

Abnormal Calibration Parameters

1 Introduction

The Calibration Parameters are central to the extensive list of diagnostics that are built in to the 8241 Silica Monitor. These parameters comprise the Zero Offset and the Calibration Factor, which are updated during each calibration. These are intended to assess the performance of the monitor, and if the performance is less than predetermined limits, i.e. large zero drift, poor or no response to silica, an Out of Service alarm is generated.

It would appear that this alarm is frequently misunderstood and therefore the inappropriate action is taken. This Technical Guide is intended to clarify the function of the calibration parameters and provide guidance as to typical causes of common problems.

2 Zero Offset

The Zero Offset parameter (displayed in $\mu\text{g l}^{-1}$) is intended to display the zero drift in the monitor between reagent replacements every 5 weeks. Each time the reagents are replaced (it is important that all four reagents are replaced at the same time) a Baseline Zero is carried out, which resets the Zero Offset to $0.0\mu\text{g l}^{-1}$. This type of calibration is intended to establish and compensate for, the background silica in the first two reagents. As each timed calibration is carried out, known as a Routine Zero, the accumulative zero drift is then displayed as the Zero Offset. The limit on the acceptable Zero Offset is $< \pm 100\mu\text{g l}^{-1}$, which is very generous, so values higher than this would indicate a very large degree of drift.

It should be remembered that a large Zero Offset value indicates an excessive drift between the last Baseline Zero and the last Routine Zero. It could be as a result of a problem that has recently occurred OR that there was a problem during the last Baseline Calibration and the monitor is in fact now operating normally. The Baseline Calibration may not have been carried out at the correct time, if at all, by some users, so the Zero Offset becomes meaningless. If a Baseline Calibration is not carried out when the solutions are replaced, a large Zero Offset could simply be caused by the different levels of background silica in the first two reagents.

3 Calibration Factor

During the chemical reaction within the monitor, all the silica is converted into a silica compound, which is blue, the optical system measures the absorbance of this coloured solution. A calibration factor of 1.0, means that the absorbance of the reacted coloured solution in the measurement cuvette is 100% of the expected value. Again limits are predetermined in the software for an acceptable calibration of $< \pm 0.2$, i.e. between 80 and 120% of the expected absorbance of the solution.

3.1 High calibration factors.

If the monitor appears to be performing normally in every other respect, the only possible cause is the calibration solution. It is simply not possible to have more blue silica compound, than there is silica in the sample in the first place. If the calibration factor is 1.9 or 6.2 then silica in the standards was 1.9 and 6.2 (respectively) times more silica in the solution than was expected. If it is suspected that the water used to make up the standard solution is high in silica, it is recommended that a much higher silica solution is prepared, so error is minimised.

3.2 Low calibration factors.

This could be either the standard solution of any one of the reagents.

3.3 Zero or very low calibration factors.

This simply means that no chemical reaction is taking place (i.e. there is no conversion from the silica in the solution into the blue coloured compound) between the silica in the sample and the reagents.

4 Possible Causes

In the event of difficulties, it is always important that the following points are checked first:

1. The reagent solutions and the sample are being pumped through the system.
2. The reagent solutions are plumbed in the correct order.
3. The solenoid valves are operating correctly, that also means they are not leaking on the closed port.
4. The drain fill sequence is OK, check that the cuvette overflows before the lamp is illuminated.
5. The pinch valve is operating and draining the cuvette each cycle.
6. The monitor responds to changes in silica, is stable on a bottle of solution.
7. The correct reagent solutions are used as per the manual, no changes must be made in any way and reagents from other monitors will be absolutely useless.

The solutions, irrespective of reassurances that are given, cause a very large percentage of problems on silica monitors out in the field. Time and time again, it is reported to us that the solutions cannot be the cause of the problem and yet time and time again they are proved wrong.

To repeat again a piece of text in the instruction manual:

In the majority of cases any problems experienced are generally found to be associated with the chemistry and liquid handling section.

The most common problems are associated with the reagent or standard solution. Any unpredictable problems may be due to the standard or reagent solutions, or their flow through the monitor. If any doubts exist regarding the integrity of these solutions, they should be replaced with freshly prepared solutions in the early stages of the faultfinding investigations.

If the monitor fails to produce results as expected, the most likely cause is the standards, either contaminated when handled or (and most likely) made up with poor quality water, possibly containing high background levels of silica and/or phosphate. Incorrectly prepared reagents may give a poor calibration factor. If the solutions are purchased from a proprietary chemical supplier, care should be taken in storing the containers; they should be date stamped, used in strict rotation, and not used after their expiry date. When measuring trace levels of silica, contamination can only be avoided by preparing and handling solutions with great care. Laboratory glassware is not to be used for low concentration solutions; use plastic instead.

5 Possible Electronic and Optical Faults

It is stressed in the instruction manual, that electronic or optical faults are **VERY** rare and when they do occur, are reasonably obvious. A procedure to carry out a Simple Electronic Response Test is given in the Instruction Manual (in Section 8.5.4) so it is not worth repeating here. This provides a full performance test of the Lamp, Photocells, Cuvette Board, and the A to D Conversion, and the Micro-processor Boards, working together as a complete system.

The electrical calibration is **VERY** unlikely to have changed, and we do not advise that you attempt a re-calibration, it is factory set, and routine re-calibration is not required. The electrical calibration procedure is only intended to calibrate the A to D converter chip, which is then self compensating for zero and span drift. The photocell input circuit on the Cuvette Board has a very low gain, and is not subject to electrical calibration.

The lamp is operating at 50% of its normal working voltage, so the life will be far greater than 10 years, so is expected to be in excess of the life of the monitor. The lamp brightness is electronically controlled, so will automatically be compensated for any effect of lamp aging and mains supply variations. Please do not remove the lamp because the alignment will need to be set-up again, although this is described in Section 8.6.2 of the manual. It may be worth checking the lamp alignment but only if it is suspected to have been disturbed.

The signals from two photocells are compared by a ratio algorithm in the software, so again any aging effect is automatically compensated. The life of the photocells is again expected to be in excess of the life of the monitor.

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