

Swab Method for TC – Determination of polluted surfaces

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1. Preface

In some technical application fields – especially in electronics, in

Food Industry, in precision engineering, in pharmacy, medicine, space technology and semiconductor production – special demands are made on the purity of the production area (surfaces, machines, tools) and the product itself.

Surface cleanliness, according to the guideline for pollution-free-room-technology VDI 2083, is defined as the absence of pollution of all kind, which could influence the process or the product respectively.

The target of all pollution-free-technological measurements is the achievement of a permissible, i.e., the process not disturbing residual pollution on workstation surfaces. As there is no covered sampling method known to us, to determine the TC-pollution on products or factory surfaces - developing an appropriate method has been determined to be necessary. In the following sections the extraction of swab samples is described in detail.

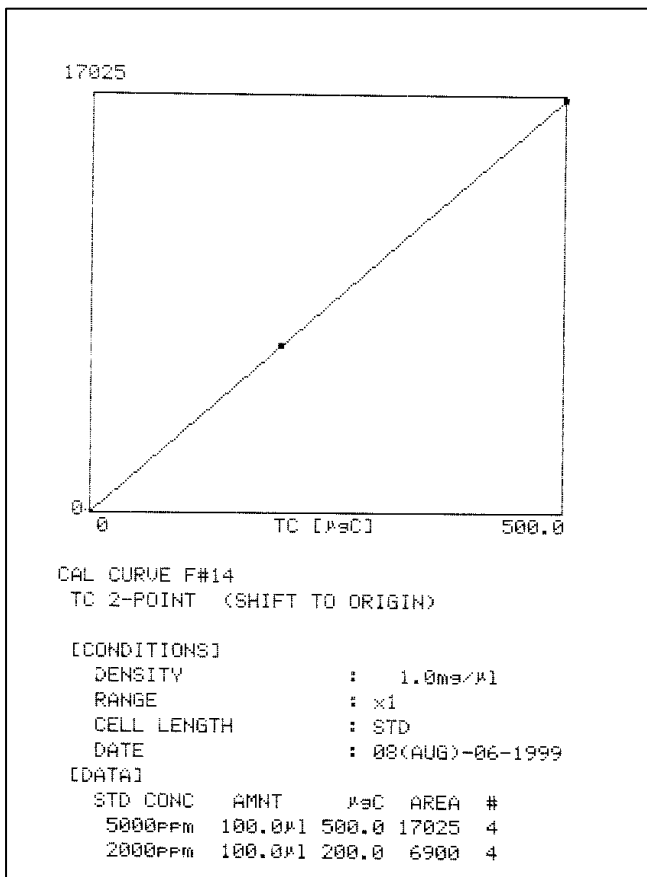


Fig. 1: Two-point-TC-Calibration curve with a 2000 ppm C- or 5000 ppm C-Glucose solution.

Instrumentation:

Shimadzu Unit: TOC – 5000 A with SSM – 5000 A
Carrier Gas: Synthetic Air (or Oxygen)
TC-furnace temperature: 900 °C
short measurement cell.

2. Cleaning of relevant surfaces

In order to determine the rest contamination, according to the below mentioned cleaning method, glass tops have been chosen, since those show no evaporation of organic substances. For the cleaning process, powder free gloves were used, made from natural rubber latex, rinsed carefully with fully deionized water (Conductivity < 1 µS/cm /VE WATER) furthermore cellulose which was exhausted with pure water (VE-WATER). The glass tops were carefully cleaned with 3-4 pieces towelling paper, before swab samples were taken with a micro filter of quartz fibre. These filter papers have a diameter of 37 mm, consisting of very pure quartz, which was pre-heated in order to remove traces of organic pollution and

which is heat resistant up to 950°C. Preferentially, the filters shall be handled only with heated tweezers and powder free gloves. The investigation of 10 filter papers revealed an average TC-baseline-pollution of 15µg/filter. This rest contamination was subtracted from all other results. If necessary, the quartz fibre filters, might be heated up together with the ceramic combustion boats, leaving a slight remaining TC-pollution which cannot be determined. Even after this pre-heating the papers are still supple and suitable for swabbing. With the swab method, described in section 4, pollution values up to 0,1 µg TC/cm² have been found.

3. Storage of the swabs

For the TC-value does not increase within the factory-new pre-heated filters, they are supposed to be stored in sealed glass containers. Plastic containers are not suitable, because, evaporating organic components (softeners etc.) can cause

contamination. Moreover, only empty and clean glass containers should be taken to the place of sampling., where all filters from the sample surface should immediately be put in one of these glass containers.

4. Swabbing method

Swab samples are real surface samples. For sampling, new, powder free gloves (made of natural rubber latex or pure-room-gloves, which are rinsed with VE water) should be worn. Here the gloves of natural rubber-latex, described in section 2 were used. In any case, each touch of the sampling surface with the hands, has to be avoided.

Implementation of Sampling:

For one sample three swabs were used.

- | | |
|------------|---|
| Swab No. 1 | was moistened with 0,5 ml VE water and then wiped off the whole glass top with it.
Swab put in a glass container. |
| Swab No. 2 | was moistened with 0,5 ml VE water and then wiped off again the whole glass top with it.
Swab put in a glass container |
| Swab No. 3 | was used for wiping dry the whole glass top
swab put in a glass container . |

Close glass container tight and label it accordingly (numbering); note also surface, appearance, kind of wiped off sample-surface.

Just so much sampling-material (swab) should be used on the surfaces until it looks optically clean. It is important, to use a dry swab at the end, in order to soak up remaining humidity from the sampling-surface.

5. Measurement Method

The quartz fibre filters which were used for the swabbing were rolled and then separately put into a pre-heated ceramic boat. Afterwards the catalytic oxidation was done in oxygen atmosphere at 900°C. The emergence of CO₂-gas during oxidation is

detected by NDIR method. The TC -concentration, which is proportional to the peak area is calculated using the calibration curve.

6. Calibration

Depending on the CO₂-concentration of the samples, three different measurement ranges can be chosen: Range 1 (most sensitive measurement range), Range 5 and Range 30 (highest

measurement range). The instrument itself chooses the measurement range automatically. TC calibration is done using two glucose-standard-solutions.

Fig.1: shows a two-point-TC-calibration curve with a 2000 ppm C- and. 5000 ppm C- glucose-solution. The measurements were done in Range 1, since the TC-value of the samples is expected within this area.

7. Measurements and Results

7.1 Samples of defined contaminated surfaces

To prove the swab method (described in section 4) several glass tops of a 980 cm² area were cleaned by the method described in section 2. Afterwards these glass tops were contaminated moreover with a standard solution of 100 µl each of a 5000 ppm C – Glucose- or potassium hydrogen phthalat standard solution. The solution used for contamination was equally spread over the glass tops with a pipette's point and then dried at room temperature. The swabbing was done with three quartz fiber. The use of a fourth swab was proved to be unnecessary because TC – measurements showed that, no further contamination

can be absorbed. The entire applied contamination was able to be captured with three swabs in these experiments. If it is a persistent contamination we are dealing with, then, more than three swabs are necessary, in order to achieve an optimal cleaning of the surfaces. Furthermore, with slighter pollution, sampling surfaces of approx. 1000 cm² or bigger are being preferred. Also, measuring the correct surface area is very important in order to obtain correct experimental values.

The measurement results are shown in table 1.

The theoretical area pollution with TC- after the defined contamination method - was calculated to be 0,51 µg/cm² TC.

Table 1:

No.	Contamination on glass	Absolute pollution with TC [µg]			Sum pollution with TC [µg]	Area [cm ²]	Measured Area pollution with TC [µg/cm ²]
		sw 1	sw 2	sw 3			
1.	500 µg Glucose	443,4	82,1	24,1	549,5	980	0,56
2.	500 µg Glucose	422,7	84,5	0,0	507,2	980	0,52
3.	500 µg Glucose	460,2	95,6	3,1	558,9	980	0,57
4.	500 µg Glucose	453,3	104,3	5,1	562,7	980	0,57
5.	500 µg PHP	447,2	85,2	3,7	536,1	980	0,55
6.	500 µg PHP	433,0	97,9	5,6	536,5	980	0,55

Short cuts: sw = Swab ; PHP = potassium hydrogen phthalate

7.2 Samples of real surface contamination

In order to get an idea of surface determination in offices, samples of vertical glass areas and horizontal plastic surfaces (desk) have been taken. The glass areas have been treated by

the normal dust-pollution for approx. one year and the desk-surfaces for about. 2 month.

The measurement-results are shown in chart 2.

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Chart 2:

No.	Surface	Absolute pollution with TC [µg]			Sum pollution with TC [µg]	Area [cm ²]	Measured area pollution with TC [µg/cm ²]
		sw 1	sw 2	sw 3			
1.	Glass (vertical)	273,1	134,4	53,1	460,6	980	0,47
2.	Glass (horizontal)	433,2	122,6	37,8	593,6	980	0,61
3.	Desk Plastic (horizontal)	591,9	276,4	159,2	1027,5	270	3,81
4.	Desk Plastic (horizontal)	712,1	190,7	72,4	975,2	270	3,61

Short cut: sw = Swab

Discussion

As shown in table 1, the measured average area pollution deviates from the theoretically expected pollution of 0,51 µg/cm² TC, with 8,49% . For the measured pollution is higher than the applied pollution, it can be said that the entire contamination of the analysed surfaces was captured. At the same time, it has to be mentioned, that the detection limit of the used instrument is at 100 µg TC. The experiments ACC. to table 1 show values from 500 – 565 µg TC. Taking into account that the measurement concentration level was at the determination limit an analysis error of < 10% is tolerable. Therefore the swab method, which is described in section 4, can be used also with similar surface concentrations of water-soluble organic contamination higher than 0,1 µg/cm².

The fact, that the deviation from the defined surface pollution was traced only in the positive direction, it might be

assumed, that in spite of careful sampling, certain contamination have been imported. These contamination can be put down to the use of deionized water (TC up to 2 ppm), to the dust-import from the atmosphere and to possible existing contamination from the gloves. If these guidelines can be improved, more specific result-data could be obtained. ACC. to chart 2, the plastic-surfaces (desk) show a 7 times higher contamination than the glass-surfaces, despite the desk-surfaces had to withstand the normal dust-pollution for a shorter time. The reason for this, is the position of the sampled surface and the type of the surface (plastic, glass) - Information , which have to be registered during sampling, in order to be able interpreting the results properly. On a further refining of the here introduced sampling- and -examination-method it is being worked currently.

Literature:

VDI 2083, Guideline for pure-room-technology (Surface purity), Page 4, February 1996



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