

CONTROL CHART ON SEMI ANALYTICAL WEIGHTING

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Abstract: Semi-analytical balance verification intends to assess the balance performance using graphs that illustrate measurement dispersion trough time, to demonstrate measurements were performed in a reliable manner. This study presents internal quality control of a semi-analytical balance (GEHAKA BG400) using control charts. From 2013 to 2016, 2 weight standards were monitored before any balance operation. This work intended to evaluate if any significant difference or bias were presented on weighting procedure over time, to check the generated data reliability. This work also exemplifies how control intervals are established.

Keywords: Weighting procedure; Quality control chart; Semi-Analytical balance.

1. INTRODUCTION

The search for better performance, for results reliability and competitiveness lead laboratories to adopt a Quality Assurance System [1,2]. ISO/IEC 17025 specifies general requirements to calibration and analysis laboratories, including sampling. Laboratories that work in accordance with this standard also follow the requirements of ISO 9001, due to 2005 revision, in order to establish the quality assurance system definitions [3]. Items 5.5 and 5.9 of ISO/IEC 17025 describe data quality assurance from test and calibration, regarding the use of certified reference materials (CRM), interlaboratory programs and/or internal quality control. Also emphasize the requirement of data register in a way that bias can be detected and, when possible, apply statistical analysis to evaluate results [4].

One of the oldest statistical technique to internal quality control is the use of quality control charts. Their applications to data from

analytical instruments assure the traceability and reliability of the analytical results [3,4].

ISO 8258/91, revised and altered to ISO 7870/13, presents the basic principles and concepts correlated to Shewhart control charts [2,4].

Aiming to achieve results reliability, using a statistical control, this study uses quality control charts, to assess the performance of a semi-analytical balance (GEHAKA BG400), in order to identify if any bias is present and to propose corrective actions, if necessary.

2. MATERIALS & METHODS

The elaboration, deployment and use of quality control charts to the semi-analytical balance assessment (GEHAKA BG400 - Figure 1), in a lab in Centro de Química e Meio Ambiente (CQMA) at IPEN/CNEN-SP, was carried out from June/2013 to 2016, in a total of

766 measurements. The semi-analytical balance specifications are presented in Table 1.



Figure 1. Semi-Analytical Balance - GEHAKA BG400

Table 1. Semi Analytical Balance Specification.

| Parameters | Specification |
|----------------|---------------|
| Dimensions | 240x260x55 mm |
| Weight | 5.2 kg |
| Maximum charge | 404 g |
| Minimum charge | 0.001 g |
| Plate diameter | 110 mm |

The process to be controlled was the semi-analytical balance response, regarding the variability provided by different analysts, environmental conditions, preventive maintenance and instrument calibration. Two different standard weights, stable and uniform, named “U” and “S” were used. Quality control charts were elaborated using the program Microsoft Excel. Central limits calculations relied on the standard weight total average.

Operation limits were established as: Satisfactory (average $\pm 1\sigma$); Warning (average $\pm 2\sigma$); and Unsatisfactory (average $\pm 3\sigma$).

Decision criteria adopted meet ISO 8258 – Shewhart Control Charts requirements, as cited below [5]:

- a) 1 or more data above superior limit or below inferior limit;
- b) 9 consecutive data in the same side of satisfactory zone;
- c) 6 consecutive data, all increasing or decreasing;
- d) 14 consecutive data alternated between below and above;
- e) 2 of 3 consecutive data in unsatisfactory zone and beyond;
- f) 4 of 5 consecutive data in warning zone or beyond it;
- g) 15 consecutive data in satisfactory zone;
- h) 8 consecutive data the warning zone.

3. RESULTS AND DISCUSSION

Figure 2 presents the histogram and measurement normal distribution performed to standard weights U and S.

Figure 3 presents control charts to standard weights U and S, with 383 measurements for each standard weight (from June/2013 to December/2016) using the semi-analytical balance. Warning limits to weight U were between 0.755 g and 0.751g; unsatisfactory limits were between 0.756 g and 0.750 g. To standard weight S, warning limits were between 0.811 g and 0.815 g; unsatisfactory limits were between 0.810 g and 0.816 g. Data over satisfactory limits could arise from measurement errors or typos; failure of unregulated instrument and/or operators failure [4,6].

Table 2 presents the descriptive statistics to standard weights U and S. No value from standard U (n = 383) neither from standard S (n = 383) were out of control.

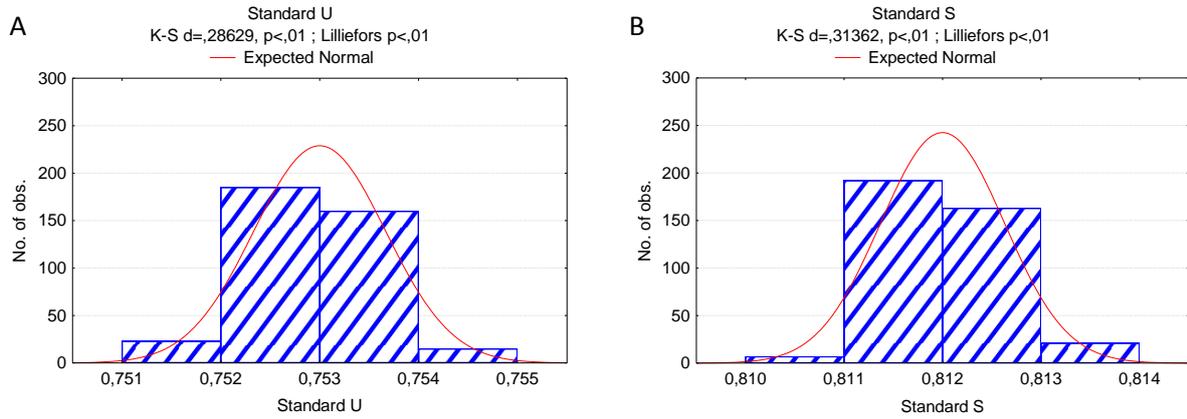


Figure 2: Histogram and expected normal distribution of standard weights (A) U and (B) S.

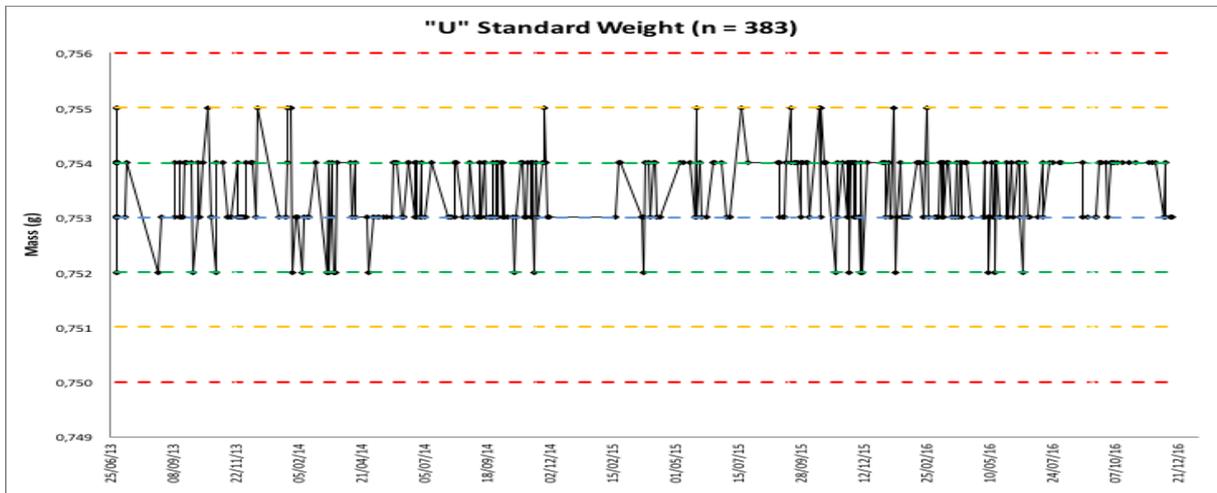


Figure 3: Standard U quality control chart.

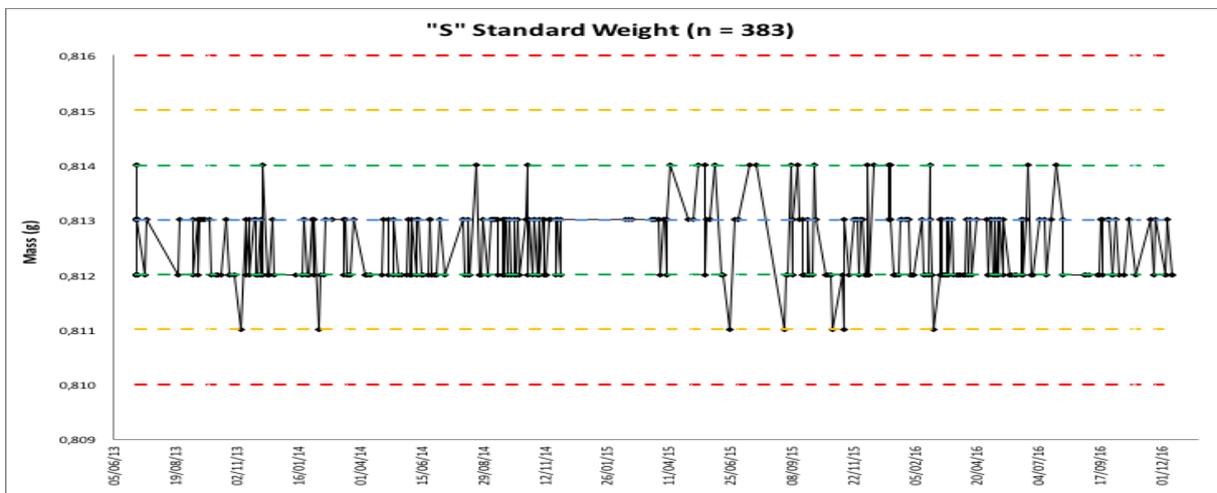


Figure 3: Standard S quality control chart.

In order to guarantee the proper functioning and durability of the semi-analytical balance, a calibration is annually performed. Last calibration was carried out in August/2016.

Table 2: Standard U and S descriptive statistics.

| | Standard U | Standard S |
|---|------------|------------|
| Average (g) | 0.753 | 0.813 |
| s (g) | 0.001 | 0.001 |
| RSD (%) | 0.133 | 0.123 |
| n | 383 | 383 |
| Median (g) | 0.753 | 0.812 |
| Maximum (g) | 0.755 | 0.814 |
| Minimum (g) | 0.752 | 0.811 |
| Number of warning data | 15 | 7 |
| % of values in the warning limit | 3.92 | 1.83 |

A periodically performed calibration and the internal quality control are necessary to ensure normal operational conditions to semi-analytical balances, as well as demonstrate its good performance.

4. CONCLUSIONS

Quality control chart, used as assessment tool for measurements performed in a semi-analytical balance, could be applied to ensure instrument correct use, its perfect conditions, even after maintenance and calibration process, performed by accredited companies (NBR ISO IEC 17025). As no unsatisfactory data was observed, no corrective actions were necessary. Warning observed values were attributed to measurements performed before equipment stabilization or to laboratory temperature changes.

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