CD-R Patent Protection:
Brand Characterisation to Identify Counterfeit Goods using Simplified Mass Spectrometry

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INTRODUCTION

CD-R discs are composed of layers of polycarbonate, a burnable dye, a reflective layer, and a protective coating. The type of burnable dye used by manufacturers is often patented with premium disc brands using dyes and other additives that give the best performance, while being stable to atmospheric conditions, such as light and humidity. Cheaper products often use less stable dye components, which can change over time, leading to possible data degradation. This burnable dye is sometimes mixed with other dyes to change its colour and appearance in order to make the disc more attractive to consumers, and to conceal the type of burnable dye used from competitors.

This competitive environment increases the manufacturers' need for reliable and reproducible testing techniques that can be used to identify differences between brands, and to detect forgeries. Ensuring the consistency of product composition is essential for maintaining product performance and quality. Many manufacturers are seeking ways to profile, or 'fingerprint' their brands by using analytical techniques capable of detecting lower quality counterfeit products that may infringe upon their patents.

A chemometric approach can quickly provide information about the similarities and differences within a chromatographic dataset. Automation of this process can greatly reduce the analysis time required, and the probability of error in this assessment. Advances in ease-of-use mass spectrometer operational software have changed working practices from complex to routine, making day-to-day MS use easier for all. This application note describes the characterisation of commercially available recordable compact discs using Time-Of-Flight Mass Spectrometry, (TOF-MS), especially designed for inexperienced users and simplified for experienced users.

SIMPLIFIED WORKFLOW

The Waters® ACQUITY UPLC® with Xevo™ QTof MS is an LC-MS/MS system that simplifies the process of acquiring and interpreting data to allow new users to feel confident when they have limited experience with TOF-MS. For the first time, Waters IntelliStart™ Software has been included with this type of mass spectrometer. With its advanced and basic settings, IntelliStart allows an experienced user to perform the initial instrument setup, which can then be accessed by less practiced analysts.

Figure 1 describes this calibration workflow and shows the report generated with a few simple mouse clicks. This workflow indicates if the instrument is performing within preset user-defined parameters. Calibration can be performed infrequently for an instrument situated in a well-regulated, temperature controlled laboratory.

LockSpray™ conditions can also be defined here using a setup wizard that can run in conjunction with the calibration.

Once the calibration profile and LockSpray have been set up and saved, further checks can be performed on these files to confirm that the instrument is still within its correct operating range. This will typically be performed before an important batch of samples to ensure precise accurate mass data.

SAMPLE PREPARATION

A simple sample extraction was employed. The CD-R was cut into small segments which were placed in a 100ml bottle. 50ml methanol was added, and the bottle was vigorously shaken for 10 min. The supernatant was filtered and placed into a Waters Certified vial, capped, and placed in the ACQUITY® Sample Manager for analysis.

LC conditions

LC system: ACQUITY UPLC

Column: ACQUITY UPLC HSS C_{18}

2.1 x 50 mm, 1.8 μm

Column temp: 40 ˚C

Sample temp: 5 ˚C

Gradient: 0.00 min 95% A

6.00 min 0% A

8.00 min 0% A

8.10 min 95% A

Mobile phase A: Water + 0.1% formic acid

Mobile phase B: Methanol + 0.1% formic acid

Weak needle wash: Water + 0.1% formic acid

Strong needle wash: Methanol + 0.1% formic acid

Total runtime: 10 min

Injection volume: 5 μL, partial loop injection with needle overfill

MS conditions

MS system: Xevo QTof MS

Ionisation mode: ESI positive

Capillary voltage: 3 kV

Sample cone: 30 V

Source Temperature: 120 ˚C

Desolvation gas: Nitrogen, 800 L/Hr, 400 ˚C

Cone gas: Nitrogen, 5 L/Hr

Lock mass compound: Leucine enkephalin

RESULTS AND DISCUSSION

Data acquisition and processing methods

Data were acquired using Waters MassLynx™ Software, v.4.1. Incorporated into MassLynx, IntelliStart Technology automates calibration and lock mass checks. Its real-time monitoring also gives the user confidence that the system is running at optimum conditions.

The data were processed using MarkerLynx™ XS Application Manager. This software enables users to overcome the time-consuming problem of identifying patterns in LC/MS data sets, by showing the similarities and differences of the detected MS traces. These characteristics define the markers within the sample set with a "mass-retention time pair". It would be difficult to characterise all of these differences without MarkerLynx XS, as they look quite similar upon inspection with the naked eye. These data can then be used for further experiments using the MS/MS capabilities of the instrument to help identify structural information[1].

ACQUITY UPLC with Xevo QTof MS System

CONCLUSIONS

- The ACQUITY UPLC with Xevo QTof MS system was used to show brand variations between commercially available CD-Rs sold in Japan and Europe using accurate mass spectrometry.

- MarkerLynx XS Software offers scientists the unprecedented ability to visualise and interpret the most complex data automatically.

- Counterfeit goods can easily be identified because the location on a MarkerLynx XS PCA plot will clearly show them as a separate group from the genuine product.

- Multi-point lock mass capability improves mass accuracy across the scanned range.

- This LC-MS/MS System reduces laboratory running costs with:

- IntelliStart ease-of-use Software
  - Simplified training of new users and daily operation with its background system monitoring.
  - Automated system setup to accurately prepare the instrument for use without human bias.

- Robust performance
  -Minimal variation over large sample batches (shown by good sample grouping) gives confidence in system reproducibility, and reduces the need for sample retests.

Reference

1. Morphet, J, Tatsuya, E. CD-R Patent Protection: Competitor Analysis Made Easy.
Waters Application Note. No. 720003161EN, July 2009.

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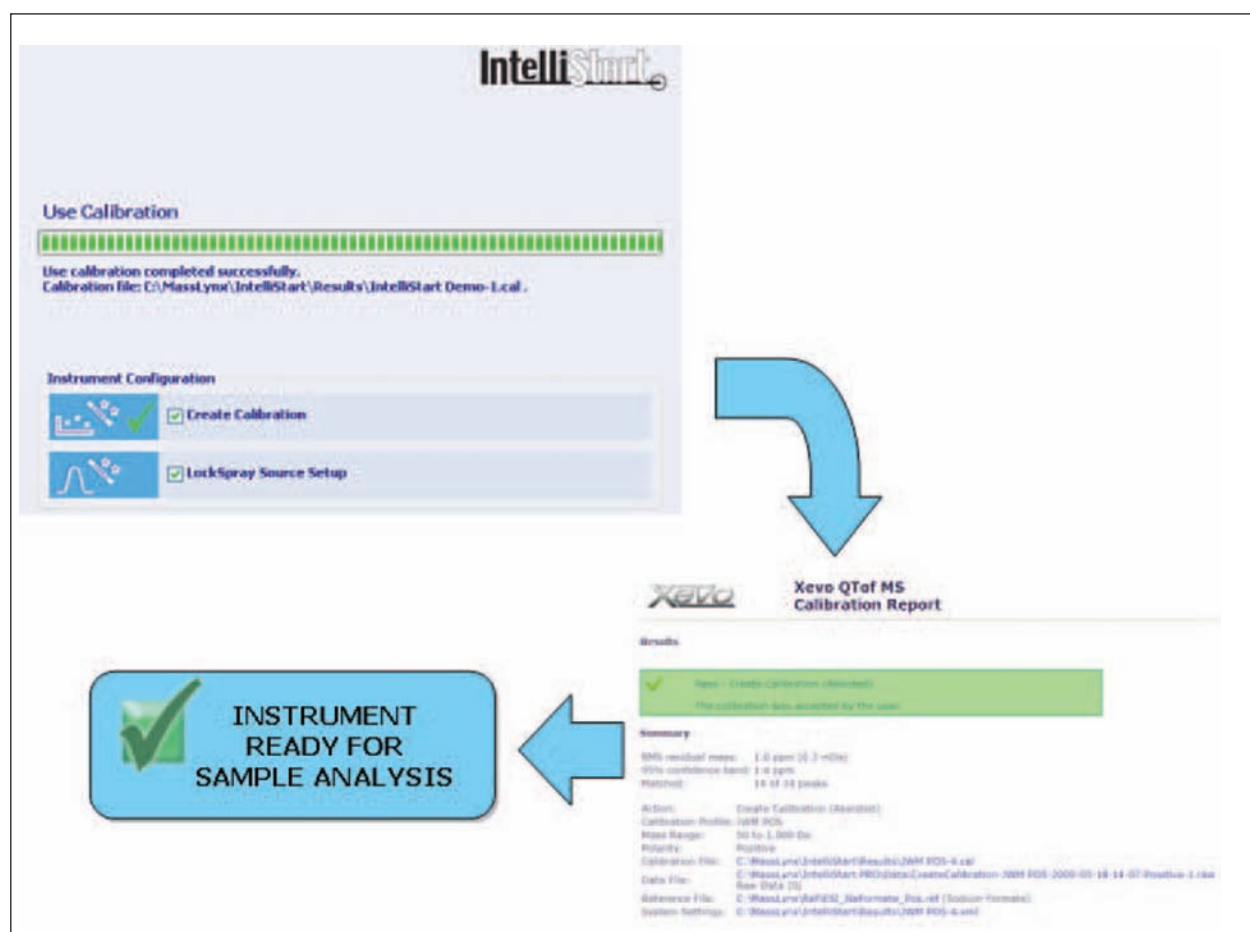Milford, MA 01757 USA
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Figure 1. Workflow showing easy calibration of Xevo QTof MS

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