Analytical Chemistry

The Approved Text
to the FECS Curriculum
Analytical Chemistry

Edited by
R. Kellner,
J.-M. Mermet,
M. Otto,
H. M. Widmer

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Foreword

Within the physical sciences, analytical chemistry has probably undergone the most dramatic expansion and development during the past few decades. Consequently, the number of analytical techniques, their degree of sophistication and areas of application have increased tremendously. This poses a problem for the chemical educator: what to include in an undergraduate course and at which level?

Since its establishment a quarter of a century ago, the Federation of European Chemical Societies (FECS) has taken a keen interest in chemical education and analytical chemistry, which is shown by the fact that these two areas are represented as divisions within the FECS structure. After conducting extensive surveys the Division of Analytical Chemistry introduced the concept of a Eurocurriculum which received a favourable response from Europe as well as from overseas. A logical follow-up and tool to implement this Curriculum Analytical Chemistry worldwide is the present textbook.

The late I.M. Kolthoff, one of the great pioneers of analytical chemistry, used the motto *theory guides, experiment decides* in his books. The present textbook aims at imparting analytical insight by a suitable blend of theoretical background, experimental and instrumental details and finally a glimpse from the real world of applications. The ultimate goal is to convey analytical thinking for problem-solving.

Our rapid technological progress and at the same time enhanced concern for the environment are guarantees that the number of analytical problems will certainly increase and become more complex. Let us hope that this timely textbook will make a contribution to the education and training of future analytical chemists in order to have enough analytical problem-solvers qualified to meet these new challenges.

September 1997

Lauri Niinistö
President FECS

Preface

Analytical chemistry is – depending on the point of view – the oldest as well as the youngest branch of chemistry, the science of the transformation of matter. Claiming to be the oldest branch of natural philosophy (as "chemistry" has been described) goes back to the use of the Aristotelian syllogism for argumentation and proof (as shown in his "Analytiken"). Claiming to be the youngest branch became a possibility as "analytics" emerged as an own scientific discipline based on application of modern knowledge theories and information science in chemistry. The emancipation of analytical chemistry from chemistry began with Robert Boyle, continued with the activities of Lavoisier, Berzelius, Wöhler and Liebig, culminated the first time 100 years ago with Wilhem Ostwald (and his work *Die wissenschaftlichen Grundlagen der Analytischen Chemie*) and led to its present autonomy as a separate, very complex and highly attractive branch of science, described in its fundamentals in this book. The development of analytical chemistry is still continuing at a dramatic rate: consider the dynamic impact of Jan Heyrovsky (Electroanalysis), Richard R. Ernst (NMR), Gerhard Binnig and Heinrich Rohrer (STM/AFM) to name just a few of today's great researchers. As a consequence of the early and continuing interest in this field, a wealth of empirical knowledge, both of fundamental and of practical importance, about the material world in and around us has been compiled.

Chemistry as a whole has evolved worldwide into a supporting pillar of human culture, industry and trade, providing numerous goods of urgent daily need for humankind, such as food, clothing, shelter, pharmaceuticals and materials essential for medical use, transport or communication. Purely empirically at first, and mainly a branch of medicine, today's chemistry is a modern experimental science underpinned by physico-chemical and mathematical laws and has itself diversified into organic chemistry, inorganic chemistry, biochemistry, food chemistry, chemical technologies, physical chemistry and lately analytical chemistry.

It is certainly true that modern analytical chemistry, with its plethora of sensitive and selective techniques, has also strongly contributed to the awareness of environmental problems and issues and to the establishment of quality control systems in industrial production, in the health area and in the environment. The world market for analytical instruments has grown significantly over the years into today's impressive 1000 billion US $ size. Under the "responsible care" program of today's chemical industry, misuse of unoptimized technologies will ultimately be banned. The principle of "sustainable development" has been accepted as a basis of the production philosophy of the major chemical companies in Europe and is under consideration elsewhere. Analytical chemistry has been given a decisive role in controling the success of this process and in preserving the ecological balance of our world today.

The Division of Analytical Chemistry (DAC) of the FECS (Federation of European Chemical Societies) defines: "*Analytical chemistry is a scientific discipline that develops and applies methods, instruments and strategies to obtain information on the composition and nature of matter in space and time*".

This textbook – besides its unique role in university education – can also be considered as an interpretation of this DAC definition of analytical chemistry and as a pillar of support of the fact that analytical chemistry is indeed an own branch of science today - an *information science* - answering the theoretically and practically important question, how the material world is composed.

In order to find answers to this question, analytical chemists use chemical, physical and biological reagents to interact with the samples under investigation. This reagent-oriented view is also the scheme adopted for this textbook for practical reasons. In some cases the methods of analysis are clearly chemical, such as acid-base titrations where chemical reagents are used exclusively or purely physical, such as x-ray fluorescence. On the other hand the reader will soon notice that in some cases there are no clear, sharp differentiations, such as in chromatography, listed here under "chemical analysis" or "scanning tunneling microscopy" which has been linked to "physical analysis" although both areas touch both chemical *and* physical aspects.

Analytical chemistry is an *in-between-science* using and depending on the laws of chemistry, physics, mathematics, information science and biology. Its aim is to decipher the information hidden in the sample under investigation, not to change this intrinsic information, hence to tell the *truth* about the composition of the material world. This sounds trivial to a scientist, but it is not in today's complex and complicated technical and environmental matrices where analytical data frequently have to be made available in *real time* and *in situ* (*in the unchanged matrix*). Pressing needs of modern world trade, industry and commerce have led to the creation of national - and more importantly - international bodies for quality assurance, such as EURACHEM and CITAC. These bodies demand that even experienced laboratories prove their technical competence by passing accreditation procedures comprising both technological standards and personal skills from time to time (see Chapter 3).

The worldwide training of analytical chemists in the analytical skills and knowledge required to meet these challenges demands a high level of education using a harmonized scientific language and worldwide agreement on the basic scientific contents to be taught. While English has today the unchallenged role as *lingua franca* in chemistry and physics, the DAC-Curriculum "Analytical Chemistry" is the first broadly accepted attempt to harmonize the basic curricula in analytical chemistry for the benefit of the chemistry students working under this scheme (see *Anal. Chem.* 1994, *66*, 98A for the basic part and *Fres. J. Anal. Chem.* 1997, *357*, 197 for the advanced part of the DAC-Curriculum).

This textbook *Analytical Chemistry* is the authentic version of the DAC-Curriculum "Analytical Chemistry" and has emerged from the former "WPAC-Eurocurriculum". Its concept is based on the balanced mix of traditional methods of chemical analysis (Part II), modern techniques of biological (also Part II) and physical analysis (Part III) as well as chemometrics (Part IV). The textbook chapters in Parts II to IV are preceded by an introductory Part I featuring general topics such as "Aims of analytical chemistry and its importance for society", "The analytical process" and "Quality assurance and quality control". The book is completed by an industrially relevant Part V "Total analysis systems" dealing with more complex "Hyphenated techniques" and "Process analysis systems" of industrial importance today and, in particular, in the future. It is a multiauthored book, in order to guarantee the highest level of competence also in the undergraduate level of modern university education. The book is devoted to the principle of combining solid foundations of scientific knowledge with flexibility towards novel analytical techniques. Its unique concept allows coverage of classical topics, such as acid-base titrations or compleximetry - which are necessary to understand modern chemical sensors technology - while also including recent, trendsetting developments in physical analysis, chemometrics and process analysis. For example, atomic force microscopy and miniaturized total analysis systems (μ-TAS) are introduced at an elementary level. Possible shortcuts in some chapters have to be seen in the light of the editorial decision to limit the size of the book to under 1000 pages and may be overcome by referring to the wealth of specialized textbooks for advanced studies that are part of the DAC-Curriculum scheme.

Besides providing chemistry students with a sound preparation for the requirements of modern industry, university studies in analytical chemistry must also provide fitness in basic academic research. Truth has an essential place in analytical chemistry. The critical student should therefore carefully study Chapters 2 ("The analytical process") and 3 ("Quality assurance and quality control"). Sir

Karl Popper's credo: "The approximation to the truth is in principle possible" can be accepted as a strong philosophical foundation of analytical chemistry as a basic science.

In the applied field, we are confronted with a worldwide flood of analytical data, resulting from the incredible number of 10 000 million analyses per year! It is the attempt of this elementary textbook and its editorial team to make clear that analytical chemistry has today more than ever a sound responsibility for the future development of our society.

Analytical data and model calculations on NO_x production in the stratosphere by supersonic jetliners, for instance, resulted in knowledge which prevented the development of a significant fleet of ozone-killing supersonic jets. This is just one example of our necessity to produce correct analytical data and to transfer them correctly into knowledge that we can use in problem solving. Correct knowledge is also the basis for wisdom, needed for instance for far reaching political decisions in general.

We are convinced, that when taught worldwide, analytical chemistry – based on an educational scheme devoted to the equilibrium between freedom and responsibility, as provided by this textbook and the DAC-Curriculum – can be a key science to provide for a safer future for mankind!

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R. Kellner, H. Malissa and E. Pungor
Chairmen of WPAC/FECS: 1993–97, 1975–81 and 1981–87
(since Sept. 1996 the WPAC continues as Division of Analytical Chemistry (DAC) of the FECS)

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Symbols

a	Auger yield
A	absorbance
b_0	intercept, background
b_1	sensitivity
$b_{1/2}$	full width at half maximum
c	concentration
C	covariance
CV	coefficient of variation
d	distance, cell thickness lattice spacing, mean deviation
D	diffusion coefficient
e	electron
E	energy, electrode potential, expectation
$E(X^r)$	rth (non-central) moment of X (or of $F(x)$)
$E\{(X-\mu)^r\}$	rth central moment of X (or of $F(x)$)
E_a	activation energy
E_B	binding energy
E_{kin}	kinetic energy
f	frequency
$f(z)$	standard normal density function
F	Fisher ratio, flow rate
$F(z)$	standard normal distribution function
G	free energy
H	plate height, enthalpy
I	intensity, nuclear angular momentum quantum number (spin), current, Kováts retention index, ionic strength
I_0	incident intensity
j	angular moment quantum number
J	coupling constant
k	rate constant, relative sensitivity factor
k'	capacity factor
K	equilibrium constant
K_M	Michaelis-Menten rate constant
l	orbital quantum number
L	distance between sample and observation screen
m	magnetic quantum number
M	multiplicity
n	order of diffraction, rotation axis, refractive index
$\bar{n}$	rotary inversion axis
N	number of counts, areal density, plate number
NA	numerical aperture
P	angular momentum of nucleus, probability
Q	number of incident projectiles
r	distribution correlation coefficient, radius
R	resolution, range of electrons, radius of diffraction, resistance
R_f	retardation factor
R_S	resolution

s	standard deviation (estimate)
S	entropy, similarity
s^2	variance (estimate)
t	time, film thickness, Student factor
$t_{1/2}$	half-life time
t_M	hold-up (mobile) time
t_R	total retention time
T	true value of the measured quantity, transmittance
T_1	spin-lattice relaxation time
T_2	spin-spin relaxation time
$\bar{u}$	average linear velocity of molecules of the mobile phase
U	DC potential, voltage
v	reaction rate, linear velocity
$\bar{v}$	average linear velocity of analyte
v_0	initial reaction rate
V	variance
V_M	hold-up (mobile) volume
V_R	total retention volume
w	peak width
x	scalar variable
$\boldsymbol{x}$	vector of x-values
$\boldsymbol{X}$	matrix of x-values
$\bar{x}$	mean, arithmetic mean or average in a set of n observations
y	variable
Y	sputter yield
z	number of elementary charges
Z	standard normal deviate, atomic number
α	significance level, selectivity factor, degree of dissociation
β	yield of ion detector, phase ratio, cumulative stability constant
χ^2	Chi-squared (distribution)
δ	chemical shift
ε	molar absorptivity
Φ	flux density, work function
λ	wavelength, radioactive decay constant
η	efficiency, abundance
γ	gyromagnetic ratio
Γ	Gamma function
μ	population mean, magnetic moment, ionic strength
ν	degrees of freedom, frequency
$\Delta\Omega$	acceptance angle of detector
θ	scatter angle, pulse angle, diffraction angle
ρ	mass density
σ	shielding constant, population standard deviation
σ^2	population variance
τ_p	pulse duration
ω	fluorescence yield, cyclotron frequency
$\varnothing$	dihedral angle

Abbreviations and Acronyms

2D	two-dimensional
AALA	American Association for Laboratory Accreditation
AAS	atomic absorption spectrometry
AC	alternating current
ADC	analog-to-digital converter
AED	atomic emission detector
AEM	analytical electron microscopy
AES	atomic emission spectrometry, Auger electron spectrometry
AFM	atomic force microscopy
AFNOR	Association Française de Normalisation
AFS	atomic fluorescence spectrometry
AOAC	Association of Official Analytical Chemists
AL	atomic layer
APCI	atmospheric-pressure chemical ionization
API	atmospheric pressure ionization
ARM	atomic resolution microscopy
ARUPS	angle resolved UPS
ATR	attenuated total reflectance
BB	broad-band (decoupling)
BCR	Bureau Communautaire de Référence
BE	reversed geometry: magnetic sector + electrostatic analyzer
BIPM	Bureau International des Poids et Mesures
BSI	British Standards Institute
CAMM	computer-aided molecular modelling
CAR	continuous addition of reagent
CCD	charge-coupled device
CCT	constant current topography
CE	capillary electrophoresis
CEN	Comité Européen de Normalisation
CENELEC	Comité Européen de Normalisation Electrotechnique
CFA	continuous flow analysis
CF-FAB	continuous flow fast-atom bombardment
CGC	capillary gas chromatography
CI	chemical ionization
CID	collision-induced dissociation
CID	charge-injection device
CITAC	Cooperation on International Traceability in Analytical Chemistry
COSY	correlation spectrometry
CPAA	charged particle activation analysis
CRM	certified reference material
CTD	charge-transfer device
CV	coefficient of variation
CW	continuous wave
DAD	diode array detection (detector)
DAS	desamino-sulphadimidine
DBE	double-bond equivalent

DC	direct current
DCI	desorption chemical ionization
DCP	direct current plasma
DCPU	dichlorophenyl urea
DEPT	distorsionless enhancement by polarization transfer
DIN	Deutsches Institut für Normung
DLI	direct liquid introduction
DMA	dynamic mechanical analysis
DSC	differential scanning calorimetry
DTA	differential thermal analysis
DTG	differential thermogravimetry
EAL	European Cooperation for Accreditation of Laboratories
EB	forward geometry: electrostatic analyzer + magnetic sector
EC	European Commission
ECD	electron capture detector
ED-XRF	energy-dispersive X-ray fluorescence (spectrometry)
EDL	electrodeless discharge lamp
EELS	electron energy loss spectrometry
EFTA	European Free Trade Association
EGA	evolved gas analysis
EI	electron impact (ionization)
EL	electro luminescence
ELD	electro luminescence display
EN	European Norm
EPA	Environmental Protection Agency (USA)
EPXMA	electron probe X-ray microanalysis
ERD	elastic recoil detection
ESA	electrostatic analyzer
ESP	electrospray
ETA	electrothermal atomizer, emanation thermal analysis
ETSI	European Telecommunication Standard Institute
EU	European Union
EXAFS	extended X-ray absorption fine structure (spectrometry)
FAB	fast atom bombardment
FD	field desorption
FDA	Food and Drug Administration (USA)
FG	functional group (chromatogram)
FIA	flow injection analysis
FID	free induction decay
FID	flame ionization detector
FIM	field ion microscopy
FIR	far infrared (radiation)
FNAA	fast neutron activation analysis
FT	Fourier transform
FT-ICR	Fourier-transform ion cyclotron resonance (spectrometry)
FTIR	Fourier transform infrared (spectrometry)
FT-MS	Fourier-transform mass spectrometry
FWHM	full width at half maximum
GC	gas chromatography
GDL	glow discharge lamp
GDMS	glow discharge mass spectrometry
GF-AAS	graphite furnace atomic absorption spectrometry
GLP	good laboratory practice
GMP	good manufacturing practice
GNP	gross national product
GO	glucose oxidase
GS	Gram-Schmidt (algorithm)
HCL	hollow cathode lamp
HPDE	high-density polyethylene
HPGe	high purity germanium (detector)
HPLC	high performance liquid chromatography
HPTLC	high performance thin-layer chromatography

ICP	inductively coupled plasma
ICP-MS	inductively coupled plasma mass spectrometry
ICTAC	International Confederation of Thermal Analysis and Calorimetry
IDF	International Dairy Federation
IDMS	isotope dilution mass spectrometry
ILAC	International Laboratory Accreditation Cooperation
INAA	instrumental neutron activation analysis
IQR	interquartile range
IR	infrared (radiation)
IRN	indicator radionuclides
ISO	International Organization for Standardization
ISO/REMCO	ISO Council Committee on Reference Materials
ISP	ionspray
ISS	ion scattering spectrometry
IUPAC	International Union for Pure and Applied Chemistry
JCPDS	Joint Committee for Powder Diffraction Standards
KRS-5	thallium-bromide-iodide (ATR crystal material)
LAMMS	laser micro mass spectrometry
LARIS	laser atomization resonance ionization mass spectrometry
LBB	Lambert-Bouguer Beer's law
LC	liquid chromatography
LEED	low energy electron diffraction
LEEM	low energy electron microscopy
LNRI SNMS	laser non-resonant ionization SNMS
LQR	lower quartile range
LRI SNMS	laser resonance ionization SNMS
LRMA	laser Raman micro analysis
m/z	mass-to-charge ratio
MALDI	matrix-assisted laser desorption/ionization
MCA	multichannel analyzer
MCT	mercury cadmium telluride
MEIS	medium energy ion scattering (spectrometry)
MID	multiple-ion detection
MIP	microwave-induced plasma
MIR	middle infrared (radiation)
MS	mass spectrometry
MS-MS	tandem mass spectrometry
M&T	Measuring and Testing Program of the EC
NAA	neutron activation analysis
NATA	National Association of Testing Authorities (Australia)
NBS	National Bureau of Standards (now NIST)
Nd:YAG	neodymium yttrium aluminium garnet (laser)
NEXAFS	near-edge X-ray absorption fine structure
NICI	negative ion chemical ionization
NIR	near infrared (radiation)
NIST	National Institute of Standards and Technology
NMR	nuclear magnetic resonance (spectrometry)
NPD	nitrogen phosphorus detection (detector)
NRA	nuclear reaction analysis
OECD	Organization for Economic Cooperation and Development
OIML	Organisation Internationale de Métrologie Légale
PA	proton affinity
PAA	photon activation analysis
PC	personal computer
PCB	polychlorobiphenyl
PCDD	polychlorodibenzodioxins
PD	plasma desorption
PDF	powder diffraction file
PE	photo electron
PFIA	process flow injection analysis
PFK	perfluorokerosene

PFTBA	perfluorotributylamine
PICI	positive ion chemical ionization
PMT	photomultiplier tube
ppb	parts per billion
ppm	parts per million
ppt	parts per trillion
PSD	position sensitive detector
PTFE	polytetrafluoroethylene
PVD	physical vapor deposition
Q	quadrupole filter
QA	quality assurance
QC	quality control
RBS	Rutherford backscattering spectrometry
REELS	reflection electron energy loss spectrometry
REM	reflection electron microscopy
RHEED	reflection high energy electron diffraction
RIC	reconstructed ion chromatogram
RIMS	resonance ionization mass spectrometry
RM	reference material
RMD	relative mean deviation
RNAA	radiochemical neutron activation analysis
RS	Raman spectrometry
RSC	Royal Society of Chemistry (UK)
RSD	relative standard deviation
SCA	single channel analyzer
SCE	standard calomel electrode
SDM	sulphadimidine
SDS-PAGE	sodium dodecylsulphonate polyacrylamide gel electrophoresis
SEC	size exclusion chromatography
SEM	scanning electron microscopy
SEM	secondary electron multiplier
SERS	surface enhanced Raman scattering
SEXAFS	surface extended X-ray absorption fine structure (spectrometry)
SFC	supercritical fluid chromatography
SHE	standard hydrogen electrode
SI	Système International (d'Unités)
SIM	selected ion monitoring
SIMS	secondary ion mass spectrometry
SIRIS	sputtered initiated resonance ionization mass spectrometry
SNMS	sputtered neutrals mass spectrometry
SOP	standard operating procedure
SPM	scanning probe microscopy
SRM	standard reference material
SRM	selective reaction monotoring
SSMS	spark source mass spectrometry
STM	scanning tunneling microscopy
STS	scanning tunneling spectrometry
TC	Technical Committee (of CEN or ISO)
TCDD	tetrachlorodibenzodioxins
TD	thermodilatometry
TEELS	transmission electron energy loss spectrometry
TG	thermogravimetry
TGA	thermogravimetric analysis
TGS	triglycine sulfate
THEED	transmission high energy electron diffraction
TIC	total ion chromatogram
TIMS	thermo-ionization mass spectrometry
TLC	thin-layer chromatography
TMA	thermomechanical analysis
TMS	tetramethylsilane
TOF	time-of-flight (mass spectrometer)
TSP	thermospray

UHV	ultrahigh vacuum
UPS	ultraviolet photoelectron spectrometry
UQR	upper quartile range
UV	ultraviolet (radiation)
VIM	vocabulaire international de métrologie
VIS	visible (radiation)
VML	vocabulaire de métrologie légale
VOX	volatile organic halogene
WD-XRF	wavelength-dispersive X-ray fluorescence (spectrometry)
WHO	World Health Organization
XAS	X-ray absorption spectroscopy
XPS	X-ray photoelectron spectrometry
XRD	X-ray diffraction
XRF	x-ray fluorescence (spectrometry)
ZAF	Z (for element number) absorption fluorescence