**APPENDIX I ZYX /CQA/SOP-039/FR-01**

**LOG BOOK OF TOC ANALYSER**

**Instrument ID : -------------------------------**

**Location : -------------------------------**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Sr. No | Date | Name of Sample | Batch No / A.R. No | Done by | Checked by | Remarks |
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**ZYX/CQA/SOP-039/FR-02 APPENDIX II**

**CALIBRATION BY POTASSIUM HYDROGEN PHTHALATE**

(Note: Calibration shall be done on the day of sample analysis)

1.0 Instrument ID . : -------------------------

2.0 Location : --------------------------

3.0 Date of Calibration : ---------------------------

4.0Preparation of Standard solution

Wt of KHP (Batch/ Lot No------------------Make-------------). -------------------gm

dissolved in -------------ml water (1000 ppm)

Dilute -----------ml of solution(1000ppm) to --------------ml of purified water.(10ppm)

Dilute -----------ml of solution (10ppm) to --------------ml of purified water (250ppb)

Dilute -----------ml of solution (10ppm) to-------------- ml of purified water (500ppb)

Dilute -----------ml of solution (10ppm) to --------------ml of purified water(750ppb)

Dilute -----------ml of solution (10ppm) to --------------ml of purified water(1000ppb)

Dilute -----------ml of solution (10ppm) to --------------ml of purified water(2000ppb)

Dilute -----------ml of solution (10ppm) to --------------ml of purified water(5000ppb)

Observations :

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sr.No | Concentration | Area Count | | | | | Average Area  Count | SD | RSD |
|  |  | I | II | III | IV | V |  |  |  |
| 1. | 10ppm |  |  |  |  |  |  |  |  |
| 2. | 0ppb |  |  |  |  |  |  |  |  |
| 3. | 250ppb |  |  |  |  |  |  |  |  |
| 4. | 500ppb |  |  |  |  |  |  |  |  |
| 5. | 750ppb |  |  |  |  |  |  |  |  |
| 6. | 1000ppb |  |  |  |  |  |  |  |  |
| 7. | 2000ppb |  |  |  |  |  |  |  |  |
| 8. | 5000ppb |  |  |  |  |  |  |  |  |
| Slope | |  | | | | |  | | |
| Intercept | |  | | | | |  | | |
| Correlation coefficient | |  | | | | |  | | |

Acceptance criteria : Linearity : Correlation coefficient should not be less than 0.98

Standard Deviation: NMT 1.0

RSD (C.V.) : NMT 5.0%

Remarks : Complies / Does not Comply

Done by : Checked By : Authorised By :

Date : Date : Date :

**APPENDIX III ZYX/CQA/SOP-039/FR-03**

**CALIBRATION BY SUCROSE (Alternate Method)**

(Note : Calibration shall be done on the day of sample analysis)

1.0 Instrument ID.. : -------------------------

2.0 Location : -------------------------

3.0 Date of Calibration :-------------------------

4.0Preparation of Standard solution

Wt of Sucrose (Batch/ Lot No------------------Make-------------)------------------.gm

dissolved in -------------ml water (50 ppm)

Dilute-------- ml of solution (50ppm) to---------------ml Purified water (250ppb)

Dilute-------- ml of solution (50ppm) to---------------ml Purified water (500ppb)

Dilute-------- ml of solution (50ppm) to---------------ml Purified water (1000ppb)

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sr.No | Concentration | Area Count | | | | | Average Area  Count | SD | RSD |
|  |  | I | II | III | IV | V |  |  |  |
| 1. | 0ppb |  |  |  |  |  |  |  |  |
| 2. | 250ppb |  |  |  |  |  |  |  |  |
| 3. | 500ppb |  |  |  |  |  |  |  |  |
| 4. | 1000ppb |  |  |  |  |  |  |  |  |
| Slope | |  | | | | |  | | |
| Intercept | |  | | | | |  | | |
| Correlation coefficient | |  | | | | |  | | |

Acceptance criteria : Linearity : Correlation coefficient should not be less than 0.98

Standard Deviation : NMT 1.0

RSD : NMT 5.0%

Remarks : Complies / Does not Comply

Done by : Checked By : Authorised By :

Date : Date :: Date :

**APPENDIX IV ZYX/CQA/SOP-039/FR-04**

SYSTEM SUITABILITY BY 1,4 BENZOQUINONE (WEEKLY)

1.0 Instrument ID :------------------- :

2.0 Location :---------------------

3.0 Date of System Suitability :----------------------

4.0 Preparation of Standard solution

4.1 Wt of 1,4 Benzoquinone (Batch/ Lot No------------------Make-------------)------------------.gm

dissolved in -------------ml water (50 ppm)

Dilute-------- ml of solution (50ppm) to---------------ml Purified water (500ppb)

* 1. Observations:

Mean Area of Sucrose----------------

Mean area of 1.4 Benzoquinone----------------.

Mean area of pure water------------------

4.3 Calculation of Response efficiency (% recovery)

% R = (Rss - Rw)

---------------- X 100

(Rs-Rw)

Where Rss = Mean area counts of 1,4 Benzoquinone solution

Rs = Mean area counts of sucrose

Rw = Mean area counts of pure water

Acceptance criteria : The system is said to be passing the test if the % recovery in between 85% to 115%.

Remarks : Complies / Does not comply

Done by ; Checked by ; Authorized by :

Date: Date : Date

**APPENDIX V ZYX/CQA/SOP-039/FR-05**

**INSCAL--------------------**

**CALIBRATION BY POTASSIUM HYDROGEN PHTHALATE, OR SUCROSE, 1.4 BENZOQUINONE**

**(HALF YEARLY RECORD)**

1.0 Instrument ID :------------------------------

2.0 Location :------------------------------

3.0 Date of Calibration :------------------------------

4.0 Next Due date ofCalibration :------------------------------

**5.0 By KHP**

5.1 Preparation of Standard solution

5.2 Wt of KHP (Batch/ Lot No------------------Make-------------)------------------.gm , dissolved in -------------ml water (1000ppm)

Dilute -----------ml of solution(1000ppm) to --------------ml of purified water.(10ppm)

Dilute -----------ml of solution (10ppm) to --------------ml of purified water (250ppb)

Dilute -----------ml of solution (10ppm) to-------------- ml of purified water (500ppb)

Dilute -----------ml of solution (10ppm) to --------------ml of purified water(750ppb)

Dilute -----------ml of solution (10ppm) to --------------ml of purified water(1000ppb)

Dilute -----------ml of solution (10ppm) to --------------ml of purified water(2000ppb)

Dilute -----------ml of solution (10ppm) to --------------ml of purified water(5000ppb)

Observations :

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sr.No | Concentration | Area Count | | | | | Average Area  Count | SD | RSD | Acceptance criteria |
|  |  | I | II | III | IV | V |  |  |  | Correlation coefficient should not be less than 0.98  SD : NMT 1.0  RSD : NMT 5.0% |
| 1. | 10ppm |  |  |  |  |  |  |  |  |
| 2. | 0ppb |  |  |  |  |  |  |  |  |
| 3. | 250ppb |  |  |  |  |  |  |  |  |
| 4. | 500ppb |  |  |  |  |  |  |  |  |
| 5. | 750ppb |  |  |  |  |  |  |  |  |
| 6. | 1000ppb |  |  |  |  |  |  |  |  |
| 7. | 2000ppb |  |  |  |  |  |  |  |  |
| 8. | 5000ppb |  |  |  |  |  |  |  |  |
| Slope | |  | | | | |  | | |
| Intercept | |  | | | | |  | | |
| Correlation coefficient | |  | | | | |  | | |

**6.0 By Sucrose**

6.1 Preparation of Standard solution

Wt of Sucrose (Batch/ Lot No------------------Make-------------)------------------.gm

dissolved in -------------ml water (50 ppm)

Dilute-------- ml of solution (50ppm) to---------------ml Purified water (250ppb)

Dilute-------- ml of solution (50ppm) to---------------ml Purified water (500ppb)

Dilute-------- ml of solution (50ppm) to---------------ml Purified water (1000ppb)

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sr.No | Concentration | Area Count | | | | | Average Area  Count | SD | RSD |
|  |  | I | II | III | IV | V |  |  |  |
| 1 | 0ppb |  |  |  |  |  |  |  |  |
| 2. | 250ppb |  |  |  |  |  |  |  |  |
| 3. | 500ppb |  |  |  |  |  |  |  |  |
| 4. | 1000ppb |  |  |  |  |  |  |  |  |
| Slope | |  | | | | |  | | |
| Intercept | |  | | | | |  | | |
| Correlation coefficient | |  | | | | |  | | |

Acceptance criteria : Linearity : Correlation coefficient should not be less than 0.98

Standard Deviation : NMT 1.0

RSD : NMT 5.0%

**7.0 By 1,4 Benzoquinone**

Preparation of Standard solution

7.1Wt of 1’4 Benzoquinone (Batch/ Lot No------------------Make-------------)------------------.gm

dissolved in -------------ml water (50 ppm)

Dilute-------- ml of solution (50ppm) to---------------ml Purified water (500ppb)

7.2 Observations:

Mean Area of Sucrose----------------

Mean area of 1.4 Benzoquinone----------------.

Mean area of pure water------------------

7.3 Calculation of Response efficiency (% recovery)

% R = (Rss - Rw)

---------------- X 100

(Rs-Rw)

Where Rss = Mean area counts of 1,4 Benzoquinone solution

Rs = Mean area counts of sucrose

Rw = Mean area counts of pure water

Acceptance criteria : The system is said to be passing the test if the % recovery in between 85% to 115%.

Remarks : Complies / Does not comply

Done by ; Checked by ; Authorized by :

Date: Date : Date :

**APPENDIX VI ZYX/CQA/SOP-039/FR-06**

**TOC SUMMARY SHEET INSCAL----------------------**

**Instrument ID : -------------------------------**

**Location : -------------------------------**

|  |  |  |  |
| --- | --- | --- | --- |
| **TEST** | **OBSERVATION** | **ACCEPTANCE CRITERIA** | **STATUS** |
| By Potassium hydrogen phthalate or  By Sucrose |  | Linearity : Correlation coefficient should not be less than 0.98  Standard Deviation : NMT 1.0  RSD : NMT 5.0% | Complies/ Does not Comply |
| By 1,4 Benzoquinone |  | The system is said to be passing the test if the % recovery in between 85% to 115% | Complies/ Does not Comply |

**Remarks:** **All the parameters for the calibration as per predefined acceptance criteria are under limit hence the instrument is suitable**

**for routine analysis**

Remarks : Complies / Does not comply

Done by ; Checked by ; Authorized by :

Date: Date : Date :