

**FACULTY OF SCIENCE**

DEPARTMENT OF CHEMICAL SCIENCE
NATIONAL DIPLOMA: ANALYTICAL CHEMISTRY

MODULE: CHEMICAL QUALITY ASSURANCE

MODULE CODES: CETQAB3/CET2BQA

CAMPUS: DFC

JANUARY EXAMINATION 2020

SUPPLEMENTARY EXAMINATION

DATE: 10TH JANUARY 2020

SESSION: 0800-1100

ASSESSORS

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EXTERNAL MODERATOR

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DURATION 180 MINUTES

MARKS 150

NUMBER OF PAGES: 7 PAGES (INCLUDING APPENDICES)

INSTRUCTIONS: CALCULATORS ARE PERMITTED (ONLY ONE PER STUDENT)

**REQUIREMENTS: ANSWER SCRIPTS
ONE SHEET GRAPH PAPER**

INSTRUCTIONS TO CANDIDATES

1. PLEASE ANSWER ALL QUESTIONS
2. STUDENTS WILL BE PENALIZED FOR OMITTED OR INCORRECT UNITS
3. STUDENTS WILL BE PENALIZED FOR INCORRECT USE OF SIGNIFICANT FIGURES

QUESTION 1

- 1.1 Briefly discuss the eight dimensions of quality. Does this improve our understanding of quality? (10)
- 1.2 Explain what is involved in the following:
- 1.2.1 Quality control (3)
 - 1.2.2 Quality Assessment (5)
 - 1.2.3 Quality Assurance (9)
- 1.3 An analytical services laboratory analysed 30 000 water samples in October 2019. The lab observed that only 1000 samples out the 30 000 fail the blind test requirements. Calculate the quality performance (%) of the laboratory. (3)

[30]**QUESTION 2**

- 2.1 Briefly discuss the following terms:
- 2.1.1 Sampling (2)
 - 2.1.2 Limit of detection (LOD) (2)
 - 2.1.3 Limit of quantitation (LOQ) (2)
 - 2.1.4 Selective sample (3)
 - 2.1.5 Sampling plan (3)
 - 2.1.6 Precision (4)
- 2.2 List the sources of contamination of samples in a laboratory environment (6)
- 2.3 Briefly define method errors and how they can be minimised (4)
- 2.4 Define systematic errors describe and how they can be identified. (4)

[30]**QUESTION 3**

- 3.1 Differentiate between analytical and non-analytical errors. (9)
- 3.2 The certified value for cholesterol in a Certified Reference Material (CRM) is $274 \pm 9.0 \text{ mg}(100\text{g})^{-1}$. The results of repeat experiments to measure the recovery of cholesterol from CRM are as follows: 271.4, 266.3, 267.8, 269.6,

- 268.7, 272.5, 269.5, 270.1, 269.7, 268.6, 268.4. The results are reported in mg (100 g)^{-1} . Calculate the bias of the method, the percentage bias and percentage recovery, percent bias in CRM. Comment on the results. (11)
- 3.3 An analyst carried out a repeated titrimetric analysis of As(V) ions in borehole water. The values obtained were normally distributed with mean value of 10.15 mg/mL and a standard deviation of ± 0.02 mg/mL Find the proportion of measurements which lie between 10.12 mg/mL and 10.20 mg/mL (8)
- 3.4 Briefly describe the two basic area properties of a standard normal curve. (2)
- (30)**

QUESTION 4

- 4.1 The precision of a method is being established, and the following data are obtained:
22.23, 22.18, 22.25, 22.09, and 22.17%. Is 22.09 a valid measurement at the 95% confidence level? (6)
- 4.2 In brief, discuss the factors that have to be considered when choosing between types of method to be used for analysing a sample in chemical analysis. (14)
- 4.3 The following table shows results of replicate potassium determination in a blood sample using atomic emission spectrophotometry (AES) and a new amperometric method. Is there a significant difference in the precision of the two methods? (Perform full calculations.) (10)

AES (mg/L)	Amperometric (mg/L)
2.09	1.82
2.01	2.05
2.06	1.99
2.12	2.13
1.97	2.06
2.00	1.83
	2.01
	2.15

[30]

QUESTION 5

- 5.1 What is good laboratory practice (GLP) for scientific practice? (6)
- 5.2 Discuss the importance of the following in quality control:
- 5.2.1 Blanks (3)
- 5.2.2 Quality control samples (3)
- 5.3.3 Blind samples (3)
- 5.4 A standard material known to contain 38.9% Hg was analysed by atomic absorption spectroscopy. The results were 38.9%, 37.4% and 37.1%. At the 95% confidence level, is there any evidence for a systematic error in the method? (10)
- 5.5 Describe Flicker noise in chemical instrumentation (5)

[30]

Appendices

Appendix 1; Equations:

$$\pm t = \frac{\bar{x}_1 - \bar{x}_2}{s_p} \sqrt{\frac{N_1 N_2}{N_1 + N_2}}$$

$$s_{\text{pooled}} = \sqrt{\frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2 + \dots + (n_k - 1)s_k^2}{n_1 + n_2 + \dots + n_k - k}}$$

$$[t] = \left| \frac{\bar{x} - \mu}{s} \right| * \sqrt{n}$$

$$s = \sqrt{\frac{\sum_1^n (x_i - \bar{x})^2}{n - 1}}$$

Appendix 2: Rejection Coefficient test ratios

No. of observations	Confidence Level (CL)		
	90%	95%	99%
3	0.941	0.970	0.994
4	0.765	0.829	0.926
5	0.642	0.710	0.821
6	0.560	0.625	0.740
7	0.507	0.568	0.680
8	0.468	0.526	0.634
9	0.437	0.493	0.598
10	0.412	0.466	0.568

Appendix 3: Tables for t and F values for various levels of probability

Table A.2 The *t*-distribution

Value of <i>t</i> for a confidence interval of Critical value of $ t $ for <i>P</i> values of number of degrees of freedom	90% 0.10	95% 0.05	98% 0.02	99% 0.01
1	6.31	12.71	31.82	63.66
2	2.92	4.30	6.96	9.92
3	2.35	3.18	4.54	5.84
4	2.13	2.78	3.75	4.60
5	2.02	2.57	3.36	4.03
6	1.94	2.45	3.14	3.71
7	1.89	2.36	3.00	3.50
8	1.86	2.31	2.90	3.36
9	1.83	2.26	2.82	3.25
10	1.81	2.23	2.76	3.17
12	1.78	2.18	2.68	3.05
14	1.76	2.14	2.62	2.98
16	1.75	2.12	2.58	2.92
18	1.73	2.10	2.55	2.88
20	1.72	2.09	2.53	2.85
30	1.70	2.04	2.46	2.75
50	1.68	2.01	2.40	2.68
∞	1.64	1.96	2.33	2.58

The critical values of $|t|$ are appropriate for a two-tailed test. For a one-tailed test the value is taken from the column for *twice* the desired *P*-value, e.g. for a one-tailed test, $P = 0.05$, 5 degrees of freedom, the critical value is read from the $P = 0.10$ column and is equal to 2.02.

Table A.3 Critical values of *F* for a one-tailed test ($P = 0.05$)

v_2	v_1												
	1	2	3	4	5	6	7	8	9	10	12	15	20
1	161.4	199.5	215.7	224.6	230.2	234.0	236.8	238.9	240.5	241.9	243.9	245.9	248.0
2	18.51	19.00	19.16	19.25	19.30	19.33	19.35	19.37	19.38	19.40	19.41	19.43	19.45
3	10.13	9.552	9.277	9.117	9.013	8.941	8.887	8.845	8.812	8.786	8.745	8.703	8.660
4	7.709	6.944	6.591	6.388	6.256	6.163	6.094	6.041	5.999	5.964	5.912	5.858	5.803
5	6.608	5.786	5.409	5.192	5.050	4.950	4.876	4.818	4.772	4.735	4.678	4.619	4.558
6	5.987	5.143	4.757	4.534	4.387	4.284	4.207	4.147	4.099	4.060	4.000	3.938	3.874
7	5.591	4.737	4.347	4.120	3.972	3.866	3.787	3.726	3.677	3.637	3.575	3.511	3.445
8	5.318	4.459	4.066	3.838	3.687	3.581	3.500	3.438	3.388	3.347	3.284	3.218	3.150
9	5.117	4.256	3.863	3.633	3.482	3.374	3.293	3.230	3.179	3.137	3.073	3.006	2.936
10	4.965	4.103	3.708	3.478	3.326	3.217	3.135	3.072	3.020	2.978	2.913	2.845	2.774
11	4.844	3.982	3.587	3.357	3.204	3.095	3.012	2.948	2.896	2.854	2.788	2.719	2.646
12	4.747	3.885	3.490	3.259	3.106	2.996	2.913	2.849	2.796	2.753	2.687	2.617	2.544
13	4.667	3.806	3.411	3.179	3.025	2.915	2.832	2.767	2.714	2.671	2.604	2.533	2.459
14	4.600	3.739	3.344	3.112	2.958	2.848	2.764	2.699	2.646	2.602	2.534	2.463	2.388
15	4.543	3.682	3.287	3.056	2.901	2.790	2.707	2.641	2.588	2.544	2.475	2.403	2.328
16	4.494	3.634	3.239	3.007	2.852	2.741	2.657	2.591	2.538	2.494	2.425	2.352	2.276
17	4.451	3.592	3.197	2.965	2.810	2.699	2.614	2.548	2.494	2.450	2.381	2.308	2.230
18	4.414	3.555	3.160	2.928	2.773	2.661	2.577	2.510	2.456	2.412	2.342	2.269	2.191
19	4.381	3.522	3.127	2.895	2.740	2.628	2.544	2.477	2.423	2.378	2.308	2.234	2.155
20	4.351	3.493	3.098	2.866	2.711	2.599	2.514	2.447	2.393	2.348	2.278	2.203	2.124

v_1 = number of degrees of freedom of the numerator; v_2 = number of degrees of freedom of the denominator.

Appendix 4: Tables for Z values for various levels of probability

Table A.1 Continued

Table A.1 Continued $\Phi(z)$ Standard normal cumulative distribution

z	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
-1.4	0.0808	0.0823	0.0838	0.0853	0.0869	0.0885	0.0901	0.0918	0.0934	0.0951
-1.3	0.0968	0.0985	0.1003	0.1020	0.1038	0.1056	0.1075	0.1093	0.1112	0.1131
-1.2	0.1151	0.1170	0.1190	0.1210	0.1230	0.1251	0.1271	0.1292	0.1314	0.1335
-1.1	0.1357	0.1379	0.1401	0.1423	0.1446	0.1469	0.1492	0.1515	0.1539	0.1562
-1.0	0.1587	0.1611	0.1635	0.1660	0.1685	0.1711	0.1736	0.1762	0.1788	0.1814
-0.9	0.1841	0.1867	0.1894	0.1922	0.1949	0.1977	0.2005	0.2033	0.2061	0.2090
-0.8	0.2119	0.2148	0.2177	0.2206	0.2236	0.2266	0.2296	0.2327	0.2358	0.2388
-0.7	0.2420	0.2451	0.2483	0.2514	0.2546	0.2578	0.2611	0.2643	0.2676	0.2709
-0.6	0.2743	0.2776	0.2810	0.2843	0.2877	0.2912	0.2946	0.2981	0.3015	0.3050
-0.5	0.3085	0.3121	0.3156	0.3192	0.3228	0.3264	0.3300	0.3336	0.3372	0.3409
-0.4	0.3446	0.3483	0.3520	0.3557	0.3594	0.3632	0.3669	0.3707	0.3745	0.3783
-0.3	0.3821	0.3859	0.3897	0.3936	0.3974	0.4013	0.4052	0.4090	0.4129	0.4168
-0.2	0.4207	0.4247	0.4286	0.4325	0.4364	0.4404	0.4443	0.4483	0.4522	0.4562
-0.1	0.4602	0.4641	0.4681	0.4721	0.4761	0.4801	0.4840	0.4880	0.4920	0.4960
0.0	0.5000	0.5040	0.5080	0.5120	0.5160	0.5199	0.5239	0.5279	0.5319	0.5359
0.1	0.5398	0.5438	0.5478	0.5517	0.5557	0.5596	0.5636	0.5675	0.5714	0.5753
0.2	0.5793	0.5832	0.5871	0.5910	0.5948	0.5987	0.6026	0.6064	0.6103	0.6141
0.3	0.6179	0.6217	0.6255	0.6293	0.6331	0.6368	0.6406	0.6443	0.6480	0.6517
0.4	0.6554	0.6591	0.6628	0.6664	0.6700	0.6736	0.6772	0.6808	0.6844	0.6879
0.5	0.6915	0.6950	0.6985	0.7019	0.7054	0.7088	0.7123	0.7157	0.7190	0.7224
0.6	0.7257	0.7291	0.7324	0.7357	0.7389	0.7422	0.7454	0.7486	0.7517	0.7549
0.7	0.7580	0.7611	0.7642	0.7673	0.7704	0.7734	0.7764	0.7794	0.7823	0.7852
0.8	0.7881	0.7910	0.7939	0.7967	0.7995	0.8023	0.8051	0.8078	0.8106	0.8133
0.9	0.8159	0.8186	0.8212	0.8238	0.8264	0.8289	0.8315	0.8340	0.8365	0.8389
1.0	0.8413	0.8438	0.8461	0.8485	0.8508	0.8531	0.8554	0.8577	0.8599	0.8621
1.1	0.8643	0.8665	0.8686	0.8708	0.8729	0.8749	0.8770	0.8790	0.8810	0.8830
1.2	0.8849	0.8869	0.8888	0.8907	0.8925	0.8944	0.8962	0.8980	0.8997	0.9015
1.3	0.9032	0.9049	0.9066	0.9082	0.9099	0.9115	0.9131	0.9147	0.9162	0.9177
1.4	0.9192	0.9207	0.9222	0.9236	0.9251	0.9265	0.9279	0.9292	0.9306	0.9319
1.5	0.9332	0.9345	0.9357	0.9370	0.9382	0.9394	0.9406	0.9418	0.9429	0.9441
1.6	0.9452	0.9463	0.9474	0.9484	0.9495	0.9505	0.9515	0.9525	0.9535	0.9545
1.7	0.9554	0.9564	0.9573	0.9582	0.9591	0.9599	0.9608	0.9616	0.9625	0.9633
1.8	0.9641	0.9649	0.9656	0.9664	0.9671	0.9678	0.9686	0.9693	0.9699	0.9706
1.9	0.9713	0.9719	0.9726	0.9732	0.9738	0.9744	0.9750	0.9756	0.9761	0.9767
2.0	0.9772	0.9778	0.9783	0.9788	0.9793	0.9798	0.9803	0.9808	0.9812	0.9817
2.1	0.9821	0.9826	0.9830	0.9834	0.9838	0.9842	0.9846	0.9850	0.9854	0.9857
2.2	0.9861	0.9864	0.9868	0.9871	0.9875	0.9878	0.9881	0.9884	0.9887	0.9890
2.3	0.9893	0.9896	0.9898	0.9901	0.9904	0.9906	0.9909	0.9911	0.9913	0.9916
2.4	0.9918	0.9920	0.9922	0.9925	0.9927	0.9929	0.9931	0.9932	0.9934	0.9936
2.5	0.9938	0.9940	0.9941	0.9943	0.9945	0.9946	0.9948	0.9949	0.9951	0.9952
2.6	0.9953	0.9955	0.9956	0.9957	0.9959	0.9960	0.9961	0.9962	0.9963	0.9964
2.7	0.9965	0.9966	0.9967	0.9968	0.9969	0.9970	0.9971	0.9972	0.9973	0.9974
2.8	0.9974	0.9975	0.9976	0.9977	0.9977	0.9978	0.9979	0.9979	0.9980	0.9981
2.9	0.9981	0.9982	0.9982	0.9983	0.9984	0.9984	0.9985	0.9985	0.9986	0.9986
3.0	0.9987	0.9987	0.9987	0.9988	0.9988	0.9989	0.9989	0.9989	0.9990	0.9990
3.1	0.9990	0.9991	0.9991	0.9991	0.9992	0.9992	0.9992	0.9992	0.9993	0.9993
3.2	0.9993	0.9993	0.9994	0.9994	0.9994	0.9994	0.9994	0.9995	0.9995	0.9995
3.3	0.9995	0.9995	0.9995	0.9996	0.9996	0.9996	0.9996	0.9996	0.9996	0.9997
3.4	0.9997	0.9997	0.9997	0.9997	0.9997	0.9997	0.9997	0.9997	0.9997	0.9998