



# Quench correction in the analysis of Organic Bonded Tritium (OBT) in biota samples

CHEN Qianyuan  
[colinsunny@163.com](mailto:colinsunny@163.com)



# Contents

- ▶ **Background**
- ▶ **Experiment**
- ▶ **Results**

# Background-Typical OBT analysis procedure



## procedure

- ▶ **Biota samples** in the ambient of an operated Nuclear Power Plant were collected, after being cleaned up and **dried** to eternal weight under the temperature of around 105 centigrade, the dried samples were **smashed** into powders, place approximately 8 grams (For fish and meat, 2 grams) of sample powder evenly into each **silica combustion boat**.



Biota sample



Dryer



Grinder



Silica boat

# Background–Typical OBT analysis



## procedure

- ▶ The totally six silica boats were inserted separately into the silica combustion tubes of the **Pyrolyser-6 Tritium&Carbon-14 extraction apparatus**, run the preset combustion program, OBT contained in the biota sample should be oxidized as tritiated water(HTO) and trapped inside of the **Coolsafe cold trap** under  $-110$  centigrade.

Combustion apparatus



Cooling Unit





# Background—Typical OBT analysis procedure



## procedure

- ▶ OBT in biota samples can be oxidized by ignition under 20 atmospheric pressure of oxygen.

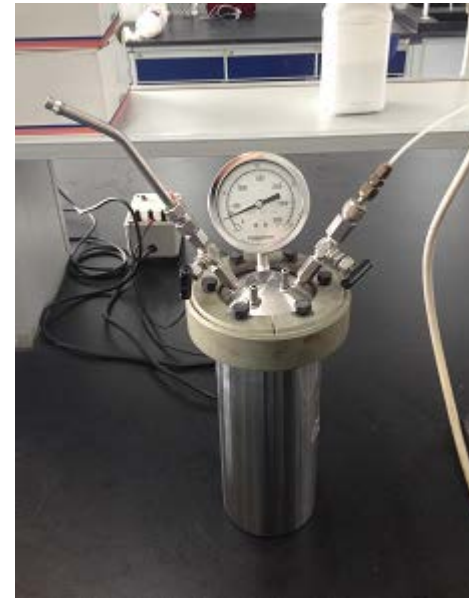
**Sample container**



**Oxygen**



**Parr oxygen bomb**



**Ignition unit**



# Background-collected HTO weight

- ▶ The sample combustion rate is calculated as follow:

$$Y_c = \frac{W_w}{9 \times W_d \times P_H}$$

$P_H$  – element content of hydrogen in biota sample, which is measured by an Elemental Analyzer

$W_w$  – collected water(H<sub>2</sub>O) weight

$W_d$  – combusted sample weight (dry)

Euro EA3000  
Elemental  
Analyzer



**Table 1 collected HTO weight and combustion rate**

Sample	combusted sample weight ( $W_d$ , g)	collected water(H <sub>2</sub> O) weight ( $W_w$ , g)	element content of hydrogen ( $P_H$ , %)	Sample combustion rate ( $Y_C$ , %)
Radish	48.0154	25.5891	6.52	90.8
Green vegetable	48.0650	24.9173	6.5	88.6
rice	48.0358	28.3154	7.19	91.1
Pine needle	42.0015	24.9153	7.59	86.8
mutton	12.0021	7.914	8.48	86.4

# Background-measurement

- ▶ Under the **normal pretreatment process**, the trapped water(HTO) should be distilled before being counted in the LSC, however, as shown in **Table 1**, after the combustion process, only less than 30 grams of water was collected, which is pretty hard to be distilled.
- ▶ Therefore, 10 grams of melted HTO was weighted into a polyethylene scintillation vial and **mixed directly** with 10 mL of **UltimaGold LLT cocktail** after fierce shaking. The sample was finally measured in a **Quantulus 1220 Liquid Scintillation Counter**.





# Background-Typical OBT analysis



## procedure

- ▶ Due to the impurity of the collected HTO, Quench index which shown as **SQP(E)** is tend to be differ from one sample to another, which means that a quench correction was required.

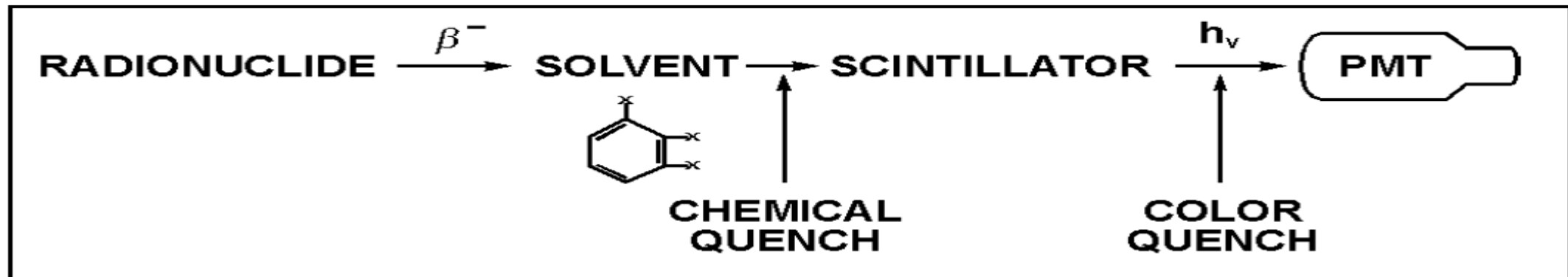
	CTIME	DTIME1	DTIME2	CUCNTS	SQP	SQP%	STIME	ID
1	100:01.774	132.417	132.592	295	608.5	0.31	1:01	XJWDM
1	100:01.774	132.419	132.598	200	683.24	0.33	1:02	XJWDM1
1	100:01.774	132.436	132.581	869	686.98	0.26	1:02	XJWDMOE
1	100:01.768	132.365	132.09	225	615.61	0.21	1:02	HZDM
1	100:01.774	132.328	132.167	195	651.84	0.32	1:02	HZDM1
1	100:01.774	132.361	132.182	397	697.99	0.17	1:02	HZDMOB
1	100:01.767	132.743	132.611	4256	687.97	0.28	1:02	XJWYCOE
1	100:01.773	132.702	132.641	3397	688.55	0.21	1:02	HZQCOB
1	100:01.773	132.457	132.636	345	589.33	0.36	1:02	XJWCY
1	100:01.770	132.607	132.68	2702	609.97	0.27	1:02	XJWCYOE
1	100:01.773	132.54	132.591	3621	560.85	0.38	1:02	XJWSZ
1	100:01.774	132.415	132.596	553	0	0	0:00	XJWDM
1	100:01.768	132.303	132.146	384	0	0	0:00	XJWDM1
1	100:01.774	132.439	132.589	1551	0	0	0:00	XJWDMOE
1	100:01.774	132.425	132.605	440	0	0	0:00	HZDM
1	100:01.774	132.433	132.611	377	0	0	0:00	HZDM1
1	100:01.773	132.439	132.6	772	0	0	0:00	HZDMOB
1	100:01.774	132.64	132.188	8408	0	0	0:00	XJWYCOE
1	100:01.773	132.687	132.621	6631	0	0	0:00	HZQCOB
1	100:01.767	132.44	132.621	568	0	0	0:00	XJWCY
1	100:01.773	132.457	132.635	560	0	0	0:00	XJWCY1
1	100:01.767	132.554	132.593	5317	0	0	0:00	XJWCYOE
1	100:01.773	132.556	132.604	7301	0	0	0:00	XJWSZ
1	100:01.774	132.474	132.651	809	605.57	0.31	1:02	XJWDM
1	100:01.774	132.444	132.626	565	682.14	0.41	1:02	XJWDM1
1	100:01.774	132.446	132.599	2252	685.2	0.22	1:02	XJWDMOE
1	100:01.768	132.371	132.086	617	615.53	0.24	1:02	HZDM
1	100:01.774	132.323	132.158	559	652.45	0.23	1:02	HZDM1
1	100:01.774	132.341	132.167	1115	701.76	0.2	1:02	HZDMOB
1	27:01.888	35.752	35.712	615	598.77	0.44	1:02	XJWCY



# Experiment–Quench principle

Any factor which reduces the efficiency of the energy transfer or causes the absorption of photons results in quenching in the sample.

Two main types of quench: **chemical quench** and **color quench**.



# Experiment–Quench curve preparation



A quench standard curve is a series of standards in which the absolute radioactivity (DPM) per vial is constant and the amount of quench increases from vial to vial. The quench is increased from vial to vial by the addition of a quenching indicator (quenching agent).

$\text{CCl}_4$ ,  $\text{CH}_3\text{NO}_2$  and  $\text{HNO}_3$ , were chosen to be the tested quenching indicator.

# Experiment–Quench curve preparation



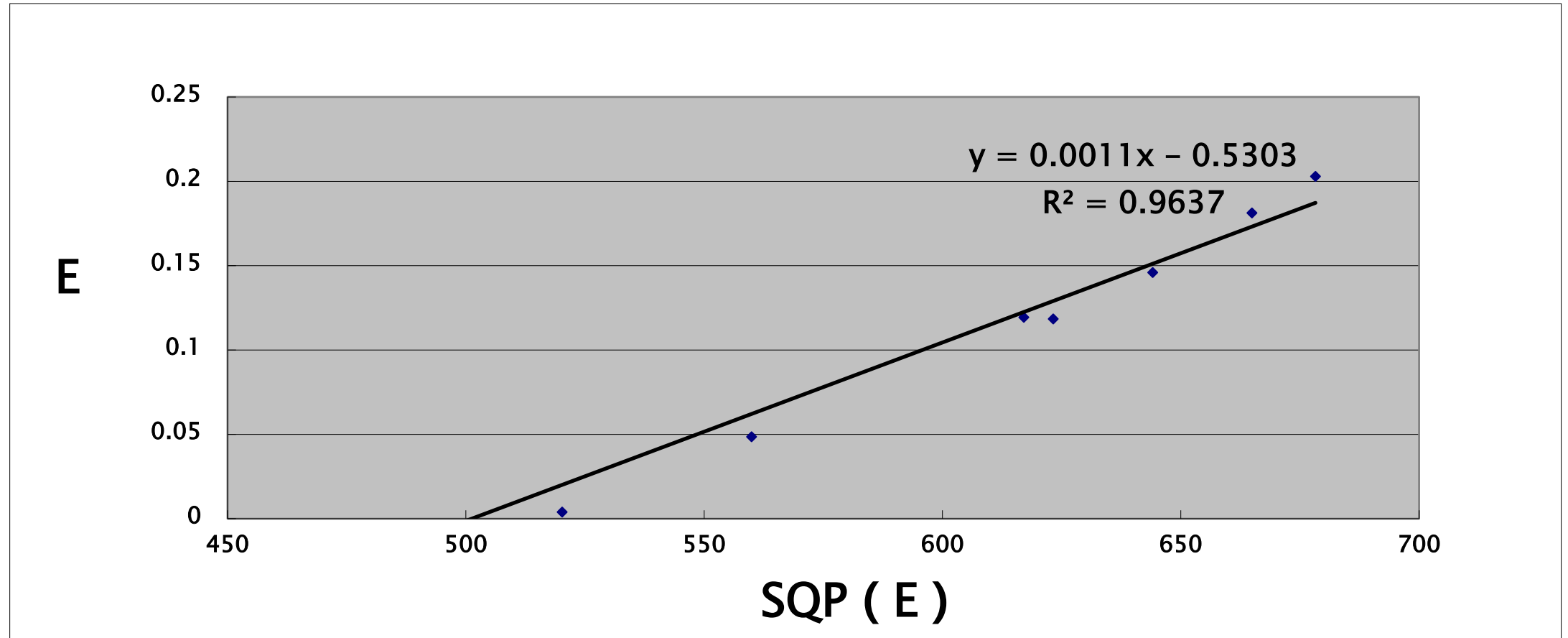
Exp. results use  $\text{CH}_3\text{NO}_2$  as quenching indicator.

Quench indicator Nitromethane ( $\text{CH}_3\text{NO}_2$ )						
No.	volumn added ( $\mu\text{L}$ )	weight (g)	cpm	SQP (E)	activity concentration (Bq/L)	Efficiency
<b>Background</b>						
1	10	10	0.871	677.8		
2	20	10	1.053	660.3		
3	30	10	0.932	647.6		
4	40	10	0.942	623.5		
5	50	10	0.866	616.6		
6	100	9.9	0.962	559.1		
7	200	9.8	1.083	516.7		
8	500	9.7	0.729	481.6		
<b>Standard</b>						
1	10	10	8.323	678.3	61.2	0.203
2	20	10	7.71	664.9	61.2	0.181
3	30	10	6.292	644.1	61.2	0.146
4	40	10	5.291	623.2	61.2	0.118
5	50	10	5.25	617.1	61.2	0.119
6	100	9.9	2.729	560	61.2	0.049
7	200	9.8	1.225	520.2	61.2	0.004
8	500	9.7	0.719	477.4	61.2	0

# Experiment–Quench curve preparation



Quench curve made by  $\text{CH}_3\text{NO}_2$  as quenching indicator.





# Experiment–Quench curve preparation



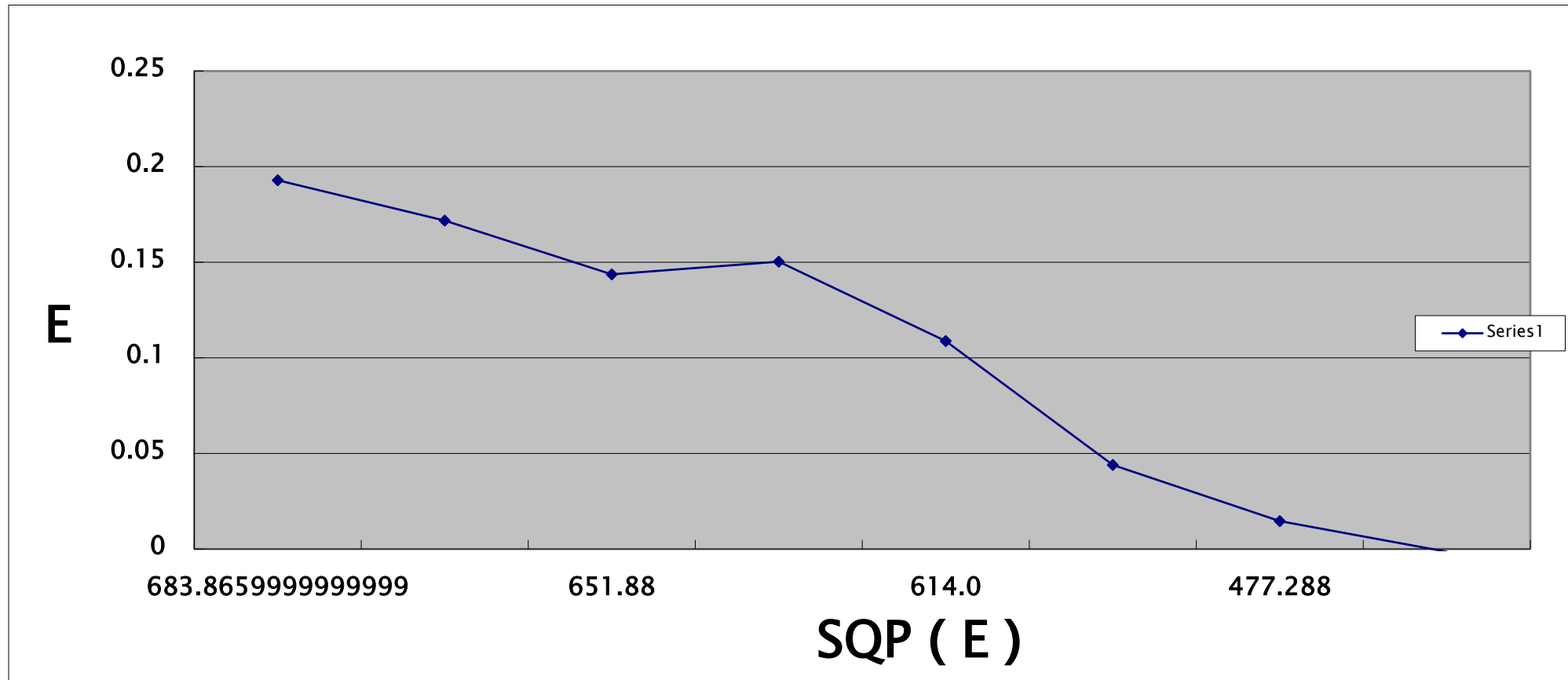
Exp. results use **CCl<sub>4</sub>** as quenching indicator.

Quench indicator <b>Tetrachloride (CCl<sub>4</sub>)</b>						
No.	volumn added (μL)	weight (g)	cpm	<b>SQP (E)</b>	activity concentration (Bq/L)	<b>Efficiency</b>
<b>Background</b>						
1	10	10	1.114	<b>695.3</b>		
2	20	10	1.119	<b>692.1</b>		
3	30	10	1.144	<b>668.9</b>		
4	40	10	1.164	<b>650.9</b>		
5	50	10	1.261	<b>633.7</b>		
6	100	9.9	1.21	<b>567.5</b>		
7	200	9.8	1.311	<b>478.7</b>		
8	500	9.7	1.453	<b>420.9</b>		
<b>Standard</b>						
1	10	10	8.197	<b>683.9</b>	61.2	<b>0.193</b>
2	20	10	7.427	<b>662.7</b>	61.2	<b>0.172</b>
3	30	10	6.42	<b>651.9</b>	61.2	<b>0.144</b>
4	40	10	6.683	<b>650.4</b>	61.2	<b>0.15</b>
5	50	10	5.255	<b>614</b>	61.2	<b>0.109</b>
6	100	9.9	2.81	<b>554.2</b>	61.2	<b>0.044</b>
7	200	9.8	1.838	<b>477.3</b>	61.2	<b>0.015</b>
8	500	9.7	1.403	<b>401.8</b>	61.2	<b>-0.001</b>

# Experiment–Quench curve preparation



Quench curve made by  $\text{CCl}_4$  as quenching indicator.



# Experiment–Quench curve preparation



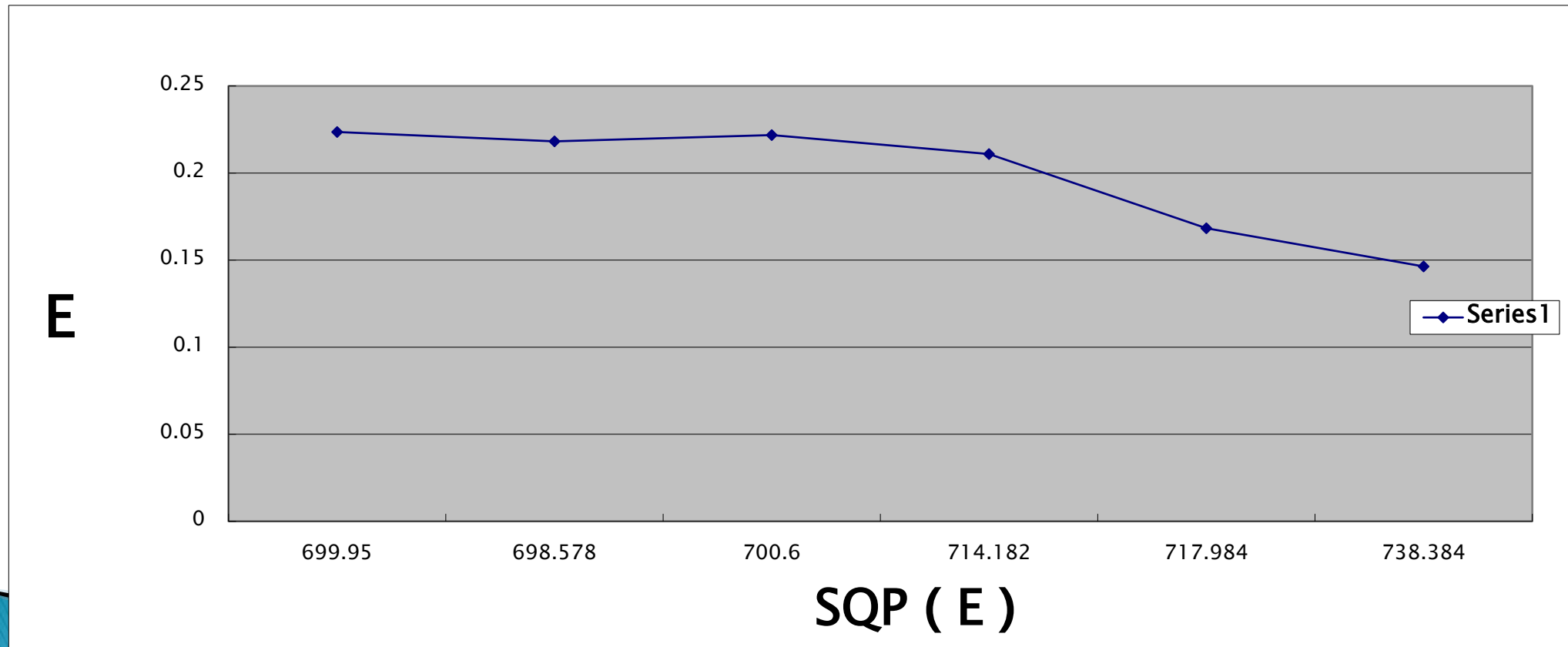
Exp. results use **HNO<sub>3</sub>** as quenching indicator.

Quench indicator <b>Nitric Acid (HNO<sub>3</sub>)</b>						
No.	volumn added(μL)	weight(g)	cpm	SQP (E)	activity concentration(Bq/L)	Efficiency
<b>Background</b>						
1	10	10	0.987	696.4		
2	30	10	0.901	700.2		
3	50	10	0.921	703.5		
4	100	10	0.835	714.2		
5	200	10	1.043	723.8		
6	500	9.9	0.982	738.2		
<b>Standard</b>						
1	10	10	9.198	700	61.2	0.224
2	30	10	8.915	698.6	61.2	0.218
3	50	10	9.067	700.6	61.2	0.222
4	100	10	8.581	714.2	61.2	0.211
5	200	10	7.224	718	61.2	0.168
6	500	9.9	6.305	738.4	61.2	0.146

# Experiment-Quench curve preparation



- ▶ Then, different weights of reagents were added into the HTO reference & background sample and measured





# Result



Quench curves selection:

- ✓ Covers the SQP(E) range of the OBT samples of our lab best was chosen.
- ✓ Has a good linear shape.

Therefore,  $\text{CH}_3\text{NO}_2$  was chosen to be the quenching indicator in the measurement of OBT in biota samples.

# Result

SOP(E) value could be checked out in the data output file of Easyview software, therefore, the correspondent counting efficiency value of the counted sample could be checked out in the quench curve [SQP(E)–Efficiency], which was to be applied to calculate the activity concentration of OBT in biota samples.



I'd like to express my sincere gratitude to the organizing committee of LSC2017 for giving me the chance to give this presentation.

Please do not hesitate to give any criticism to my work.

**Thank you and welcome to China!**