

File
Inv. of gas of low Emission
Black Note book.

Schenectady, July 14, 1954

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SYRACUSE

Enclosed with this letter are the mass spectrometer analyses and experimental data used for the analyses of five 12-inch cathode-ray tubes.

When the tubes were received, they were open to the atmosphere. It was necessary to pump each one of them at least over night in order to get the pressure low enough to start the analyses. The large amount of water vapor present was responsible for this long pump-out period. We were able to speed up the evacuation process to some extent by heating the tube to 100°C while pumping on it.

The purpose of the experiment was to heat the cathode-ray tube in steps to 400°C, measuring the amount of gas given off during each interval and analyzing the gas on a mass spectrometer. The heating steps used were:

- (1) Room temp. to 150°^oC
- (2) 150°^oC for 15 min.
- (3) 150°^oC - 300°^oC
- (4) 300°^oC for 15 min.
- (5) 300°^oC - 400°^oC
- (6) 400°^oC for 15 min

Special heating mantles provided by Syracuse covered the bottom, sides and neck of the tube. An iron-constantan thermocouple placed against the side of the tube was used to measure the temperature. Temperature was recorded at five-minute intervals throughout the run.

Upon removing the heating mantles at the end of the first run, we found that the pyrex tubing connected to the neck of the cathode-ray tube had collapsed almost completely. This indicated that the temperature of the mantle covering the neck of the tube was much higher than that of the mantles covering the sides and bottom of the tube. From then on we inserted a metal thermometer into the mantle covering the neck of the tube, and controlled its temperature separately.

The pressure measurements given for the amount of gas evolved during each heating step are only approximate values. A thermocouple gage was used for the measurements in the micron region and a mercury manometer for those in the millimeter range.

After a run was completed on a tube, the tube was cooled to room temperature and brought up to atmospheric pressure. It was allowed to stand at atmospheric pressure at least over night before a second run was made on it. The second run was carried out in the same manner as the first. Each tube was run twice.

Mr. H. L. McLeland

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In regard to the mass spectra obtained, the results indicate that the gases evolved in heating the tubes consist mainly of hydrogen, water vapor, carbon monoxide, carbon dioxide, nitric oxide, methane, ethylene, and propanal.

The spectra of the gases given off at higher temperatures (from 300°C up) consist of peaks at almost every mass up to 126. From mass 60 up the peaks are quite small and are contributed to by a number of different compounds. This makes it very difficult to identify them all very accurately. However, a good deal of effort was employed in making the best possible identification of these constituents.

There are a very few peaks that were not identified. These are the peaks that occur at masses 93, 94, 95, 96 and 107, 108, 109. However, these are present in extremely small quantities.

In most cases mass 58 has been attributed to propanal. In some cases mass 41 has been attributed to methyl cyanide and mass 27 to hydrogen cyanide; in other cases butene seemed to fit into the analysis at mass 41 and ethylene at mass 27.

We have never run pure hydrogen fluoride in our instrument, and consequently do not have a sensitivity or cracking pattern calibration for it. I have simply indicated its presence in a run. I can assure you, though, that only a fraction of a percent is present in any case.

The mass spectrometer analyses and experimental data are tabulated on separate sheets for each tube.

As I stated before, we have made every attempt to identify all the possible constituents. Perhaps you will have some questions or suggestions after you have looked over our results. We shall be glad to discuss this further with you.

Jane C. Doolittle

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JED:ml

Enclosures

cc: MJ Ozeroff SYRACUSE
C Dichter SYRACUSE
TW Dietze 37-219
ET Connor 37-323
JM Coleman 37-323
CQ Lemmond 37-287

Cathode-Ray Tube C9E2C# - Not Aluminized

	Run I						Run II					
	Rm Temp-	150°C 150°C	150°C- 300°C 300°C	300°C- 400°C 400°C	400°C 15 min.		Rm Temp-	150°C 150°C	150°C- 300°C 300°C	300°C- 400°C 400°C	400°C 15 min.	
Toluene			0.01	0.04	0.03							
1-Hexyne			0.1	0.1	0.06							
Benzene			0.01	0.01								
n-Pentane				0.07								
Pentene-2				0.02	0.04							
n-Butane			0.02		0.06	0.02					0.04	0.1
Butene-2		0.01	0.07	0.03		0.01					0.03	0.06
Ethyl Alcohol	0.01	0.05	0.04	0.01	0.01	Trace						
Carbon Dioxide	9.8	8.4	12.3	13.5	16.6	18.9	7.6	17.5	33.6	37.9	26.2	22.0
Propane	0.03	0.02		0.05	0.09	0.08			0.03	0.04	0.02	
Methyl Cyanide				0.4	0.6	0.4						
Nitric Oxide	0.05	0.02		0.1	0.04	0.03	0.06	0.1	0.09	0.04	0.02	Trace
Carbon Monoxide	0.06	0.02		0.7	1.7	3.1	0.2	0.1	0.08	0.2	0.5	1.7
Ethylene	0.01	0.01	0.3				Trace			0.03	0.3	0.5
Hydrogen Cyanide				0.6	1.3	1.0						
Hydrogen Fluoride	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present
Water	89.9	91.5	87.0	83.8	77.4	70.5	92.0	82.2	66.2	61.7	72.3	72.6
Methane	0.02	0.02	0.07	0.2	0.3	0.5					0.09	0.3
Hydrogen	0.09	0.1	0.2	0.4	1.9	5.4	0.1	0.09	0.09	0.1	0.6	2.8

Cathode-Ray Tube C9E2C# - Not Aluminized

1st Run - March 12, 1954

2nd Run - March 18, 1954

Time	Potentiometer Reading (mv x5)	Temp. °C	Pressure	Time	Potentiometer Reading (mv x5)	Temp. °C.	Pressure
1:30	0.26	26		9:55	0.25	24	
1:35	0.81	78		10:00	0.78	74	
1:40	1.40	132	140 μ	10:05	1.34	126	
1:45	1.42	133		10:10	1.68	157	160 μ
1:50	1.63	153		10:15	1.68	157	
1:55	1.63	153		10:20	1.68	157	
2:00	1.63	153	175 μ	10:25	1.65	154	
2:05	1.60	150		10:30	1.61	151	155 μ
2:10	1.57	147		10:35	1.56	146	
2:15	1.53	144		10:40	1.70	159	
2:20	1.90	177		10:45	2.12	197	
2:27	2.58	238		10:50	2.67	246	
2:30	2.95	271		10:55	3.20	294	180 μ
2:35	3.10	285	4.5 mm	11:00	3.24	298	
2:40	3.06	282		11:05	3.17	291	
2:45	2.99	275		11:10	3.33	306	
2:50	3.18	292		11:15	3.25	299	155 μ
2:55	3.08	283		11:20	3.13	288	
3:00	3.18	292		11:25	3.42	314	
3:05	3.09	284	160 μ	11:30	3.83	351	
3:10	3.00	276		11:35	4.26	390	
3:15	3.22	296		11:40	4.30	394	160 μ
3:20	3.69	339		11:45	4.22	386	
3:25	4.21	385		11:50	4.31	395	
3:30	4.20	384	1 mm.	11:55	4.33	396	
3:35	4.09	375		11:60	4.26	390	140 μ
3:40	4.32	396					
3:45	4.28	392					
3:50	4.28	392	160 μ				

Cathode-Ray Tube C9E3C# - Not Aluminized

	Run I						Run II						
	Rm Temp	150°C- 150°C 15 min.	150°C- 300°C	300°C- 7 min.	300°C- 400°C	400°C 15 min.		Rm Temp	150°C- 150°C 15 min.	150°C- 300°C	300°C 15 min.	300°C- 400°C	400°C 15 min.
Toluene				Trace	0.02	0.02							Trace
Spiropentane			0.03	0.1	0.2	0.07							
n-Butane	0.02	Trace	0.02	0.04	0.09	0.2		0.01	0.01	0.01	0.02	0.03	0.05
Butene-2								Trace	Trace	0.01	0.02	0.03	0.04
Butadiene				Trace	0.08								
Ethyl Alcohol	Trace			Trace	Trace			0.02	0.02	0.01	0.01		
Carbon Dioxide	4.0	7.4	17.3	17.5	18.7	19.8		11.4	17.4	28.9	29.2	24.3	23.6
Propane					0.05			0.01	0.02	0.03	0.03	0.02	
Propylene												0.03	0.04
Methyl Cyanide			0.07	0.3	0.6	0.3							
Nitric Oxide	0.03	0.01	0.05	0.05	0.03	0.03		0.1	0.1	0.07	0.03	0.01	0.01
Carbon Monoxide	0.06	0.05	0.1	0.6	1.7	3.1		0.2	0.2	0.1	0.1	0.2	0.7
Ethylene	Trace							0.02	0.01	0.04	0.07	0.4	0.6
Hydrogen Cyanide			0.2	0.7	1.6	0.9							
Hydrogen Fluoride	Present	Present	Present	Present	Present	Present		Present	Present	Present	Present	Present	Present
Water	95.7	92.4	82.0	79.9	74.1	67.3		88.2	82.2	70.7	70.4	74.5	73.0
Methane	Trace	Trace	0.06	0.2	0.3	0.5						0.07	0.2
Hydrogen	0.1	0.1	0.2	0.6	2.5	7.8		0.1	0.09	0.08	0.1	0.5	1.8

Cathode-Ray Tube C9E3C# - Not Aluminized

1st Run - March 19, 1954

2nd Run - March 24, 1954

Time	Potentiometer Reading (mv x5)	Temp. ^o C.	Pressure	Time	Potentiometer Reading (mv x5)	Temp. ^o C.	Pressure
10:20	0.80	77		9:40	0.26	26	
10:25	1.25	118		9:45	0.72	69	
10:30	1.65	154	5 mm.	9:50	1.27	120	
10:35	1.60	150		9:55	1.74	163	150 μ
10:40	1.60	150		10:00	1.78	166	
10:45	1.55	145		10:05	1.78	166	
10:50	1.65	154	160 μ	10:10	1.77	165	150 μ
10:55	1.60	150		10:15	1.91	178	
11:00	1.65	154		10:20	2.49	230	
11:05				10:25	3.03	279	
11:10	3.25	299	4 mm.	10:30	3.31	304	170 μ
11:15	3.30	303		10:35	3.26	299	
11:20	3.15	290		10:40	3.26	299	
11:25	3.20	294		10:45	3.23	297	160 μ
11:30	3.25	299		10:50	3.16	291	
11:35	3.20	294	150 μ	10:55	3.50	321	
11:40 } *	3.15	290		11:00	4.03	369	
11:45 } *	3.25	299	125 μ	11:05	4.37	400	160 μ
11:55	3.30	303		11:10	4.32	396	
12:00	3.65	335		11:15	4.38	401	
12:05	4.00	367		11:20	4.24	388	
12:10	4.35	398	200 μ	11:25	4.37	400	160 μ
12:15	4.27	391					
12:20	4.21	385					
12:25	4.40	403					
12:30	4.24	388	155 μ				

* Sample for 300^oC run was accidentally pumped away. So the temperature was held at 300^oC for an additional 7 or 8 minutes, and then analyzed on the spectrometer.

Cathode-Ray Tube C9E4C# - Aluminized

	Run I						Run II					
	Rm Temp- 150°C	150°C 15 min.	150°C- 300°C	300°C 15 min.	300°C- 400°C	400°C 15 min.	Rm Temp- 150°C	150°C 15 min.	150°C- 300°C	300°C 15 min.	300°C- 400°C	400°C 15 min.
1-Nonene				0.03								
Isopropyl Benzene				Trace	0.01	0.03						
n-Octane				0.1								
1-Octene		0.08		0.1	0.2	0.2						
4-Octyne				0.04	0.06	0.06						
Ethylbenzene				Trace	Trace	0.07						
2,2-Dimethylpentane			0.02		0.04	0.04						
1-Heptene			0.06	0.2	0.3	0.2						
2,3,3-Trimethyl-1-butene			0.01									
1-Heptyne				0.01		0.09						
Toluene				0.02	0.06	0.03						
n-Hexane			0.07			0.3					0.01	0.03
1-Hexene			0.09			0.08						
4-Methyl-cis-2-pentene											Trace	
Cyclohexene		0.01	0.04	0.04	0.09	0.1					0.01	0.03
1,3-Cyclohexadiene				0.03	0.08	0.1						
Benzene					0.04	0.1						
Tert. Butyl Alcohol				0.2							0.02	0.04
n-Pentane			0.2	0.4	0.4	1.0						
Neopentane	0.06						Trace	Trace	Trace		0.06	0.08
Isopentane	0.05											
Isopropyl Alcohol				0.1								
Propanal			1.1	9.0	13.8	10.4	0.07	0.07	0.20	0.4	1.2	0.2
n-Butane	0.03	0.07	0.02		0.5	0.2	0.02	0.02	0.03	0.1	0.1	0.2
Butene-2	0.02						Trace	Trace	0.02	0.03	0.08	0.1
Ethyl Alcohol	0.01	0.04	0.09	0.4	0.5	0.1	0.01	0.02	0.03	0.03	0.03	0.03
Carbon Dioxide	2.3	11.3	24.4	21.7	18.2	18.1	13.5	17.8	31.8	34.3	33.9	30.4
Propane		0.06	0.3		0.7				0.02	0.04	0.05	0.07
Propylene					1.1							
Methyl Cyanide				0.2		0.7						
Air	13.8											
Oxygen			0.02	0.04	0.05	0.08	0.05	0.02		0.01	Trace	0.02
Nitric Oxide	0.02	2.3	4.6	1.2	0.2	0.2	0.2	0.3	0.1	0.02	0.02	0.06
Carbon Monoxide	0.3	0.8	1.7	1.2	2.2	5.3	0.4	0.4	0.5	1.0	2.6	
Ethylene	Trace		0.8	1.2	2.0	2.7		Trace	0.05	0.6	0.9	
Hydrogen Cyanide												
Hydrogen Fluoride	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present
Water	83.1	83.8	64.6	59.0	50.4	40.8	85.8	81.4	67.2	63.7	59.2	52.6
Methane	0.02	0.08	0.06	0.7	1.5	2.4			0.04	0.08	0.3	0.8
Hydrogen	0.1	1.5	1.8	3.9	7.5	16.4	0.1	0.1	0.2	0.9	3.4	11.8
trans-2-Pentene									Trace	0.04		.06

Cathode-Ray Tube C9E4C# - Aluminized

1st Run - March 17, 1954

2nd Run - March 23, 1954

Time	Potentiometer Reading (mv x5)	Temp. ^o C	Pressure	Time	Potentiometer Reading (mv x5)	Temp. ^o C	Pressure
9:50	0.25	24		9:35	1.04	99	
9:55	0.95	90		9:40	1.50	141	170 μ
10:00	1.50	141	200 μ	9:45	1.68	157	
10:05	1.62	152		9:50	1.70	159	
10:10	1.40	132		9:55	1.71	160	
10:15	1.70	159		10:00	1.68	157	160 μ
10:20	1.60	150		10:05	1.81	169	
10:25	1.55	145	200 μ	10:10	2.45	226	
*				10:15	2.88	265	
10:45				10:20	3.23	297	170 μ
10:50	1.95	181		10:25	3.24	298	
10:55	1.80	168		10:30	3.23	297	
11:00	1.75	164		10:35	3.25	299	150 μ
11:05	1.80	168		10:40	3.37	309	
11:10	2.55	235		10:45	3.78	347	
11:15	3.10	285		10:50	4.16	381	
11:20	3.26	299	200 μ	10:55	4.38	401	160 μ
11:25	3.10	285		11:00	4.33	396	
11:30	3.30	303		11:05	4.37	400	
11:35	3.20	294		11:10	4.30	394	150 μ
11:40	3.30	303	200 μ				
11:45	3.20	294					
11:50	3.55	326					
11:55	4.08	374					
12:00	4.38	401	200 μ				
12:05	4.28	392					
12:10	4.27	391					
12:15	4.27	391					
12:20	4.32	396	190 μ				

* Mercury diffusion pump on sample system failed temporarily.

Cathode-Ray Tube C9E6C# - Aluminized

	Run I						Run II					
	Rm Temp-	150°C 150°C	150°C- 300°C	300°C 15 min.	300°C- 400°C	400°C 15 min.	Rm.Temp-	150°C 150°C	150°C- 300°C	300°C 15 min.	300°C- 400°C	400°C 15 min.
Isopropyl Benzene					0.01							
n-Octane				0.03	0.01							
1-Octene					0.1	0.1						
4-Octyne					0.04							
Ethyl Benzene					Trace							
1-Heptene				0.2	0.2							
1-Heptyne					0.04							
Toluene			Trace	0.02	0.03	0.1						0.03
n-Hexane			0.02	0.07	0.1	0.3						
1-Hexene				0.04								
4-Methyl-cis-2-pentene	Trace	Trace	Trace									
Cyclohexene	Trace	Trace	0.02	0.06	0.08	0.2						
1,3-Cyclohexadiene				0.03	0.08							
Benzene					0.03							0.04
Tert. Butyl Alcohol				0.2								
n-Pentane	0.04	0.04	0.09	0.6	0.6	0.9						0.2
Neopentane	0.01	0.02		0.04								
cis-2-Pentene				0.03								
Spiropentane												
1-Pentyne												0.06
Propanal	0.02	0.08	0.9	11.3	13.8	10.2						1.4
n-Butane			0.1		0.6	0.6						0.2
Butene-2	Trace		0.06									
Ethyl Alcohol	0.01	0.01	0.08	0.6	0.4		0.01	0.02	0.02	0.03	0.04	0.03
Carbon Dioxide	12.0	14.2	26.9	17.8	17.3	15.9	11.2	17.8	29.6	29.9	31.6	31.4
Propane	0.04	0.07	0.2	0.2	0.1	1.2	0.01	0.02	0.08	0.1	0.4	0.1
Propylene										0.03	0.1	
Methyl Cyanide			0.09	0.4		0.8						
Oxygen		Trace	0.03	0.06				0.05	0.01	Trace	0.02	0.01
Nitric Oxide	0.1	1.1	4.2	0.5	0.1		0.2	0.3	0.1	0.04	0.04	0.06
Air						3.2						
Carbon Monoxide	0.1	0.2	2.4	2.8	2.8	5.1	0.1	0.9	0.6	0.9	1.2	3.6
Ethylene	0.04	0.06			2.3	2.4	0.01	0.01	0.04	0.1	0.5	0.9
Hydrogen Cyanide			0.3	0.5								
Hydrogen Fluoride	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present
Water	87.3	82.9	63.0	60.0	51.8	42.0	84.9	80.8	69.3	68.3	64.9	60.9
Methane	0.03	0.03	0.06	0.8	1.2	1.8				0.1	0.3	0.8
Hydrogen	0.2	1.4		3.7	7.2	19.6	0.3	0.2	0.2	0.2	0.2	0.2

Cathode-Ray Tube C9E6C# - Aluminized

1st Run - March 16, 1954

2nd Run - March 22, 1954

Time	Potentiometer Reading (mv x5)	Temp. °C	Pressure	Time	Potentiometer Reading (mv x5)	Temp. °C	Pressure
11:05	0.61	59		9:45	0.42	41	
11:10	1.60	150		9:50	1.21	114	
11:15	1.60	150	125 μ	9:55	1.59	149	160 μ
11:20	1.43	134		10:00	1.68	157	
11:25	1.50	141		10:05	1.60	150	
11:30	1.60	150		10:10	1.54	144	
11:35	1.55	145	125 μ	10:15	1.69	158	170 μ
11:40	1.55	145		10:20	2.56	236	
11:45	2.05	190		10:25	2.87	264	
11:50	2.50	231		10:30	3.28	301	180 μ
11:55	3.07	282		10:35	3.18	292	
12:00	3.62	332	190 μ	10:40	3.23	297	
12:05	3.22	296		10:45	3.31	304	
12:10	3.40	312		10:50	3.20	294	160 μ
12:15	3.30	303		10:55	3.28	301	
12:20	3.26	299	150 μ	11:00	3.75	344	
12:25	3.17	291		11:05	4.20	384	
12:30	3.60	331		11:10	4.22	386	160 μ
12:35	3.95	362		11:15	4.37	400	
12:40	4.35	398	175 μ	11:20	4.36	399	
12:45	4.25	389		11:25	4.28	392	
12:50	4.40	403		11:30	4.30	394	170 μ
12:55	4.20	384					
1:00	4.10	376	150 μ				

Cathode-Ray Tube Cl5E1C# - Aluminized

	Run I						Run II					
	Rm Temp-	150°C 150°C	150°C- 300°C 300°C	300°C- 400°C 400°C	400°C 15 min.		Rm Temp-	150°C 150°C	150°C- 300°C 300°C	300°C- 400°C 400°C	400°C 15 min.	
Isopropyl Benzene				Trace	0.01							
n-Octane			0.1	0.02								
4-Octyne			0.02	0.02	0.02							
Ethylbenzene				Trace	Trace							
1-Heptene			0.2	0.2	0.1							
Toluene		Trace	Trace	Trace	0.03	0.05						0.04
n-Hexane					0.08	0.2						
1-Hexene					0.07	0.09						
4-methyl-cis-2-pentene			0.01									
Cyclohexene			0.03	0.05	0.07	0.09						
1,3-Cyclohexadiene						0.06						0.04
Benzene						0.03						0.04
n-Pentane			0.09	0.3	0.5	0.6						
Neopentane	Trace	0.01	0.02									0.1
cis-2-pentene			0.04		0.05	0.1						0.2
trans-2-pentene		0.02										
1-Pentene												
Propanal												
n-Butane	0.02	0.06	0.8	5.9	11.4	9.5						0.03
Butene-1	Trace	Trace	0.2	0.2	0.5	0.6	0.02	0.03	0.3	0.9	1.9	0.06
Ethyl Alcohol	Trace	0.02	0.07	0.3	0.5	0.2	0.02	0.02	0.06	0.1	0.2	2.6
Propane	-	0.03	0.05	0.3	0.1	0.4	0.03	0.03	0.03	0.08	0.08	0.4
Carbon Dioxide	6.8	10.1	21.5	23.0	20.2	19.9	9.0	15.3	26.2	22.4	21.0	0.03
Propylene					1.1	0.7			0.03	0.09	0.1	0.03
Methyl Cyanide				0.2		0.5						
Oxygen				0.01	0.06	0.07	0.06					
Nitric Oxide	0.09	1.2	6.3	2.7	0.3	0.2	0.02	0.01	0.02	0.02	0.04	Trace
Ethylene	Trace	0.05	0.7	1.0	1.8	2.1	0.03	0.03	0.04	0.1	1.4	0.08
Carbon Monoxide		0.3	2.4	2.8	5.0	7.6	0.1	0.08	0.3	0.4	1.1	2.7
Hydrogen Fluoride	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present
Water	92.7	86.9	65.7	59.5	50.7	42.9	90.6	84.5	73.2	75.4	70.1	58.3
Methane	0.02		0.04	0.6	1.0	1.5			0.04	0.1	0.4	1.0
Hydrogen	0.3	1.3	1.6	3.0	6.0	12.8	0.1	0.08	0.2	0.5	4.0	12.5

Cathode-Ray Tube C15ELC# - Aluminized

1st Run - March 25, 1954

2nd Run - March 26, 1954

Time	Potentiometer Reading (mv x5)	Temp. °C	Pressure	Time	Potentiometer Reading (mv x5)	Temp. °C	Pressure
8:45	0.89	85		9:25	0.60	58	
8:50	1.46	137	160 μ	9:30	1.31	124	
8:55	1.63	152		9:35	1.61	151	125 μ
9:00	1.63	152		9:40	1.70	159	
9:05	1.62	152		9:45	1.53	144	
9:10	1.60	150	160 μ	9:50	1.82	170	140 μ
9:15	2.01	187		9:55	2.31	214	
9:20	2.90	267		10:00	2.97	273	
9:25	3.20	294	180 μ	10:05	3.24	298	150 μ
9:30	3.26	299		10:10	3.29	302	
9:35	3.22	296		10:15	3.18	292	
9:40	3.32	305		10:20	3.26	299	
9:45	3.28	301	180 μ	10:25	3.12	287	160 μ
9:50	3.52	323		10:30	3.52	323	
9:55	3.79	348		10:35	3.99	366	
10:00	4.32	396		10:40	4.32	396	160 μ
10:05	4.30	394	180 μ	10:45	4.21	385	
10:10	4.34	397		10:50	4.25	389	
10:15	4.35	398		10:55	4.40	403	
10:20	4.38	401		11:00	4.30	394	160 μ
10:25	4.32	401	200 μ				