

# Tetrahydrocannabinol (THC) in Saliva

## Mass-Spectrometer

The National Institute on Drugs of Abuse [NIDA] and The Substance Abuse and Mental Health Services Administration [SAMSHA] determine appropriate detection levels for GCMS confirmation. The levels vary depending on sample matrix and target compound. One of the most challenging GCMS applications is the determination of THC in saliva. Detection for THC at low ppb [ng/ml] levels is required due to several factors, including: 1) rapid elimination of THC from saliva from time of exposure, 2) low amount of drug for 'active' dose, and 3) small sample volume available at time of collection.

This application demonstrates an example of how the Shimadzu GCMS QP-2010 can provide the instrument performance necessary to complete this analysis.

## Experimental

### Equipment

This analysis was performed using the Shimadzu QP-2010 Mass-Spectrometer, the standard GC-2010 Gas Chromatograph with Advanced Flow Control and using a high-pressure injection technique, and the AOC-20i/20s auto injector and sample tray.

Using the standard SPL-2010 split/splitless injection port on the GC-2010 as a high-pressure injector, the Method was performed with a splitless injection using a Restek 3.4mm. id. SILTEK liner. These results were obtained using a 15m X 0.25mm X .25 um phase DB-IMS column.

### Standards

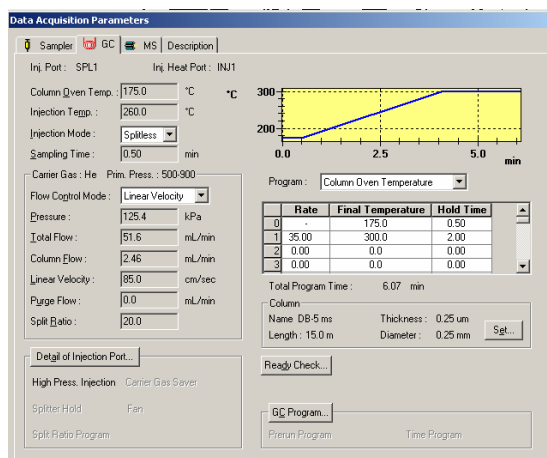
The Standards were prepared from spiked saliva matrix, extracted as 0.400ml and dried. Extract were then reconstituted and derivatized with 40ul BSTFA to form the trimethylsilane derivative [TMS] of THC. The concentrations used for the 0.400ml extracted sample volumes were 0.100ppb – 2.50ppb. Tri-deuterated THC-d3 was spiked at 1.25ppb into all samples as internal standard used for quantification. 1.0ul injections were made for all sample extracts analyzed and a splitless sampling time of 0.5 minutes was used to load THC onto the column. The calibration curve was run and the results processed to determine the response and linearity for the range stated.

The QP-2010 method file setup specified that the mass filter was to operate in SIM mode, and data acquired at mass 371.20, 386.25 and 303.15 was used for THC detection; mass 374.30 and 389.25 were used to detect THC-D3 internal standard. The dwell time was set to 40 ms per ion. The detector gain was set to 0.5kV offset from tune result, providing detector gain of 1.35kV.

## GC

The GC-2010 was set up with a single-rate temperature profile and the Advanced Flow Control was programmed to utilize constant linear velocity after the programmed high-pressure injection and splitless sample loading time. Using high-pressure injection of 175 kPa, fast temperature ramping of 35 C/min and high column flow rate, the peaks eluted from the column into the mass spectrometer ion source in less than 3.00 minutes. Additionally, the programmable septum purge flow was initially set to 0.0ml/min flow for true splitless sampling. The septum purge was then programmed to 3.0 and split flow opened to a 20:1 ratio at 0.50 minutes.

## GC Control Display



# Results

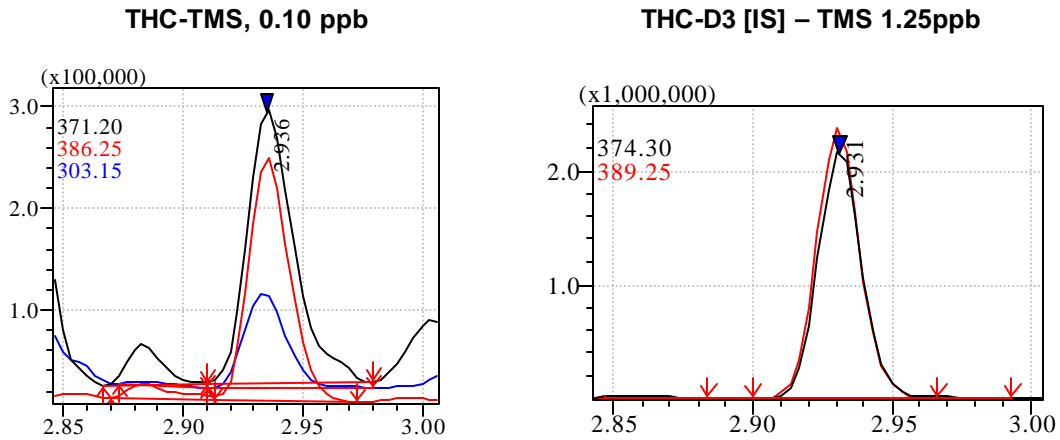
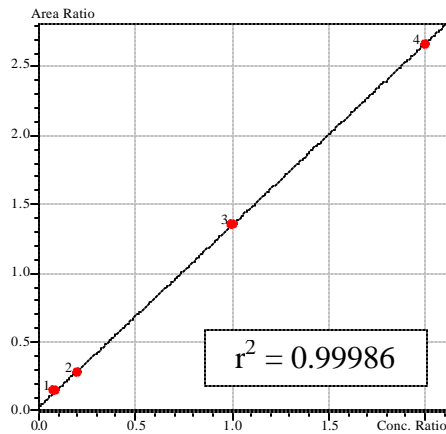


Figure 1: Chromatogram of 0.10ppb calibration standard and calibration for THC-Saliva Method on the QP-2010 GC/MS



AREA COUNT COMPARISON	
0.10ppb	QP-2010
THC-D3 [IS]	2410896
THC	362088
Detector gain	1.35 kV

Figure 2: Calibration result for THC-Saliva Method on the Shimadzu QP-2010 GC/MS

With one 1uL injected into the GCMS, the expected detection limit for this compound is 0.01ppb using the same method shown. For lower levels, adjusting the detector gain up to 1.7kV would increase the signal without significantly increasing the noise level of the system. Detection at this level is characterized by 3/1 s/n for the ion of quantitation with isotopic ratios consistent with calibrated amounts. When used for higher levels, the system needs to be recalibrated for the new detection levels. For detections of 0.05ppb and above, only 100uL of initial sample volume would be needed.