Repeatable Cannabidiol Isolate Method

Application Note

Quick, Accurate Analysis via Polyarc and GC/FID

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Abstract

The Polyarc microreactor for an existing gas chromatograph with a Flame Ionization Detector (GC/FID system) allows for a quick and accurate analysis of Cannabidiol (CBD) isolate purity. With a method time under twenty minutes, the purity of CBD isolate was determined to be 98.95% with an RSD value of 1% (n=3). With the Polyarc microreactor providing universal carbon detection via FID, CBD isolate was accurately quantitated at 9,416 ppm (mg/kg), matching the expected theoretical value of 9,831 ppm – which was calculated assuming no impurities present. When including impurities in the theoretical calculation, the experimental determined recovery was 97%.

Introduction

Since the 2018 Farm Bill, the United States has seen an explosion of hemp-related products, ranging from topical and cosmetic applications, to food and beverages, as well as in the dietary and vitamin supplements industries. Blended varieties of cannabis products and extraction methods also vary widely, resulting in an industry with many options.

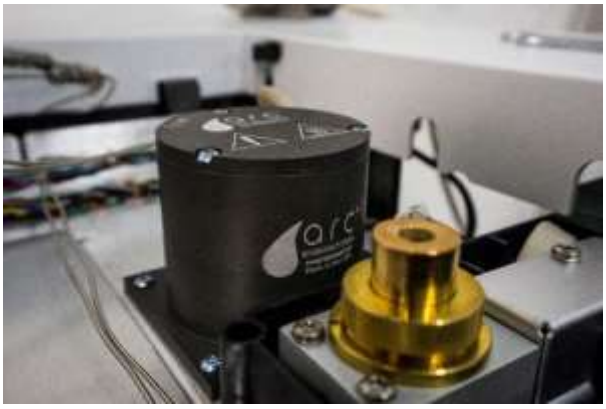

Figure 1. Polyarc microreactor on an Agilent 7890 GC.

Extraction methods commonly include either use of a solvent or supercritical liquid (commonly CO_2) to extract the desired product from the raw biomass, seed, or flower. This raw extract is used to produce a variety of raw or 'full-spectrum' CBD which includes terpenes, oils and cannabinoids including CBD. Manufacturers can then blend other ingredients or distill the raw extract to isolate pure CBD.

In this application note, a Polyarc microreactor is used with a GC/FID to accurately detect CBD isolate in a quick and repeatable method, accurate within less than 1% variance between run to run.

Experimental

An Agilent 7890A GC equipped with a multimode inlet (Agilent G3454-64000), a Phenomenex inert inlet liner (AG2-0A13), capillary-optimized FID, and Polyarc microreactor (ARC PA-RRC-A02) were used for the analysis. Helium (99.999%, Praxair) was used for carrier and FID makeup. Air (zero grade, VICI DBS Generator) and H_2 (99.999%, VICI DBS Hydrogen Generator) were supplied to the ARC Electronic Flow Control module (PA-MFC-A09) and to the FID. The effluent of the GC column was connected to the Polyarc, which was then connect to the capillary-optimized FID.

CBD samples were prepared for GC analysis by dilutions in undecane (Sigma Aldrich 99.99%). CBD was weighed and diluted to 9,831 ppm with a heptane spike, 10,152 ppm, to serve as an internal standard. A blank standard with undecane and heptane was also prepared.

GC conditions

Front inlet	Split Mode on MMI
Inlet temperature	250 °C
Inlet liner	Phenomenex AG2-0A13
Carrier gas	He; 2 mL/min constant flow
Septum purge flow	3 sccm
Oven	90 °C (hold 2 min) to 250 °C at 10 °C/min (hold 5 min)
Column	HP-5 (30 m x 0.25 mm x 0.25 µm film)
Syringe	10 µL
Injection volume	1 µL
Split Ratio	25:1

FID conditions

Temperature	315 °C
H_2 Flow Rate	1.5 sccm
Air Flow Rate	350 sccm
Makeup Flow Rate (He)	5 sccm

Polyarc® System conditions

Temperature Setpoint	293 °C
H_2 Flow Rate	35 sccm
Air Flow Rate	2.5 sccm

Analysis Procedure

Results were first analyzed comparing area percentages of CBD relative to the sum of CBD and total impurities. The concentration of CBD was then analyzed relative to an internal heptane standard – which is possible through the Polyarc microreactor. Methane produced from combustion-reduction reactions in the Polyarc is measured with the FID resulting in an equimolar carbon response. The concentration of each analyte can therefore be calculated from the concentration/area ratio of an arbitrary standard using the following equation:

$$C_A = C_s \left(\frac{Area_A}{Area_S}\right)\left(\frac{\#C_S}{\#C_A}\right)\left(\frac{MW_A}{MW_S}\right)$$

where:

C_A = Wt. % of analyte
$Area_A$ = Integrated peak area of the analyte
Mw_A = Molecular weight of the analyte
Mw_S = Molecular weight of the standard
$\#C_S$ = Number of carbon atoms for standard
$\#C_A$ = Number of carbon atoms for analyte

*See "Quantification with the Polyarc.pdf" at https://www.activatedresearch.com/documents/ for more information.

Results and Discussion

Three injections of the CBD isolate sample and a respective blank reference were performed. The area percentages of CBD to impurities are tabulated below in Table 1.

CBD Peak Area Percent

Injection	CBD	Impurity A	Impurities B
One	98.91%	0.74%	0.35%
Two	98.94%	0.75%	0.30%
Three	99.00%	0.71%	0.30%
Average	98.95%	0.73%	0.32%

Table 1. List of peak areas percentages of CBD and impurities present per sample injection. Each injection was followed with a blank injection. Impurity B is the sum of the remaining non-A impurities.

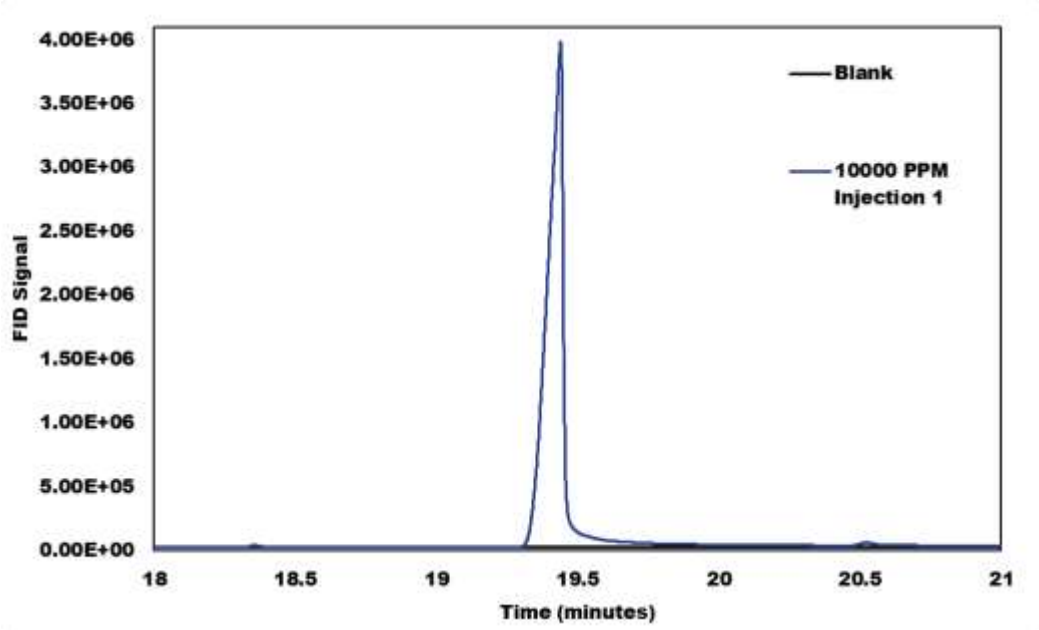

Figure 2. Polyarc/FID chromatogram of CBD isolate peak.

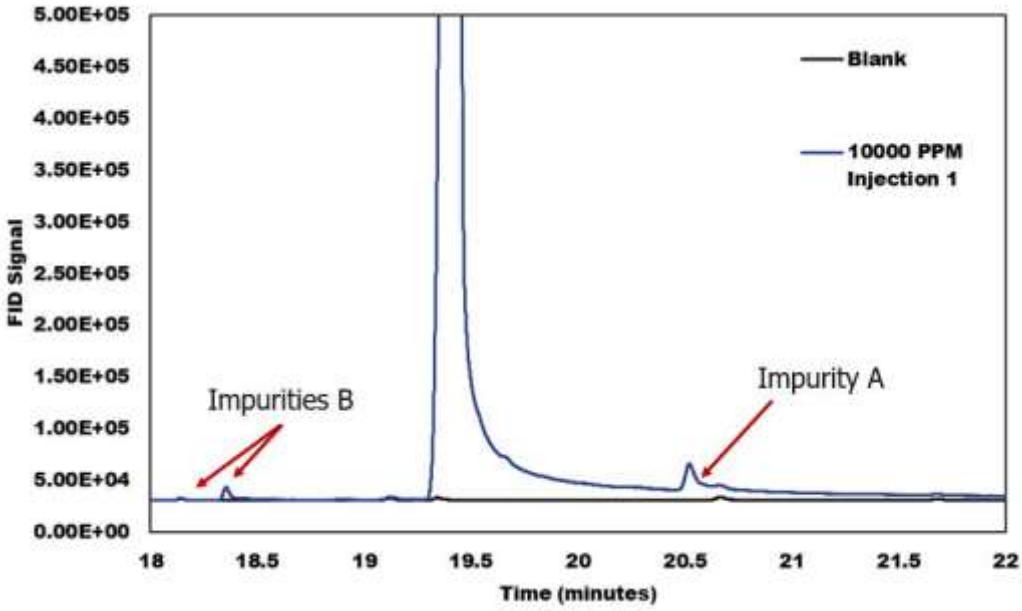

Figure 3. Polyarc/FID chromatogram of CBD peak with impurities A and B at minute 20.5 and 18.4 respectively.

Peak Area			
Injection	**CBD**	**Impurity A**	**Impurities B**
One	1.63E+08	1.22E+06	5.69E+05
Two	1.66E+08	1.26E+06	5.10E+05
Three	1.65E+08	1.18E+06	4.95E+05
% RSD	1.05%	3.28%	7.46%

Table 2. Peak areas per injection number with resulting RSD values.

Based on the peak area results, as shown in Table 2, concentration of CBD isolate was determined to be 9,416 ppm with around 100 ppm total of impurities. The response factor was determined to be 0.94, very close to the theoretical value of 1. That is, the relative response factor of the species to the internal standard (heptane), where area is the integrated detector response, and concentration is the molar carbon concentration in the mixture. The final response factor of 0.94 is within GC error +_10% when compared to the theoretical uniform response factor of 1. See below for response factor equation.

$$RF = \frac{{}^{area}/_{concentration}}{{}^{area_{standard}}/_{concentration_{standard}}}$$

Conclusions

The Polyarc microreactor can determine CBD isolate purity either in peak area percent or relative ppm within 20 minutes, requiring only a simple dilution in undecane or similar hydrocarbon. Furthermore, no complex standard is required for quantitation. Heptane was used to as an internal standard spike for same injection quantification.

Contact Us

For more information or to purchase a Polyarc® system, please contact us at 612-787-2721 or contact@activatedresearch.com.

Please visit our website for details and additional technical literature.

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