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STATISTICAL PROCESS CONTROL BASED PERFORMANCE EVALUATION OF ON-LINE ANALYSERS

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On-line analyzers can provide accurate and timely information for process control and monitoring. Statistical Process Control (SPC) techniques can be effectively utilized to support the development and maintenance of these tools. The D6299-10 ASTM standard details how on-line analyzers should be validated. The applicability of this standard is demonstrated through the analysis of industrial data collected from an on-line gas chromatograph. The results confirm that automatized SPC can effectively improve the reliability of advanced process control systems.

Keywords: on-line analyzer, software sensor, statistical process control, SPC, process monitoring

Introduction

Process variables characterizing and influencing product quality have a significant role in process control and optimization. Off-line laboratory tests mostly take more than two hours. This time delay can cause control problems resulting in economic loss. In such situations, an improved on-line monitoring system is required. On-line analyzers eliminate the dependence on laboratory data. Analysers are valuable instruments for real time control because of their fast response time (1-4 minutes) (see *Fig. 1*) [1].

Quality control techniques can be effectively used to support the development of on-line analyzers [2] and advanced process control systems [3]. The D 6299 ASTM standard (Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance) provides information for the design and operation statistical quality control (QC) tools to monitor and control of analytical measurement systems using a collection of statistical quality control (SQC) tools [4].

The goal of the performance monitoring is the periodic comparison of the on-line analyzer's results to the reference value of the same sample measured by laboratory test methods. Precision and bias (see *Fig. 2*) are calculated to provide information for updating test methods as well as for indicating areas of potential improvements.

Control charts and other statistical techniques can be used for performance monitoring. Statistical estimates of the measurement system precision and bias can be calculated on the basis of periodically updated data. Plotting and interpreting these test results can ascertain the in-statistical-control status of the measurement system [5]. On-line Statistical Process Control (SPC) based real-time validation of measurement systems has been already re-

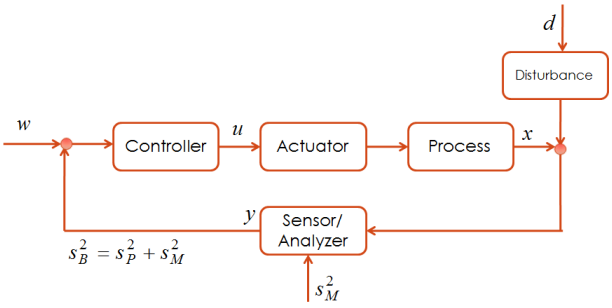

Figure 1: Soft sensors and on-line analyzers enable feedback control

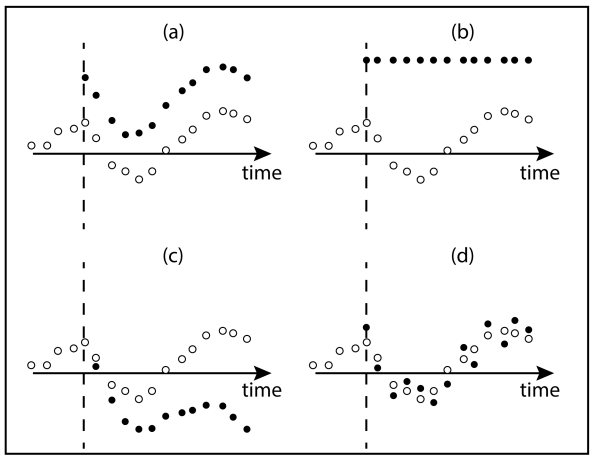

Figure 2: Type of faults. The dashed line shows when the fault occurs. ○: data free of fault; ● corrupted data for the following cases: (a) bias, (b) complete failure, (c) drifting, and (d) precision degradation [4]

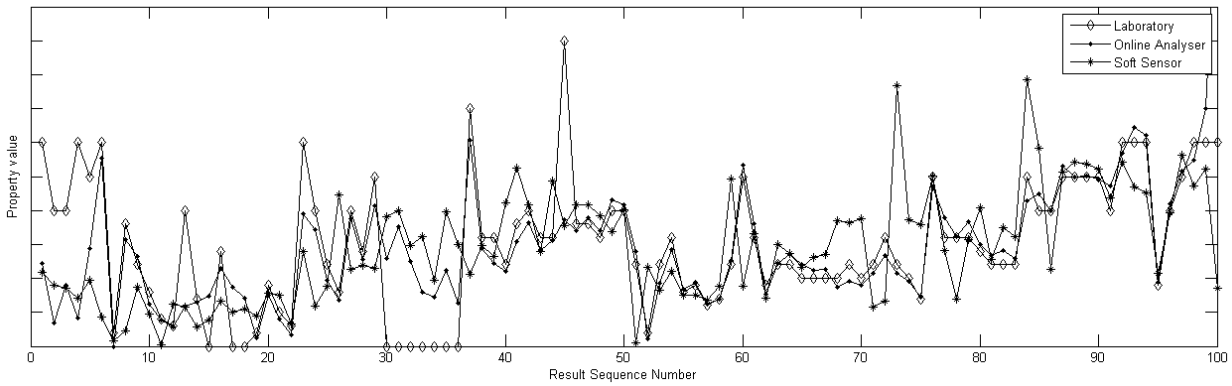

Figure 3: Illustrative example: MOL aromatic block gas chromatograph IP benzene content (m/m %), Lab, Analyser and APC soft sensor

ported in 1997 [6]. The SPC based approach can also be applied for off-line performance evaluation [7]. This approach has a wide range of application areas like steel industry [8], electronic device assembling [9], and buildings' energy demand monitoring [10].

The goal of this paper is to present the theoretical background and application details of SPC based performance evaluation of on-line analyzers. The applicability of the concepts is illustrated by a case study based on data collected from an on-line gas chromatograph of the MOL Plc (see *Fig. 3*) . Typical patters that show out of control status of the system are also presented. To check the normality of the residuals, an easily applicable and interpretable tool is proposed. The developed demonstration tools are available at the website of the authors (www.abonyilab.com).

Control Chart based Evaluation of System Performance

The studied D6299 practice is devoted to a special testing of analyzers [4]. Quality Control (QC) test specimen samples from a specific lot are introduced and tested in the analytical measurement system on a regular basis to establish system performance history in terms of both stability and precision.

The control chart is one of the seven basic tools of QC. Control charts - also known as SHEWHART charts - are used to determine if process is in a state of statistical control. The analysis of the control chart indicates whether the process is currently under control. This means that these charts are used to check the stability of the production. In stable operation, the variations of the process and quality variables are only random, normally distributed variables. In these cases, no corrections of the control parameters are needed. When the chart indicates that the monitored process is out-of-control, the analysis of the chart can help to determine the sources of the variation. Typically, control charts are used for time-series data, though they can be used for data that have logical comparability [11]. In this section, we present these charts and detail how these should be applied for the performance assessment of on-line analyzers.

Control Charts

Run Chart

The run chart is a plot of sample values in chronological order (*Fig. 4*). The run chart can be used to screen data for unusual patterns such as continuous trending in either direction, unusual clustering, and cycles. The run chart of the data used in our case study is shown in *Fig. 3*. The plotted time series shows the signal of the on-line analyzer and the related laboratory measurements.

I Chart

The I (individual) chart is a run chart to which control limits and center line have been added (see top panel of *Fig. 5*). The center line is based on the mean of the samples,

$$\bar{I} = \frac{\sum_{i=1}^{n} I_i}{n} \tag{1}$$

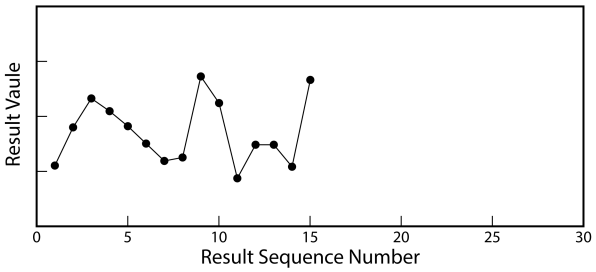

Figure 4: Example for a run chart

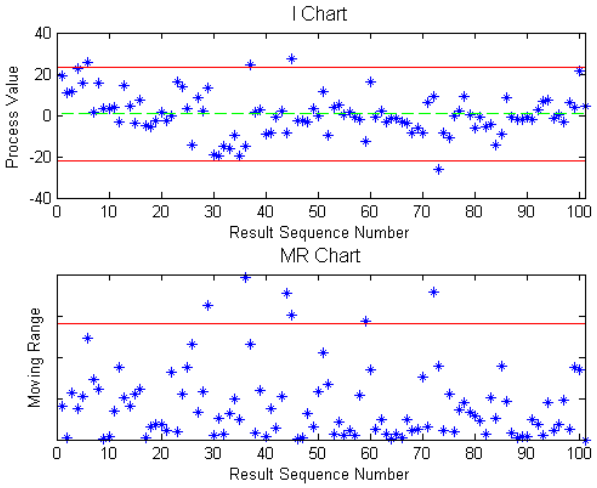

Figure 5: I and MR charts show out of control samples of the gas chromatograph IP benzene content (m/m) %

while the upper and lower control limits are based on the estimated variance (range of the data) $\overline{MR}$ [12]

$$\overline{MR} = \frac{\sum_{i=1}^{n-1} |I_{i+1} - I_i|}{n-1}, \tag{2}$$

$$UCL_I = \bar{I} + 2.66\overline{MR}, \tag{3}$$

$$LCL_I = \bar{I} - 2.66\overline{MR}. \tag{4}$$

Individual values that are outside the upper or lower control limits are indications of an unstable system, and efforts should be made to determine the cause [5]. Optionally, any one of the following occurrences should be considered as potential signs of instability:

1. Two out of three consecutive results on the I chart that are more than $1.77\overline{MR}$ distant from the center line in the same direction;
2. Five consecutive results on the I chart that are more than $0.89\overline{MR}$ distant from the center line in the same direction;
3. Eight or more consecutive points in the I chart that fall on the same side of the center line.

MR Chart

MR (Moving Range) charts are also used to detect unusual patterns by plotting the sequential range of two values given by

$$\Delta_i = MR_i = |I_i - I_{i-1}|, \tag{5}$$

and connecting each point (see *Fig. 6* for an example). There is no lower control limit for an MR chart [5]. The upper control limit for the MR chart is given by

$$UCL_{MR} = 3.27\overline{MR}. \tag{6}$$

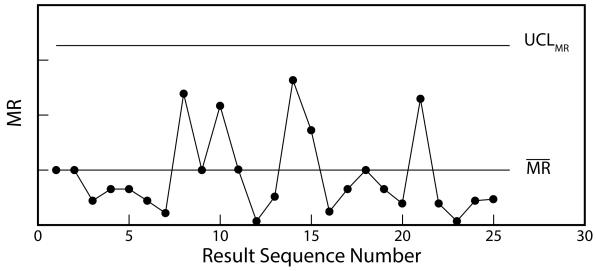

Figure 6: Example for a MR chart

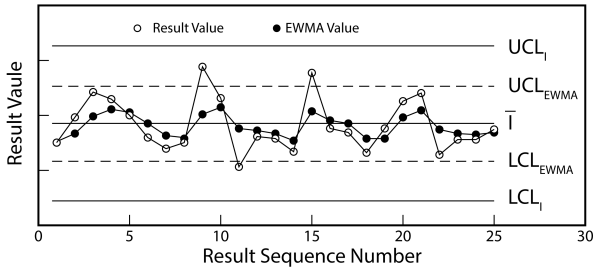

Figure 7: Example for an EWMA chart

EWMA chart

An EWMA (Exponentially Weighted Moving Average) chart is used to enhance the sensitivity in detecting mean shifts that are small relative to the measurement system precision (see *Fig. 7* for an example). Each $EWMA$ value is a weighted average of the current result and previous results with the weights decreasing exponentially with the age of the reading [5]:

$$EWMA_1 = I_1, \tag{7}$$

$$EWMA_i = (1-\lambda)EWMA_{i-1} + \lambda I_i, \tag{8}$$

where λ is the exponential weighting factor. For application of this practice, a λ value of 0.4 is recommended.

The control limits for the EWMA chart are calculated using a weight (λ) as follows:

$$UCL_\lambda = \bar{I} + 2.66\overline{MR}\sqrt{\frac{\lambda}{2-\lambda}}, \tag{9}$$

$$LCL_\lambda = \bar{I} - 2.66\overline{MR}\sqrt{\frac{\lambda}{2-\lambda}}. \tag{10}$$

The complexity of multivariate and autocorrelated processes makes it difficult to use standard control charts. To construct simple and interpretable charts, dimensional reduction could also be used as SIMOGLU and MARTIN have done [13], but in case of autocorrelated data, model-based control charts should be applied as KIM and JITPITAKLER have done in their research [14].

Pretreatment of Test Results

Assessment, control charting, and evaluation are applied only to appropriately pretreated test results. The purpose

of pretreatment is to standardize the control chart scales so as to allow for data from multiple check standards to be compared on the same chart. For QC sample test results, no data pretreatment is typically used since results for different QC samples are generally not plotted on the same chart.

In our case, the difference between the measurements and their accepted reference values (ARVs) are monitored. ARV serves as an agreed-upon reference for comparison and that is derived based on (1) a theoretical value, based on scientific principles, (2) an assigned value, based on experimental work, or (3) a consensus value, based on collaborative experimental work under the auspices of a scientific or engineering group.

$$I = \text{test result} - ARV. \tag{11}$$

Assessment of Initial Results

In the initial phase of the application assessment techniques are applied to test results collected during the startup phase of or after significant modifications to a measurement system. It is required to perform the following assessment after at least 15 pretreated results have become available. The purpose of this assessment is to ensure that these results are suitable for deployment of control charts.

Pretreated results should first be visually screened for values that are inconsistent with the remainder of the data set, such as those that could have been caused by transcription errors. Those flagged as suspicious should be investigated. Discarding data at this stage must be supported by evidence gathered from the investigation. If after discarding suspicious pretreated results there are less than 15 values remaining, collect additional data and start over.

The next step is to examine the pretreated results for non-random patterns such as continuous trending in either direction, unusual clustering, and cycles. One way to do this is to plot the results on a run chart and examine the plot. If any non-random pattern is detected, investigate for and eliminate the root cause(s).

Typical Control Chart Patterns

In the previous sessions, the main charts used for performance measurement were presented. In SPC, the Western Electric Rules are the decision rules for detecting "out-of-control" or non-random conditions on control charts. Locations of the observations relative to the control chart control limits (typically at ± 3 standard deviations) and centerline indicate whether the process in question should be investigated for assignable causes. The Western Electric Rules were codified by a specially-appointed committee of the manufacturing division of the Western Electric Company and appeared in the first edition of its Statistical Quality Control Handbook in 1956 [15]. Their

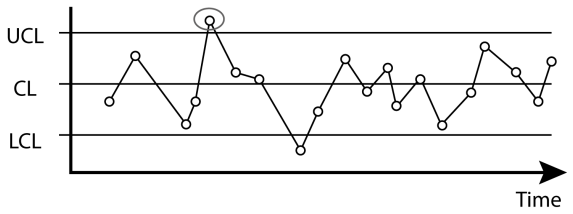

Figure 8: This process is out of control because a point is either above the UCL or below the LCL. For example, in *Fig. 5*-B at sequence number 28 there is a unique point below the LCL

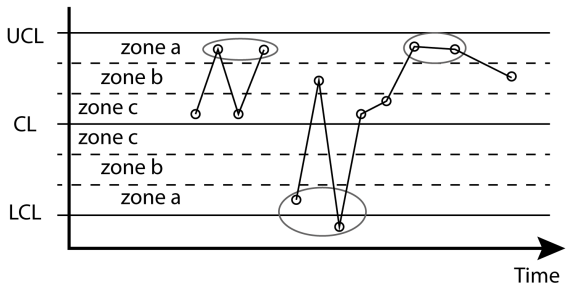

Figure 9: In this case the system produce 2 out of 3 consecutive points either in or beyond zone A. The process on *Fig. 5*-B shows this behaviour at sequence number 45 where the values are close to the Upper Control Limit.

purpose was to ensure that line workers and engineers interpret control charts in a uniform way [5, 12]. The eight standard Western electric rules are:

1. The most recent point plots outside one of the 3-sigma control limits (see *Fig. 8*). If a point lies outside either of these limits, there is only a 0.3% chance that this was caused by the normal process.

2. Two of the three most recent points plot outside and on the same side as one of the 2-sigma control limits (see *Fig. 9*). The probability that any point will fall outside the warning limit is only 5%. The chances that two out of three points in a row fall outside the warning limit is only about 1%.

3. Four of the five most recent points plot outside and on the same side as one of the 1-sigma control limits. In normal processing, 68% of points fall within one sigma of the mean, and 32% fall outside it. The probability that 4 of 5 points fall outside of one sigma is only about 3%.

4. Eight out of the last eight points plot on the same side of the center line, or target value. Sometimes you see this as 9 out of 9, or 7 out of 7. There is an equal chance that any given point will fall above or below the mean. The chances that a point falls on the same side of the mean as the one before it is one in

two. The odds that the next point will also fall on the same side of the mean is one in four. The probability of getting eight points on the same side of the mean is only around 1%.

5. Six points in a row increasing or decreasing. The same logic is used here as for rule 4 above. Sometimes this rule is changed to seven points rising or falling.

6. Fifteen points in a row within one sigma. In normal operation, 68% of points will fall within one sigma of the mean. The probability that 15 points in a row will do so, is less than 1%.

7. Fourteen points in a row alternating direction. The chances that the second point is always higher than (or always lower than) the preceding point, for all seven pairs is only about 1%.

8. Eight points in a row outside one sigma. Since 68% of points lie within one sigma of the mean, the probability that eight points in a row fall outside of the one-sigma line is less than 1% (see *Fig. 10*).

Normality Checks

Since the control chart and limits prescribed in this practice are based on the assumption that the data behavior is adequately modeled by the normal distribution, it is recommended that a test of this normality assumption be conducted. One way to do this is to use a normal probability plot and the ANDERSON-DARLING Statistic [16]. If the results show obvious deviation from normality, the statistical control charting techniques described are not directly applicable to the measurement system [5].

Quantile-quantile plot (q-q plot) is a graphical tool for comparing two probability distributions by plotting their quantiles against each other. The normality plot of the process can be obtained by comparing the empirical distribution of the data against a standard normal distribution

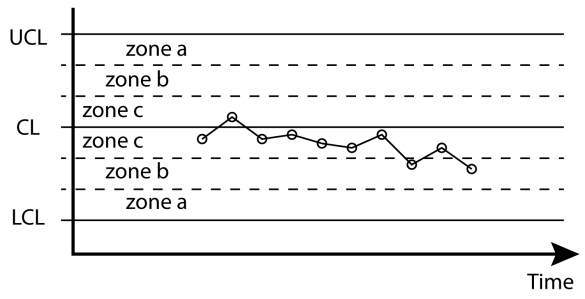

Figure 10: Long runs (8 or more consecutive points) either above or below the centerline. (See *Fig. 5*-A in range 28-40)

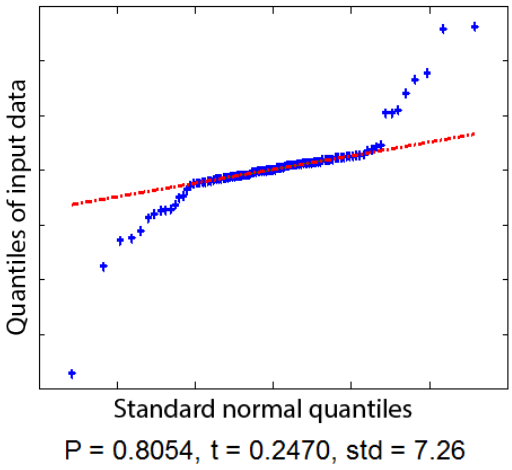

Figure 11: Normality check of the studied gas chromatograph.

(see *Fig. 11*). When the results are normally distributed, the plot should be approximately linear. Major deviations from linearity are an indication of non-normal distributions of the differences [17, 16].

Conclusions

We showed that statistical process control can be effectively used to support the development and maintenance of on-line process analyzers. A case study is presented based on the analysis of data taken from the chemical process industry. The proposed concept has been implemented in MATLAB. The results illustrate the applicability the developed tools in improving the reliability of advanced process control systems.

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