

## Thermal Desorption Technical Support

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#### **Key Words:**

Field and Laboratory Emission Cell (FLEC®), Emissions

#### **Overview**

This paper describes test cells such as the Field and Laboratory Emission Cell (FLEC®) in comparison to small chambers for materials emissions testing and reviews the results of multiple inter-laboratory (round-robin) studies involving both types of test equipment.

The paper begins by providing an introduction to emission cells, describing their typical design/construction and operating parameters. It also summarises the benefits and limitations of chambers/cells and where each could be considered most applicable. Extensive references are included.

A description of emission kinetics is also provided and introduces the reader to the principles behind gas phase mass transfer (external diffusion) and source phase mass transfer (internal diffusion) emission models. These principles help explain how experimental parameters, (such as air velocity, air exchange rate, load factor, etc.) are likely to impact surface emissions rates for dry and wet materials/products.

The paper concludes by comparing the test cell and chamber results from the inter-laboratory studies and showing that the degree of correlation between emission test data from chambers and cells is generally satisfactory (*i.e.* within 25% difference). Possible reasons for any significant differences that were observed are also presented.



**Field and Laboratory Emission Cell (FLEC®)**

#### **Trademarks**

FLEC® is a registered trademark of Chematec, Denmark

# Emission cells and comparison to small chambers for materials emissions testing

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**Abstract** Materials and products for indoor use need to be evaluated for possible release of volatile chemicals in order to ensure a healthy indoor climate. The demand for standardised methods has resulted in several guidelines for emission testing by use of emission cells and small chambers. Both chambers and cells can provide reliable and useful materials emission data. A number of interlaboratory studies show satisfactory comparison between data from cells and chambers, particularly, when the emission process is dominated by internal diffusion within the test specimen. The ease-of-use, high throughput, and relatively low cost of operation of emission cells facilitate quality control at the manufacturing site, in addition to field measurements of surface emissions. Laboratory performance issues and sample inhomogeneity remain two major obstacles to harmonization of emission testing.

## Vergleich von Emissionszellen und Prüfkammern zur Bestimmung des Emissionsverhaltens von Materialien

**Zusammenfassung** Im Hinblick auf ein gesundes Innenraumklima müssen Materialien und Produkte für Innenanwendungen auf die mögliche Freisetzung flüchtiger organischer Verbindungen getestet werden. Dabei hat die Forderung nach standardisierten Methoden maßgeblich zur Entwicklung von Richtlinien für Emissionsprüfungen mittels Emissionszellen und Prüfkammern beigetragen. Sowohl Kammern als auch Zellen können nützliche und zuverlässige Emissionsdaten liefern. Vergleichsstudien zeigten in vielen Fällen eine befriedigende Übereinstimmung zwischen den Ergebnissen von Emissionsprüfungen mit Kammern und Zellen, insbesondere, wenn der Emissionsprozess durch Diffusion im Material kinetisch bestimmt wird. Die leichte Handhabung, der hohe Probendurchsatz und die relativ geringen Kosten lassen den Einsatz von Emissionszellen besonders im Herstellungsprozess bei der Qualitätskontrolle als geeignet erscheinen. Ein weiterer Vorteil ist die Möglichkeit von zerstörungsfreien Emissionsmessungen vor Ort. Die unterschiedlichen Arbeitsabläufe in Testlaboratorien und die Inhomogenität von Materialien sind zwei wesentliche Punkte, die sich auf die Vergleichbarkeit von Emissionsmessungen nachteilig auswirken.

### 1 Introduction to emission cells

Emission cells are small portable devices for the determination of volatile organic compounds (VOCs) and semi volatile organic compounds (SVOCs) emitted from indoor materials/

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products. They differ from small chambers inasmuch as they have one open wall or "face" which is placed onto the planar surface of the material under test such that the material surface effectively becomes part of the emission cell. The air inlet of the emission cell is designed such that the flow of air is directed over the entire surface of the test specimen before exiting the cell through a central exhaust point.

The emission cell concept was pioneered in 1991 by a Scandinavian team. Their objective was to address the need for a small, versatile and easy-to-use tool for both non-destructive on-site (field) measurements of surface emissions and laboratory emissions tests [1; 2]. A schematic view of the Field and Laboratory Emission Cell (FLEC) which resulted from this project is shown in Figure 1. It is circular and con-

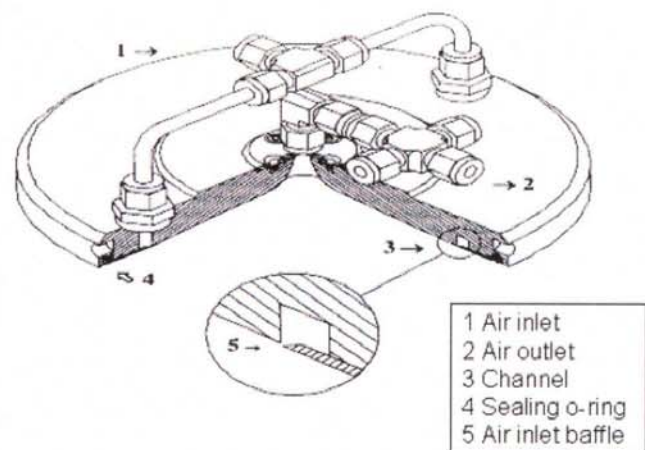


Figure 1. Schematic view of the Field and Laboratory Emission Cell (FLEC).

structed of polished, acid-resistant stainless steel. Air enters from a baffle around the perimeter (see inset). Few other types of emission cells have been reported to date. Kayser et al. [3] describe a device with rectangular shape and laminar air flow. Uhde et al. [4] have coupled a self-constructed emission cell with an online detection system.

Emission cells can be used for materials/products with a planar surface. Typical applications include flooring materials (wood-block, carpeting, vinyl flooring etc.), wood-based panels, sealants, textiles, adhesives, paints, coatings, plastic beads, concrete levelling compounds, wall paper, plastic sheeting, and structural foams. Cells can be placed directly onto rigid products/surfaces and are held in place by the weight of the cell itself compressing the sealing gasket or o-ring. In the laboratory, compressible products are placed into a test specimen holder, such that the weight of the cell rests on the rim of the holder and does not compress the product itself. This ensures that, whether the material is compressible or rigid, a planar sample surface is always presented to the cell at the right height – i. e. without bulging up into the cell and impacting the internal volume or other parameters (for examples see [2]). The application of FLECs in

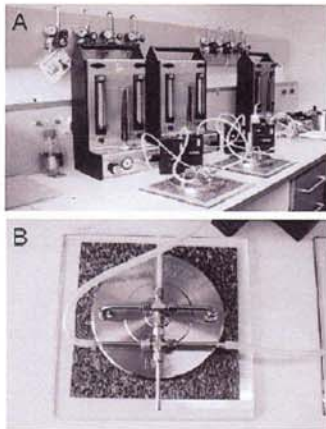


Figure 2. Application of the FLEC in the laboratory for emission testing of vinyl flooring. A: Experimental setup with air supply unit; B: Top view of the experiment. Photos: Wolkoff

the laboratory for testing of flooring materials is shown in Figure 2.

The high cost and relative complexity of small to large test chamber investigations renders them less suitable for routine quality control of emissions in most industrial labs. Moreover, appropriately equipped/experienced thirdparty test laboratories cannot usually offer sufficiently fast turn-around to provide a convenient alternative – i. e. results from a third party lab are unlikely to arrive back with the manufacturer until after the material under test has left production, at which point it would be too late to address any issues identified by the tests. It is therefore desirable to have a more simple measuring system, such as an emission cell that can be used by industry to carry out meaningful emission testing and quality assurance on site [5].

Field application of emission cells also allows emissions from the surface of suspect building products to be identified and quantified during use – i. e. after installation. Relevant information can then be fed back to the manufacturer as quickly as possible thus minimising the need and cost of recalls or other corrective actions. A comparison of the technical parameters of emission cells (FLEC) and emission chambers is presented in Table 1.

## 2 Harnessing the versatility of chambers and cells for studies of physical processes

Due to their flexibility, small chambers (20 l) and emission cells offer a variety of useful experimental configurations. For example, *Jann et al.* [6; 7] have used small chambers connected in series to study the emission of biocides from treated wood. *Meininghaus and Uhde* [8] have combined a 20 l chamber with a FLEC to observe the diffusion of a VOC mixture through a test specimen and to determine diffusion coefficients. *Meininghaus et al.* [9] have installed two FLECs face-to-face, separated by the test specimen to be tested. This configuration allowed for a quick screening of the sorption capacity and permeability of indoor materials. A similar test was also performed by *Meininghaus et al.* [10] using CLIMPAQ-type chambers. *Clausen et al.* [11] have combined FLECs to study the emission of phthalates from vinyl flooring. In this case, sorption experiments were carried out by connecting two FLECs in series and by connecting two FLECs face-to-face (double FLEC).

## 3 Dynamics of emission kinetics

It is well-known that building products are sources of VOC and SVOC emissions. However, the important questions are:

- what specific pollutants are emitted that effect occupants (i. e. health and annoyance, e. g. odour);
- how their emission profiles change over time (and associated modelled indoor concentrations) and
- are the emission test results meaningful – i. e. are the results independent of test conditions?

In regards to point (c), a primary objective of emission testing with either chambers or cells is measurement of a specific emission rate ( $\mu\text{g}\cdot\text{m}^{-2}\text{h}^{-1}$ ) at a given time for the material. However, the test method and equipment may interact with the test specimen and influence the result.

Table 1. Emission cell (FLEC) vs. small chamber – Comparison of technical parameters and discussion of performance data. (SER: specific emission rate,  $k_g$ : gas-phase mass transfer coefficient,  $k_s$ : source-phase mass transfer coefficient)

Parameter	FLEC	Chamber ( $\leq 1 \text{ m}^3$ )	Impact of difference
air change rate in $\text{h}^{-1}$	~ 200 to 600	typically 0.5 to 1.0	not significant, realistic air velocities
air supply in l/min	0.2 to 1.0	0.5 to 20	> 500 l chambers more expensive to run than cells or smaller chambers
air velocity in m/s	0.01 to 0.1	> 0.1 to 0.3	different air velocities result in different SERs for external diffusion emissions ( $k_g \gg k_s$ )
distribution of air	constant, but uneven distribution	uneven, varies with sample orientation	for cells and the smallest chambers: multi-tests are needed for mats with point sources
sample area	177 $\text{cm}^2$	depends on loading	cells more likely to require repeat tests on inhomogeneous materials
loading in $\text{m}^2/\text{m}^3$	510	0.5 to 1	counter balances difference in exchange rate
volume	35 $\text{cm}^3$	20 l to 1 (5) $\text{m}^3$	no still air in cell – minimises sink effects
equilibration time	minutes to hours	hours to days	cells allow higher experimental throughput

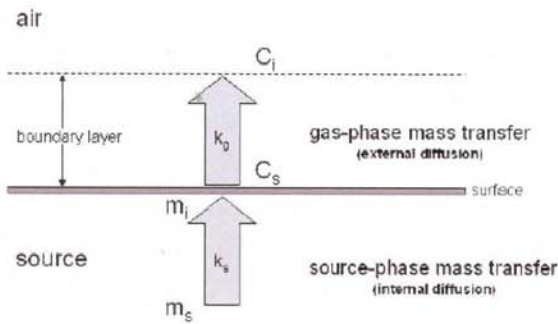


Figure 3. Diagram of the kinetic processes involved in the mass transfer model of VOCs from material surfaces (adopted from Sparks et al. [13]).

The dynamics of emission processes from building product surfaces has been studied in detail. A number of physical and empirical models of different complexity have been described [12]. The emission can be characterized by two fundamental physical processes (see Figure 3) [13]:

- Gas-phase mass transfer (i. e. external diffusion),
- Source-phase mass transfer (i. e. internal diffusion).

The gas-phase mass transfer model (a) is based on molecular diffusion across a laminar boundary layer as described in Eq. (1).

$$SER = \frac{D}{\delta} \cdot (C_s - C_i) = k_g \cdot (C_s - C_i) \quad (1)$$

$SER$  is the specific emission rate,  $D$  is the diffusion coefficient,  $\delta$  is the thickness of the boundary layer,  $C_s$  is the concentration of the target VOC at the source surface,  $C_i$  is the concentration of the target VOC in the air and  $k_g$  is the gas-phase mass transfer coefficient.

Process (b) is limited by diffusion from the interior of the source to the surface and can be described by Eq. (2).

$$SER = k_s \cdot (m_s - m_i) \quad (2)$$

Here,  $m_s$  is the mass of the target VOC in the source,  $m_i$  is the mass of the target VOC at the surface and  $k_s$  is the source-phase mass transfer coefficient.

In terms of the comparability of emissions test results obtained from different chambers and cells, three different scenarios have to be considered. For  $k_g \gg k_s$  the emission is controlled by the external diffusion process and the thickness of the boundary layer  $\delta$  is directly related to the air velocity above the surface. This applies to most wet-applied or liquid products during the drying/curing phase. In this case the air flow conditions in the test facility might influence the test result. This drying/curing phase of wet-applied or liquid products typically lasts between one and 14 days. For  $k_g \ll k_s$  the emission is controlled by the internal diffusion process and the influence of the air flow condition in the test facility should be negligible. This applies to most materials manufactured in the solid-phase and to wet applied or liquid products after they have dried or cured. A more difficult situation arises for  $k_g \approx k_s$  or if the ratio  $k_s/k_g$  changes over time. For an ageing product,  $k_s/k_g$  will normally reach infinity over time. For discussion of comparability of different experimental set-ups see Section 4.

Sink effects are another analytical variable that can significantly influence emissions test results. Emissions test equipment will invariably have an inherent sink (wall) effect, the

nature and extent of which will depend upon the chamber/cell properties and on the physical properties of the emitted compounds (e. g. polarity and volatility). Sink effects and recovery measurements are required by most relevant standard methods [14 to 16], but may still not be adequately or mathematically taken into account in routine testing and modelling. To study sink effects in chambers, standard recovery experiments (e. g. using permeation or diffusion-controlled sources of target VOCs within the chamber or cell) can be supplemented by "thermally desorbing" the empty chamber, after use – i. e. using elevated temperatures to desorb any organic compounds which have condensed on the chamber surfaces and collecting the vapours on a Tenax tube [17]. Larger chambers (1 m<sup>3</sup>) also offer the possibility of fogging experiments [18]. Emission cells are designed to minimise sink effects (see below) and can be assessed for recovery using standard tests and by using post-test thermal desorption as above. In addition, the smooth internal surface of the FLEC cell can be easily rinsed with a suitable solvent and the extract analysed.

#### 4 General discussion of emission cells in comparison to small chambers

Emission cells like the FLEC cannot be used for sculpted or moulded materials nor for whole objects (e. g. cell phones). They are limited to planar materials or to those products which can be made to present a relatively planar surface to the cell. The air velocity over the sample surface does vary from point to point [19; 20], but this is also the case in small chambers [21]. However, specific emission rates controlled by internal diffusion are broadly independent of surface velocity, so both cells and small chambers can produce meaningful, reproducible and comparable data despite the uneven distribution [20]. The variability of surface air velocity may mean that equilibration times for highly textured surfaces in emission cells need to be extended from 15 to 20 minutes to ~2 hours, but similar issues also affect small chambers.

The only real concern relating to non-uniform air velocity for internal diffusion controlled emissions relates to materials with strong point-source emissions such as knot-holes in wood [19; 22]. In these cases multiple repeat tests would be advised with either small chambers or cells.

Though uneven, the pattern of air velocity distribution within a FLEC cell is reproducible at different flow rates [19]. This allows it to be used for comparing emissions from external diffusion controlled systems, but encumbers comparison with test data for the same materials obtained using small chambers – see later under performance data.

The relatively small size of most emission cells (that shown in Figure 1 only allows 177 cm<sup>2</sup> area of test specimen to be exposed) means that multiple tests are required on (highly) inhomogeneous or jointed materials. Depending on size, this is less likely to be an issue with small chambers – especially those that are > 100 l. Within the restrictions/limitations described above, emission cells provide a simple and effective tool for testing emissions from many common materials/products. For example; because the emission cell is simply placed directly onto most products, it eliminates sample orientation and edge sealing issues. The features of emission cells like the FLEC are well-documented in the literature and include:

Table 2. Parallel emissions testing with emission chambers and the FLEC.  
(ss = stainless steel)

Chamber size/type – Duration/time of testing	Building product	Compound	Comments	Ref.
250 l ss to 3 weeks	vinyl flooring	cyclohexanone, phenol, TXIB	satisfactory correlation between different FLECs and one chamber – some apparent material inhomogeneity	[1]
50 l ss to 2 weeks	paint, wax	ethylene glycol, Texanol, TVOC	satisfactory correlation, especially after ~ 50 hrs; note: FLEC SERs generally higher (possibly due to min. sink effects)	[30]
1 m <sup>3</sup> ss or glass to 1 week	wood	terpenes	satisfactory correlation with glass chamber in one test; apparent inhomogeneity in another test and/or domination of external diffusion emission (velocity dependent); note: FLEC SERs generally higher	[31]
34 m <sup>3</sup> wood walls, PVC floor to 20 hours	floor polish	2-(2-ethoxyethoxy)ethanol	modeled (FLEC) and measured peak concentrations (34 m <sup>3</sup> ) appeared after 30 and 100 min, respectively and differed by a factor of 2	[32]
1 m <sup>3</sup> , 187 l ss to months and office air to months	linoleum floor	hexanal	unsatisfactory correlation; higher SER in chambers, partly assigned to edge effects; satisfactory correlation between measured hexanal SER in office and FLEC	[33]
1 m <sup>3</sup> ss to ca. 13 days	wood based products	formaldehyde	satisfactory correlation (correlation coefficient = 0.98)	[34]
European round-robin (18 labs.) up to 1 m <sup>3</sup> ss/glass to 48 hours	carpet PVC paint	VOCs	main causes of discrepancies were: i) analytical errors, ii) sorption on walls, iii) inhomogeneity of the building products. Also variable film thicknesses of paint were used <i>Note: 28 hours may not be sufficient to reach equilibrium</i>	[28]
20 m <sup>3</sup> , 1 m <sup>3</sup> and 20 l ss to 28 days	UV-cured lacquers	TVOC	FLEC time/concentration profile was highest during first 10 days, thereafter super-impossible with that for 20 l to 20 m <sup>3</sup> . 1 m <sup>3</sup> chambers showed lowest time/concentration profiles	[35]
German round-robin (9 labs) up to 1 m <sup>3</sup> ss to 28 days	UV-cured acrylic lacquer	sum of VOCs and SVOCs and individual VOCs	observed differences ≤ 15 %	[26; 27]
ADSEC (stainless steel)	wood based	formaldehyde	satisfactory correlation (> 0.99)	[36]
1 m <sup>3</sup> ss, (51 l glass) to 24 hours	paint on steel plate	higher aldehydes, decanol	satisfactory comparison (variation ≤ 15 %)	[37]
round-robin (8 labs.) to up to 200 days	lacquer on MDF	VOCs	recovery generally better than 90 % in FLEC; satisfactory analyses of spiked tubes; apparent material inhomogeneity	[29]

ss = stainless steel, TXIB = texanol di-isobutyrate, MDF = medium density fibreboard

- Minimal sink effects and > 90 % recovery of VOCs [2; 23]. While there is little specific data on recoveries from small chambers in the published literature, chambers are typically expected to offer > 80 % recovery of VOCs. The internal geometry of emission cells means that there are no volumes of still air which helps minimise sink effects and optimise recovery.

- Tests are more rapid – typically 15 to 30 minutes equilibration for smooth surfaces and 15 to 20 minutes vapour collection compared with several (up to 24) hours for small chambers and days for large chamber tests.

- The relatively small size and low air flow (< 1 l/min) of emission cells mean that parameters are easy to control or change (e. g. temperature, humidity, e. g. see [24]). They are also readily reproduced and convenient to monitor. In this respect emission cells are not dissimilar to the smallest test chambers, but chambers (> 100 l) do require more careful control.

- Minimal cleaning is required between tests and cells are easy-to-clean when needed.

- Cells facilitate field use, testing of composite sample testing and testing at elevated temperature, relative humidity, or enriched ozone [25].

For all the above reasons, emission cells facilitate quality control at the manufacturing site.

VOC emissions controlled by internal diffusion (dry products/materials) are largely independent of surface air velocity, provided the rate is fast enough to prevent build up of contaminants at the test specimen surface. Table 2 presents a summary of the results from interlaboratory (round robin-type) studies of emissions for a range of material types. Correlation between data from chambers and cells is generally satisfactory (i. e. within 25 % difference), especially for dry products where the dominating emission process is internal diffusion. For example, Jann et al. [26; 27] have studied VOC emissions from UV-cured lacquer in different chambers and in the FLEC

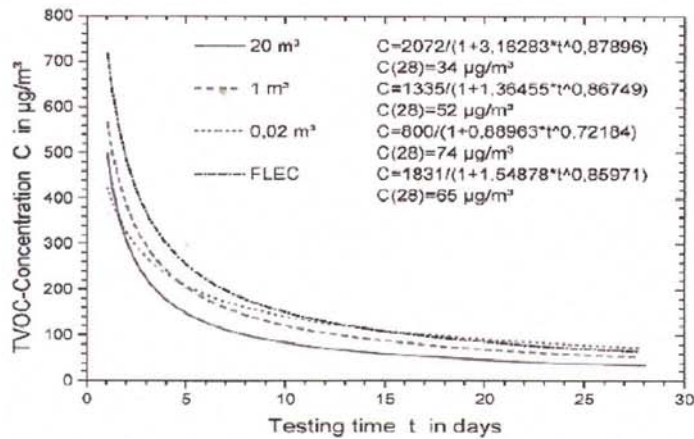


Figure 4. Emission of VOCs from a UV-cured lacquer applied on solid wood (alder) – comparison of 20 m<sup>3</sup> chamber, 1 m<sup>3</sup> chamber, 20 l chamber and FLEC. Figure reproduced with permission of Dr. Oliver Jann, BAM, Berlin [27].

and report a good comparability of the results. The time vs. concentration curves over 28 days are presented in Figure 4. However, in the case of drying or curing products, the primary emission process is normally external diffusion (i. e.  $k_g \gg k_s$ ), which is significantly affected by both surface air velocity and the sample loading factor ( $m^2 \cdot m^{-3}$ ) (and associated vapour concentration within the chamber/cell). The exact timing of emissions testing and selection of specimen storage conditions prior to testing are also critical for wet samples. Although emission test data for wet samples obtained using an emission cell such as FLEC or any given type of chamber can thus be made to be reproducible by applying rigorous control of all parameters prior to and during testing, the extreme sensitivity of the results to such a multitude of variables, does call into the question the validity of testing wet samples during the curing or drying stage. Discrepancies between chambers and cells have been observed in round-robin studies, but this is true for all interlaboratory comparisons of emissions testing (whether carried out using emission cells, small chambers, or a mixture of the two) and is not primarily caused by differences in chamber/cell design – Laboratory performance and material inhomogeneity currently appear to be the most significant factors [28; 29].

## 5 Conclusions

This article has highlighted the fact that the frequently asked question, "...is the emission cell comparable to the test chamber..." is deceptive and could equally be asked of two different sizes/formats of emission chambers or emission cells. The fact is that both chamber and cell can provide reliable emission data under well-controlled test conditions. Extensive field experience of the FLEC emission cell since its introduction has shown that the relative ease, with which the key parameters can be controlled/reproduced, is one of the main reasons, in addition to speed, that make it suitable for use as a routine industrial quality assurance tool. Experience gained during the interlaboratory studies described, has shown the FLEC emission cell to be suitable for many emissions testing applications and to be a useful supplement to small chambers. Acceptance of emission cells is now reflected by their inclusion by CEN into EN 13419-2 as

a "horizontal" standard (multi-product/application) for use by industry and service laboratories in compliance with the European Construction Products Directive. Similar draft standards are currently proceeding through the latter stages of balloting in both ASTM and ISO (DIS 16000-10). However, additional, well-regulated intercomparative testing of various small chambers and test cells, under controlled/harmonised conditions of analytical method, VOC recovery and specified homogeneity of the test specimen would make an important contribution to our understanding of chamber/cell characteristics. Such an activity should be strongly supported.

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## Buchbesprechung

**The Art of Scientific Writing.** Von Hans F. Ebel, Claus Bliefert und William E. Russey. 2. Aufl. Weinheim: Wiley-VCH 2004, XII + 595 S. Preis: 34,90 €.

Lernen Sie Schreiben in drei Wochen – wer hat sie nicht schon einmal gesehen, diese Anzeigen für Kurse, die zu belletristischen Erfolgen führen sollen. Ob dieser Erfolg sich dann tatsächlich einstellt, ist wohl leider nicht in jedem Fall gesichert. Dass aber im wissenschaftlichen und technischen Bereich Schreiben eine Kunst ist, die man auch erlernen kann, zeigt der vorliegende, jetzt neu aufgelegte Band. Seit seiner ersten Auflage 1987 haben die elektronischen Medien das Publikationswesen grundlegend verändert. Dementsprechend haben die Autoren, die alle aus dem Bereich der Chemie stammen, ihre grundlegende Darstellung des Ablaufs und nahezu aller Aspekte wissenschaftlichen Publizierens um Themen wie Onlinemedien und Software aktualisiert.

Das Buch gliedert sich im ersten Teil nach den verschiedenen Typen von Publikationen: Report, Dissertation, Zeit-

schriftenbeitrag und Monografie. Dabei wird neben der allgemeinen Darstellung des Mediums viel Wert auf praxisgerechte Hinweise zur Manuskripterstellung gelegt. Besonders der Dialogcharakter einer Veröffentlichung wird herausgestrichen: Nicht nur die Autorensicht ist wichtig, der Leser ist ja das Ziel der Publikation, das der Autor immer im Auge haben sollte. Im zweiten Teil werden als Komponenten einer Veröffentlichung detailliert Text, Formeln, Abbildungen, Tabellen und Literaturhinweise beschrieben. Die Autoren weisen hier ausführlich auf den heute selbstverständlichen Softwareeinsatz hin und geben dazu Tipps und Übersichten. Insgesamt bietet das Buch für Studierende eine gründliche Einführung in Hintergründe und Methodik des Publizierens mit vielen Praxistipps. Gleichzeitig gibt es aber auch erfahrenen Autoren viele nützliche Anregungen in einer umfassenden Zusammenstellung. Wer also Publizieren nicht nur als lästige Verpflichtung empfinden möchte, dem kann das vorliegende Buch nur empfohlen werden.

Dr. rer. nat. Ralf Michaelis