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WP419

Development of a compact TLC sampler for MS and its applications

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1. Overview

A compact TLC (Thin-Layer Chromatography) sampler for MS analysis is developed with the features of simplicity, low cost and low cross contamination.

2. Introduction

The coupling of TLC-MS plays an important role in compound synthesis, purification and identification. A better TLC sampler for MS would make the analysis process more efficient. Unlike the traditional manual operation way that is time-consuming and non-quantitative, or some commercial product that is complex and expensive, this poster provides a more compact and easy-to-operate solution.

The whole system is integrated into a compact device without external gas, pump or PC control, and the operations of sample extraction and sampler cleaning are simplified to one-click. The extracted sample could be analyzed offline, yet the analysis result matches well with its position on TLC plate, the risk of clogging or cross contamination is minimized with the use of disposable parts.

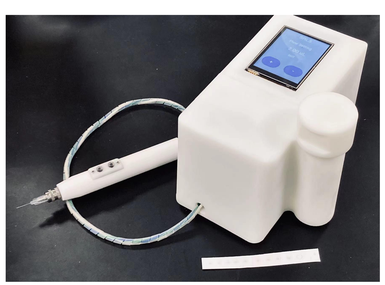

Figure 1. Appearance of TLC sampler

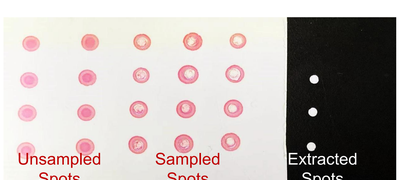

Figure 2. Picture of unsampled, sampled and extracted spots

3. Methods

The device comprises a sampler, a solvent injection mechanism and a filter.

- The sampler is used to cut down and hold a fixed size of adsorbent from TLC support.
- The injection mechanism is used to inject solvent for sample extraction, and the volume of solvent is limited to ensure the concentration of extracted sample.
- The filter is attached to the front end of sampler, the filtered sample could be introduced into MS by various ways. Especially, fast TLC detection (< 1min per sample) is achieved by the coupling of this device with our homemade compact MS using direct analysis ion source, the test results listed in part 4 are all obtained on this platform.
- The sampler could be self-cleaned by solvent flushing, and the filter is designed to be disposable to avoid clogging and cross contamination.

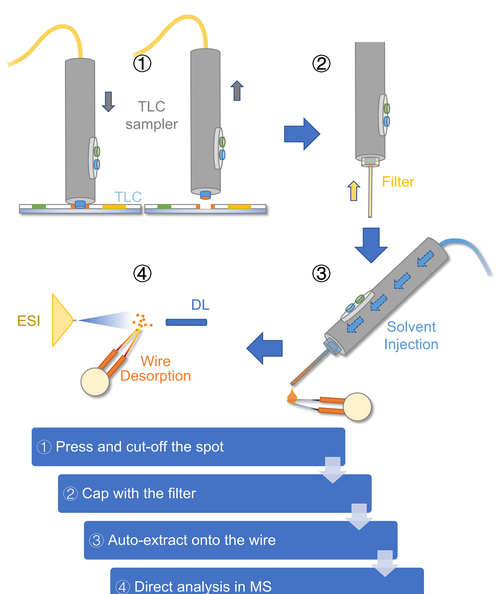

Figure 3. Operation procedure of TLC-MS analysis

4. Results

4-1. Extraction efficiency

10 to 1000ppm methyl red is used for the evaluation of extraction efficiency of the device. The extraction efficiency is calculated with the ratio of detected sample amount (m_{ext}) to sample amount on the cut-down adsorbent (m_{cut}), where m_{ext} could be estimated from sample concentration on linear curve. Table 1 shows the test result, an average rate of 70% could be achieved.

Table 1. TLC extraction result of 10 to 1000ppm methyl red

Test sample on TLC		m_{cut}	m_{ext}	Extraction efficiency
Concentration	Intensity			
10 ppm	9.40E+05	5μg	3.5μg	70%
100 ppm	2.06E+06	50μg	31.6μg	63%
200 ppm	5.58E+06	100μg	120μg	102%
500 ppm	1.00E+07	250μg	231μg	70%
1000 ppm	1.26E+07	500μg	297μg	59%

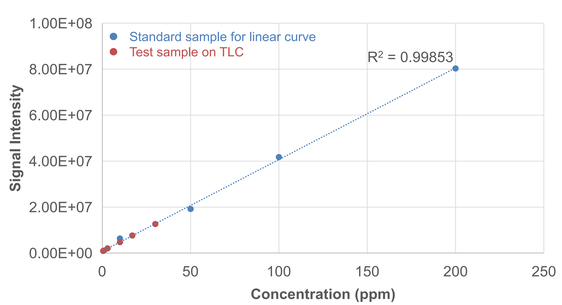

Figure 4. Estimated concentration of extracted samples on linear curve

4-2. Repeatability

200 ppm methyl red is used to evaluate the repeatability of this method, and 6 sample spots are detected to calculate the coefficient of variation. As shown in Figure 5, CV of these 6 detections reaches 13.2%, which counts in the stability of our compact MS and direct analysis ion source.

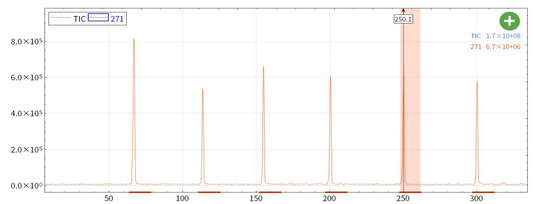

Figure 5. Repeatability of 6 detections on TLC

4-3. Application on practical samples

Figure 6 gives an example of component checking on practical TLC plate sent by our collaborator that is used in their daily synthesis work. The TLC plate is already developed, and the interested spots are labeled. Analysis results could be clearly linked to the position on TLC, and no cross contamination is found.

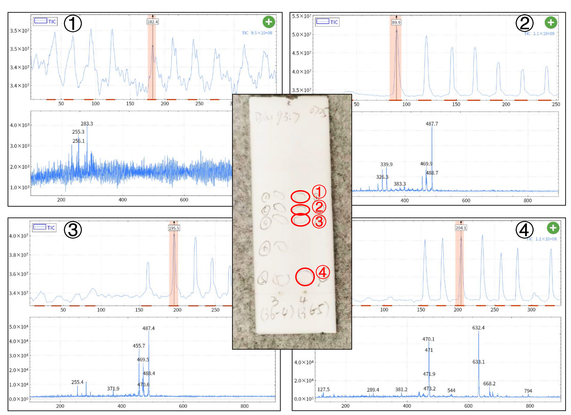

Figure 6. Example of practical TLC detection

5. Conclusions

A compact TLC sampler is developed for MS analysis. Its high extraction efficiency, good repeatability, low contamination and easy handling make it a proper tool for the customers who have high demand of TLC detection.

Acknowledgement

The authors wish to thank Shimadzu Cooperation for funding this project.