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National Physical Laboratory

Applying Trend Analysis Techniques to Quality Control Measurements in Radionuclide Metrology

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LSUF, NPL, 24th January 2006

Quality Control – ISO 17025:2005

5.9 Assuring the quality of test and calibration results

5.9.1 The laboratory shall have quality control procedures for monitoring the validity of tests and calibrations undertaken. The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results. This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

a) regular use of certified reference materials and/or **internal quality control** using secondary reference materials…

*NOTE The selected methods should be **appropriate** for the type and volume of the work undertaken.*

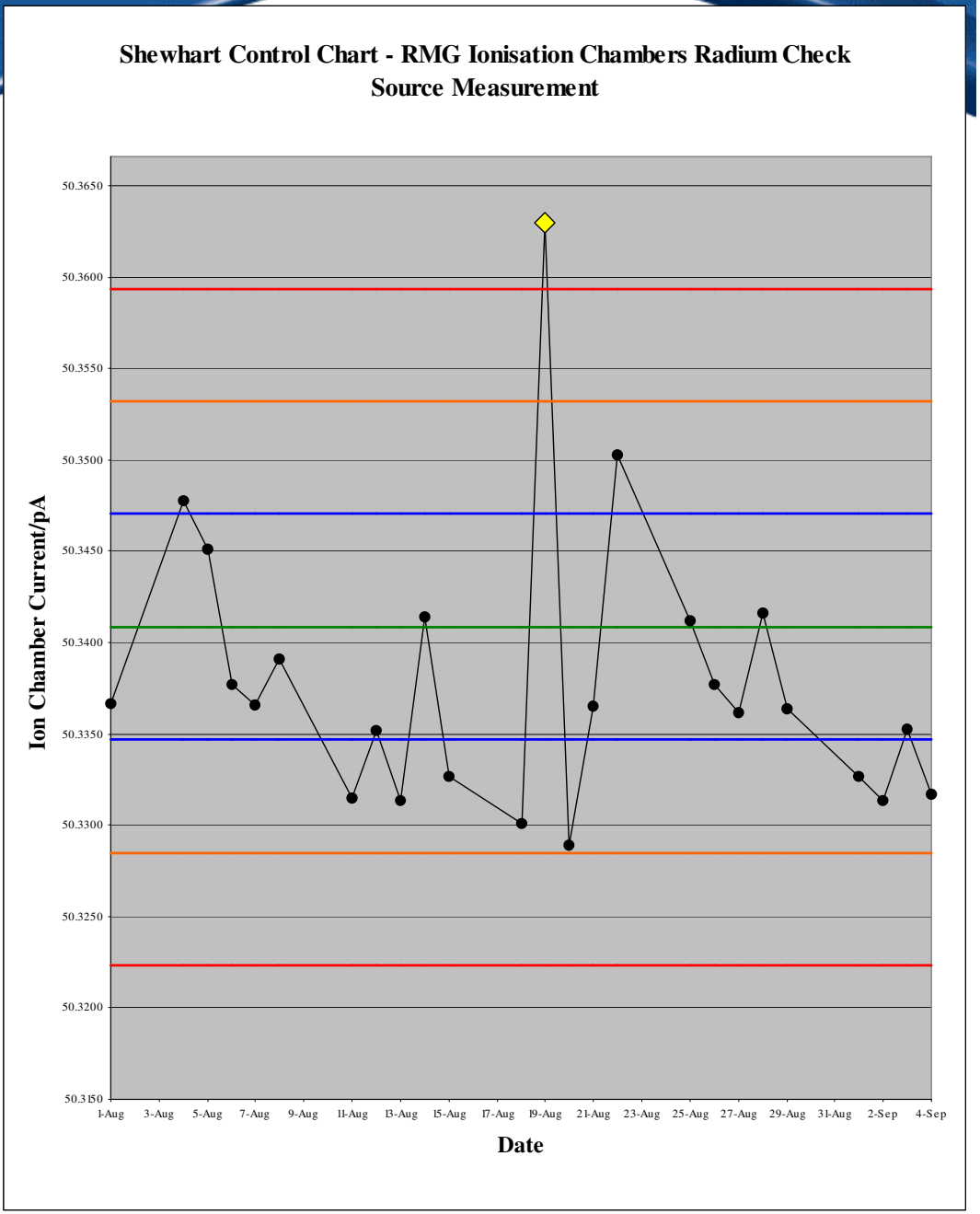
Two Techniques Commonly Used (More Are Available)

- Shewhart – more easily interpreted
- CuSum (Cumulative Summation) – more sensitive to change

Use of Shewhart charts

- Common usage is to set 2σ and 3σ limits, based on 11-31 replicate results
- Action taken if 3σ limit is exceeded – expected 3 times in 1000
- Warning is registered if 2σ but not 3σ limit is exceeded – expected 5 times in 100
- Straightforward, but no 'warning signals' before warning or action levels breached

- 3σ exceeded – action required immediately, since the procedure is out of control. Measurement should stop until the cause of the action limit violation is identified and corrected.



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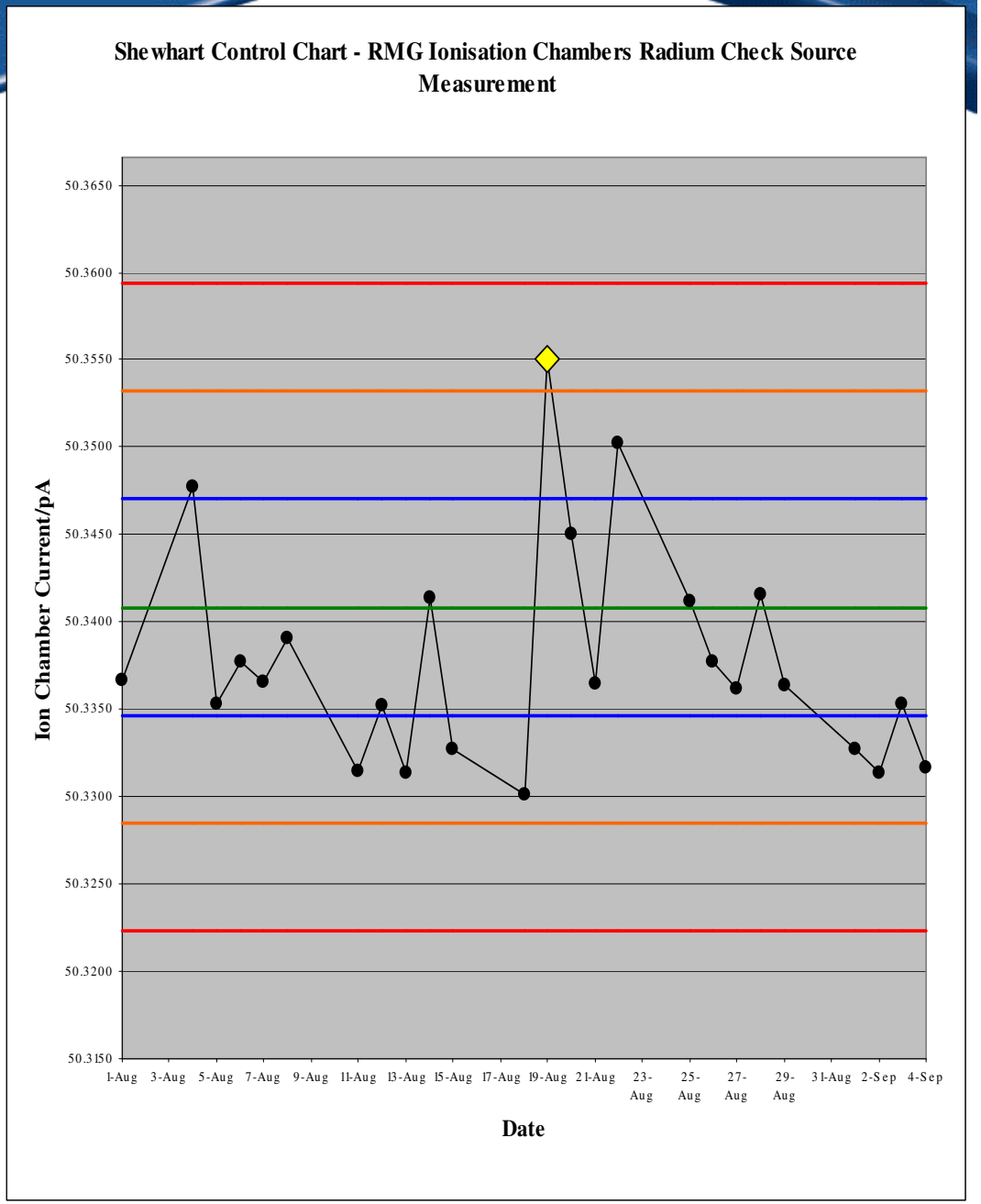
- 2σ exceeded – situation needs to be closely monitored



ISO 8258:1991 – Shewhart Control Charts

- Introduces the concept of Trend Analysis
- Relies on the range observed in measurement:

$$r_j = |x_{(j+1)} - x_j|$$

$$X = \frac{\sum_j x'_j}{N'}$$

$$R = \frac{\sum_j r'_j}{(N-1)'}$$

ISO 8258:1991 – Shewhart Control Charts

- Action limit set at: $X \pm R \times 2.66$
- Warning limit set at: $X \pm R \times 1.77$
- 'Information' limit set at: $X \pm R \times 0.89$

Based on a series of single observations; limits vary depending on the batch size and subgroup

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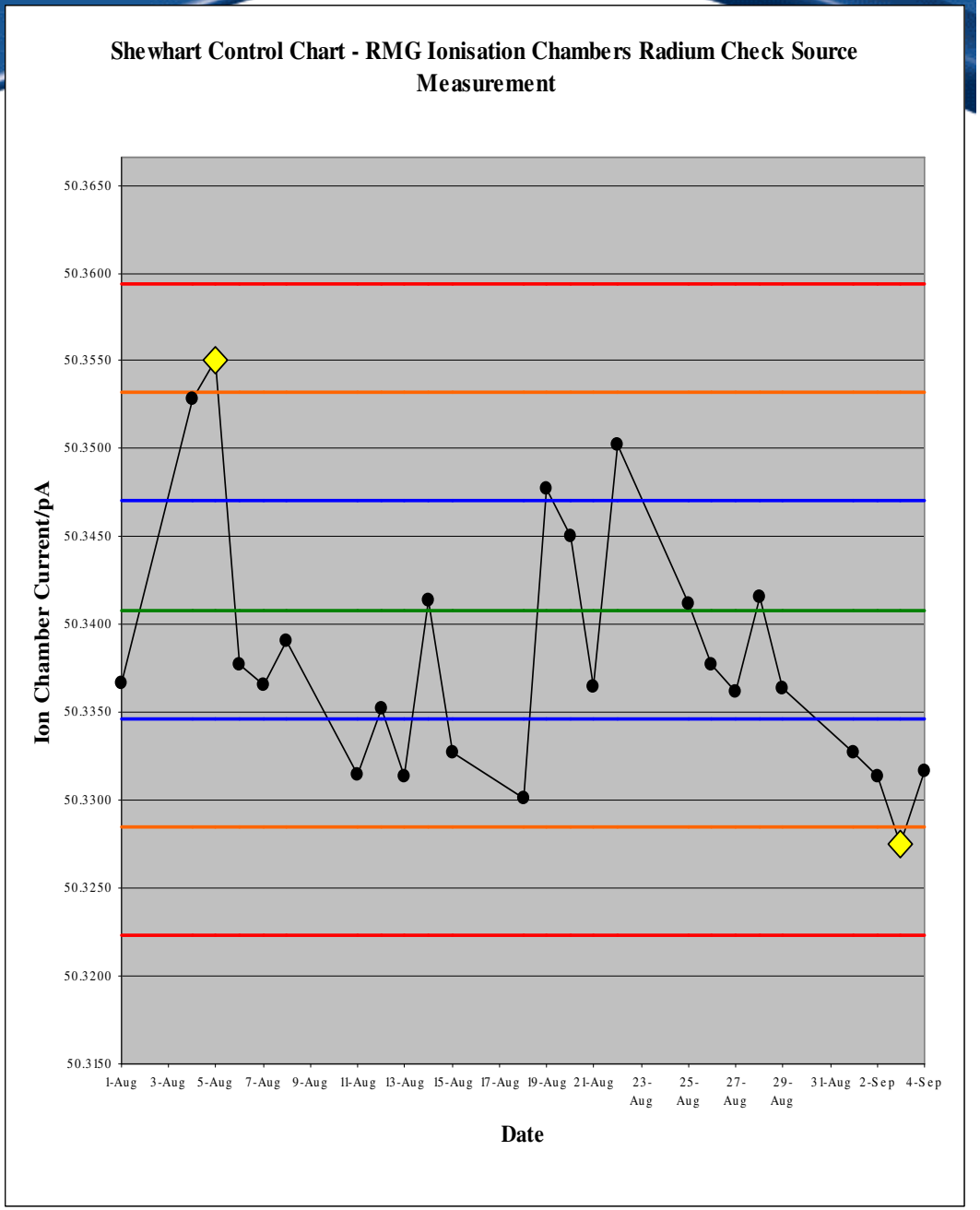
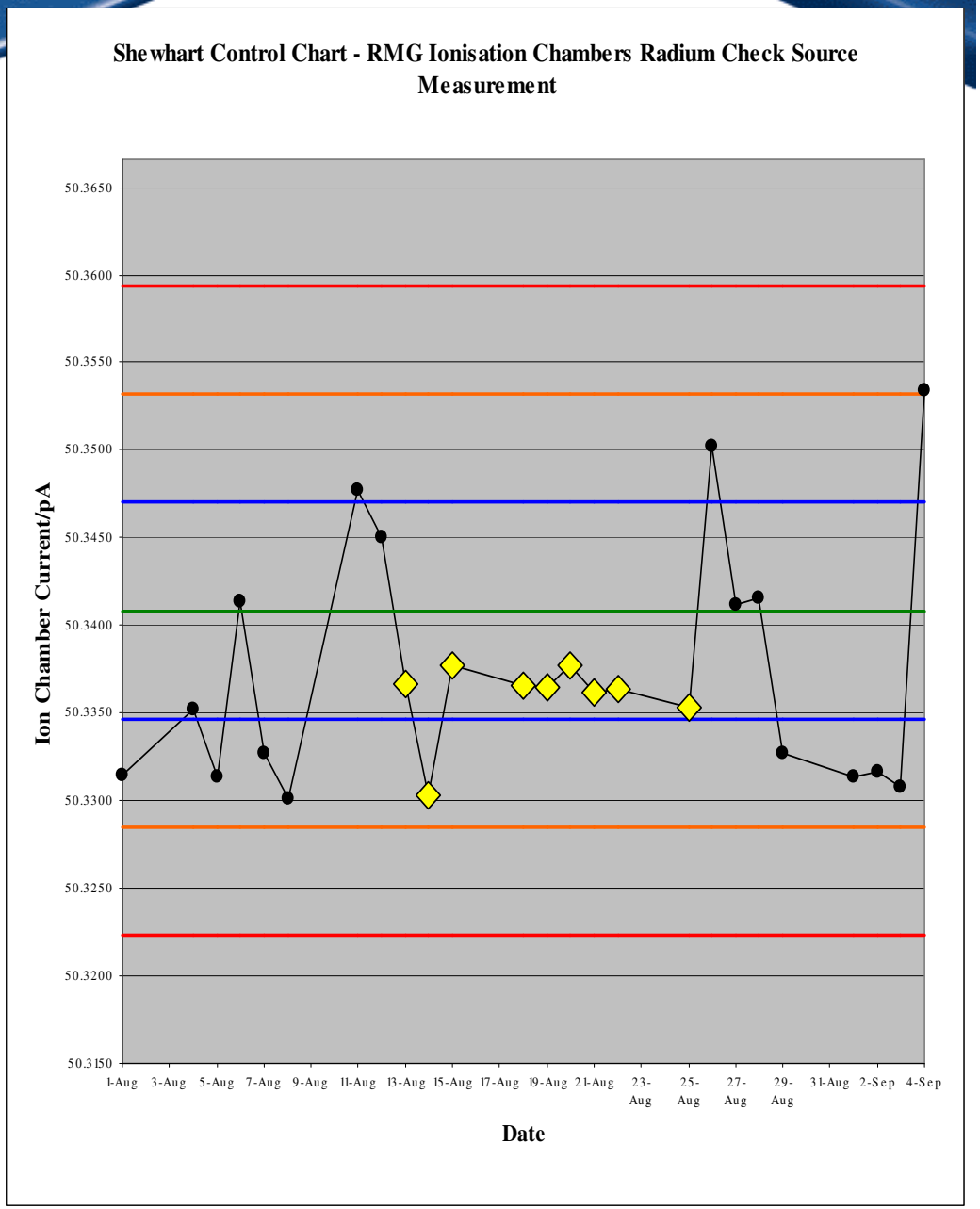
- Warning Limit Exceeded twice in under 42 measurements. Measurement should stop until the cause of the action limit violations are identified and corrected. (ISO 8259:1991 suggests 2 out of 3 is an action point)



- Nine points in a row on one side of the mean. This may indicate that there has been a shift in the mean; work can continue, however the cause of the shift needs to be identified and the limits re-set if justified



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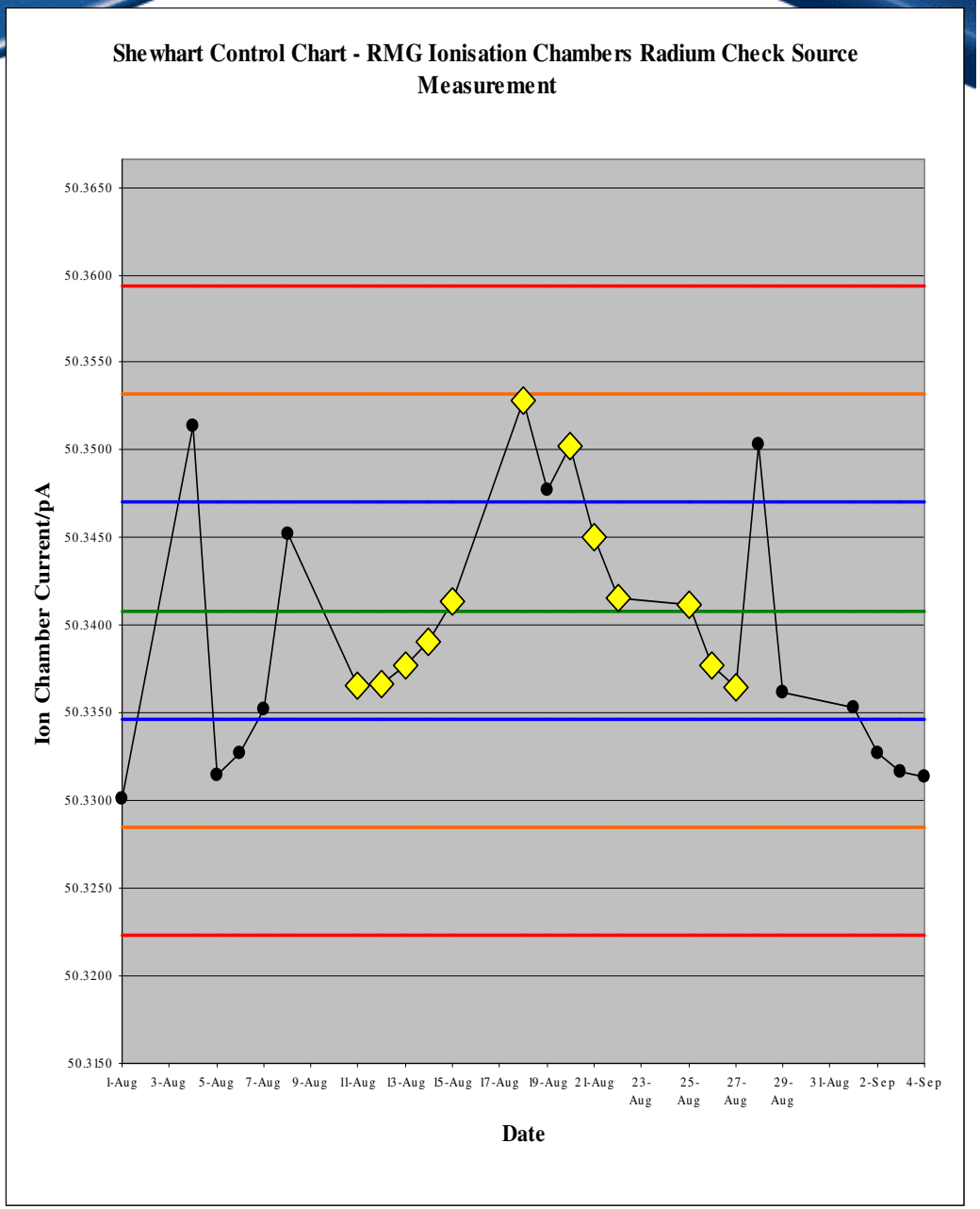
- Six points in a row steadily increasing or decreasing. Suggests that the measurements are shifting with time; needs investigation, since the action and warning limits will eventually be exceeded



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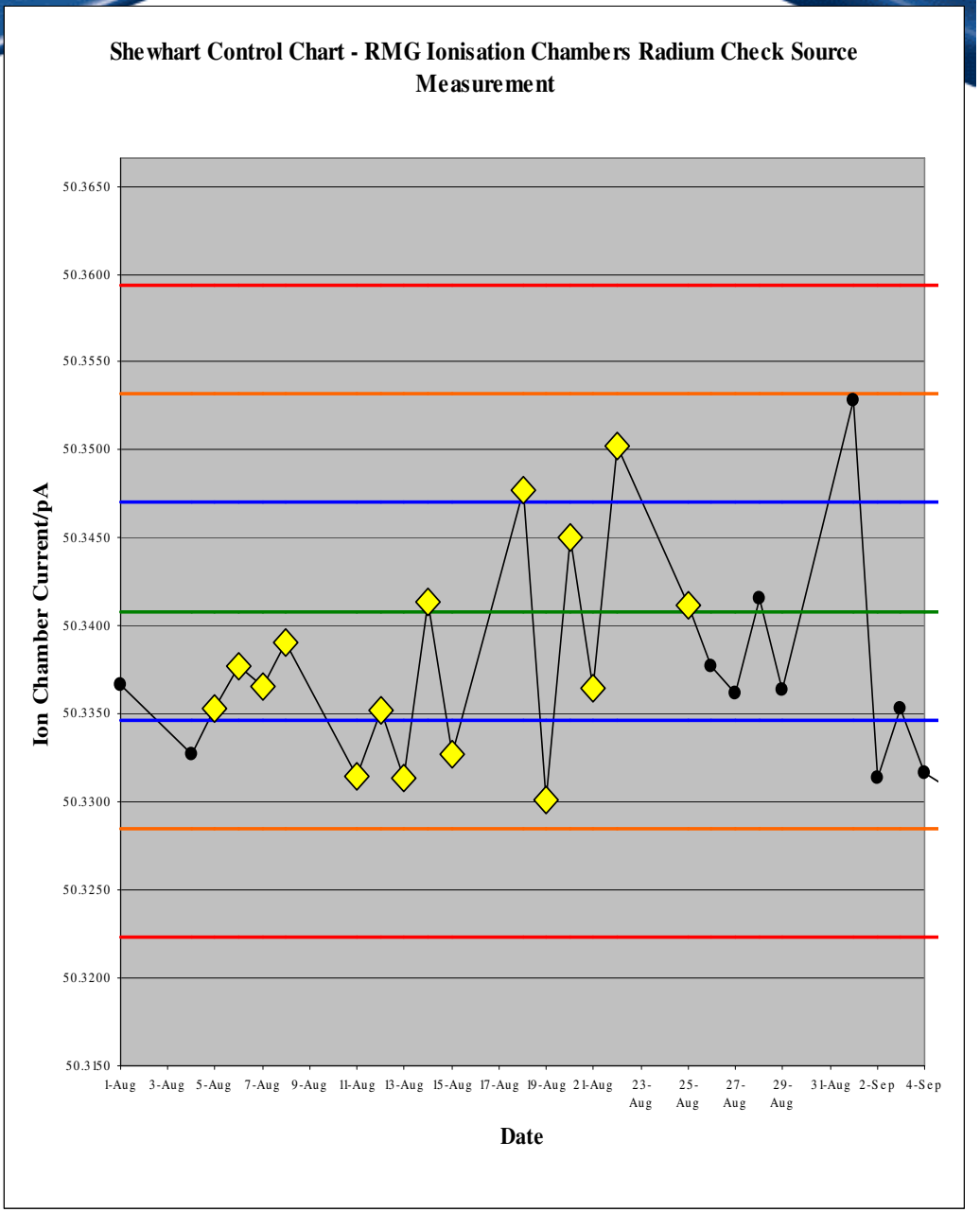
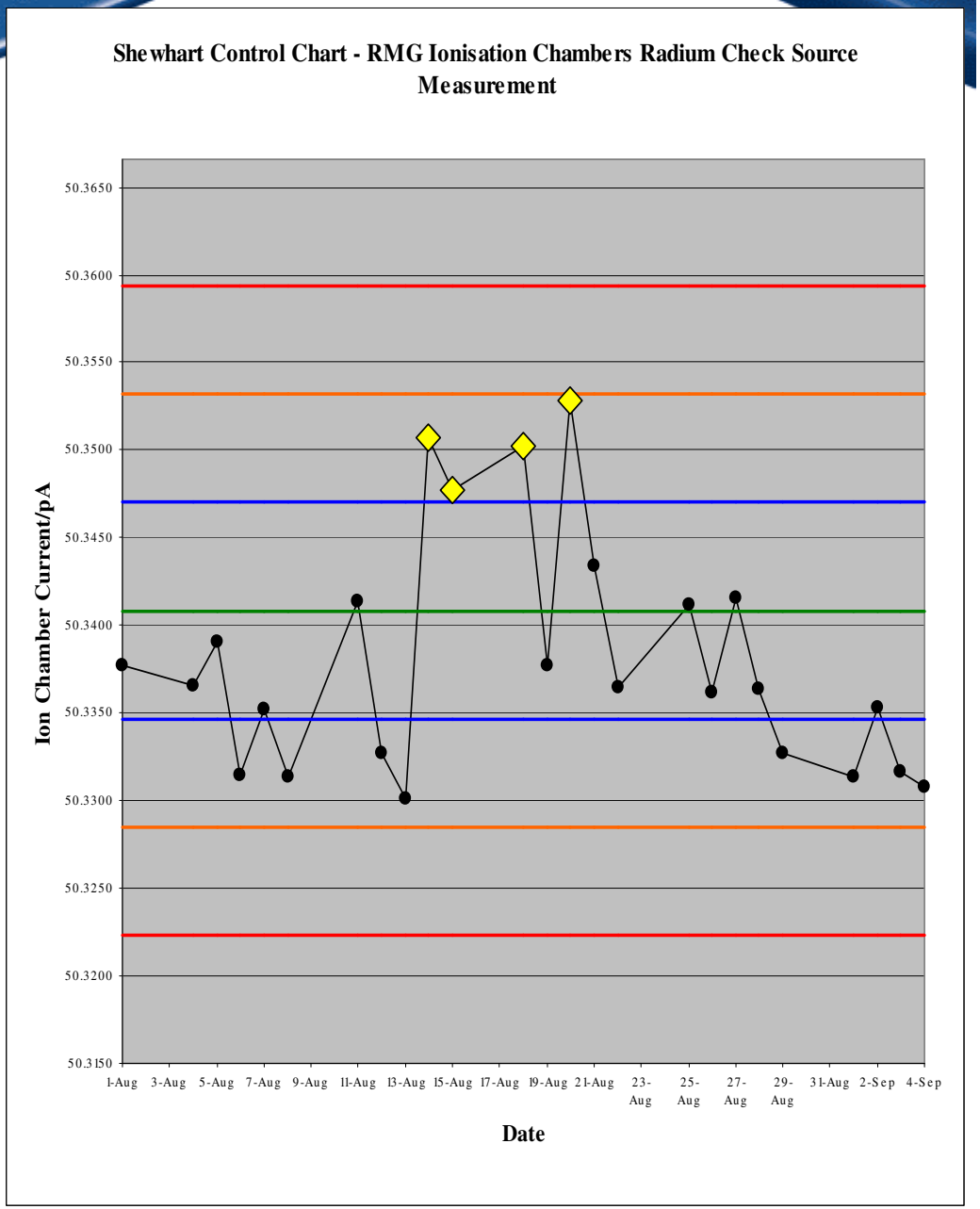
- Fourteen points in a row alternating up and down. Suggests a cyclical change in conditions; may be due to two different operators



- Four out of five points in a row exceeding the information level. This may indicate a mean shift



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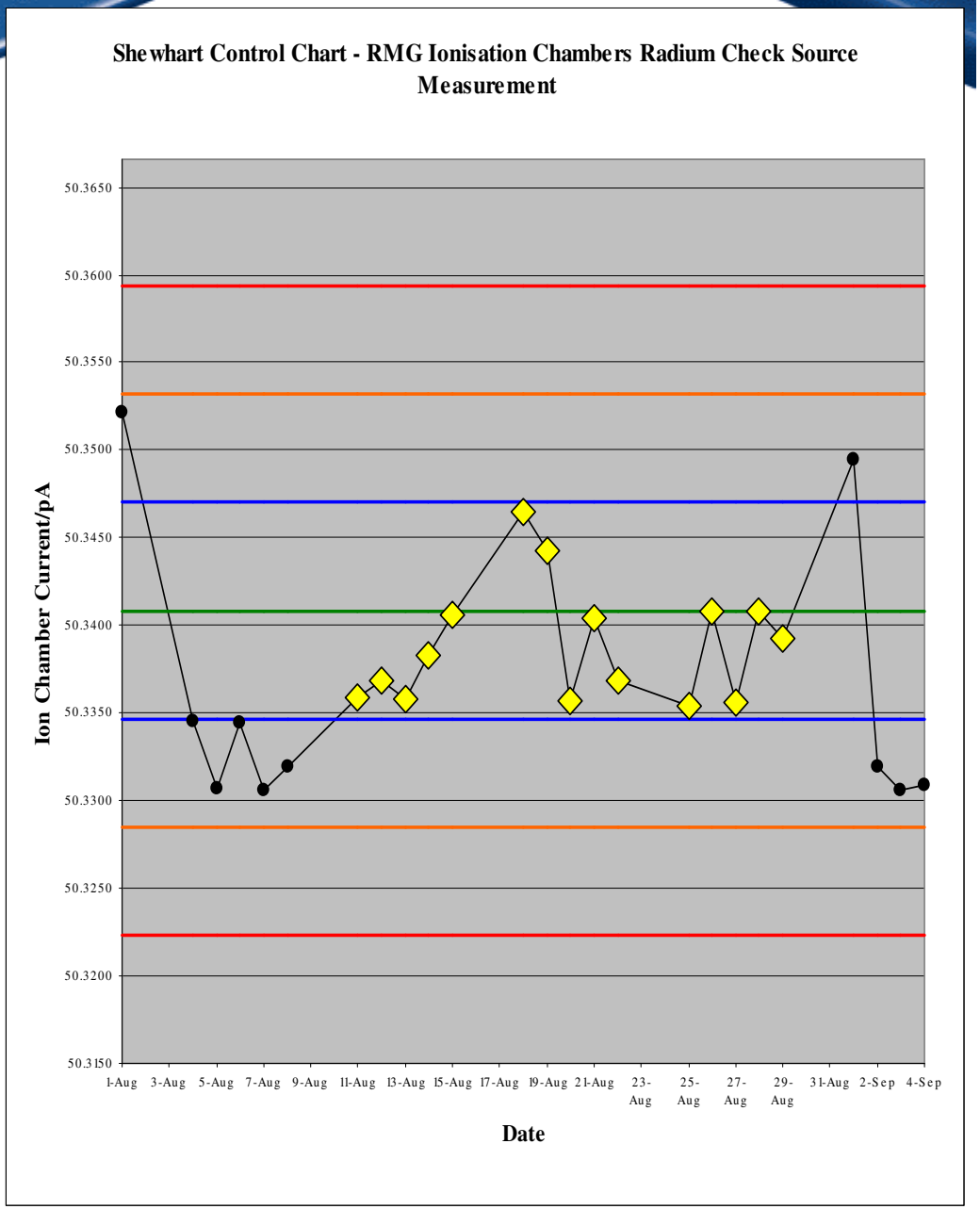
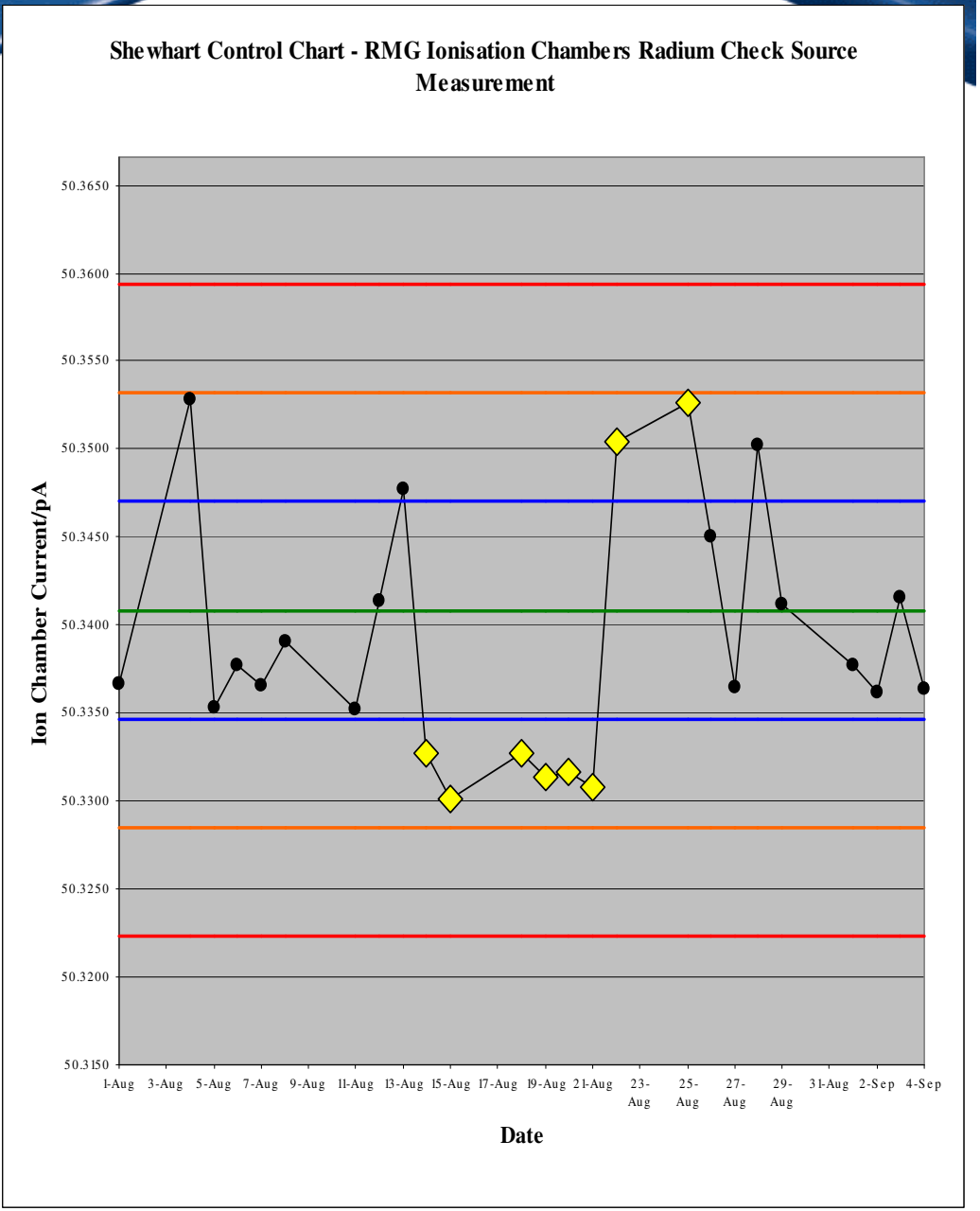
- Fifteen points in a row between the lower and upper information levels. This may arise after the procedure has been operated for some time and may require the limits to be re-set



- Eight points in a row either side of the mean with none between the upper and lower information levels. This may indicate a mean shift.



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In practice.

- Use software to record and present the data
- Require operators to manually carry out a check (use a paper-based check list) for the above conditions
- Carry out investigations as necessary and institute improvement actions

However, it is incorrect simply to:

- Repeat a measurement and find the chart to be ‘in control’
- Reset the chart parameters without at least seeking a reason
- Rely on the software to do it for you
- Rely on manufacturer’s software

In addition:

- Use a CuSum Chart:

$$c_1 = x_1 - X$$
$$c_2 = c_1 + x_2 - X$$
$$c_3 = c_2 + x_3 - X$$
$$\vdots$$
$$c_i = c_{i-1} + x_i - X$$

- Deviation from control become clear very rapidly

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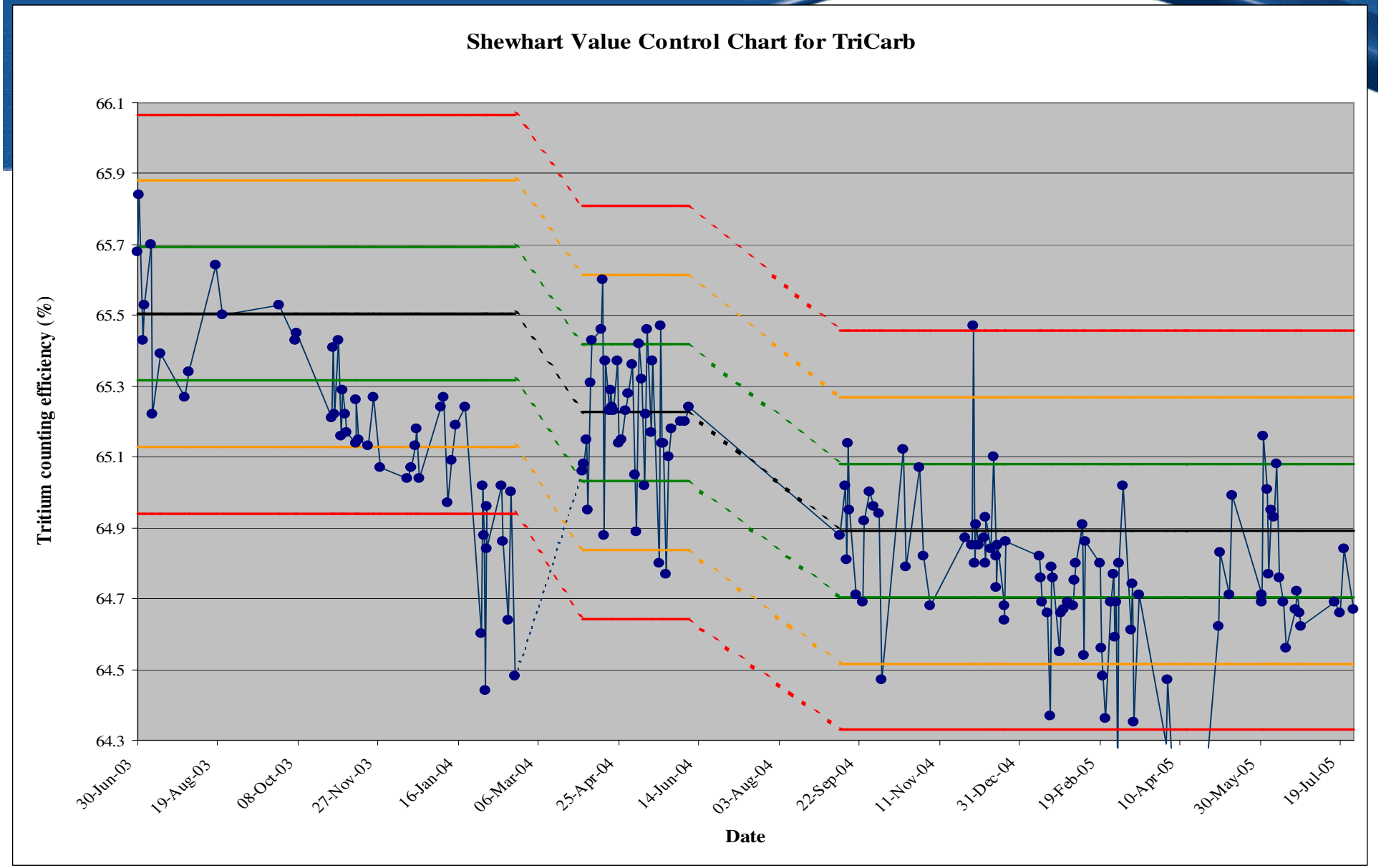

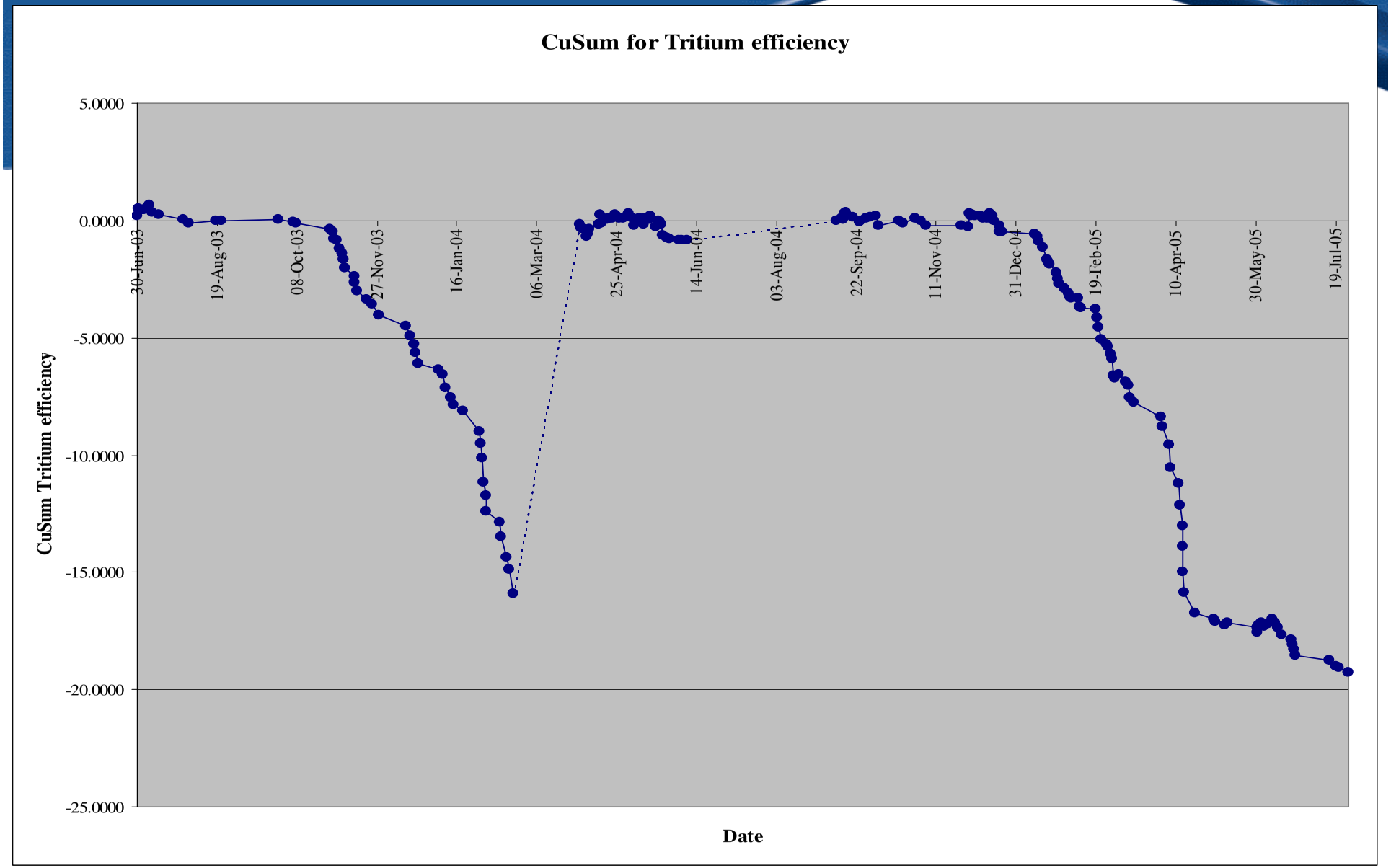
CuSum for Tritium efficiency
5.0000
0.0000
-5.0000
-10.0000
-15.0000
-20.0000
-25.0000
CuSum Tritium efficiency
30-Jun-03
19-Aug-03
08-Oct-03
27-Nov-03
16-Jan-04
06-Mar-04
25-Apr-04
14-Jun-04
03-Aug-04
22-Sep-04
11-Nov-04
31-Dec-04
19-Feb-05
10-Apr-05
30-May-05
19-Jul-05
Date

Tritium Check Source for LSC

- Shewhart Chart gives first warning on 6th November – 9 points in a row, one side of the mean – (and many thereafter)
- CuSum Chart gives first warning around 1st November – problem is clear from this point on
- On-board Q/C software gave first fault message on 30th January – '3σ' limit violation

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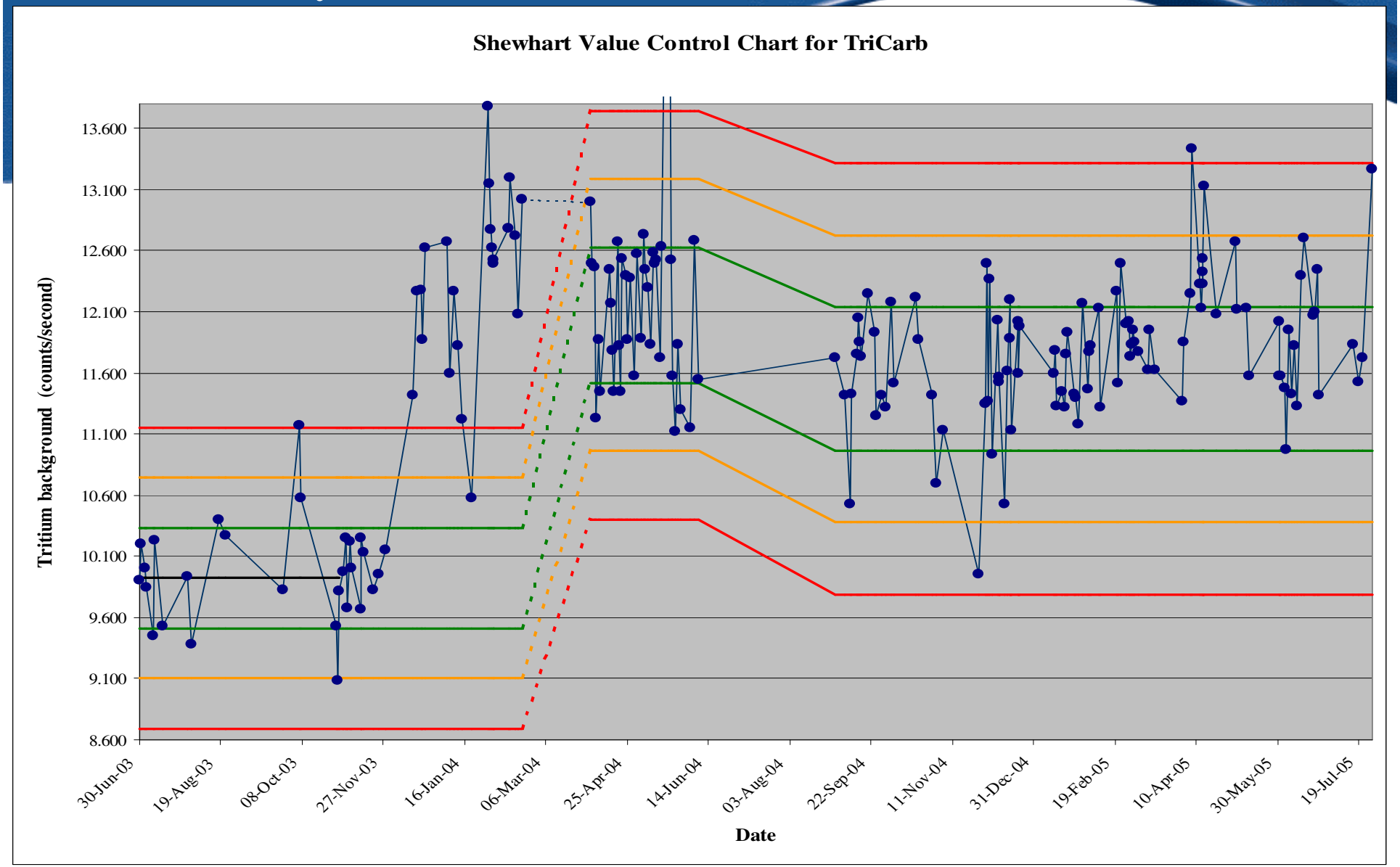

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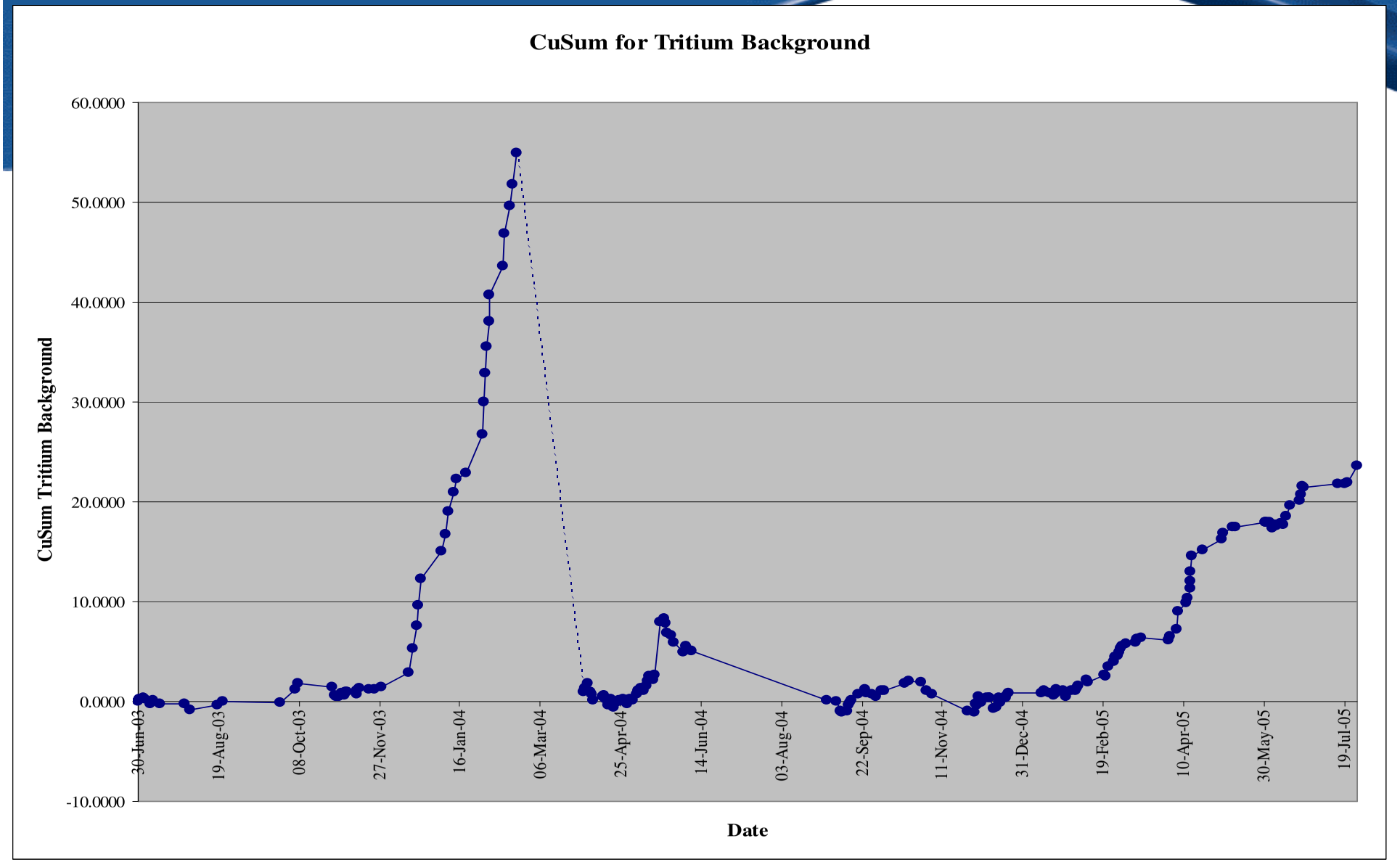

Tritium Background for LSC

- Shewhart Chart gives first warning on 15th December – action limit violation – (event on 8th October assigned to $^{99}Mo/^{99m}Tc$ source in vicinity)
- CuSum Chart gives first warning around 18th December – problem is clear from this point on (event on 8th October does not affect CuSum)
- On-board Q/C software gave first fault message on 15th February – '3σ' limit violation

Analysis of data

- Engineer called out in February
- Deteriorating Photo-Multiplier tube identified
- ^{14}C check source did not return comparable data, apart from on action limit violation that did not have an assignable cause
- Subsequent measurements with other systems indicate general instability of ^{3}H measurements – observed at other establishments
- Further investigations reveal 'hidden' parameters that low energy β-emitters are very sensitive to (such as DBB)
- Essential to know the system well before use

Conclusions

- Full implementation of ISO 8258:1991 allows operators to far better understand measurement systems
- Potential faults can be trapped by preventative actions rather than corrective actions
- Simple to use and interpret
- Applicable to similar systems – α-spectrometers, Germanium γ-spectrometers, NaI(Tl) γ-spectrometers, ionisation chambers &c
- Customer calibrations and source generation can be carried out with greater confidence

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Questions and Feedback