4. Future Developments

Our laboratory is continuing to study new chiral selectors for capillary electrophoresis and stationary phases for liquid chromatography. These new tools will have to be more selective and more rugged to analyze in routine complicated samples such as biological matrices, especially in the case of liquid chromatography.

Furthermore, the application of capillary electrophoresis and electrochromatography as well as μ-liquid chromatography in chiral analysis and in phytochemistry is under development in the LCAP. These powerful methods will permit to miniaturize the total analytical system and reduce the consumption of organic polluting solvents.

The coupling of separation methods with all kinds of selector and the development of new sample preparation techniques is needed to enhance selectivity and sensitivity. Finally, our laboratory is involved in the use of the mass spectrometry coupled to capillary electrophoresis, gas and liquid chromatography.

Received: July 17, 1997

[1] E. Varesio, J.-L. Veuthey, *J. Chromatogr., A* **1995**, *717*, 219.
[2] E. Varesio, J.Y. Gauvrit, R. Longeray, P. Lantéri, J.-L. Veuthey, *Electrophoresis* **1997**, *18*, 931.
[3] S. Rudaz, J.-L. Veuthey, *J. Pharm. Biomed. Anal.* **1996**, *14*, 1271.
[4] P. Nussbaumer, I. Kapetanidis, P. Christen, *Plant Cell Rep.*, in press.
[5] S. Cherkaoui, L. Mateus, P. Christen, J.-L. Veuthey, *J. Chromatogr., B*, **1997**, *696*, 283.
[6] S. Cherkaoui, L. Mateus, P. Christen, J.-L. Veuthey, *Chromatographia*, in press.
[7] A. Brachet, O. Muñoz, M. Gupta, J.-L. Veuthey, *Phytochemistry*, in press.
[8] M. Kohler, W. Haerdi, P. Christen, J.-L. Veuthey, *J. High Resol. Chromatogr.* **1997**, *20*, 62.
[9] M. Kohler, W. Haerdi, P. Christen, J.-L. Veuthey, *Phytochem. Anal.*, in press.

Chimia 51 (1997) 797–799

ISSN 0009–4293

Chemical Analysis in the Life Cycle of Products

Helene Felber and **Bruno Wampfler***

Abstract. Chemical analysis has an important function in the life-cycle of consumer goods and technical products. In this paper the variety of analytical chemistry provided at EMPA St. Gallen is shown by three typical examples concerning the two product categories plastic packaging materials and textile detergents. In addition EMPA is engaged in an overall promotion of the accuracy and reliability of amount-of-substance data.

1. Introduction

Large numbers of consumer goods and technical products are a constant part of our daily lives. We expect these products to be there for our benefit and that they in no way cause us any harm or injury either when they are being produced, during use or during the disposal phase. Irrespective of whether they are made of naturally produced biological material or synthetic material, all such products consist of a combination of basic chemical elements whose characteristics are determined by their chemical composition.

Chemical analysis has a central function in the total life cycle of a product (*Fig. 1*). It is an important instrument when carrying out market research and it also provides support for technical research and development of a product. Chemical analysis is used to characterise raw materials for manufacture and to control production processes. It helps to determine the optimum application range of a product and provides information on possible harmful side effects to man and the environment during the useful phase (*e.g.* release of harmful substances). It also provides information for deciding whether or how a product can be disposed of, whether it can be recycled, incinerated, composted or whether it has to be deposited at a landfill site.

**Correspondence*: Dr. B. Wampfler
EMPA
Swiss Federal Laboratories for Materials Testing and Research
Lerchenfeldstrasse 5
CH–9014 St. Gallen
Tel.: +41 71 274 77 86
Fax: +41 71 274 77 88
E-Mail: bruno.wampfler@empa.ch

2. Chemical Analysis at EMPA St. Gallen

EMPA St. Gallen is engaged in the chemical analysis of consumer goods and various technical products. Special attention is paid to packaging material, plastic products, washing and cleaning agents, wood preservatives, paints, varnishes and several more. The diversity of chemical analyses necessary for characterising such products can be illustrated by three examples.

2.1. *Plastic Packaging Material (Examples 1 and 2)*

The analysis of a plastic packaging includes the characterisation of the plastic itself using polymer or structure analysis methods such as SEC, TMA, DSC, FTIR, and also determining qualitatively and quantitatively the presence of organic and inorganic additives such as antioxygenes, UV stabilisers, softeners, colorants, filling material, and also trace elements. Usually chromatographic separation methods are used such as HPLC and GC coupled with suitable sampling and detection technologies (pyrolysis, head space or DAD, FID, MS).

Example 1: The Analyst as Detective

Very often investigations have to be carried out due to cases of damage. For instance, in a recent case consumers complained to a brewery that its beer had a strange smell and that it was not drinkable. Very soon the plastic bottle top came under suspicion. It was believed that trace compounds could have contaminated the beer. The GC/MS analysis indicated among others the presence of tribromphenol and tribromanisol. But where did these compounds come from? After systematic

4. Future Developments

Our laboratory is continuing to study new chiral selectors for capillary electrophoresis and stationary phases for liquid chromatography. These new tools will have to be more selective and more rugged to analyze in routine complicated samples such as biological matrices, especially in the case of liquid chromatography.

Furthermore, the application of capillary electrophoresis and electrochromatography as well as µ-liquid chromatography in chiral analysis and in phytochemistry is under development in the LCAP. These powerful methods will permit to miniaturize the total analytical system and reduce the consumption of organic polluting solvents.

The coupling of separation methods with all kinds of selector and the development of new sample preparation techniques is needed to enhance selectivity and sensitivity. Finally, our laboratory is involved in the use of the mass spectrometry coupled to capillary electrophoresis, gas and liquid chromatography.

Received: July 17, 1997

[1] E. Varesio, J.-L. Veuthey, *J. Chromatogr., A* **1995**, *717*, 219.

[2] E. Varesio, J.Y. Gauvrit, R. Longeray, P. Lantéri, J.-L. Veuthey, *Electrophoresis* **1997**, *18*, 931.

[3] S. Rudaz, J.-L. Veuthey, *J. Pharm. Biomed. Anal.* **1996**, *14*, 1271.

[4] P. Nussbaumer, I. Kapetanidis, P. Christen, *Plant Cell Rep.*, in press.

[5] S. Cherkaoui, L. Mateus, P. Christen, J.-L. Veuthey, *J. Chromatogr., B*, **1997**, *696*, 283.

[6] S. Cherkaoui, L. Mateus, P. Christen, J.-L. Veuthey, *Chromatographia*, in press.

[7] A. Brachet, O. Muñoz, M. Gupta, J.-L. Veuthey, *Phytochemistry*, in press.

[8] M. Kohler, W. Haerdi, P. Christen, J.-L. Veuthey, *J. High Resol. Chromatogr.* **1997**, *20*, 62.

[9] M. Kohler, W. Haerdi, P. Christen, J.-L. Veuthey, *Phytochem. Anal.*, in press.

Chimia 51 (1997) 797–799
© Neue Schweizerische Chemische Gesellschaft
ISSN 0009–4293

Chemical Analysis in the Life Cycle of Products

Helene Felber and **Bruno Wampfler***

Abstract. Chemical analysis has an important function in the life-cycle of consumer goods and technical products. In this paper the variety of analytical chemistry provided at EMPA St. Gallen is shown by three typical examples concerning the two product categories plastic packaging materials and textile detergents. In addition EMPA is engaged in an overall promotion of the accuracy and reliability of amount-of-substance data.

1. Introduction

Large numbers of consumer goods and technical products are a constant part of our daily lives. We expect these products to be there for our benefit and that they in no way cause us any harm or injury either when they are being produced, during use or during the disposal phase. Irrespective of whether they are made of naturally produced biological material or synthetic material, all such products consist of a combination of basic chemical elements whose characteristics are determined by their chemical composition.

Chemical analysis has a central function in the total life cycle of a product (*Fig. 1*). It is an important instrument when carrying out market research and it also provides support for technical research and development of a product. Chemical analysis is used to characterise raw materials for manufacture and to control production processes. It helps to determine the optimum application range of a product and provides information on possible harmful side effects to man and the environment during the useful phase (*e.g.* release of harmful substances). It also provides information for deciding whether or how a product can be disposed of, whether it can be recycled, incinerated, composted or whether it has to be deposited at a landfill site.

**Correspondence*: Dr. B. Wampfler
EMPA
Swiss Federal Laboratories for Materials Testing and Research
Lerchenfeldstrasse 5
CH–9014 St. Gallen
Tel.: +41 71 274 77 86
Fax: +41 71 274 77 88
E-Mail: bruno.wampfler@empa.ch

2. Chemical Analysis at EMPA St. Gallen

EMPA St. Gallen is engaged in the chemical analysis of consumer goods and various technical products. Special attention is paid to packaging material, plastic products, washing and cleaning agents, wood preservatives, paints, varnishes and several more. The diversity of chemical analyses necessary for characterising such products can be illustrated by three examples.

2.1. *Plastic Packaging Material (Examples 1 and 2)*

The analysis of a plastic packaging includes the characterisation of the plastic itself using polymer or structure analysis methods such as SEC, TMA, DSC, FTIR, and also determining qualitatively and quantitatively the presence of organic and inorganic additives such as antioxygenes, UV stabilisers, softeners, colorants, filling material, and also trace elements. Usually chromatographic separation methods are used such as HPLC and GC coupled with suitable sampling and detection technologies (pyrolysis, head space or DAD, FID, MS).

Example 1: The Analyst as Detective

Very often investigations have to be carried out due to cases of damage. For instance, in a recent case consumers complained to a brewery that its beer had a strange smell and that it was not drinkable. Very soon the plastic bottle top came under suspicion. It was believed that trace compounds could have contaminated the beer. The GC/MS analysis indicated among others the presence of tribromphenol and tribromanisol. But where did these compounds come from? After systematic

CHIMIA 51 (1997) Nr. 10 (Oktober)

questioning of all production and logistics operators and several analyses of various materials (plastic, wood) the problem was solved. The beer bottle-tops had been packed by the manufacturer into plastic bags. During transport from the manufacturer to the beer filling plant the plastic bags were stored for a considerable time on old wooden palettes which were contaminated with tribromphenol and tribromanisol (the second compound was possibly due to bacterial methylation of the first compound). Both materials diffused from the wood through the polyethylene foil into the beer bottle-top and from there into the beer.

Example 2: The Analyst as Partner in an Interdisciplinary Project

Current investigations are being carried out in collaboration with industrial companies and research institutes dealing with bottles made of polyethylene terephthalate (PET) to study whether food packaging can be recycled for reuse as food packaging. The main task is to prevent diffusion of harmful substances from contaminated recycled material into the drink. It should be possible to solve this problem using a three-layer bottle: a recycled layer in the middle with a layer on each side made of new material (*Fig. 2*). Microscopic investigations show that a three-layer bottle can be easily blown without the danger of the recycled material penetrating the inner and outer layers. The current work is studying whether migration of contaminant is possible through the inner layer into the liquid contained in the bottle. For these tests the middle layer was contaminated with a three-layer foil containing polar to nonpolar volatile organic compounds. Pockets were formed from the foil, filled with solvent and then closed again. After different storage times at slightly raised temperatures, samples will be taken and the contaminants in the solvent determined using gas chromatography. The results will provide information as to whether the concept using three-layer packaging can be used or not.

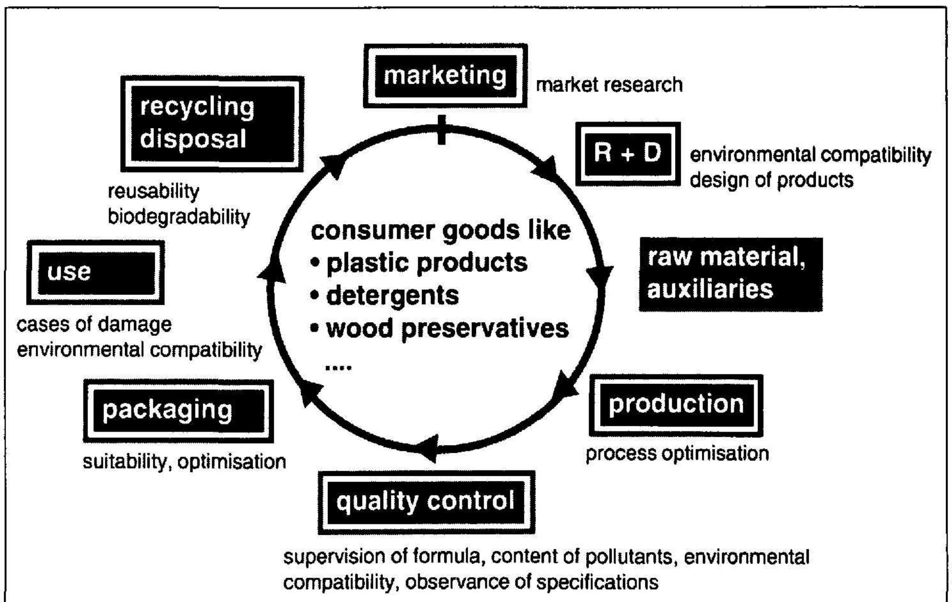

Fig. 1. *Chemical analysis in the life cycle of consumer goods and technical products.* The areas marked in dark frames are those in which EMPA St. Gallen is active.

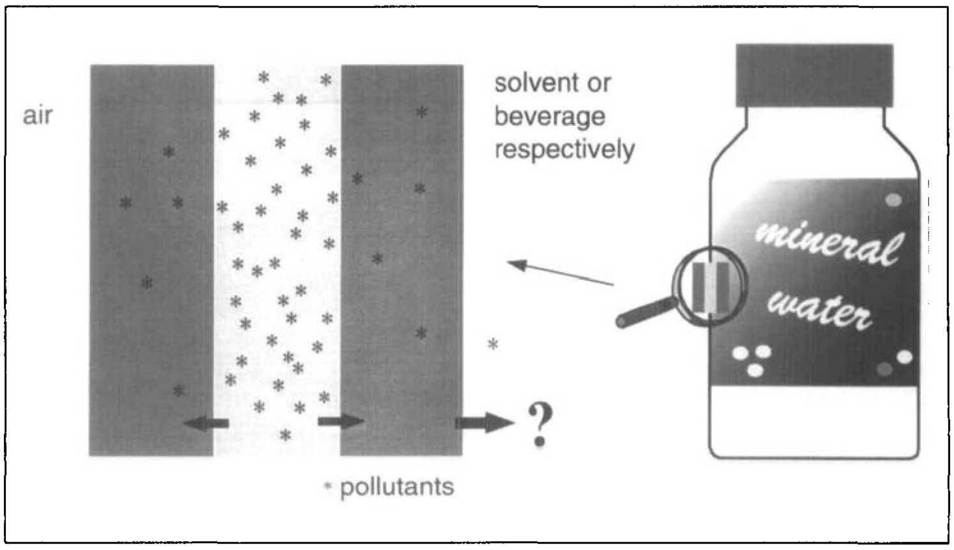

Fig. 2. *Model for the migration of contaminants through a polymer layer in a three-layer food packaging*

2.2. *Textile Detergents (Example 3)*

Textile detergents usually consist of up to 20 individual substances some of which are a mixture of several hundred isomers. The substances range from inorganic components (zeolite, various silicates, carbonate, perborate) to defined organic substances (phosphonate, citrate, TAED) and to complex composites or polymer compounds (surfactants, polycarboxylate, odiferous agents, *etc.*). Therefore, the analysis has to meet very high demands. The inorganic components can usually be determined well and precisely employing classical and electrochemical procedures such as gravimetry, titration, potentiometry, polarography. Individual elements like silicium or aluminium (in silicates, zeolite) are quantified using atom spectrometric methods such as AAS or ICP/OES. While simpler organic molecules can usually be determined using a defined method (*e.g.* TAED with GC or HPLC, phosphonate and citrate with ion chromatography), surfactant analysis, in particular, demands a sophisticated combination of various separation and coupling techniques (isolation by extraction, separation by specific ion exchangers, LC or GC, identification using FTIR, NMR, MS, usually after derivation).

Example 3: The Analyst Providing Ecological Services

What is the purpose of detergent analysis? First of all we could mention checking production quality. Because in these cases the composition of the product is known, the analysis is comparatively easy. Some institutes and service laboratories all over the world provide such analysis services at unbelievably low prices. If, however, the important factor for a client is a comparison of products for ecological product assessment, an analysis has to be carried out on products whose contents are unknown. With the exception of large detergent manufacturers, there are very few laboratories worldwide that are in a position to carry out such analyses. EMPA is one of them. For service laboratories the

CHIMIA 51 (1997) Nr. 10 (Oktober)

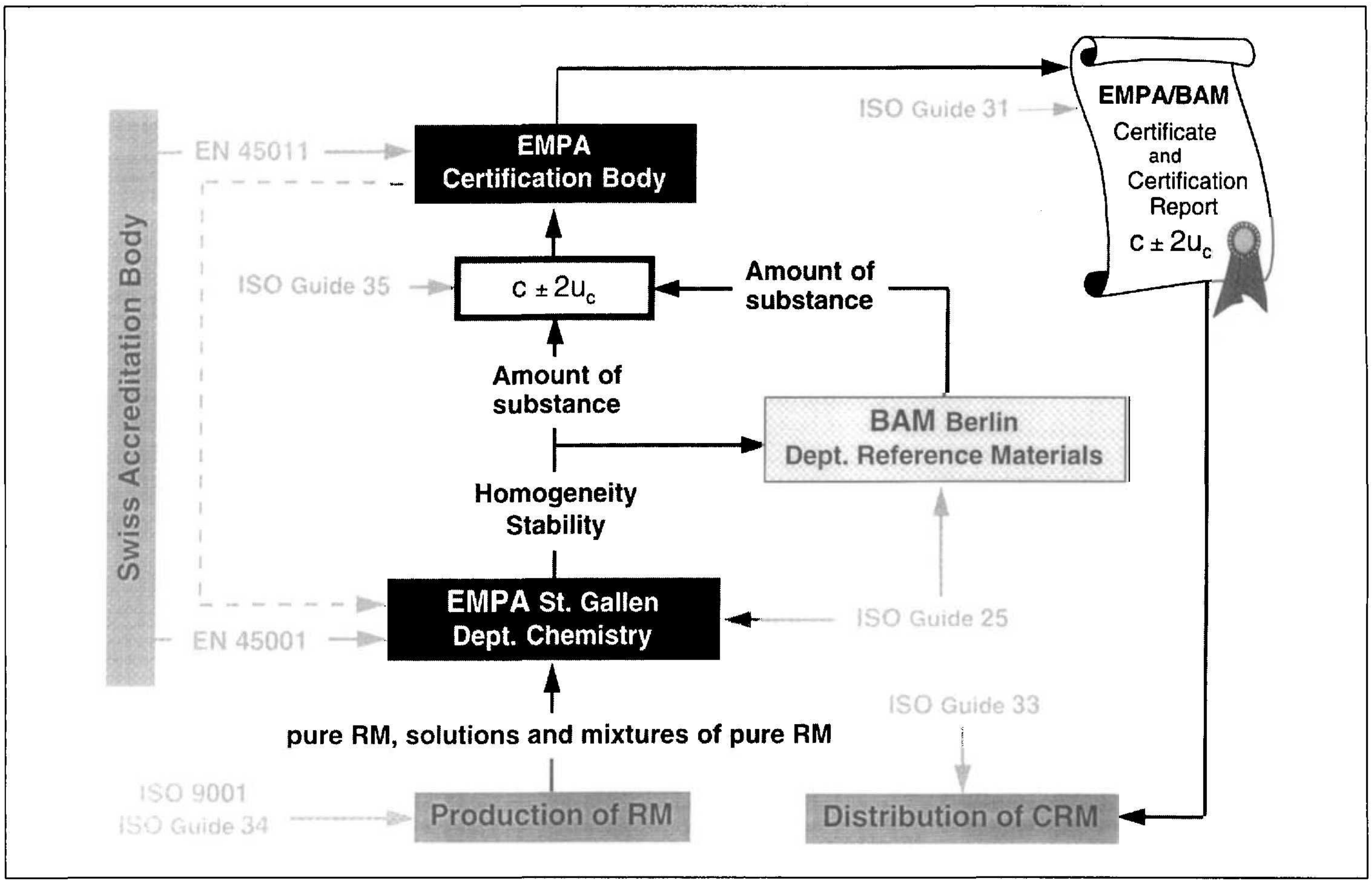

Fig. 3. *Procedure for the certification of reference material at EMPA St. Gallen according to ISO and EN guidelines.* RM: reference material, CRM: certified reference material, c: certified value, u_c: combined uncertainty.

greatest difficulty with such analysis tasks is the remoteness of the product, meaning that the latest trends with respect to the substances and the composition are not known. Technical literature helps very little because the information required is published too late. On the other hand the analytic laboratories of detergent manufacturers are completely familiar with these trends because of their close association with development laboratories and because of their intensive market research activities. Competitors' products are acquired daily on the worldwide market and analysed. Service laboratories, whose clients are usually small and medium-size companies, or institutes performing ecological assessment studies can only combat this difficulty by increasing their analytic capacity.

2.3. *The Quality of Chemical Analysis Test Results* (Amount of Substance Measurements)

When we consider how chemical analysis data effects the 'life' of a product, then it is important that we must ensure that this data is as correct as possible. It is a general problem of modern chemical analysis that, using efficient technologies, a great deal of amount of substance data can be acquired in a short period of time, but at the same time very little knowledge is available on the accuracy and reliability of this data. For this reason it is hardly surprising that the results of many interlaboratory tests indicate widely varying data on the amount of substances. This problem is not only due to the use of modern analysis technology but because, in contrast to physical measuring techniques, chemistry does not have an accepted system which permits the data to be traced back to a common measurement basis. It was not until 26 years ago that the unit of measurement for the amount of a substance, the mole, was defined and accepted into the international system of units (SI). Currently worldwide organisations of chemists (Eurachem, CITAC) and metrologists (CCQM, Euromet, *etc.*) are making great efforts to determine a recognised measurement basis and a concept for traceability. EMPA, as independent Swiss laboratory, is also engaged in these efforts. Irrespective of what concept is finally accepted, it is certain that determining the uncertainty of measurement in a complete analysis process is a decisive link in the chain. In 1993 ISO defined how measurement uncertainty has to be calculated. Two years later Eurachem prepared a guide which gives instructions for implementing the ISO guidelines. Experience has shown, however, that the guidelines from ISO and Eurachem are still not well known and hardly implemented at all. For this reason know-how transfer is another of our main tasks. In this connection it is of particular importance that the practical demonstration of how the uncertainty of measurement can be calculated is based on procedures carried out daily. Most of the latest analysis procedures employ methods which are related to a reference quantity (reference material), and, therefore, the quality of this reference material has a decisive influence on the reliability of the results. Currently insufficient certified reference materials traceable to the SI are available on the market. Many commercially available reference materials cannot meet their specifications and, therefore, they make a large contribution to the uncertainty of measurement and unfortunately thus also to the incorrectness of chemical analysis data. In order to correct this situation, EMPA, in close cooperation with the German Federal Office for Material Research and Testing (BAM), certifies reference material strictly according to ISO guidelines 30–35 (*Fig. 3*).