UNIVERSITY OF JOHANNESBURG

FACULTY OF SCIENCE

DEPARTMENT OF CHEMICAL SCIENCES

NATIONAL DIPLOMA: ANALYTICAL CHEMISTRY

MODULE: CHEMICAL QUALITY ASSURANCE

MODULE CODES: CETQAB3/CET2BQA

CAMPUS: DFC

NOVEMBER EXAMINATION 2019

MAIN EXAMINATION

DATE: 16TH NOVEMBER 2019 **SESSION: 0830-1130**

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EXTERNAL MODERATOR **DR M MUJURU**

DURATION 180 MINUTES **MARKS 150**

NUMBER OF PAGES: 8 (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 SIGNFICANT FIGURES

QUESTION 1

1.1 Describe the following terms as they are used in quality management

1.1.1 External quality assessment (3)
1.1.2 Quality control (2)
1.1.3 Quality improvement (1)
1.1.4 Quality assurance (4)

1.2 A juice canning equipment is designed to produce 2 000 000 units per month but managed to deliver only 1 900 000 units in the month of November. What is the quality performance of the equipment? (3)

1.3 If you are provided with 10 replicated data from an assay of a rock sample. Briefly describe how you are going to determine the following

1.3.1 Average (4)
1.3.2 Median (3)
1.3.3 Mode (4)

1.4 List the three basic measures of dispersion used in statistical quality control. (3)

1.5 what are the procedures which can be used to detect systematic errors (3)

[30]

QUESTION 2

2.1 Differentiate the meaning of the following terms as they are commonly used in chemical quality assurance.

2.1.1 Precision and accuracy (4)
2.1.2 Repeatability and reproducibility (2)

2.2 Briefly describe the sources of the following errors and how to reduce them.

2.2.1 Instrumental errors (3)
2.2.2 Method error (3)
2.2.3 Personal error (2)

2.3 Explain the difference between relative error (E_r) and coefficient of variation (relative standard deviation (RSD) (4)

2.4 Three students (A, B and C) were given the same soil sample to analyse for phosphorus. Each of the three students performed more than 30 replicates analysis. Each set of data obtained by teach student was plotted on a graph as Range of measured values (horizontal) vs Percentage of measurements (vertical axis). Comment on the following observations:

2.4.1 Student A data produced a curve with a kurtosis symmetrically by the mean (4)
2.4.2 Student B data produced a curve with a positive kurtosis (4)
2.4.3 Student C data produced a curve with negative kurtosis (4)

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QUESTION 3

3.1 Briefly describe the four basic properties of a standard normal curve. (5)

3.2 A student carried out a repetitive titrimetric analysis of calcium ions in borehole water. The Ca^{2+}(aq) levels obtained were normally distributed with the mean value of 11.16 mg/L and a standard deviation of ±0.03 mg/L. Find the proportion (%) of the measurements which lie between 11.13 mg/L and 11.26 mg/L. ($Z = \frac{x-\bar{x}}{s}$) (5)

3.3 A student carried out titrimetric analysis of vitamin C in a juice produced from a continuous production line and obtained the concentrations shown in the table below:

n	Time	x_i (mg/L)	(x_i - $\bar{x}$)	(x_i - $\bar{x}$)^2
1	1	10.08		
2	20	10.11		
3	40	10.09		
4	60	10.10		
5	80	10.12		
6	100	10.90		
7	120	9.80		
8	140	11.20		
9	160	8.80		
10	180	10.30		
Totals				

3.3.1 Redraw and complete the table by filling in the missing data in the table in your answer booklet. (4)

3.3.2 Calculate using the data from the table:
(a) the mean, (2)
(b) standard deviation, (2)
(c) variance; and (2)

(d) relative standard deviation of the data. (2)

3.3.3 Given the following equations:

$Control\ Limits = \bar{x} \pm 3s/\sqrt{n}$; $Control\ Limits = \bar{x} \pm 3s/\sqrt{n}$

(a) Calculate the Control limits and the warning limits (4)

(b) Plot a control chart of x_i versus time (min) (4)

(c) Comment briefly on the process stability (2)

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QUESTION 4

4.1 Define the following terms and describe how they are determined in analytical chemistry experiments.

4.1.1 Limit of detection (LOD) (4)

4.1.2 Limit of Decision (CC_{α}); and (4)

4.1.3 Limit of quantitation (LOQ) (4)

4.2 The following set of alcohol analyses on separate aliquots of a pooled serum were reported: 103, 106, 107, and 124 meq/L. One value appears suspect. Identify this value and determine if it can be ascribed to accidental error, at 95% confidence level? (5)

4.3 The following replicates of selenium determination on a tissue extract using amperometric analysis and new calorimetric method were reported. Is there a significant difference between the precisions of the two methods at 95% confidence level? (5)

Amperometric method (mg/dm^3)	Colorimetric (mg/dm^3)
10.9	10.5
10.3	9.8
10.7	11.4
10.1	11.5
9.8	9.4
11.3	10.2
	11.3
	9.2

4.4 Compare Analog and Digital analytical equipment in terms of

4.3.1 Power consumption (2)

4.3.2 Errors (2)

[30]

QUESTION 5

5.1 Briefly describe the following instrumental noises.
5.1.1 Thermal or Johnson Noise (2)
5.1.2 Shot noise (2)
5.1.3 Flicker noise (3)
5.1.4 Environmental noise (7)

5.2 An analyst analysed a soil sample for phosphorus using Microwave induced Plasma Optical Emission Spectrometer (MIP-OES) and validated the method by analysing the same sample using Ion chromatography (IC). The student obtained a mean concentration of 55.75±0.111 µg/kg S and 53.45 ±0.0105 µg/kg S with MIP–OES and IC respectively. Which of the equipment is more sensitive in terms of signal to noise ratio? (4)

5.3 Briefly describe the following samples, giving a typical example where such are sample is required.
5.3.1 Selective sample (4)
5.3.2 Random sample (5)
5.3.3 Composite sample (3)

[30]

APPENDICES

Appendix 1; Equations:

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

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

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

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

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 *t* for a confidence interval of Critical value of $\|t\|$ for *P* 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 ($P = 0.05$)

ν_2	ν_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

ν_1 = number of degrees of freedom of the numerator; ν_2 = number of degrees of freedom of the denominator.

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

Table A.1 Continued F(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	0.2389
−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	0.6579
0.5	0.6915	0.6950	0.6965	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