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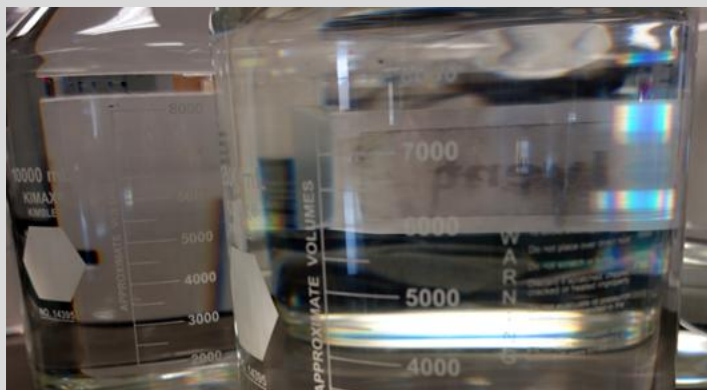
If you find that you are still experiencing issues with low micron counts, then you can alternatively filter the solvent prior to use. We recommend you use an in-line filter (see Figure 1).


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

Adjusting the Solvent Verification Behaviour & Limit Settings

If you are routinely testing samples that have ISO 4406 cleanliness levels below 14/12/10 it is suggested that you adjust the Solvent Verification Limits in the APC instrument software. To do so;

1. Launch the instrument software in admin mode.
2. Click the [Edit Parameters] button from the admin menu.
3. From the Micron Settings tab select the Solvent Verification Limits (see Figure 2).
4. Set the 4, 6 and 14µm settings to the following values; 4µm : 10, 6µm: 2, 14µm: 1
5. Click Save Settings and close the System Parameter Configuration dialog.

Figure 2 – *The Micron Settings tab of the Edit Parameters dialog showing the Solvent Verification Limits settings.*
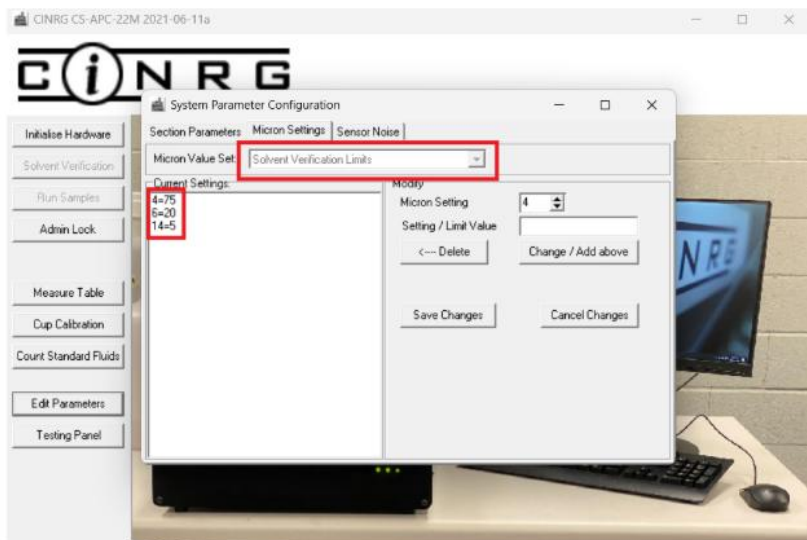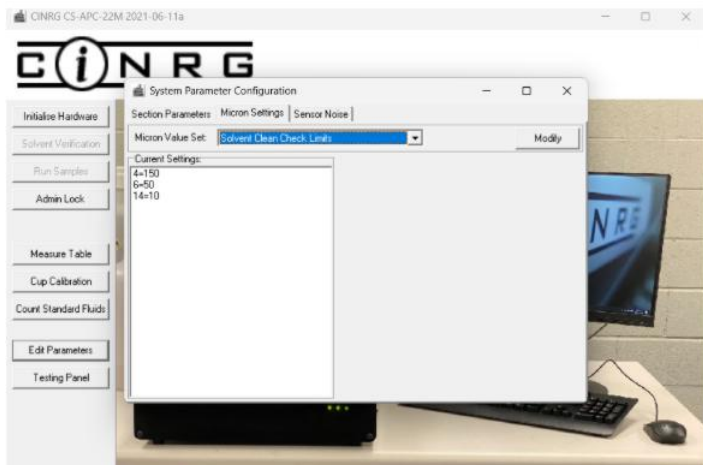
In the same menu, you'll also find "Solvent Clean Check Limits." These are used during auto-cleaning cycles, triggered when the >4 µm count from a sample exceeds the Solvent_Check_Threshold value. When that happens, the system performs additional cleaning and checks until the solvent passes the set limits (see Figure 3).

Figure 3 – *The Solvent Clean Check Limits on the Micron Settings tab of the Edit Parameters dialog.*

In the Section Parameters | Cleaning there are several parameters to initiate additional cleaning cycles when a 4µm count is exceeded (see Figure 4). This can also be combined with a solvent check (Solvent_Check_Threshold), that will initiate a cycle of performing extra cleans for dirty samples, and a verification of the solvent counts prior to proceeding to test the next sample. The Max_Solvent_Checks will allow you to indicate how many times the cleaning and solvent verification takes places if the solvent check fails. See Section 7 – System Parameters of your APC instrument user manual for details on these settings.

Figure 4 – *The Section Parameters tab of the Edit Parameters dialog showing the Cleaning settings.*