



APLAC T072

Food Synthetic Dyestuff Proficiency Testing Program

FINAL REPORT

**China National Accreditation Service
for Conformity Assessment (CNAS)**

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FINAL REPORT

1. Instruction

This report summarizes the results of APLAC Food Synthetic Dyestuff Proficiency Testing Program T072.

This program was conducted in accordance with the simultaneous scheme specified in *ISO/IEC 17043:2010*, covered quantitative tests on **Sunset Yellow** and **Tartrazine** contents in orange juice samples. This program applies only to the use of interlaboratory comparisons of the purpose of proficiency testing. The aim of this program is to assess the performance of individual laboratories for specific tests. It does not include determining the effectiveness and precision of testing methods.

2. Main Relevant Parties

Asia Pacific Laboratory Accreditation Cooperation (APLAC) approved and financially supported the program.

2.1 Organizing and Coordinating Bodies

APLAC T072 Program was organized by China National Accreditation Service for Conformity Assessment (CNAS) and coordinated by Technical Centre of Shenyang Entry-Exit Inspection and Quarantine Bureau of People's Republic of China.

2.2 Summary of Participants and Their Accreditation Situations

The members of APLAC, EA, IAAC and some other laboratory accreditation bodies were invited to nominate laboratories to participate in this program. A total of 39 laboratories from 25 economies were nominated.

6 laboratories could not report any results till the the deadline, while 33 laboratories reported the testing results of Sunset Yellow and Tartrazine contents in the three samples, in which 12 laboratories were accredited for testing Sunset Yellow and Tartrazine contents by their accreditation bodies.

Each participating laboratory was endowed with an exclusive lab code. All references to the laboratories associated with this program are with these codes, respectively. The numbers of laboratories with respect to their accreditation bodies are listed in Table 1.

Table 1 Summary of Participants

Economy	Accreditation Body	Number of Labs
Argentina	ORGANISMO ARGENTINO DE ACREDITACIÓN (OAA)	1
Australia	National Association of Testing Authorities (NATA)	2
Brunei	Ministry of Development	1
Canada	Standards Council of Canada	1
China	China National Accreditation Service for Conformity Assessment (CNAS)	2
Chinese Hong Kong	Hong Kong Accreditation Service (HKAS)	2
Colombia	ONAC	2
Czech	Czech Accreditation Institute	2
El Salvador	Consejo Nacional de Ciencia y Tecnología-CONACYT	2
Estonia	Estonian Accreditation Centre	1
Finland	FINAS Finnish Accreditation Service	1
Germany	DAkkS Deutsche Akkreditierungsstelle GmbH	2
Guatemala	Oficina Guatemalteca de Acreditación – OGA	2
Israel	ISRAC (Israel Accreditation)	1
Italy	ACCREDIA	2
Japan	The Japan Accreditation Board for Conformity Assessment (JAB)	1
Korea	KOLAS	1
Mongolia	Accreditation Department, MASM	1
Poland	Polish Centre for Accreditation	2
Romania	RENAR – Romanian Accreditation Association	1
Sweden	SWEDAC	1
Switzerland	Swiss Accreditation Service	2
Thailand	Bureau of Laboratory Accreditation, Department of Science Service (BLA-DSS)	3
U. S.	American Association for Laboratory Accreditation (A2LA)	2
Viet Nam	Bureau of Accreditation, Viet Nam	1

3. Statistical Design

3.1 Agreement of Testing Methods

Sunset Yellow and Tartrazine are widely used as food synthetic dyestuff in juice. Unfortunately, Sunset Yellow and Tartrazine are provided with middling toxicity, which is harmful to human health.

There are various methods for quantifying Sunset Yellow and Tartrazine contents in orange juice samples. In this situation, the samples in this program should be analyzed with preferably selected methods and it was recommended to use HPLC, HPLC-MS methods. In this program, HPLC, HPLC-MS and new UPLC methods were used by the participants, no significant difference was found among the datasets obtained by using different methods.

3.2 Three Samples Employed

The accuracy of testing results may be different on samples with different content levels. Consequently, discrepancy probably occurs for samples with different content levels tested in different laboratories.

In this program, three samples with different contents of Sunset Yellow and Tartrazine were employed, in which two levels were designed. The accuracy of testing results at these different levels will be examined in this proficiency testing program.

Split-level design was used in this program, at one level, two samples with slightly different contents were employed. When the agreement of participants' results is well enough, the **laboratory repeatability** could be assessed.

3.3 Methods for Statistical Assessments

Robust statistical method was employed to assess the ability of participating laboratories to implement relevant tests of Sunset Yellow and Tartrazine contents. Individual, within-lab and between-labs z-scores were intended to use as the assessments of the ability for an individual participating laboratory. Details are shown in Appendix D.

For each gross sample, the assigned value and the standard deviation for proficiency assessment were consensus values from participants. Statistical graphs, such as histograms and Youden plots were regarded as references to the overall effectiveness of this program.

3.4 Measurement Uncertainty

To ensure the **traceability**, the measurement uncertainty of the assigned values were evaluated and limited in sample preparation.

Perhaps some participants were not used to evaluate or report measurement uncertainty, for each content, about 5 laboratories did not report measurement uncertainty. Except it, the relative measurement uncertainty reported by the participants covers a wide range, from the minimum 0.2% to the maximum 17.3% (i.e. the ratio of max/min is about 85), it shows quite inconsistent. Obviously, we can not explain it by the discrepancy in equipments, technical operation, or other environmental disturbance, only evaluation methods or skill should be paid attention to.

The measurement uncertainty reported by the participants in this program was shown in Annex B (Fig. B.29~B.34)

4. Description of Samples

4.1 Preparation and Distribution

Each **gross sample** was prepared by adding designed amounts of Sunset Yellow and Tartrazine (Sunset Yellow CAS Registering Number: 2783-94-0, Tartrazine CAS Registering Number: 1934-21-0) in super-water to the blank orange juice. Then, the orange juice samples were mixed well to make it homogeneous.

Respectively, each gross sample was divided and packed into sealed plastic bottles covered with dark packaging to make into testing samples.

The testing samples were randomly labeled and recorded. Testing samples packed with the Instructions for Participants (see Appendix E) were distributed directly to accreditation bodies as requested.

Note: For the convenience of description in this report, these three gross samples are denoted as Gross Sample C, Gross Sample D and Gross Sample E, the testing samples prepared from each corresponding gross sample are denoted as Sample C, Sample D and Sample E, and the results obtained on these samples are denoted as Result C, Result D and Result E, respectively.

4.2 Homogeneity and Stability

The samples randomly selected from each gross sample were determined for their homogeneity and stability. Details of sample homogeneity and stability check are described in Appendix C.

5. Testing Results and Assessments

5.1 Methods for Assessments

To assess performances of participants, individual z-score for each result was calculated, within-lab and between-labs z-scores for each pair of results (Result C and Result E) were derived based on the normalized difference and normalized sum of the paired results (also see Appendix D). As a general rule, according to *ISO/IEC 17043*, any absolute value of individual, within-lab or between-labs z-score falls in the range of 2 to 3 is regarded as a questionable result, while any absolute value of z-score exceeds 3 is regarded as an unsatisfactory result/pair of results.

The participants are encouraged to review, if any, the questionable or unsatisfactory results. It is suggested that the participants refer to the main technical aspects given in clause 6 of this report.

5.2 Statistics for Assessments

32 participating laboratories reported testing results of Sunset Yellow and Tartrazine contents in samples C, D and E, the results are listed together with the testing methods in Appendix A. The robust mean, robust standard deviation and **standard uncertainty** for individual results C, D, E, normalized sum and normalized difference were estimated and listed in Table 2-1 and Table 2-2.

Table 2-1 Statistics for Assessments of Sunset Yellow

Results	Total Number of Results	Robust Mean, mg/kg	Robust Standard Deviation, mg/kg	Standard Uncertainty, mg/kg
Result C	32	87.1	9.8	2.2
Result D	32	137.9	16.2	3.6
Result E	32	61.2	7.2	1.6
Normalized Sum of Result C and E	32	105.2	11.5	2.5
Normalized Difference between Result C and E	32	17.8	2.4	0.5

Table 2-2 Statistics for Assessments of Tartrazine

Results	Total Number of Results	Robust Mean, mg/kg	Robust Standard Deviation, mg/kg	Standard Uncertainty, mg/kg
Result C	33	123.1	12.7	2.8
Result D	33	72.9	8.6	2.0
Result E	33	106.6	9.5	2.1
Normalized Sum of Result C and E	33	162.7	15.1	3.3
Normalized Difference between Result C and E	33	11.2	2.4	0.5

A total of 9(28.1%) laboratories made questionable or unsatisfactory results, in which 6(18.8%) laboratories reported unsatisfactory results. The lab codes with questionable results are listed in Table 3.

Table 3 Laboratories Made Questionable or Unsatisfactory Results

z-scores	Sunset Yellow		Tartrazine	
	Questionable ($2 \leq z < 3$)	Unsatisfactory ($ z \geq 3$)	Questionable ($2 \leq z < 3$)	Unsatisfactory ($ z \geq 3$)
z-score of sample C (z_C)	T072-011	T072-007	T072-010	T072-007, T072-025
z-score of sample D (z_D)	T072-004, T072-010, T072-011			T072-010, T072-017
z-score of sample E (z_E)	T072-011	T072-007	T072-036	T072-007, T072-025
Between-labs z-score z_b (C&E)	T072-011	T072-007		T072-007, T072-025
Within-lab z-score z_w (C&E)	T072-002, T072-025	T072-004, T072-007, T072-010, T072-017	T072-002	T072-004, T072-010, T072-017, T072-025, T072-035

6. Technical Commentary

In order to facilitate the participants to find the reasons of unsatisfactory results and improve their testing performance, main factors that influence the analytical results of Sunset Yellow and Tartrazine contents are described as following

- Purity and storage of standard solution;
- Testing conditions;
- Calculation of results.

In this program, some participants used lower purity of standard solution, it might lead to higher results. The lower purity might be caused either by reference materials, or by improper storage of standard solution.

Extracting operation, such as improper choice and usage of extracting solvents and polyamide powder, might lead to inaccurate results. Higher results might be obtained due to lack of cleaning-up steps. Improper chromatography determination conditions might lead to inaccurate results, too.

According to some participants' records for this program, the method of quantitative analysis with one standard concentration point was used, if the peak area of the testing point was far from that of the standard point, then the inaccurate results could be obtained.

Repeatability of testing results was assessed by using the plots of range against average shown in Annex B, some participants obtained results with poor dispersion, the repeatability of testing method, replicate operations and other random factors should be investigated. Oppositely, some laboratories reported very same replicate results, as shown in Annex A, the authenticity of these results was doubtful.

In this program, also, the exact reasons of some unsatisfactory results could not be found, because the information the participants provided was very limit.

APPENDIX A

Summary of Testing Results

The testing results on Sunset Yellow and Tartrazine contents obtained from the participants with corresponding testing methods and the z-scores are listed in Table A.1 and Table A.2.

In these tables, some abbreviations and symbols were used. The symbols z_C , z_D and z_E denote individual z-scores of result C, result D, result E, respectively. The symbols z_b and z_w denote between-labs z-scores and within-lab z-scores of paired results C and E, respectively. The symbol “#” indicates a z-score derived from a questionable result/pair of results, while “\$” follows a z-score derived from an unsatisfactory result/pair of results. The abbreviations Norm. Sum and Norm. Diff. denote normalized sum and normalized difference of paired result C and E as described in Appendix D, respectively.

Table A.1 Testing Results of Sunset Yellow with Their Assessments

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
T072-001	Z 231 (C)	94.5	95.2	94.7	94.5	94.7	0.70	0.77	115.3	0.88	18.6	0.36	HPLC-SPD	5
	Z 183 (D)	154	152	153	155	154	3.00	1.00						
	Z 809 (E)	67.7	67.3	69.1	69.4	68.4	2.10	1.00						
T072-002	Z 210 (C)	102.0	103.2	97.46	95.62	99.57±12%	7.58	1.27	117.4	1.06	23.4	2.42#	UPLC-PDA	0.5
	Z 169 (D)	168.2	164.4	162.8	146.3	160.42±12%	21.90	1.39						
	Z 783 (E)	72.38	69.52	62.77	61.50	66.42±12%	10.88	0.72						
T072-004	Z 506 (C)	88.49	94.95	92.58		92.01±3.27,K=1	6.46	0.50	102.6	-0.23	27.6	4.16\$	HPLC-UV	0.1
	Z 334 (D)	100.37	98.11	92.39		96.95±4.11,K=1	7.98	-2.53#						
	Z 764 (E)	53.49	49.35	56.28		53.04±3.49,K=1	6.93	-1.15						
T072-005	Z 346 (C)	77.7	78.9	77.3	76.6	77.6±10.9	2.30	-0.97	94.1	-0.96	15.6	-0.90		5
	Z 497 (D)	120.0	124.1	121.7	127.8	123.4±17.3	7.80	-0.90						
	Z 746 (E)	57.4	54.6	54.6	55.2	55.5±7.8	2.80	-0.80						
T072-006	Z 353 (C)	97	98	98	98	98±9.8	1.00	1.11	118.1	1.12	20.5	1.17	HPLC-DAD	10
	Z 831 (D)	160	158	158	158	159±15.9	2.00	1.30						
	Z 870 (E)	68	69	69	69	69±6.9	1.00	1.08						
T072-007	Z 243 (C)	117	122	120	123	120	6.00	3.41\$	165.6	5.25\$	4.8	-5.51\$	LC-SPD	1
	Z 239 (D)	127	126	127	125	126	2.00	-0.72						
	Z 908	112	115	114	114	114	3.00	7.33\$						

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
T072-008	(E)													
	Z 104 (C)	78.339	77.925	78.074	77.903	78.06±2.18	0.44	-0.92	94.1	-0.97	16.3	-0.61	LC-UV	0.011
	Z 966 (D)	124.066	124.005	124.398	124.112	124.15±3.22	0.39	-0.85						
	Z 760 (E)	54.809	55.211	55.033	54.907	54.99±1.64	0.40	-0.87						
T072-009	Z 221 (C)	81.96	81.65	81.97	81.93	81.9±1.1,K=2	0.32	-0.53	99.2	-0.52	16.6	-0.48	HPLC-MWD	0.16
	Z 932 (D)	131.10	131.36	131.46	131.44	131.3±1.6,K=2	0.36	-0.41						
	Z 676 (E)	58.32	58.40	58.37	58.33	58.4±0.8,K=2	0.08	-0.40						
T072-010	Z 047 (C)	86.4	87.6			87.0±1.10	1.20	-0.01	112.9	0.67	10.2	-3.21\$		
	Z 996 (D)	178.8	179.4			179.1±1.82	0.60	2.55#						
	Z 980 (E)	72.0	73.2			72.6±1.6	1.20	1.59						
T072-011	Z 588 (C)	63.7	63.7	63.7	63.7	63.7±3.4	0.00	-2.39#	75.1	-2.62#	15.0	-1.17	HPLC-DAD	3
	Z 418 (D)	104	104	104	104	104±5	0.00	-2.10#						
	Z 814 (E)	42.5	42.5	42.5	42.5	47.5±2.5	0.00	-2.62#						
T072-012	Z 223 (C)	78.92	79.43			79.17	0.51	-0.81	93.6	-1.01	18.4	0.27		10
	Z 517 (D)	128.78	128.38			128.58	0.40	-0.58						
	Z 794 (E)	53.97	52.37			53.17	1.60	-1.13						
T072-013	Z 021 (C)	82.2	86.8	83.9	87.8	85.2±7.0,K=2,	5.60	-0.19	104.0	-0.10	16.5	-0.54	LC-UV	1
	Z 007 (D)	133	134	134	133	134±14,K=2,	1.00	-0.24						
	Z 742 (E)	63.4	60.3	62.6	61.3	61.9±4.0,K=2,	3.10	0.09						

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
T072-014	Z 140 (C)	88.74	88.62	89.88	88.61	88.96±0.91	1.27	0.19	106.9	0.15	18.9	0.51		0.3
	Z 416 (D)	142.74	143.35	144.21	143.38	143.42±1.47	1.47	0.34						
	Z 670 (E)	62.09	62.24	62.20	62.18	62.17±0.64	0.15	0.13						
T072-015	Z 415 (C)	91.8	91.8	91.6	91.8	91.8±1.92	0.20	0.48	110.9	0.49	19.0	0.51	LC-PDA	9.88
	Z 730 (D)	147	147	147	147	147±1.92	0.00	0.56						
	Z 974 (E)	65.1	64.9	65.1	64.7	65.0±1.92	0.40	0.52						
T072-016	Z031 (C)	85.2	84.8	85.2	84.6	84.9±3.8	0.60	-0.23	102.0	-0.28	18.1	0.15	LC-UV	3.0
	Z 247 (D)	136.9	136.6	136.7	136.0	136.5±6.0	0.90	-0.09						
	Z 942 (E)	59.2	59.3	59.3	59.2	59.3±2.6	0.10	-0.27						
T072-017	Z 365 (C)	105.0	110.0	105.0	98.0	104.7±15.7	12.00	1.79	122.2	1.48	25.9	3.45\$	LC-DAD	2
	Z 193 (D)	143.0	138.0	140.0	143.5	141.1±21.2	5.50	0.20						
	Z 792 (E)	68.6	68.5	68.9	66.5	68.1±10.2	2.40	0.96						
T072-020	Z 403 (C)	88.9	90.2	88.8	90.3	89.6±0.8	1.50	0.25	108.0	0.24	18.7	0.42		5
	Z 305 (D)	138.6	138.9	140.4	141.3	139.8±1.3	2.70	0.12						
	Z 802 (E)	63.3	63.1	63.3	62.6	63.1±0.3	0.70	0.26						
T072-021	Z 386 (C)	85.78	85.54	84.23	83.47	84.76±4.48	2.31	-0.24	103.2	-0.17	16.6	-0.47	HPLC-MWD	0.20
	Z 750 (D)	136.96	138.08	137.04	137.28	137.34±10.44	1.12	-0.03						
	Z 955 (E)	61.49	61.03	61.46	60.89	61.22±2.11	0.60	0.00						
T072-022	Z 550	85.6	85.6	85.1	83.2	85.3±5.07	2.40	-0.18	104.3	-0.08	16.3	-0.60	HPLC-UV	0.72

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
	(C)													
	Z 539 (D)	133.5	132.8	132.8	131.7	132.7±7.75	1.80	-0.32						
	Z 865 (E)	61.9	62.6	62.2	62.2	62.2±3.62	0.70	0.13						
T072-023	Z 171 (C)	74.76	73.64	70.47	70.95	72.46±7.26	4.29	-1.49	86.1	-1.66	16.4	-0.57	HPLC-DAD	2.00
	Z 269 (D)	109.77	104.04	100.00	112.94	106.44±14.25	12.94	-1.95						
	Z 941 (E)	47.26	51.42	48.45	49.85	49.24±5.42	4.16	-1.68						
T072-024	Z 245 (C)	76	77	77	76	77±7.7	1.00	-1.03	92.6	-1.09	16.3	-0.63		1
	Z 636 (D)	122	124	124	121	123±12.3	3.00	-0.92						
	Z 961 (E)	54	54	54	54	54±5.4	0.00	-1.01						
T072-025	Z 464 (C)	108.7	104.8	104.6	105.0	105.8±3.1	4.10	1.91	126.3	1.83	23.3	2.37#	LC-UV	0.6
	Z 816 (D)	169.2	169.8	170.6	168.0	169.4±1.7	2.60	1.95						
	Z 848 (E)	72.8	72.4	73.1	73.0	72.8±0.5	0.70	1.61						
T072-026	Z 094 (C)	80.50	80.87	79.93	80.83	80.53±1.05	0.94	-0.67	97.0	-0.71	16.9	-0.36	LC-DAD	3.50
	Z 134 (D)	138.93	138.12	138.92	138.98	138.73±1.41	0.86	0.05						
	Z 615 (E)	56.87	56.73	56.92	56.06	56.64±0.69	0.86	-0.64						
T072-027	Z 192 (C)	101.06	102.12	101.79	101.67	101.66±13.63	1.06	1.48	122.4	1.49	21.4	1.56	HPLC-DAD	0.32
	Z 203 (D)	160.98	163.07	162.40	162.82	162.32±21.77	2.09	1.51						
	Z 743 (E)	71.34	71.25	71.25	71.68	71.37±9.57	0.43	1.41						
T072-029	Z 149 (C)	84.8	85.7	84.2	84.8	84.9±13.9	1.50	-0.23	102.2	-0.26	17.8	0.03		2.1

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
T072-030	Z 968 (D)	137.7	137.4	135.8	137.2	137.0±20.9	1.90	-0.06	108.2	0.26	18.0	0.12	HPLC-DAD	
	Z 991 (E)	60.5	58.5	59.3	60.5	59.7±10.3	2.00	-0.22						
	Z 289 (C)	90	89	86	92	89	6.00	0.22						
T072-031	Z 705 (D)	143	143	142	143	143	1.00	0.30	101.1	-0.35	16.7	-0.45		0.6
	Z 904 (E)	65	64	63	63	64	2.00	0.35						
	Z 392 (C)	83.6	83.2	83.5	82.9	83.3±2.0	0.70	-0.39						
T072-032	Z 429 (D)	133.9	133.8	133.8	133.7	133.8±2.7	0.20	-0.25	114.2	0.78	18.3	0.24		0.483
	Z 909 (E)	59.7	59.5	59.5	59.9	59.7±1.3	0.40	-0.22						
	Z 503 (C)	93.869	93.181	95.572	92.204	93.7±4.4(K=2)	3.37	0.67						
T072-034	Z 638 (D)	153.835	156.892	155.555	146.170	153.1±7.0(K=2)	10.72	0.94	92.8	-1.08	15.7	-0.86	LC-DAD	5
	Z 867 (E)	66.120	68.298	70.614	66.366	67.8±4.4(K=2)	4.49	0.92						
	Z 389 (C)	77.43	76.30	76.95	76.22	76.73±7.7	1.21	-1.06						
T072-035	Z 855 (D)	120.25	122.43	121.14	122.18	121.50±12.2	2.18	-1.01	104.6	-0.05	18.0	0.10		10
	Z 713 (E)	55.22	53.69	54.97	54.09	54.49±5.4	1.53	-0.94						
	Z 384 (C)	86	87	85	88.7	86.7	3.70	-0.04						
T072-036	Z 946 (D)	149	143	140	160	148	20.00	0.62	98.2	-0.61	16.4	-0.58		20
	Z 675 (E)	63	57	61	64	61	7.00	0.00						
	Z 412 (C)	79.24	79.83	82.46	82.49	81.00RSD2.1%	3.25	-0.62						

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
	Z 614 (D)	145.06	152.26	144.87	145.16	146.84RSD3.6%	7.39	0.55						
	Z 940 (E)	58.87	55.89	57.3	59.25	57.83RSD2.7%	3.36	-0.48						
T072-038	Z 598 (C)	88.7	89.3	89.8	89.2	89.3±0.4	1.10	0.22	107.1	0.17	19.2	0.60	LC-DAD	2
	Z 610 (D)	142.7	141.5	142.8	142.0	142.2±0.5	1.30	0.27						
	Z 726 (E)	62.1	62.1	62.4	62.1	62.2±0.1	0.30	0.13						
T072-039	Z 380 (C)	83.7	82.6	83.8	82.9	83.2±1.0	1.20	-0.40	101.7	-0.31	16.0	-0.75	LC-UV/DAD	1.0
	Z 988 (D)	133.7	138.7	130.1	131.3	133.5±6.0	8.60	-0.27						
	Z 948 (E)	61.4	62.1	61.9	56.9	60.6±4.0	5.20	-0.09						

Table A.2 Testing Results of Tartrazine with Their Assessments

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
T072-001	Z 231 (C)	135	138	136	135	136	3.00	1.02	181.7	1.27	10.6	-0.24	HPLC-DAD	5
	Z 183 (D)	78.6	75.9	75.4	75.2	76.3	3.40	0.40						
	Z 809 (E)	120	120	120	124	121	4.00	1.52						
T072-002	Z 210 (C)	137.8	138.7	141.8	138.1	139.1±10%	4.00	1.27	180.5	1.18	16.3	2.12 #	UPLC-PDA	0.5
	Z 169 (D)	81.52	80.21	84.77	76.91	80.85±10%	7.86	0.93						
	Z 783 (E)	122.9	118.4	112.0	111.2	116.1±10%	11.70	1.00						
T072-004	Z 506 (C)	125.32	137.83	128.41		130.52±6.52,k=1	12.51	0.59	160.4	-0.15	24.2	5.42 \$	HPLC-UV	0.1
	Z 334 (D)	67.17	69.94	65.88		67.66±2.07,k=1	4.06	-0.61						
	Z 764 (E)	95.77	94.24	98.90		96.30±2.38,k=1	4.66	-1.09						
T072-005	Z 346 (C)	109.5	110.9	109.1	108.8	109.6±14.2	2.10	-1.06	145.9	-1.11	9.1	-0.89		5
	Z 497 (D)	60.9	62.4	62.1	64.6	62.5±8.1	3.70	-1.21						
	Z 746 (E)	97.3	98.5	95.5	96.0	96.8±12.6	3.00	-1.04						
T072-006	Z 353 (C)	126	126	127	126	126±12.6	1.00	0.23	166.9	0.28	11.3	0.06	HPLC-DAD	10
	Z 831 (D)	75	74	74	74	74±7.4	1.00	0.13						
	Z 870 (E)	109	110	110	110	110±11.0	1.00	0.36						

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
T072-007	Z 243 (C)	79	82	81	83	81	4.00	-3.30\$	103.1	-3.96\$	11.8	0.28	LC-SPD	1
	Z 239 (D)	72	72	73	70	72	3.00	-0.13						
	Z 908 (E)	63	65	65	65	64	2.00	-4.45\$						
T072-008	Z 104 (C)	114.560	114.229	115.039	114.315	114.54±2.16	0.81	-0.67	151.3	-0.76	10.7	-0.19	LC-UV	0.013
	Z 966 (D)	66.588	66.481	66.724	66.680	66.62±1.22	0.24	-0.73						
	Z 760 (E)	99.252	99.601	99.252	99.381	99.38±1.80	0.35	-0.76						
T072-009	Z 221 (C)	116.24	117.09	116.25	116.20	116.4±1.5,K=2	0.89	-0.52	154.9	-0.51	9.7	-0.62		0.16
	Z 932 (D)	68.59	67.87	68.55	68.61	68.4±1.1,K=2	0.74	-0.52						
	Z 676 (E)	102.26	103.28	102.16	103.19	102.7±1.5,K=2	1.12	-0.41						
T072-010	Z 047 (C)	150.0	149.4			149.7±1.54	0.60	2.10#	191.1	1.89	20.6	3.91\$		
	Z 996 (D)	121.2	119.4			120.3±1.25	1.80	5.53\$						
	Z 980 (E)	121.8	119.4			120.6±1.62	2.40	1.48						
T072-011	Z 588 (C)	109	109	109	109	109±3	0.00	-1.11	144.0	-1.24	10.1	-0.44	HPLC-DAD	2
	Z 418 (D)	62.5	62.5	62.5	62.5	62.5±1.7	0.00	-1.21						
	Z 814 (E)	94.7	94.7	94.7	94.7	94.7±2.4	0.00	-1.26						
T072-012	Z 223 (C)	120.46	120.56			120.51	0.10	-0.20	159.8	-0.19	10.6	-0.24		10

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
	Z 517 (D)	70.79	70.77			70.78	0.02	-0.24						
	Z 794 (E)	105.69	105.38			105.53	0.31	-0.11						
	Z 021 (C)	116	121	117	121	119±9,k=2	5.00	-0.32						
T072-013	Z 007 (D)	70.4	74.3	73.3	73.3	72.8±4.7,k=2	3.90	-0.01	159.1	-0.24	9.2	-0.83	LC-UV	1
	Z 742 (E)	106	107	104	108	106±6,k=2	4.00	-0.06						
	Z 140 (C)	120.07	125.16	119.51	121.79	121.63±1.36	5.65	-0.11						
T072-014	Z 416 (D)	73.15	74.28	70.94	73.70	73.01±0.81	3.34	0.02	161.0	-0.11	11.0	-0.07		0.3
	Z 670 (E)	107.33	108.08	104.84	103.91	106.04±1.18	4.17	-0.06						
	Z 415 (C)	136	136	135	136	136±0.90	1.00	1.02						
T072-015	Z 730 (D)	80.9	80.8	80.9	81	80.9±0.90	0.20	0.94	180.3	1.17	12.0	0.35	LC-PDA	9.54
	Z 974 (E)	119	119	119	119	119±0.90	0.00	1.31						
	Z 031 (C)	113.8	112.7	113.8	113.0	113.3±5.0	1.10	-0.77						
T072-016	Z 247 (D)	65.1	65.2	65.7	65.5	65.3±2.9	0.60	-0.88	149.8	-0.85	10.4	-0.33	LC-UV	3.0
	Z 942 (E)	98.2	99.0	98.8	98.2	98.6±4.4	0.80	-0.84						
	Z 365 (C)	137.5	134.0	135.0	133.0	134.9±20.2	4.50	0.93						
T072-017	Z 193 (D)	100.0	105.0	105.0	110.0	105.0±15.8	10.00	3.75\$	168.5	0.39	22.3	4.62\$	LC-DAD	2
	Z 792	107.6	104.2	102.0	100.0	103.4±15.5	7.60	-0.34						

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
T072-020	(E)													
	Z 403 (C)	117.2	116.7	116.3	117.5	116.9±0.5	1.20	-0.49	155.4	-0.48	9.9	-0.53		5
	Z 305 (D)	70.7	69.8	70.7	70.6	70.5±0.4	0.90	-0.27						
	Z 802 (E)	102.8	103.3	102.1	103.3	102.9±0.6	1.20	-0.39						
T072-021	Z 386 (C)	126.20	126.65	125.32	124.48	125.66±8.90	2.17	0.21	166.7	0.27	11.0	-0.06	HPLC-MWD	0.20
	Z 750 (D)	74.95	75.38	74.70	74.48	74.88±3.14	0.90	0.24						
	Z 955 (E)	109.91	109.95	110.07	110.22	110.04±6.64	0.31	0.36						
T072-022	Z 550 (C)	120.7	119.7	116.9	120.0	119.3±7.11	3.80	-0.30	159.5	-0.21	9.2	-0.83	HPLC-UV	0.49
	Z 539 (D)	70.5	70.0	73.1	69.8	70.8±4.40	3.30	-0.24						
	Z 865 (E)	104.9	106.1	105.3	108.8	106.3±6.45	3.90	-0.03						
T072-023	Z 171 (C)	106.04	105.83	109.36	108.67	107.48±14.29	3.53	-1.23	145.2	-1.16	6.8	-1.83	HPLC-DAD	2.00
	Z 269 (D)	58.75	59.21	61.68	60.73	60.09±8.20	2.93	-1.49						
	Z 941 (E)	99.64	96.17	98.84	96.96	97.90±13.00	3.47	-0.92						
T072-025	Z 464 (C)	168.4	165.2	170.9	164.1	167.1±4.9	6.80	3.47\$	215.8	3.53\$	20.5	3.88\$	LC-UV	2.8
	Z 816 (D)	91.3	83.6	88.0	95.1	89.5±7.7	11.50	1.94						
	Z 848 (E)	135.1	136.3	139.8	141.4	138.1±4.5	6.30	3.33\$						
T072-026	Z 094 (C)	123.48	123.52	123.47	123.26	123.43±0.55	0.26	0.03	165.1	0.16	9.5	-0.71	LC-DAD	4.04

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
	Z 134 (D)	71.76	71.81	71.79	71.89	71.81±1.13	0.13	-0.12						
	Z 615 (E)	109.98	110.01	110.13	110.06	110.04±0.67	0.15	0.36						
	Z 192 (C)	131.79	132.72	132.79	133.38	132.4±18.32	1.59	0.74						
T072-027	Z 203 (D)	75.39	76.33	75.66	76.91	76.06±10.52	1.52	0.37	174.8	0.81	12.4	0.53	HPLC-DAD	0.32
	Z 743 (E)	114.90	114.64	114.92	113.99	114.8±15.88	0.93	0.87						
	Z 149 (C)	119.1	120.8	119.8	119.8	119.9±18.7	1.70	-0.25	158.0	-0.31	11.5	0.14		1.1
T072-029	Z 968 (D)	69.7	70.5	69.8	70.6	70.2±11.8	0.90	-0.31						
	Z 991 (E)	102.8	104.0	101.0	106.4	103.6±16.5	5.40	-0.32						
T072-030	Z 289 (C)	121	119	114	123	119	9.00	-0.30	157.2	-0.37	11.5	0.13	HPLC-DAD	
	Z 705 (D)	69	70	70	70	70	1.00	-0.36						
	Z 904 (E)	106	102	102	102	103	4.00	-0.38						
T072-031	Z 392 (C)	115.0	114.9	114.7	114.0	114.7±2.8	1.00	-0.66	153.2	-0.63	9.1	-0.89		0.8
	Z 429 (D)	67.7	67.7	67.8	67.6	67.7±1.4	0.20	-0.60						
	Z 909 (E)	102.0	101.8	101.8	101.9	101.9±2.0	0.20	-0.50						
T072-032	Z 503 (C)	116.116	114.314	114.539	112.558	114.4±4.5(K=2)	3.56	-0.68	153.4	-0.62	8.4	-1.15		0.271
	Z 638 (D)	69.144	70.069	68.404	64.863	68.1±4.2(K=2)	5.21	-0.55						
	Z 867	102.87	103.09	105.19	98.818	102.5±4.8(K=2)	6.38	-0.43						

Lab Code	Sample No.	Testing Results (mg/kg)					Range	Z-score	Normalized Sum	Zb	Normalized Difference	Zw	Testing Method	LOD (mg/kg)
		1	2	3	4	Mean								
	(E)	9	6	7										
T072-034	Z 389 (C)	116.68	114.62	115.39	117.54	116.06±11.6	2.92	-0.55	154.5	-0.54	9.6	-0.64	LC-DAD	5
	Z 855 (D)	70.37	71.40	67.62	72.00	70.35±7.0	4.38	-0.29						
	Z 713 (E)	103.36	98.55	103.96	103.88	102.44±10.2	5.41	-0.44						
T072-035	Z 384 (C)	169	143	128	124	141	45.00	1.42	180.5	1.19	18.9	3.22 \$		10
	Z 946 (D)	99	92	82	70	86	29.00	1.50						
	Z 675 (E)	121	129	109	98	114	31.00	0.81						
T072-036	Z 412 (C)	136.84	141.70	144.60	146.00	142.28RSD2.8%	9.16	1.52	190.8	1.87	10.4	-0.31		20
	Z 614 (D)	84.37	92.82	82.08	85.00	89.21RSD5.4%	10.74	1.91						
	Z 940 (E)	128.22	123.76	127.04	131.09	127.53RSD2.4%	7.33	2.21#						
T072-038	Z 598 (C)	129.1	128.7	130.7	130.3	129.7±0.8	2.00	0.52	171.1	0.56	12.3	0.47	LC-DAD	3
	Z 610 (D)	75.1	76.1	76.7	75.1	75.8±0.8	1.60	0.34						
	Z 726 (E)	112.2	111.4	112.5	113	112.3±0.6	1.60	0.60						
T072-039	Z 380 (C)	112.9	113.2	113.8	115.1	113.8±1.5	2.20	-0.73	150.4	-0.81	10.5	-0.27	LC-UV/DAD	2.0
	Z 988 (D)	65.0	67.0	62.6	61.7	64.1±4.0	5.30	-1.02						
	Z 948 (E)	98.5	100.5	101.7	94.9	98.9±5.0	6.80	-0.81						

APPENDIX B

Graphical Display of Testing Results

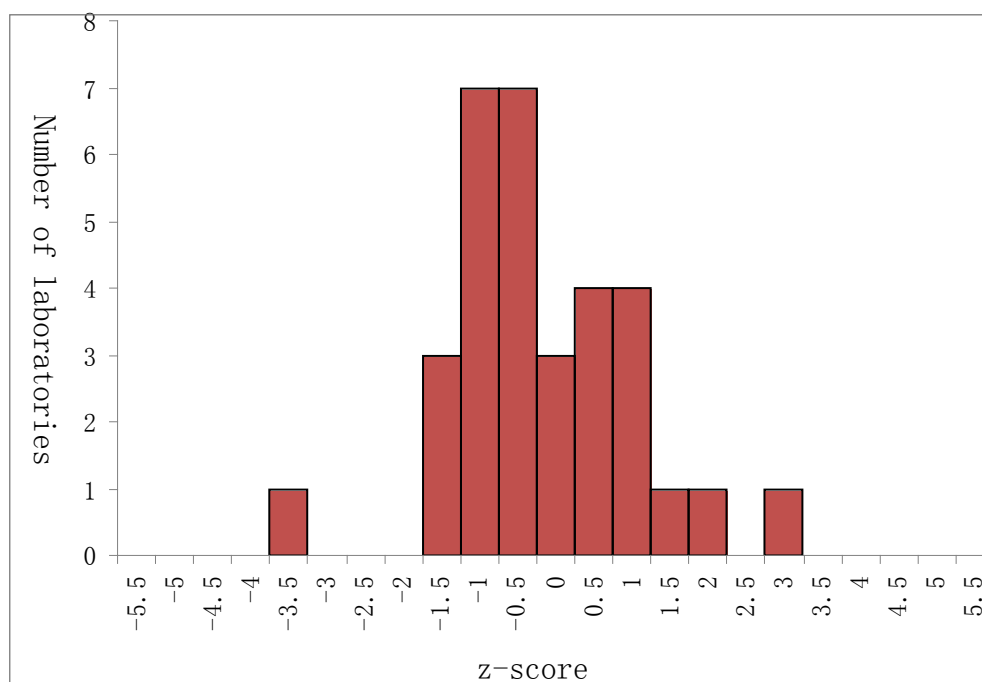
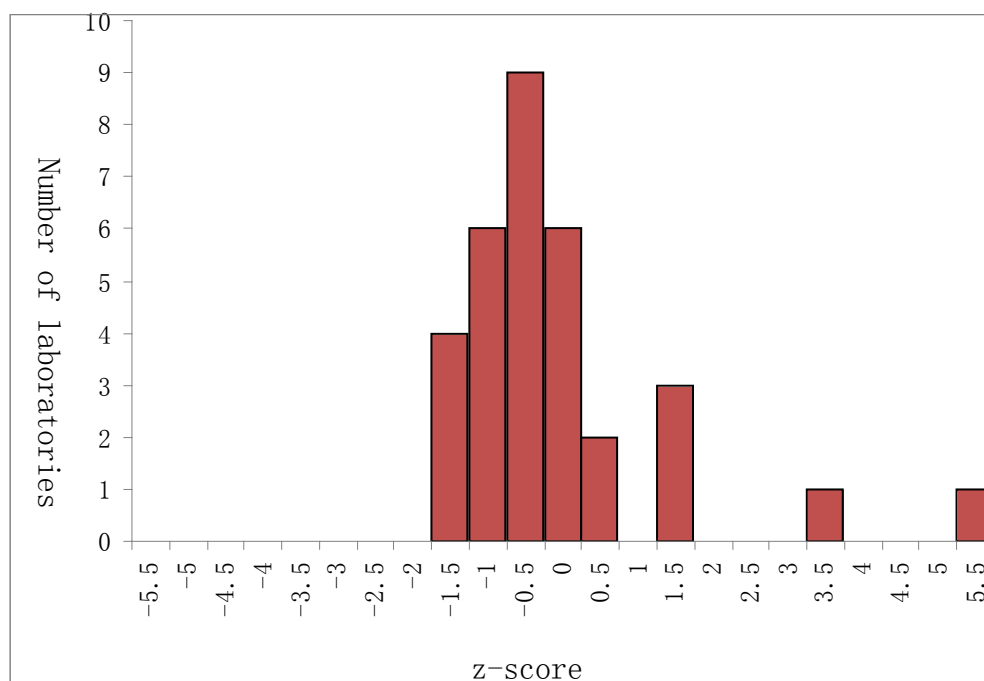
Here, graphs were made in order to give an open-and-shut display of testing results and their statistics.

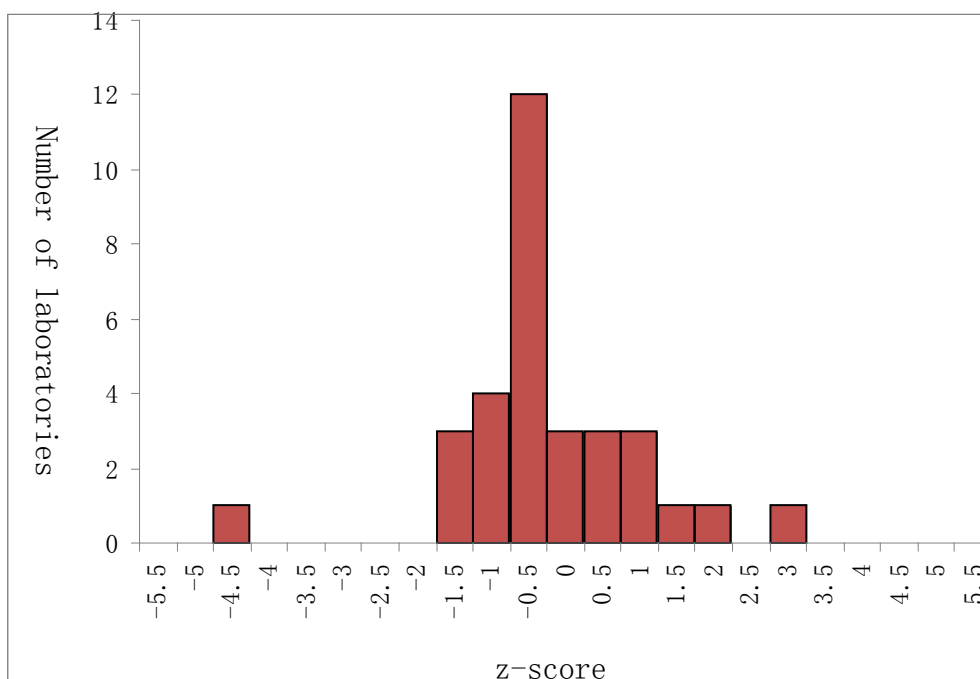
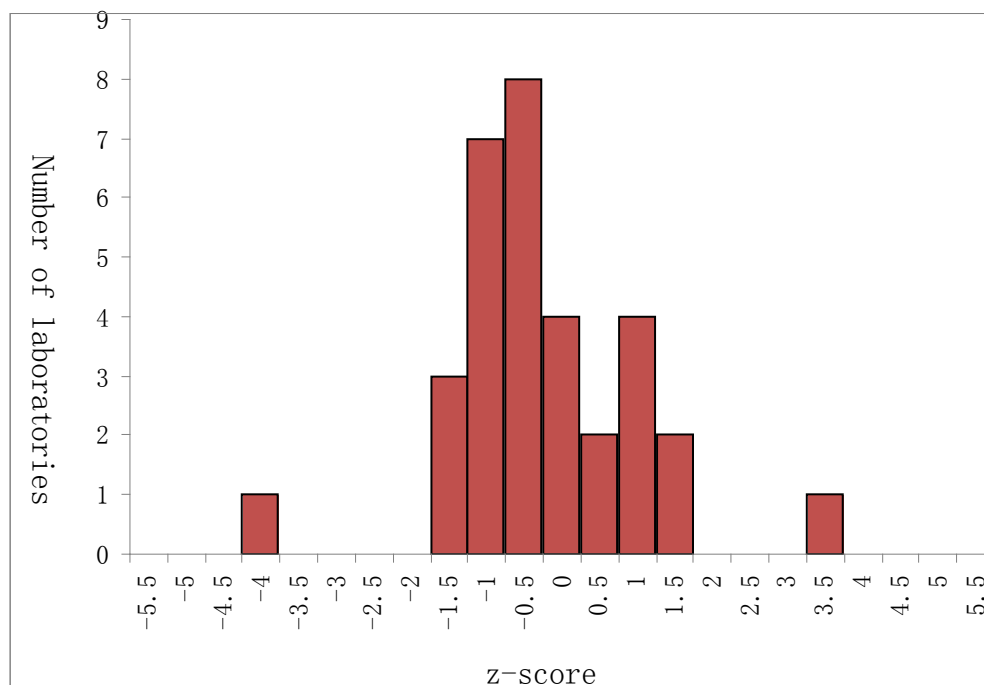
Histograms of z-scores (B.1~B.10) show the distributions of testing results.

Ordered z-score bar charts (B.11~B.20) are given in order to facilitate visual identification of the questionable and unsatisfactory results.

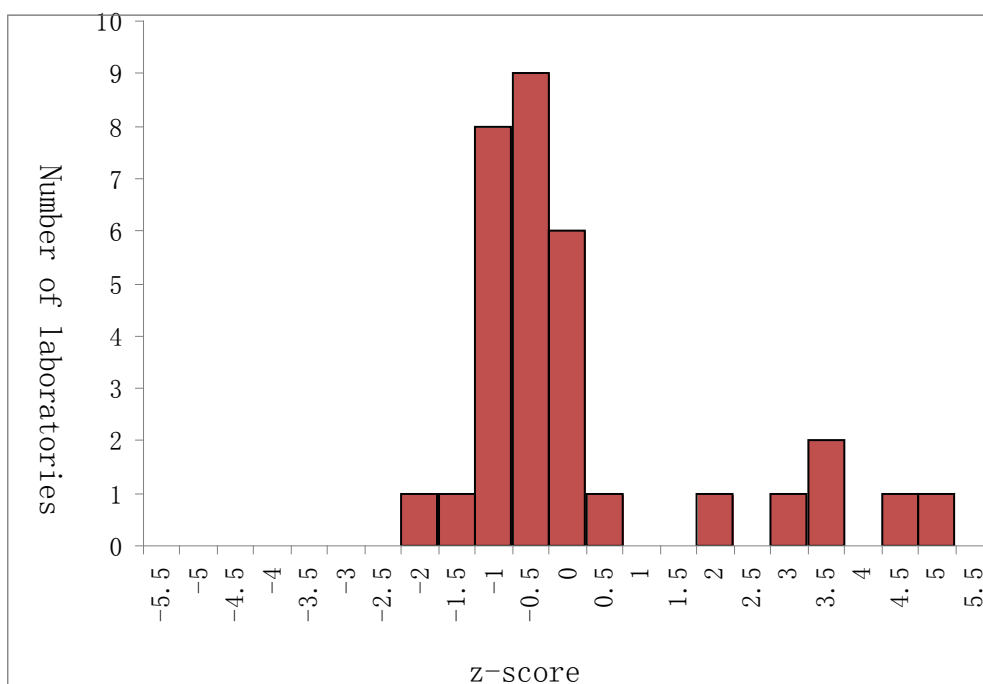
Youden Plots of paired z-scores (B.21~B.22) distinctly reveal questionable and unsatisfactory results, points outside the ellipses indicate their well-separated from the rest of the data, where points far out along the major axis of the ellipses imply that the results are subject to bias or large variation, and those points far away from the major axis represent participants whose repeatability is poor. Generally, labs with large absolute between-labs z-scores should be outside the ellipse in quadrants 2 and 4, while labs with large absolute within-lab z-scores should be outside the ellipse in quadrants 1 and 3. Some points are too far away from the ellipses to be drawn on the Youden Plots, the corresponding lab codes are drawn together with arrows that indicate their orientations.

The Plots of range against average of replicate testing results (B.23~B.28) show unusual dispersion of testing results, points outside the outer ring (which might not shown in the plots due to range axis scale cut down) indicate poor repeatability of testing results. The participants who obtained the results should investigate the random factors which influence the results.

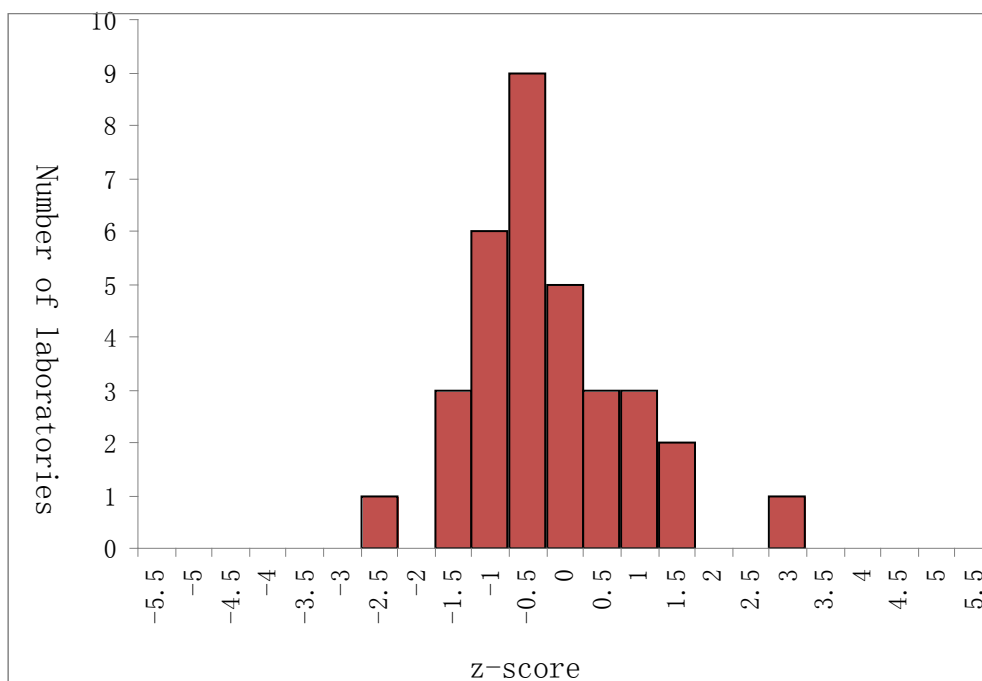
B.1 Distribution of Testing Results on Tartrazine Content in Sample C**B.2 Distribution of Testing Results on Tartrazine Content in Sample D**

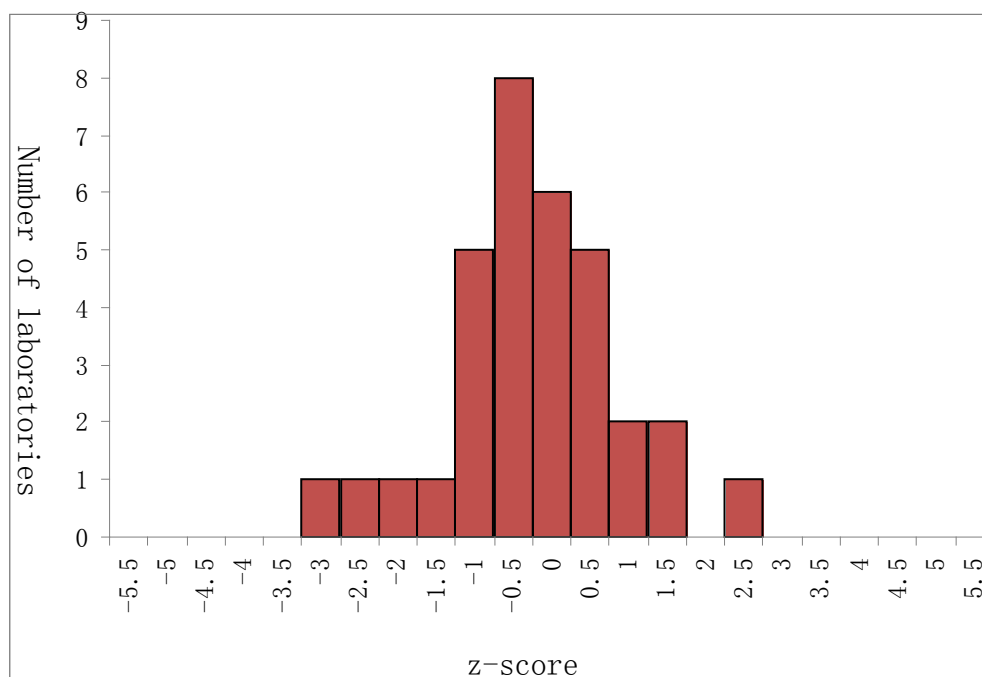
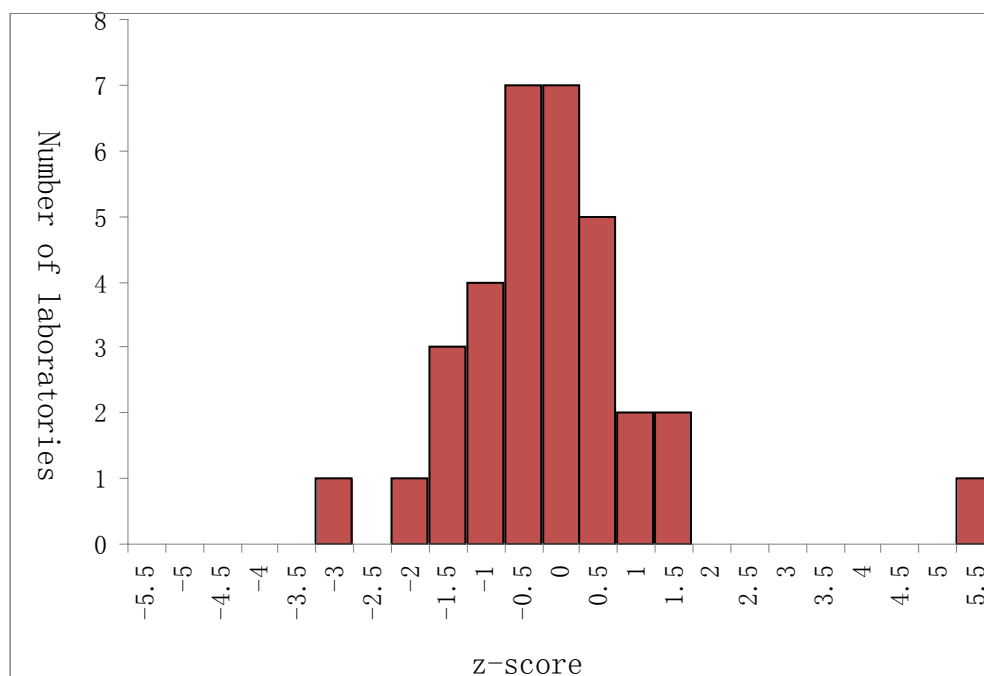
B.3 Distribution of Testing Results on Tartrazine Content in Sample E**B.4 Distribution of Normalized Sum of Paired Results C and E on Tartrazine Content**

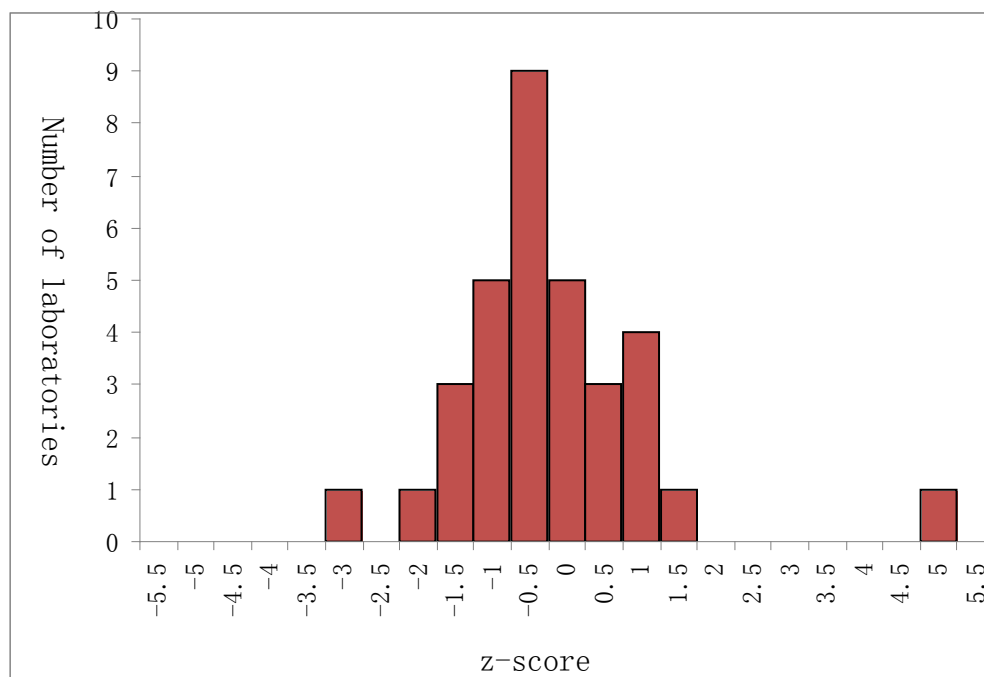
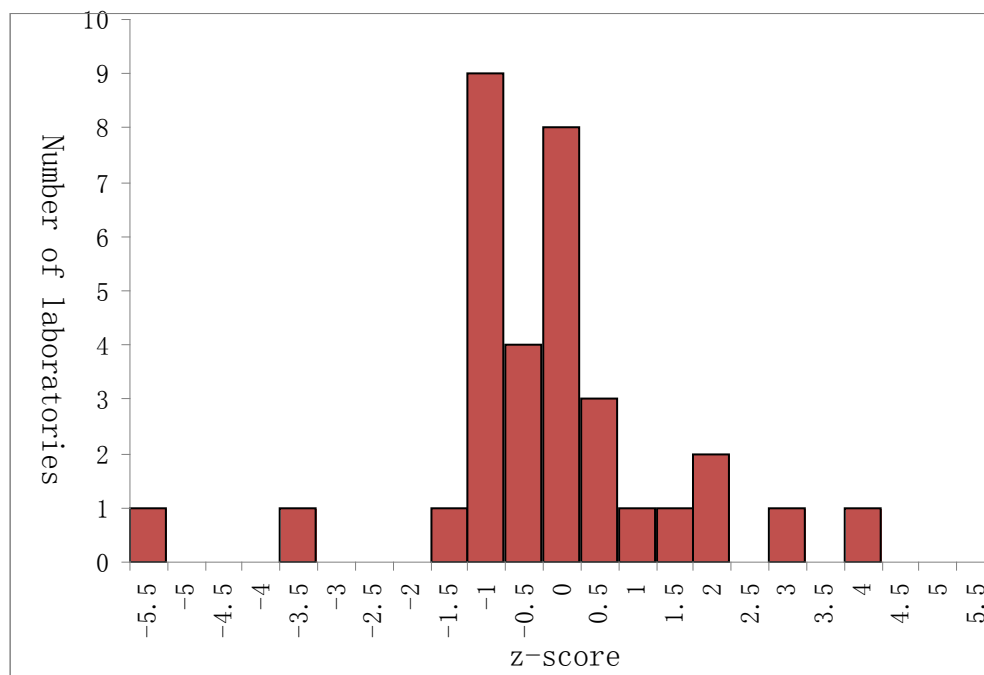
B.5 Distribution of Normalized Difference of Paired Results C and E on Tartrazine Content



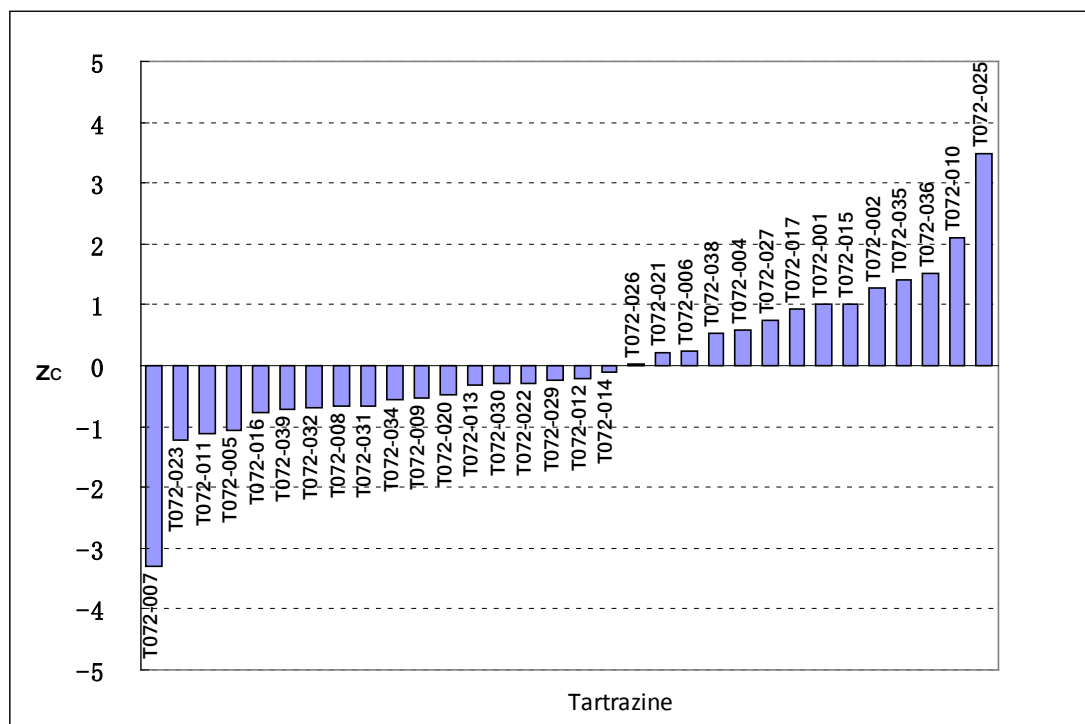
B.6 Distribution of Testing Results on Sunset Yellow Content in Sample C



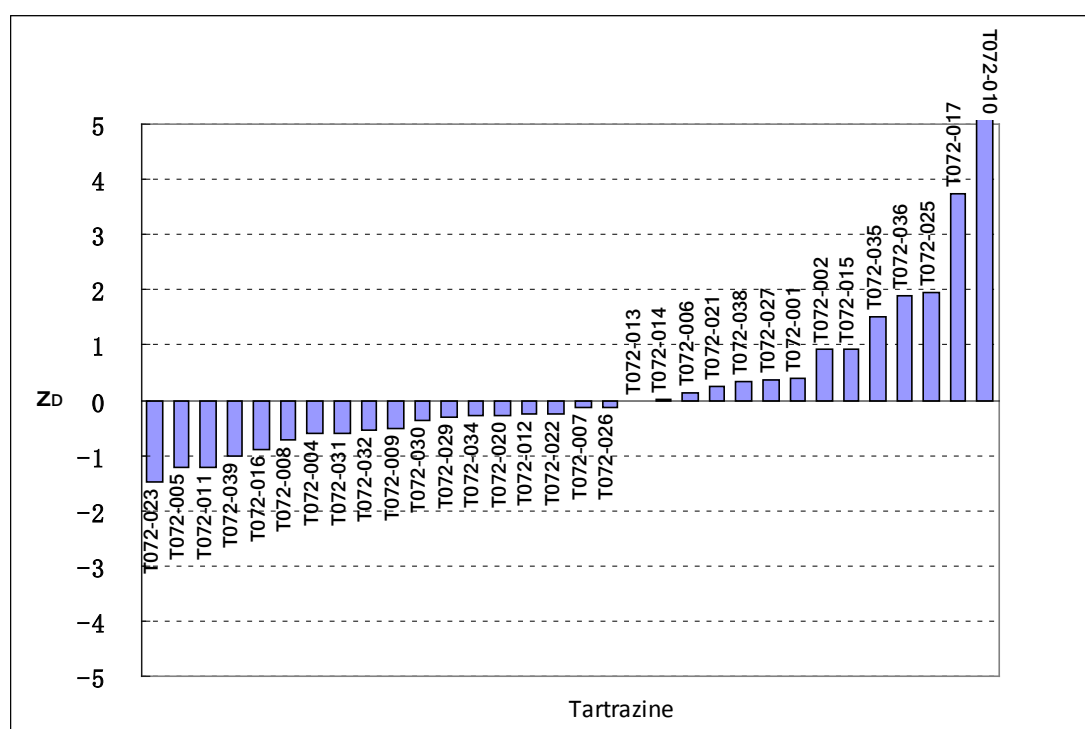
B.7 Distribution of Testing Results on Sunset Yellow Content in Sample D**B.8 Distribution of Testing Results on Sunset Yellow Content in Sample E**

B.9 Distribution of Normalized Sum of Paired Results C and E on Sunset Yellow Content**B.10 Distribution of Normalized Difference of Paired Results C and E on Sunset Yellow Content**

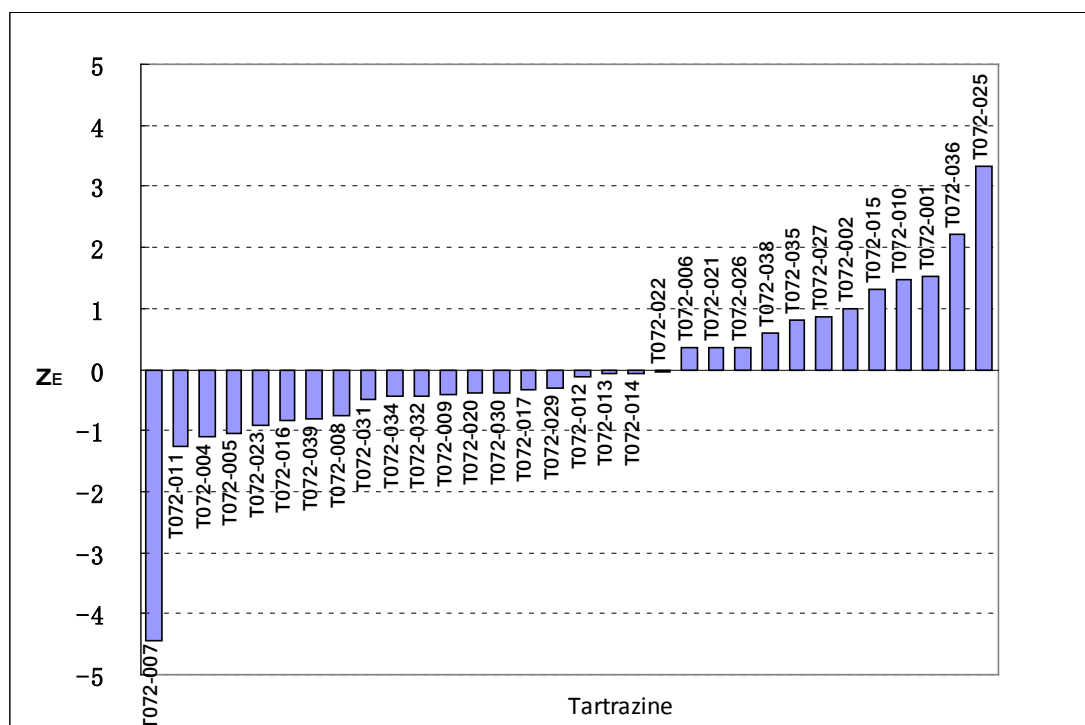
B.11 Ordered z-scores Bar Chart of Testing Results on Tartrazine Content in Sample C



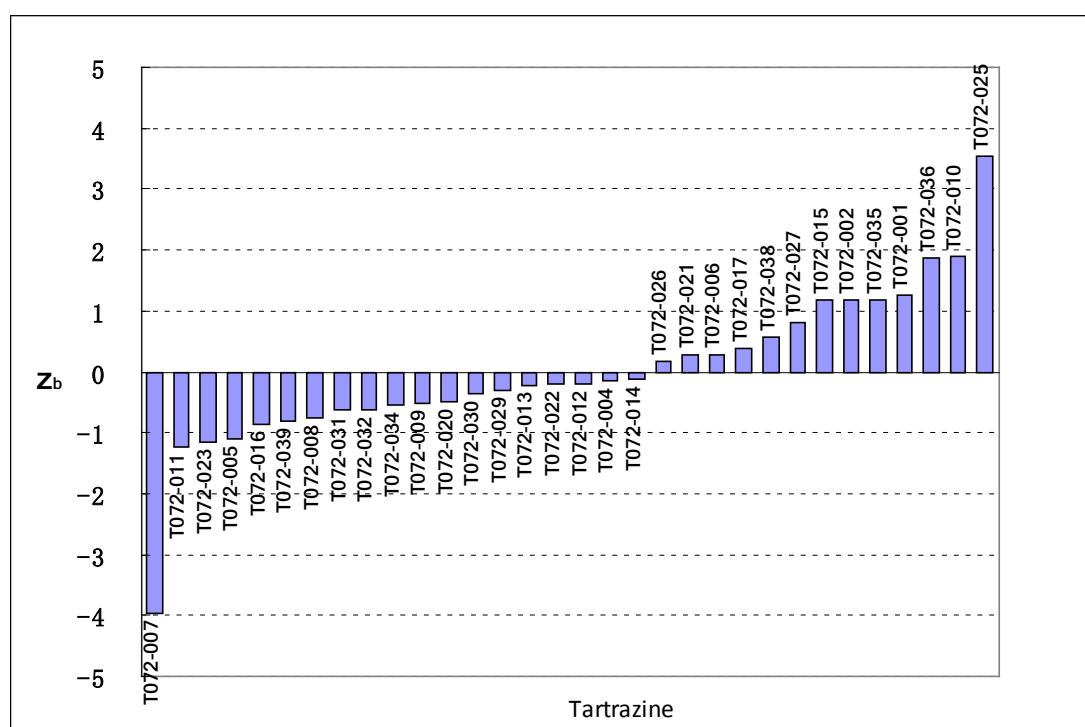
B.12 Ordered z-scores Bar Chart of Testing Results on Tartrazine Content in Sample D



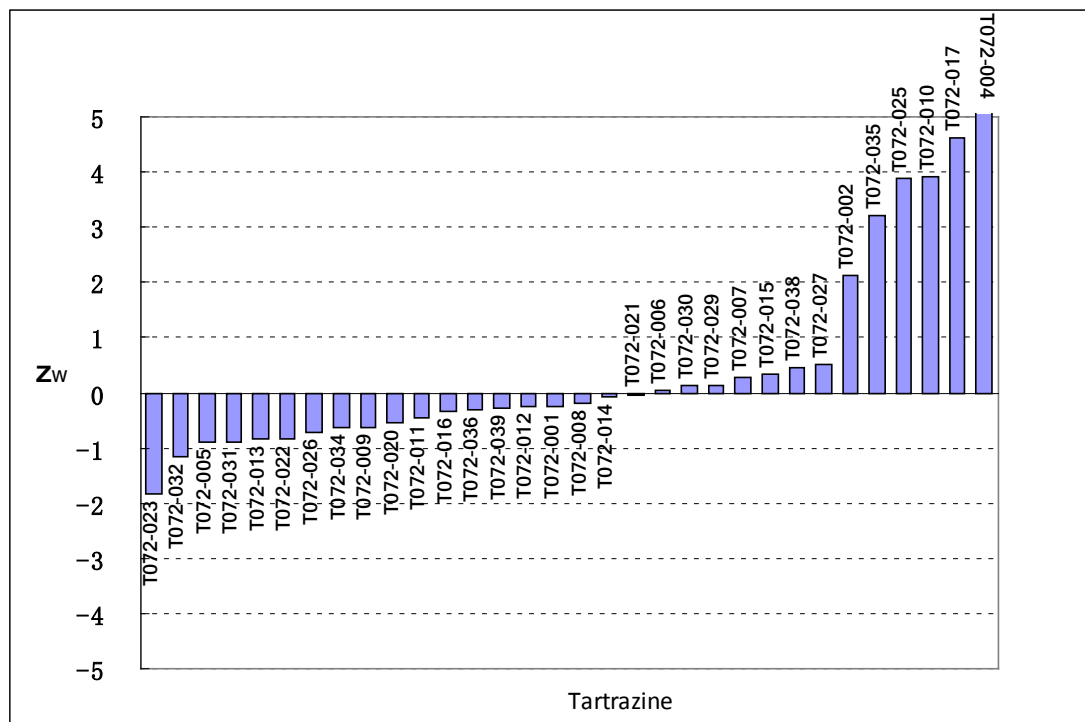
B.13 Ordered z-scores Bar Chart of Testing Results on Tartrazine Content in Sample E



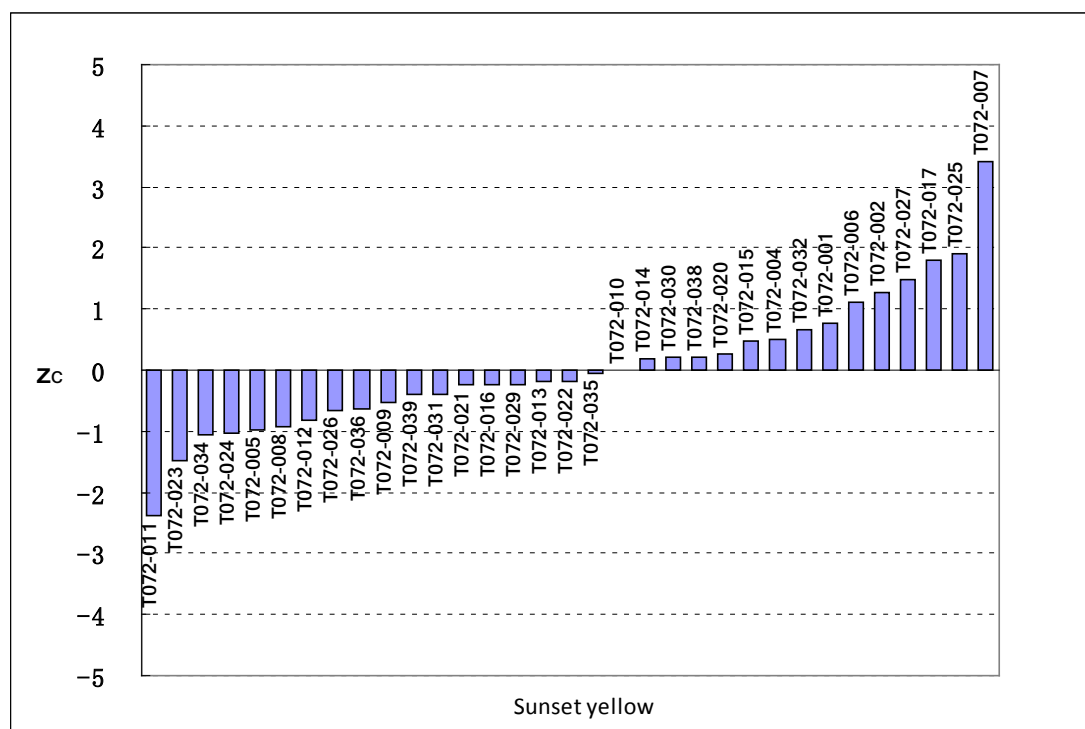
B.14 Ordered Between-labs z-scores Bar Chart of Paired Results C and E on Tartrazine Content



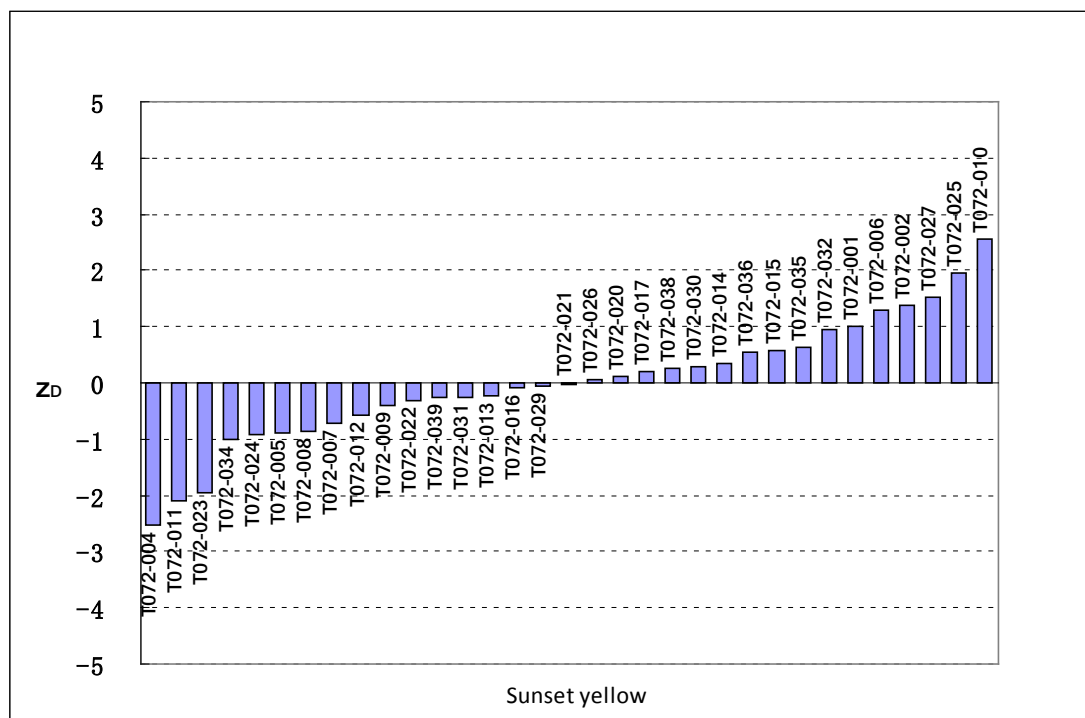
B.15 Ordered Within-lab z-scores Bar Chart of Paired Results C and E on Tartrazine Content



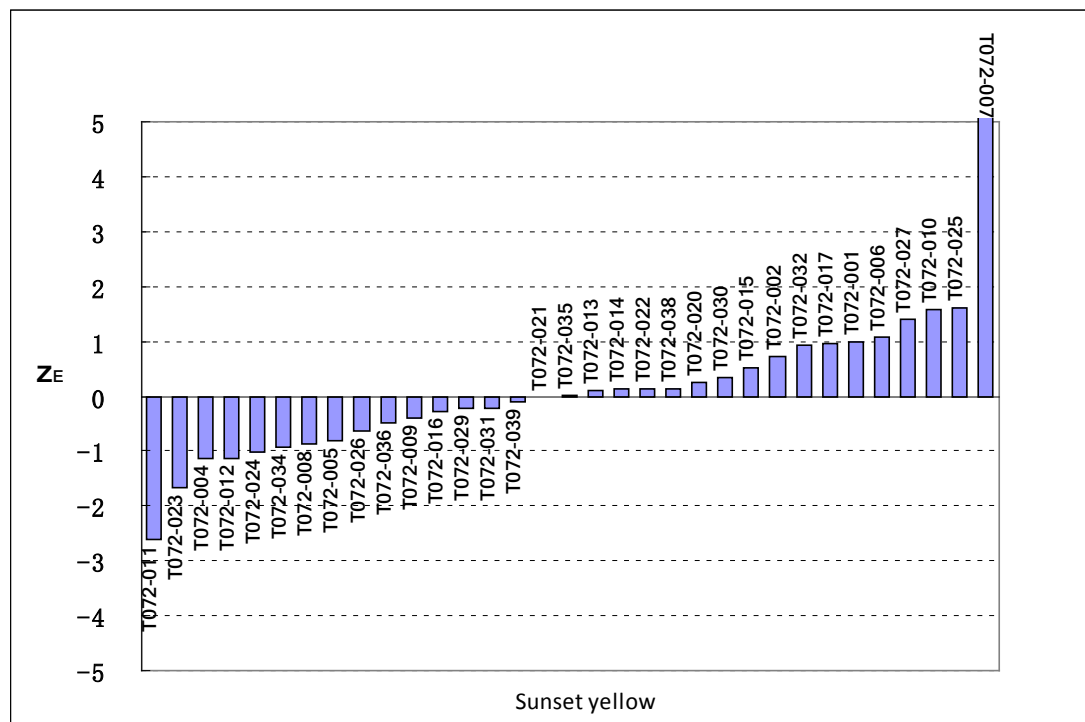
B.16 Ordered z-scores Bar Chart of Testing Results on Sunset Yellow Content in Sample C



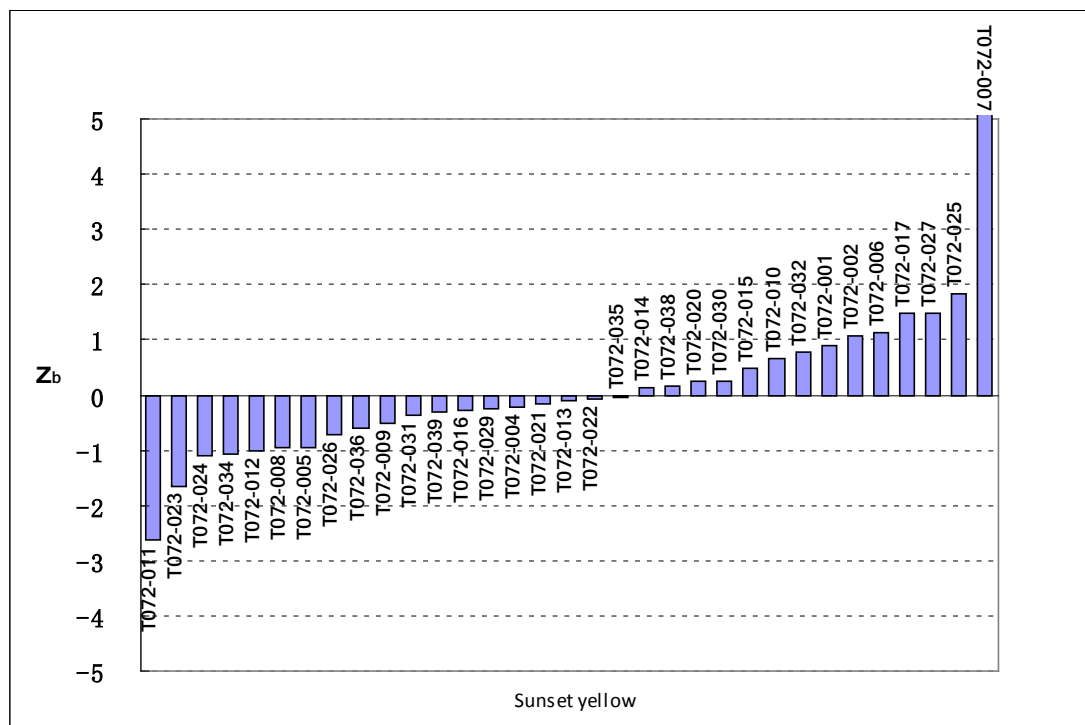
B.17 Ordered z-scores Bar Chart of Testing Results on Sunset Yellow Content in Sample D



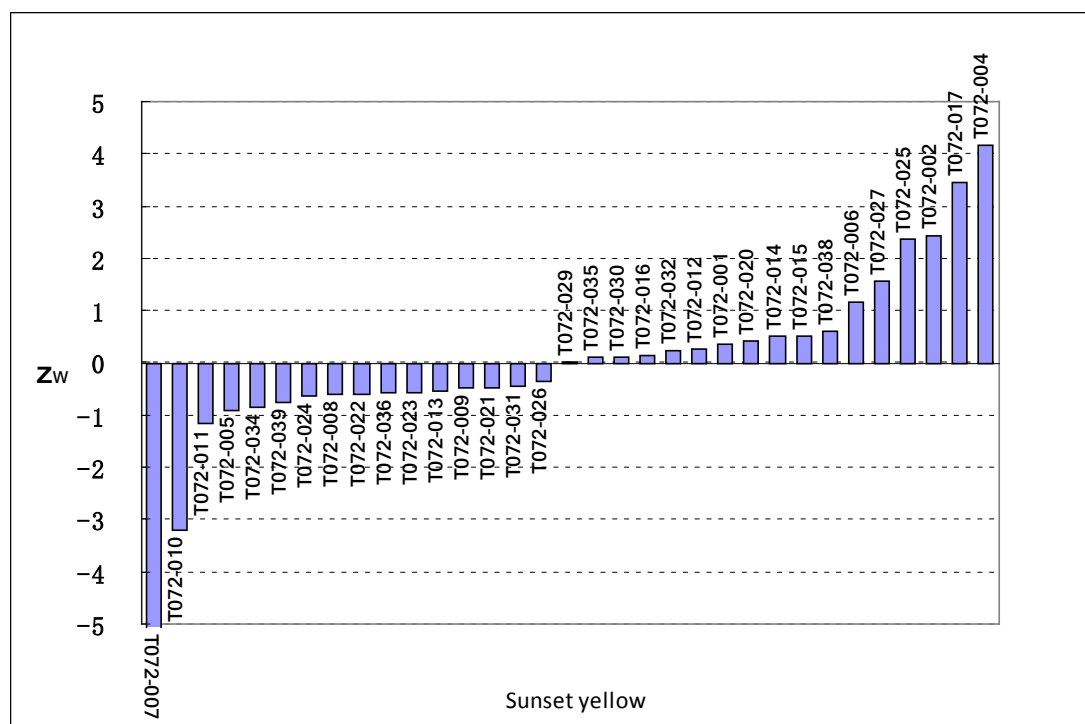
B.18 Ordered z-scores Bar Chart of Testing Results on Sunset Yellow Content in Sample E



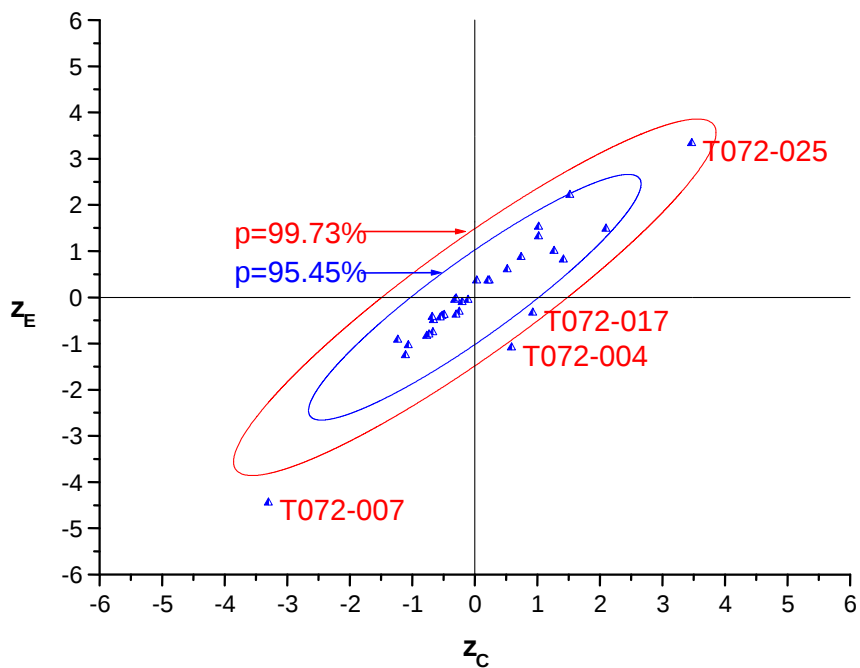
B.19 Ordered Between-labs z-scores Bar Chart of Paired Results C and E on Sunset Yellow Content



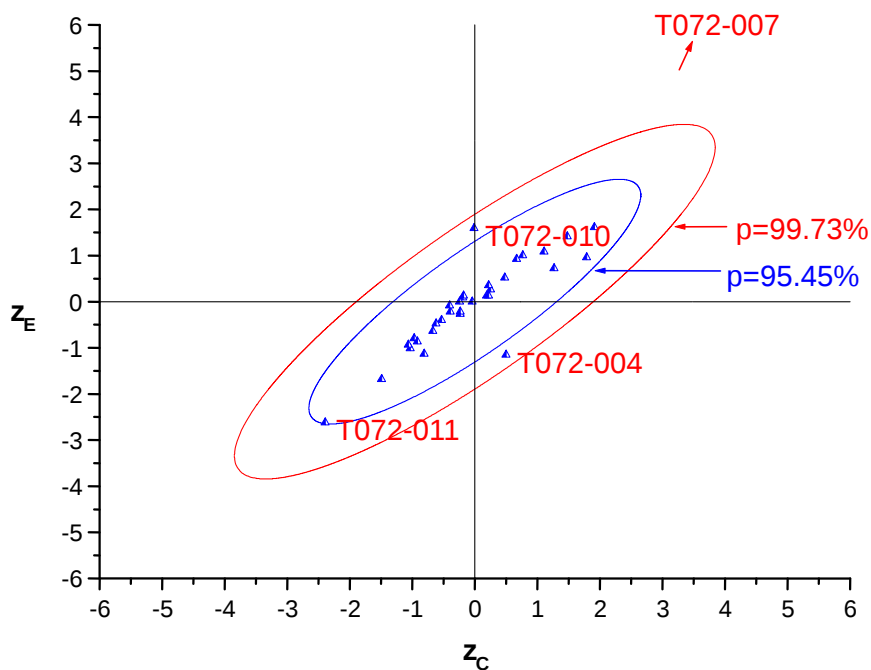
B.20 Ordered Within-lab z-scores Bar Chart of Paired Results C and E on Sunset Yellow Content



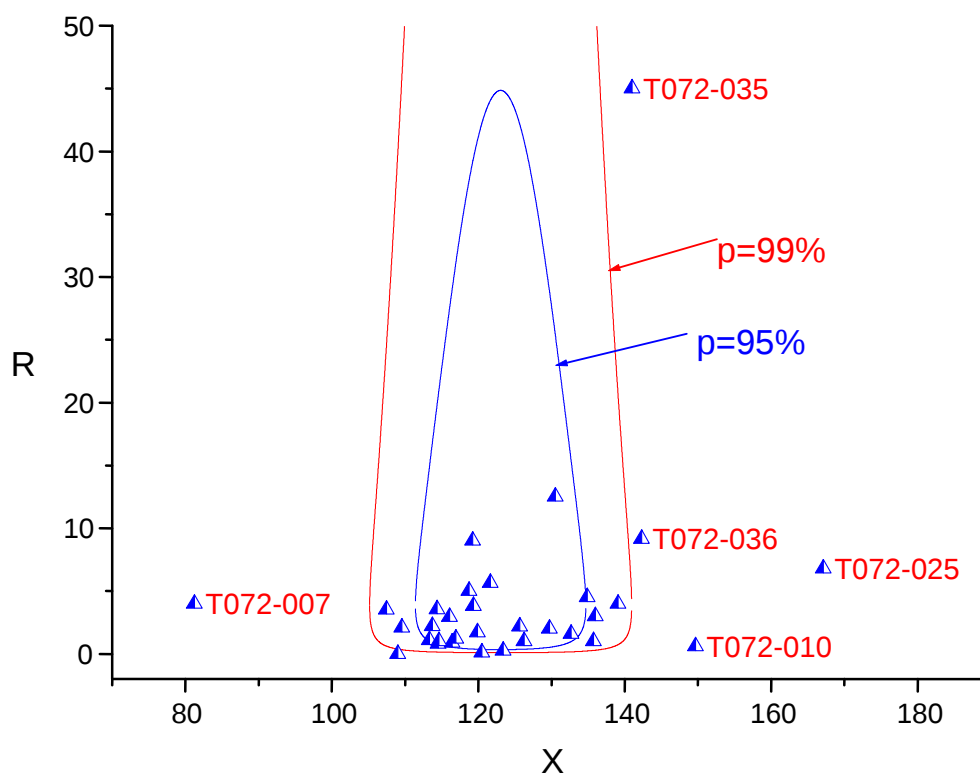
B.21 Youden Plot of z-scores of Tartrazine Results



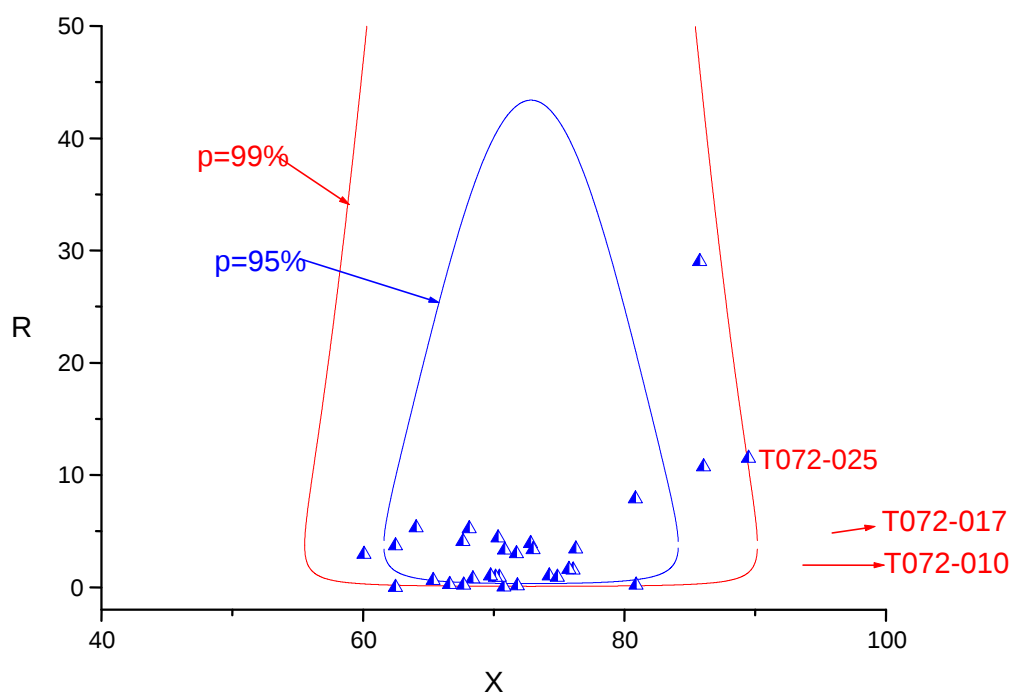
B.22 Youden Plot of z-scores of Sunset Yellow Results



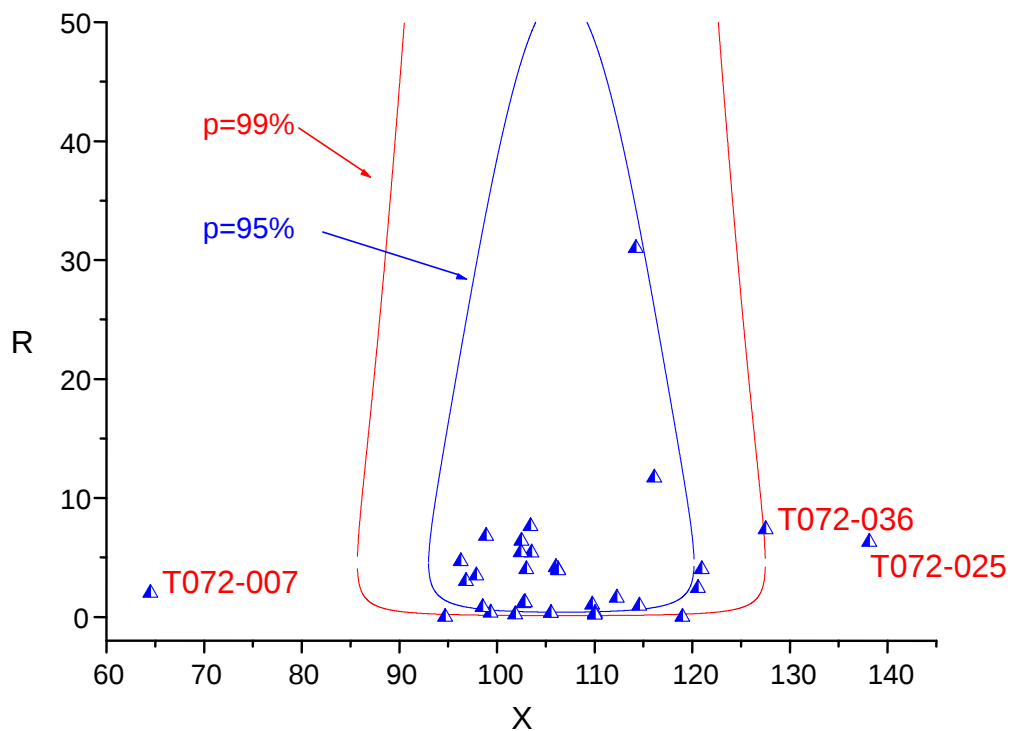
B.23 Plot of Ranges against Averages on Tartrazine Contents in Sample C



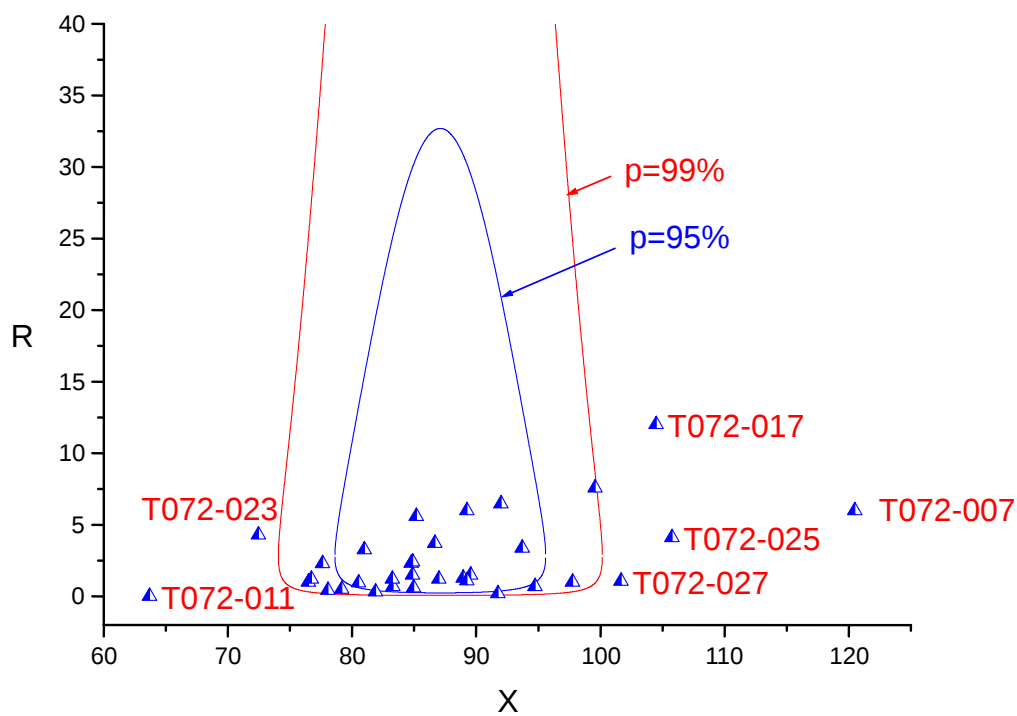
B.24 Plot of Ranges against Averages on Tartrazine Contents in Sample D



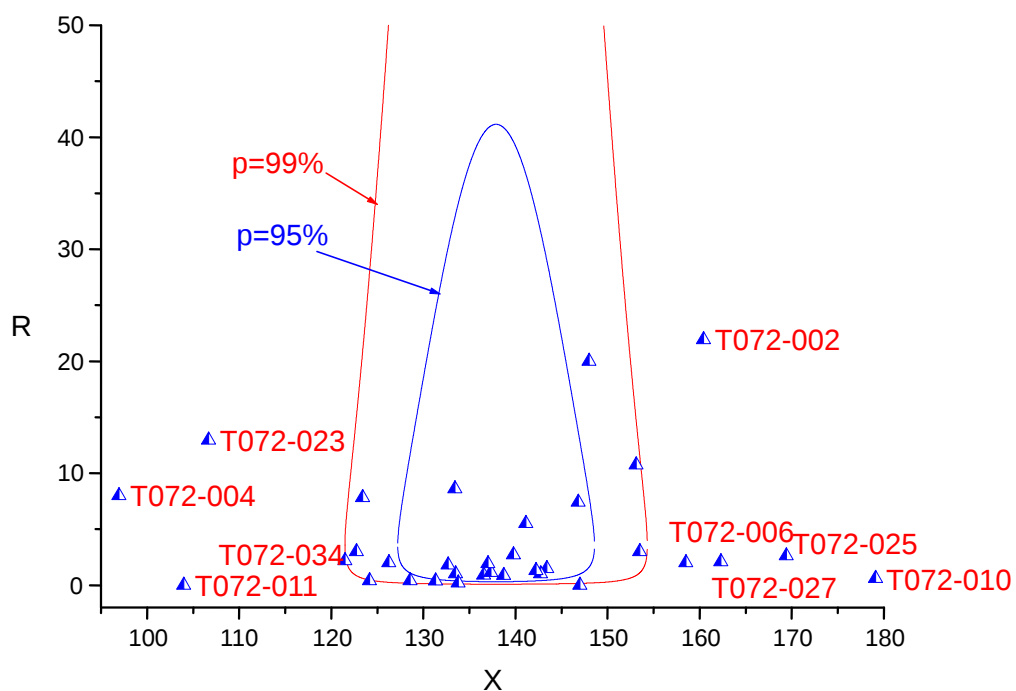
B.25 Plot of Ranges against Averages on Tartrazine Contents in Sample E



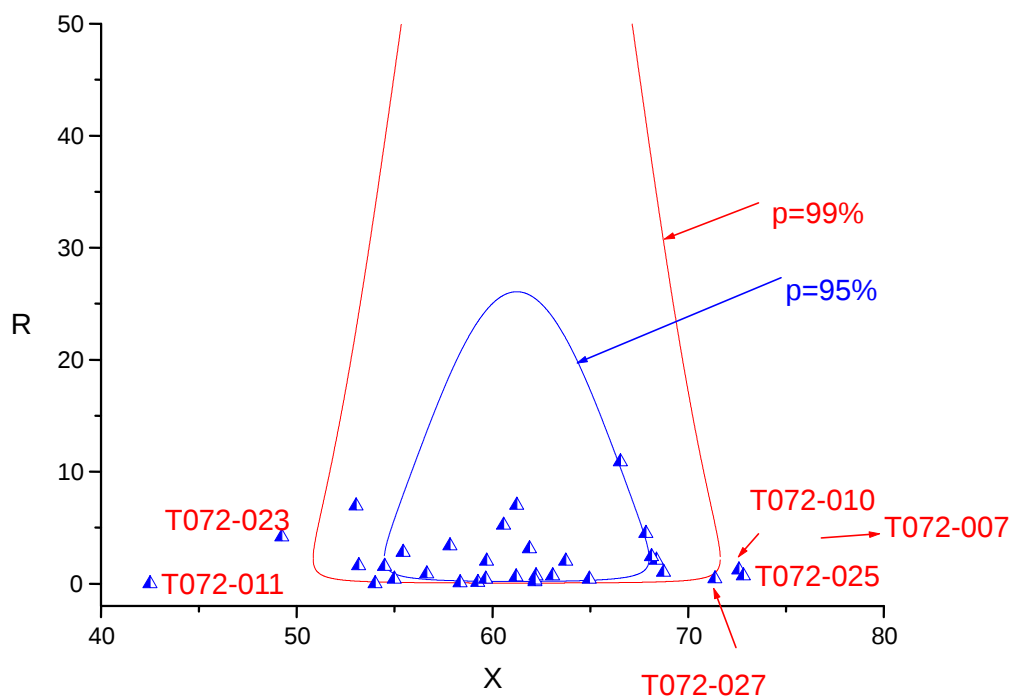
B.26 Plot of Ranges against Averages on Sunset Yellow Contents in Sample C



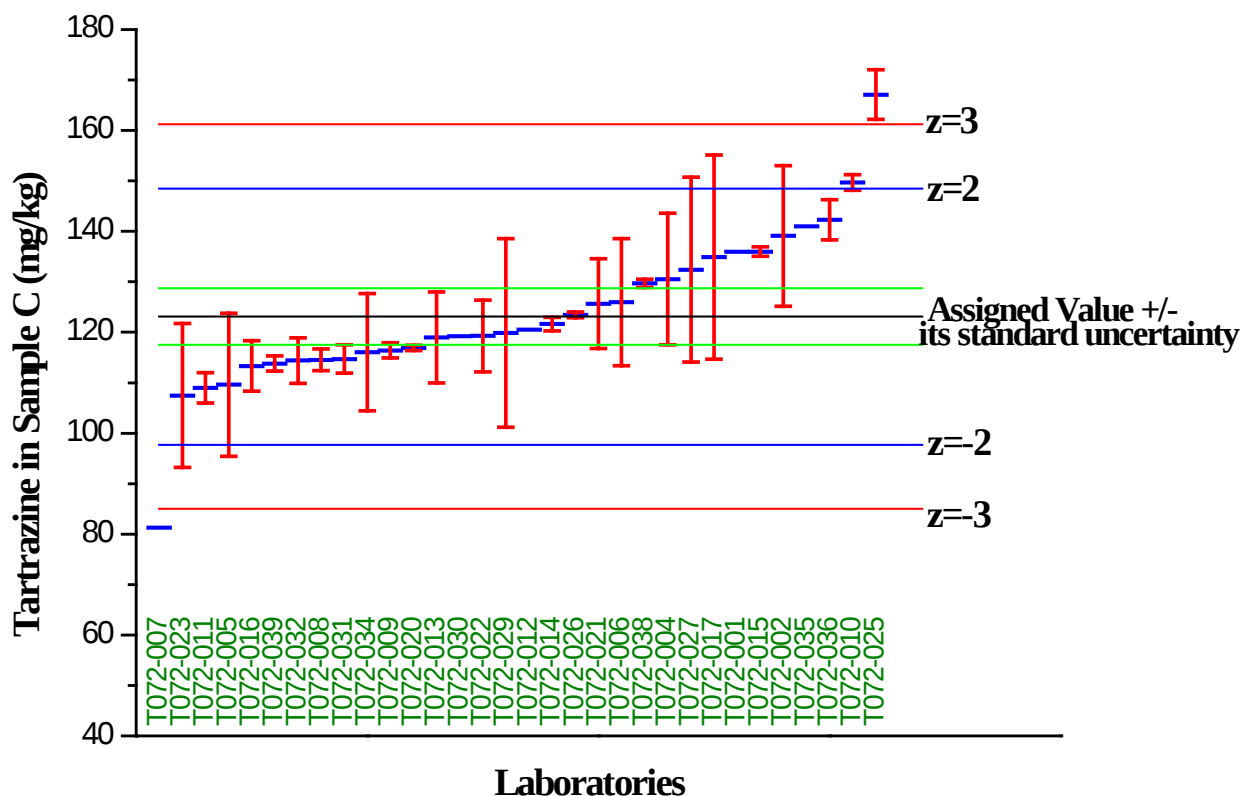
B.27 Plot of Ranges against Averages on Sunset Yellow Contents in Sample D



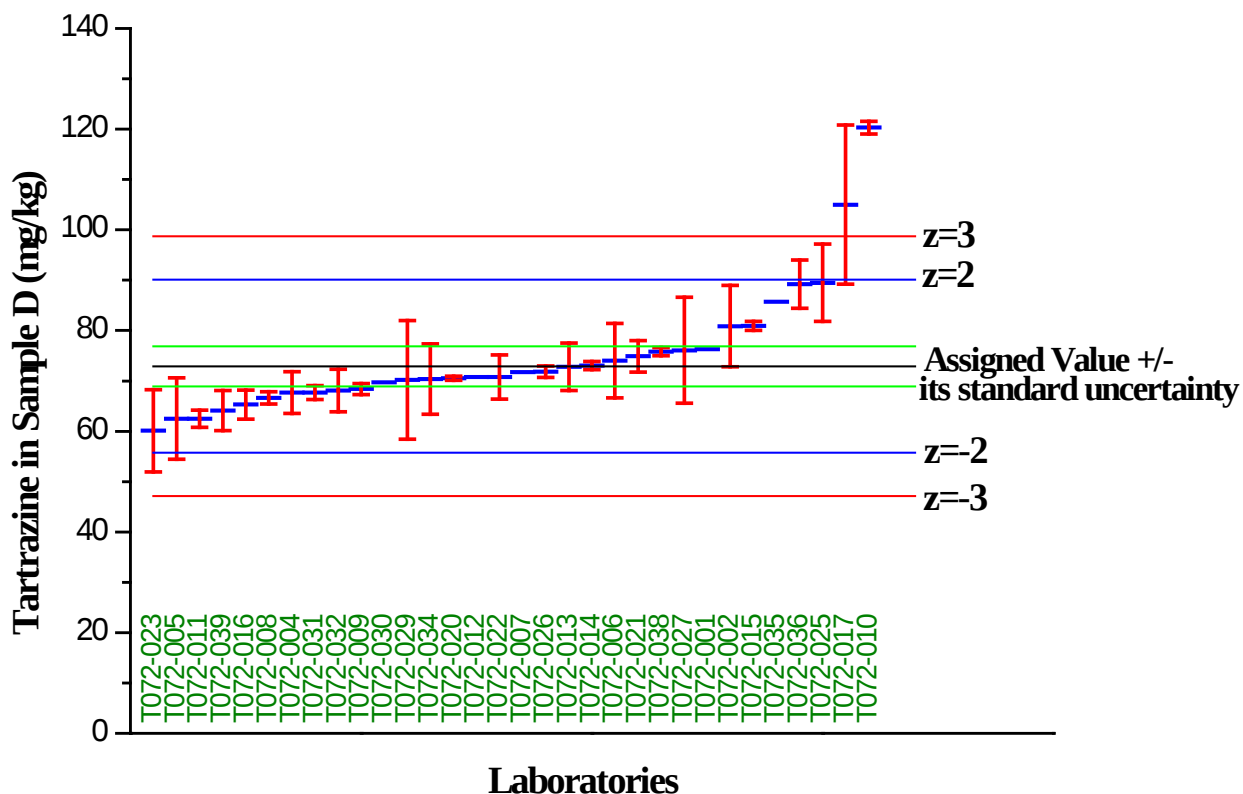
B.28 Plot of Ranges against Averages on Sunset Yellow Contents in Sample E



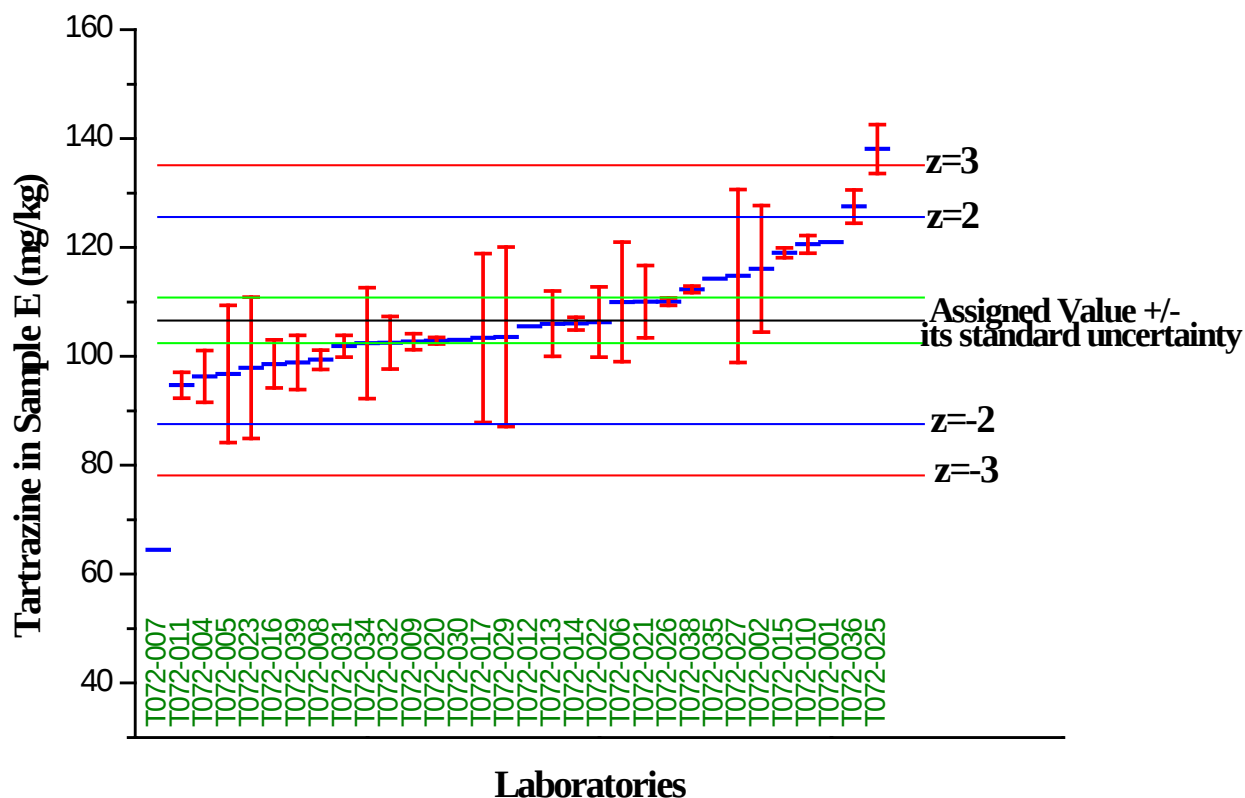
B.29 Ordered Results C on Tartrazine with Measurement Uncertainty



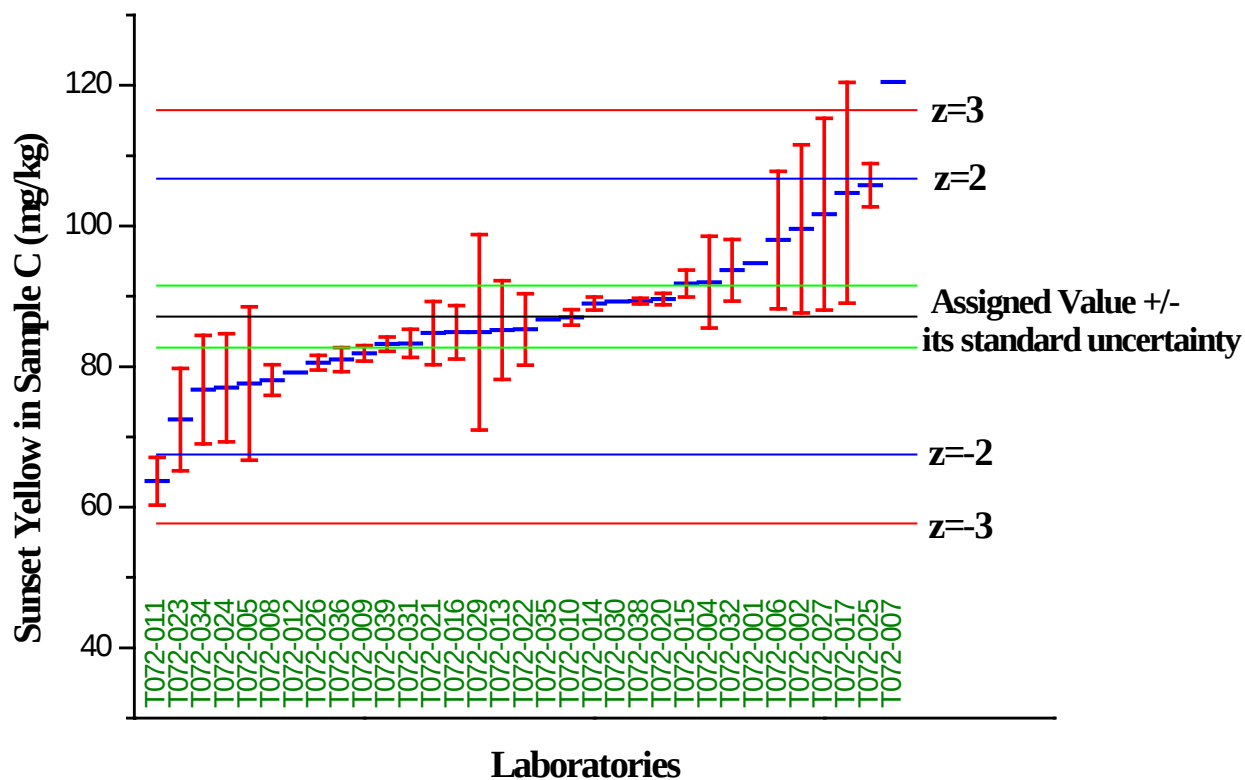
B.30 Ordered Results D on Tartrazine with Measurement Uncertainty

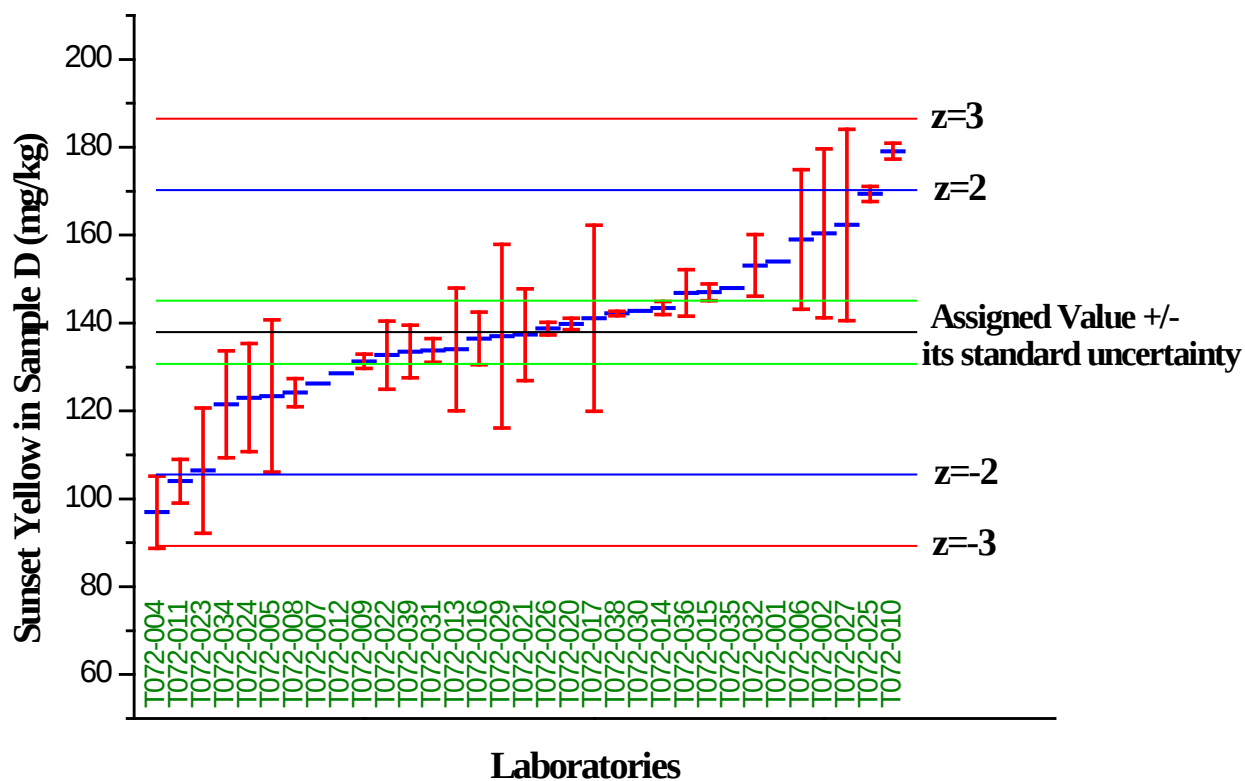
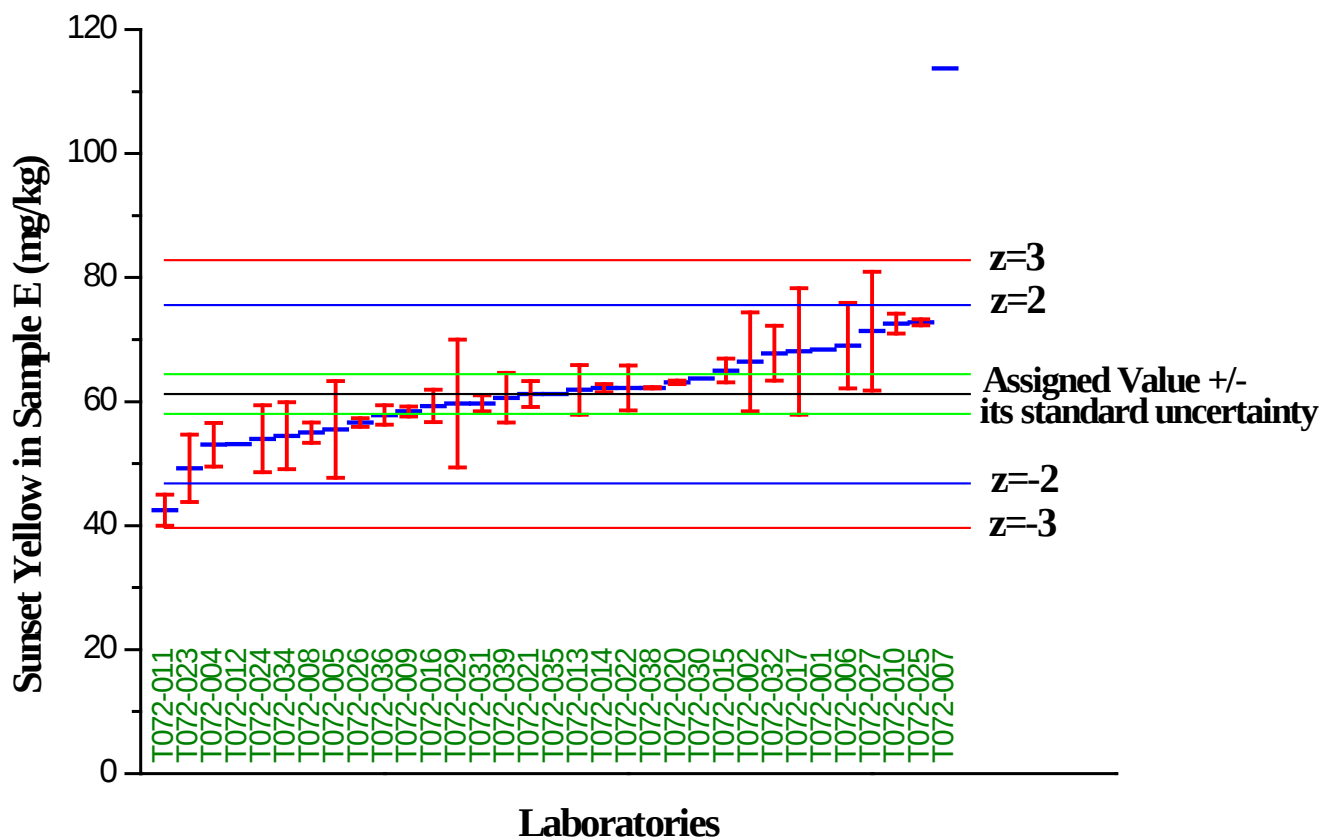


B.31 Ordered Results E on Tartrazine with Measurement Uncertainty



B.32 Ordered Results C on Sunset Yellow with Measurement Uncertainty



B.33 Ordered Results D on Sunset Yellow with Measurement Uncertainty**B.34 Ordered Results E on Sunset Yellow with Measurement Uncertainty**

APPENDIX C

Homogeneity and Stability Checks

C.1 Homogeneity Testing

For each gross sample, 10 samples were randomly selected and tested. Each sample was divided into two test portions, all test portions were tested in random order on the repeatability condition, that is to say, the testing was carried out during a short period by one person using the same testing method and apparatus in one laboratory. The results on each sample are listed in Table C.1~C.6.

The **between-samples standard deviations** (s_s) were calculated using the following formulae, where g is the number of samples used:

The **sample average** is $\bar{x}_t = (x_{t1} + x_{t2}) / 2$

The **between-test-portion range** is $w_t = |x_{t1} - x_{t2}|$

General average is $\bar{\bar{x}} = \sum \bar{x}_t / g$

The standard deviation of general average is $s_x = \sqrt{\sum (\bar{x}_t - \bar{\bar{x}})^2 / (g - 1)}$

The within-sample standard deviation is $s_w = \sqrt{\sum w_t^2 / 2g}$

The between-samples standard deviation is $s_s = \sqrt{s_x^2 - s_w^2 / 2}$

Table C.1 Homogeneity Testing Results of Tartrazine Sample C

Sample	$x_{t1}(\text{mg/kg})$	$x_{t2}(\text{mg/kg})$	$\bar{x}_t (\text{mg/kg})$	$w_t(\text{mg/kg})$	$w_t^2, (\text{mg/kg})^2$
1	116.29	116.90	116.6	0.61	0.3721
2	117.31	116.29	116.8	1.02	1.0404
3	117.62	117.17	117.4	0.45	0.2025
4	116.71	115.17	115.9	1.54	2.3716
5	116.22	115.78	116.0	0.44	0.1936
6	118.15	117.13	117.6	1.02	1.0404
7	116.42	116.42	116.4	0.00	0.0000
8	116.95	117.92	117.4	0.97	0.9409
9	117.50	116.85	117.2	0.65	0.4225
10	118.22	116.37	117.3	1.85	3.4225
$\bar{\bar{x}} = 116.87\text{mg/kg}$, Robust Mean=121.15mg/kg					
$s_x = 0.61\text{mg/kg}$, $s_w = 0.71\text{mg/kg}$, $s_s = 0.35\text{mg/kg}$, Robust SD=26.23mg/kg,					
$s_s/\text{Robust SD} = 0.013 < 0.3$					

Table C.2 Homogeneity Testing Results of Tartrazine Sample D

Sample	$x_{t1}(\text{mg/kg})$	$x_{t2}(\text{mg/kg})$	$\bar{x}_t (\text{mg/kg})$	$w_t(\text{mg/kg})$	$w_t^2, (\text{mg/kg})^2$
1	67.25	68.10	67.68	0.85	0.7225
2	70.25	69.49	69.87	0.76	0.5776
3	68.40	68.58	68.49	0.18	0.0324
4	68.59	68.29	68.44	0.30	0.09
5	68.70	68.72	68.71	0.02	0.0004
6	68.46	67.35	67.91	1.11	1.2321
7	67.60	68.28	67.94	0.68	0.4624
8	68.61	68.16	68.39	0.45	0.2025
9	68.07	67.63	67.85	0.44	0.1936
10	69.01	67.26	68.14	1.75	3.0625
$\bar{\bar{x}} = 68.34\text{mg/kg}$, Robust Mean=72.68mg/kg					
$s_x = 0.63\text{mg/kg}$, $s_w = 0.57\text{mg/kg}$, $s_s = 0.48\text{mg/kg}$, Robust SD=18.87mg/kg,					
$s_s/\text{Robust SD} = 0.030 < 0.3$					

Table C.3 Homogeneity Testing Results of Tartrazine Sample E

Sample	$x_{t1}(\text{mg/kg})$	$x_{t2}(\text{mg/kg})$	$\bar{x}_t (\text{mg/kg})$	$w_t(\text{mg/kg})$	$w_t^2, (\text{mg/kg})^2$
1	105.10	104.43	104.77	0.67	0.4489
2	103.88	103.79	103.84	0.09	0.0081
3	103.50	104.51	104.01	1.01	1.0201
4	103.23	103.29	103.26	0.06	0.0036
5	104.75	105.27	105.01	0.52	0.2704
6	105.30	105.60	105.45	0.30	0.0900
7	103.87	105.89	104.88	2.02	4.0804
8	104.53	104.76	104.65	0.23	0.0529
9	104.93	103.11	104.02	1.82	3.3124
10	105.51	105.72	105.62	0.21	0.0441
$\bar{\bar{x}} = 104.55\text{mg/kg}$, Robust Mean=104.59mg/kg					
$s_x = 0.75\text{g/kg}$, $s_w = 0.68\text{mg/kg}$, $s_s = 0.57\text{mg/kg}$, Robust SD=21.97mg/kg,					
$s_s/\text{Robust SD} = 0.0026 < 0.3$					

Table C.4 Homogeneity Testing Results of Sunset Yellow Sample C

Sample	$x_{t1}(\text{mg/kg})$	$x_{t2}(\text{mg/kg})$	$\bar{x}_t (\text{mg/kg})$	$w_t(\text{mg/kg})$	$w_t^2, (\text{mg/kg})^2$
1	81.73	79.10	80.42	2.63	6.9169
2	81.42	81.56	81.49	0.14	0.0196
3	81.95	83.28	82.62	1.33	1.7689
4	80.19	81.16	80.68	0.97	0.9409
5	81.19	80.45	80.82	0.74	0.5476
6	84.13	82.53	83.33	1.60	2.5600
7	82.11	82.11	82.11	0.00	0.0000
8	81.32	81.74	81.53	0.42	0.1764
9	82.53	81.98	82.26	0.55	0.3025
10	82.21	80.83	81.52	1.38	1.9044
$\bar{\bar{x}}=81.68\text{mg/kg}$, Robust Mean=84.54mg/kg					
$s_x=0.92\text{g/kg}$, $s_w=0.87\text{mg/kg}$, $s_s=0.68\text{mg/kg}$, Robust SD=17.55mg/kg,					
$s_s/\text{Robust SD}=0.038<0.3$					

Table C.5 Homogeneity Testing Results of Sunset Yellow Sample D

Sample	$x_{t1}(\text{mg/kg})$	$x_{t2}(\text{mg/kg})$	$\bar{x}_t (\text{mg/kg})$	$w_t(\text{mg/kg})$	$w_t^2, (\text{mg/kg})^2$
1	134.08	132.39	133.2	1.69	2.8561
2	131.35	129.22	130.3	2.13	4.5369
3	130.27	132.43	131.4	2.16	4.6656
4	132.37	133.04	132.7	0.67	0.4489
5	130.77	132.28	131.5	1.51	2.2801
6	133.38	129.77	131.6	3.61	13.0321
7	131.06	130.50	130.8	0.56	0.3136
8	129.38	131.81	130.6	2.43	5.9049
9	133.52	131.98	132.8	1.54	2.3716
10	134.69	132.31	133.5	2.38	5.6644
$\bar{\bar{x}}=131.83\text{mg/kg}$, Robust Mean=134.72mg/kg					
$s_x=1.14\text{g/kg}$, $s_w=1.45\text{mg/kg}$, $s_s=0.51\text{mg/kg}$, Robust SD=29.99mg/kg,					
$s_s/\text{Robust SD}=0.017<0.3$					

Table C.6 Homogeneity Testing Results of Sunset Yellow Sample E

Sample	$x_{t1}(\text{mg/kg})$	$x_{t2}(\text{mg/kg})$	$\bar{x}_t (\text{mg/kg})$	$w_t(\text{mg/kg})$	$w_t^2, (\text{mg/kg})^2$
1	60.53	60.42	60.48	0.11	0.0121
2	59.02	59.13	59.08	0.11	0.0121
3	57.78	60.58	59.18	2.80	7.8400
4	58.85	57.80	58.33	1.05	1.1025
5	60.57	60.61	60.59	0.04	0.0016
6	59.80	59.85	59.83	0.05	0.0025
7	59.23	60.19	59.71	0.96	0.9216
8	59.90	59.35	59.63	0.55	0.3025
9	60.51	56.88	58.70	3.63	13.1769
10	60.51	60.39	60.45	0.12	0.0144
$\bar{\bar{x}}=59.60\text{mg/kg}$, Robust Mean=59.45mg/kg					
$s_x=0.78\text{g/kg}$, $s_w=1.08\text{mg/kg}$, $s_s=0.14\text{mg/kg}$, Robust SD=12.41mg/kg,					
$s_s/\text{Robust SD}=0.011<0.3$					

Comparing the between-samples standard deviation (s_s) with the corresponding standard deviation for proficiency assessment (Robust SD), all s_s are less than 0.3 Robust SD, it indicates that the variation between samples could be neglected in subsequently statistical analysis and proficiency assessments, in another word, the samples are homogenous enough.

C.2 Stability Testing

For Gross Sample C, D and E, 12 packaged testing samples were selected for stability testing, respectively. The samples were placed at room temperature (18~25 degrees C) for 2 days, then putted into an attemperator with a temperature of 40 degrees C for 7 days and placed at about 4 degrees C until stability testing finished, on the date 30, 60 and 100 beginning from the day of homogeneity testing, every 3 samples per date were tested on Sunset Yellow and Tartrazine contents.

For all stability testing, the person, apparatus, testing method and laboratory were same as the homogeneity testing. The testing results of stability check are listed in Table C.7, every absolute differences from the general average of homogeneity tests were calculated using following formulae:

The sample average is $\bar{y}_t = (y_{t1} + y_{t2}) / 2$

General average is $\bar{\bar{y}} = \sum \bar{y}_t / g$

The difference between stability and homogeneity testing results is $|d| = |\bar{\bar{y}} - \bar{\bar{x}}|$

Comparing with the standard deviations for proficiency assessment (Robust SD) in this program, all maximal absolute differences between the stability testing results and the corresponding homogeneity testing results ($|d|$) for each sample are less than 0.3 Robust SD, it indicates that the variation of samples could be neglected in subsequently statistical analysis and proficiency assessments.

As a conclusion, all samples are stable enough for the purposes of this program.

Table C.7 Stability Testing Results

Unit: mg/kg

Tartrazine Sample C						Tartrazine Sample D					
Day	y _{t1}	y _{t2}	$\bar{y}_t$	$\bar{\bar{y}}$	d	Day	y _{t1}	y _{t2}	$\bar{y}_t$	$\bar{\bar{y}}$	d
30	114.08	116.89	114.08	113.36	3.51	30	69.47	66.21	67.84	68.90	0.56
	111.82	111.71	111.82				70.31	68.62	69.47		
	112.39	113.28	112.39				68.65	70.12	69.39		
60	115.07	114.18	115.07	114.38	2.49	60	68.35	69.24	68.79	69.82	1.48
	113.49	113.17	113.49				71.32	69.41	70.37		
	114.50	115.85	114.50				70.53	70.06	70.30		
100	113.90	112.90	113.90	113.28	3.59	100	68.70	70.31	69.51	69.60	1.26
	114.21	112.62	114.21				70.45	69.84	70.15		
	112.65	113.39	112.65				69.35	68.95	69.15		
$\bar{\bar{x}}$ =116.87, Robust SD =26.23, max d / Robust SD =0.14<0.3						$\bar{\bar{x}}$ =68.34, Robust SD =18.87, max d / Robust SD =0.078<0.3					
Tartrazine Sample E						Sunset Yellow Sample C					
Day	y _{t1}	y _{t2}	$\bar{y}_t$	$\bar{\bar{y}}$	d	Day	y _{t1}	y _{t2}	$\bar{y}_t$	$\bar{\bar{y}}$	d
30	107.52	103.67	105.59	105.39	0.89	30	82.82	84.80	83.81	81.32	0.36
	106.32	105.62	105.97				79.91	79.95	79.93		
	102.89	106.35	104.62				80.67	79.75	80.21		
60	105.77	107.29	106.53	105.74	1.24	60	80.81	79.89	80.35	80.46	1.22
	107.36	105.32	106.34				79.25	79.90	79.58		
	103.48	105.24	104.36				81.32	81.58	81.45		
100	106.50	107.35	106.93	106.00	1.50	100	79.34	81.52	80.43	80.43	1.25
	104.68	107.29	105.99				79.35	80.03	79.69		
	105.62	104.53	105.08				78.68	79.86	79.27		
$\bar{\bar{x}}$ =104.55, Robust SD =21.97, max d / Robust SD =0.068<0.3						$\bar{\bar{x}}$ =81.68, Robust SD =17.55, max d / Robust SD =0.071<0.3					
Sunset Yellow Sample D						Sunset Yellow Sample E					
Day	y _{t1}	y _{t2}	$\bar{y}_t$	$\bar{\bar{y}}$	d	Day	y _{t1}	y _{t2}	$\bar{y}_t$	$\bar{\bar{y}}$	d
30	128.83	130.10	129.46	130.70	0.90	30	61.58	59.02	60.30	60.97	1.37
	130.59	132.56	131.58				63.10	60.12	61.61		
	129.87	132.25	131.06				60.35	61.64	61.00		
60	130.95	129.28	130.12	130.63	0.97	60	59.50	58.36	58.93	60.30	0.70
	131.87	129.76	130.82				62.13	60.72	61.43		
	129.36	132.54	130.95				59.68	61.42	60.55		
100	129.77	133.10	131.43	132.17	0.57	100	58.23	61.38	59.81	61.58	1.98
	131.23	132.51	131.87				63.21	63.75	63.48		
	133.85	132.59	133.22				61.35	61.54	61.45		
$\bar{\bar{x}}$ =131.83, Robust SD =29.99, max d / Robust SD =0.032<0.3						$\bar{\bar{x}}$ =59.60, Robust SD =12.41, max d / Robust SD =0.16<0.3					

APPENDIX D

Statistical Assessment Procedures

This appendix outlines the robust statistical assessment procedures used in this program.

D.1 Distribution of Testing Results

Since it was designed that all statistical assessments on participants' proficiency are based on the results obtained from participants, the assumption that the results follow a Normal distribution should be achieved. The histograms of z-scores for results in Appendix B.1~B.5 show approximate normal distribution, they were plotted by grouping and counting the numbers of z-scores with an interval of 0.5.

D.2 Summary Statistics Used in This Program

In most cases, robust statistics are not highly influenced by the extreme results comparing with the classical statistics. In this program, robust statistical method was used for evaluating the most important summary statistics, the robust mean and standard deviation, for proficiency assessment. The algorithm in this program is reproduced from ISO 5725-5 as follows:

Denote the p items of data, sorted into increasing order, by:

$$x_1, x_2, \dots, x_i, \dots, x_p$$

Denote the robust average and robust standard deviation of these data by x^ and s^* .*

Calculate initial values for x^ and s^* as:*

$$x^* = \text{median of } x_i \text{ (} i = 1, 2, \dots, p \text{)} \quad (\text{D.1})$$

$$s^* = 1.483 \text{ median of } |x_i - x^*| \text{ (} i = 1, 2, \dots, p \text{)} \quad (\text{D.2})$$

Update the values of x^ and s^* as follows. Calculate:*

$$\varphi = 1.5 s^* \quad (\text{D.3})$$

For each x_i ($i = 1, 2, \dots, p$), calculate:

$$x_i^* = \begin{cases} x^* - \varphi & \text{if } x_i < x^* - \varphi \\ x^* + \varphi & \text{if } x_i > x^* + \varphi \\ x^* & \text{if otherwise} \end{cases} \quad (\text{D.4})$$

Calculate the new values of x^ and s^* from:*

$$x^* = \sum x_i^* / p \quad (\text{D.5})$$

$$s^* = 1.134 \sqrt{\sum (x_i - x_i^*)^2 / (p - 1)} \quad (\text{D.6})$$

where the summation is over i .

The robust estimates x^ and s^* may be derived by an iterative calculation, i.e. by updating the values of x^* and s^* several times, until the process converges. Convergence may be assumed when there is no change from one iteration to the next in the third significant figure of the robust standard deviation and of the equivalent figure in the robust average. This is a simple method to program on a computer.*

In order to express the characteristics of population, standard uncertainty of robust mean was evaluated in this program by formula (D.7).

$$u(x^*) = 1.23s^* / \sqrt{p} \quad (D.7)$$

D.3 Statistics for Assessment

A z-score is normalized value which gives a “score” to each result, relative to the other numbers in the group, a z-score value close to zero means that the result agrees well with those from the other laboratories. When robust summary statistics are used, it is a robust z-score which is calculated by formula (D.8).

$$z = \frac{x - x^*}{s^*} \quad (D.8)$$

For instance, a z-score for Result C is calculated by

$$z_C = \frac{x_C - x_C^*}{s_C^*} \quad (D.9)$$

According to statistical design in this program, split-level samples were used. Within-lab z-score (denoted as z_w) and between-labs z-scores (denoted as z_b) that are for evaluating both sources of variation -- the variability within a laboratory and the variability between laboratories were calculated by formulae (D.10) and (D.11).

$$z_w = \frac{\text{Norm.Diff.} - x^*(\text{Norm.Diff.})}{s^*(\text{Norm.Diff.})} \quad (D.10)$$

$$z_b = \frac{\text{Norm.Sum} - x^*(\text{Norm.Sum})}{s^*(\text{Norm.Sum})} \quad (D.11)$$

where, Norm. Diff. is normalized difference between paired results, which is calculated by $\text{Norm.Diff.} = (x_B - x_A) / \sqrt{2}$, while Norm. Sum is normalized sum of paired results, which is calculated by $\text{Norm.Sum} = (x_B + x_A) / \sqrt{2}$, in which x_B is the result obtained from the sample B with a higher robust mean, x_A is the result from the paired sample A with a lower robust mean.

D.4 Procedures for Drawing Youden Plots

Youden Plots for z-scores were constructed by plotting z-scores obtained from one gross sample against z-scores obtained from another gross sample, and by plotting confidence ellipses with two different confidence levels and straight lines marked the robust means of each gross sample in the same plots. The method for calculating the confidence ellipses is based on Jackson (Jackson, J. Edward, Quality control methods for two related variables, Industrial Quality Control, January, 1956.) and robust statistics are used. For convenience, only the calculating method for ellipses in Youden Plot for z_B against z_A is given in formula (D.12).

$$z_B = z_A \cdot \hat{\rho} \pm \sqrt{(1 - \hat{\rho}^2)(T^2 - z_A^2)} \quad (D.12)$$

where,

$\hat{\rho}$ is the estimation of correlation coefficient of the two sets of data, it can be calculated by formula (D.13).

$$\hat{\rho} = \frac{\sum (z_A - \bar{z}_A)^2 \sum (z_B - \bar{z}_B)^2}{\sum (z_A - \bar{z}_A)(z_B - \bar{z}_B)} \quad (D.13)$$

T is related to projective range on the horizontal axis ($-T \leq z_A \leq T$) that can be derived from formula (D.14). When confidence level is given, for example, 5% or 1%, $F_{(1-\alpha),2,(n-1)}$, which is the tabulated $(1-\alpha)$ -quantile of the F-distribution with 2 and $(n-1)$ degrees of freedom can be obtained from related statistical tables.

$$T^2 = 2 \frac{n-1}{n-2} F_{(1-\alpha),2,(n-1)} \quad (D.14)$$

D.5 Procedures for Drawing Plots of Repeatability Ranges

In this PT scheme, participants were requested to carry out 4 replicate measurements on each analyte in each sample, almost participants reported 4 replicate results, so that we could produce a plot to identify any laboratories whose average and range are unusual, as described in ISO 13528. The plot of repeatability ranges was constructed by plotting the range of replicate results for each laboratory r_i against the corresponding average x_i for each laboratory.

Let

$\bar{X} = x^*$ the robust average of $x_1, x_2, \dots, x_p$, as calculated by Algorithm A in ISO 13528

$\bar{R} = r^*$ the robust pooled value of $r_1, r_2, \dots, r_p$, as calculated by Algorithm S in ISO 13528

and assume that the data are Normally distributed.

Under the null hypothesis that there is no difference between laboratories in the population values of either the laboratory means or the within-laboratory ranges, the statistic

$$\left(\sqrt{n} \frac{x_i - \bar{X}}{\bar{R}} \right)^2 + \left[\sqrt{2(n-1)} \ln \left(\frac{r_i}{\bar{R}} \right) \right]^2 \quad (D.15)$$

has approximately the χ^2 distribution with 2 degrees of freedom.

Hence a critical region with a significance level of 1 % may be drawn on the graph by plotting

$$r = \bar{R} \exp \left[\pm \frac{1}{\sqrt{2(n-1)}} \sqrt{\chi_{2,0.99}^2 - \left(\sqrt{n} \frac{x_i - \bar{X}}{\bar{R}} \right)^2} \right] \quad (D.16)$$

on the range axis against x on the average axis for

$$x = \bar{X} - \bar{R} \sqrt{\frac{\chi_{2,0.99}^2}{n}} \quad \text{to} \quad x = \bar{X} + \bar{R} \sqrt{\frac{\chi_{2,0.99}^2}{n}} \quad (D.17)$$

APPENDIX E

Models of Instructions, Forms and Interim Reports

In this appendix, models of Instructions to Participants, Receipt Form and Results Sheet are copied and attached. Due to confidential requests, the parts, which include participants' information, were deleted.

INSTRUCTIONS TO PARTICIPATING LABORATORIES

To ensure that results from this programme can be analysed properly, participating laboratories are asked to strictly adhere to the following instructions.

1. Participating laboratories are supplied with three samples, which are in sealed plastic bottles covered with dark packaging and labelled as “PT Sample”. All samples contain sunset yellow and tartrazine. The matrix is orange juice. The names and structures were listed in Annex A of this file. The concentration of sunset yellow or tartrazine in each sample is in the range of 40~300mg/kg.
2. Upon receipt of the samples, participating laboratories should carefully inspect the samples for any physical damages and defects. If presence of abnormality is detected, please contact the coordinator at yaojiab@lniciq.gov.cn immediately. New sample(s) will be replaced if necessary.
3. If the conditions of the samples are satisfactory, participating laboratories shall promptly acknowledge the receipt of the samples by returning the “Receipt Form” electronically to the coordinator at yaojiab@lniciq.gov.cn.
4. Analysis may commence as soon as the sample is received. The sample should be stored in the original packaging at 0-4℃ until analysis commences.
5. The following analyses are to be performed. Analysis results, in mg/kg on as-received basis, and other information should be reported on the given “Results Sheet”.
 - i. Sunset yellow [the limit of quantitation (LOQ) of sunset yellow should be better than 20mg/kg.]
 - ii. Tartrazine [the limit of quantitation (LOQ) of tartrazine should be better than 20mg/kg.]

Participating laboratories should be aware of analyte stability and perform the analyses in an appropriate order.

6. All samples must be analyzed and reported. The samples could be analyzed with preferably selected methods and it is recommended to use HPLC, HPLC-MS methods or other suitable methods. Whatever method you selected, it should be recorded on the TESTING RECORD FORMS. It is recommended that each sample should be mixed, prior to test, thoroughly and respectively. It is suggested that the mass of each duplicated test portion should be exceed 5g. Please record relevant information in TESTING RECORD FORMS in details, all you report will be benefit for analyzing testing results.
7. For each sample, please report 4 duplicated test results and their mean result with uncertainty of sunset yellow and tartrazine respectively on the RESULT SHEET. The test results shall be corrected by recovery.

8. Please fill out all relevant information, then email or fax the RESULTS SHEET to the coordinator of this program and your accreditation body NO LATER THAN THE DEADLINE 21st Feb 2010. And the TESTING RECORD FORMS, relevant spectrums, your preferred form of Measurement Uncertainty Report and any supporting documents should be submitted to your accreditation body before the deadline. At the same time, email or mail all the documents to the coordinator of this program at yaojiab@lnciq.gov.cn.

(Notes: 1. Under normal circumstances, results submitted after the deadline will not be accepted. 2. Owing to possible poor transmission quality, results submitted by fax are generally not acceptable.)

For enquires, please contact the coordinator at yaojiab@lnciq.gov.cn.

Annex A (For Introduction to Participants)

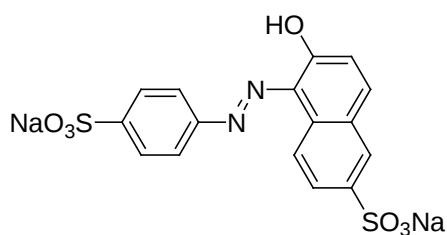
Structure of sunset yellow and tartrazine to Be Analyzed

1. sunset yellow

CAS Registering Number: 2783-94-0

Molecular Formula: $C_{16}H_{10}N_2Na_2O_7S_2$, molecular weight : 452.37

Structure:

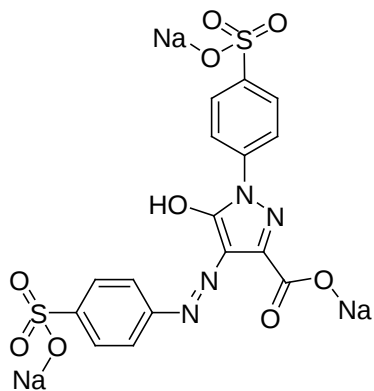


2. tartrazine

CAS Registering Number: 1934-21-0

Molecular Formula: $C_{16}H_9O_9N_4S_2Na_3$, molecular weight : 534.37

Structure:



RECEIPT FORM

We are pleased to announce that in accordance with the distribution schedule of APLAC food synthetic dyestuff proficiency testing program T072. we have the samples on 28th Jan. 2010 through TNT Express, along with INSTRUCTIONS TO PARTICIPANTS, RECEIPT FORM, RESULTS SHEET and TESTING RECORD FORMS.

In order to monitor status of the samples, we kindly ask each participating laboratory on receipt of the samples to fill out this RECEIPT FORM and return a copy by email or fax URGENTLY to:

Mr. Yao Jiabiao

Fax No. : +86 024-24123659

E-mail: yaojiab@lnciq.gov.cn

Thank you in advance for your cooperation.

NAME OF LABORATORY:

LAB CODE: **T072**—

DATE SAMPLE RECEIVED:

SAMPLE AMOUNT:

The samples are labelled as follows: Z ,Z ,Z

Inspect the packaging. Was there any visible damage? Yes No

If yes, or any other problem is available, describe the problem below.

Signature:

Date:

.

Results Sheet

Lab Code: T072—

SAMPLE RECEIVED DATE: _____ REPORT DATE: _____

SAMPLE PROCESSED DATE: From _____ to _____

Results of Sunset Yellow Contents in Samples

Sample	Testing Results(mg/kg)				Mean Result with Uncertainty (mg/kg)	Limit of Quatitation (mg/kg)
	1	2	3	4		
Z						
Z						
Z						

Results of Tartrazine Contents in Samples

Sample	Testing Results(mg/kg)				Mean Result with Uncertainty (mg/kg)	Limit of Quatitation (mg/kg)
	1	2	3	4		
Z						
Z						
Z						

Signature:**Date:**

Return this Results Sheet (including the Testing Record Form) by no later than the deadline 21st Feb. 2010 (we will confirm it by local postmark) by fax or email, and then mail to:

Mr. Yao Jiabiao

Address: 106, Dong Binhe Road, Shen He Qu, ShenYang110016, P. R. China

Phone: +86-24-24123659

Fax(Auto) : +86-24-24123659

E-mail: yaojiab@lnciq.gov.cn

Testing Record Forms

Lab Code: T072—

Is the method used for the results submitted in your scope of accreditation?			sunset yellow		☐ yes	☐ no
			tartrazine		☐ yes	☐ no
Reference:	sunset yellow	☐ standard/ ☐ journal/ ☐ book	year	No./Vol /Edition	pages	
	tartrazine	☐ standard/ ☐ journal/ ☐ book	year	No./Vol /Edition	pages	
Protocol for Quantification	sunset yellow	☐ External Standards ☐ Internal Standards ☐ Others (specify): _____				
	tartrazine	☐ External Standards ☐ Internal Standards ☐ Others (specify): _____				
Reference Materials	sunset yellow	Supplier(s): Purity:				
	tartrazine	Supplier(s): Purity:				
Reagents and Materials	sunset yellow					
	tartrazine					
Extraction	sunset yellow	Extraction Method:				

		Instruments used:
	tartrazine	Extraction Method: Instruments used:
Cleanup(if necessary)	sunset yellow	Cleanup Method: Instruments used:
	tartrazine	Cleanup Method: Instruments used:
Concentration	sunset yellow	Concentration Method: Instruments used:
	tartrazine	Concentration Method: Instruments used:
Operating Condition	sunset yellow	Equipment Model: _____ Detector: _____ Wavelength: _____ Column (type and specification): _____ MobilePhase: _____ Flow Speed: _____ mL/min Column

		Temperature: _____ °C Injection Volume: _____ μL
	tartrazine	Equipment Model: _____ Detector: _____ Wavelength: _____ Column (type and specification): _____ MobilePhase: _____ Flow Speed: _____ mL/min Column Temperature: _____ °C Injection Volume: _____ μL
Testing Sample	sunset yellow	Mass of Test Portions (in g) _____
	tartrazine	Mass of Test Portions (in g) _____
Chromatogram		Chromatograms Attached: _____ Pages The retention time of sunset yellow _____ min The retention time of tartrazine _____ min
MS characteristic parameters		sunset yellow: quantitative ion _____ qualitative ion _____ tartrazine: quantitative ion _____ qualitative ion _____
Calculating Formulae		sunset yellow tartrazine
Additional Information		Method usually used for Extraction (including instruments used): ☐ Solvent extraction ☐ Immerging method ☐ Shaking method ☐ Homogenization ☐ Soxhlet extraction ☐ SFE ☐ Other (specify): _____ Method usually used for Purification (including instruments used): ☐ L-L partition ☐ Column adsorption ☐ SPE ☐ GPC ☐ SFE ☐ Other (specify): _____

	Method usually used for Concentration (including instruments used): ☐ Speedy centrifugation ☐ On-line concentration ☐ Rotary evaporation ☐ N₂ blowing instrument ☐ natural evaporation ☐ K.D concentration instrument ☐ Other (specify): _____ 10 items most usually detected in your lab (including pesticide or veterinary drug residues): Do you usually use on-line analysis? ☐ Yes ☐ No Are the reference materials usually used in your lab certificated reference materials? ☐ Yes ☐ No Do you usually report the testing results with measurement uncertainty? ☐ Yes ☐ No Which method do you usually use in your determination? ☐ Screening method ☐ Detecting method ☐ Confirming method (Extra sheet may be added if necessary)
--	---

Signature:

Date:



APLAC Food Synthetic Dyestuff Proficiency Testing Program T072

Interim Report

Date of Issue : May 12, 2010

In this APLAC proficiency testing program, the contents of sunset yellow and tartrazine in orange juice samples were tested.

Three gross samples C, D and E were prepared and divided respectively to testing samples C, D and E, then every three testing samples C, D and E were distributed to the participating laboratories. There are a slightly higher content of sunset yellow and tartrazine in Sample C than that in Sample E, while Sample D bears a significant lower content of sunset yellow and a significant higher content of tartrazine than those in Samples C and Sample E.

The results for the tests on content of sunset yellow and tartrazine reported from the participating laboratories are listed in Table 1 and Table 2.

The statistics given in Table 3 were calculated by robust statistical methods based on the results listed in Table 1 and Table 2. The z-scores can be calculated by the following formulae. In Formula 1, Result is an individual result listed in Table 1 and Table 2, the normalized sum or normalized difference of a pair of results which can be calculated by Formula 2 and Formula 3, respectively. Paired results are the results on paired Sample C and Sample E.

Formula 1: $z\text{-score} = (\text{Result} - \text{Robust Mean}) / \text{Robust Standard Deviation}$

Formula 2: $\text{Normalized Sum} = (\text{Result C} + \text{Result E}) / \sqrt{2}$

Formula 3: $\text{Normalized Difference} = (\text{Result C} - \text{Result E}) / \sqrt{2}$

As a general rule, any z-score outside the range of -2 to 2 indicates a questionable result/pair of results, while an outlier is any result/pair of results with a z-score outside the range of -3 to 3.

Please confirm this interim report NO LATER THAN June 10, 2010 with your lab's address, Lab Code No. in this program, email, phone and fax number, we will use them for sending the final report. Thanks for your cooperation.

If you have any question on any aspect of your test, or on this PT program, please feel free to contact the persons listed below.

PT Manager:

Mr. Cao Zhijun

E-mail: caozj@cnas.org.cn

Coordinators:

Mr. Zheng Jiang

Mr. Yao Jiabiao

Fax: +86 024 24123659

Phone: +86 024 24123659

E-mail: yaojiab@inciq.gov.cn

Table 1 Reported Results of Content of Tartrazine

LabCode	Sample No.	Testing Results(mg/kg)				Mean Result(mg/kg)	Z- score	Normalized Sum	Zb	Normalized Difference	Zw
		1	2	3	4						
T072-001	Z 231 (C)	135	138	136	135	136	1.02	181.7	1.27	10.6	-0.24
	Z 183 (D)	78.6	75.9	75.4	75.2	76.3	0.40				
	Z 809 (E)	120	120	120	124	121	1.52				
T072-002	Z 210 (C)	137.8	138.7	141.8	138.1	139.1±10%	1.27	180.5	1.18	16.3	2.12
	Z 169 (D)	81.52	80.21	84.77	76.91	80.85±10%	0.93				
	Z 783 (E)	122.9	118.4	112.0	111.2	116.1±10%	1.00				
T072-004	Z 506 (C)	125.32	137.83	128.41		130.52±6.52,k=1	0.59	160.4	-0.15	24.2	5.42
	Z 334 (D)	67.17	69.94	65.88		67.66±2.07,K=1	-0.61				
	Z 764 (E)	95.77	94.24	98.90		96.30±2.38,K=1	-1.09				
T072-005	Z 346 (C)	109.5	110.9	109.1	108.8	109.6±14.2	-1.06	145.9	-1.11	9.1	-0.89
	Z 497 (D)	60.9	62.4	62.1	64.6	62.5±8.1	-1.21				
	Z 746 (E)	97.3	98.5	95.5	96.0	96.8±12.6	-1.04				
T072-006	Z 353 (C)	126	126	127	126	126±12.6	0.23	166.9	0.28	11.3	0.06
	Z 831 (D)	75	74	74	74	74±7.4	0.13				
	Z 870 (E)	109	110	110	110	110±11.0	0.36				
T072-007	Z 243 (C)	79	82	81	83		-3.30	103.1	-3.96	11.8	0.28
	Z 239 (D)	72	72	73	70		-0.13				
	Z 908 (E)	63	65	65	65		-4.45				
T072-008	Z 104 (C)	114.560	114.229	115.039	114.315	114.54±2.16	-0.67	151.3	-0.76	10.7	-0.19
	Z 966 (D)	66.588	66.481	66.724	66.680	66.62±1.22	-0.73				
	Z 760 (E)	99.252	99.601	99.252	99.381	99.38±1.80	-0.76				
T072-009	Z 221 (C)	116.24	117.09	116.25	116.20	116.4±1.5,K=2	-0.52	154.9	-0.51	9.7	-0.62
	Z 932 (D)	68.59	67.87	68.55	68.61	68.4±1.1,K=2	-0.52				
	Z 676 (E)	102.26	103.28	102.16	103.19	102.7±1.5,K=2	-0.41				
T072-010	Z 047 (C)	150.0	149.4			149.7±1.54	2.10	191.1	1.89	20.6	3.91
	Z 996 (D)	121.2	119.4			120.3±1.25	5.53				
	Z 980 (E)	121.8	119.4			120.6±1.62	1.48				
T072-011	Z 588 (C)	109	109	109	109	3	-1.11	144.0	-1.24	10.1	-0.44
	Z 418 (D)	62.5	62.5	62.5	62.5	1.7	-1.21				
	Z 814 (E)	94.7	94.7	94.7	94.7	2.4	-1.26				
T072-012	Z 223 (C)	120.46	120.56			120.51	-0.20	159.8	-0.19	10.6	-0.24
	Z 517 (D)	70.79	70.77			70.78	-0.24				
	Z 794 (E)	105.69	105.38			105.53	-0.11				
T072-013	Z 021 (C)	116	121	117	121	119±9; K=2, norm	-0.32	159.1	-0.24	9.2	-0.83
	Z 007 (D)	70.4	74.3	73.3	73.3	72.8±4.7; K=2, norm	-0.01				
	Z 742 (E)	106	107	104	108	106±6; K=2, norm	-0.06				
T072-014	Z 140 (C)	120.07	125.16	119.51	121.79	121.63±1.36	-0.11	161.0	-0.11	11.0	-0.07
	Z 416 (D)	73.15	74.28	70.94	73.70	73.01±0.81	0.02				
	Z 670 (E)	107.33	108.08	104.84	103.91	106.04±1.18	-0.06				

LabCode	Sample No.	Testing Results(mg/kg)				Mean Result(mg/kg)	Z- score	Normalized Sum	Zb	Normalized Difference	Zw
		1	2	3	4						
T072-015	Z 415 (C)	136	136	135	136	136±0.90	1.02	180.3	1.17	12.0	0.35
	Z 730 (D)	80.9	80.8	80.9	81	80.9±0.90	0.94				
	Z 974 (E)	119	119	119	119	119±0.90	1.31				
T072-016	Z031 (C)	113.8	112.7	113.8	113.0	113.3±5.0	-0.77	149.8	-0.85	10.4	-0.33
	Z 247 (D)	65.1	65.2	65.7	65.5	65.3±2.9	-0.88				
	Z 942 (E)	98.2	99.0	98.8	98.2	98.6±4.4	-0.84				
T072-017	Z 365 (C)	137.5	134.0	135.0	133.0	134.9±20.2	0.93	168.5	0.39	22.3	4.62
	Z 193 (D)	100.0	105.0	105.0	110.0	105.0±15.8	3.75				
	Z 792 (E)	107.6	104.2	102.0	100.0	103.4±15.5	-0.34				
T072-020	Z 403 (C)	117.2	116.7	116.3	117.5	116.9±0.5	-0.49	155.4	-0.48	9.9	-0.53
	Z 305 (D)	70.7	69.8	70.7	70.6	70.5±0.4	-0.27				
	Z 802 (E)	102.8	103.3	102.1	103.3	102.9±0.6	-0.39				
T072-021	Z 386 (C)	126.20	126.65	125.32	124.48	125.66±8.90	0.21	166.7	0.27	11.0	-0.06
	Z 750 (D)	74.95	75.38	74.70	74.48	74.88±3.14	0.24				
	Z 955 (E)	109.91	109.95	110.07	110.22	110.04±6.64	0.36				
T072-022	Z 550 (C)	120.7	119.7	116.9	120.0	119.3±7.11	-0.30	159.5	-0.21	9.2	-0.83
	Z 539 (D)	70.5	70.0	73.1	69.8	70.8±4.40	-0.24				
	Z 865 (E)	104.9	106.1	105.3	108.8	106.3±6.45	-0.03				
T072-023	Z 171 (C)	106.04	105.83	109.36	108.67	107.48±14.29	-1.23	145.2	-1.16	6.8	-1.83
	Z 269 (D)	58.75	59.21	61.68	60.73	60.09±8.20	-1.49				
	Z 941 (E)	99.64	96.17	98.84	96.96	97.90±13.00	-0.92				
T072-025	Z 464 (C)	168.4	165.2	170.9	164.1	167.1±4.9	3.47	215.8	3.53	20.5	3.88
	Z 816 (D)	91.3	83.6	88.0	95.1	89.5±7.7	1.94				
	Z 848 (E)	135.1	136.3	139.8	141.4	138.1±4.5	3.33				
T072-026	Z 094 (C)	123.48	123.52	123.47	123.26	123.43±0.55	0.03	165.1	0.16	9.5	-0.71
	Z 134 (D)	71.76	71.81	71.79	71.89	71.81±1.13	-0.12				
	Z 615 (E)	109.98	110.01	110.13	110.06	110.04±0.67	0.36				
T072-027	Z 192 (C)	131.79	132.72	132.79	133.38	132.4±18.32	0.74	174.8	0.81	12.4	0.53
	Z 203 (D)	75.39	76.33	75.66	76.91	76.06±10.52	0.37				
	Z 743 (E)	114.90	114.64	114.92	113.99	114.8±15.88	0.87				
T072-029	Z 149 (C)	119.1	120.8	119.8	119.8	119.9±18.7	-0.25	158.0	-0.31	11.5	0.14
	Z 968 (D)	69.7	70.5	69.8	70.6	70.2±11.8	-0.31				
	Z 991 (E)	102.8	104.0	101.0	106.4	103.6±16.5	-0.32				
T072-030	Z 289 (C)	121	119	114	123		-0.30	157.2	-0.37	11.5	0.13
	Z 705 (D)	69	70	70	70		-0.36				
	Z 904 (E)	106	102	102	102		-0.38				
T072-031	Z 392 (C)	115.0	114.9	114.7	114.0	114.7±2.8	-0.66	153.2	-0.63	9.1	-0.89
	Z 429 (D)	67.7	67.7	67.8	67.6	67.7±1.4	-0.60				
	Z 909 (E)	102.0	101.8	101.8	101.9	101.9±2.0	-0.50				
T072-032	Z 503 (C)	116.116	114.314	114.539	112.558	114.4±4.5(K=2)	-0.68	153.4	-0.62	8.4	-1.15
	Z 638 (D)	69.144	70.069	68.404	64.863	68.1±4.2(K=2)	-0.55				
	Z 867 (E)	102.879	103.096	105.197	98.818	102.5±4.8(K=2)	-0.43				

LabCode	Sample No.	Testing Results(mg/kg)				Mean Result(mg/kg)	Z- score	Normalized Sum	Zb	Normalized Difference	Zw
		1	2	3	4						
T072-034	Z 389 (C)	116.68	114.62	115.39	117.54	116.06±11.6	-0.55	154.5	-0.54	9.6	-0.64
	Z 855 (D)	70.37	71.40	67.62	72.00	70.35±7.0	-0.29				
	Z 713 (E)	103.36	98.55	103.96	103.88	102.44±10.2	-0.44				
T072-035	Z 384 (C)	169	143	128	124		1.42	180.5	1.19	18.9	3.22
	Z 946 (D)	99	92	82	70		1.50				
	Z 675 (E)	121	129	109	98		0.81				
T072-036	Z 412 (C)	136.84	141.70	144.60	146.00	142.28RSD2.8%	1.52	190.8	1.87	10.4	-0.31
	Z 614 (D)	84.37	92.82	82.08	85.00	89.21RSD5.4%	1.91				
	Z 940 (E)	128.22	123.76	127.04	131.09	127.53RSD2.4%	2.21				
T072-038	Z 598 (C)	129.1	128.7	130.7	130.3	129.7±0.8	0.52	171.1	0.56	12.3	0.47
	Z 610 (D)	75.1	76.1	76.7	75.1	75.8±0.8	0.34				
	Z 726 (E)	112.2	111.4	112.5	113	112.3±0.6	0.60				
T072-039	Z 380 (C)	112.9	113.2	113.8	115.1	113.8±1.5	-0.73	150.4	-0.81	10.5	-0.27
	Z 988 (D)	65.0	67.0	62.6	61.7	64.1±4.0	-1.02				
	Z 948 (E)	98.5	100.5	101.7	94.9	98.9±5.0	-0.81				

Table 2 Reported Results of Content of Sunset Yellow

LabCode	Sample No.	Testing Results(mg/kg)				Mean Result(mg/kg)	Z- score	Normalized Sum	Zb	Normalized Difference	Zw
		1	2	3	4						
T072-001	Z 231 (C)	94.5	95.2	94.7	94.5	94.7	0.77	115.3	0.88	18.6	0.36
	Z 183 (D)	154	152	153	155	154	1.00				
	Z 809 (E)	67.7	67.3	69.1	69.4	68.4	1.00				
T072-002	Z 210 (C)	102.0	103.2	97.46	95.62	99.57±12%	1.27	117.4	1.06	23.4	2.42
	Z 169 (D)	168.2	164.4	162.8	146.3	160.42±12%	1.39				
	Z 783 (E)	72.38	69.52	62.77	61.50	66.42±12%	0.72				
T072-004	Z 506 (C)	88.49	94.95	92.58		92.01±3.27,K=1	0.50	102.6	-0.23	27.6	4.16
	Z 334 (D)	100.37	98.11	92.39		96.95±4.11,K=1	-2.53				
	Z 764 (E)	53.49	49.35	56.28		53.04±3.49,K=1	-1.15				
T072-005	Z 346 (C)	77.7	78.9	77.3	76.6	77.6±10.9	-0.97	94.1	-0.96	15.6	-0.90
	Z 497 (D)	120.0	124.1	121.7	127.8	123.4±17.3	-0.90				
	Z 746 (E)	57.4	54.6	54.6	55.2	55.5±7.8	-0.80				
T072-006	Z 353 (C)	97	98	98	98	98±9.8	1.11	118.1	1.12	20.5	1.17
	Z 831 (D)	160	158	158	158	159±15.9	1.30				
	Z 870 (E)	68	69	69	69	69±6.9	1.08				
T072-007	Z 243 (C)	117	122	120	123		3.41	165.6	5.25	4.8	-5.51
	Z 239 (D)	127	126	127	125		-0.72				
	Z 908 (E)	112	115	114	114		7.33				
T072-008	Z 104 (C)	78.339	77.925	78.074	77.903	78.06±2.18	-0.92	94.1	-0.97	16.3	-0.61
	Z 966 (D)	124.066	124.005	124.398	124.112	124.15±3.22	-0.85				
	Z 760 (E)	54.809	55.211	55.033	54.907	54.99±1.64	-0.87				
T072-009	Z 221 (C)	81.96	81.65	81.97	81.93	81.9±1.1,K=2	-0.53	99.2	-0.52	16.6	-0.48
	Z 932 (D)	131.10	131.36	131.46	131.44	131.3±1.6,K=2	-0.41				
	Z 676 (E)	58.32	58.40	58.37	58.33	58.4±0.8,K=2	-0.40				
T072-010	Z 047 (C)	86.4	87.6			87.0±1.10	-0.01	112.9	0.67	10.2	-3.21
	Z 996 (D)	178.8	179.4			179.1±1.82	2.55				
	Z 980 (E)	72.0	73.2			72.6±1.6	1.59				
T072-011	Z 588 (C)	63.7	63.7	63.7	63.7	3.4	-2.39	75.1	-2.62	15.0	-1.17
	Z 418 (D)	104	104	104	104	5	-2.10				
	Z 814 (E)	42.5	42.5	42.5	42.5	2.5	-2.62				
T072-012	Z 223 (C)	78.92	79.43			79.17	-0.81	93.6	-1.01	18.4	0.27
	Z 517 (D)	128.78	128.38			128.58	-0.58				
	Z 794 (E)	53.97	52.37			53.17	-1.13				
T072-013	Z 021 (C)	82.2	86.8	83.9	87.8	85.2±7.0; K=2, norm	-0.19	104.0	-0.1	16.5	-0.54
	Z 007 (D)	133	134	134	133	134±14; K=2, norm	-0.24				
	Z 742 (E)	63.4	60.3	62.6	61.3	61.9±4.0; K=2, norm	0.09				
T072-014	Z 140 (C)	88.74	88.62	89.88	88.61	88.96±0.91	0.19	106.9	0.15	18.9	0.51
	Z 416 (D)	142.74	143.35	144.21	143.38	143.42±1.47	0.34				
	Z 670 (E)	62.09	62.24	62.20	62.18	62.17±0.64	0.13				
T072-015	Z 415 (C)	91.8	91.8	91.6	91.8	91.8±1.92	0.48	110.9	0.49	19.0	0.51
	Z 730 (D)	147	147	147	147	147±1.92	0.56				
	Z 974 (E)	65.1	64.9	65.1	64.7	65.0±1.92	0.52				

LabCode	Sample No.	Testing Results(mg/kg)				Mean Result(mg/kg)	Z- score	Normalized Sum	Zb	Normalized Difference	Zw
		1	2	3	4						
T072-016	Z031 (C)	85.2	84.8	85.2	84.6	84.9±3.8	-0.23	102.0	-0.28	18.1	0.15
	Z 247 (D)	136.9	136.6	136.7	136.0	136.5±6.0	-0.09				
	Z 942 (E)	59.2	59.3	59.3	59.2	59.3±2.6	-0.27				
T072-017	Z 365 (C)	105.0	110.0	105.0	98.0	104.7±15.7	1.79	122.2	1.48	25.9	3.45
	Z 193 (D)	143.0	138.0	140.0	143.5	141.1±21.2	0.20				
	Z 792 (E)	68.6	68.5	68.9	66.5	68.1±10.2	0.96				
T072-020	Z 403 (C)	88.9	90.2	88.8	90.3	89.6±0.8	0.25	108.0	0.24	18.7	0.42
	Z 305 (D)	138.6	138.9	140.4	141.3	139.8±1.3	0.12				
	Z 802 (E)	63.3	63.1	63.3	62.6	63.1±0.3	0.26				
T072-021	Z 386 (C)	85.78	85.54	84.23	83.47	84.76±4.48	-0.24	103.2	-0.17	16.6	-0.47
	Z 750 (D)	136.96	138.08	137.04	137.28	137.34±10.44	-0.03				
	Z 955 (E)	61.49	61.03	61.46	60.89	61.22±2.11	0.00				
T072-022	Z 550 (C)	85.6	85.6	85.1	83.2	85.3±5.07	-0.18	104.3	-0.08	16.3	-0.60
	Z 539 (D)	133.5	132.8	132.8	131.7	132.7±7.75	-0.32				
	Z 865 (E)	61.9	62.6	62.2	62.2	62.2±3.62	0.13				
T072-023	Z 171 (C)	74.76	73.64	70.47	70.95	72.46±7.26	-1.49	86.1	-1.66	16.4	-0.57
	Z 269 (D)	109.77	104.04	100.00	112.94	106.44±14.25	-1.95				
	Z 941 (E)	47.26	51.42	48.45	49.85	49.24±5.42	-1.68				
T072-024	Z 245 (C)	76	77	77	76	77±7.7	-1.03	92.6	-1.09	16.3	-0.63
	Z 636 (D)	122	124	124	121	123±12.3	-0.92				
	Z 961 (E)	54	54	54	54	54±5.4	-1.01				
T072-025	Z 464 (C)	108.7	104.8	104.6	105.0	105.8±3.1	1.91	126.3	1.83	23.3	2.37
	Z 816 (D)	169.2	169.8	170.6	168.0	169.4±1.7	1.95				
	Z 848 (E)	72.8	72.4	73.1	73.0	72.8±0.5	1.61				
T072-026	Z 094 (C)	80.50	80.87	79.93	80.83	80.53±1.05	-0.67	97.0	-0.71	16.9	-0.36
	Z 134 (D)	138.93	138.12	138.92	138.98	138.73±1.41	0.05				
	Z 615 (E)	56.87	56.73	56.92	56.06	56.64±0.69	-0.64				
T072-027	Z 192 (C)	101.06	102.12	101.79	101.67	101.66±13.63	1.48	122.4	1.49	21.4	1.56
	Z 203 (D)	160.98	163.07	162.40	162.82	162.32±21.77	1.51				
	Z 743 (E)	71.34	71.25	71.25	71.68	71.37±9.57	1.41				
T072-029	Z 149 (C)	84.8	85.7	84.2	84.8	84.9±13.9	-0.23	102.2	-0.26	17.8	0.03
	Z 968 (D)	137.7	137.4	135.8	137.2	137.0±20.9	-0.06				
	Z 991 (E)	60.5	58.5	59.3	60.5	59.7±10.3	-0.22				
T072-030	Z 289 (C)	90	89	86	92		0.22	108.2	0.26	18.0	0.12
	Z 705 (D)	143	143	142	143		0.30				
	Z 904 (E)	65	64	63	63		0.35				
T072-031	Z 392 (C)	83.6	83.2	83.5	82.9	83.3±2.0	-0.39	101.1	-0.35	16.7	-0.45
	Z 429 (D)	133.9	133.8	133.8	133.7	133.8±2.7	-0.25				
	Z 909 (E)	59.7	59.5	59.5	59.9	59.7±1.3	-0.22				
T072-032	Z 503 (C)	93.869	93.181	95.572	92.204	93.7±4.4(K=2)	0.67	114.2	0.78	18.3	0.24
	Z 638 (D)	153.835	156.892	155.555	146.170	153.1±7.0(K=2)	0.94				
	Z 867 (E)	66.120	68.298	70.614	66.366	67.8±4.4(K=2)	0.92				

LabCode	Sample No.	Testing Results(mg/kg)				Mean Result(mg/kg)	Z- score	Normalized Sum	Zb	Normalized Difference	Zw
		1	2	3	4						
T072-034	Z 389 (C)	77.43	76.30	76.95	76.22	76.73±7.7	-1.06	92.8	-1.08	15.7	-0.86
	Z 855 (D)	120.25	122.43	121.14	122.18	121.50±12.2	-1.01				
	Z 713 (E)	55.22	53.69	54.97	54.09	54.49±5.4	-0.94				
T072-035	Z 384 (C)	86	87	85	88.7		-0.04	104.6	-0.05	18.0	0.10
	Z 946 (D)	149	143	140	160		0.62				
	Z 675 (E)	63	57	61	64		0.00				
T072-036	Z 412 (C)	79.24	79.83	82.46	82.49	81.00RSD2.1%	-0.62	98.2	-0.61	16.4	-0.58
	Z 614 (D)	145.06	152.26	144.87	145.16	146.84RSD3.6%	0.55				
	Z 940 (E)	58.87	55.89	57.3	59.25	57.83RSD2.7%	-0.48				
T072-038	Z 598 (C)	88.7	89.3	89.8	89.2	89.3±0.4	0.22	107.1	0.17	19.2	0.60
	Z 610 (D)	142.7	141.5	142.8	142.0	142.2±0.5	0.27				
	Z 726 (E)	62.1	62.1	62.4	62.1	62.2±0.1	0.13				
T072-039	Z 380 (C)	83.7	82.6	83.8	82.9	83.2±1.0	-0.40	101.7	-0.31	16.0	-0.75
	Z 988 (D)	133.7	138.7	130.1	131.3	133.5±6.0	-0.27				
	Z 948 (E)	61.4	62.1	61.9	56.9	60.6±4.0	-0.09				

Table 3 Statistics for the Test on Contents of Tartrazine and Sunset Yellow

Tartrazine				Sunset Yellow			
	Total Number of Results	Robust Mean, mg/kg	Robust Standard Deviation, mg/kg		Total Number of Results	Robust Mean, mg/kg	Robust Standard Deviation, mg/kg
Result C	33	123.1	12.7	Result C	33	87.1	9.8
Result D	33	72.9	8.6	Result D	33	137.9	16.2
Result E	33	106.6	9.5	Result E	33	61.2	7.2
Normalized Sum of Result C and E	33	162.7	15.1	Normalized Sum of Result C and E	33	105.2	11.5
Normalized Difference between Result C and E	33	11.2	2.4	Normalized Difference between Result C and E	33	17.8	2.4