Analysis of airborne particles

and deposits in production processes

Elemental analysis can help to identify the source of contamination in cleanrooms. Often the contamination consists of airborne particles or deposits in production equipment.This makes sampling a challenge when the production processes cannot be disturbed. After sampling, several analytical techniques can be used for characterization of the material found.The most common approach is acid digestion, followed by quantitative analysis using Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). Laser Ablation (LA) however forms a good alternative for wet-chemical analysis of solids as it solves many of the problems associated with the conventional acid-digestion method.

Philips Innovation labs

PHILIPS

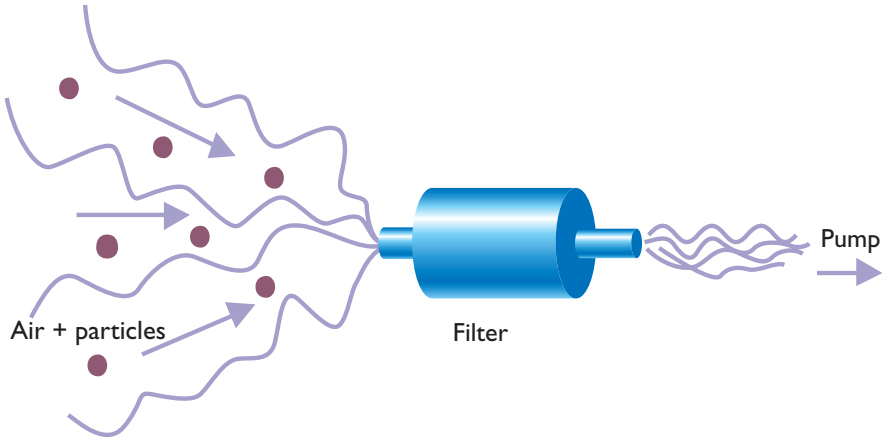


Fig. 1: Principle of air sampling.

Micro-analysis

Contamination problems occurring in a production or development process can only be solved if the source of the problem is well known. Small amounts of material are normally present, which are difficult to sample. In this note two different sampling techniques will be discussed. The first technique is sampling airborne particles using micro-pore filters. The second technique is sampling small amounts of material using a cotton bud. Both techniques sample only a very small amount (< 1 µg) of material. This material is then analyzed using Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS).

LA-ICP-MS

LA-ICP-MS is a micro analytical technique for the determination of main and trace elements (wt%-µg/g) in solid materials. A pulsed laser beam is used to sample a small quantity of material by a physical process (ablation). This process generates very fine solid particles (< 0.5 µm) leaving behind a tiny ablation crater (10-250 µm in diameter). The sampled material is transported by a flow of argon gas into an argon plasma (ICP) for ionization. A mass spectrometer is used for separation and detection of the elements present.

The technique requires no sample preparation. Almost all elements in the periodic table (Li-U) can be detected at concentration levels ranging from sub-µg/g to wt%-level in one analysis. This makes LA-ICP-MS very suitable for the elemental analysis of totally unknown samples, even if very little material is present.

Sampling with micro-pore filters

The analysis of airborne particles in a working area is important for a number of reasons. Such particles can contaminate products during production and the identification of their source is essential to eliminate problems. Particles in exhaust systems are also of interest as their analysis can help to quantify the total amount of material that is brought into the environment as a result of work inside a plant.

Trapping

Particles have to be trapped first. To do so, a known amount of air is sampled using a micro-pore filter. Previous study showed that especially cellulose acetate filters contain very low levels of inorganic contaminants. Air is pumped through the filter using a standard air-sampling pump (fig. 1). Particles deposit on the filter, after which the filter (fig. 2) can be analyzed.

LA-ICP-MS analysis

Laser ablation forms a welcome alternative for the normally used wet chemical analysis procedure where the total filter is digested before analysis. It is less time-consuming, there is no loss of trace elements and contamination due to sample pre-treatment is avoided. Filters used for sampling are immediately transported to the sample chamber and directly analyzed. In the sample chamber an argon flow creates a miniature cleanroom, which avoids unwanted contamination. The filter surface is sampled (fig. 3) by scanning a high-energy laser beam across it and the removed (= ablated) material is transported to the ICP-MS for detection. During ablation the signals of the elements of interest are followed. In figure 4 the relative intensity of several elements present in the filter is shown against the ablation time. It can be seen that the filter is contaminated with As, Cd, Pb,

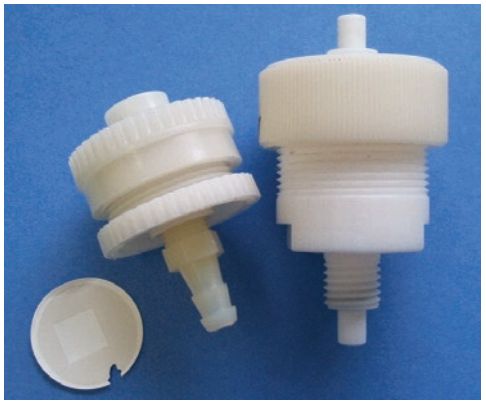


Fig. 2: Filter and holders used for trapping the particles.

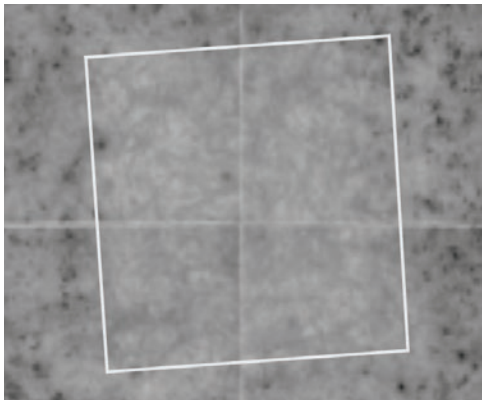


*Fig. 3: Filter after ablation of a 400 * 400 µm area.*

Main component	Detection limit (ng/filter)
Ca	200
Ni	0.25
Zn	0.9
As	2
Cd	0.1
Pb	0.35

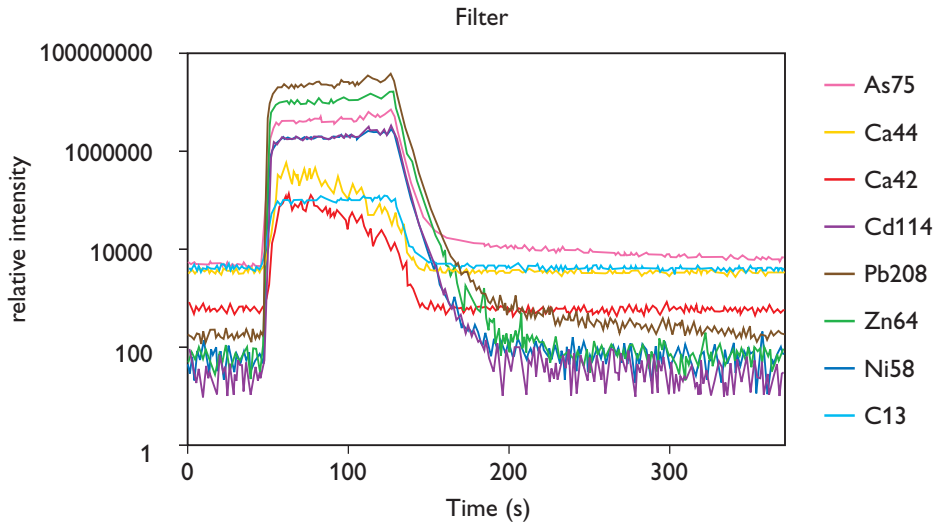
Table 1: Detection limits for some elements when sampled with filters and analyzed with LA-ICP-MS.

Zn and Ni. For this particular analysis, the background signals on ^{42}Ca en ^{44}Ca are not stable. The signal for ^{13}C is stable and forms a reliable internal standard for the analysis.

A good calibration is critical for accurate and precise analysis. Calibration is performed using a standard filter prepared by dripping an aqueous standard solution on the filter. The real samples consist of particles on filters instead of material impregnated in filters. Therefore only semi-quantitative analysis is possible with a relative accuracy of about 10%. Since ICP-MS is a very sensitive detection technique, analysis is possible at levels that are of practical use (ng/filter) (Table 1).

Fig. 5: Sampling material using a cotton bud.

Fig. 4: LA-ICP-MS signal for several elements followed during ablation of a sampling filter.



Sampling using cotton buds

If a deposit is formed in a production process, it is often formed in inaccessible places. Because the deposits are present in very small amounts, it is difficult to sample them properly. Samples are easily contaminated, which hinders accurate elemental analysis.

Using a cotton bud for sampling (fig. 5) in combination with LA-ICP-MS detection, most problems can be avoided. Previous research showed that several brands of cotton buds are very clean and contain almost no inorganic contaminations. The long stick of the cotton bud makes it easier to physically sample the deposit. LA-ICP-MS makes it possible to analyze these very small amounts of totally unknown material.

Trapping

When a problem occurs at a production site and deposits are formed, the cotton buds are sent in special containers to the site. The dirty surfaces are sampled by touching them with the cotton buds. The samples are then returned in the same special containers to avoid contamination.

LA-ICP-MS analysis

In the lab the cotton buds are analyzed using LA-ICP-MS. The elements detected as well as the ratios of these elements can help identify the source of the problem.

Most elements in the cotton are present at very low concentrations (sub-ppm). When a cotton bud with sampled material is ablated and compared with a “blank” one, the elemental composition of

the sampled material can be deduced. Because no exact standards are available, the concentrations are determined in µg/g in relation to the amount of C (cotton). Using this semi-quantitative method, the ratios of the different elements can be determined quantitatively.

The example shown in table 2 contains data for a typical analysis. In combination with some background information from the production site the conclusion could be drawn that the deposit formed in the process contains "frit glass", which is high in boron and zinc. With this information the problem occurring during processing could be identified and solved.

	cotton (µg/g)	cotton + sampled material (µg/g)
Main component	**C**	**C**
Li	< 3	100000
B	< 10	220000
Na	2500	2500
Mg	200	400
Al	2000	2000
Si	< 1000	350000
K	1000	10000
Ti	200	500
Mn	50	25
Cu	40	30
Zn	40	180000
Ga	< 10	200
Sr	100	100
Zr	15	400
Mo	25	30
Sn	10	30
Ce	< 3	150
Ba	250	230
Pb	< 3	1000
Most other elements	< 3	< 3

Table 2: Comparison between a "blank" cotton bud and a cotton bud with sampled material. Elements that are characteristic for the sampled material are indicated in red. Values are semi-quantitative.

Philips Innovation labs Material Analysis lab
offers a full range of analytical methods and expertise to support both research and manufacturing, serving customers by taking an integral, solution-oriented approach.

World-class expertise – working for you

For more information:
Phone: +31 40 27 40455
E-mail: innovationlabs@philips.com
www.innovationlabs.philips.com

Application Note 12
August 2016

Philips Innovation labs