

Note d'application

Rapid Detection of Illicit Material Within Simulated Seized Drug Samples by RADIANT™ ASAP Mass Detector

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Main

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Abstract

The growing scale of the illicit drug market has created an increasing demand for fast and reliable methods to identify seized drug samples. As forensic laboratories receive a growing number of submissions, the need to deliver rapid, accurate, and scientifically robust results has become more critical than ever. Efficient screening techniques are therefore essential for supporting timely forensic investigations.

Traditional confirmatory techniques such as GC-MS and LC-MS require extensive preparation and longer turnaround times. Rapid ambient ionization approaches such as atmospheric solids analysis probe (ASAP)

provide faster screening for high-throughput forensic workflows. Counterfeit pharmaceutical products frequently contain undeclared excipients, increasing identification complexity, and risk. To address these challenges, a streamlined analytical approach was applied to evaluate seized drug samples using the Waters RADIANT ASAP Mass Detector, with data processing performed in [SpectralWorks™ AnalyzerPro™ XD Software](#). This method enabled clear differentiation between counterfeit and authentic pharmaceutical products within simulated seizure scenarios. Counterfeit and genuine acetaminophen (paracetamol) tablets, along with a combined acetaminophen-caffeine formulation, were examined to demonstrate the capability of the technique, and the resulting data highlight its robustness and efficiency for rapid screening, confident identification, and reliable discrimination of drug-related materials.

Benefits

- Simple and easy-to-use, minimizing the requirement of an expert user or extensive training programs
- Minimal sample preparation is required
- Rapid analysis for incorporating collection of data at multiple cone voltages for enhanced specificity
- Rapid analysis using AnalyzerPro XD Software to deliver real-time library matching within seconds for confident decision making
- Compact benchtop footprint-space efficient design suitable for both laboratory and on-site usage

Introduction

There is a growing worldwide concern about illicit substances. Recent estimates indicate that the number of people who used drugs increased by approximately 70 million between 2013 and 2023, rising from 5.2% to 6% of the global population aged 15–64.¹ The continued proliferation of both traditional and novel drug substances presents an ongoing challenge for authorities worldwide, making the analysis of seized materials essential to effectively monitor and control, minimizing the potential risk to humans. Seized materials from the illicit drug supply chain exhibit substantial variability, including both counterfeit formulations and materials adulterated with secondary substances.² Notably, paracetamol has been documented as a long-standing and widespread adulterant in heroin samples. Therefore, rapid detection and confirmation of these illicit substances is vital for minimizing potential harm to human health.

RADIAN ASAP Mass Detector, a compact mass detector developed by Waters, integrates the simplicity of the ASAP with the molecular specificity of mass spectrometry and was used to investigate a simulated seized drug sample. The seized drug samples included a range of tablets: over-the-counter (OTC) paracetamol products with and without caffeine, as well as counterfeit paracetamol tablets produced in an unregulated drug manufacturing laboratory. The AnalyzerPro XD Software, together with the Waters Seized Drug Reference Library was used for real-time analysis to confirm the presence of paracetamol and, also to show differentiation between the sample types.

Experimental

Sample Materials

Preparation of Simulated Counterfeit Samples

Three laboratory prepared tablets were generated to simulate counterfeit products which reflect formulation variability commonly observed in illicit drug products using paracetamol (Sigma-Aldrich, UK) and adulterated with additional excipients including Kollitab™ DC 87 (BASF, Germany), PROSOLV™ EASYtab™ SP (JRS Pharma, Germany), and Starch 1500™ (Colorcon, USA) (Figure 1). Samples were prepared under controlled laboratory conditions for analytical evaluation only.

For the counterfeit tablets, the active pharmaceutical ingredient and excipients were placed into an amber glass jar and blended within a Turbula mixer (WAB, Switzerland) for 5 minutes. The powder was passed through a 500 µm laboratory sieve (EndecoHs, UK) then transferred back into the amber glass jar and blended for another 5 minutes. For the Starch 1500 excipient batch, 1% w/w magnesium stearate was added as a processing aid and mixed for further 2 minutes.

Tablets were produced using a single-station press (Riva Minipress) fitted with 10 mm round, flat-faced tooling. The tablets were collected, placed in a 30 mL vial and labeled.

Commercial Tablets

Commercially available paracetamol tablets were acquired from nearby licensed dispensaries (Figure 1 tablets D-G).

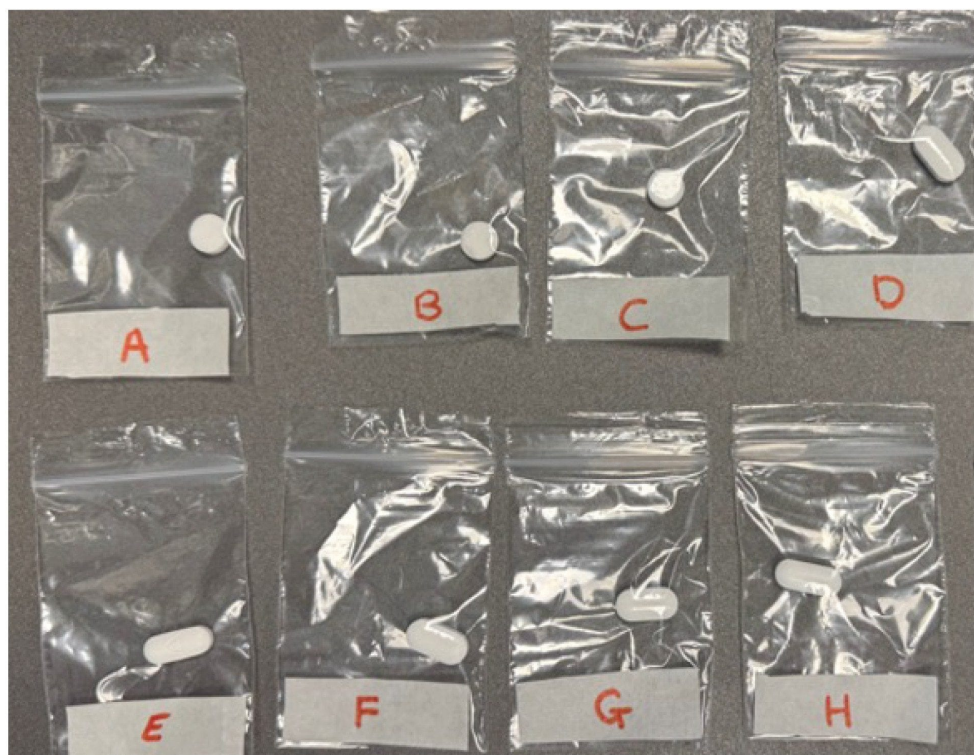


Figure 1. Simulated seized drug samples contain tablets, A-C counterfeits paracetamol tablets, D-G commercial paracetamol and H paracetamol and caffeine.

Sample Preparation

Samples were introduced in two ways; firstly, through the “rubbing method” and then using the Waters seized drug protocol³, the “dipping method”.

Method 1- “Rubbing” Method

For each sample, a new glass capillary was selected and baked out using the AnalyzerPro XD Software. This was then run as a blank to ensure no targets were detected. The glass capillary was subsequently ‘rubbed’ along the top of the tablet three times (Figure 2) and inserted into the ion source. Each sample was analyzed five times.

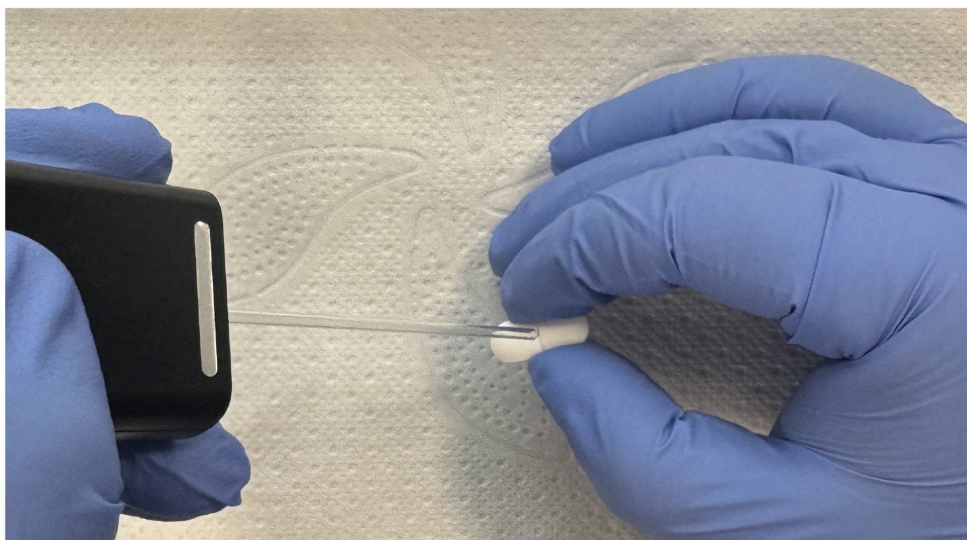


Figure 2. Rubbing method.

Method 2-“Dipping” Method

Each of the eight samples (A–H) was individually pulverized within its original bag, after which a 10mg subsample was transferred into a separate glass vial, dissolved in 1mL of methanol, and subsequently diluted to a final working concentration of 25µg/mL.

A new glass capillary was selected and baked out using the AnalyzerPro XD Software, followed by a methanol blank. A “dipping” method was used for each sample, where the clean capillary was dipped just below the surface of the liquid sample to a depth of approximately 1 mm for approximately 3 seconds. The glass capillary was then inserted into the ion source. Each sample was analyzed five times.

RADIAN ASAP Mass Detector Analysis

The analytical parameters used for data acquisition are summarized in Figure 3.

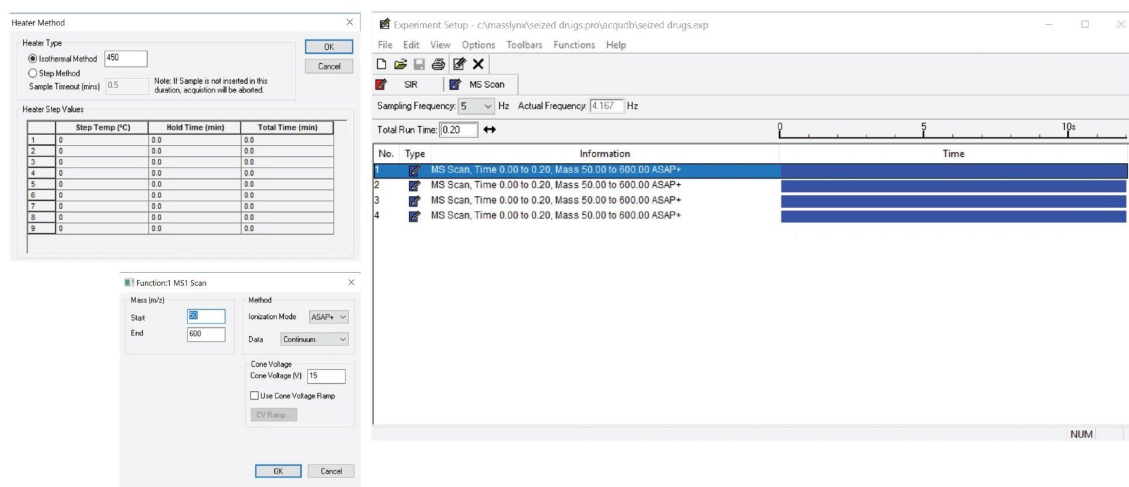


Figure 3. Seized tablets were acquired in full-scan mode over a range of m/z of 50-600 at four cone voltages, (15, 25, 35 and 50V).

Data Processing with AnalyzerPro XD Software

Acquired data was processed using AnalyzerPro XD Software, which provides real-time spectral library matching of acquired mass spectral data. Each spectrum is automatically compared against the curated Waters Seized Drug Reference Library using an advanced matching algorithm that assigns a confidence score to potential identifications.

The identification process evaluates multiple criteria, including forward and reverse spectral matching, relative and absolute ion-intensity thresholds, molecular mass verification, and ion-ratio consistency. Information from spectra acquired at all four cone voltages is then combined using a weighted scoring approach to generate a final confidence score, expressed as a percentage, for each compound identification.

Results and Discussion

Figure 4 demonstrates the successful identification of paracetamol following the "rubbing method" (method 1) across all tablet samples, achieved through Target Component Analysis using the Waters Seized Drugs Reference Library workflow in AnalyzerPro XD Software. All identifications were returned with confidence

ratings of 91% or higher.

	Data File	Status	Found	Vial Position	Sample Reference	Sample Description	Category
1	Bakeout 001	Processed	 No Target Detected	1			
2	Blank 001	Processed	 No Target Detected	2			Blank
3	A 001	Processed	 Paracetamol 98.1%	3	A counterfeit	Rubbed along A surface	A
4	Bakeout 002	Processed	 No Target Detected	4			
5	Blank 002	Processed	 No Target Detected	5			Blank
6	B 001	Processed	 Paracetamol 90.9%	6	B counterfeit	Rubbed along B surface	B
7	Bakeout 003	Processed	 No Target Detected	7			
8	Blank 003	Processed	 No Target Detected	8			Blank
9	C 001	Processed	 Paracetamol 97.8%	9	C counterfeit	Rubbed along C surface	C
10	Bakeout 004	Processed	 No Target Detected	10			
11	Blank 004	Processed	 No Target Detected	11			Blank
12	D 001	Processed	 Paracetamol 95.9%	12	D OTC	Rubbed along D surface	D
13	Bakeout 005	Processed	 No Target Detected	13			
14	Blank 005	Processed	 No Target Detected	14			Blank
15	E 001	Processed	 Paracetamol 98.7%	15	E OTC	Rubbed along E surface	E
16	Bakeout 006	Processed	 No Target Detected	16			
17	Blank 006	Processed	 No Target Detected	17			Blank
18	F 001	Processed	 Paracetamol 94.4%	18	F OTC	Rubbed along F surface	F
19	Bakeout 007	Processed	 No Target Detected	19			
20	Blank 007	Processed	 No Target Detected	20			Blank
21	G 001	Processed	 Paracetamol 95.1%	21	G OTC	Rubbed along G surface	G
22	Bakeout 008	Processed	 No Target Detected	22			
23	Blank 008	Processed	 No Target Detected	23		Blank	Blank
24	H 001	Processed	 Paracetamol 96.3%	24	H OTC	Rubbed along H surface	H

Figure 4. Target results for all samples using “rubbing” method.

Following the Waters seized drug protocol, the simulated seized drugs were put into solution and diluted for the “dipping” method. Figure 5 shows that all samples tested positive for paracetamol, with an increased confidence levels ranging from 96–99%. The increased confidence observed for the dipping method likely reflects improved analyte extraction and more reproducible ionization compared to surface sampling.

	Data File	Status	Found	Vial Position	Sample Reference	Sample Description	Category
1	Bakeout 001	Processed	 No Target Detected	1			
2	Blank 001	Processed	 No Target Detected	2		Blank	
3	A 001	Processed	 Paracetamol 98.1%	3	A counterfeit	A 25 ug/ mL in methanol	A
4	Bakeout 002	Processed	 No Target Detected	4			
5	Blank 002	Processed	 No Target Detected	5		Blank	
6	B 001	Processed	 Paracetamol 98.0%	6	B counterfeit	B 25 ug/ mL in methanol	B
7	Bakeout 003	Processed	 No Target Detected	7			
8	Blank 003	Processed	 No Target Detected	8		Blank	
9	C 001	Processed	 Paracetamol 96.4%	9	C counterfeit	C 25 ug/ mL in methanol	C
10	Bakeout 004	Processed	 No Target Detected	10			
11	Blank 004	Processed	 No Target Detected	11		Blank	
12	D 001	Processed	 Paracetamol 98.6%	12	D OTC	D 25 ug/ mL in methanol	D
13	Bakeout 005	Processed	 No Target Detected	13			
14	Blank 005	Processed	 No Target Detected	14		Blank	
15	E 001	Processed	 Paracetamol 98.9%	15	E OTC	E 25 ug/ mL in methanol	E
16	Bakeout 006	Processed	 No Target Detected	16			
17	Blank 006	Processed	 No Target Detected	17		Blank	
18	F 001	Processed	 Paracetamol 99.3%	18	F OTC	F 25 ug/ mL in methanol	F
19	Bakeout 007	Processed	 No Target Detected	19			
20	Blank 007	Processed	 No Target Detected	20		Blank	
21	G 001	Processed	 Paracetamol 98.6%	21	G OTC	G 25 ug/ mL in methanol	G
22	Bakeout 008	Processed	 No Target Detected	22			
23	Blank 008	Processed	 No Target Detected	23		Blank	
24	H 001	Processed	 Paracetamol 97.6%	24	H OTC	H 25 ug/ mL in methanol	H

Figure 5. Target results for all samples using “dipped” method.

To compare the seized drug samples, which included both counterfeit and commercially available tablets, data collected using the Waters seized drug protocol were analyzed by principal component analysis (PCA). As shown in Figure 6, the PCA clearly separated counterfeit tablets from commercial products based on their underlying chemical profiles. PCA separation is attributed to differences in excipient composition rather than the presence of paracetamol, demonstrating sensitivity to formulation-level variation.

PCA scores from results

Pareto scaling with Log transform and 95% confidence ellipses

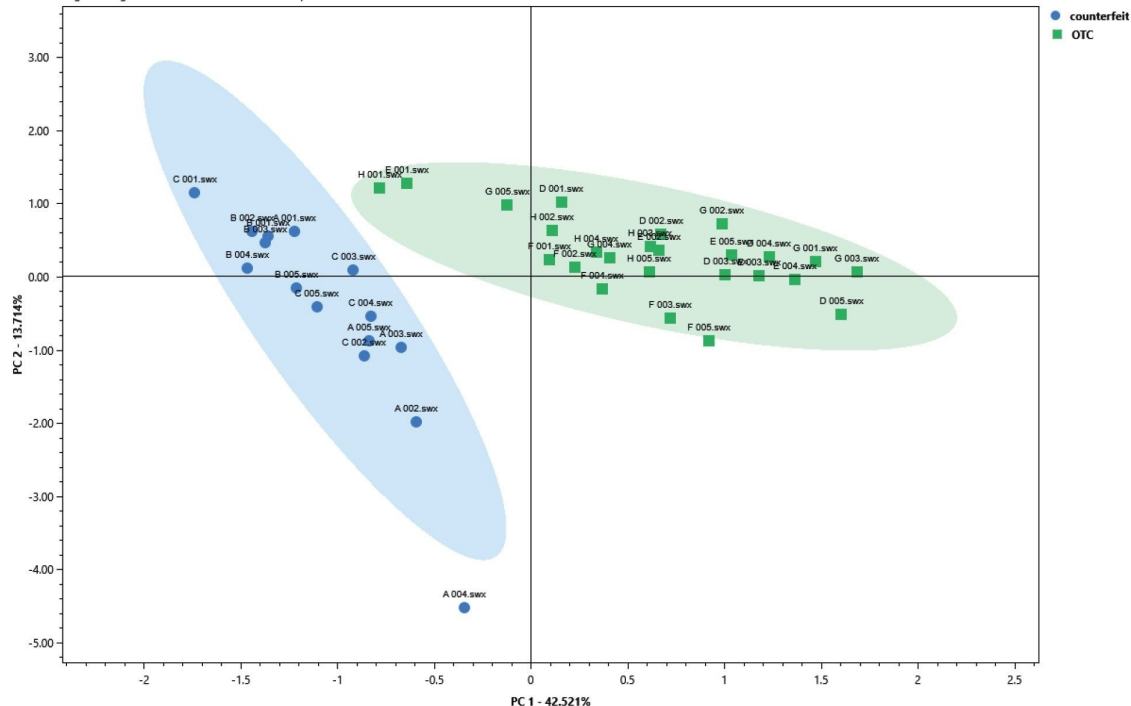


Figure 6. PCA of OTC and counterfeit tablets.

Figure 7 shows the comparison of tablet H vs tablet F; caffeine (m/z 195) is seen exclusively in the volcano plot for category H, with further confirmation from the bar chart.

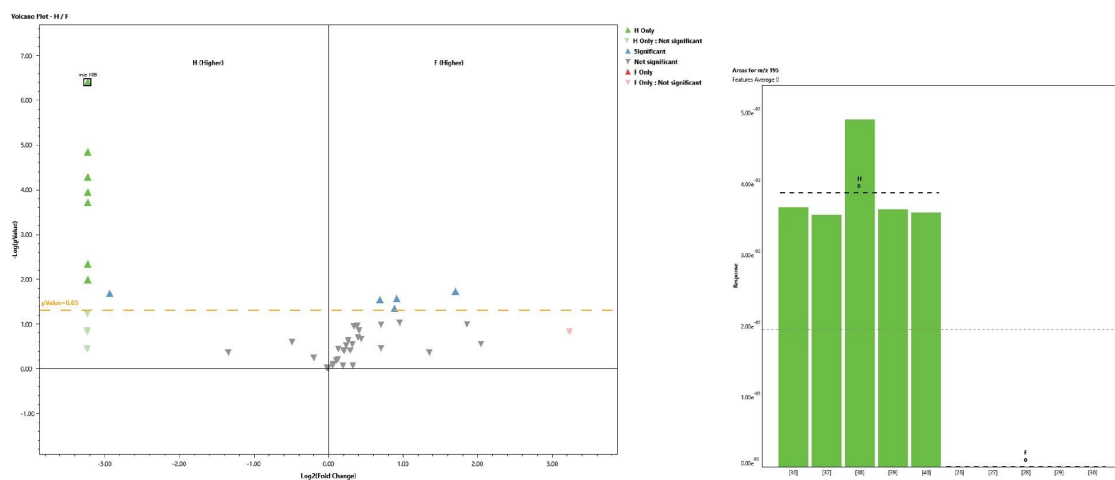


Figure 7. Volcano plot and bar chart for caffeine for tablet "H" compared to tablet "F".

Conclusion

The RADIAN ASAP Mass Detector, along with data analysis in AnalyzerPro XD Software, matched with Waters Seized Drugs Reference Library, allowed for accurate identification of paracetamol within commercial and counterfeit tablets within a simulated seized drug scenario. Additionally, the statistical workflow enabled clear differentiation between the OTC paracetamol tablets and the counterfeit samples. It also allowed identification of which OTC tablet contained caffeine.

While this study utilizes controlled simulated samples, further assessment using a wider variety of real-world seized materials would be valuable to capture the full range of potential sample variability.

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