

TRAINING COURSE

How to Develop Stability Indicating HPLC Methods



This course will enable you to understand how HPLC methods work, how to develop a new HPLC method, and how to show that a new or existing HPLC method (e.g. a pharmacopeial method) is stability indicating.

Course overview:

Pharmaceuticals need to be assessed for stability to support the assigned shelf life. Therefore, when analysing stability samples obtained from these studies, analytical methods are required which are stability indicating, i.e. there is a measureable response which correlates with degradation (if present). HPLC is a popular technique for monitoring the decrease in drug and corresponding increase in degradation products due to its separating abilities. However, the HPLC method must be developed carefully to ensure that degradation products are both separated and detected appropriately.

This two day training course is designed to provide a thorough understanding of how to develop HPLC methods specifically designed for stability indicating analysis of pharmaceuticals. The course will describe strategies for performing forced degradation studies and selecting optimal HPLC method parameters to ensure that all relevant degradation products are separated. The same strategies may be applied to existing methods to demonstrate that they are stability indicating. The course is ideal for those who have some experience of using HPLC.

This course focuses on reversed phase mode separations.

Learning Objectives:

1. Define the objectives for the development of a stability indicating HPLC analytical method.
2. Effectively assess all the available relevant information relating to the desired method.
3. Perform forced degradation studies to prepare samples that will be used for the method development.
4. Select suitable scouting conditions to find a suitable column and mobile phase system and investigate stressed samples.
5. Optimise the chromatographic conditions to result in the best possible separation.

Open-enrolment training courses:

You can attend one of our open enrolment training courses at the following locations:

- Hilton Garden Inn Heathrow, London, UK;
- Metro Hotel Dublin Airport, Dublin, Ireland; and
- GLS Campus Berlin, Germany.

The courses are available throughout the year, refer to the MTS website for full details.

On-site training courses:

We can deliver the course at your site including any required customisation to meet your specific requirements. Contact us to discuss your training needs and for a quotation.

Use of case studies and real life scenarios:

Case studies are employed to allow consideration of real life scenarios. Delegates are invited to bring along any real life examples that they would like advice on during the training. These may be discussed during group exercises, or, where intellectual property is an issue, privately with the trainer.

This course is suitable for:

Anyone who has some experience of using HPLC and wants to know more about how HPLC methods work, and how to develop new HPLC methods, particularly for stability indicating purposes.

For example:

- Analytical chemists;
- Laboratory managers/ supervisors;
- Quality control analysts/ managers;
- etc.



Included in the course fees:

- Comprehensive course hand-outs;
- Certificate of Attendance;
- Access to training resources via e-MTS;
- Optional post training assessment (leading to Certificate of Training);
- Post training support;
- Lunch and refreshments (for open-enrolment courses only).

Course Agenda & Outline

Day 1

Timings

(approximate): Content:

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| 0900 to 1030 | Introduction: Stability of pharmaceuticals & available regulatory guidance <ul style="list-style-type: none">• The stability of pharmaceuticals and how this is investigated is discussed to introduce the concept of a stability indicating method.• Common strategies for HPLC method development are compared and the benefits of using a strategic approach are discussed.• Available regulatory guidance relating to stability indicating methods is reviewed. |
| 1030 to 1045 | <i>Refreshment break</i> |
| 1045 to 1130 | Step 1: Setting suitable objectives for the HPLC method <ul style="list-style-type: none">• Defining the requirement for a stability indicating HPLC method: the analytes; the sample to be tested; the type of test required; and the purpose of the test.• Consideration of restrictions and preferences for the method, e.g. available equipment, run time, etc.• Setting appropriate criteria for the method: Goals for the separation in terms of resolution (R), efficiency (N) and capacity factor (k'); |
| 1130 to 1230 | Step 2: Assessing all available information <ul style="list-style-type: none">• Identifying potential sources of information which may be useful during method development.• Assessing the effects of the structure of the analyte and in particular: how molecular weight and polarity of analytes are related to the most suitable type of HPLC and why reversed phase mode is preferred; how pKa affects the choice of mobile phase; analyte solubility. |
| 1230 to 1315 | <i>Lunch</i> |
| 1315 to 1500 | Step 2 continued: <ul style="list-style-type: none">• How the properties of the analyte impacts on the choice of detector; detector options for stability indicating HPLC methods.• Assessing available information relating to interferences which are likely to be encountered for the method.• Determination of what is known regarding the degradation profile of the active pharmaceutical ingredient. |
| 1500 to 1515 | <i>Refreshment break</i> |

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| 1515 to 1645 | Step 3: Selecting suitable samples <ul style="list-style-type: none"> Identifying suitable samples for the HPLC method development. Performing forced degradation studies: Selection of suitable samples, stress conditions and timings. Preparation of the test sample(s) to be used for method development. |
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Day 2

Timings

(approximate): **Content:**

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| 0900 to 1030 | Step 4: Performing scouting experiments to select suitable initial conditions <ul style="list-style-type: none"> Separation theory for reversed phase HPLC including discussion of the parameters which affect selectivity, e.g. mobile phase composition, %B, gradient time and steepness, temperature, pH etc. Selecting columns which give different selectivity – tools for column comparison. The effects of HPLC method chromatographic parameters: e.g. column attributes, mobile phase composition, temperature, flow rate, injection volume, etc. |
| 1030 to 1045 | <i>Refreshment break</i> |
| 1045 to 1230 | <ul style="list-style-type: none"> Selecting initial conditions for stability indicating HPLC method development: stationary phase and mobile phase. Designing scouting experiments: consideration of the requirements of the method, selection of suitable stationary phase and mobile phase combinations and set-up of the scouting experiments. Interpretation of scouting experiments: how to identify promising potential conditions for the method, e.g. measurement of resolution, peak shape, etc. |
| 1230 to 1315 | <i>Lunch</i> |
| 1315 to 1500 | Step 5: Optimising the method to define method parameters which achieve the desired separation <ul style="list-style-type: none"> Adjusting method parameters to achieve the desired separation, i.e. optimising the separation. Using multi-segment gradients for complex mixtures of analytes. Using computer modelling to optimise the separation. |
| 1500 to 1515 | <i>Refreshment break</i> |
| 1515 to 1645 | <ul style="list-style-type: none"> Potential solutions for very polar analytes. Assessing peak purity for analyte peaks. Assessing the robustness of the method. Designing an appropriate system suitability test. |
