Process Analytical Chemistry

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Control, Optimization, Quality, Economy

With 97 figures

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Preface

Industrial process analytics is, like the whole field of analytical chemistry, an integral and essential part of every industrial company based on chemical reactions. It provides decision aids in the series of process steps, so its results have decisive technical, economic and ecological effects. Therefore, this part of analytics must hence be included in the teaching of modern "applied" analytical chemistry. Only in this way can the student, particularly the advanced one, recognise the real relevance of this scientific matter, and learn the way the methods of analytics are applied for solutions to problems in actual practice: a discrepancy between university training and working life realities is avoided and the variety in "instrumental" (physical) analytics becomes more comprehensible.

This book is therefore aimed, on the one hand, at advanced students of analytical chemistry, chemical engineering (chemical process technology), material sciences and, on the other hand, at the analytical practitioner, the chemical engineer and the process engineer who requires information about the possibilities and methods of process analytics and knowledge about their efficiency, e.g. with regard to industrial process optimization. In this case, the chemical engineers and chemical engineering technicians working in small and medium-sized companies are also included, these having to solve process analytical problems without an experienced analyst by their sides. For this purpose, lists of suppliers added to particular chapters, which naturally cannot make any claims to completeness, are intended to be helpful, and can make practical problem solving easier. For the advanced students interested in analytics this volume is intended to impart insights into the necessity for the development and the application of analytical methods in industrial practice, and it can possibly serve the purpose of orientation for advanced studies.

This presentation of industrial process analytics is supposed to be a supplement to proven text books and manuals and is made in the knowledge that more recent books in the field of industrial analytics are scarce.

For the description of the contents of the first chapters, divided according to the aggregate states, the scientific basis of the methods is briefly presented and after that the state-of-the-art is described by examples. The references annexed to each chapter are intended to provide more profound access to the matter. The publications cited can provide further help.

In some chapters examples for the interaction and the mutual dependence and reciprocal influencing of various disciplines are described. In this way, for example, the future analyst or chemical engineer is directly acquainted with the necessity of an interdisciplinary dialogue.

In a concluding chapter the integration of quality assurance - a concept which in connection with the European Market has become of considerable importance for the whole industry and for the analytics - into process engineering and process analytics is dealt with.

Finally it should be mentioned that, when dealing with particular sub-areas, the scientific basis of the analytical methods is generally presupposed.

For stimulating discussions and helpful indications the undersigned would like to thank most sincerely Prof. Dr. Manfred Grasserbauer. Furthermore, special thanks to Dr. J. Flock, Dortmund, for preparing numerous figures. Finally, many thanks to the Springer-Verlag for the unbroken realization of this project without any problem.

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1 Introduction

1.1 Fields of Application of Industrial Analytics

First of all, the term of (chemical) "analytics" should be briefly explained in order to be able to classify that special part of analytical chemistry [1], the *industrial process analytics (process analytical chemistry)*, which will be treated here. By "analytics" we understand the obtaining of information not only on the qualitative and/or quantitative composition, but also on the geometrical structure of substances [1], including the sampling and the preparation of the material to be investigated and the very difficult and time-consuming evaluation of measured results [2] (chemometrics [3]). This processing of analytical data includes, in special cases, the process step of data reduction in order to obtain a plausible and directly comprehensible result. From this characterization it follows that analytics goes far beyond the field of traditional analytical chemistry [4, 5].

The analytical *result* is, in certain circumstances, not only useful for the industrial client (process engineer) or the researcher but at the same time it is of importance for the consumer (items of everyday use), the legislator [6] (cf., e.g., the German Chemicals Act "Chemikaliengesetz"; see Sect. 9.2.2) and/or the media as representatives of the public interest (environmental relevance) [7]. In order to reach the analytical goal a strategy is required which has its starting point in the description of the object and after that defines the methods for

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1 General *definition*: "Analytical chemistry" is the science of the synoptical micro- and/or macrological observation and informational processing of the material-related and reagent-dependent signals from the chemical, physical or biochemical reactions between sample and reagent which leads to the clarification of the substance.

Definition by K. Cammann *(Competition: "Analytical chemistry – today's definition and interpretation", 1992)*: Analytical Chemistry is defined as the self-reliant, chemical sub-discipline which develops and delivers appropriate methods and tools to gain information on the composition and structure of matter, especially concerning type, number, energetic state and geometrical arrangement of atoms and molecules in general or within any given sample volume.

Definition of the Working Party of Analytical Chemistry (WPAC) of the FECS (EUROANALYSIS VIII, Edinburgh (UK), 1993: "Analytical chemistry is a scientific discipline which develops and applies methods, instruments and strategies to obtain information on the composition and nature of matter in space and time."

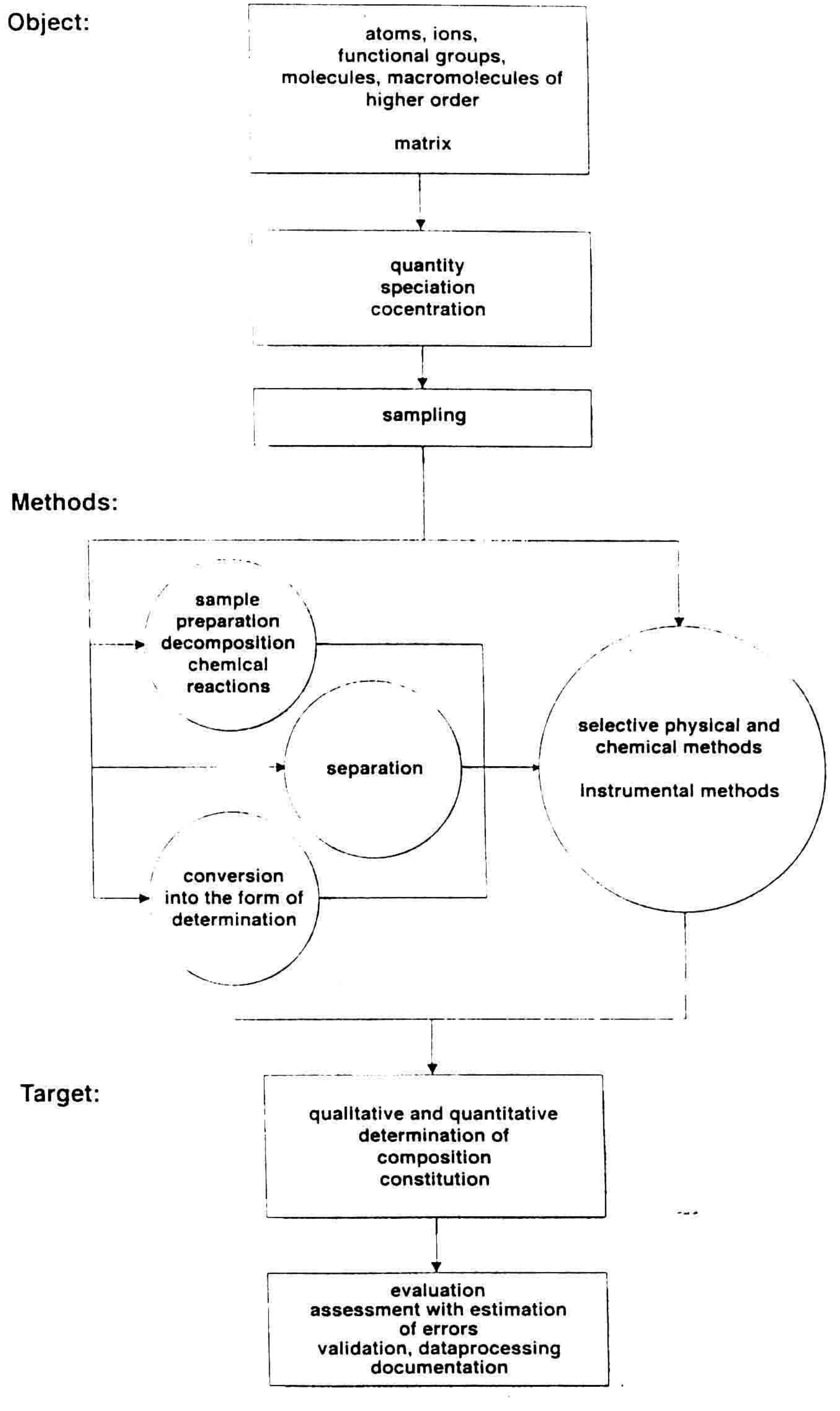

Fig. 1.1. Strategy of analytics

reaching the goal (Fig. 1.1). With regard to the methods it must be noted that modern analytics is characterized by a variety which, by a combination of methods, permits the solution of the most complicated questions. As an example of the temporal development of this broadening of analytical methodology, attention should be drawn to the steel industry as one of the basic materials industries (Fig. 1.2). While in 1950 only purely chemical methods were used, the widespread introduction of atomic spectroscopy started in 1960; in 1970 phase analytics was already of great significance and the application of gas chromatography as well as of infrared spectroscopy began; since 1980 surface analytics became increasingly important. The factors decisive for these developments will be explained at a later stage.

The title of this book *"Process Analytical Chemistry"* already outlines the programme for the subject field [8] to be dealt with and contains a subject-oriented claim which is to be defined below. According to it, process analytical chemistry [9] is to be understood as part of the instrumental analytics used in process engineering which means the application of multi-element and/or multi-method concepts [10]. This field of process analytics – or rather "chemical process analytics" – is thus demarcated as against the process-accompanying measurement of physical variables such as temperature, pressure, viscosity, etc., which only under certain conditions can be regarded as process analytics. The (chemical) process analytics covers discontinuously and continuously working methods, whereby in-line and on-line procedures [2] are promoted and developed more and more [11]. The latter are gaining increasingly in technical and economic significance, in the course of which, during the development phase, considerable material-related problems frequently have to be solved [12].

The field of application of *industrial analytics* as a whole[13] naturally goes far beyond the area of process analytics in the stricter sense already outlined [14, 15]. Complementary to the purely process-accompanying and product-describing investigations comes the analysis of

- raw materials of the most varied type (possibly including samples from ore prospecting or raw material production),
- by-products of various process stages,
- competitive products in various markets,
- auxiliary materials such as boiler feed-water, water fit for industrial use, lubricants, fuels, gaseous, liquid and solid fuels, construction and painting materials,
- waste gases and waste water including their assessment with regard to environmental matters and legal regulations,

2 in-line = investigation in the production flow (without sample taking)
on-line = investigation of partial quantities continuously sampled and analysed
off-line = investigation of samples discontinuously sampled and analysed without direct (automatic) linking to the process
at-line = quick testing near the process

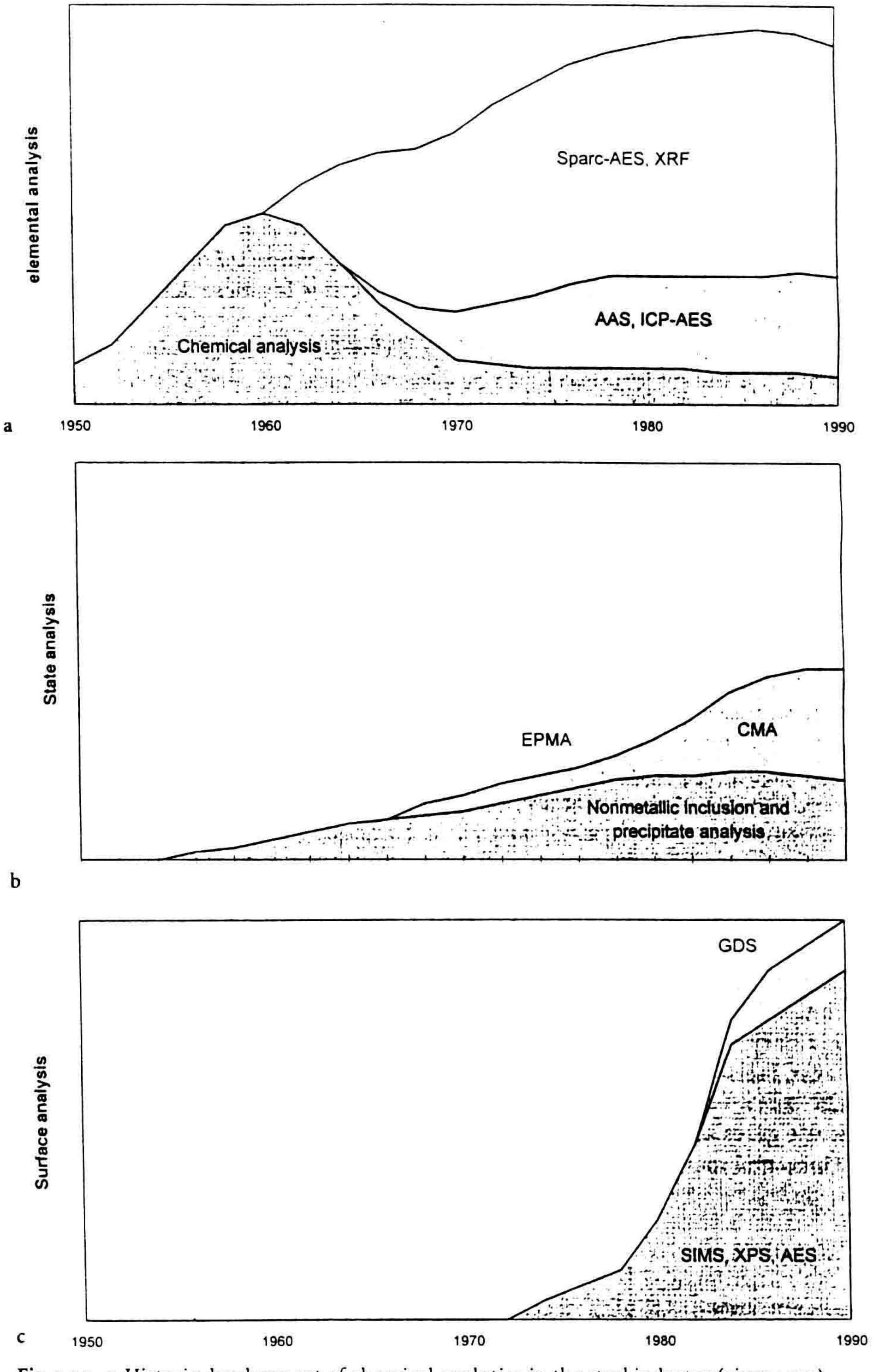

Fig. 1.2 a–c. Historic development of chemical analytics in the steel industry (since 1950)

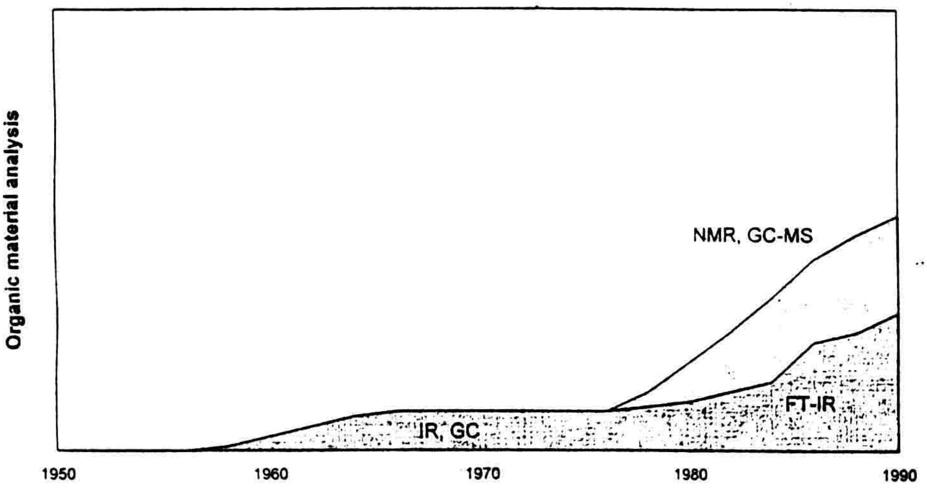

Fig. 1.2 d. Historic development of chemical analytics in the steel industry (since 1950)

- detergents and cleansing agents for the most varied purposes (cleaning of machine parts, plants, workshops, laundries, etc.),
- samples from the fields of ergonomics and industrial medicine.

(This list cannot make any claim to completeness due to the difference in the analytical requirements in the individual sectors of industry).

The levels of investment and efforts for specific research projects by the most important industrial branches to safeguard their future business differ widely and depend on a number of technical and economic factors. In any case, these future-safeguarding measures include, to a not inconsiderable extent, development and application of analytical methods. The same applies to competition between various materials which is characterized by a large range of substitution tendencies (Fig. 1.3). Here, too, analytics plays an important part in the characterization of conventional and newly developed materials as well as the description of their chemical properties.

The activities mentioned comprise, in addition, problem-related research and development work and the training of staff and of new young personnel (professional training of chemical laboratory assistants, preparation of application-orientated dissertations and theses).

Considerable technical and economic importance [11] is attached to *process analytical chemistry*, as will be shown by a number of impressive examples. The technical importance lies partly in the fact that this field of analytics enables the description and control of technical processes and the characterization of the products. The economic aspect consists of the creation of preconditions for cost minimization of process technology. Moreover, this area of analytics delivers a considerable contribution to quality control of products which will be looked at more closely elsewhere.