CS-APC-2 / 3 / 22M

Bulletin No.: CS-APC-SUPPORT-ZERO-COUNTS

Subject: Adjusting Solvent Verification Settings to Eliminate Negative Counts

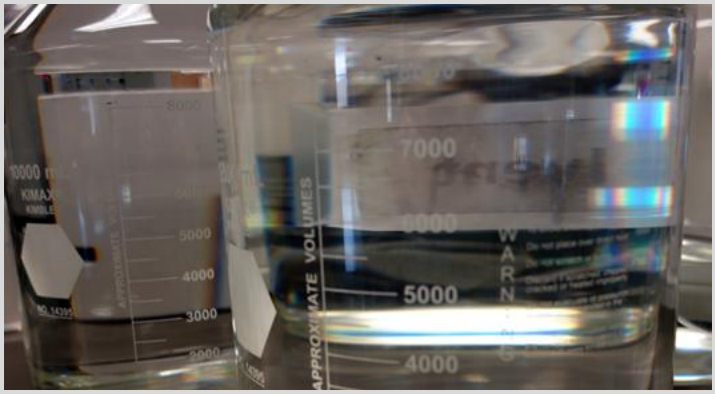
Background
You are experiencing unusually low or even zero 4µm particle counts (see the example below);

>4 = 0
>6 = 3540
>10 = 202
>14 = 45.5
>21 = 6.7
>38 = 0.15
>70 = 0

Issue
When solvent verification limits are much higher than the actual cleanliness of the solvent, the system will accept count data that is within the high limits but not representative of the actual cleanliness of the solvent. When this happens the reported counts on all samples will be lower than they should be because the corrections applied for the solvent background counts will be too high. This is not as issue for samples with high particle counts (i.e. > 15/13/11) as the error is minimal and insignificant, but this is not the case with very clean samples (i.e. < 14/12/10).

The solvent verification limit parameters (Micron setting parameters) should be set based on the cleanest samples expected to be analyzed on the system and the highest dilution ratio used to dilute the samples. The parameters in the particle counter must be set to ensure the method requirement for the solvent not contributing more than 12.5% of the counts in the diluted sample is met on all samples.

Guidelines on Solvent Cleanliness
The cleanliness of your solvent can have a significant impact on your sample particle count results if you are using very high dilution ratios (i.e. 5:1 or higher) and/or testing samples with very low particle counts (i.e. ISO 4406 <14/12/10). Ideally when you make up a batch of solvent allow the solvent to stand for 24 hours prior to use. This ensures that the vast majority of the particles have settled out of the solvent, and this is usually sufficient for most testing requirements.



If you find that you are still experiencing issues with low micron counts, then you can alternatively filter the solvent prior to use. We recommend you use an in-line filter (see Figure 1).

Figure 1 – *A typical low cost in-line liquid filter (courtesy Sigma-Aldrich)*

Setting Solvent Verification Limits

Solvent verification is the process in which the counts/ml of the particle sizes of interest in the system diluent are determined. It is an especially important process as the counts/ml determined during this process are used when correcting the count data measured on the diluted sample to represent the counts that would be expected to be measured if the sample had been analyzed undiluted.

Any error in diluent counts will obviously affect the accuracy of the final count data. The accuracy is also dependent on the sample dilution ratio since the smaller the “Undiluted” sample becomes the larger the correction for dilution and diluent background counts becomes.

To obtain decent quality results for any sample that is suitable to be processed on the auto diluting particle counter it is particularly important to establish valid solvent verification limits that are used by the software to determine if the solvent is suitable for use on the system. These limits must be set to values that ensure the diluent complies with the cleanliness requirements specified in the ASTM D7647 test procedure.

The following procedure is recommended when setting the solvent verification limits on the CINRG auto diluting particle counters.

1. Decide on the ISO Code of the cleanest sample that is expected to be processed on the CINRG particle counter. In the example that follows a code of 15/13/11 is selected.
2. Decide what the highest dilution ratio is likely to be when diluting samples (1:1,1:2 or 1:3) wand then use table 2 to determine the limits needed to be applied to meet the ASTM D7647 diluent cleanliness requirements i.e. Diluent must not contribute more 12.5% of the counts in the diluted sample.
3. Enter the limits determined from table 2 as the solvent verification micron settings parameter in the system software.
4. If the ISO code selected was 15/13/11 and sample are to be diluted on a 1:1 basis the solvent verification limits would be set in the software as shown below.

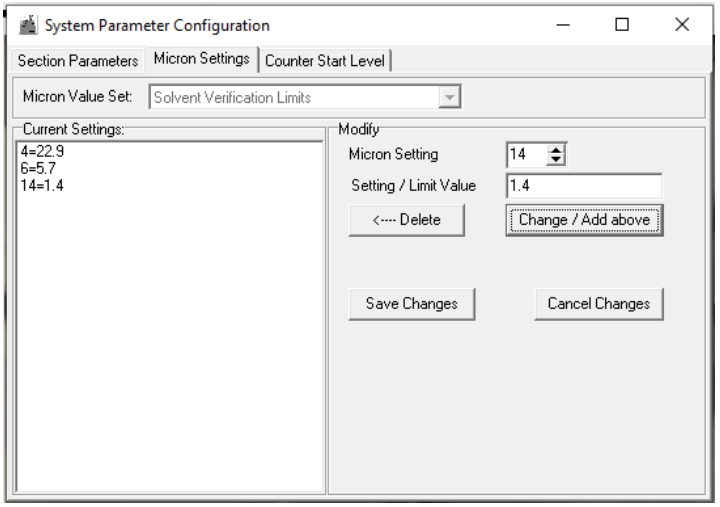


Figure 2 – *Micron Settings tab of the the System Parameter Configuration for adjusting the solvent verification limits.*

Table 1 – *ISO 4406 Cleanliness Code & count ranges.*

ISO 4406 CODE #	MORE THAN	UP TO AND INCUDING
24	80000	160000
23	40000	80000
22	20000	40000
21	10000	20000
20	5000	10000
19	2500	5000
18	1300	2500
17	640	1300
16	320	640
15	160	320
14	80	160
13	40	80
12	20	40
11	10	20
10	5	10

Table 2 – *Max solvent counts based on dilution ratio.*

Max Solvent Count 1:1 Dil	Max Solvent Count 1:2 Dil	Max Solvent Count 1:3 Dil
11429	5714	3810
5714	2857	1905
2857	1429	952
1429	714	476
714	357	238
357	179	119
185.7	92.9	62
91.4	45.7	30
45.7	22.9	15
22.9	11.4	7.6
11.4	5.7	4
5.7	2.9	1.9
2.9	1.4	1
1.4	0.7	0.5
0.7	0.4	0.2

Setting Solvent Verification Limits (continued)

5. After setting the solvent verification limits go to the "Section parameters" for solvent verification and set the "Max_Invalid_Results" and "Required_Valid_Resilts" parameters to their maximum settings. These values will be 6 and 12, respectively.
6. After setting and saving these parameters run a solvent verification to see if the diluent is clean enough to meet the solvent verification limits set.
7. If the diluent cannot meet the limits it will need to be filtered through a suitable filtration system and retested.
8. When the solvent meets the solvent verification requirements, examine the data to determine if the average values are representative.
9. If there are early results included in the data set from which the average is determined that are significantly higher than the average, then the average is not representative, and the solvent verification should be repeated to get a more representative average.
10. If the average count results are significantly below the solvent verification limit, the solvent verification limits should be reset manually in the Micron settings system parameter to reflect the cleanliness of the solvent.
11. When the solvent verification limits have been set up correctly. The solvent verification section parameter for required valid results can be set to 9.

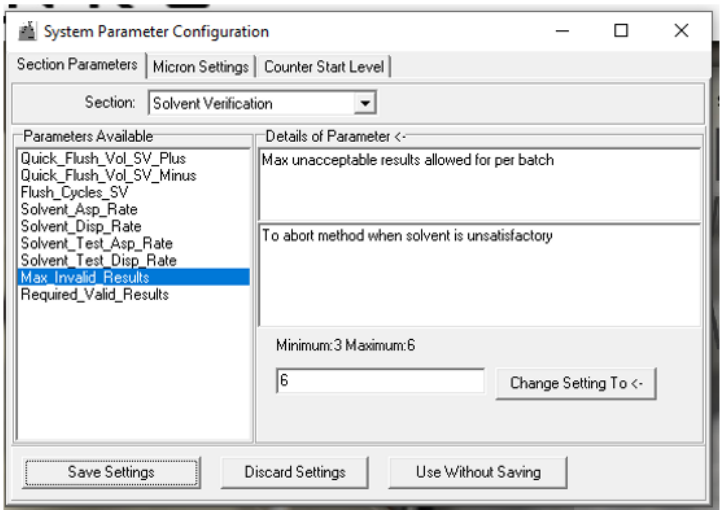


Figure 3 – *Setting the Max_Invalid_Results and Required_Valid_Results settings for Solvent Verification process.*

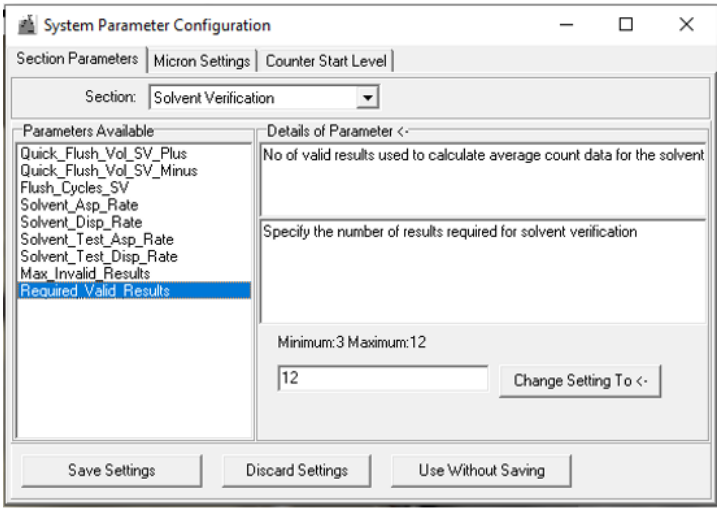


Figure 4 – *Setting the Required_Valid_Results setting for the Solvent Verification process.*