Accredited Programme Title: Research Chemistry Continuing Education Programme at GSK Module Specification Form

<table>
<thead>
<tr>
<th>Unit/Module Title</th>
<th>Techniques for Purification and Analysis</th>
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</thead>
<tbody>
<tr>
<td>Code</td>
<td>GSK/Chem 5</td>
</tr>
<tr>
<td>Date of initial approval event</td>
<td></td>
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<tr>
<td>Proposed Level</td>
<td>HE Level 4 / NQF Level 7</td>
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<tr>
<td>Proposed General Credit Value</td>
<td>5 credits</td>
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<tr>
<td>Brief Rationale</td>
<td>This module highlights the options available for the purification and analysis of compounds prepared within GSK.</td>
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<tr>
<td>Description of Learner (target audience)</td>
<td>The module provides a continuing framework of learning for new staff entering the company, primarily Chemistry graduates. However it is also suitable for those who have gained industrial experience elsewhere, and wish to become aware of the available purification techniques, and improve their skills in the interpretation of analytical data. This group may include staff who initially joined the company without a first degree, but who have achieved an equivalent qualification by part time study.</td>
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<tr>
<td>Learning Hours</td>
<td>50 (10 of which are in a “classroom setting”, with further learning gained “on the job”)</td>
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**Learning Outcomes**

An Introduction to Purification Techniques and Analytical Facilities

Including a “hands-on” session with a purification expert.

On completion of these sessions, participants should be able to:

- Demonstrate an awareness of the range of methods available for the purification of synthesised compounds, for example, silica-based chromatography, ion exchange methods, reverse phase methods (A4, A6, A7, B1b, B1c, B1d, B2i, B2j, Cn),
  - Including the selection of the most appropriate technique(s) for a particular purification
- Demonstrate an awareness of the main techniques used for the routine analysis of synthetic compounds, i.e. Nuclear Magnetic Resonance (NMR) and Liquid Chromatography-Mass Spectrometry (A6, A7,B1d, B2i, B2j, Cn)
  - Including the implementation of best practice for the preparation of samples for analysis (e.g. selection of appropriate solvent)

NMR for Recently Graduated Chemists

On completion of these sessions, participants should be able to:

- Demonstrate an understanding of the information that can be obtained from the interpretation of a standard proton NMR experiment, and the limitations of the experiment (A4, A6, A7, B1b, B1c, B1d, B2j, Cn)
- Appreciate the influence of one or more chiral centres on the proton NMR spectrum of a compound (B1d, B2k, Cn)
  - Understand the use of chiral shift reagents to estimate compound enantiomeric and/or diastereoisomeric purity/composition
- Understand the influence of the salt form of the compound being analysed on the resultant spectrum (B1d, B2k, Cn)
- Appreciate the effects on the spectrum of molecular properties, such as restricted rotation (B1d, B2k, Cn)
- Understand the application of the Karplus equation to the interpretation of the spectra of carbocyclic and related systems (B1d, B2k, Cn)
- Appreciate the role of experiments utilising the nuclear Overhauser effect to assist in structure elucidation (B1d, B2k, Cn)

* Whilst the specific “teaching sessions” are extremely important and informative for the participant, the real “training” derives from the application of the theory to the situations encountered by the participants on a daily basis. (B1b, B1c, B1d, B1e, B1g, B1h, B2j, Cm, Cq, Cs, Ct)

Therefore, it would be expected that the participants would be working for 6-12 months to gain the required level of aptitude described in the Learning Outcomes above.

<table>
<thead>
<tr>
<th>Indicative Unit content</th>
<th>The subject matter is of relevance to chemists and includes:</th>
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<tr>
<td></td>
<td>• An understanding of the theory underlying the commonly used purification techniques.</td>
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<td></td>
<td>• Hands-on experience of some of these techniques.</td>
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<td></td>
<td>• An understanding of the use of LC-MS as a key analytical technique.</td>
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<tr>
<td></td>
<td>• A detailed understanding of the use of proton NMR spectroscopy in structure elucidation and evaluation of compound purity.</td>
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</table>

| Assessment specification | - Within an agreed timeframe (i.e. 6-12 months of starting work), participants should be confident in the use of a range of purification techniques; they should also be able to utilise the key analytical techniques (LC-MS and NMR) to ascertain the identity and purity of compounds. The assessment will be based on the judgement of their supervisor and supervisor’s line manager, with input from other colleagues as required, based on their daily use of the techniques in question. |
|                         | - The participant will compile a body of experimental evidence demonstrating the application of relevant purification and analytical techniques which will be assessed by the supervisor and the supervisor’s line manager. |

The participant’s supervisor will provide written justification regarding their assessment. The supervisor’s line manager will review, discuss with the supervisor, and ultimately ratify the recommendations of the participant’s supervisor.

The external examiner will have access to:
- Examples where the knowledge acquired has been applied in the workplace, including:– chromatograms, LC-MS traces and NMR spectra demonstrating that appropriate techniques have been used to achieve the relevant standard of purity
  - Laboratory notebook
- Worked tutorial problems (NMR tutorial)
- The supervisor’s and supervisor’s line manager’s assessment summaries
| Learning support/indicative reading and resources | Purification methodology is referenced in “The Chemists’ Toolbox”, a GSK resource available in every research chemistry laboratory.  
Hands-on sessions delivered by in-house experts are used to demonstrate key purification techniques in action. The experts may then be consulted at any future date.  
During the two sessions that comprise the NMR course, the theory covered is reinforced by tutorial work.  
Copies of reference material are also provided from the NMR course.  
The participant’s direct supervisor and other colleagues (including the module leaders) can be consulted for their expertise in both purification and analytical methods. |
Techniques for Purification and Analysis Module Assessment Form

Candidate Identification
Number:………………………………………………………………………………………………………………

Supervisor:………………………………………………………………………………………………………………

Assessment Criteria

Fail
Unable to independently select the most appropriate purification technique from the available range and/or operate the technique. Needs support from supervisor in interpreting routine spectroscopic data.

Pass
Consistently selects and executes effective purifications with minimal intervention from supervisor or other experts. Demonstrates the ability to interpret routine spectroscopic data accurately and without supervision, but is able to recognise when the data are more complex and need to be discussed with a local expert.

Supervisor’s evidence
Please refer to the items in the participant’s portfolio that provide the relevant evidence of achievement

Assessment of the participant in the workplace

Independent identification and successful application of the most appropriate technique for compound purification on more than one occasion

Most appropriate analytical techniques selected to provide confidence in the structural identity and assessment of purity
Understood the information obtained from liquid chromatography/mass spectrometry (LC-MS), and recognised the limitations of the technique.

Interpreted proton nuclear magnetic resonance (NMR) spectra, taking into account factors such as possible stereoisomerism, used these in structure elucidation and in purity determination and appreciated when and why it is useful to use advanced techniques, such as nuclear Overhauser effects.

Recommendation: Pass or Fail
Please outline any particular areas of strength or, if fail, which of the above were not of the required standard.

Signed: (Supervisor) Date:

Comments from Supervisor’s Line Manager