

Pesticide Analysis in Chili and Cumin Powders using Verex™ Filter Vials by HPLC-MS/MS with Luna® C18 Columns

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Overview

Undissolved sample particulates can damage expensive equipment, valves, columns and pumps. They can also lead to erratic analytical results. Pre-filtering samples prior to analysis is critical in preventing column and frit blockage, undue wear on valve seals, and abnormally high operating pressures. Moreover, sample and solvent filtration will lead to an overall improvement in baseline, quantitation, reproducibility, and detection limits

In this application, chili powder and cumin powder were analyzed for pesticides. Samples were filtered using Verex Filter Vials and then run by HPLC-MS/MS to determine if there was a loss of sample or unwanted extractables by using the filter vials. When compared to the pesticide analytes without a filtration device, the filtered analytes using the Verex Filter Vials showed similar results.

LC-UV Conditions

Column: Luna 3 µm C18

Dimension: 150 x 4.6 mm

Part No.: [00F-4784-Y0](#)

Pressure (bar): 250

Mobile Phase: A = 0.1 % Formic Acid and 10 mM Ammonium Acetate in water
B = 0.5 % Formic Acid in Methanol

Gradient:	Time (min)	%B
	0	5
	0.1	5
	0.5	60
	3.5	75
	8	85
	10.5	95
	12	95
	12.1	5
	18	5

Flow Rate: 0.4 mL/min
0.35 mL/min, steps 3-5

Temperature: 20 °C

Instrument: Agilent® 1100 LC Quaternary Pump

Detection: MS/MS

Detector: 5500 QTRAP® (SCIEX®)

Injection Volume: 10 µL



Sample Preparation

Standards	Prepare:	1 mg/mL solutions of all 18 standards in water.
	Mix:	40 µL of every standard except 5-OH thiabendazole, tolyfluanid, quinoxifen, thiophanate-methyl, and etoxaole. From these take 150 µL and put into mix with others.
	Prepare:	10x diluted mixture by mixing 100 µL with 900 µL of 1:2 water/acetonitrile.
	Prepare:	50x and 100x diluted mixture from the 10x.
Samples	Mix:	1 g of either chili powder or cumin powder with 10 mL of water followed by 15 mL of acetonitrile in separate 50 mL centrifuge tubes.
	Spin:	3600 RPM for 10 minutes.
	Take:	1.5 mL of the supernatant from each and place into separate vials. Keep leftover supernatant and pellet.
	Spike:	30 µL of 10x standard mix and add 270 µL of either the chili powder or cumin powder supernatant.
	Load:	450 µL of either chili powder or cumin powder into Verex™ Filter Vial 0.45 µm RC ARO-F203-12 , or 0.45 µm NY ARO-F207-12
	Inject:	1 µL for standard mixture, 1 µL for Chili Powder supernatant only, or 3 µL of Cumin powder only and mixtures with spiked standards.

MRM Transitions

Peak No.	Analyte	Q1 (m/z)	Q3 (m/z)
1	Pymetrozine 1	218	105
1	Pymetrozine 2	218	78
2	5-OH Thiabendazole 1	218	190.9
2	5-OH Thiabendazole 2	218	147
3	Thiophanate-methyl 1	343	151
3	Thiophanate-methyl 2	343	192
4	Simazine 1	202.1	96.1
4	Simazine 2	202.1	104.1
5	Triazophos 1	314.1	162.1
5	Triazophos 2	314.1	97.1

Peak No.	Analyte	Q1 (m/z)	Q3 (m/z)
6	Metolachlor 1	284.1	176.2
6	Metolachlor 2	284.1	134.1
7	Tolyfluanid 1	347	137
7	Tolyfluanid 2	347	237.9
8	Coumaphos 1	363	227.1
8	Coumaphos 2	363	307.1
9	Pyraclostrobin 1	388	194
9	Pyraclostrobin 2	388	163
10	Prochloraz 1	376.1	308
10	Prochloraz 2	376.1	70.1



MRM Transitions

Peak No.	Analyte	Q1 (m/z)	Q3 (m/z)	Peak No.	Analyte	Q1 (m/z)	Q3 (m/z)
11	Phosalone 1	368	182.1	15	Trifloxystrobin 1	409	186
11	Phosalone 2	368	110.9	15	Trifloxystrobin 2	409	206
12	Thiobencarb 1	258.1	125	16	Prallethrin 1	301.2	133
12	Thiobencarb 2	258.1	89	16	Prallethrin 2	301.2	105
13	Pirimiphos-methyl 1	306.1	108	17	Quinoxifen 1	308	197
13	Pirimiphos-methyl 2	306.1	164.1	17	Quinoxifen 2	308	162
14	Clofentezine 1	303	138	18	Etoxazole 1	360.1	141
14	Clofentezine 2	303	102	18	Etoxazole 2	360.1	57.2

Figure 1. Full scan of pesticides in chili powder and cumin powder.

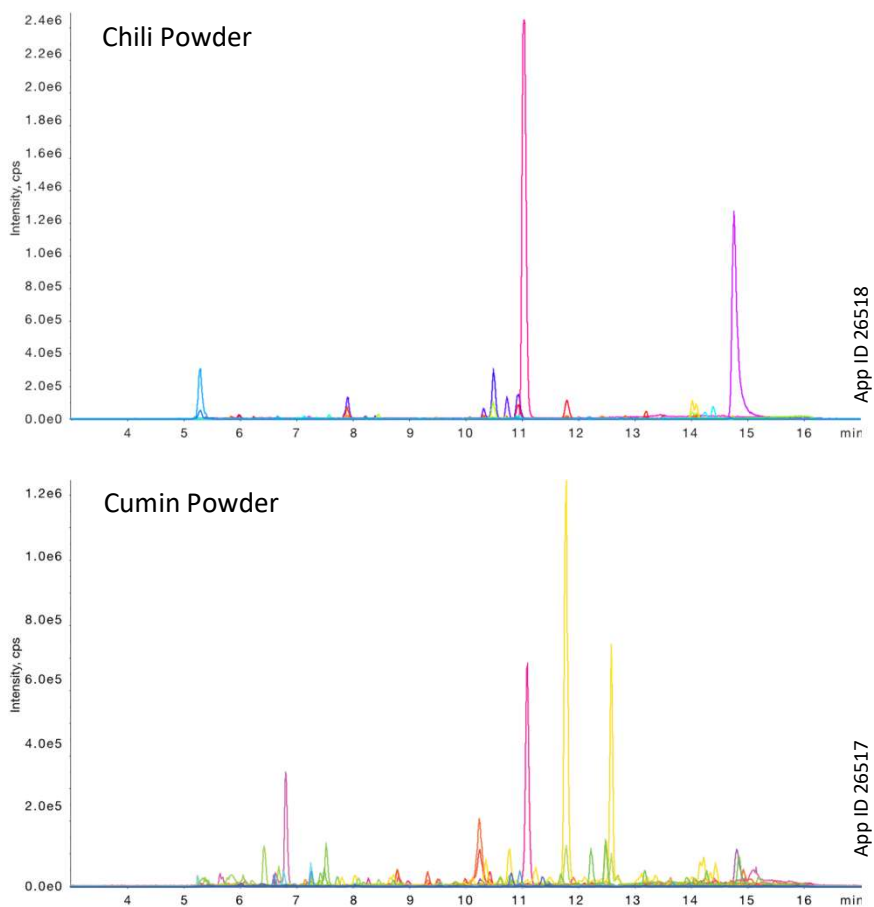


Figure 2. Prallethrin in chili powder and cumin powder with and without the use of Verex™ Filter Vials.

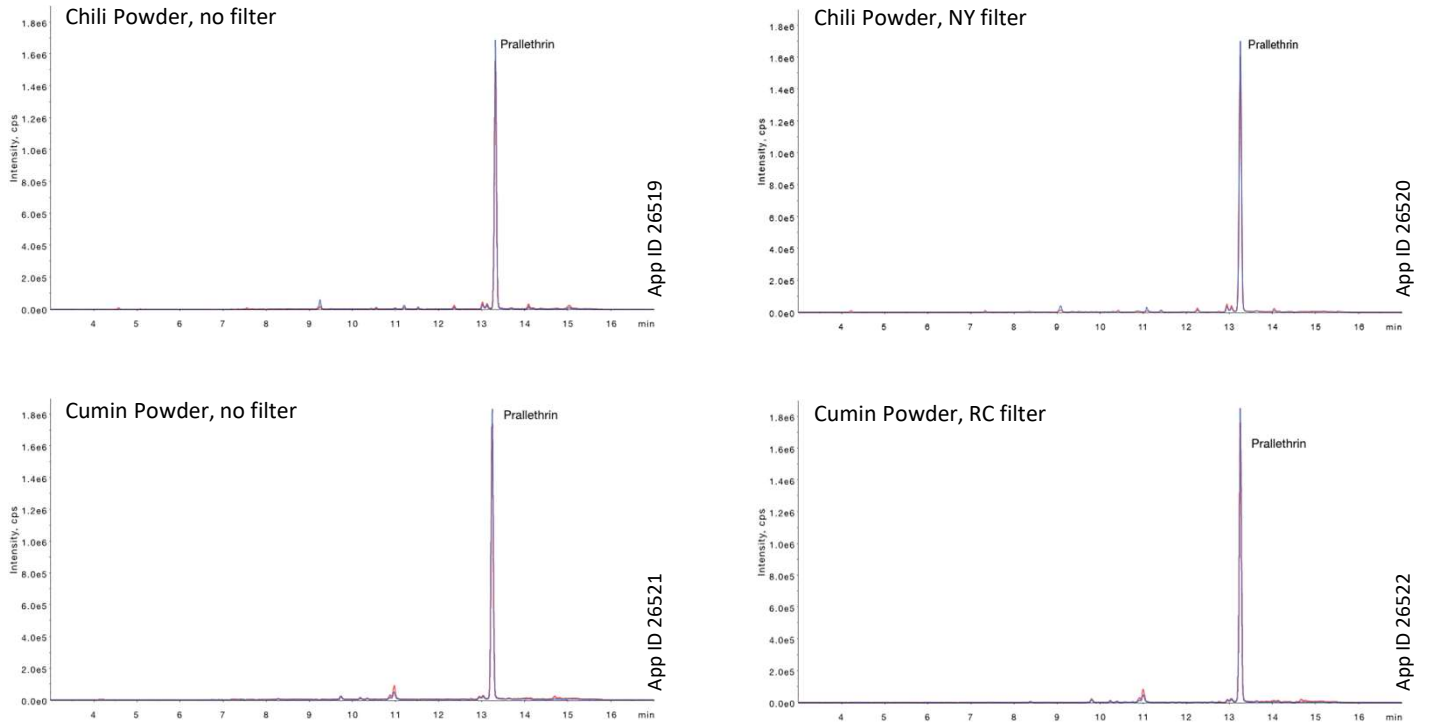


Figure 3. Metolachlor in chili powder and cumin powder with and without the use of Verex Filter Vials.

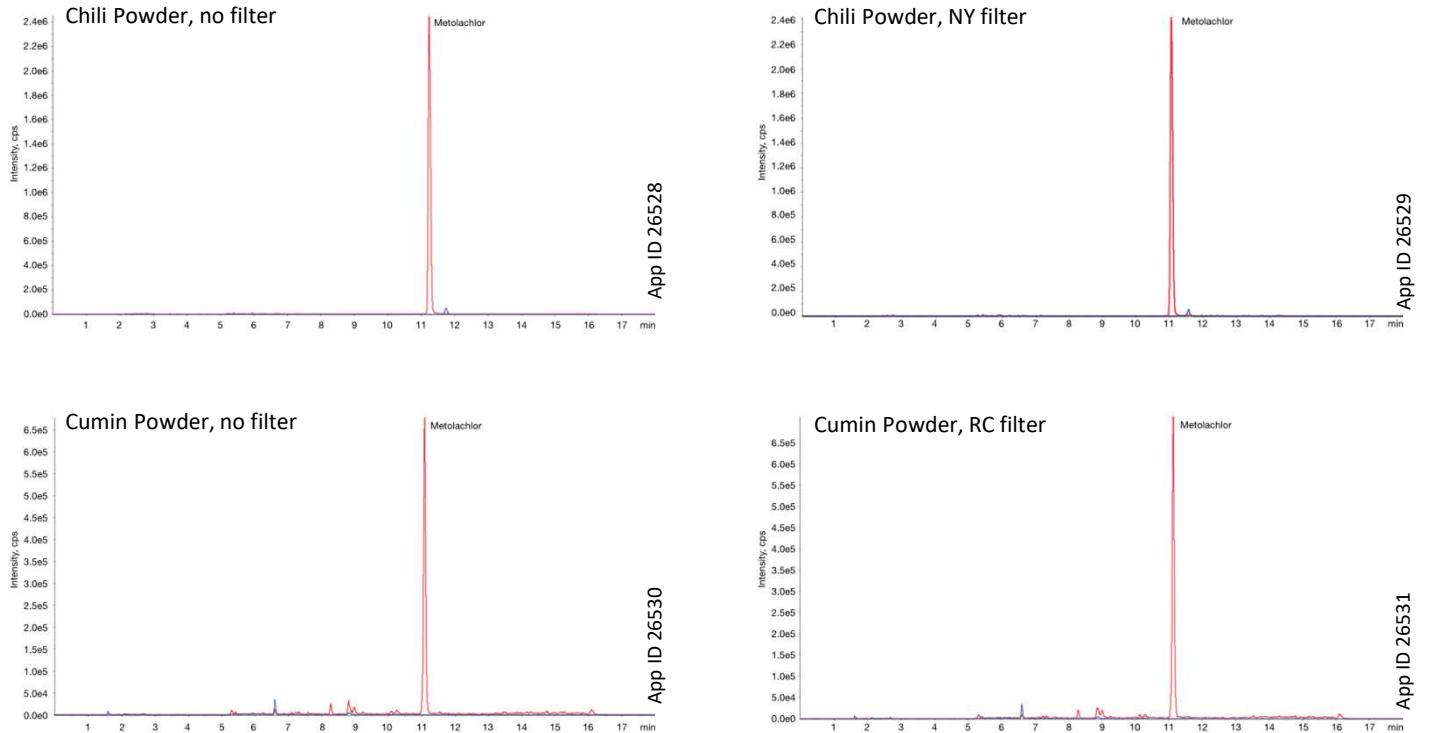


Figure 4. Thiobencarb in chili powder and cumin powder with and without the use of Verex™ Filter Vials.

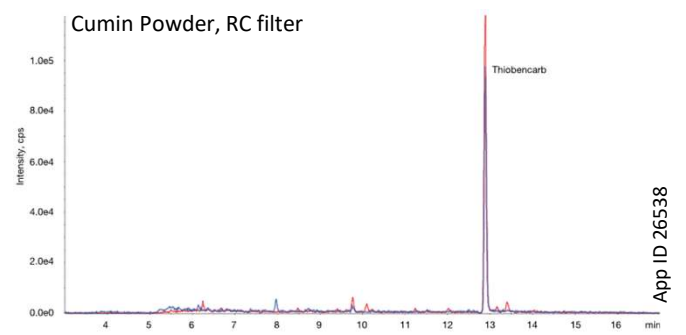
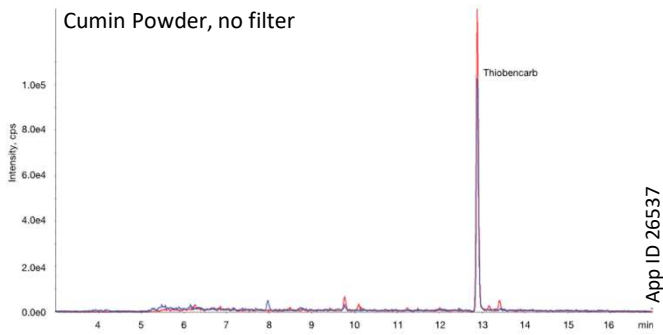
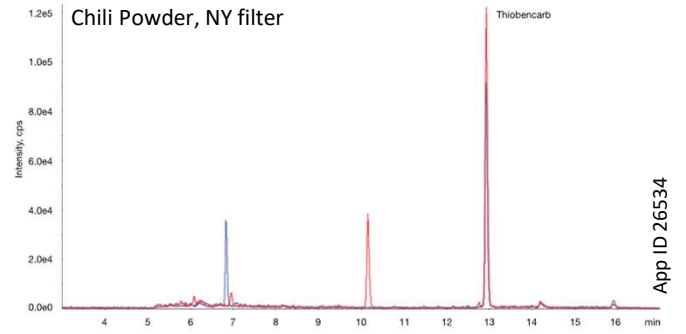
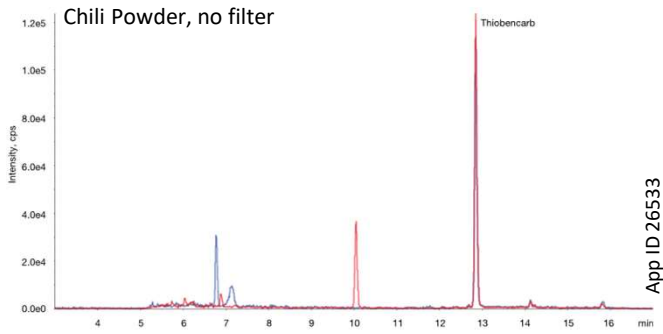
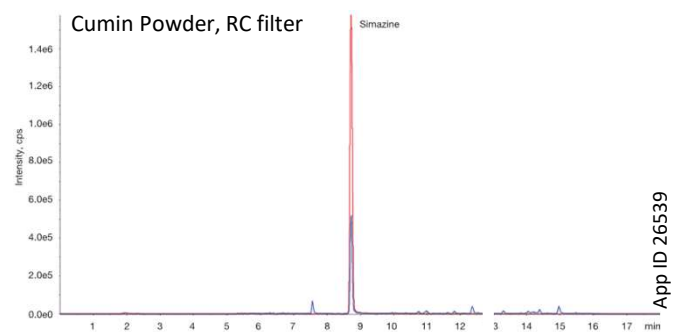
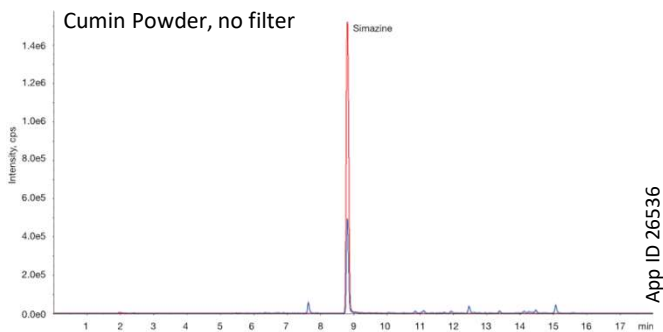
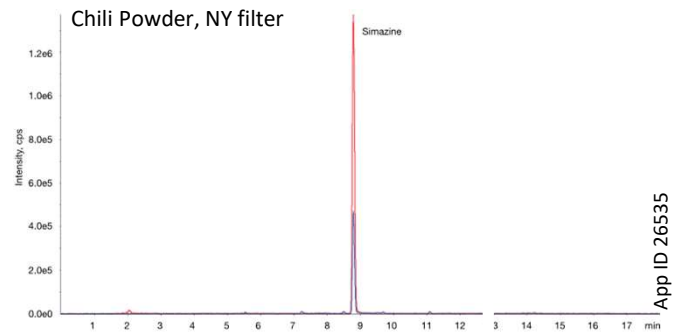
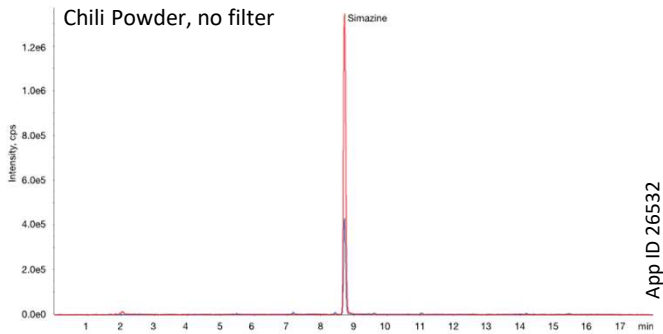


Figure 5. Simazine in chili powder and cumin powder with and without the use of Verex Filter Vials.



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