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Review

Life cycle management of analytical methods

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ABSTRACT

In modern process management, the life cycle concept gains more and more importance. It focusses on the total costs of the process from invest to operation and finally retirement. Also for analytical procedures an increasing interest for this concept exists in the recent years. The life cycle of an analytical method consists of design, development, validation (including instrumental qualification, continuous method performance verification and method transfer) and finally retirement of the method. It appears, that also regulatory bodies have increased their awareness on life cycle management for analytical methods. Thus, the International Council for Harmonisation of Technical Requirements for Pharmaceuticals for Human Use (ICH), as well as the United States Pharmacopeial Forum discuss the enrollment of new guidelines that include life cycle management of analytical methods. The US Pharmacopeia (USP) Validation and Verification expert panel already proposed a new General Chapter ⟨1220⟩ *"The Analytical Procedure Life-cycle"* for integration into USP. Furthermore, also in the non-regulated environment a growing interest on life cycle management is seen. Quality-by-design based method development results in increased method robustness. Thereby a decreased effort is needed for method performance verification, and post-approval changes as well as minimized risk of method related out-of-specification results. This strongly contributes to reduced costs of the method during its life cycle.

Contents

Abbreviations: AMMS, accurate mass spectrometry; AQbD, analytical quality-by-design; ASME, American Society of Mechanical Engineers; ATP, analytical target profile; CQA, critical quality attributes; DoE, design of experiments; DQ, design qualification; DS, design space; FDA, US Food and Drug Administration; FMEA, failure mode effect analysis; GMP, good manufacturing practice; HILIC, hydrophilic interaction liquid chromatography; HPLC, high performance liquid chromatography; ICH, International Council for Harmonisation of Technical Requirements for Pharmaceuticals for Human Use; ISO, International Organization for Standardization; IQ, installation qualification; LLOQ, lower limit of quantification; LOD, limit of detection; MODR, method operable design region; MS/MS, tandem mass spectrometry; NIR, near infrared (ν = 13,000–4,000 cm^{-1}, 800–2,500 nm); OFAT, one-factor-at-a-time; OQ, operational qualification; Ph.Eur., European Pharmacopoeia; PM, prioritization matrix; PQ, performance qualification; PQRI, Product Quality Research Institute; QbD, quality-by-design; QRM, quality risk management; QSAR, quantitative structure activity relationship; QSPR, quantitative structure property relationship; QSRR, quantitative structure retention relationship; QTPP, quality target product profile; RP, reversed phase; TMU, target measurement uncertainty; UHPLC, ultra high performance liquid chromatography; ULOQ, upper limit of quantification; URS, user requirement specification; USP, United States Pharmacopeia.

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1. Introduction

Analytical methods are used to generate data in various fields of application. As these data are used as basis for decisions, their validity is extremely important. Thus, methods need to be able to provide these data in constant quality. According to a database search at Web-of-Science for "life cycle management" in topic, the concept of life cycle management was mentioned in 1975 for the first time [1] in scientific literature. Therein, Baglow, affiliated at the Canadian National Defense Headquaters, refers to life cycle management with focus of total costs of ownership and operation of an equipment or system. In the following years, increasing interest is observed resulting in growing numbers of scientific publications as illustrated in Fig. 1. In total, the search at Web-of-Science yielded 940 manuscripts. In our review we focus on methods in the pharmaceutical context and therefore on methods of chemical analyses. The search using the term "life cycle management" and "pharma*" yielded 58 articles, with the first publication dated in 2003 (Fig. 1). Refining the database search on "life cycle management" to the Web-of-Science category "Chemistry Analytical" 14 articles are identified in the database.

The transfer of the life cycle concept to analytical methods is illustrated in Fig. 2. It includes quality-by-design (QbD) approaches in method development, validation and operational use and may be considered as link between method development and method validation [2,3].

Up to now, four stimuli articles regarding the analytical life cycle have been published by the United States Pharmacopeial Forum [4–7]:

- "Lifecycle Management of Analytical Procedures: Method Development, Procedure Performance Qualification, and Procedure Performance Verification"
- "Fitness for Use: Decision Rules and Target Measurement Uncertainty"
- "Analytical Control Strategy"

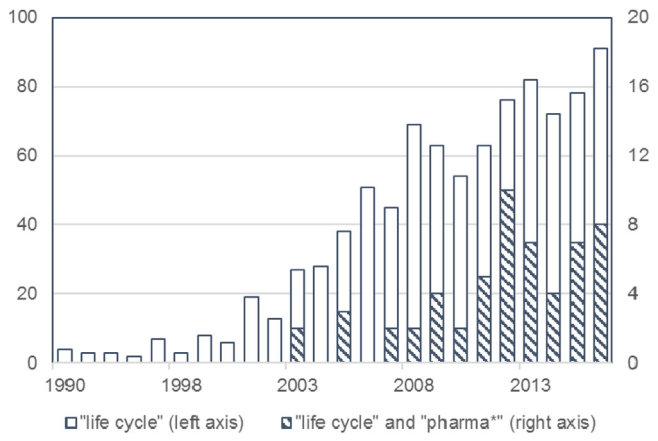

Fig. 1. Number of citations 1990–2016 according to Web-of-Science, 2017/04/25, termed used in search "life cycle" (white bars) or "life cycle" and "pharma*" (hatched bars).

- "Analytical Target Profile: Structure and Application Throughout The Analytical Lifecycle".

Only recently, the US Pharmacopeia (USP) Validation and Verification expert panel proposed a new General Chapter ⟨1220⟩ *"The Analytical Procedure Lifecycle"* for integration into USP [8]. In this process the transfer of modern concepts of a life cycle model, based on process validation (US Food and Drug Administration (FDA) Guidance for Industry: Process Validation, and International Council for Harmonisation of Technical Requirements for Pharmaceuticals for Human Use (ICH) guidelines Q8, Q9, Q10, Q11 and Q12 [9–14]), to analytical methods was discussed. A comprehensive view on the method and its risk management is integrated to ensure valid data generation and improved quality of the method throughout all stages of the method.

Application of life cycle management concepts to analytical procedures provides an opportunity to use the knowledge gained from the application of scientific and quality risk management to continuous improvement and assurance of data quality. Analytical method life cycle management combines activities of analytical method development, improvement, qualification, validation, transfer and maintenance related to Good Manufacturing Practice (GMP) production [15].

The life cycle approach for an analytical procedure is an extension of the current guidelines, taking advantage of the QbD approach [6].

2. Analytical target profile (ATP)

As stated by Martin et al. in their stimulus article [8] "*a fundamental component of the lifecycle approach to analytical procedures is having a predefined objective that stipulates the performance requirement for the analytical procedure. These requirements are described in the ATP*".

The ATP may be seen as a reference point of the life cycle approach of an analytical method as it is already mentioned as "set analytical requirement" in the Eurachem Guide [16] since 1998 (Fig. 3). It is comparable to the quality target product profile (QTPP), which is defined in ICH Q8 [12], but transferred to analytical methods, or the critical quality attributes (CQA) as mentioned in ICH Q5E [17] for biotechnological products. The ATP is a predefined written record of the requirements of an analytical method. It should be established prior to method development and be linked to the purpose of the method, not to a specific analytical technique. That implies that any analytical procedure that conforms to the ATP is acceptable [18].

The ATP criteria should be based on the intended use of the analytical method. Customer specifications or regulatory requirements and guidelines may be used as basis for the ATP. If no external requirements are preset, the laboratory should select appropriate methods (ISO/IEC 17025, 5.4.2). In case of quantitative methods the ATP is very often based on the target measurement uncertainty (TMU), which is the maximum acceptable uncertainty in the reportable result that must be achieved by the method in order to make decisions with confidence [4,19]. TMU may also be transferred to qualitative methods as decision limit and detection capability [20].

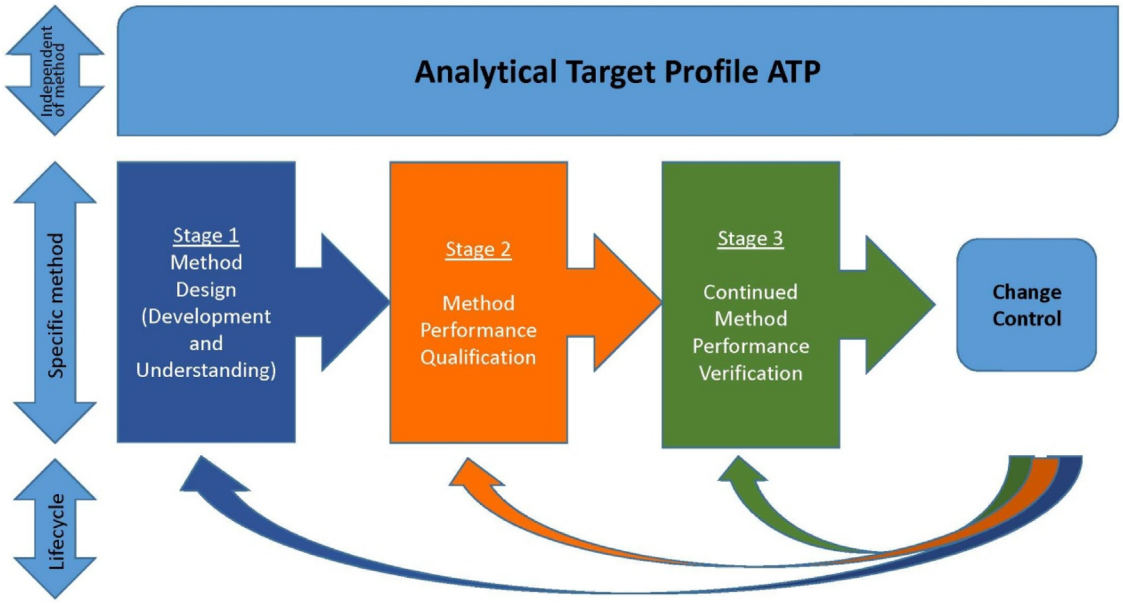

Fig. 2. Life cycle management in analytical methods.

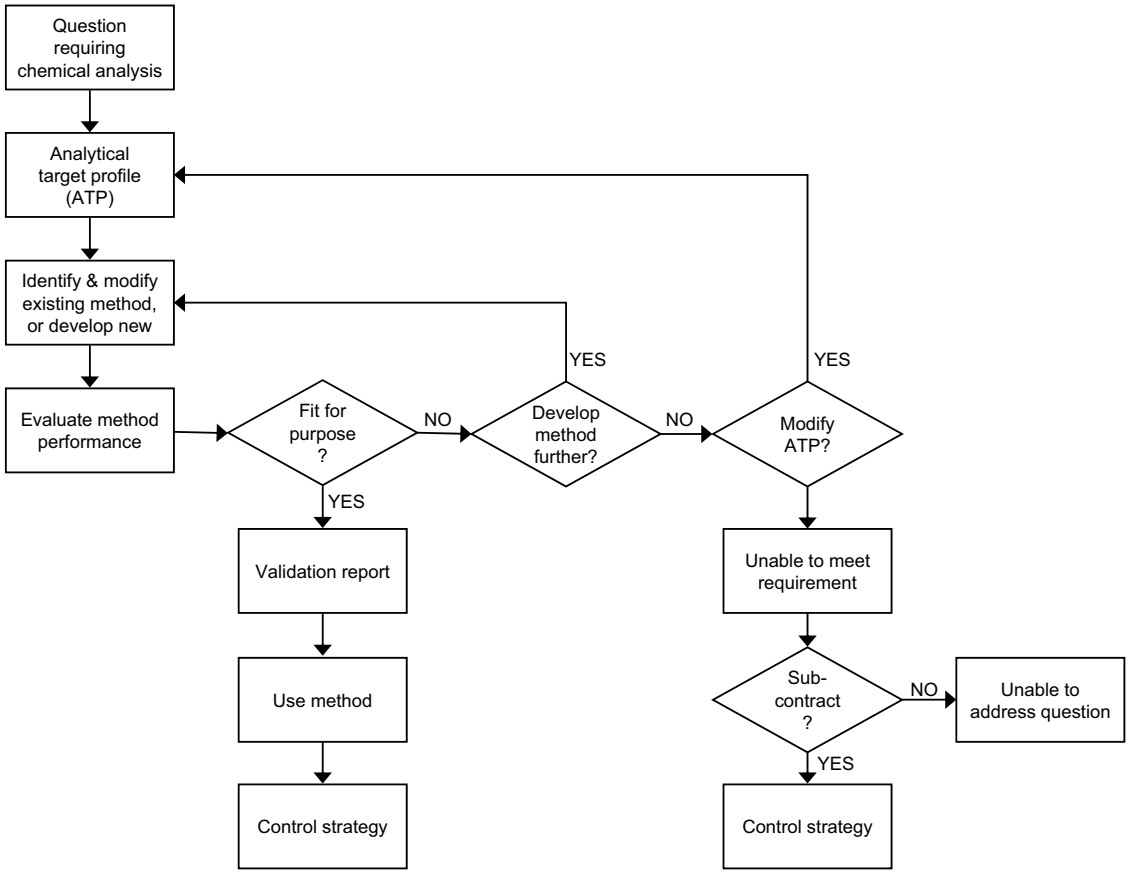

Fig. 3. Method development and validation process based on analytical need described in the analytical target profile (ATP), modified from [16].

Therefore, key to the assessment of compliance is the concept of "decision rules". These rules give a prescription for the acceptance or rejection of a product based on the measured quantity value, its uncertainty and the specification limit or limits, taking into account the acceptable level of the probability of making a wrong decision [4,19]. The concept of decision rules is also described in consensus standard documents such as the "Guideline for Decision Rules" of the American Society of Mechanical Engineers (ASME), the Eurachem Guide "Use of Uncertainty Information in Compliance Assessment", and "Guide to the Expression of Uncertainty in Measurement (GUM)" of the International Organization for Standardization (ISO) [21–23].

Depending on the intended use of the method, typical requirements of a method may include the performance criteria accuracy,

precision, selectivity, sensitivity, linearity, and/or robustness, but also sample turn-around time, throughput capacity, total costs for analysis and ease of operations.

As one example of an ATP for an impurity method of a drug product, Barnett et al. [6] proposed an accuracy of 100.0% ± 3.0% and a precision of ≤1.0% based on the requirement to accurately quantify the drug substance in the presence of impurities and excipients. Once the ATP has been defined, an analytical technique that is most likely capable of delivering analytical data/results compliant to the ATP needs to be selected based on the analyst's knowledge. Subsequently the life cycle may be continued with method design and a risk assessment.

As prerequisite the ATP should be considered in all stages of the analytical method's life cycle as outlined in Fig. 2 [6].

3. Method design

The next step in the life cycle of an analytical method is related to the design of a method. For successful method development, it is important to understand method fundamentals. Sound knowledge of the key variables and how they may influence the analysis is required [62]. Furthermore, it is important to consider all aspects in the development stage, including sample preparation as well as preparation of reference solutions [24] to ensure that the final method is robust and fit-for-purpose. Thus, method design will not only consider the ATP but also the capabilities and educational status of the analytical laboratory. Together with the following topic, method development, method design is considered as stage 1 of the life cycle by Martin el al. [8].

4. Method development

Strategies in method development are strongly influenced by the type of analyses chosen in the previous decision process of method design. A similar effort as for the development of the analytical method should be spend on the development of the sample preparation procedure. Suitable sample preparation protocols for complex samples are often crucial for the outcome of the full method. When too little effort is taken, poor or irreproducible recovery may be observed as well as method robustness problems, increased effort for method transfer, or even shortened instrument lifetime [24,25]. Most of the analytical techniques require homogenous solutions of the analyte, thus dissolution of solid samples is necessary. Only a few techniques such as Raman or near infrared (NIR) spectroscopy allow for direct analysis of solid samples [26,27]. Further steps of sample preparation may be intended to reduce matrix components, and increase selectivity of the full method. Bioanalytical samples often require further preconcentration of the analytes or sometimes also cleavage of conjugates prior to analyses. Common sample preparation steps include (supported) liquid-liquid or solid phase extraction, immune purification, hydrolysis, and sometimes derivatization of the analytes is required prior to analysis in addition (e.g. for inclusion of a fluorophore etc.). A summary of different sample preparation techniques for pharmaceutical products and bioanalytical procedures can be found in the literature [24,28–40].

As high performance liquid chromatography (HPLC) based methods are considered as most relevant analytical techniques in current pharmaceutical applications, they are used within this article to exemplify different possibilities. A transfer to other techniques may be done based on analogies.

4.1. Instrumentation for straight forward method development

There are several components of a HPLC system that are used to facilitate method development [41]. HPLC systems can be modular or integrated, and utilize either isocratic or gradient solvent delivery. As mobile phase and column screening is a key step in method development, systems used for convenient investigation of starting conditions for further method optimization consist of gradient solvent delivery manager, solvent switching valves, column oven with valve for multiple column selection, an automatic sample manager, and if possible multiple detector capabilities including photo-diode array and mass spectrometry.

This system is capable of delivering mobiles phases (usually 3 organic solvents and 6 buffers with different pH values) consisting of different polarities, pHs and four or more columns operated at different temperatures [42]. Additionally different detectors may help to identify the analytes and to evaluate the best choice in the light of the ATP. Mass spectrometric detection may be used to facilitate peak tracking even if not considered as detector for the finally provided method.

Equivalent and/or orthogonal columns with respect to any selected column can be evaluated by using the hydrophobic subtraction model (Product Quality Research Institute (PQRI) approach) as proposed by Snyder et al. [43]. Over 650 columns have been tested, characterized and added to the database [44]. This database is available on the USP website [http://www.usp.org/pqri-approach-column-equiv-tool, accessed 17/04/24].

In general, HPLC systems have mainly remained unchanged for at least 30 years. The most remarkable improvement was achieved by the introduction of ultra-high performance liquid chromatography (UHPLC) systems. Due to their potential of an operation at a pressure up to 20,000 psi (1,300 bar), these systems allow for use of columns packed with sub–2 μm particles [45–56]. Even if rarely operated at real ultra-high pressure in routinely used methods, UHPLC systems are considered as the standard equipment for the method development laboratory, mainly due to reduced system dispersion and dwell volumes as well as improved precision and sensitivity (10). Furthermore faster analysis times with acceptable resolution, lower solvent consumption and deceased limits of quantification may be achieved [57]. This also meets the request for improved and faster information gain in an economic climate where cost control is a primary concern [58].

4.2. Traditional method development approach

Strategic method development depends on the knowledge and complexity of the sample, the analyst's experience, and intuition, availability of materials such as columns and solvents, as well as the goals of the separation. An identification of critical method parameters is required to focus on relevant experiments. In the past, choosing conditions for a final separation (method development) was often carried out by a trial-and-error approach [59], for example by varying one-factor-at-a-time (OFAT) and examine the resolution of peaks until a suitable method was found. This approach is very time-consuming, especially in cases where multiple parameters are identified as crucial for method development. This generally leads to very high numbers of experiments as basis for reasonable method development. Additionally, this approach often results in a non-robust performance ("new" peaks, disappearance of other peaks and changes in critical peak pairs), especially when transferred into another laboratory because interactions between chromatographic parameters (factors) were not considered [60–62].

Hence, the traditional method development strategy has a high risk in method failure (e.g. non-confirmed out-of-specification result) and always requires an extensive revalidation protocol after

method transfer or alternative method development. Thereby it may result in increasing costs of the method [63].

Thus, there is a remarkable desire to develop a chromatographic method in a more systematic approach of screening columns and mobile phase buffers to gain knowledge about the influential parameters and to set the optimized conditions for the separation.

4.3. Systematic method development

A more systematic approach compared to the traditional way uses statistical methods and software assistance as tools for fast and efficient test planning in method development. In the last couple of years lots of research was conducted on systematic method development strategies [41,64–68]. A systematic approach is also recommended by the FDA's "*Pharmaceutical Current Good Manufacturing Practices (cGMPs) for the 21st Century – a Risk Based Approach*" initiative [69]. This was promoted after FDA identified that pharmaceutical manufacturing problems are not fully understood and that the implementation of new state-of-the-art technologies was slower than in other industries [70,71]. The initiative resulted in the development of a series of new guidelines issued by the ICH:

The ICH guidelines Q8 and Q9, both issued in 2005, provide guidance in pharmaceutical development and risk management, while the 2008 issued Q10 guideline describes a holistic and integrated pharmaceutical quality system [10–12]. In 2012 the Q11 guideline [13] on development and manufacture of drug substances was added. These guidelines were intended to modernize the pharmaceutical industries approach for development and manufacturing of pharmaceuticals to a more scientific and risk-based approach [72]. Although the ICH guideline Q8 does not explicitly mention analytical method development, a QbD approach in pharmaceutical development is requested, which may be seen as recommendation to imply QbD for analytical methods as well. QbD, as defined by the revised ICH guideline Q8(R2), is "*a systematic approach to development that begins with predefined objectives and emphasizes product and process understanding and process control, based on sound science and quality risk management*" [12]. QbD is a concept first outlined by Joseph Juran [73], who stated that *"product features and failure rates are largely determined in planning for quality"*. This means that quality must be designed into a product or a process and cannot be tested into it.

The QbD concept can be extended to analytical methods and results in a systematic approach that includes definition of method goals, risk assessment, specification of a design space (DS), implementing a control strategy and continuous improvement to increase method robustness and knowledge (Fig. 4). To distinguish this from the QbD concept for processes, it is often called analytical quality-by-design (AQbD) in recent publications [63,74–83].

AQbD includes an early risk assessment to clearly identify method parameters that have an impact on the performance of the analytical method but also risks associated with variability such as sample preparation, instrument configuration, and environmental conditions [63]. The quality risk management (QRM) process is described in detail in ICH Q9 guideline [11] and comprises of risk assessment, risk control, and risk review. Risk assessment using "Fishbone" (Ishikawa) diagram or failure mode effect analysis (FMEA) and prioritization matrix (PM) may be employed throughout various stages in the development of an analytical method to assess method factors with the highest effect on method performance and define which (if any) require additional investigation [72,84,85].

Using the QbD approach the fundamentals of a systematic method development have not changed. However, there is an increased demand to design adequate quality into the method, e.g. by Design-of-Experiments (DoE) strategies. The introduction of an early risk-assessment helps to identify critical analytical parameters and to concentrate on them in method development [60,86]. A deeper understanding of what we are doing and why we are doing it in the laboratory is required. The idea is to invest more time, consideration and good scientific know-how into the early stages of a method in order to prevent problems later on (e.g. frequently non-confirmed out-of-specification results due to the non-robustness of the method) [6,87].

The novelty and prospect in this approach is that modifications within the Design Space (DS) or Method Operable Design Region (MODR) of a specific method can be seen as an adjustment and not a (post-approval) change [60,85].

4.3.1. Chemometry based method development

In a full or fractional factorial design a set of experiments (DoE) are carried out in which one or more factors are changed at the same time. Using statistical tools the effect of each factor on the separation can be calculated and the data be used to find the optimum conditions in a method. Typical examples are the widespread use of the Plackett-Burman design that accounts for the interdependence of different factors. A multi-step approach is needed as pairs of factors are included in this design, only. As alternative central composite, Rechtschaffen and Box-Behnken design may be applied for method development [82,83,88–95]. Rakic et al. [93] found the central composite design as superior to two-level and three-level full factorial as well as Box-Behnken design in chromatographic method development. They obtained significantly better models with concomitantly reduced numbers of experiments needed for model building.

By using DoE (e.g. full or fractional factorial designs, Plackett-Burman design) the most critical parameters (influencing factors) are optimized simultaneously to assess the effect of the critical parameters individually and in combination. As an example a DoE for three (p) method parameters at two (n) levels leads to 8 ($E = n^p$) experimental runs [18].

The output of the DoE leads to the identification of a region of robust operating conditions, the so-called design space (DS) or method operable design region (MODR) [12,63,72,80,81,96–102].

After choosing a working point within the DS (or MODR) method verification is performed to confirm the ability of the method to meet the requirements of the previously defined method goals (Analytical Target Profile, ATP). Further method validation in compliance to applicable regulations [103] is mandatory.

Chemometry based method development procedures are universal methods and also useful for the development of methods other than reversed phase (RP-)HPLC [75,82,83,94,95,104–112], i.e. other chromatographic, but also electrophoretic, spectroscopic or biochemical methods [113–121].

4.3.2. Modelling software assisted method development

As alternative for method development, software assistance may be used to predict chromatograms at selected conditions [122]. Examples of commercially available solutions especially designed for RP-HPLC method development are DryLab (Molnár-Institute, Berlin, Germany), Fusion LC Method Development (S-Matrix Corporation, Eureka, CA, USA), ChromSword (Dr. Galushko Software Entwicklung GmbH, Muehltal, Germany), ACD/AutoChrom, or ACD/LC simulator (both Advanced Chemistry Development, Inc.,Toronto, Canada). Based on a small number of experiments these software applications can predict the movement of peaks in reversed-phase liquid chromatography separation when changing the mobile phase composition, pH, temperature, flow rate, or column dimensions and particle size [60,96,123–129].

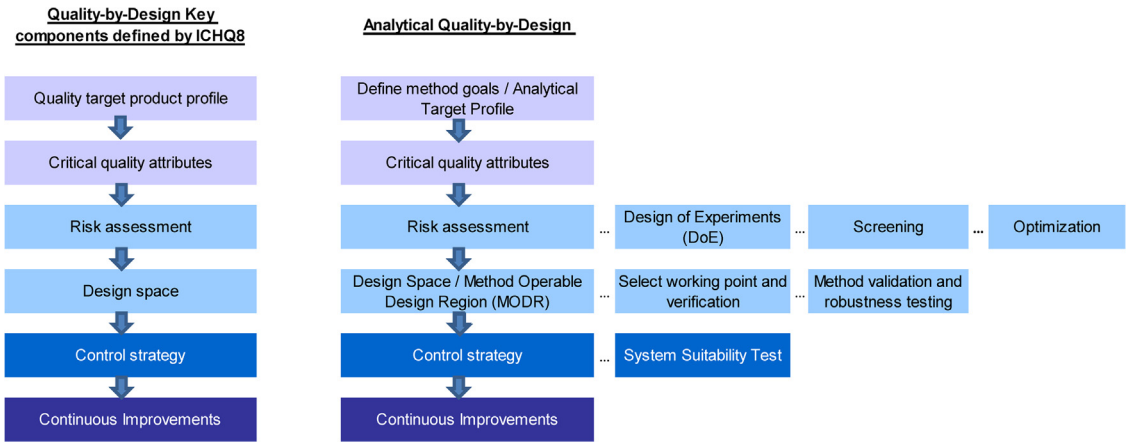

Fig. 4. Quality-by-design procedures for products as mentioned in ICH guideline Q8 and transfer to analytical quality-by-design.

4.4. Method development based on analyte based predictions

Another strategy in HPLC method development is based on the molecular structure, or physicochemical properties such as logP, logD and pKa of the sample components to estimate their retention and thereby optimal separation conditions [65,130,131].

Already in the 1980s retention time prediction in chromatography was discussed in scientific literature [132–134]. In recent years a growing interest in retention time prediction is documented by lots of publications [77,131,135–164]. Quantitative structure activity (QSAR), property (QSPR) or retention relationships (QSRR) may provide the basis for the estimation of retention times [77,135,141–147,152]. With the emerging interest in metabolomics or non-targeted analyses in general, structure based retention time prediction gains even more interest. Most of the methods used in this context utilize HPLC hyphenated to accurate mass spectrometry (AMMS). Even if AMMS is capable to identify a compound by its chemical composition (empirical formula) and eventually give structural information due to fragment assignments by tandem mass spectrometry MS/MS, there may still be lots of isomeric compounds that remain undistinguished. To further reduce the number of potential candidates for most confident identification (level 1 [165]) structure based retention time prediction may be of great value [143,144,147,149,166–168].

5. Method validation (method performance qualification)

Following method development, method validation is required. It is classified as stage 2 of the life cycle by Martin el al. [8]. Recommendations for pharmaceutical analysis may be found in the ICH guideline Q2 "Validation of Analytical Procedures: Text and Methodology" [103]. Adapted from the terms in FDA Guidance for Industry on Process Validation [9] "method validation" is also called "method performance qualification". This guideline was revised in 2011 to better align with the US Food and Drug Administration's "*Pharmaceutical Current Good Manufacturing Practices (cGMPs) for the 21st Century – a Risk Based Approach*" initiative and the ICH Q8, 9 and 10, and comprised a product life cycle concept [10–12,69].

As mentioned earlier, not only the process quality management can benefit from the concept of a product life cycle but also method validation [169–172]. Consequently, Ermer and Ploss [171] define method validation as "*the collection and evaluation of data and knowledge from the method design stage throughout its life cycle of use which established scientific evidence that a method is capable of consistently delivering quality data*".

The purpose of method performance qualification is to confirm that the method will operate (in routine use) as intended and meets the previously defined ATP criteria. It should be performed in the laboratory, which will be using the procedure routinely and in this case it may replace the current method transfer approach [173]. Commonly considered parameters in method validation include selectivity, linearity (calibration model), accuracy (bias), precision, limit of detection (LOD), lower and upper limit of quantification (LLOQ and ULOQ), stability, recovery, robustness, matrix effects [103,174–177]. Their individual relevance strongly depends on the ATP. Assistance for the experimental setup for method validation may be taken from organizations such as ICH [103] for pharmaceutical product analysis, FDA guidance or EU EMA guideline for bioanalytical methods [177–179] or international toxicologists [175]. Due to the fundamental differences in the techniques applied specific guidelines or at least consensus papers are available for validation of hematological and flow cytometric methods [180,181].

Within a life cycle concept of analytical methods an integrated approach of QbD method development may also result in an integrated data collection suitable for method validation [182–186].

6. Method transfer

Transfer of processes to an alternative site occurs at some stage in the life cycle of most products, from development, scale-up, manufacturing, production and launch, to the post-approval phase [187]. Analogous procedures may also be envisioned for the transfer of analytical methods [6]. The current approach for method transfer includes comparative testing, method co-validation, method verification or revalidation or a transfer waiver as alternative strategies [188–192] as requested by USP General Chapter ⟨1224⟩ Transfer of Analytical Procedures [193].

As stated by Nethercote and Ermer [170] within the life cycle concept the transfer of analytical procedures may be considered as method performance verification and integrates actions that are determined by risk assessment. This is also claimed as additional advantage of the life cycle management and continuous method verification [170,172]. Specific attention needs to be payed for methods such as ligand binding or immunoassays, where results may be strongly influenced by the platform and some critical reagents used [194].

7. Continuous method performance verification

Once method development and validation are completed, a method control strategy is established based on the risk assessment and data available in the life cycle approach. The method is then implemented for routine use where continuous monitoring of the method performance over the time is established (e.g. by using

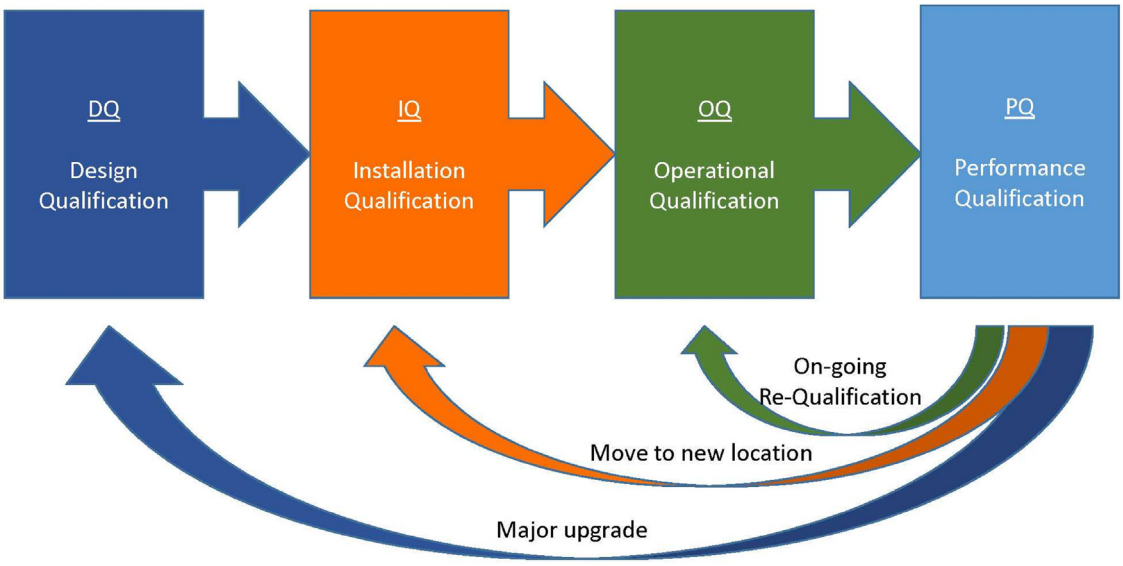

Fig. 5. Analytical instrument qualification in the "4Q model".

control charts) and improvements may take place when needed [63,84].

As important aspect in the life cycle approach method performance verification is used to examine how the method operates in routine use and that the resulting data are fit for its intended use (meaning accurate and precise). A statement on "*verifying an acceptable level of performance of an analytical system in routine or continuous use*" can be found in USP General Chapter ⟨1010⟩ "*Analytical data – Interpretation and Treatment*" [193]. It includes a continuous program for routine monitoring of analytical performance data and can be achieved through tracking of real samples or standards results (trend analysis charts), trending of system suitability data, assessing precision from stability studies, and/or analysis of a reference batch [5,193,195,196]. Verification and validation experiments should also demonstrate the robustness of the method across the parameter range from low to high through target values of variables [63].

If data indicate that the method is not operating as expected (e.g. causing lab related out-of-specification results), an identification of the root cause of the variation should be evaluated. The outcome of this investigation may result in a change of the method and thereby improvement of the (new) method performance [197,198]. The nature of the change dictates the action that is required: it may be a change to the method design (stage 1) and/or causes re-validation (stage 2).

One of the advantages of using the quality-by-design approach for method development is that post-approval changes within the method operable design region may be seen as an adjustment and do not need regulatory approval.

8. Fitness for purpose concept

To ensure high quality of analytical measurements Wenclawiak et al. [199] state they "*should be made using methods and equipment which have been tested to ensure they are fit for purpose*". This may also be transferred to qualified personnel and their working environment. Thus, meeting the predefined ATP and constant evaluations are highly important throughout the life cycle of an analytical method. According to EURACHEM [16] "*method validation enables chemists to demonstrate that a method is 'fit for purpose'*".

To evaluate the fitness for purpose of an analytical method data gained during method performance qualification and verification need to be judged in the light of the preset ATP [200–202]. According to Traple et al. and Guigues et al. the level of confidence of analytical data and the resulting measurement uncertainty should be used as measure [201–203]. Furthermore, other parameters of the ATP need to be considered as well.

However, qualification of instruments and systems can positively or negatively influence the analytical life cycle as well. If an analytical system is not installed correctly, the environment is not suitable for the instrument, or the instrument is not operated correctly, the analytical data/results are not valid. Thus, system suitability testing should be considered as integral part of analytical procedures [103].

According to USP General Chapter ⟨1058⟩ [204] the analytical instrument qualification can be seen as the base for reliable and consistent data (data quality). Focusing on the QbD principles, a qualification process based on the "4Q model" is typically used to demonstrate that a an analytical instrument is fit for purpose [204,205]. The 4Q model qualification process consists of four phases.

As illustrated in Fig. 5, the qualification process starts with the Design Qualification (DQ), in which the requirements for the instrument (user requirement specification, URS) are compared with the specification of the instrument manufacturer. After ordering and delivery of system, the Installation Qualification (IQ) phase starts with the documentation of delivered components, the installation of all modules as well as training provided for the users. During the Operational Qualification (OQ) the instrument is tested under standardized conditions, thus, confirming the correct operation of the instrument in the light of its specification. Later on, the Performance Qualification (PQ) addresses the suitability of the instrument under actual conditions of use and based on good scientific practice [205].

The full 4Q qualification process is performed every time a new instrument is implemented into a laboratory. Requalification of an existing instrument after a specified time period within the life cycle of the instrument, typically linked to preventive maintenance procedures, is necessary to prove that the system is still fit for purpose. In addition, when the location of an instrument has changed or the instrument undergoes major repairs or modifications, relevant IQ, OQ and/or PQ tests should be repeated [86].

According to USP ⟨1058⟩ "*Analytical Instrument Qualification*" [193], laboratory equipment is categorized in the risk groups A to C:

Group A: Standard laboratory equipment (magnetic stirrers, evaporators, etc.) with no measurement capability and no need for calibration. Qualification processes are not necessary.

Group B: Standard laboratory equipment with measurement capability that need calibration (e.g. balances, pH meters and thermometers). Conformance to user requirements are documented during IQ and OQ phases.

Group C: Complex instruments and computerized analytical systems. Conformance to user requirement is documented through all qualification phases. In addition to dissolution testers and spectrometers, all HPLC systems are classified as category C instruments.

As part of the risk management the classification is used to establish the level of qualification activities necessary to demonstrate fitness for intended use. For example, in HPLC analysis a frequent system suitability test – as required by pharmacopeia chapters European Pharmacopoeia (Ph.Eur.) 2.2.46 [206] and USP⟨621⟩ [193] – can be seen as an ongoing performance qualification for its intended use. Therefore, trending of system suitability data in control charts helps to identify and understand potential issues and take preventive actions before a major problem occurs [72,204,207].

9. Method retirement

As final stage of the life cycle of a method its termination should also follow quality management principles. On the one hand data storage needs to be considered especially if the method was used in regulated environment. However, more and more research organizations disclaim raw data storage worth the attention as well. To be able to reevaluate data even years after the termination of the original analytical method, storage of the relevant software needs to be considered as well [208]. A method may be completely terminated due to changes in the scope of a laboratory, or a new method may be based on the concluded, to meet newly defined ATP requirements [209]. If a new method is based on the retired one, some parts of the earlier lifecycle may be used to start the new.

10. Conclusions and future perspectives

The implementation of the ICH guidelines Q8 to Q11 within the pharmaceutical industry is intended to modernize the current approach for development and manufacturing of pharmaceuticals to a more scientific and risk-based approach. Although the ICH guideline Q8 does not explicitly mention analytical method development, a quality-by-design (QbD) approach in pharmaceutical development is requested [14]. Therefore, the QbD concept may be extended to analytical methods. Stimuli articles to the USP follow this trend.

While the concepts in ICH Q8, Q9, Q10 and Q11 provide opportunities for a more science and risk-based approach for assessing changes across the life cycle, several gaps exist which limit full realization of intended benefits. The envisioned post-approval flexibility has not been achieved yet. Therefore, a new proposed ICH guideline Q12 will provide guidance to facilitate the management of post-approval changes in a more predictable and efficient manner across the product life cycle. Adoption of this new ICH guideline will promote innovation and continual improvement, and strengthen quality assurance and reliable supply of a product [Q12].

In addition, the enrollment of further ICH guidelines and a new general chapter of the USP are already discussed to give guidance explicitly to the life cycle of analytical procedures.

11. Remarks

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