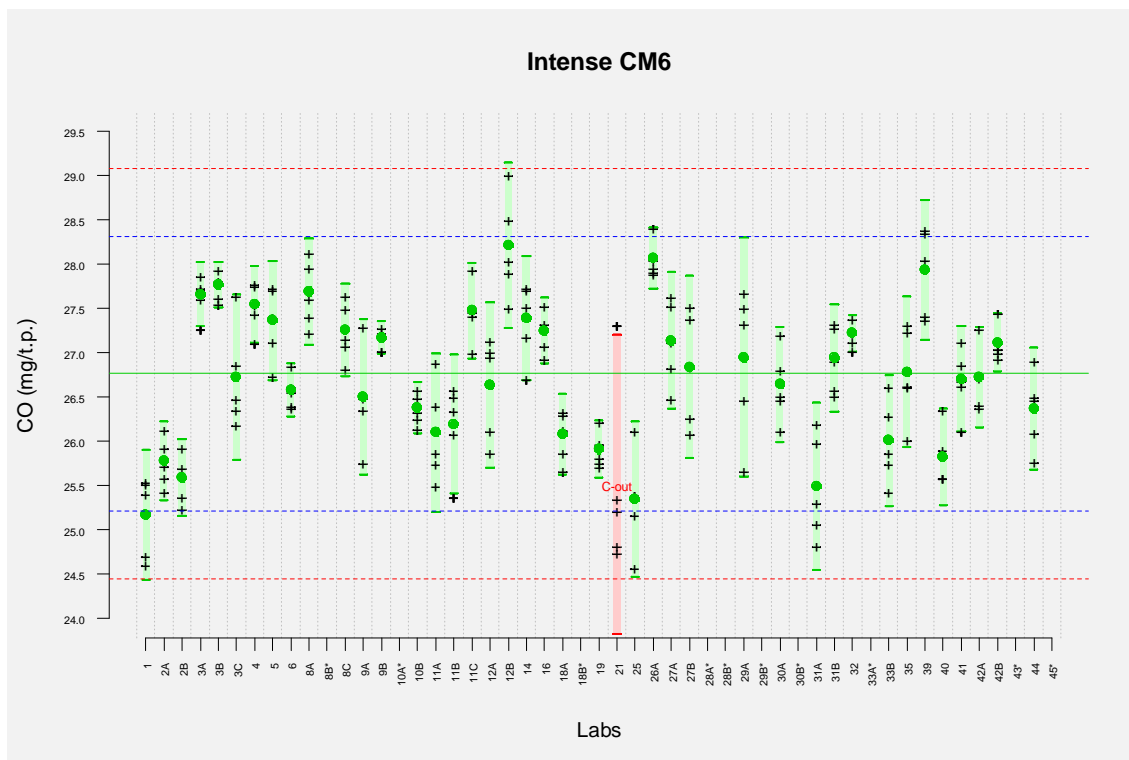
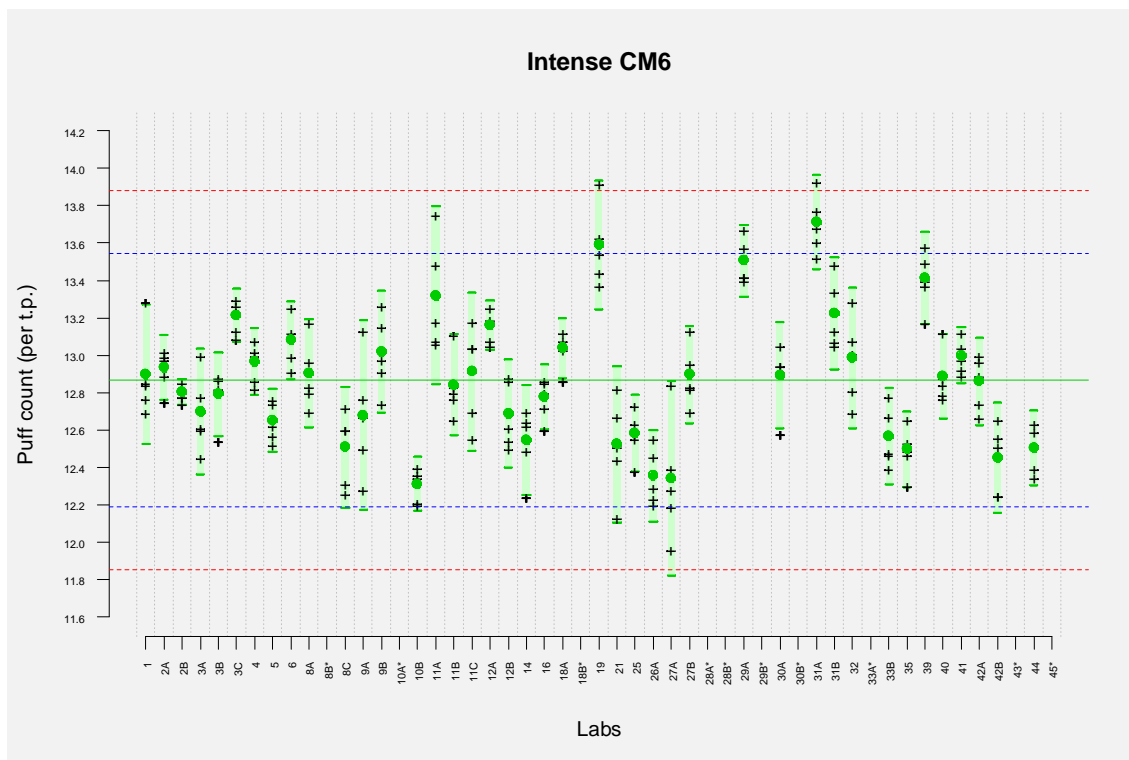


Figure 20: CM6 Individual observations of mainstream "Intense" PUFF Count



5.2.2 CM7

Figure 21: CM7 Individual observations of mainstream “Intense” TPM

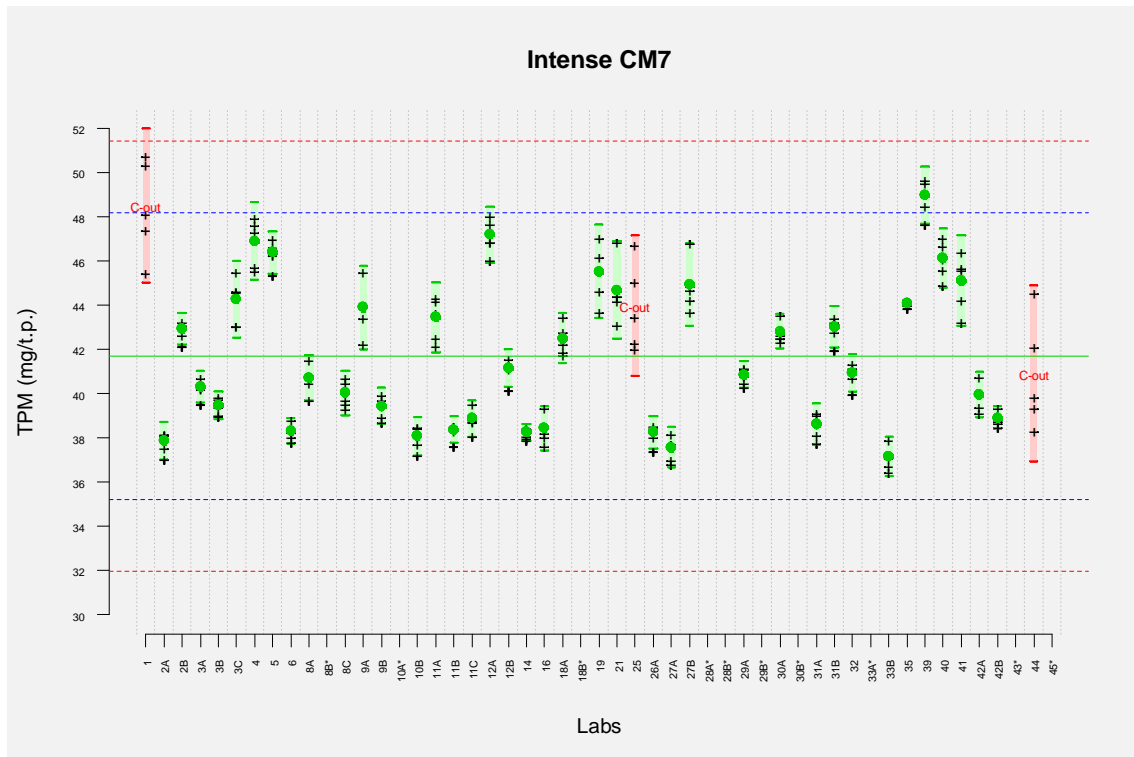


Figure 22: CM7 Individual observations of mainstream “Intense” WATER

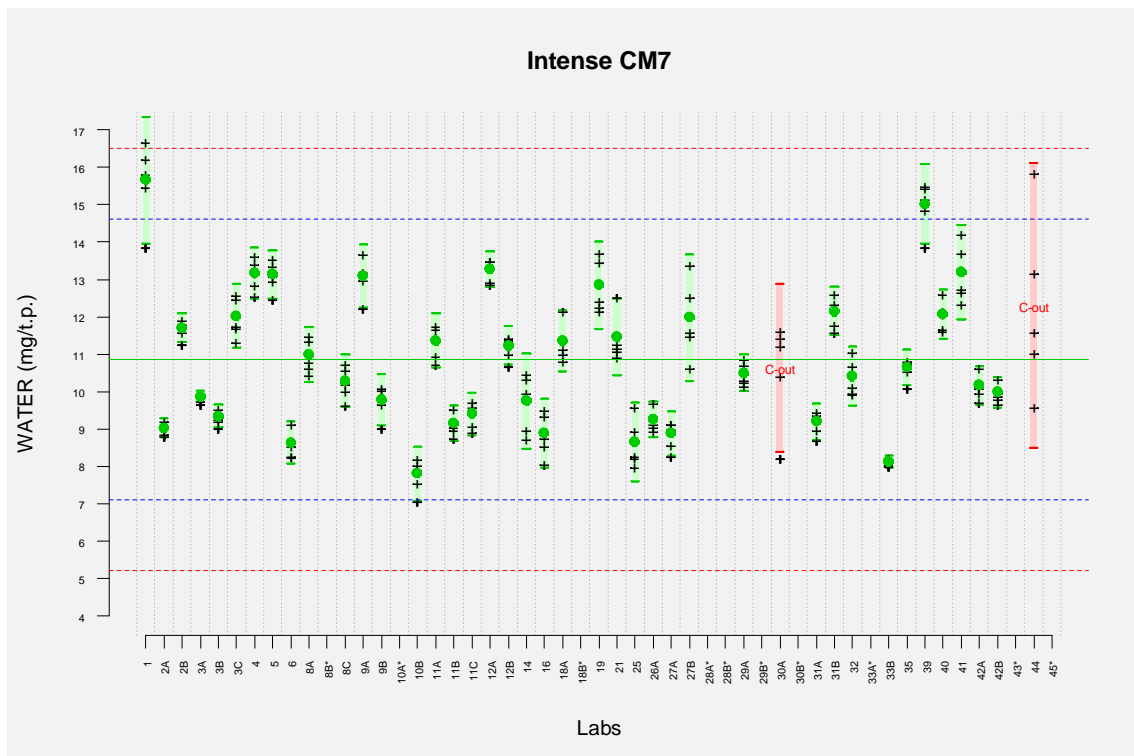


Figure 23: CM7 Individual observations of mainstream “Intense” Nicotine

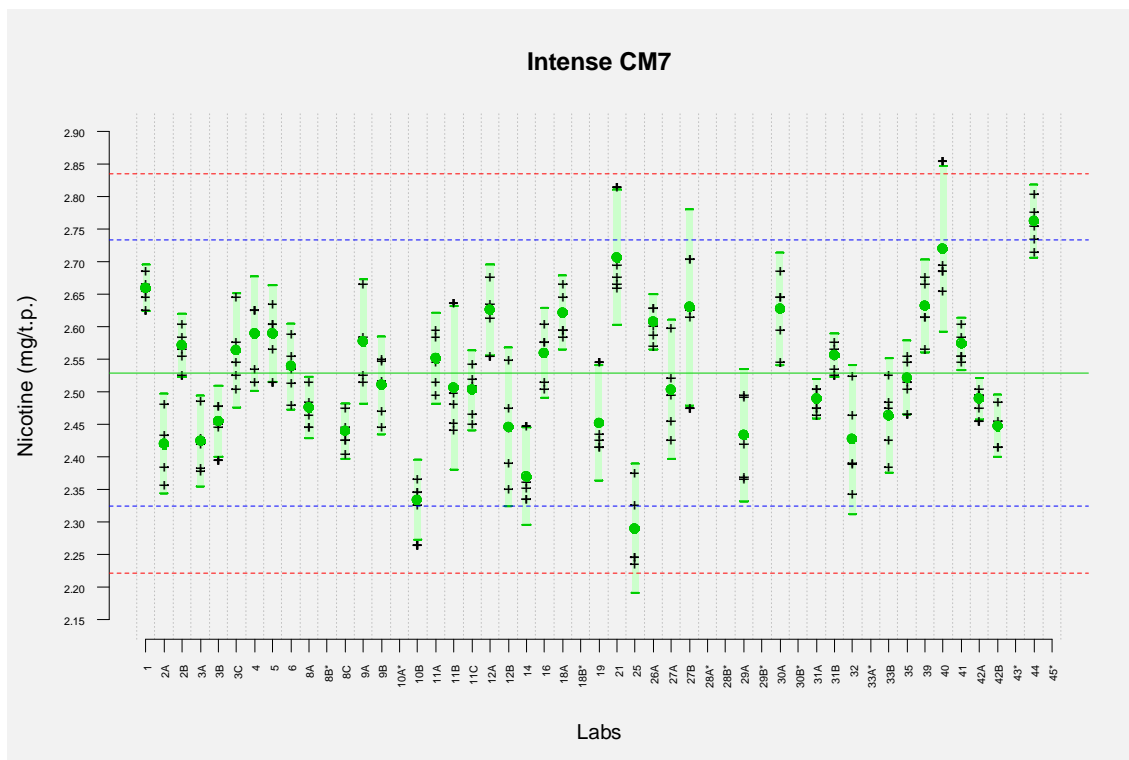


Figure 24: CM7 Individual observations of mainstream “Intense” NFDPM

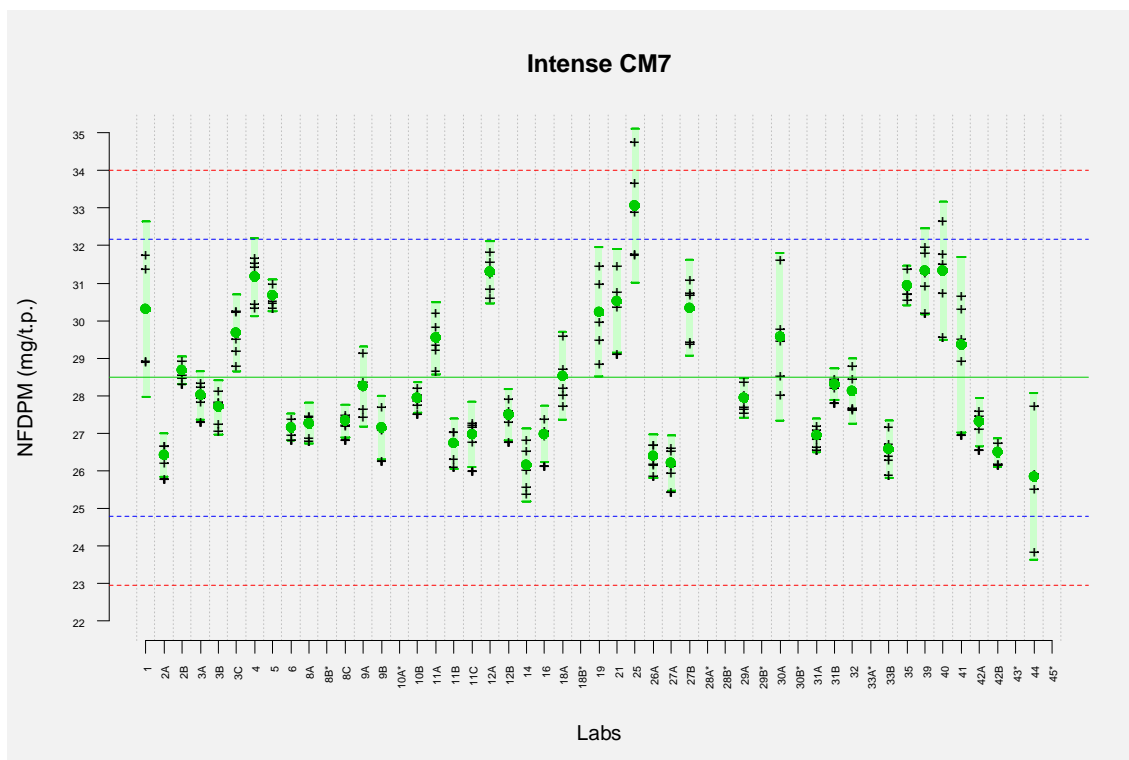


Figure 25: CM7 Individual observations of mainstream “Intense” CO

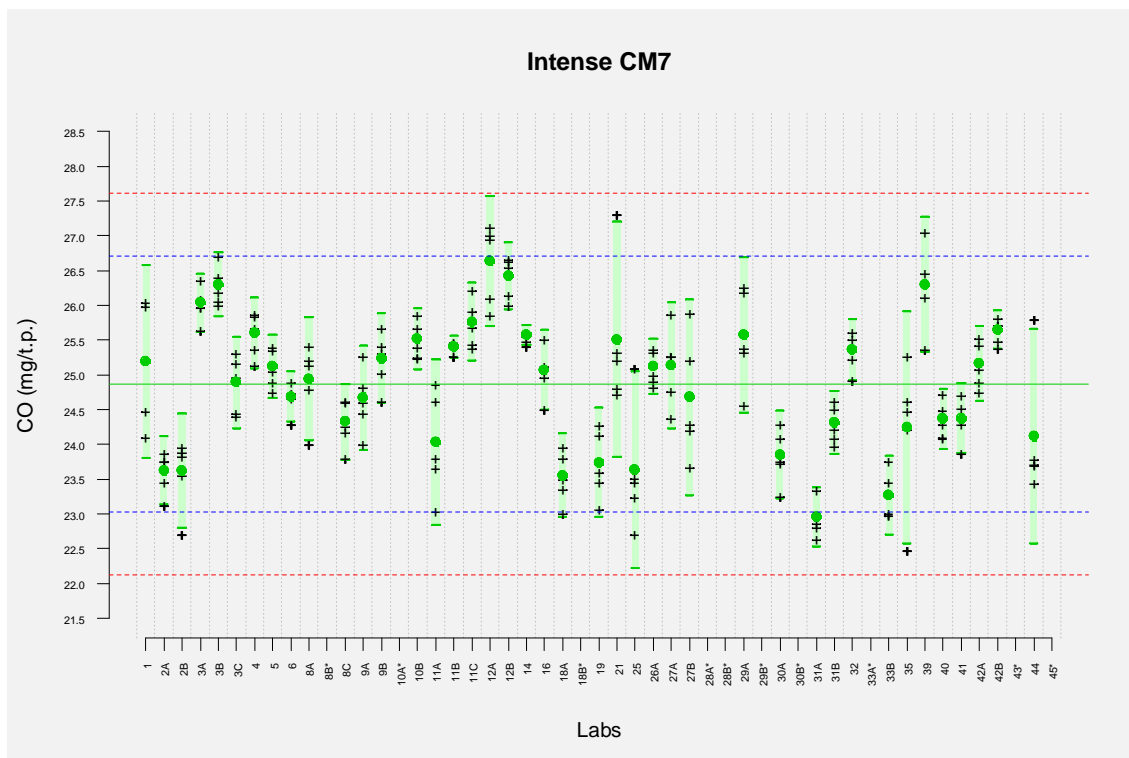
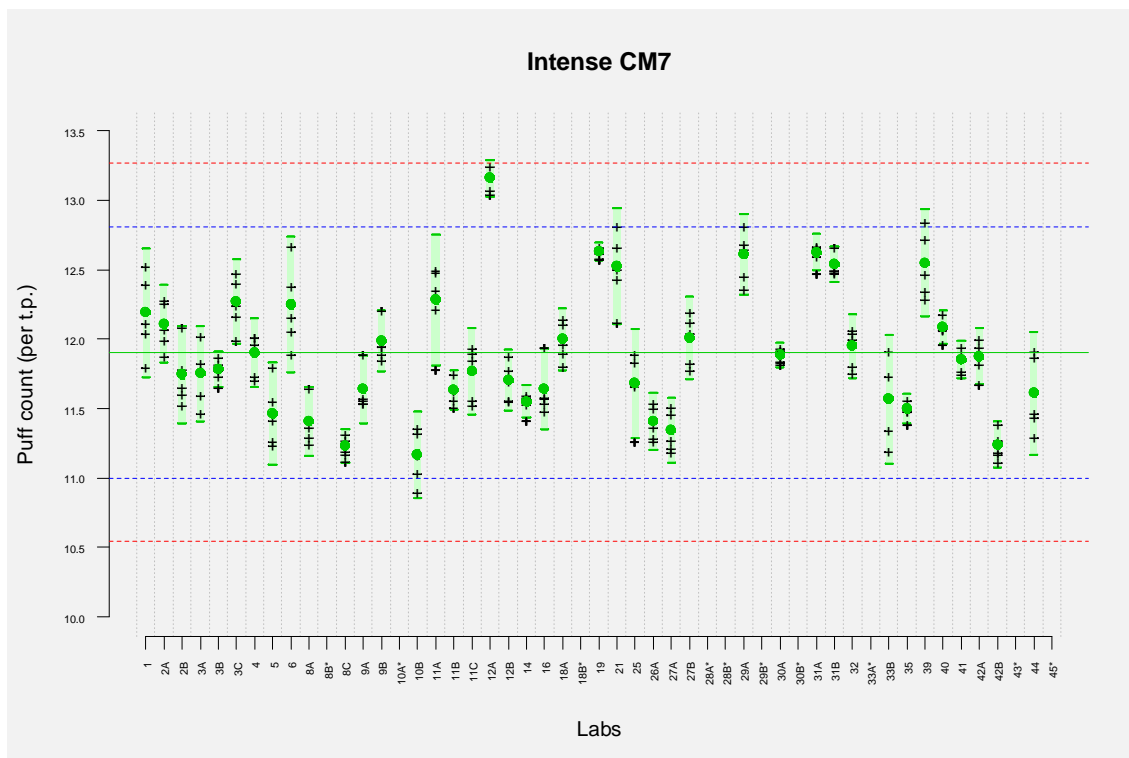


Figure 26: CM7 Individual observations of mainstream “Intense” PUFF Count



6. COMPARISON

6.1 Comparisons of Linear with Rotary smoking machines for test piece results of CM6 and CM7

It should be noted that a statistical evaluation of differences in linear and rotary smoking machine results on the basis of just one product (CM7 is similar to CM6) could be misleading. Furthermore, a simplistic differentiation between linear and rotary smoking machines could also be misleading as both machine types used in this study comprise different models produced by different manufacturers. Furthermore it has to be noted that each measurement from a rotary machine represents a single result from a single pad onto which the smoke of twenty (or ten in intense smoking regime) test pieces is trapped. Linear smoking machines require 4 pads to collect mainstream TPM from the same number of test pieces and hence each measurement on a linear machine is the average of 4 results. This has to be taken into account when interpreting statistical differences in repeatability between machine classes.

Due to the aforementioned reasons a pure statistical comparison seems to have limited value.

Graphical comparison of smoke data separated for linear and rotary smoking machines are available in appendix G.

6.2 Comparison with previous studies

This section compares the results of this study with previous collaborative studies of different test pieces conducted by the Routine Analytical Subgroup under the ISO smoking regime. It has to be noted that the number of smoking machines, smoking machine types and participants varies throughout the studies.

Table 15 lists the averages, reproducibility and repeatability limits for the test pieces CM4, CM5, CM6 and CM7 under ISO smoking regime.

Table 15: Averages, repeatability, reproducibility of CORESTA collaborative studies from 2004 to 2011 for different test pieces

	Year	2004	2005	2006	2007	2008	2010	2011	2011
	Test Piece	CM4	CM5	CM5	CM6	CM6	CM6	CM6	CM7
Average Level	Puff Number	9,10	7,72	7,64	9,18	9,15	9,09	9,00	8,36
	TPM	17,53	16,71	16,58	17,71	17,54	17,37	17,43	16,37
	Water	2,23	2,44	2,44	1,91	1,87	1,81	1,84	1,78
	Nicotine	1,295	1,078	1,083	1,384	1,390	1,366	1,362	1,236
	NFDPM	13,98	13,20	13,03	14,40	14,28	14,18	14,21	13,34
	CO	13,14	14,07	13,88	14,82	14,83	14,56	14,42	12,93
Repeatability [r]	Puff Number	0,42	0,38	0,34	0,37	0,37	0,39	0,33	0,38
	TPM	0,95	1,13	1,14	0,84	0,83	0,81	0,94	0,93
	Water	0,48	0,53	0,55	0,43	0,43	0,40	0,47	0,41
	Nicotine	0,080	0,075	0,080	0,096	0,080	0,074	0,084	0,075
	NFDPM	0,75	0,87	0,75	0,92	0,71	0,63	0,80	0,77
	CO	0,78	1,01	1,04	1,07	0,82	0,92	0,95	0,95
Reproducibility [R]	Puff Number		0,78	0,93	0,89	0,88	0,82	0,69	0,86
	TPM	1,89	2,58	2,78	1,81	1,77	1,54	1,72	1,77
	Water	1,51	1,21	1,22	1,07	1,11	1,09	0,98	0,97
	Nicotine	0,158	0,140	0,147	0,167	0,131	0,150	0,173	0,150
	NFDPM	1,90	2,17	2,29	1,92	1,80	1,53	1,92	1,88
	CO	1,98	1,82	1,62	1,84	1,64	1,96	1,83	1,94

The data in Table 15 indicate that the mean values for the listed smoke parameters are quite stable for the CM6 test piece over time. Only CO mean values suggest a trend towards lower figures which might be caused by an aging effect. The CM7 test piece shows comparable reproducibility and repeatability data with the CM6 test piece for all parameters. This is underlined by the Mandel's k graphs (Appendix E) that suggest no difference in variability between CM6 and CM7 in the 2011 collaborative study.

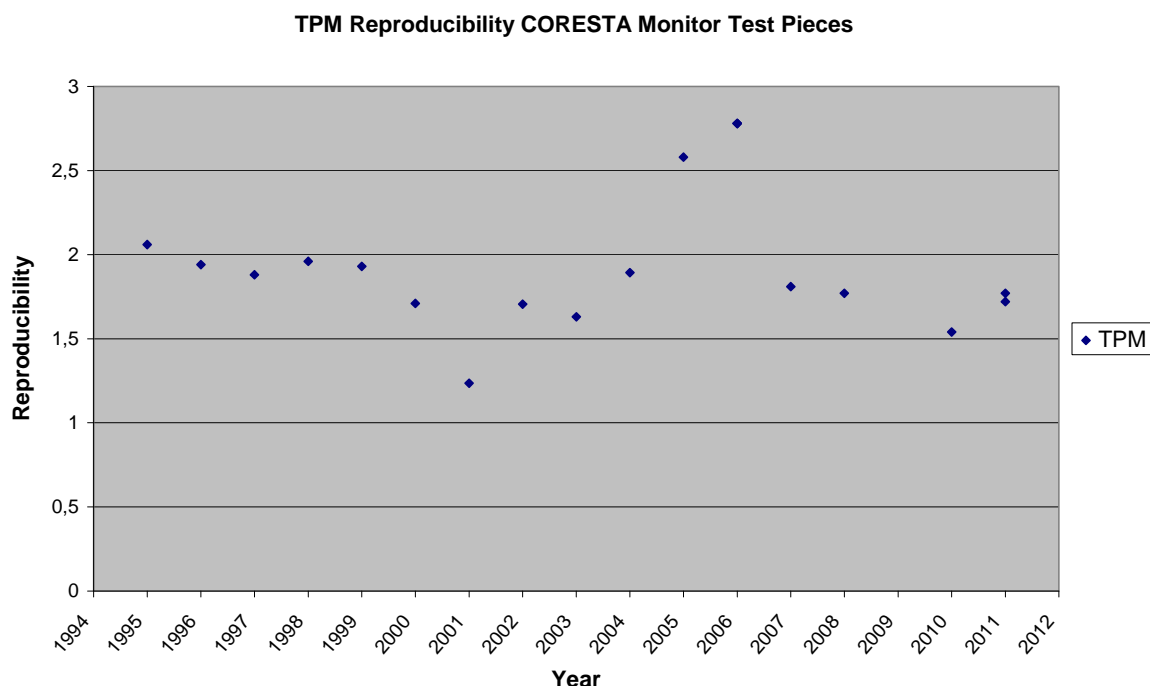
The variation in weight is somewhat higher for the CM7 (see Table 16) compared with CM6 test pieces but lower as for the CM5. As the smoke data do not indicate a significantly higher reproducibility compared with CM6 the variation might be acceptable.

Table 16: Average weight data, repeatability and reproducibility for test pieces CM5, CM6, CM7

Study	CM5 2006	CM6 2008	CM6 2010	CM6 2011	CM7 2011
Weight [mg/t.p.]	898,8	974,0	973,8	971,8	957,2
Repeatability [r]	15,8	11,2	14,0	12,1	13,0
Reproducibility [R]	21,9	17,4	17,6	18,0	20,0

A comparison of the reproducibility for the parameter TPM for all studies since 1995 (Figure 27) shows only two increased figures for reproducibility limit of TPM. These data (2005 and 2006) relate to the test piece CM5 which had noticeable higher weight variations (Table 16) compared to other test pieces. TPM reproducibility for the CM7 test piece is in line with CM6 and lower or comparable with other test pieces in previous collaborative studies.

Figure 27: TPM Reproducibility of CORESTA collaborative studies for various test pieces



7. CONCLUSION

In summary the analytical results for the CM7 test piece lead to the conclusion that it can be used as a monitor test piece in smoke analysis.

APPENDIX A - List of Participating Laboratories

Altadis Maroc – ITG*	KT&G
Arista Europe	Laboratoire National De Metrologie Et D'Essais
Arista Laboratories Inc.	Laboratorio Agroalimentario Sevilla
BAT Germany GmbH	Labstat International
BAT GR&D	Landewyck Group sarl.
Borgwaldt KC GmbH	Lorillard Tobacco Company
C.I.T. Monte Paz S.A.	Massalin Particulares
Covance*	MST/Vectis
Filtrona Technology Centre	PSS Stellenbosch
Food & Consumer Product Safety Authority, region South	PT HM Sampoerna Tbk.
Imperial Tobacco Joure**	R.J. Reynolds Tobacco Company (Lancaster Laboratories)
Imperial Tobacco Nottingham	Reemtsma/Imperial Tobacco Central Lab
Imperial Tobacco Polaska S.A.	Souza Cruz RPC-Americas
Japan Tobacco Inc.	Seita Imperial Tobacco Group
JTI Gallaher U.K.	SWM Intl
JTI Germany GmbH	TDR Rovinj, Croatia
Karelia Tobacco Company	

* CM7 ISO Regime Only

** CM6 ISO Regime Only

APPENDIX B – Experimental Protocol

Experimental Protocol Annual Study of the CORESTA Monitor Test Piece CM6 and CM7– February 28, 2011

Experimental protocol

1. INTRODUCTION

The CORESTA Routine Analytical Chemistry Sub Group has been given the responsibility to organize the annual testing of the CORESTA Monitor test piece. This year the study will include the existing test piece CM6 and also the newly produced CM7.

2. OBJECTIVES

This year's study will have expanded objectives that include smoking the monitor test piece(s) under both ISO and a specified intense smoking regime. The study objectives are:

- to measure mainstream smoke yields of nicotine-free dry particulate matter (NFDPM), nicotine and carbon monoxide (CO) when smoked under ISO and a specified intense smoking regime
- to establish the conditioned weight of CM6 and CM7 prior to smoking
- to determine intra- and inter-laboratory variability for the measured smoke yields and to continue this comparison over time
- to obtain statistical information about possible differences in smoke yields from different smoking machines
- Qualify CM7 for sale

3. NORMATIVE REFERENCES

The relevant Standards for ISO smoking:

Definitions and standard conditions	ISO 3308:2000
Conditioning	ISO 3402:1999
TPM / NFDPM	ISO 4387:2000/Amd 1:2008
Carbon monoxide	ISO 8454:2007
Nicotine	ISO 10315:2000
Water (GC)	ISO 10362-1:1999

Intense smoking (in addition to the ISO Standards above):

Ventilation zone blocking, puffing parameters and number of test pieces smoked	Health Canada Method T-115 paragraph smoked16.1 http://www.hc-sc.gc.ca/hc-ps/alt_formats/hecs-sesc/pdf/tobac-tabac/legislation/reg/indust/method/_main-principal/nicotine-eng.pdf
--------------------------------------------------------------------------------	--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------

4. METHODS

4.1. General guidance

As a general guide, experiments conducted under each puffing regime should be conducted according to ISO standard requirements. Exceptions to this guiding principle include the puffing parameters applied for intense smoking regime, the cigarette vent blocking applied, the number of test pieces smoked per sample and the butt length for smoking. **The study (the smoking results) and the daily use of the monitor are based on a fixed butt length of 32 mm.**

Please note that any operating conditions (a) not specified in the above ISO standards, or regarded as optional, (b) not specified in the intense method documents provided as guidelines and (c) any circumstances that may have influenced test results should be noted in the test report.

Puffing parameter and vent blocking specifications for each puffing regime are summarized in Table 2. Note: Although the CORESTA Monitor Test Pieces are unventilated it is advised to follow the Health Canada procedures exactly and tape the filter ventilation zone.

Table 1: Smoking Regime Specifications

Smoking Regime	Puff Volume (mL)	Puff Frequency* (s)	Puff Duration (s)	Vent Blocking (%)	Description
1	35 ± 0.3	60 ± 0.5	2 ± 0.2	0	ISO
2	55 ± 0.5	30 ± 1	2 ± 0.2	100	Intense
* Time from the start of one puff to the start of the next puff.					

4.2. Sampling

The study will use the CORESTA Monitor CM6 and CM7. Each participating laboratory is responsible for procuring their own supply of CM6 through the normal sources (Borgwaldt KC or Cerulean – Appendix 1), however as CM7 is not yet available for sale it will be provided to each participating laboratory. The smoking plans will use 200 test pieces for each rotary smoking machine or 220 test pieces for each linear smoking machine included in the study. A composite of all packs should be prepared for conditioning according to ISO 8243:2006. If a substantial period of time is expected to occur between the times when the test pieces are smoked with the different smoking regimes, then two separate composite samples should be prepared, one for each smoking regime.

4.3. Ventilation Zone Blocking

The blocking of the filter ventilation zone will be achieved by wrapping a single layer of invisible tape, 19.0 mm wide or equivalent, around the complete circumference of the filter and tipping paper of the test articles.

4.4. Butt Marking

For the purpose of this study the CM test pieces (CM6 and CM7) will be butt marked to a fixed butt length of 32 mm.

4.5. Conditioning

The composite samples should be conditioned for at least 48 hours but no more than 10 days under the following conditions:

Temperature: $22 \pm 1^{\circ}\text{C}$ Relative humidity: $60 \pm 3\%$

Temperature and relative humidity of the conditioning enclosure should be checked and recorded for every smoking run.

Test cigarettes to be smoked for the determination of NFDPM, nicotine and CO should be selected at random from the conditioned test pieces.

Any cigarettes with visible defects should be discarded.

4.6. Weight of the Test

The test articles will be weighed for ISO smoking only since the ventilation zone blocking tape applied for intense smoking may vary between laboratories. The weight of the test piece after conditioning is to be determined by weighing all test pieces to be smoked just before they are placed at the smoking machine. The result is reported as the mean weight of the 20 test pieces for linear or rotary smoking machine at ISO puffing parameters.

4.7. Smoking Machine

Where smoking laboratories have more than one type of smoking machine (i.e. linear and rotary variants) they are encouraged to include as many types in the study as possible.

4.8. Smoking Plans

The smoking plans will incorporate two products (CM6 and CM7), shown as M1 and M2 in the smoking plans. These plans are designed to smoke the products in all ports for each puffing regime, spread over a number of days. If possible, each participating laboratory should use only one operator (for each smoking machine type) throughout the course of the study.

For ISO smoking, one test result is defined as the mean yield obtained from smoking 20 test pieces in a single smoking machine run, therefore from a rotary machine it is the result from one run smoking 20 test pieces and from a linear machine it is the average of four ports, smoking five test pieces per port. This is repeated on five separate days resulting in 100 test pieces being smoked (Tables 2-4)

For Intense smoking, the number of test pieces smoked per filter pad is reduced to avoid overloading the pad. As a consequence, the number of smoking

runs/ports has to be increased so that the same nominal number of test pieces are smoked. Thus, a test result from a rotary machine is the mean of two runs, each of 10 test pieces, and from a linear machine it is the average of eight ports, smoking three test pieces per port (Tables 5-7). This is repeated on five separate days resulting in 100 test pieces being smoked on a rotary machine and 120 on a linear machine.

The smoking plans will use 220 of each test piece for each linear machine variant and 200 for each rotary machine variant included in the study.

Note that the side seam of the test pieces should be positioned at random and not in a fixed position since it has been observed that some laboratory staff habitually place test articles in a particular orientation. Ports not occupied by the test pieces are left blank, which may be assigned to any other test article that is to be smoked by the laboratory, or the ports may be left empty.

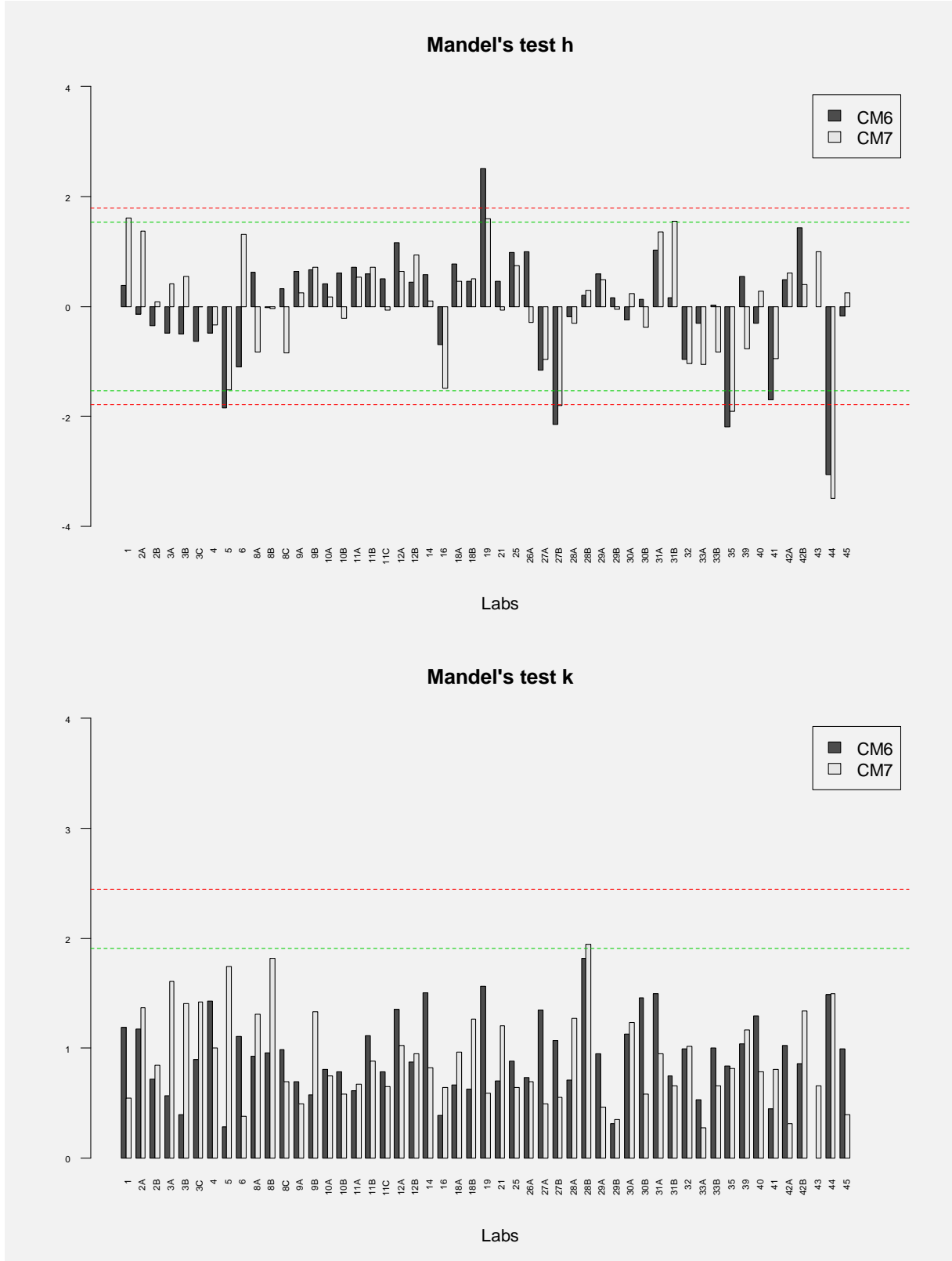
APPENDIX C – Departures from Experimental Protocol

No departures from the experimental protocol were reported.

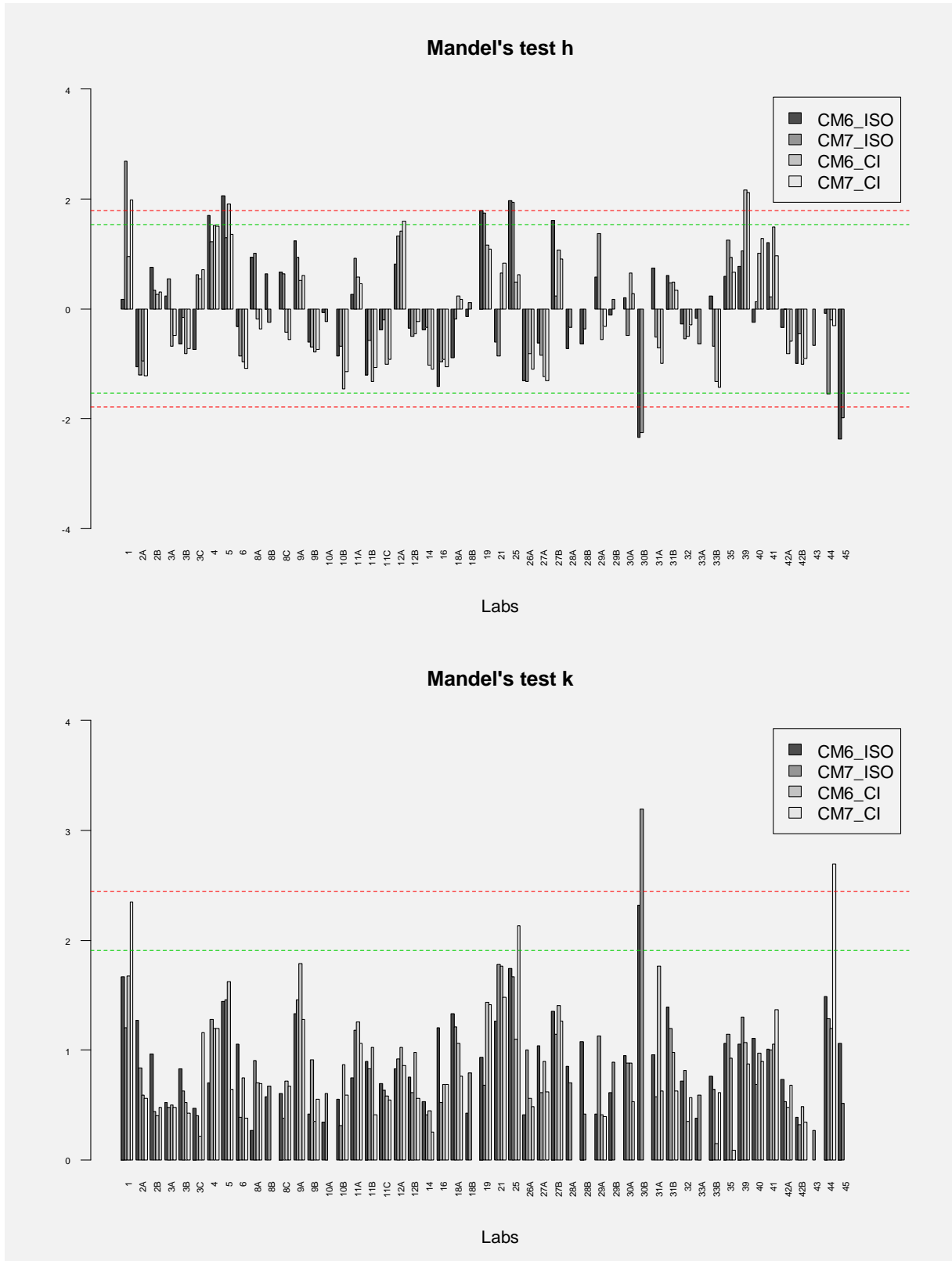
APPENDIX D – RAW DATA SET

APPENDIX E – MANDEL'S GRAPH

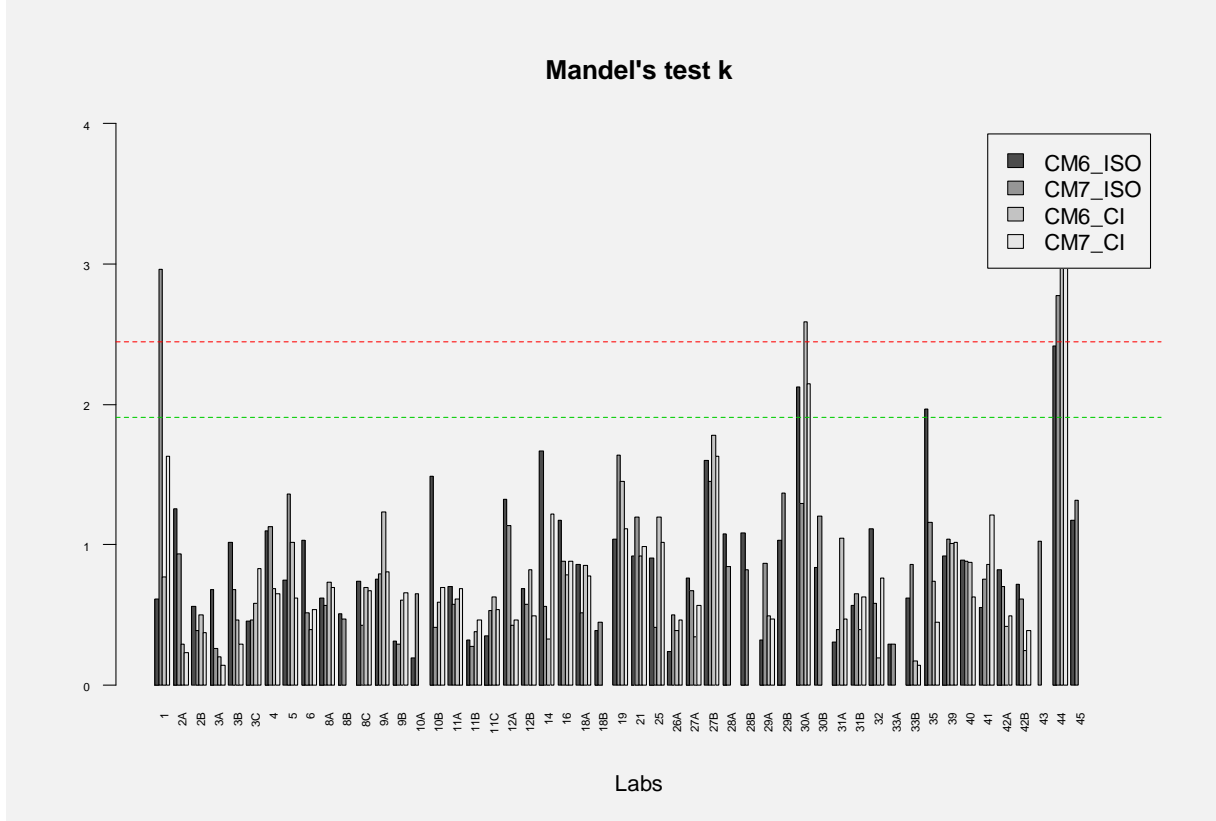
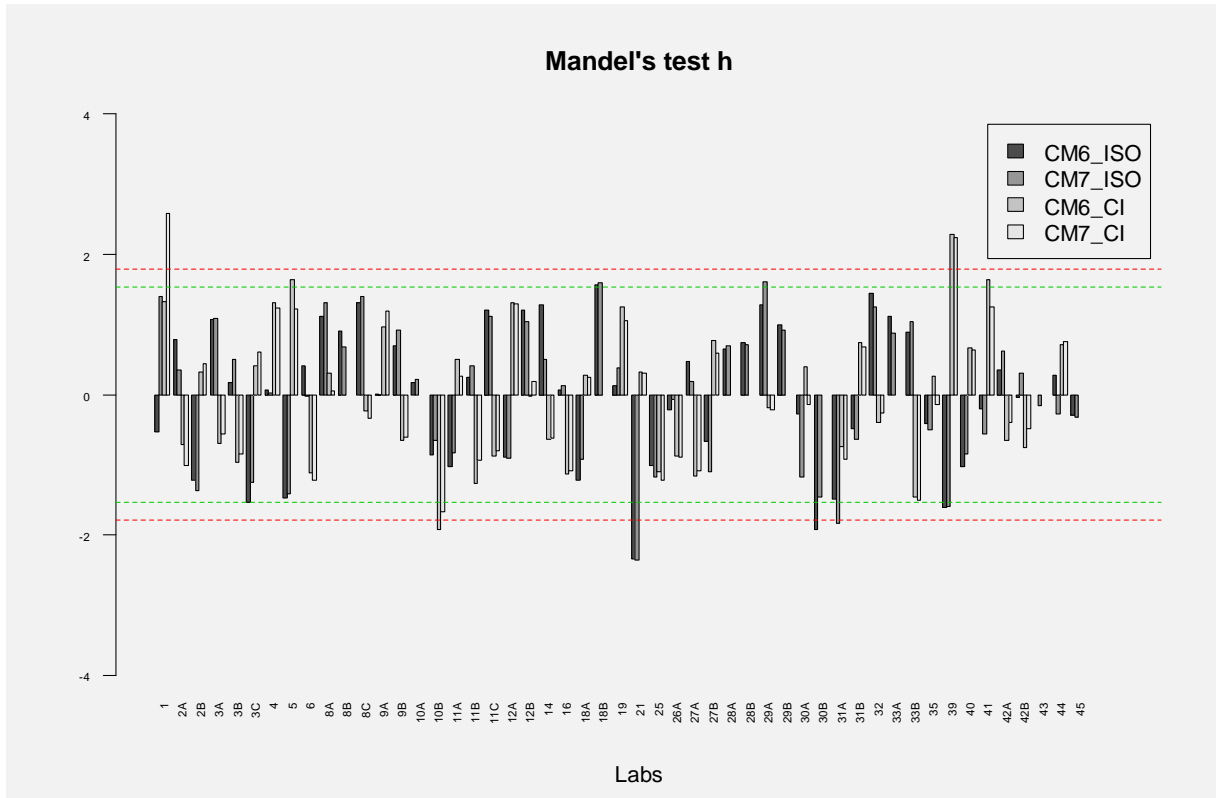
1. WEIGHT



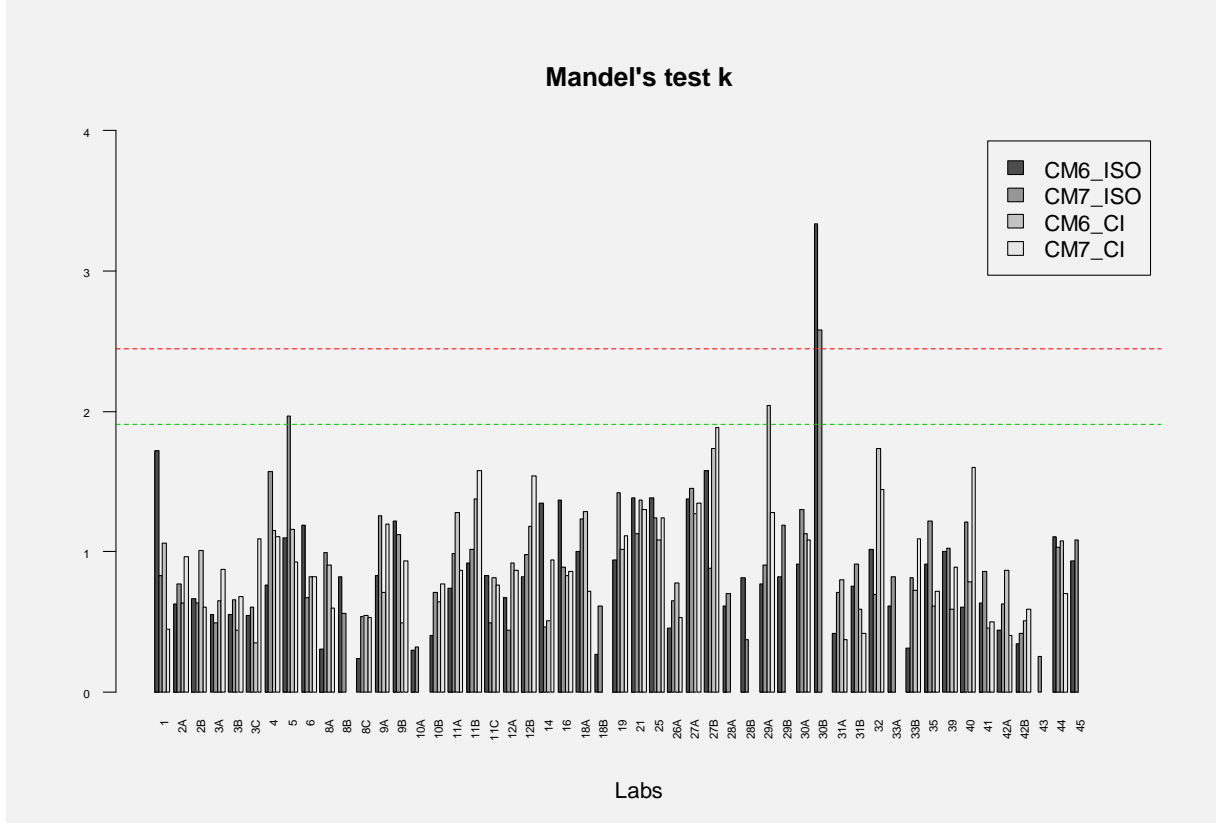
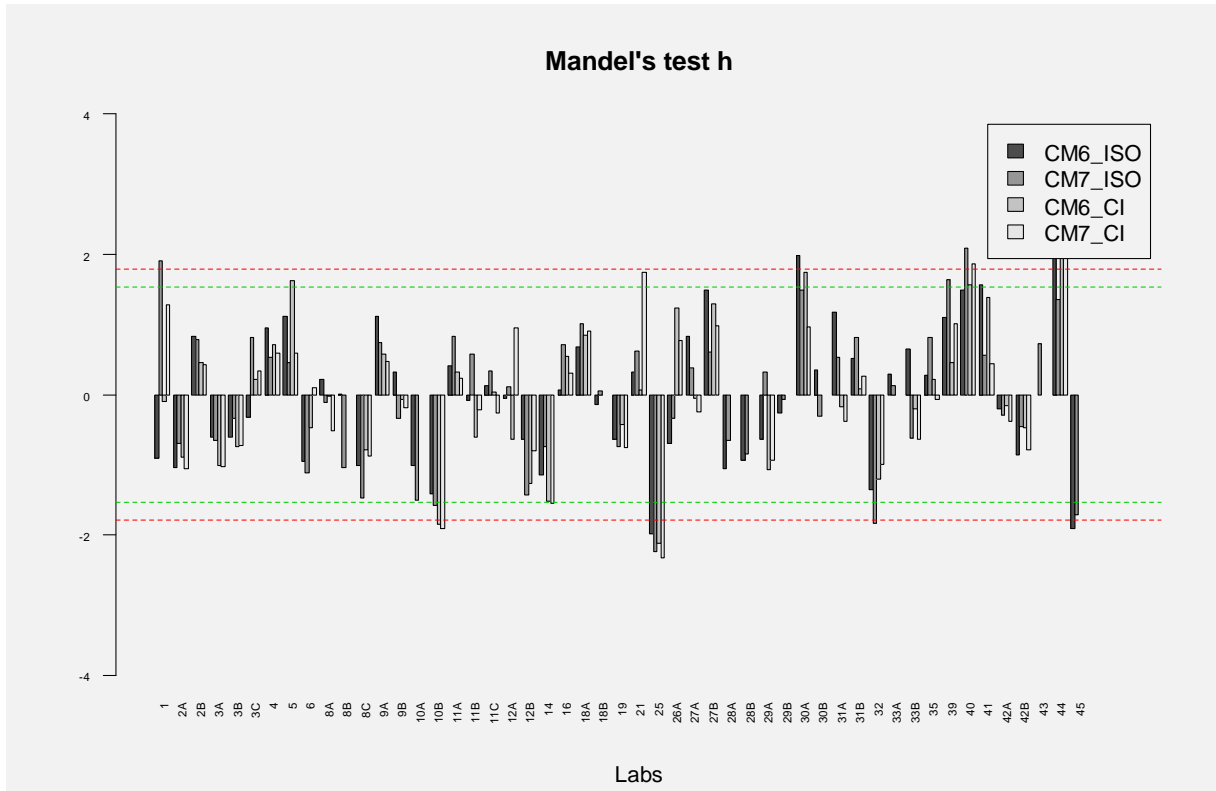
2. TPM



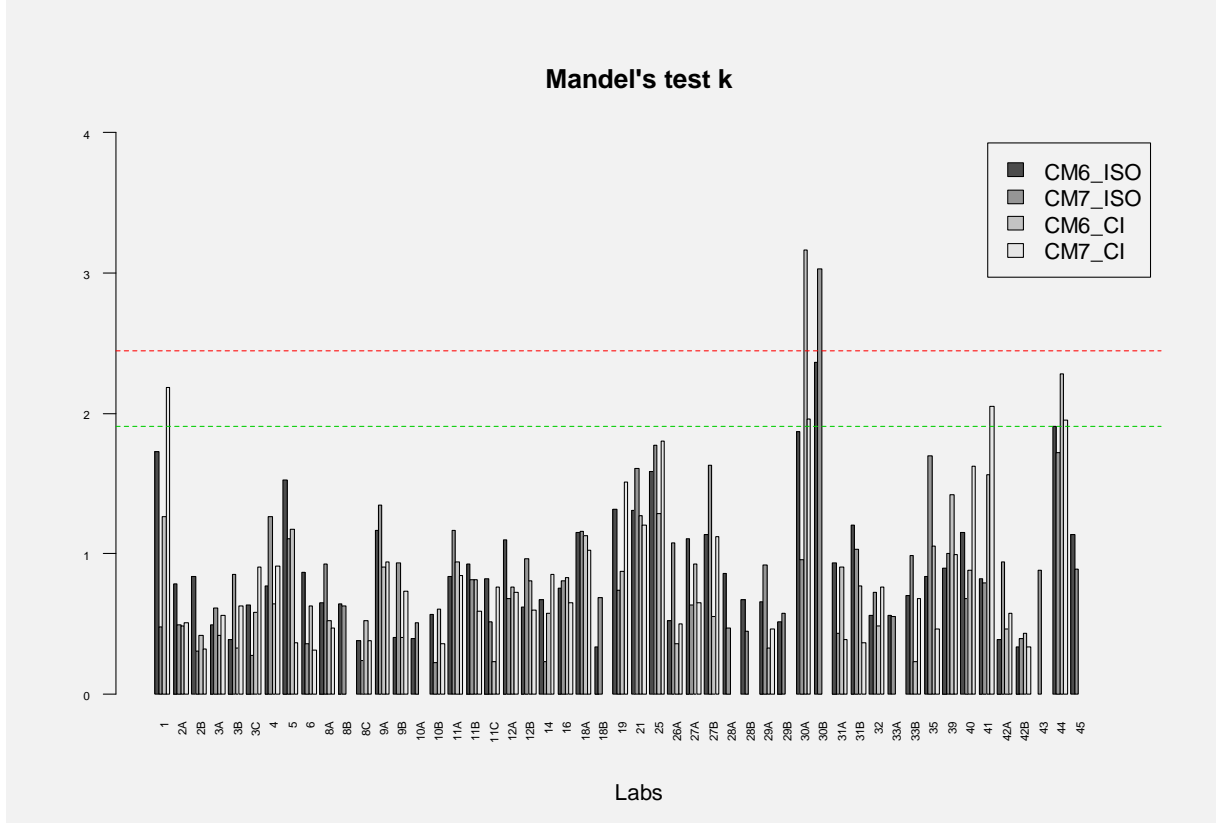
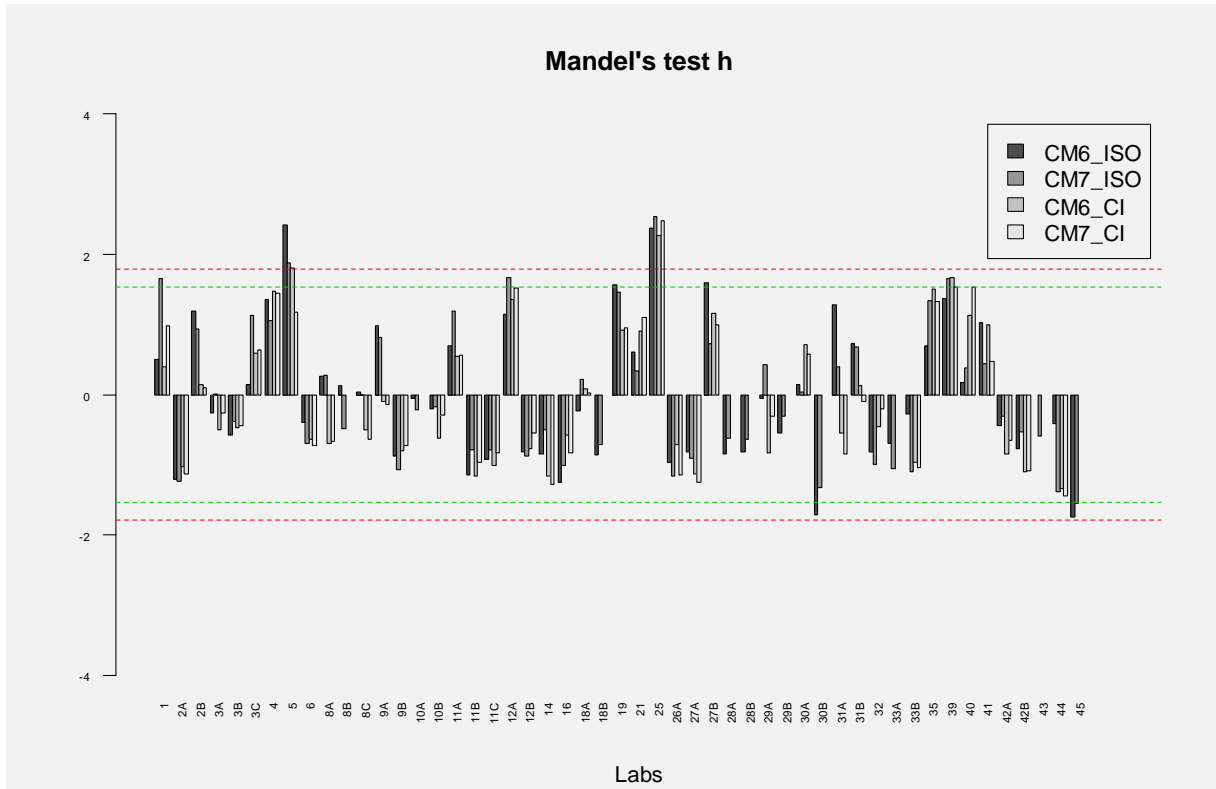
3. WATER



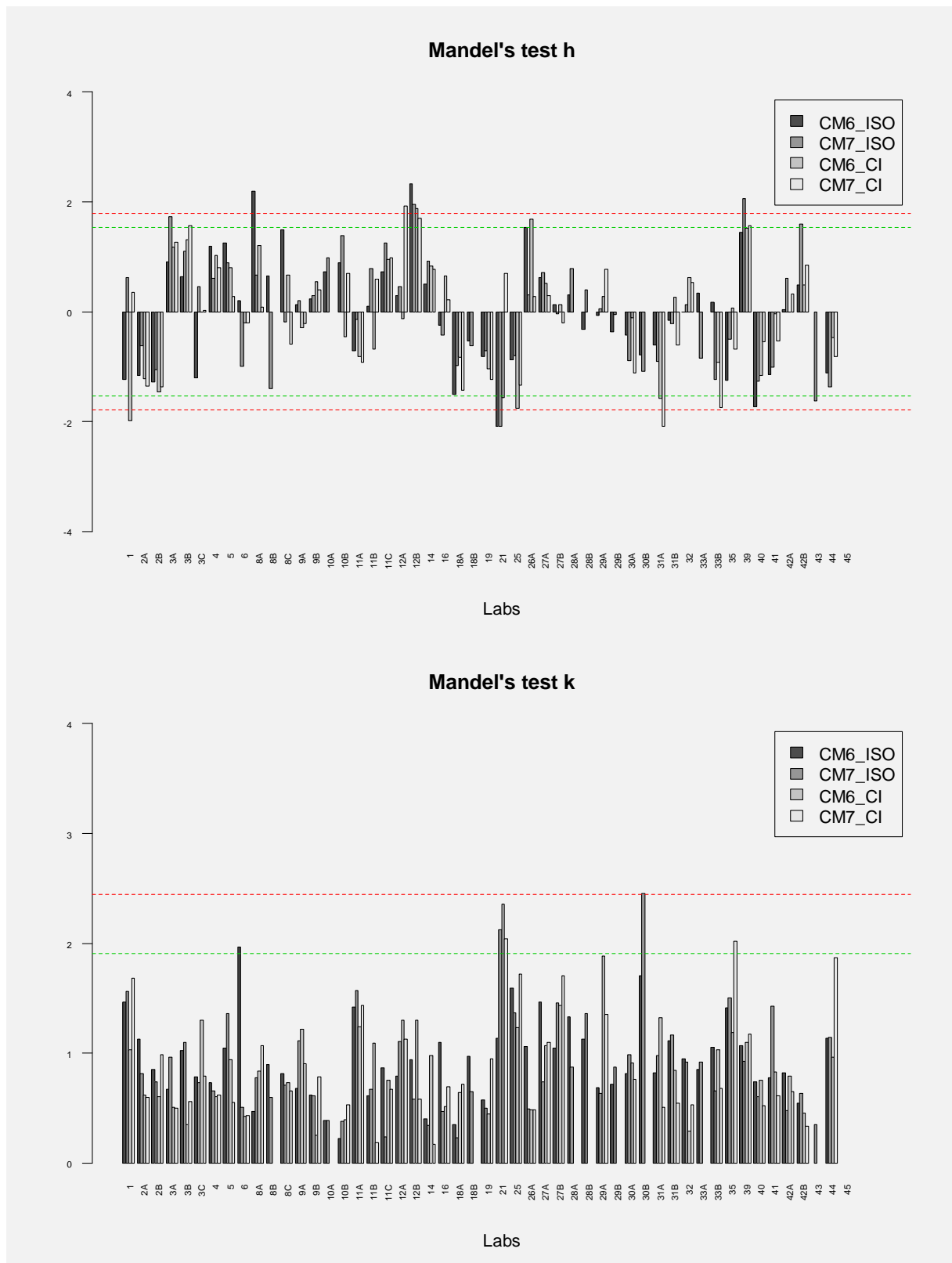
4. NICOTINE



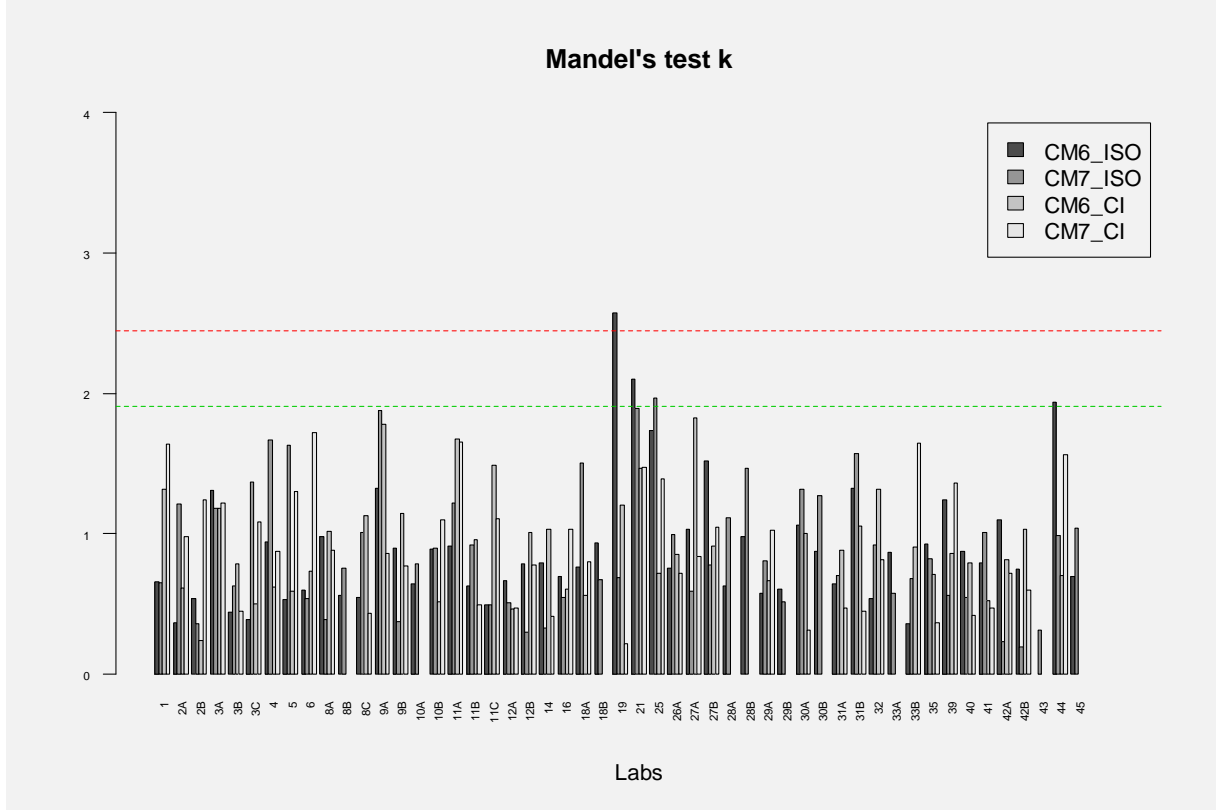
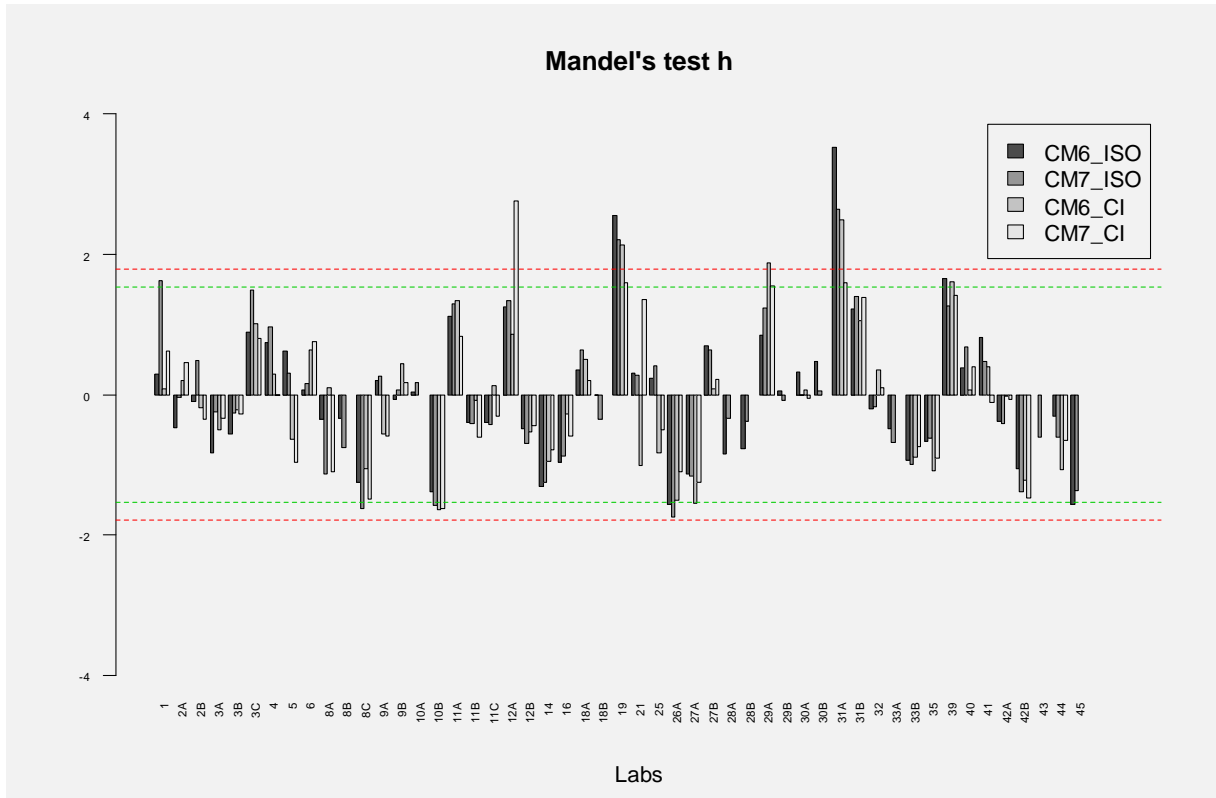
5. NFDPM



6. CO

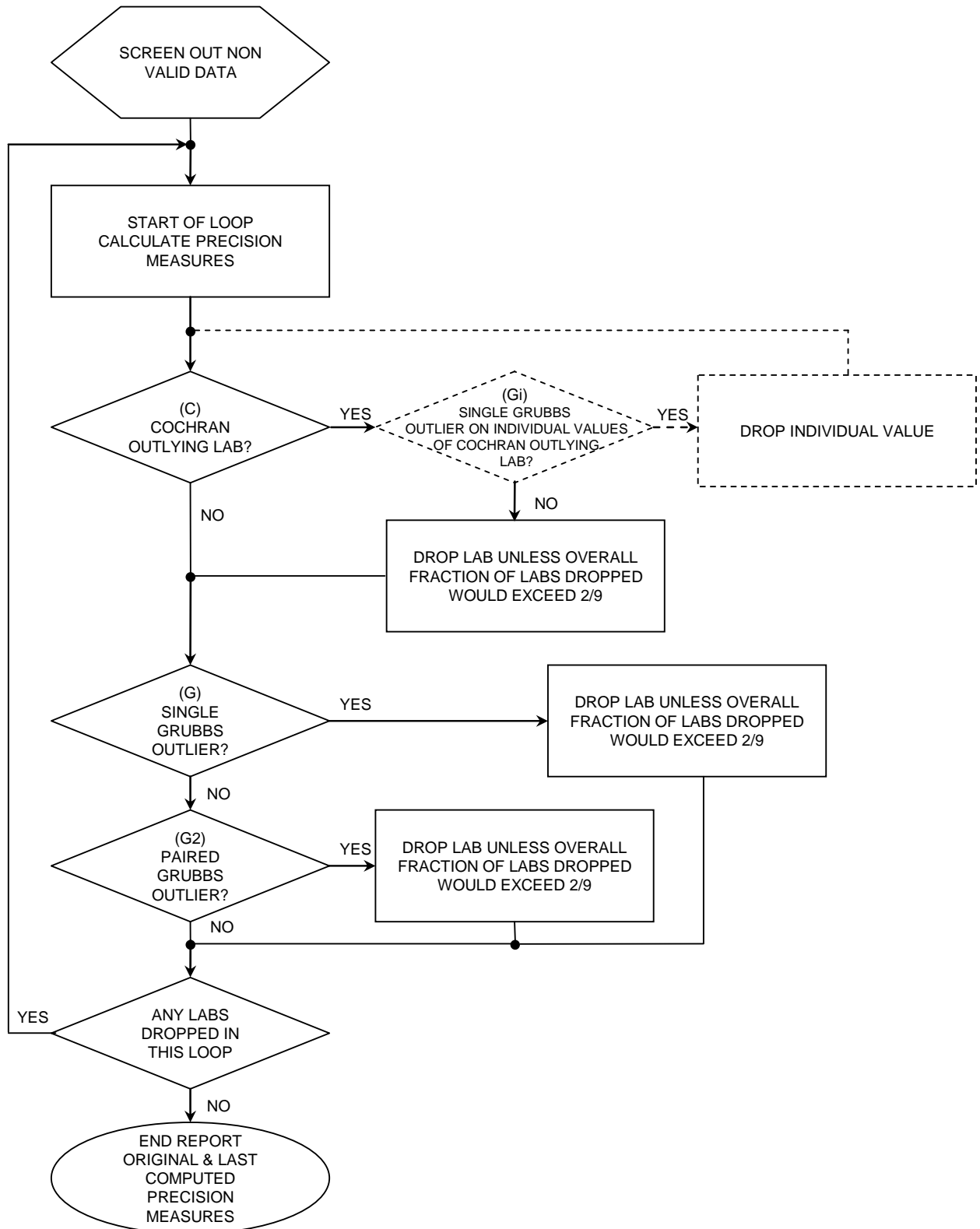


7. PUFF



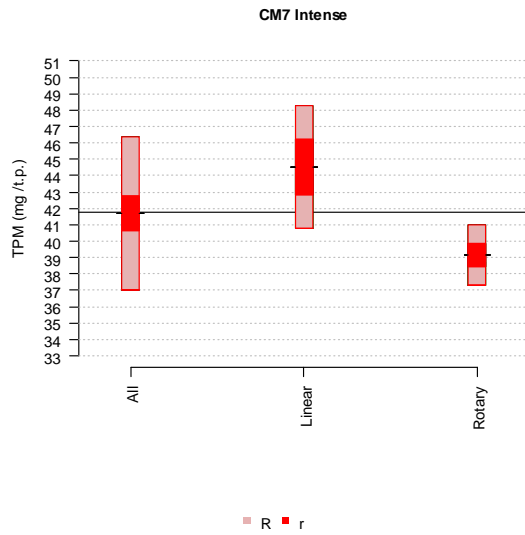
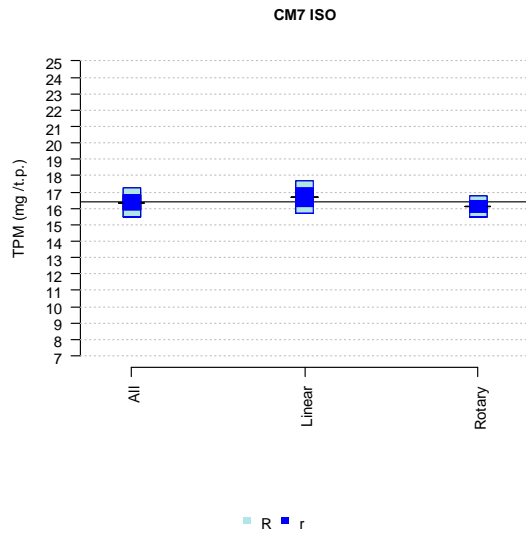
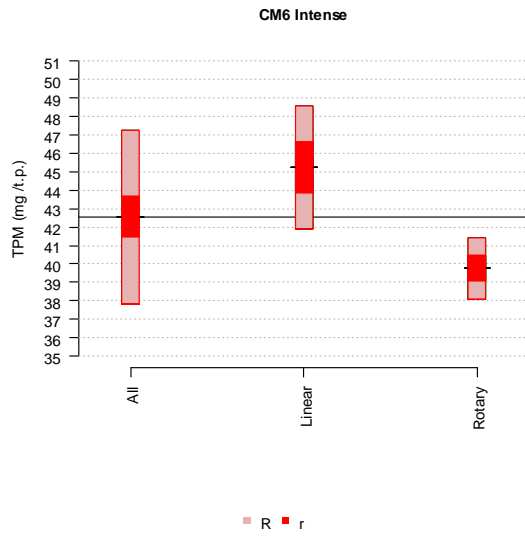
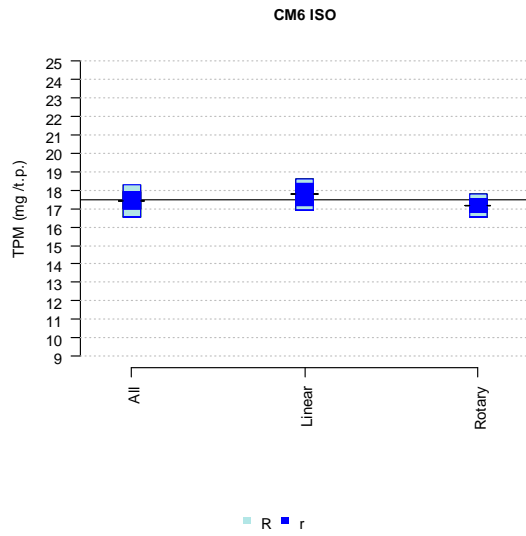
APPENDIX F – IUPAC 1994 HARMONIZED STATISTICAL PROCEDURE

IUPAC 1994 HARMONIZED STATISTICAL PROCEDURE

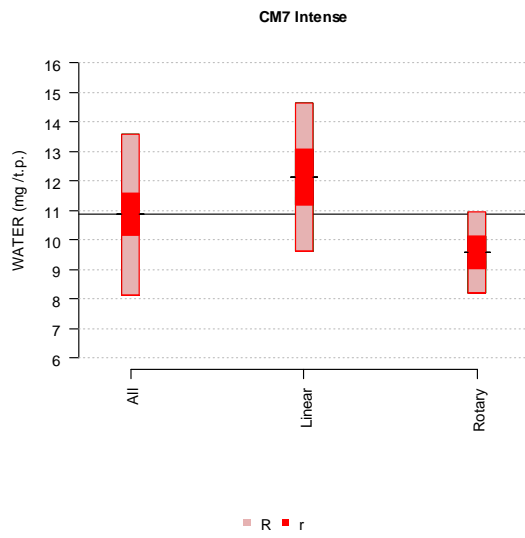
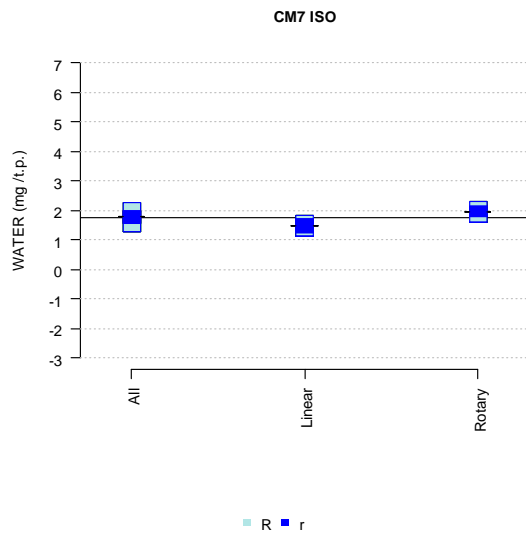
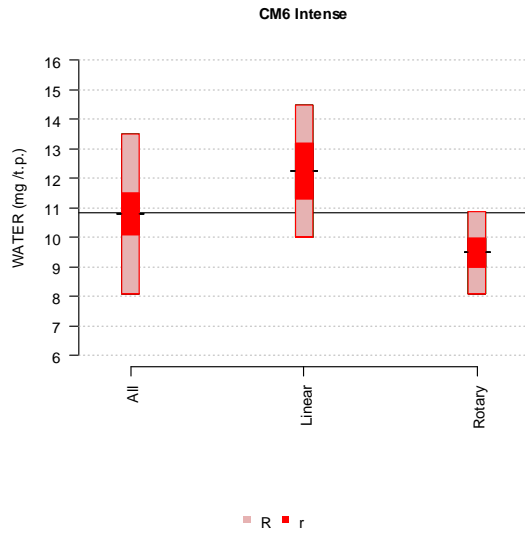
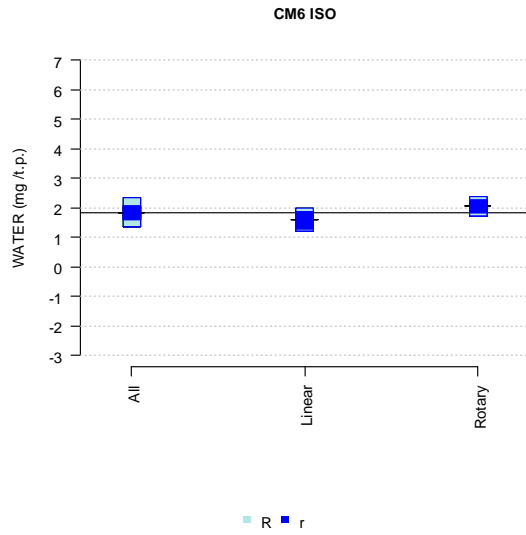


APPENDIX G - LINEAR – ROTARY COMPARISONS

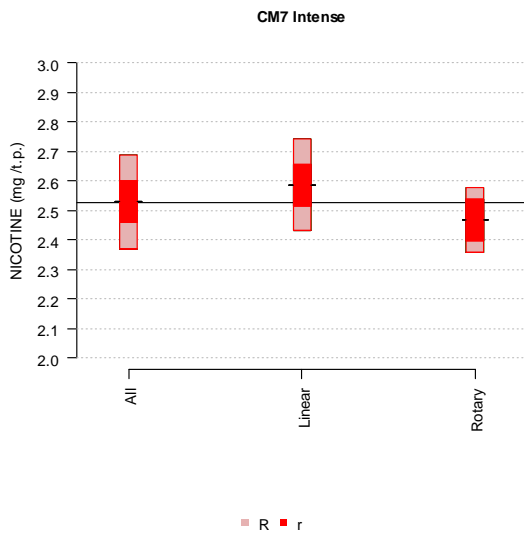
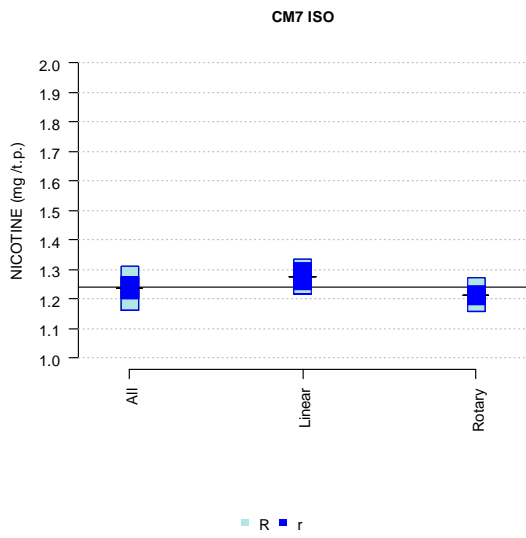
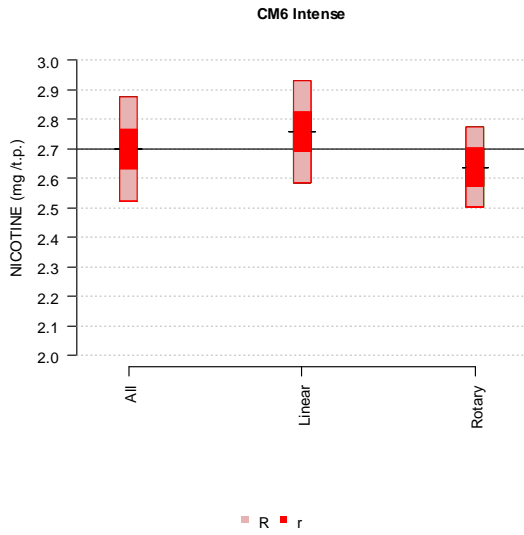
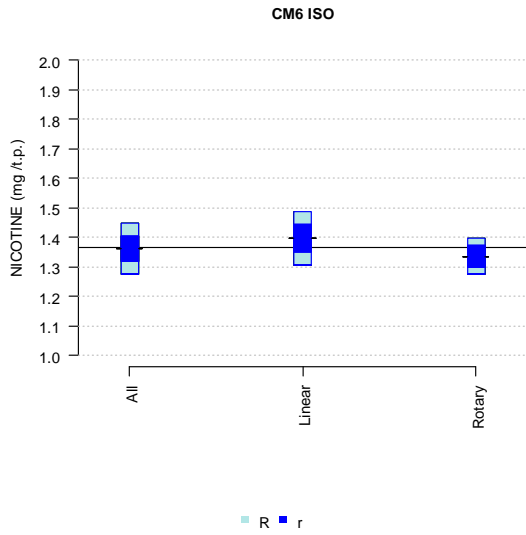
1. TPM



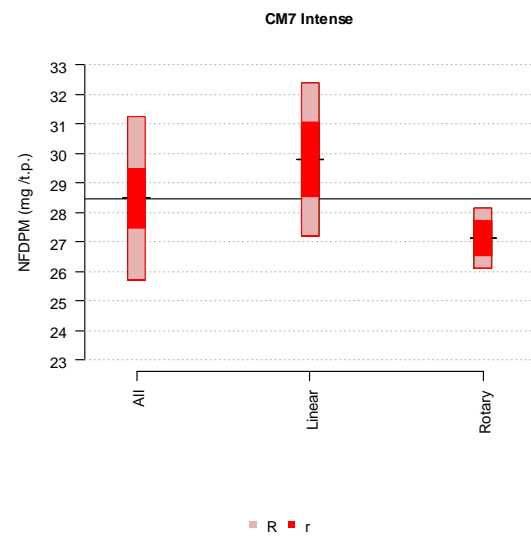
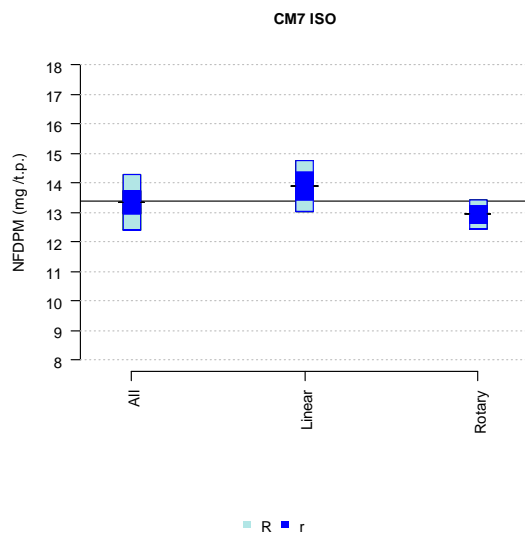
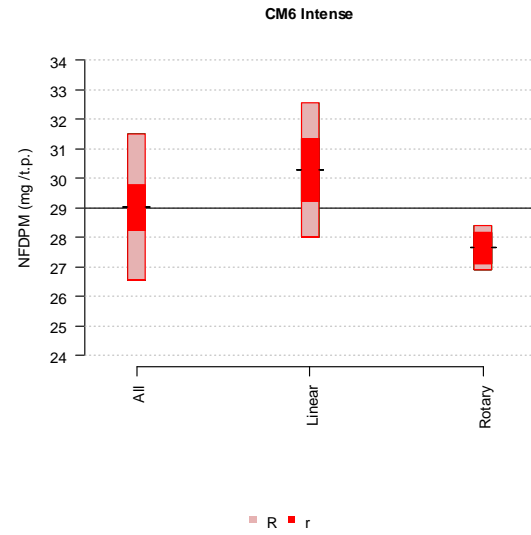
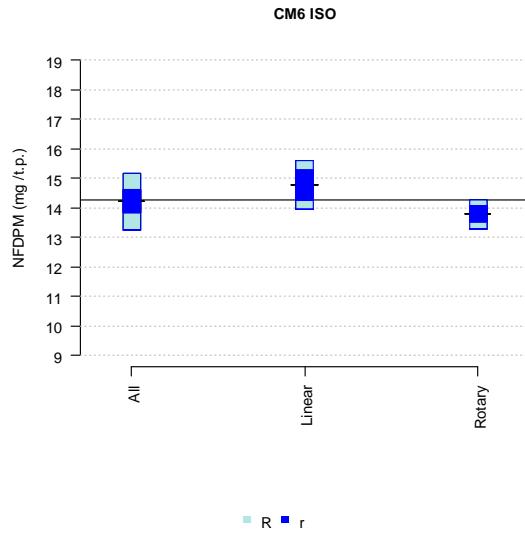
2. WATER



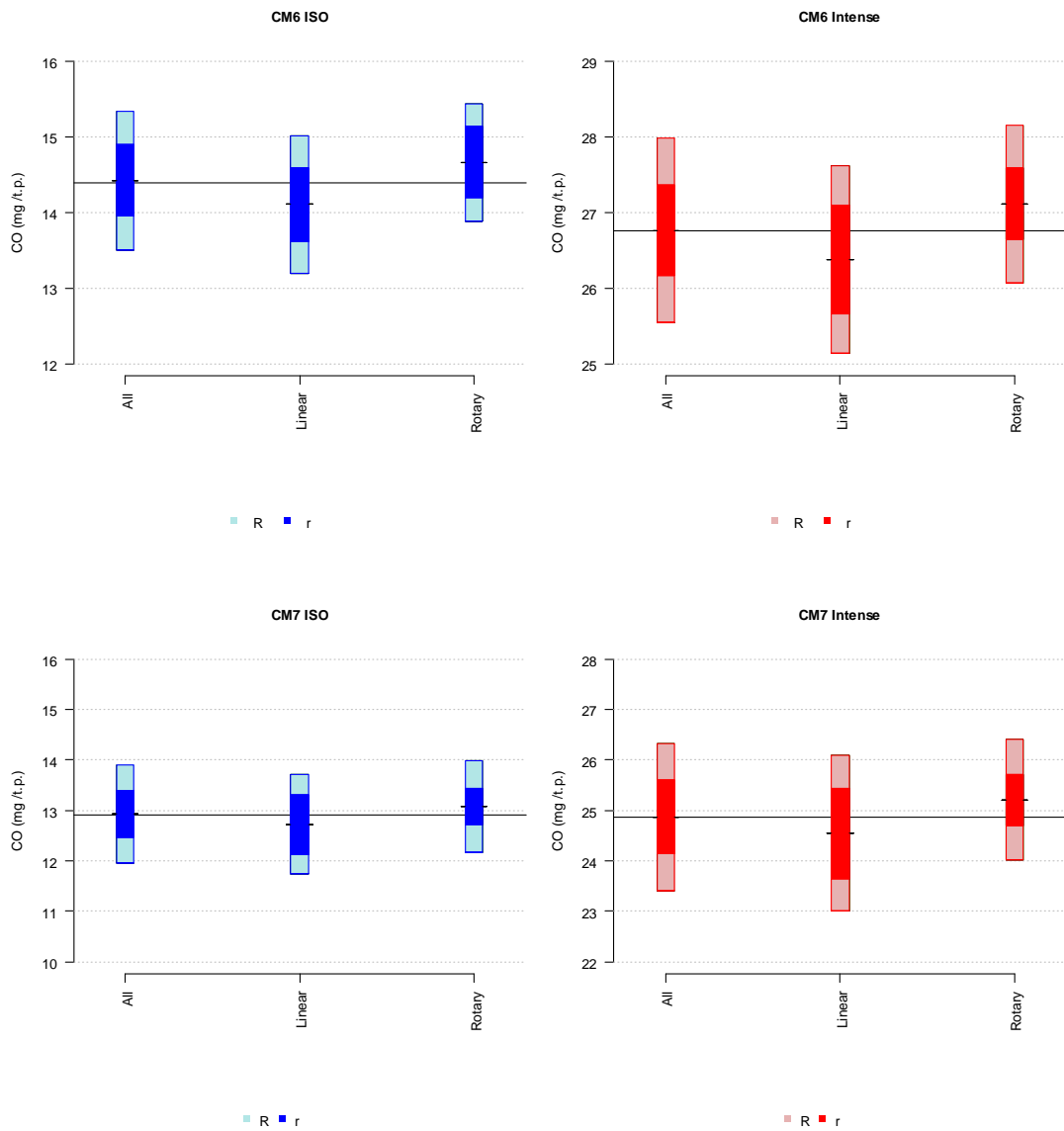
3. NICOTINE



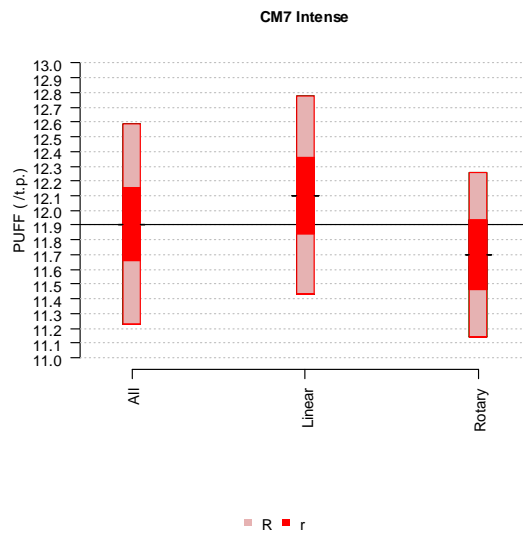
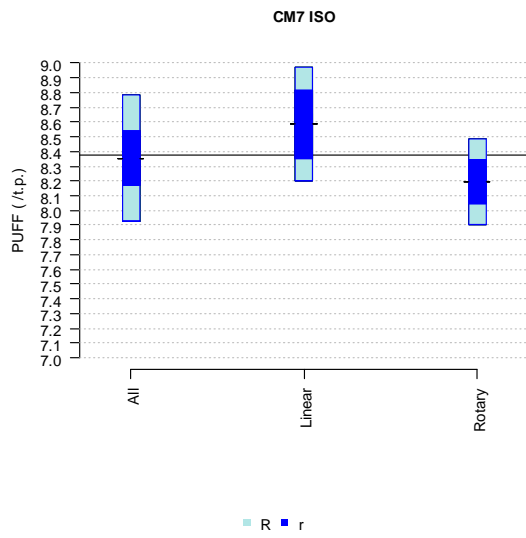
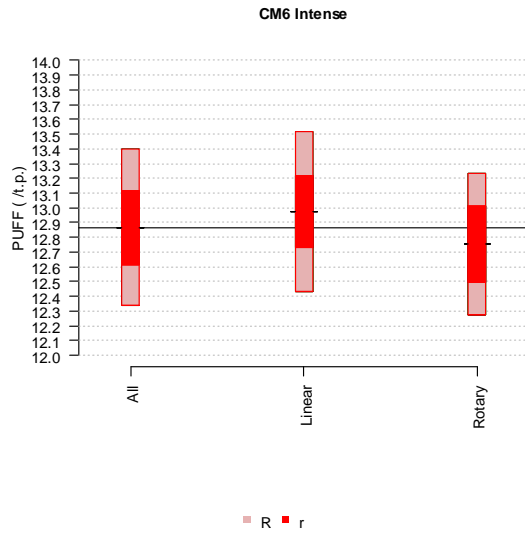
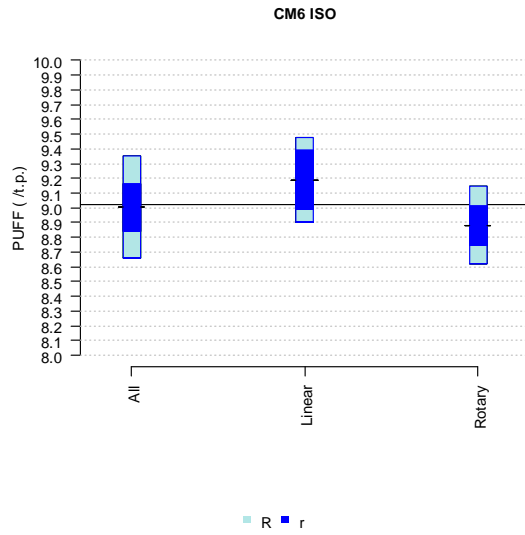
4. NFDPM



5. CO



6. PUFF



APPENDIX H - ROTARY SMOKING MACHINES

Smoking regime	Coresta Monitor	Parameter	No of Labs	Mean	Repeatability		Reproducibility		Outlying Labs			Stragglings Labs	No data
					sr	r	sR	R	C	G/G2	Gi		
ISO	CM6	TPM	28	17.2	0.26	0.73	0.46	1.30	30B				43
		WATER	28	2.1	0.15	0.43	0.23	0.66		30B			43
		Nicotine	28	1.33	0.027	0.076	0.044	0.124	30B				43
		NFDPM	28	13.8	0.20	0.56	0.35	0.98	30B				43
		CO	28	14.7	0.33	0.94	0.55	1.54				8A, 12B	43, 45
		Puff Count	29	8.9	0.09	0.26	0.19	0.53					43
	CM7	TPM	29	16.1	0.24	0.69	0.45	1.28	30B				
		WATER	30	2.0	0.13	0.36	0.24	0.69				10B, 30B	
		Nicotine	29	1.21	0.022	0.062	0.040	0.114	30B				
		NFDPM	29	12.9	0.21	0.59	0.35	0.98	30B				
		CO	28	13.1	0.25	0.71	0.64	1.81	30B				45
		Puff Count	30	8.2	0.11	0.30	0.21	0.58					
Intense	CM6	TPM	20	39.8	0.50	1.42	1.17	3.30					
		WATER	20	9.5	0.35	0.99	0.99	2.80					
		Nicotine	20	2.64	0.046	0.131	0.095	0.269				29A	
		NFDPM	20	27.6	0.38	1.06	0.53	1.49					
		CO	19	27.1	0.33	0.94	0.74	2.09	29A				
		Puff Count	20	12.8	0.18	0.52	0.34	0.95					
	CM7	TPM	20	39.2	0.50	1.42	1.30	3.68					
		WATER	20	9.6	0.39	1.10	0.96	2.71					
		Nicotine	20	2.47	0.049	0.139	0.077	0.218				14	
		NFDPM	20	27.1	0.42	1.18	0.73	2.06					
		CO	20	25.2	0.36	1.00	0.85	2.39				2A, 29A, 33B	
		Puff Count	20	11.7	0.17	0.48	0.40	1.12					

1. ISO smoking regime

1.1. CM6

Figure 1: CM6 Individual observations of mainstream “ISO” conditioned WEIGHT

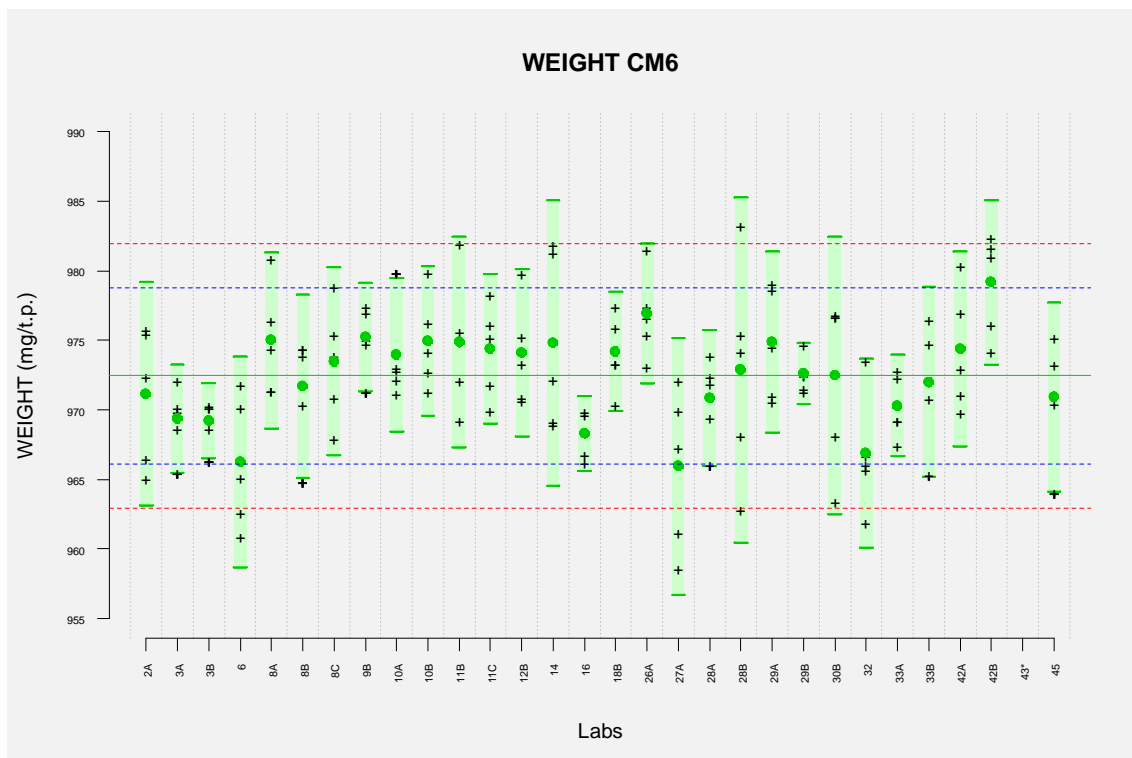


Figure 2: CM6 Individual observations of mainstream “ISO” TPM

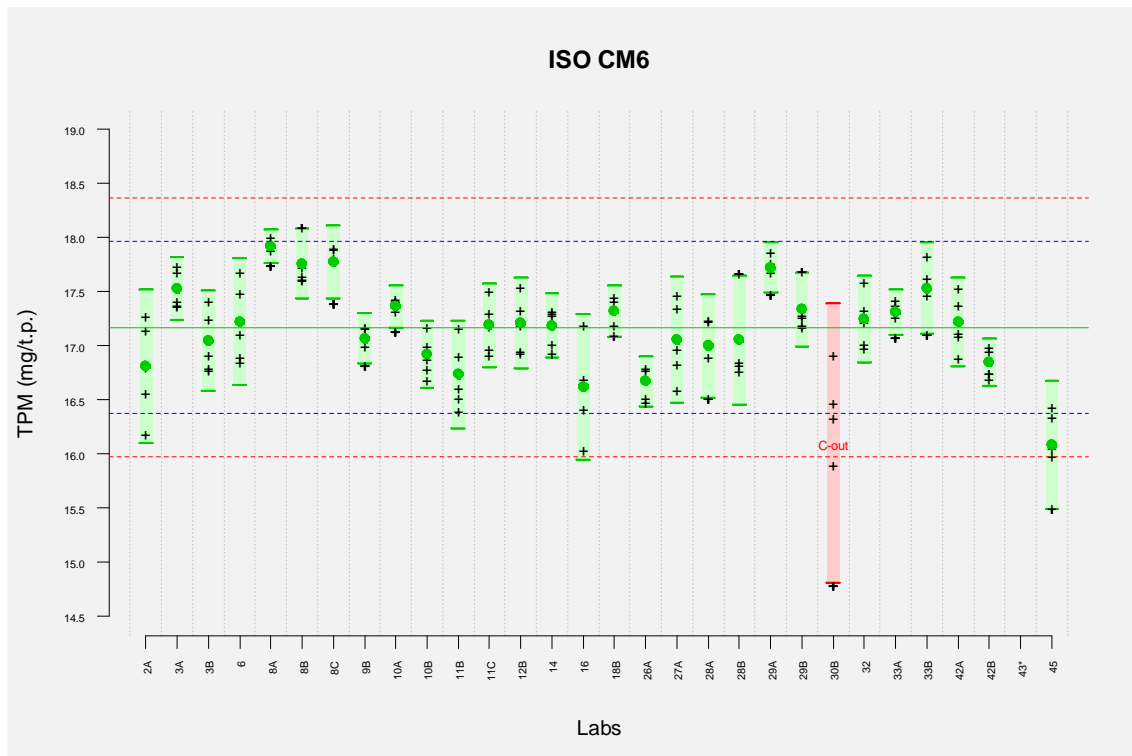


Figure 3: CM6 Individual observations of mainstream “ISO” WATER

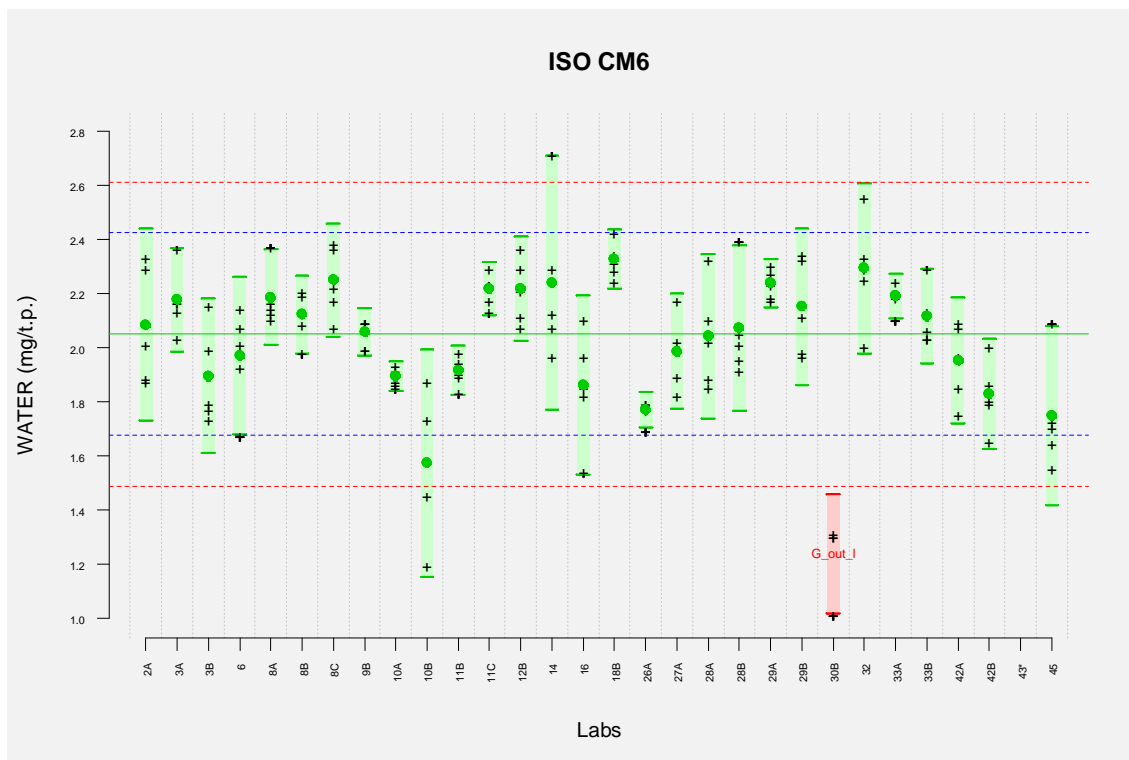


Figure 4: CM6 Individual observations of mainstream “ISO” Nicotine

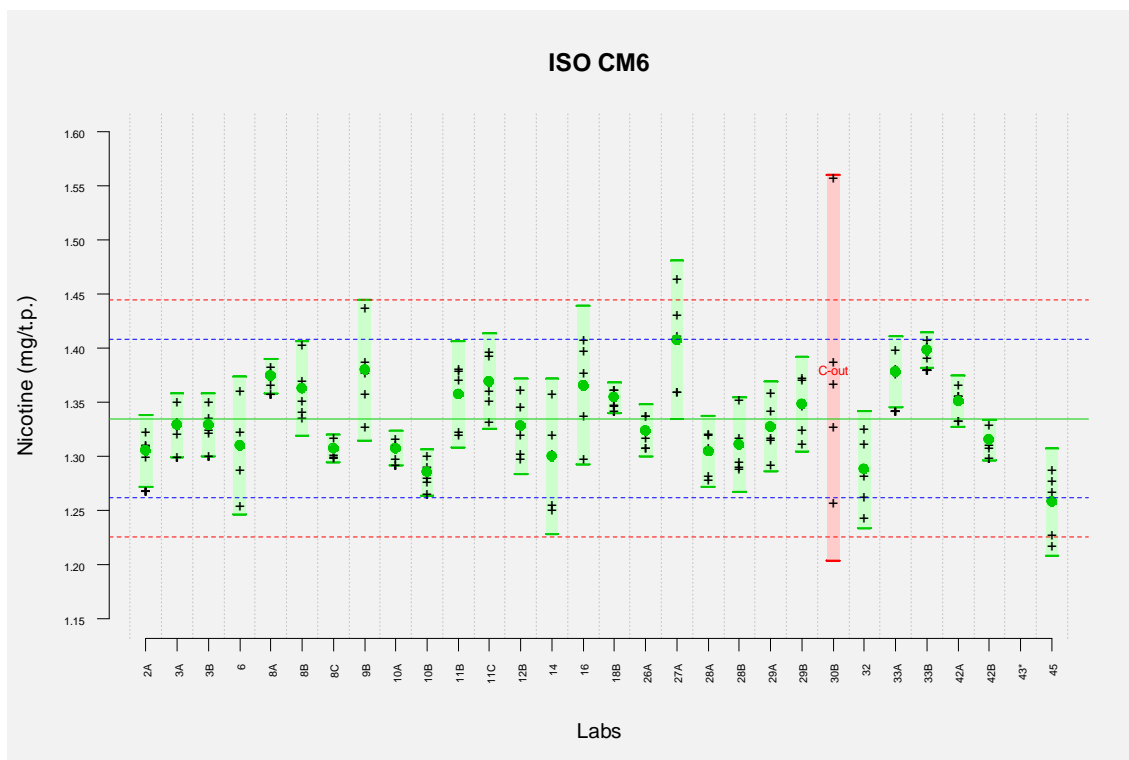


Figure 5: CM6 Individual observations of mainstream “ISO” NFDPM

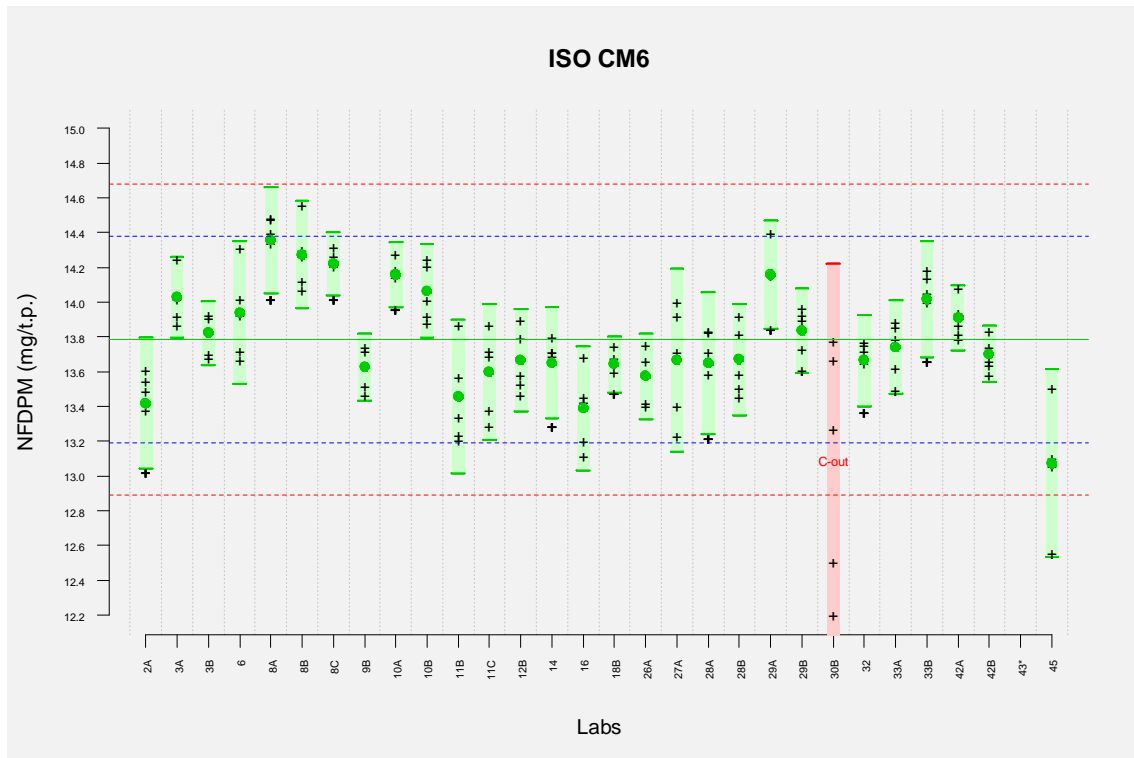


Figure 6: CM6 Individual observations of mainstream “ISO” CO

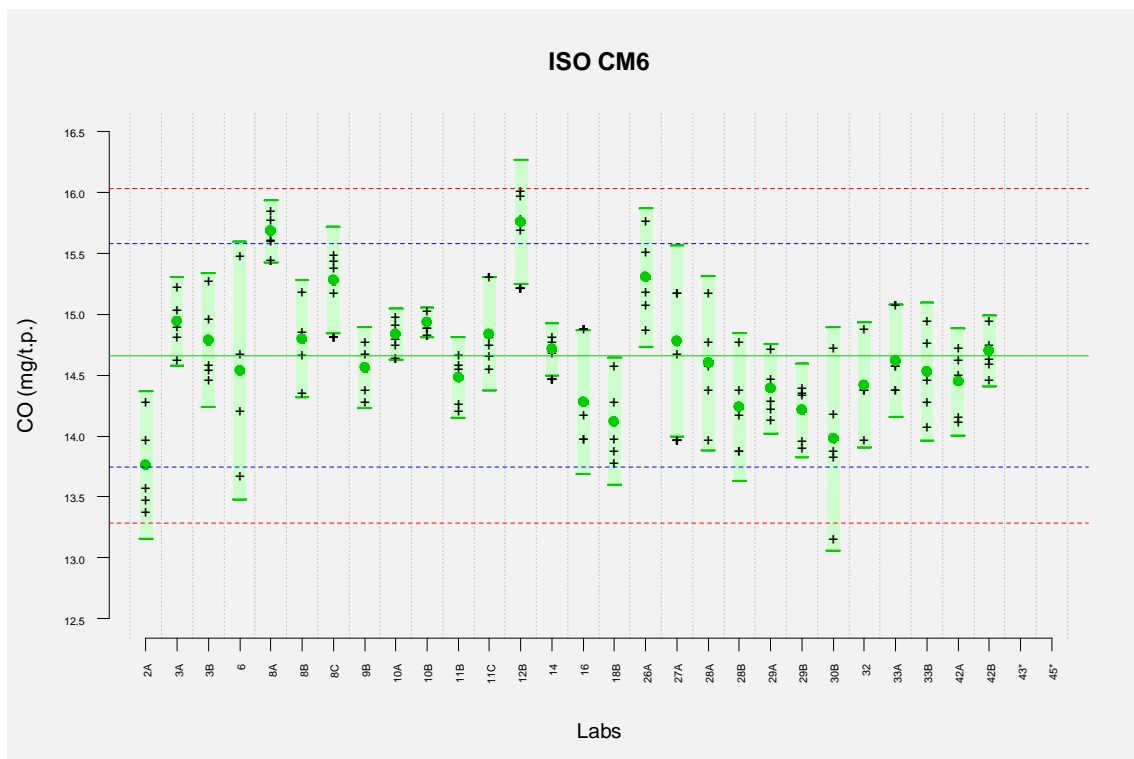
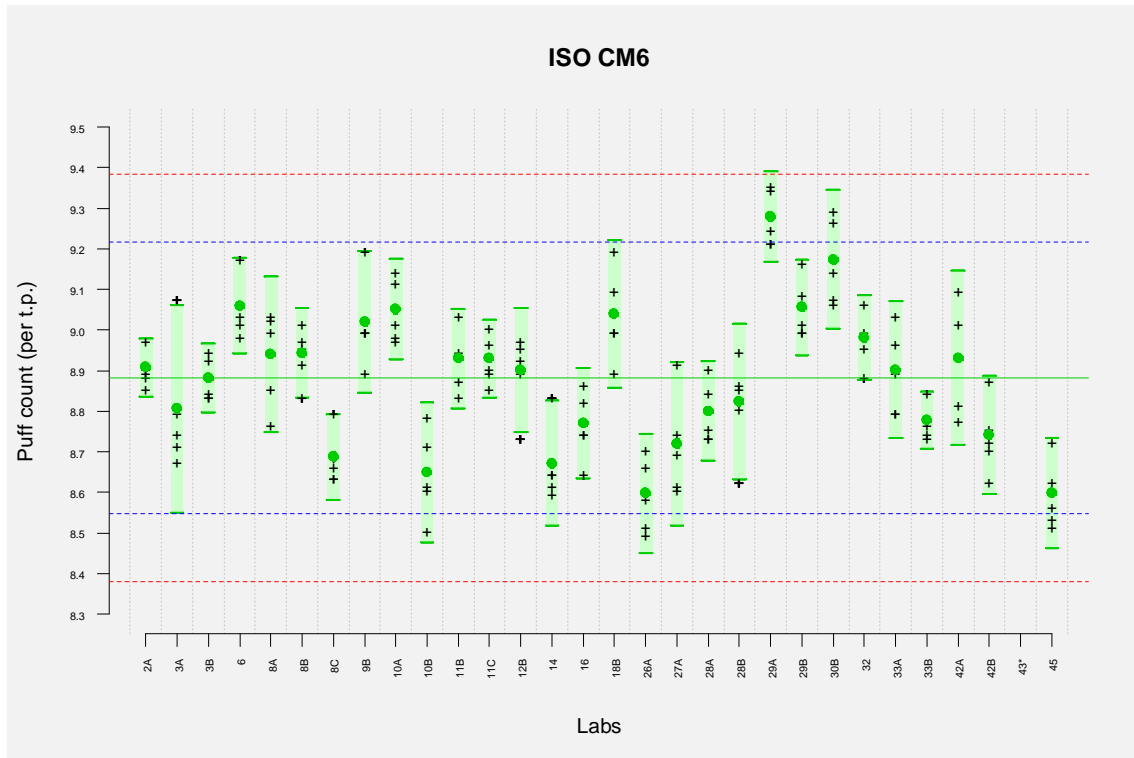


Figure 7: CM6 Individual observations of mainstream “ISO” PUFF Count



1.2. CM7

Figure 8: CM7 Individual observations of mainstream “ISO” conditioned WEIGHT

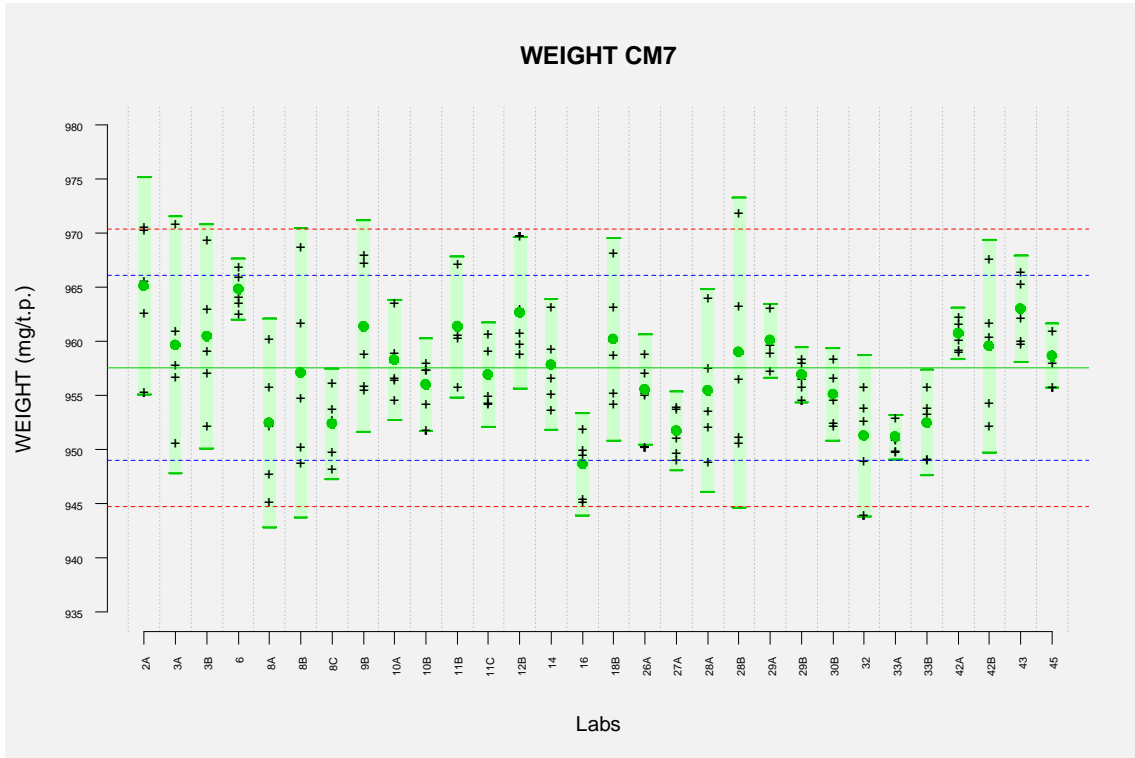


Figure 9: CM7 Individual observations of mainstream “ISO” TPM

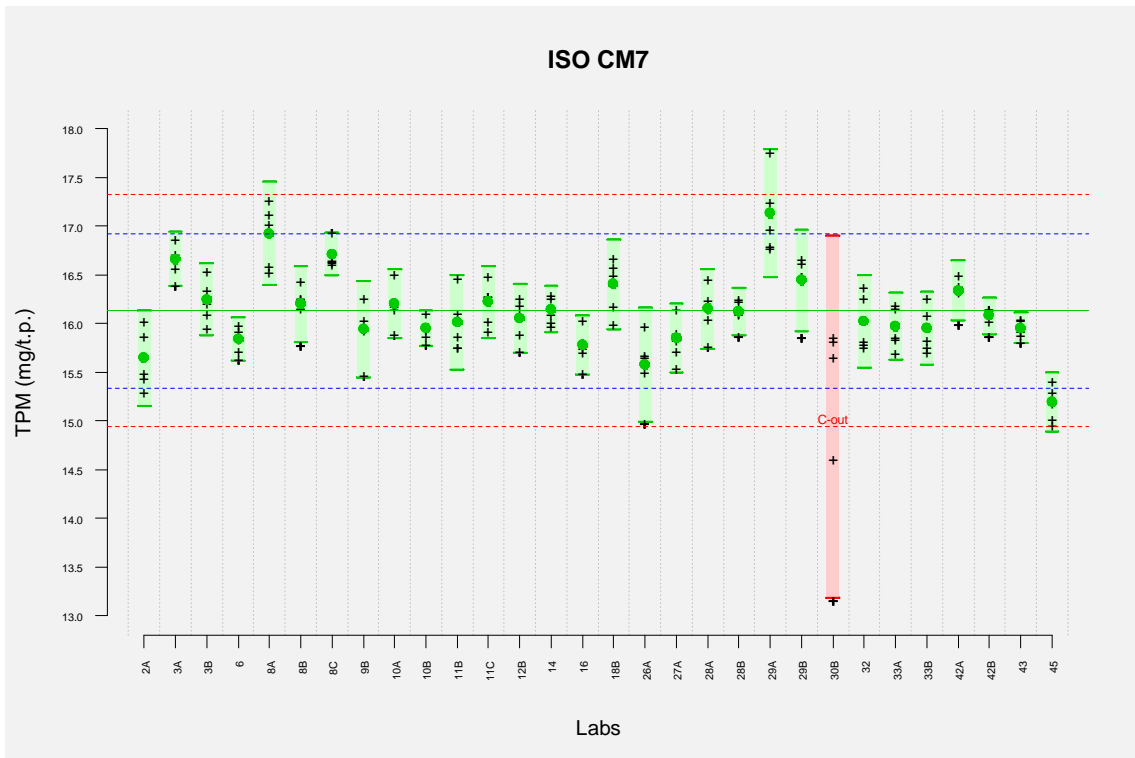


Figure 10: CM7 Individual observations of mainstream "ISO" WATER

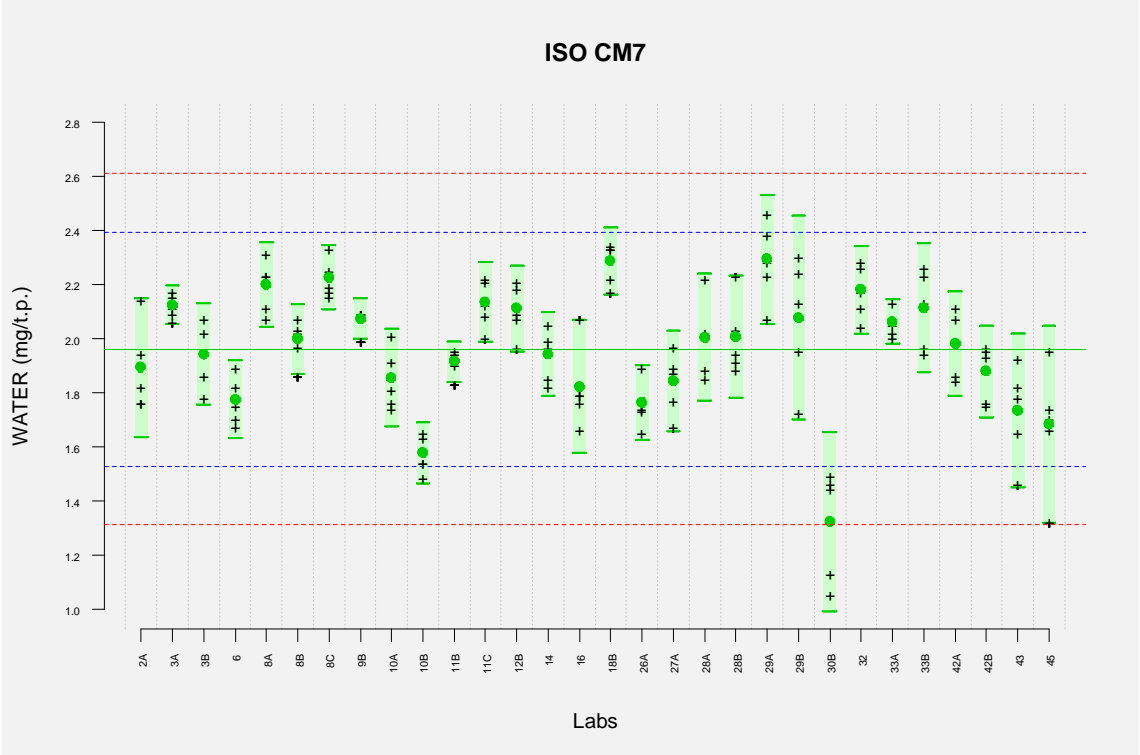


Figure 11: CM7 Individual observations of mainstream "ISO" Nicotine

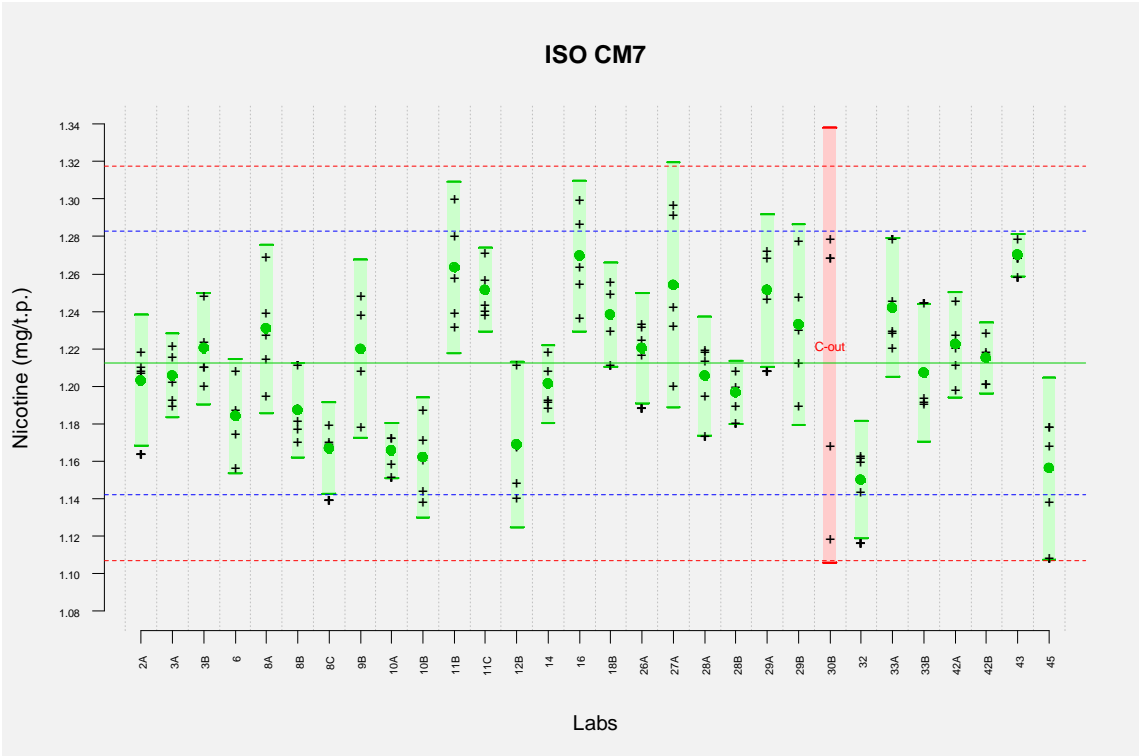
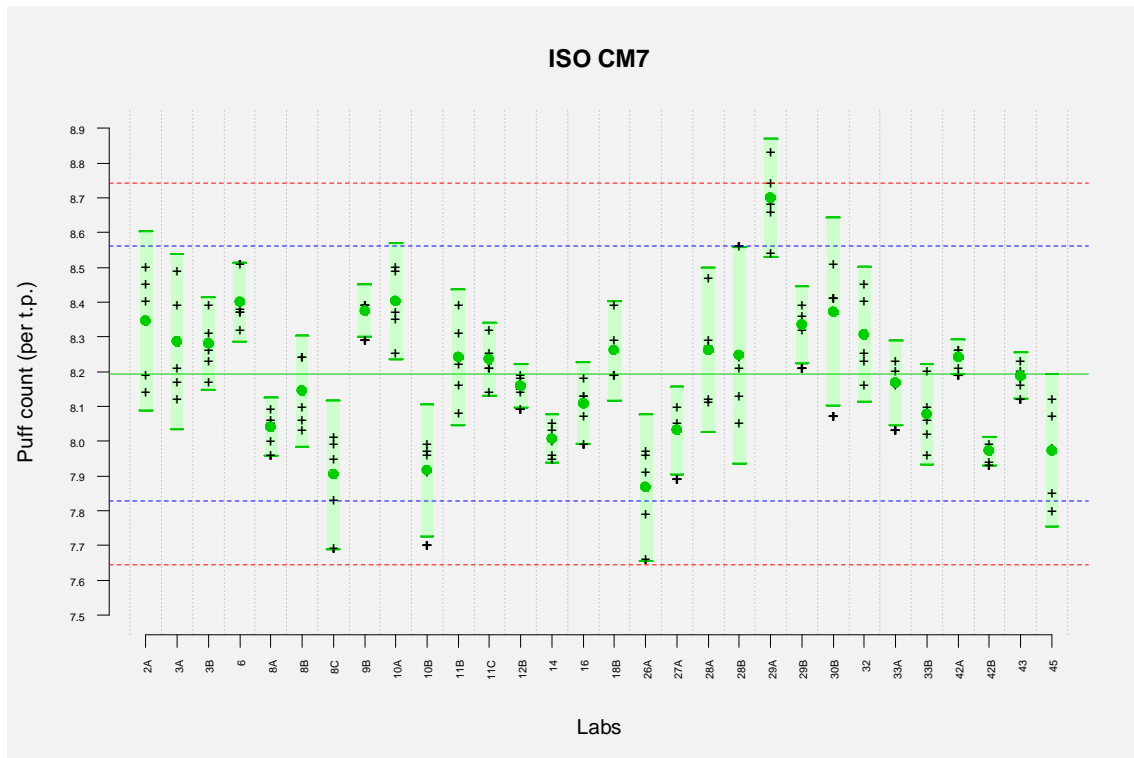


Figure 14: CM7 Individual observations of mainstream “ISO” PUFF Count



2. Intense smoking regime

2.1. CM6

Figure 15: CM6 Individual observations of mainstream “Intense” TPM

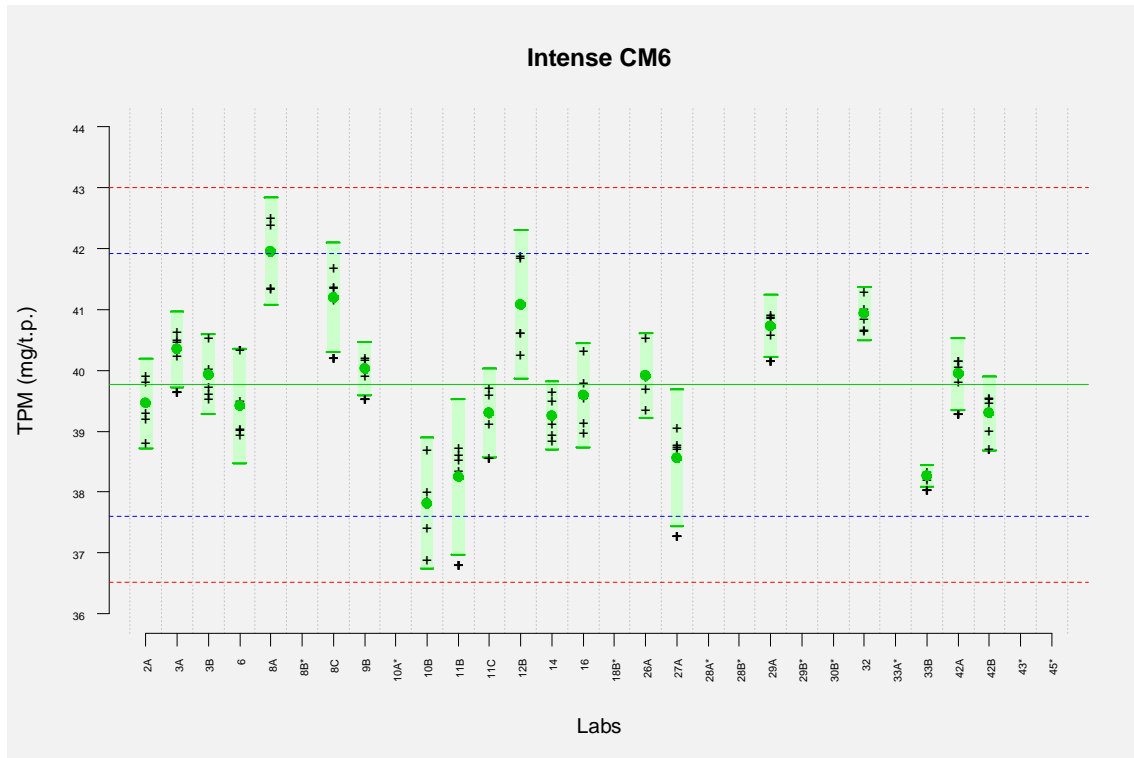


Figure 16: CM6 Individual observations of mainstream “Intense” WATER

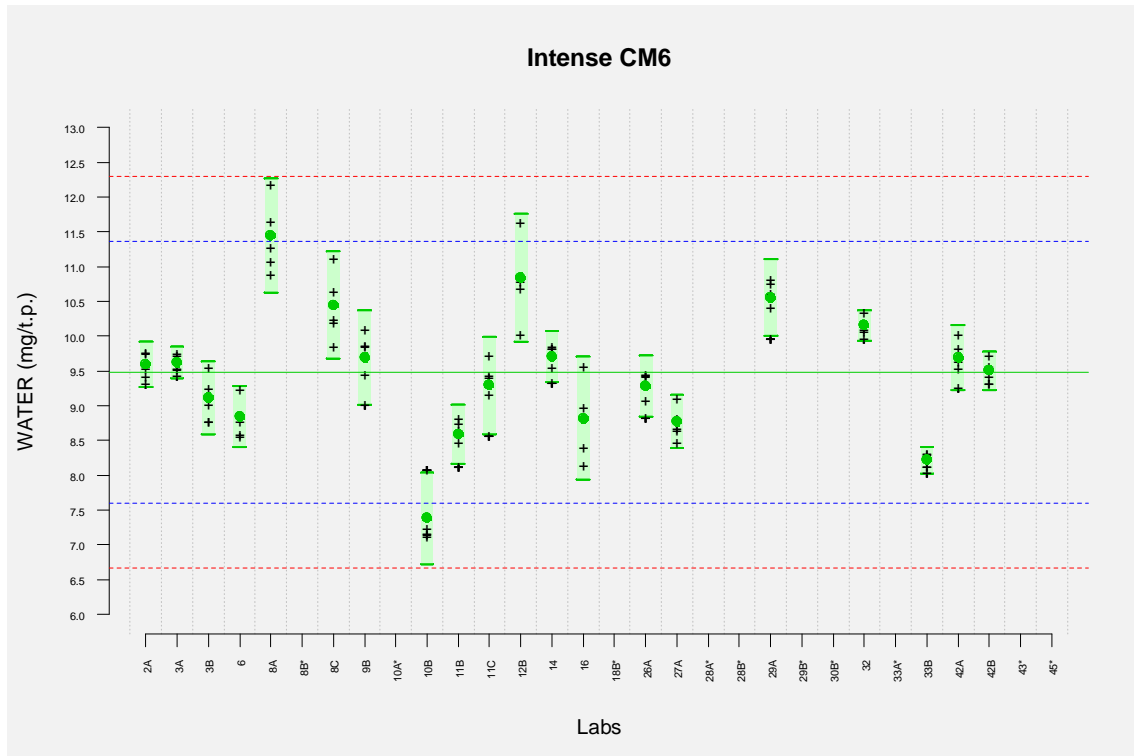


Figure 17: CM6 Individual observations of mainstream “Intense” Nicotine

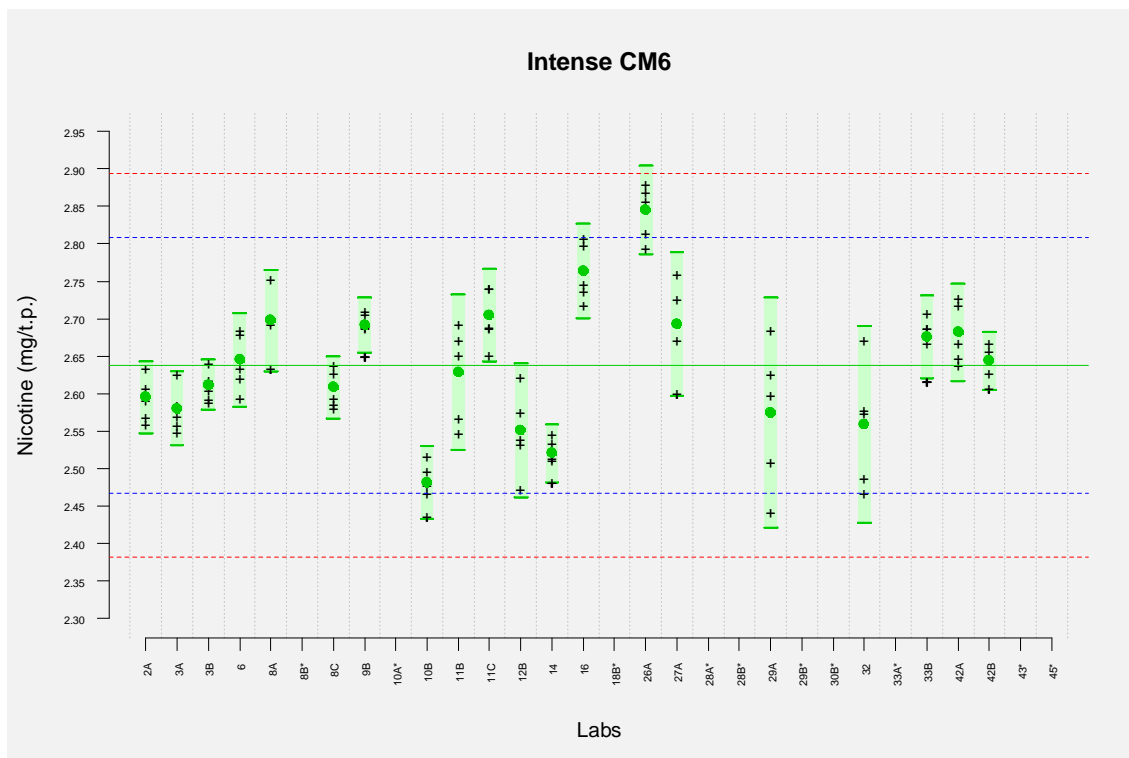


Figure 18: CM6 Individual observations of mainstream “Intense” NFDPM

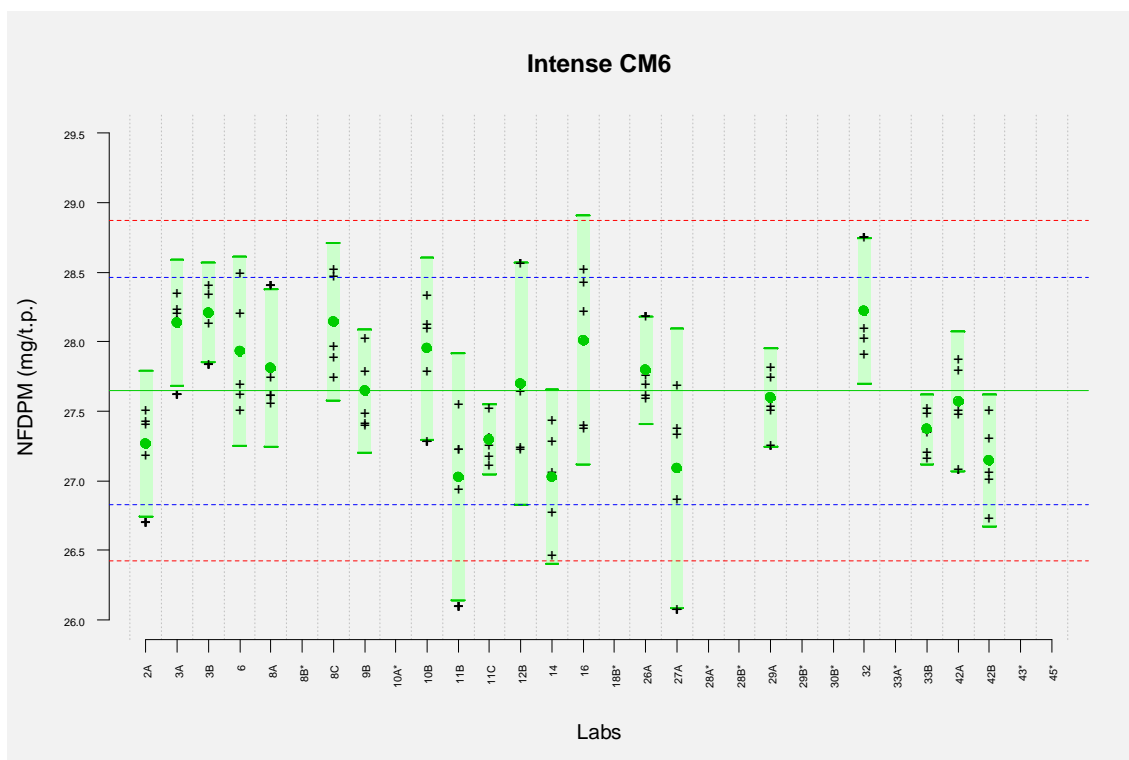


Figure 19: CM6 Individual observations of mainstream "Intense" CO

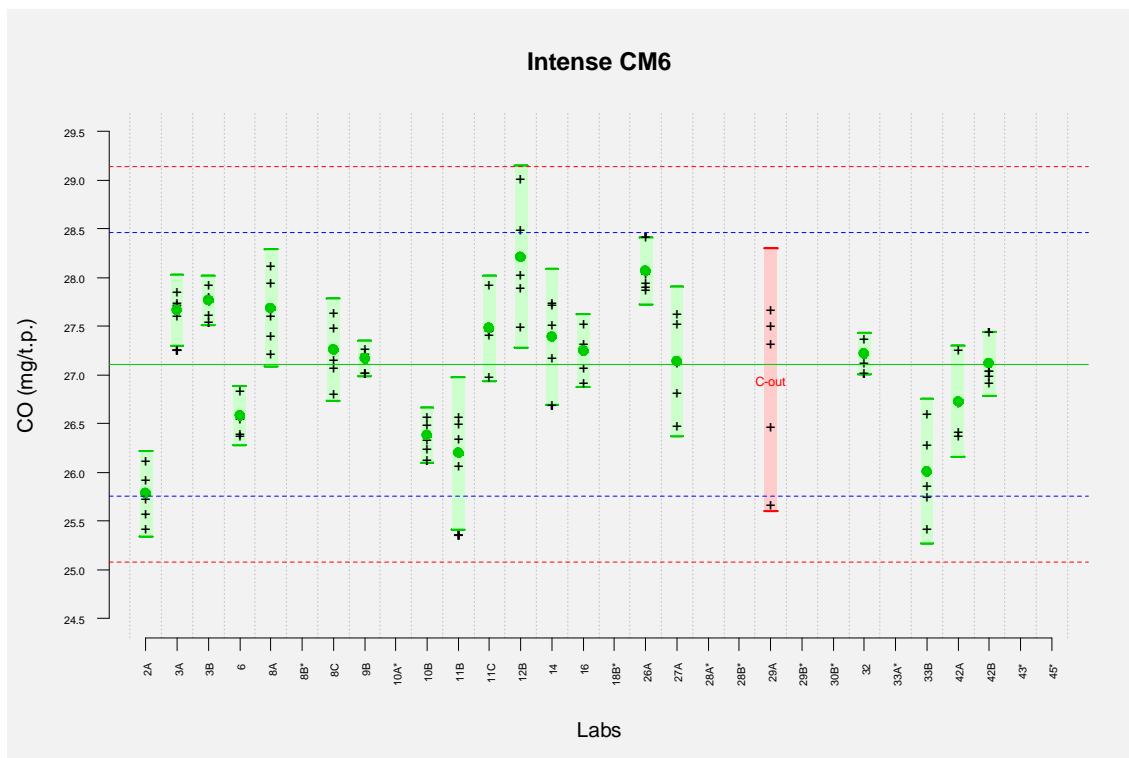


Figure 20: CM6 Individual observations of mainstream "Intense" PUFF Count

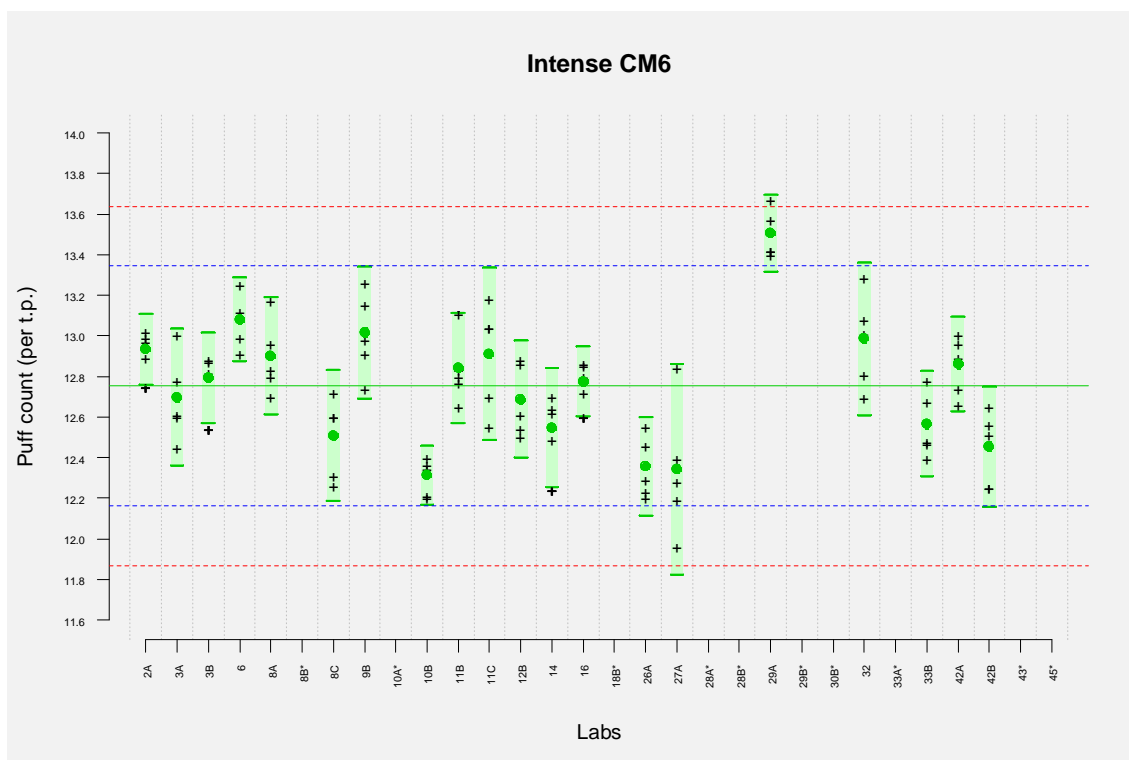


Figure 23: CM7 Individual observations of mainstream “Intense” Nicotine

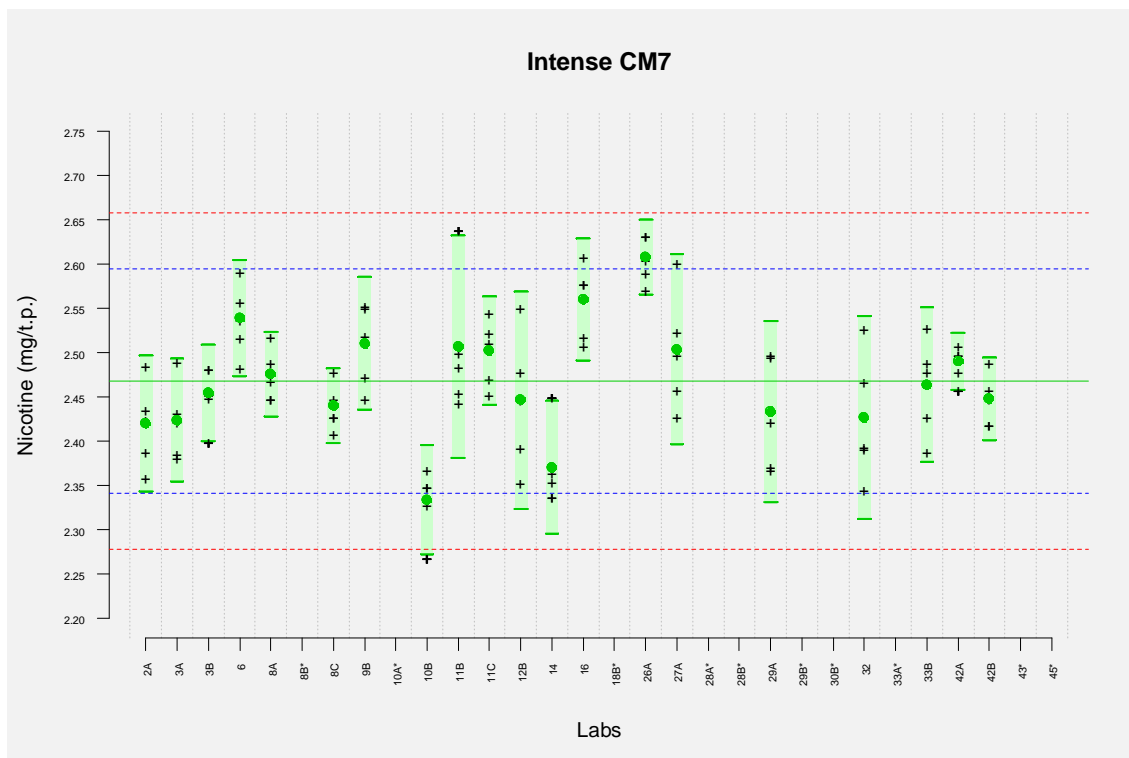


Figure 24: CM7 Individual observations of mainstream “Intense” NFDPM

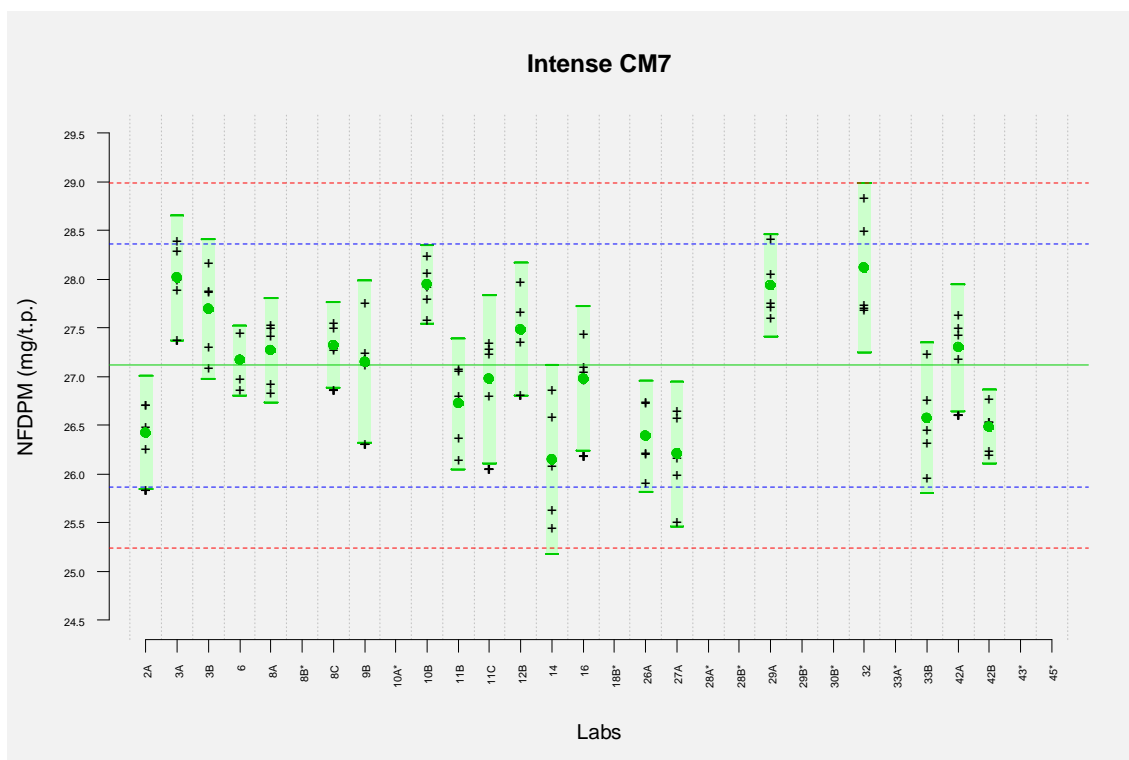


Figure 25: CM7 Individual observations of mainstream "Intense" CO

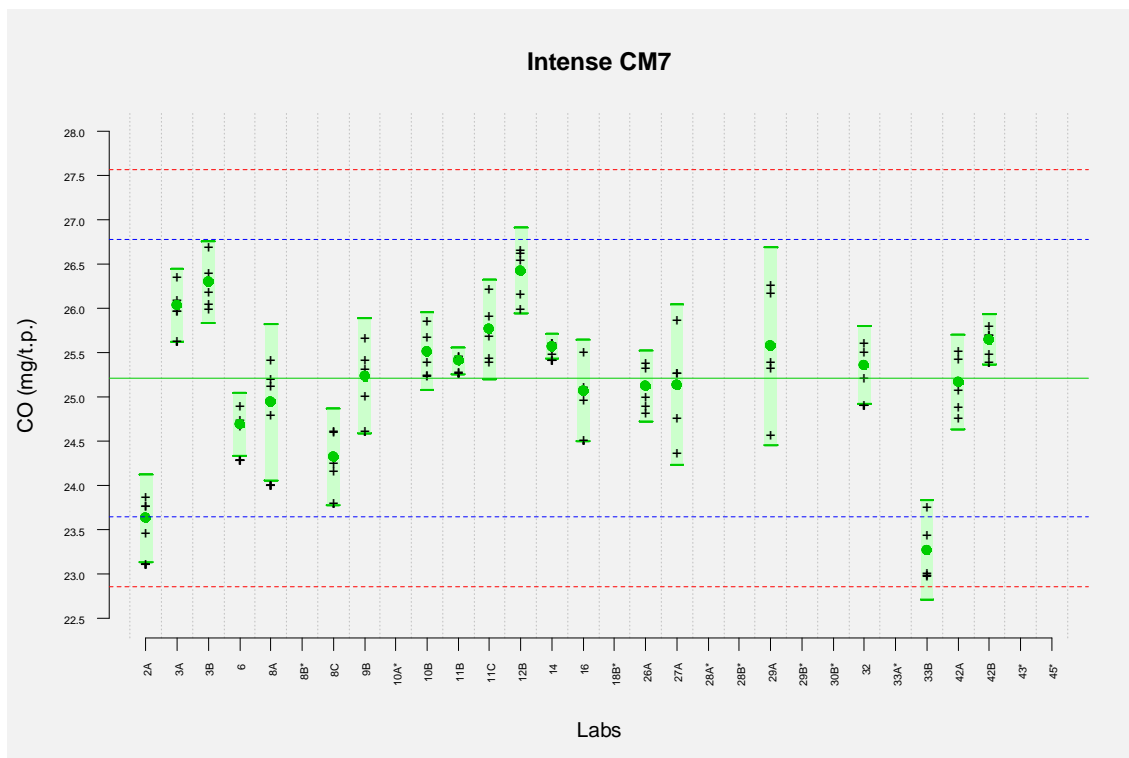
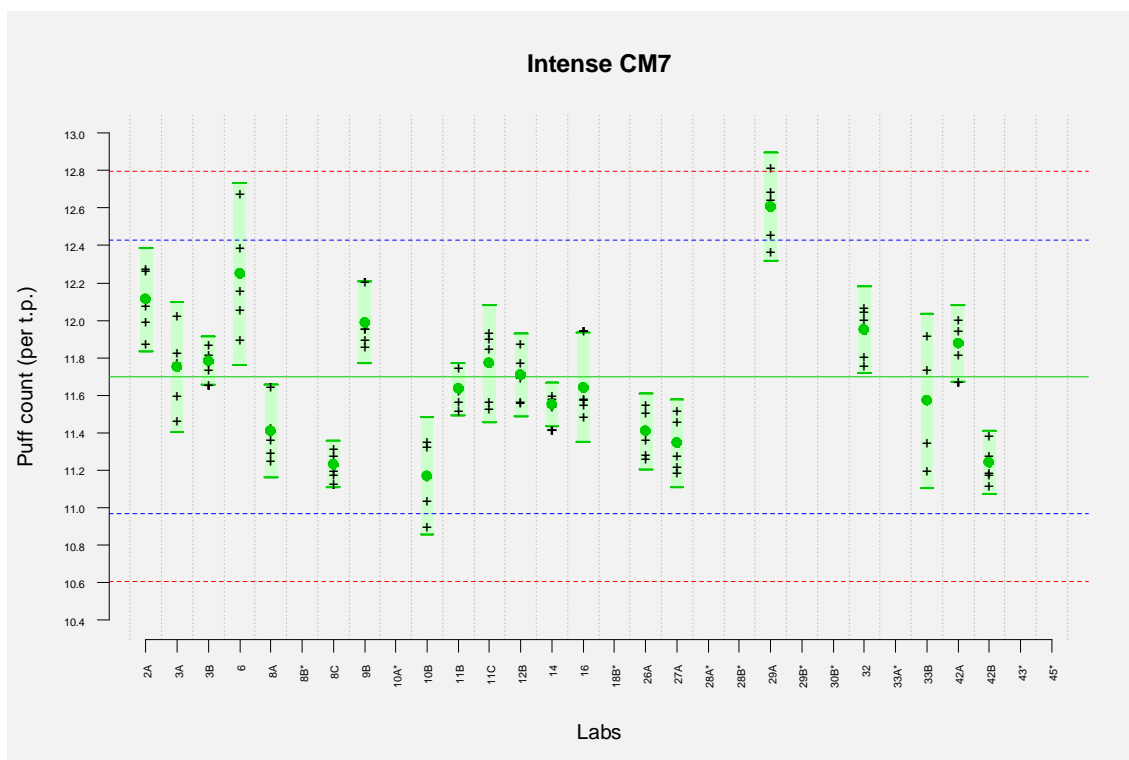


Figure 26: CM7 Individual observations of mainstream "Intense" PUFF Count



APPENDIX I - LINEAR SMOKING MACHINES

Smoking regime	Coresta Monitor	Parameter	No of Labs	Mean	Repeatability		Reproducibility		Outlying Labs			Stragglings Labs	No data
					sr	r	sR	R	C	G/G2	Gi		
ISO	CM6	TPM	21	17.8	0.41	1.16	0.61	1.73					
		WATER	21	1.6	0.20	0.58	0.28	0.80				44	
		Nicotine	21	1.40	0.033	0.094	0.063	0.179					
		NFDPM	21	14.8	0.37	1.03	0.58	1.64					
		CO	21	14.1	0.34	0.97	0.64	1.82					
	Puff Count	19	9.2	0.14	0.40	0.20	0.58		19, 31A			19, 31A	
	CM7	TPM	21	16.7	0.42	1.18	0.69	1.95					
		WATER	19	1.5	0.17	0.48	0.26	0.73	1, 44				
		Nicotine	20	1.28	0.031	0.089	0.041	0.116		25			
		NFDPM	21	13.9	0.34	0.96	0.60	1.70			1		44
CO		21	12.7	0.42	1.19	0.70	1.97						
Puff Count	21	8.6	0.16	0.46	0.27	0.77							
Intense	CM6	TPM	21	45.2	0.97	2.75	2.36	6.67					
		WATER	19	12.2	0.67	1.89	1.59	4.50	30A, 44				
		Nicotine	21	2.76	0.048	0.136	0.123	0.347					
		NFDPM	20	30.3	0.75	2.13	1.61	4.57					44
		CO	21	26.4	0.51	1.44	0.88	2.48	30A				
	Puff Count	21	13.0	0.17	0.49	0.38	1.08						
	CM7	TPM	21	44.5	1.20	3.39	2.65	7.51					44
		WATER	20	12.1	0.66	1.86	1.78	5.04	44				30A
		Nicotine	21	2.59	0.050	0.142	0.110	0.310					19, 25
		NFDPM	21	29.8	0.90	2.53	1.84	5.20			1		
CO		21	24.5	0.63	1.79	1.09	3.08						
Puff Count	21	12.1	0.18	0.52	0.48	1.35							

1. ISO smoking regime

1.1. CM6

Figure 1: CM6 Individual observations of mainstream “ISO” conditioned WEIGHT

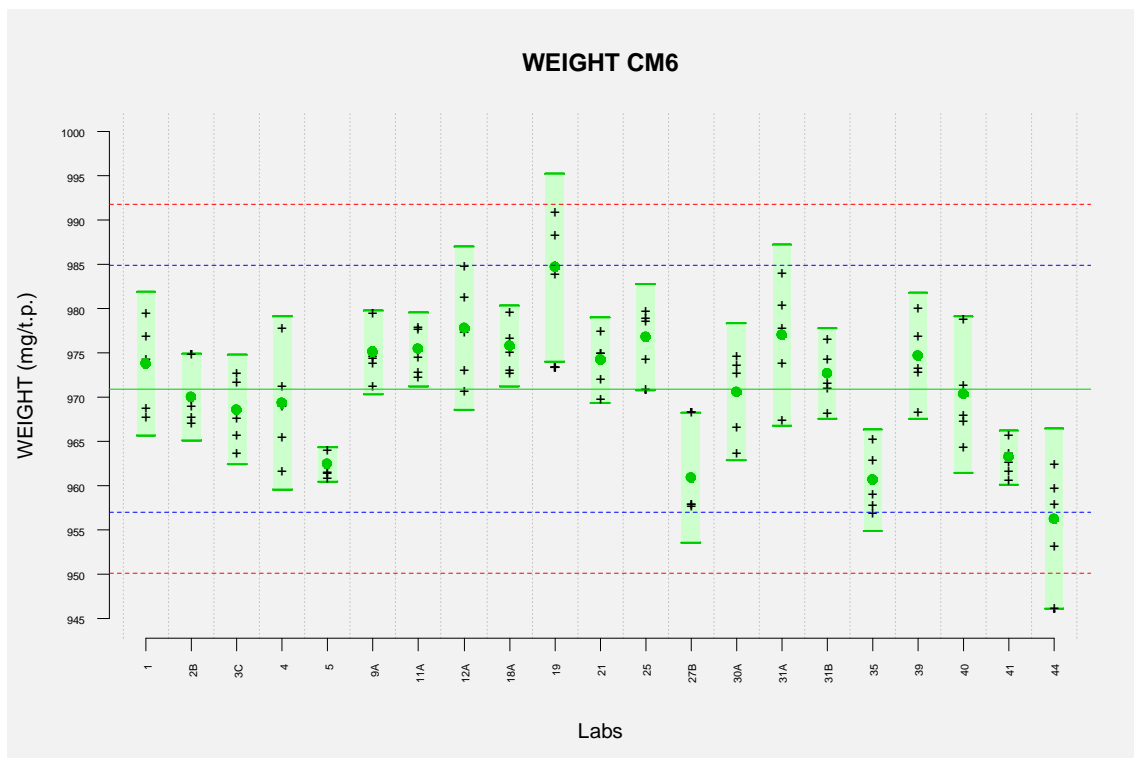


Figure 2: CM6 Individual observations of mainstream “ISO” TPM

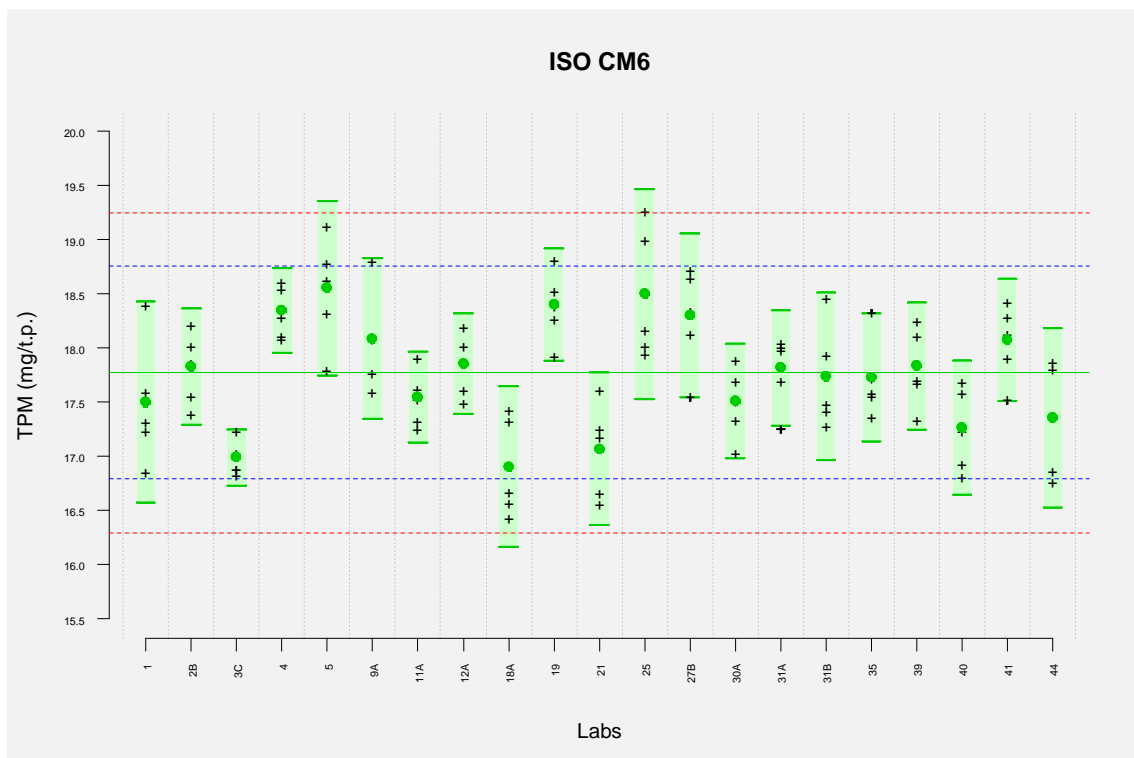


Figure 3: CM6 Individual observations of mainstream “ISO” WATER

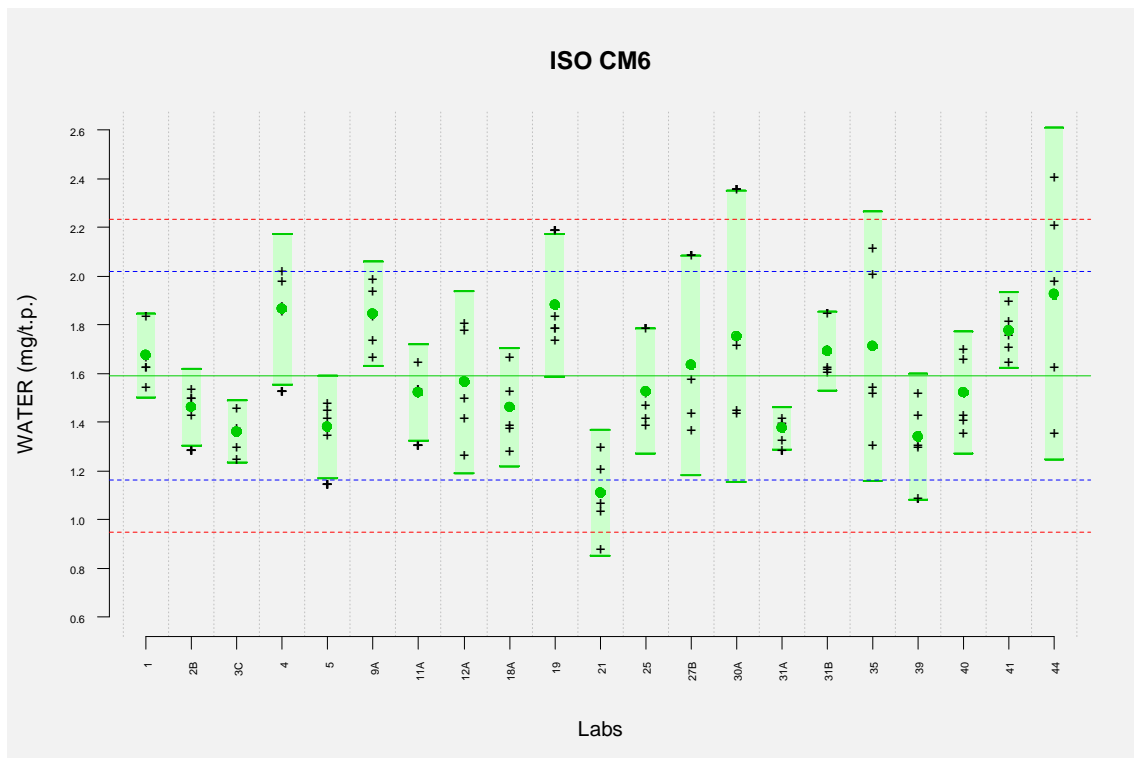


Figure 4: CM6 Individual observations of mainstream "ISO" Nicotine

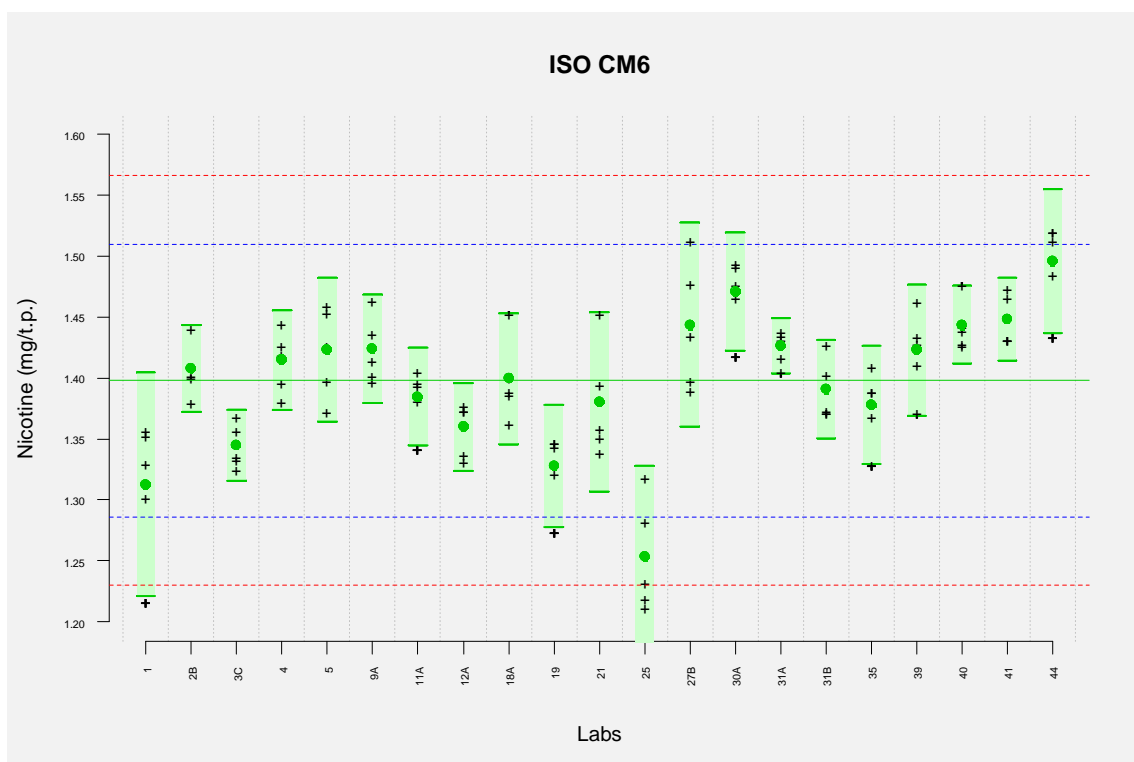


Figure 5: CM6 Individual observations of mainstream “ISO” NFDPM

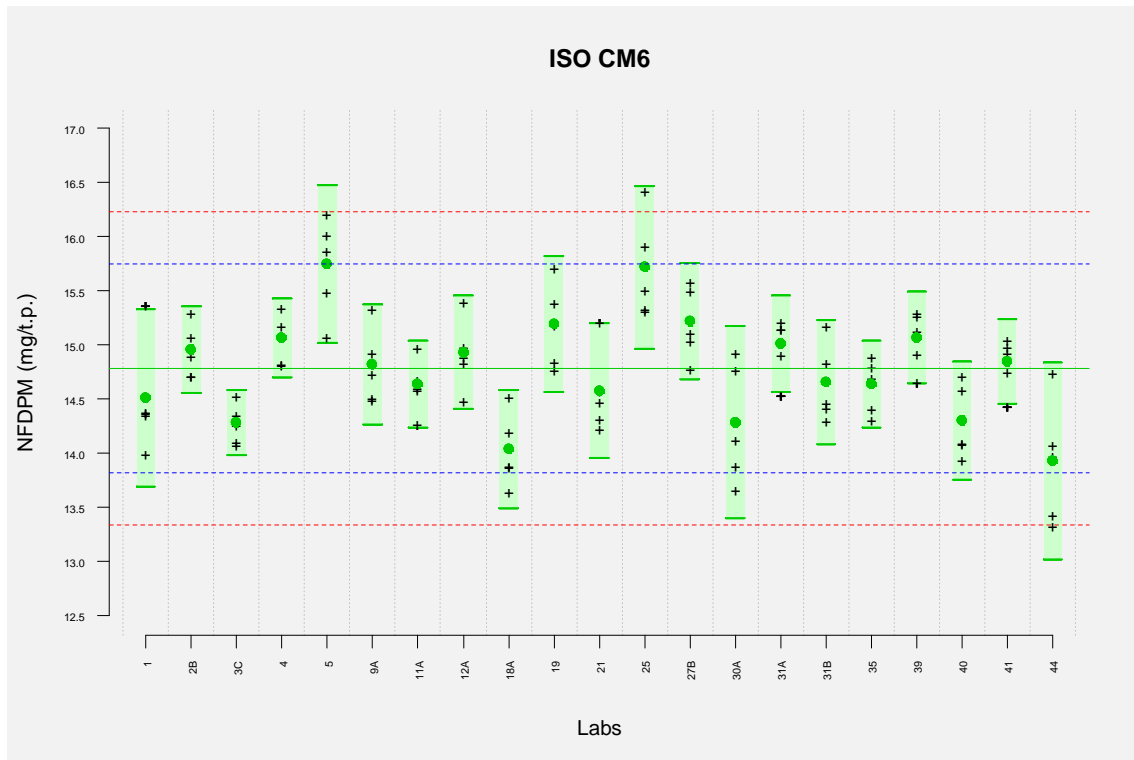


Figure 6: CM6 Individual observations of mainstream “ISO” CO

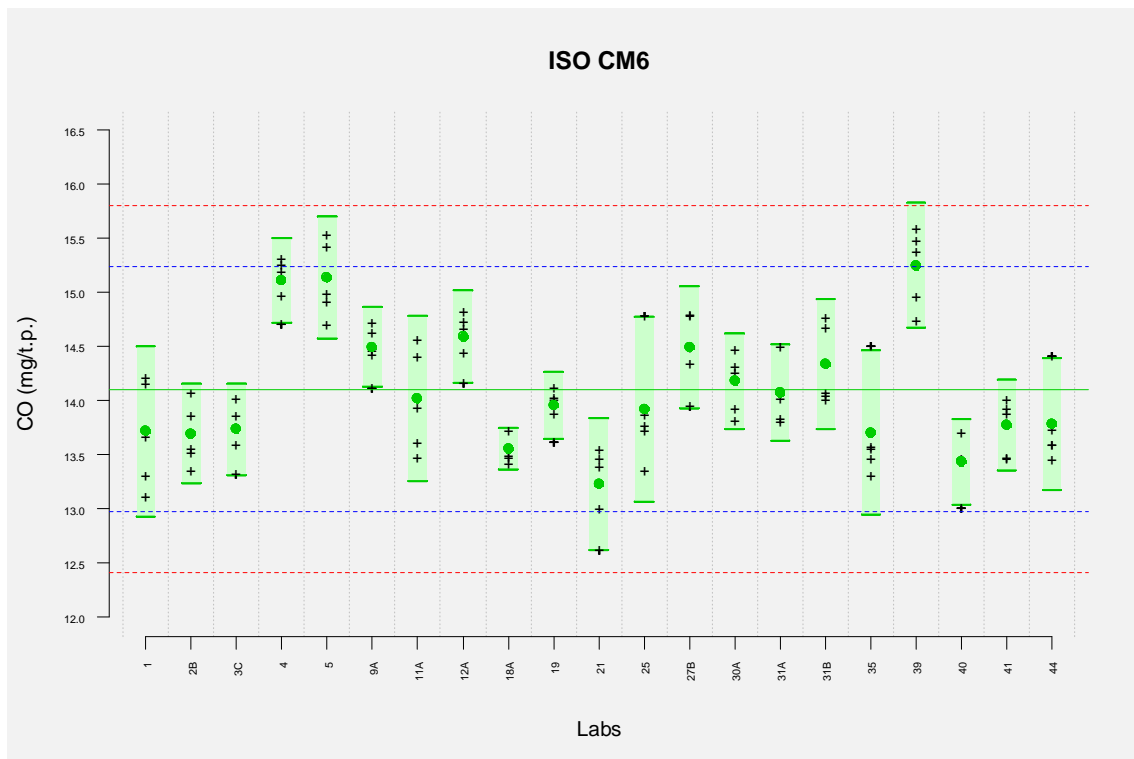
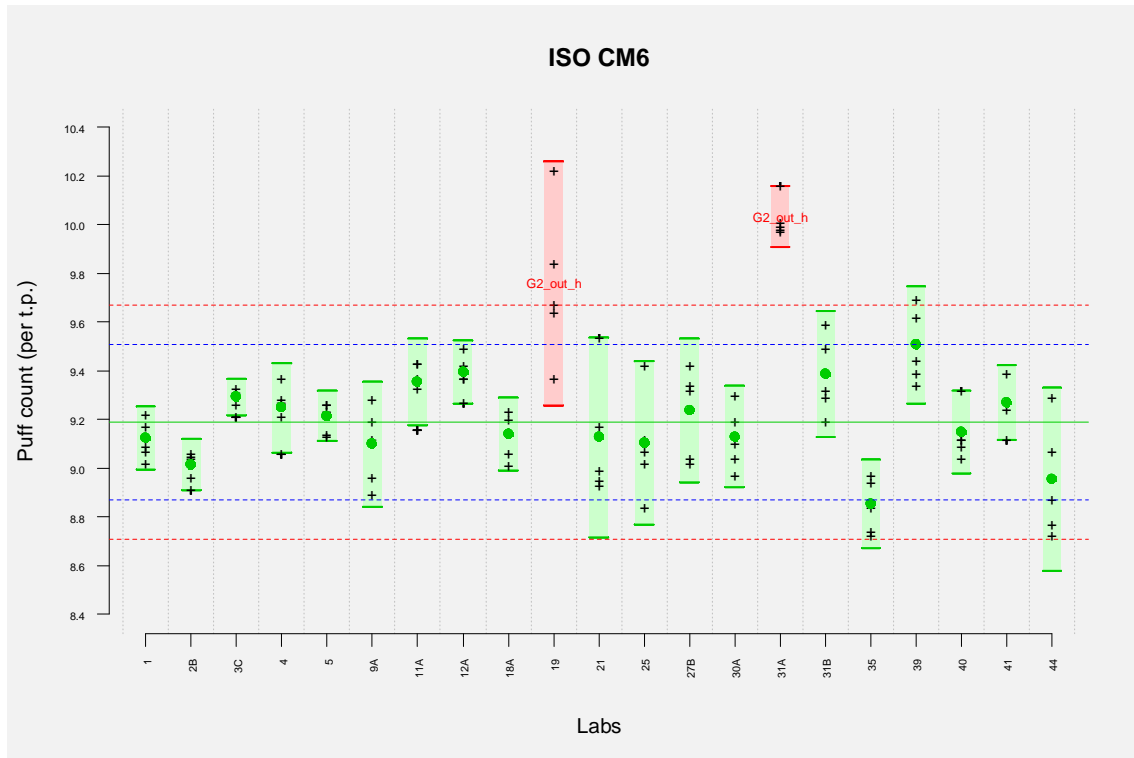


Figure 7: CM6 Individual observations of mainstream “ISO” PUFF Count



1.2. CM7

Figure 8: CM7 Individual observations of mainstream “ISO” conditioned WEIGHT

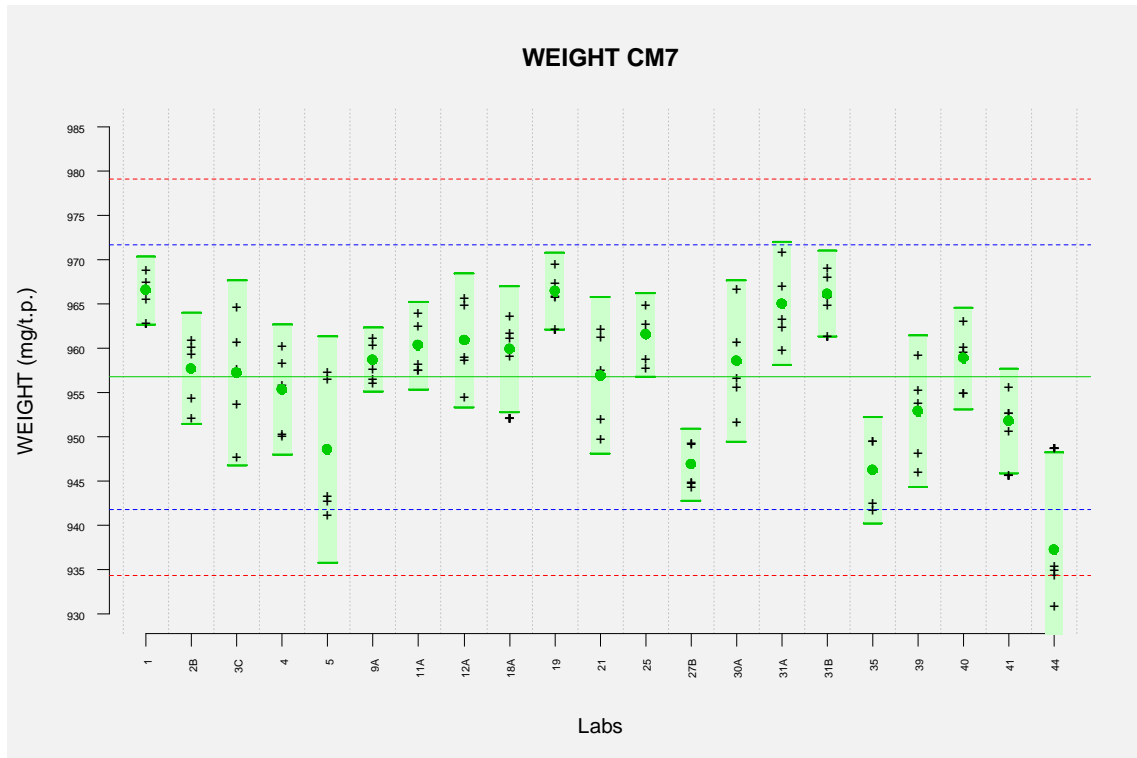


Figure 9: CM7 Individual observations of mainstream “ISO” TPM

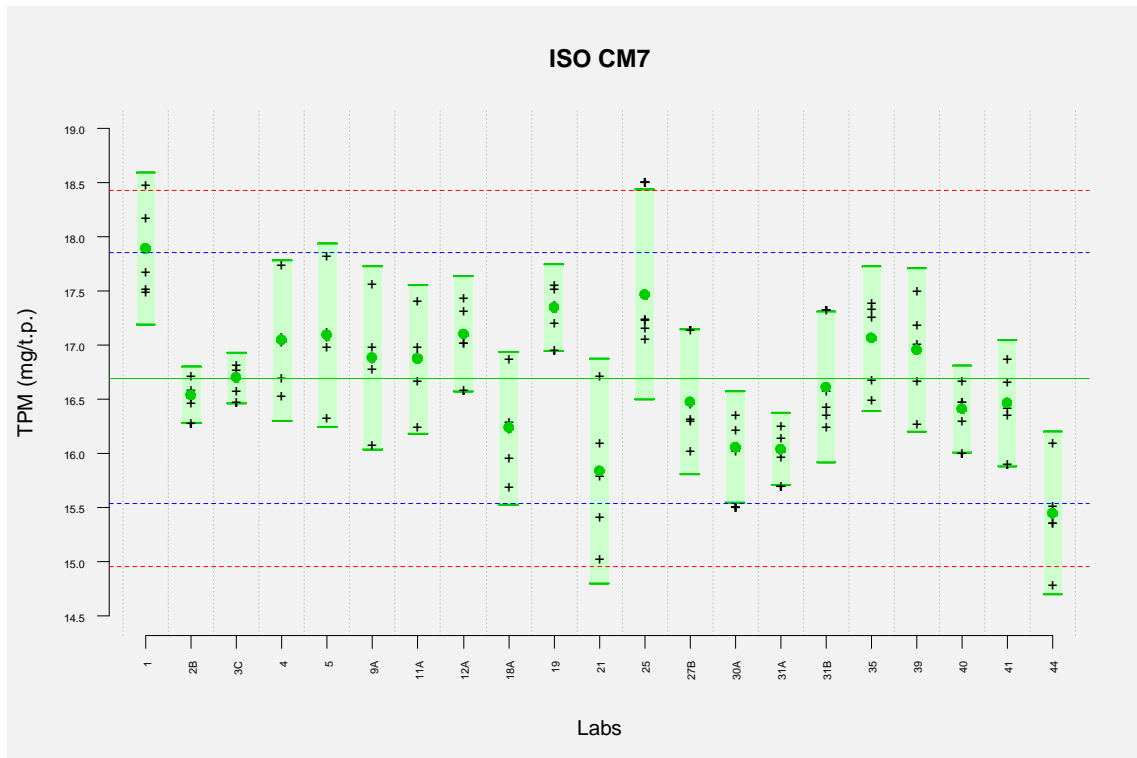


Figure 10: CM7 Individual observations of mainstream "ISO" WATER

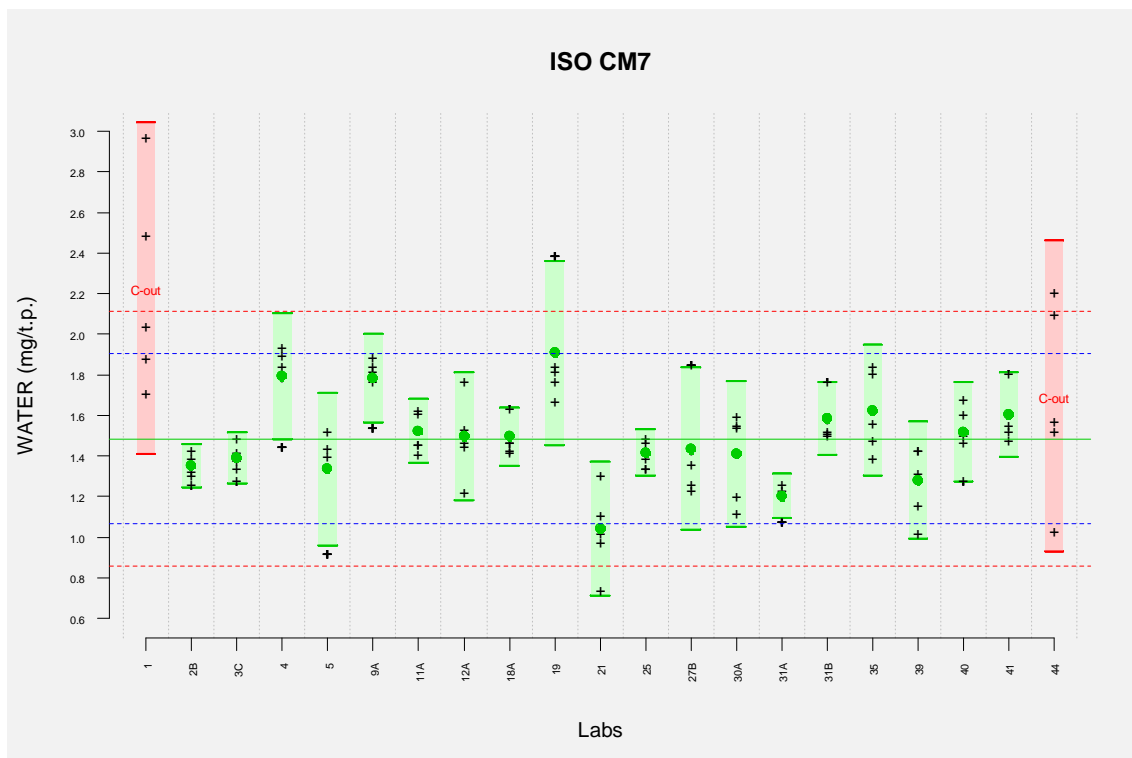


Figure 11: CM7 Individual observations of mainstream "ISO" Nicotine

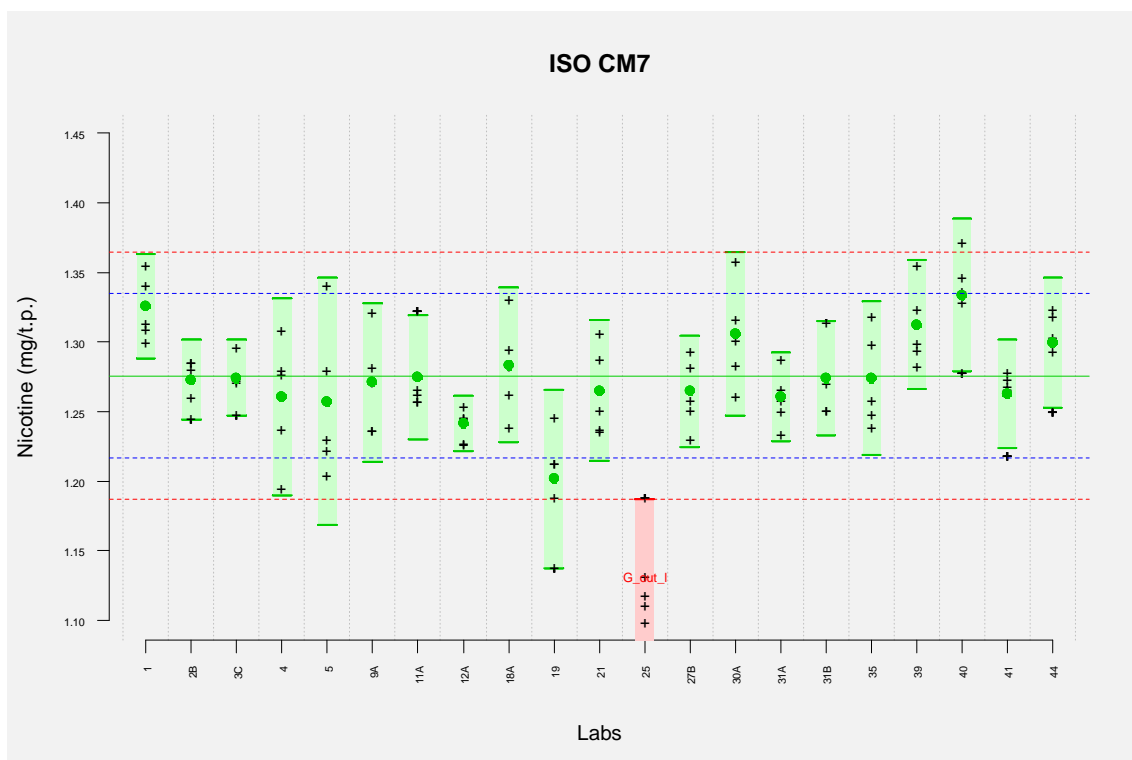


Figure 12: CM7 Individual observations of mainstream "ISO" NFDPM

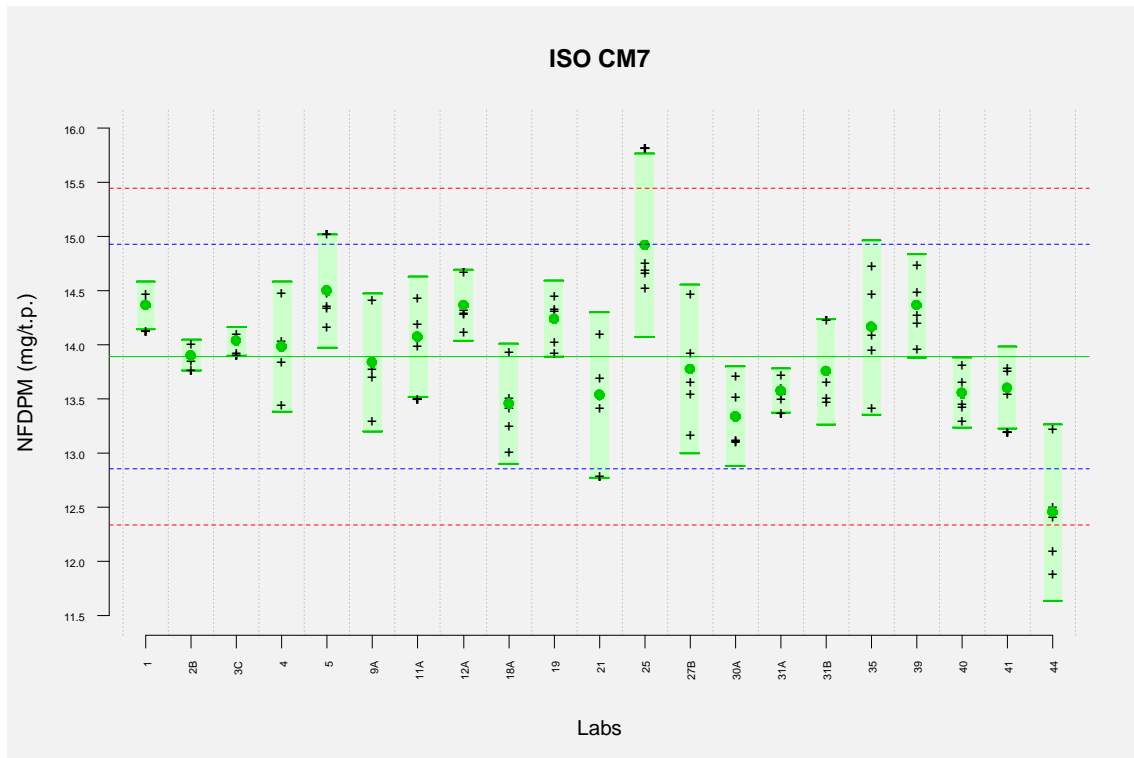


Figure 13: CM7 Individual observations of mainstream "ISO" CO

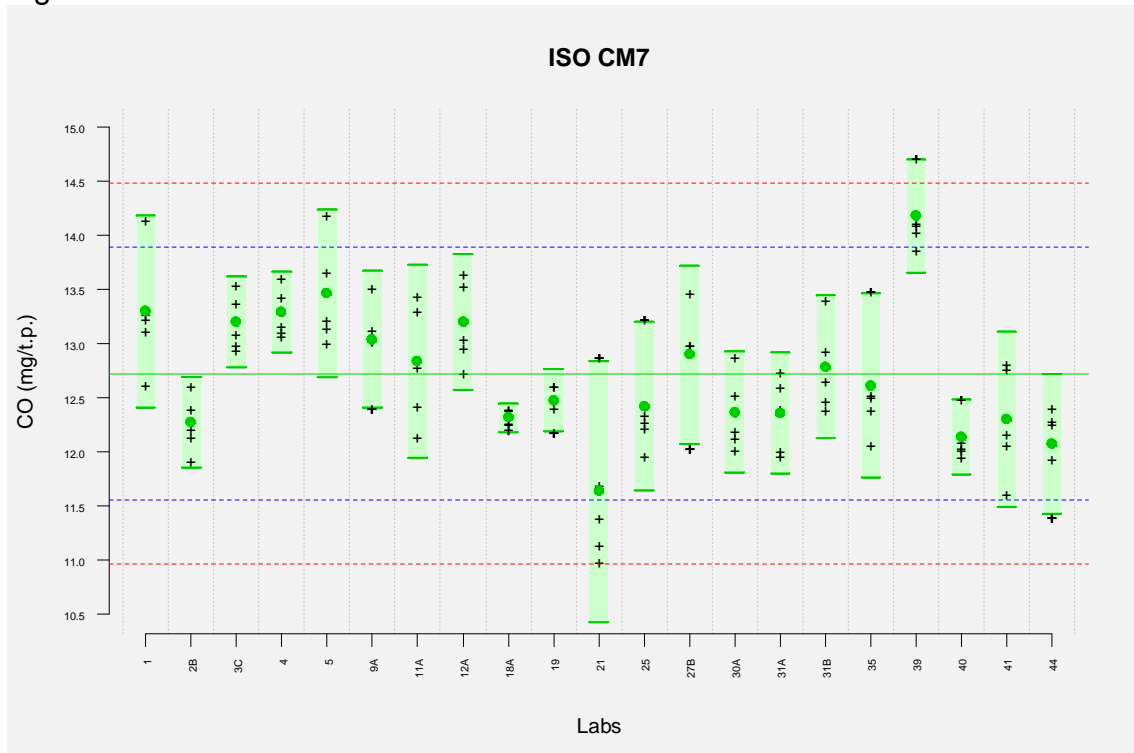
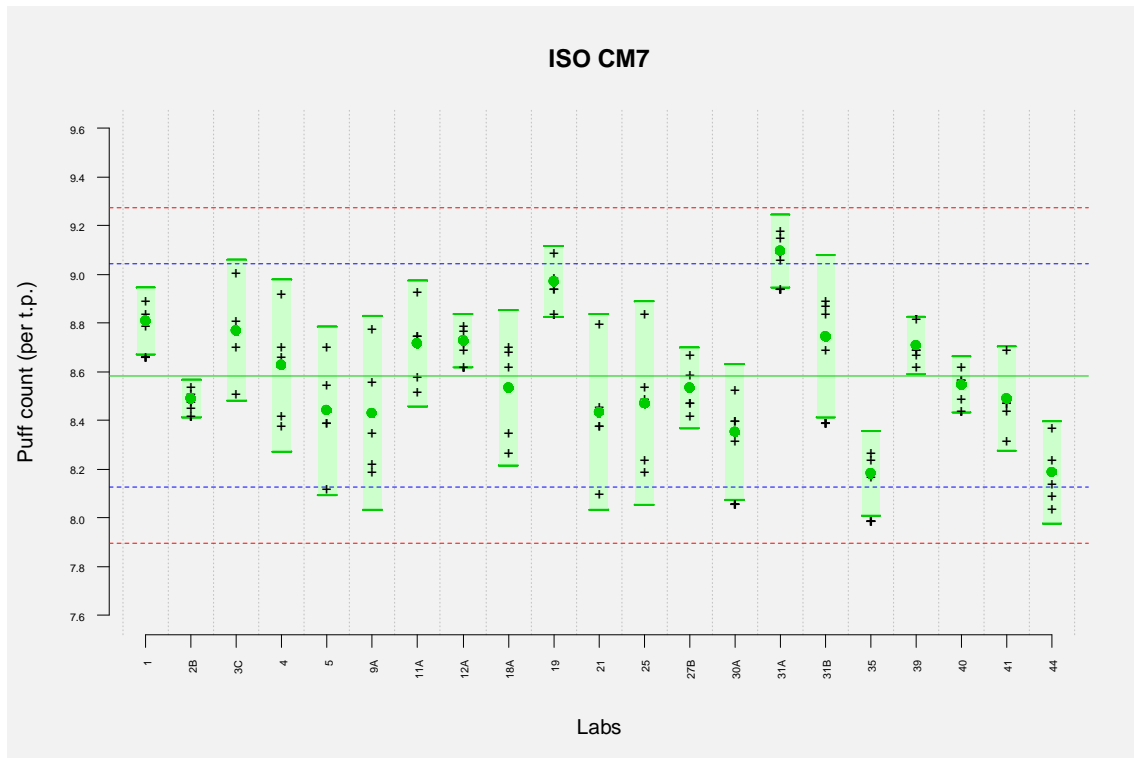


Figure 14: CM7 Individual observations of mainstream “ISO” PUFF Count



2. Intense smoking regime

2.1. CM6

Figure 15: CM6 Individual observations of mainstream “Intense” TPM

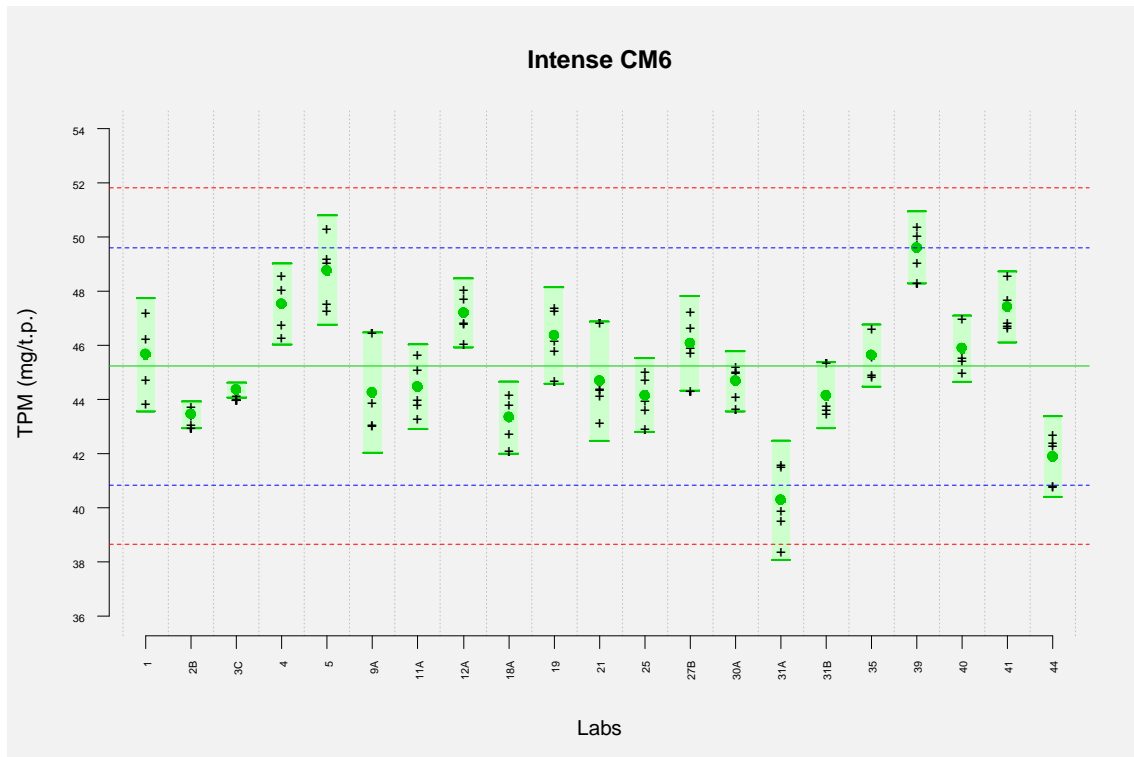


Figure 16: CM6 Individual observations of mainstream “Intense” WATER

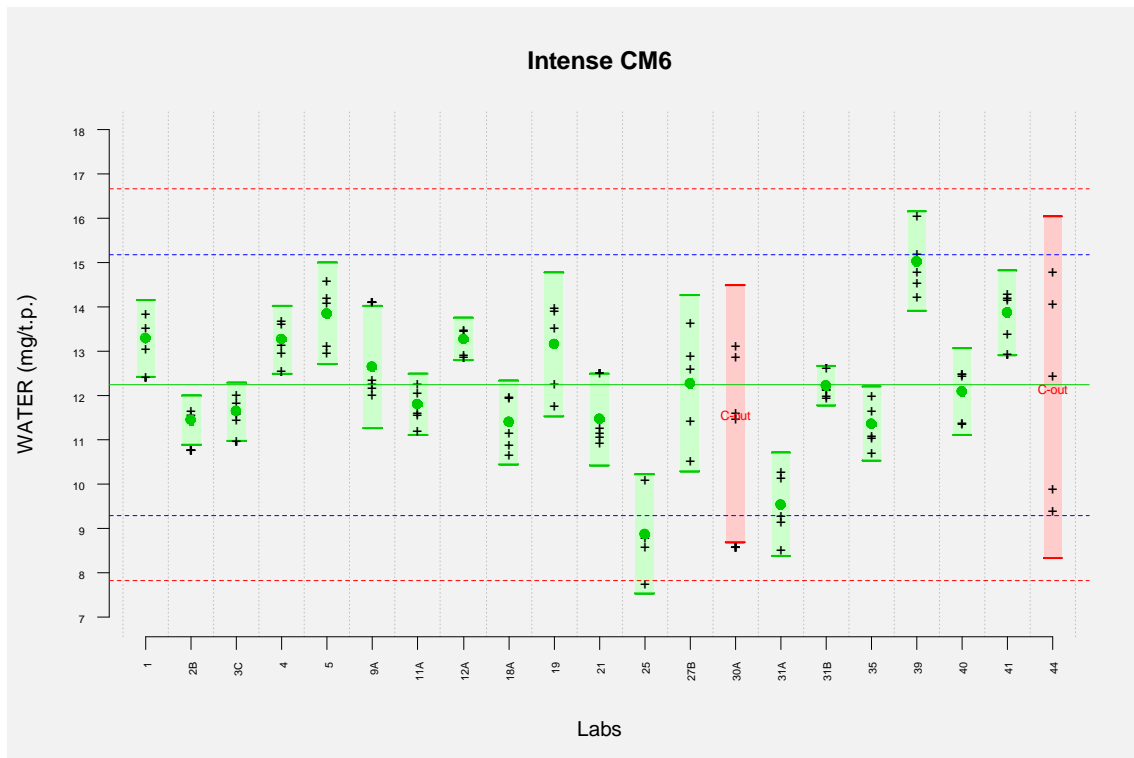


Figure 17: CM6 Individual observations of mainstream “Intense” Nicotine

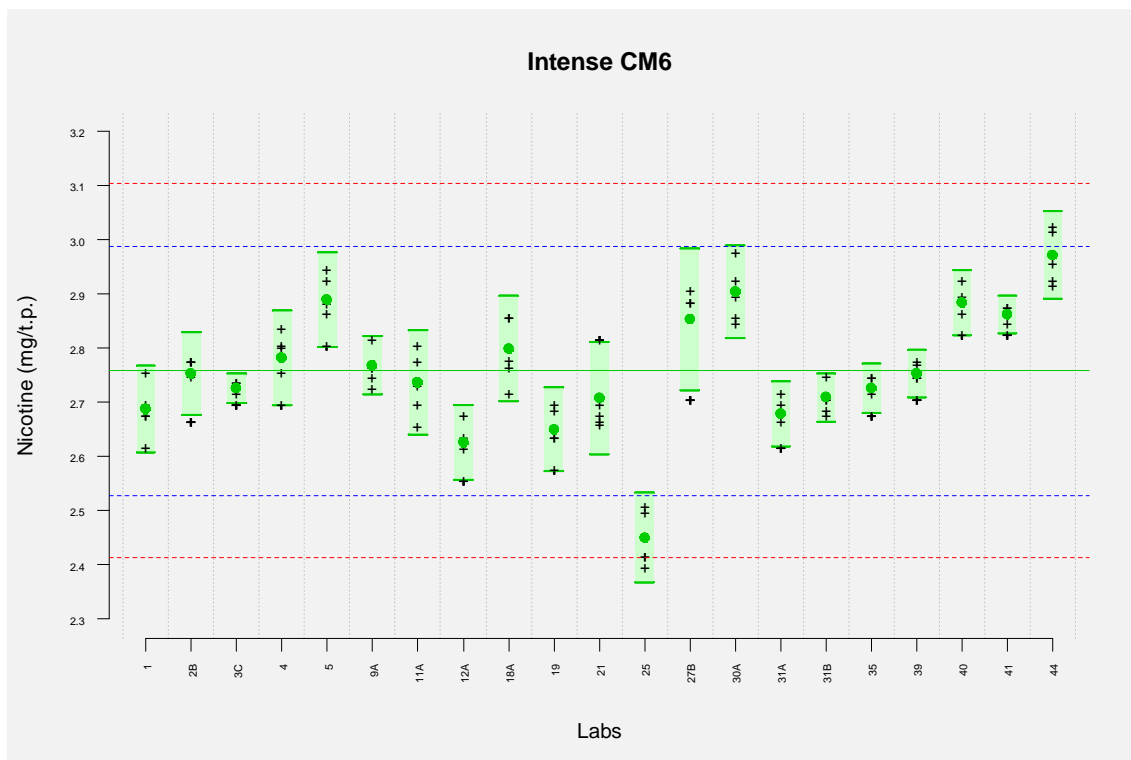


Figure 18: CM6 Individual observations of mainstream “Intense” NFDPM

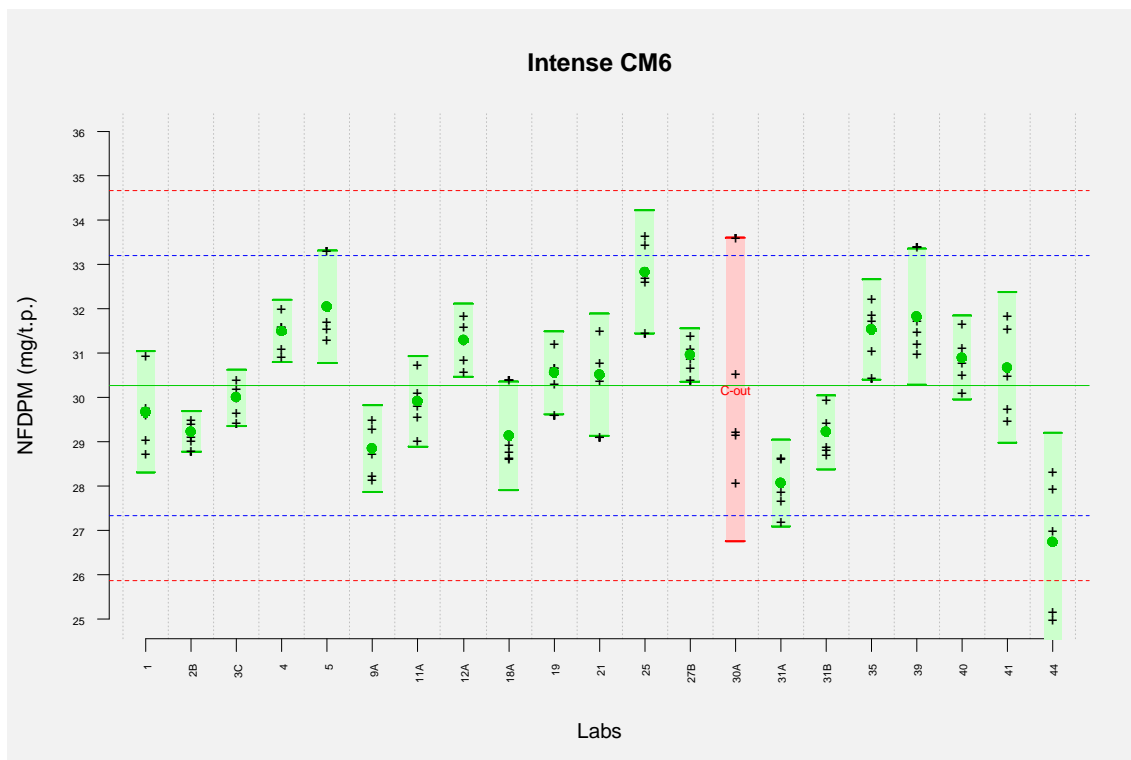


Figure 19: CM6 Individual observations of mainstream “Intense” CO

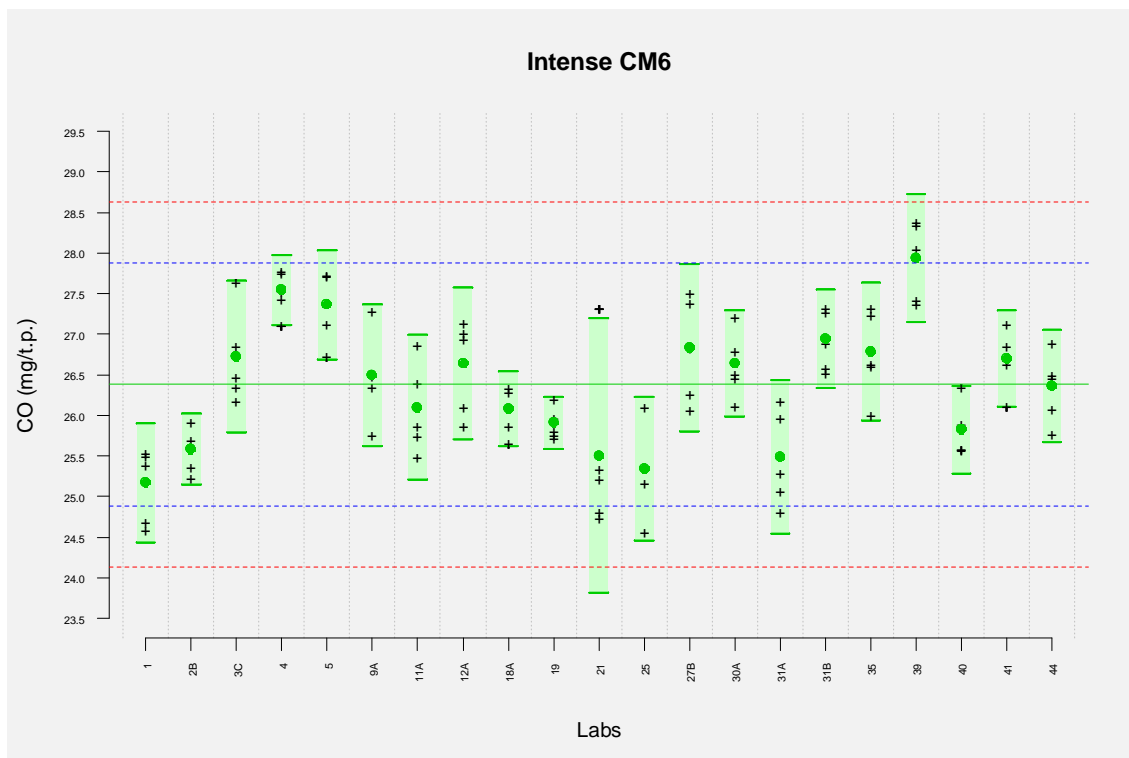
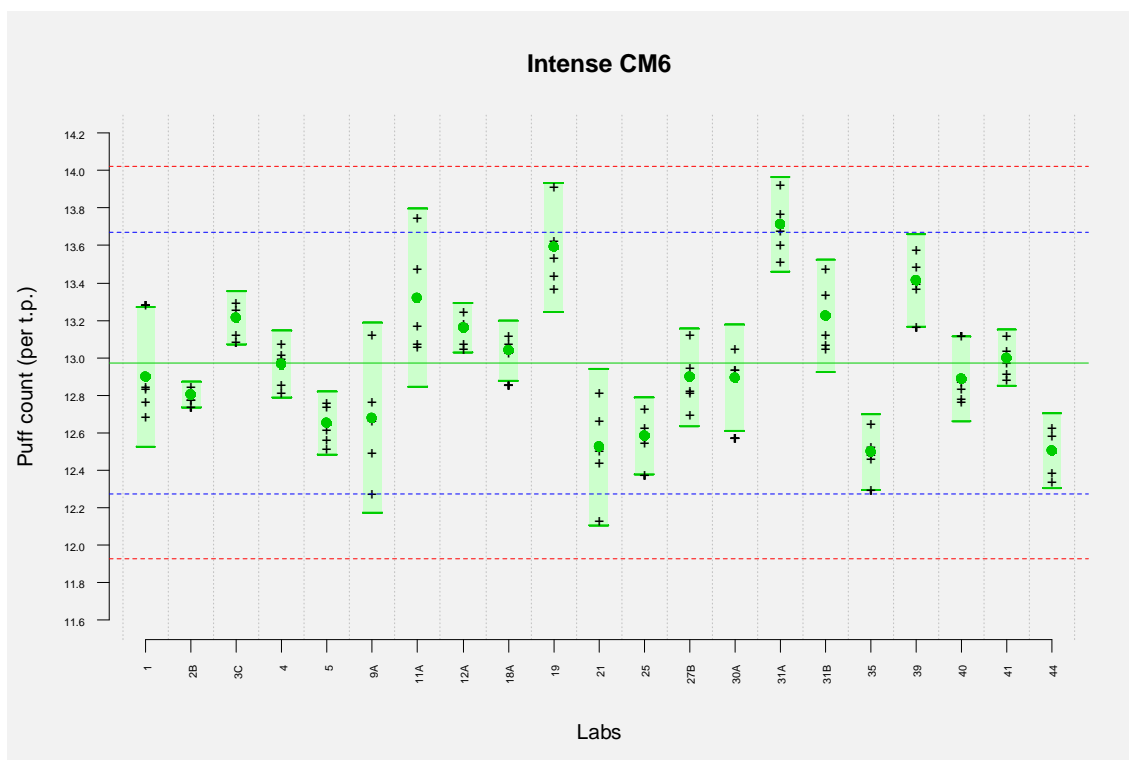


Figure 20: CM6 Individual observations of mainstream “Intense” PUFF Count



2.2. CM7

Figure 21: CM7 Individual observations of mainstream "Intense" TPM

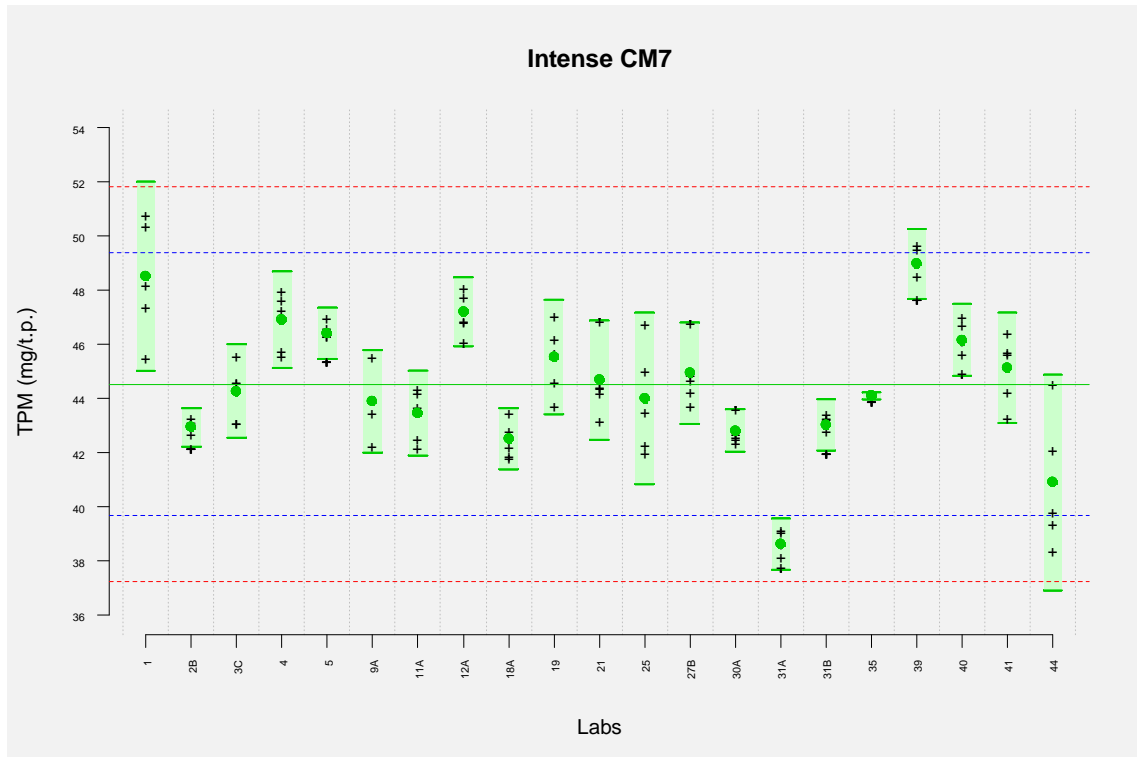


Figure 22: CM7 Individual observations of mainstream "Intense" WATER

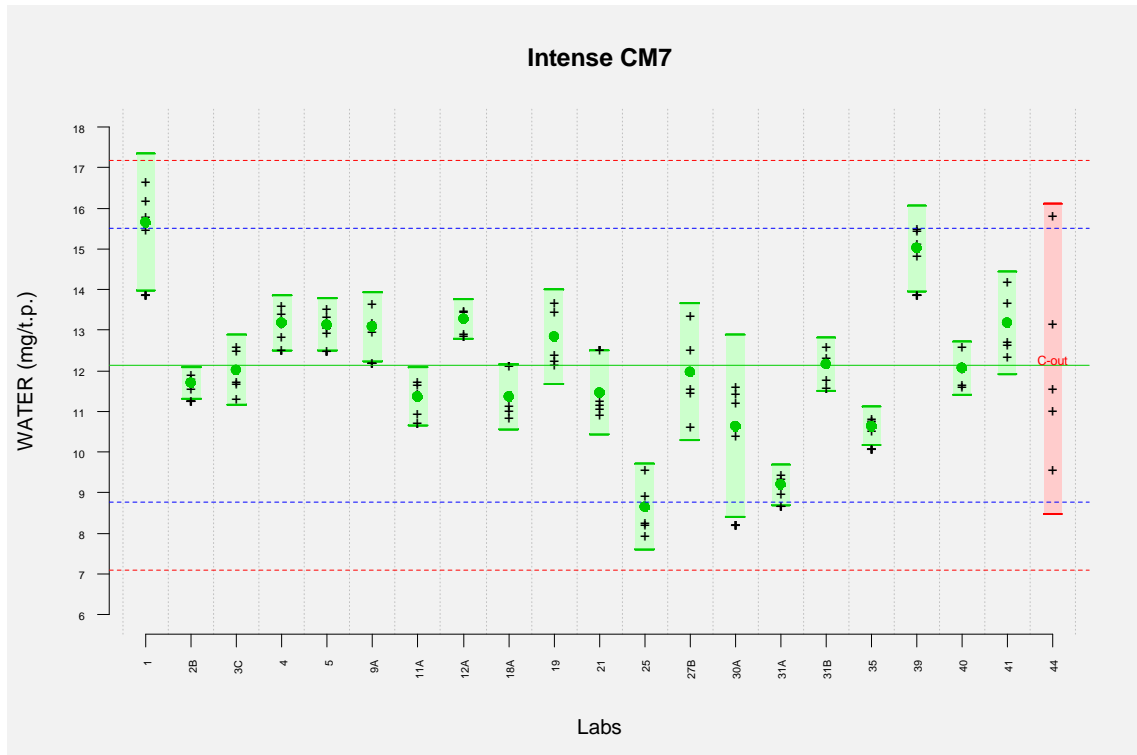


Figure 25: CM7 Individual observations of mainstream "Intense" CO

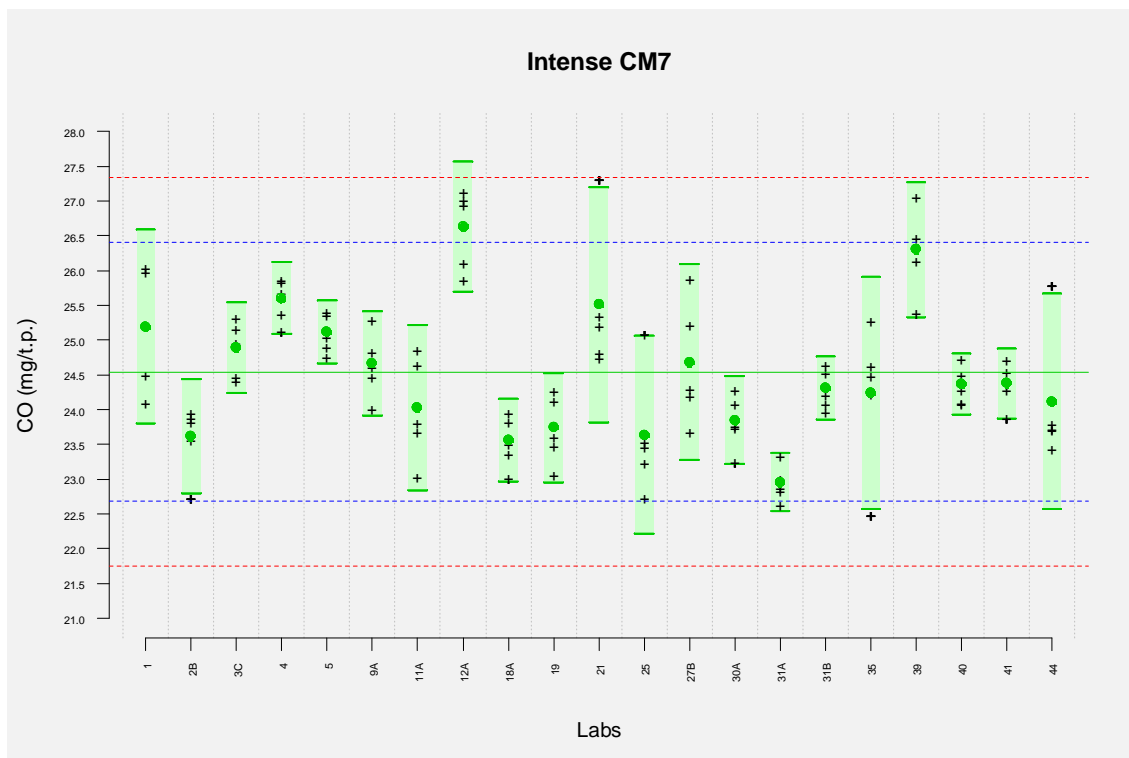


Figure 26: CM7 Individual observations of mainstream "Intense" PUFF Count

