LCMS QP-8000

On-Column Detection Limit for Testosterone by APCI LC/MS

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Introduction

Atmospheric Pressure Chemical Ionization (APCI) LC/MS offers users a highly sensitive and specific means for the determination of non-polar and semi-polar analytes including steroids such as testosterone.

This brief investigation demonstrates the ability of the Shimadzu QP-8000 for the rapid development of a high sensitivity LC/MS method for testosterone. Furthermore, these data were produced by the students in the Technical Support Training Class given in August of 1999. This underscores the ease of method development utilizing this LC/MS instrument.

Method

Testosterone was obtained from Sigma Chemical Co. (St. Louis, MO) and was serially diluted in 75:25 methanol:water, 1% formic acid yielding solutions with concentrations of 10 pg/µL, 1 pg/µL, and 100 fg/µL. The LCMS QP-8000 was tuned by flow injection of these testosterone solutions with a manual injection valve.

After sensitivity optimization, a reversed phase, C-18 column, 2 mm i.d. X 10 cm, Shimadzu Shim-Pack was connected to the SIL 10ADvp autosampler and injections were made of the 100 fg/µL testosterone solution. The mobile phase used was 75:25 methanol:water 1% formic acid.

Results and Discussion

Figure 1 illustrates the result of a triplicate injection of 10 µL of the 100 fg/µL testosterone solution, or 1 pg per injection on-column. The signal-to-noise ratio, S/N, was 25 at 3σ and the RMS S/N was 41.

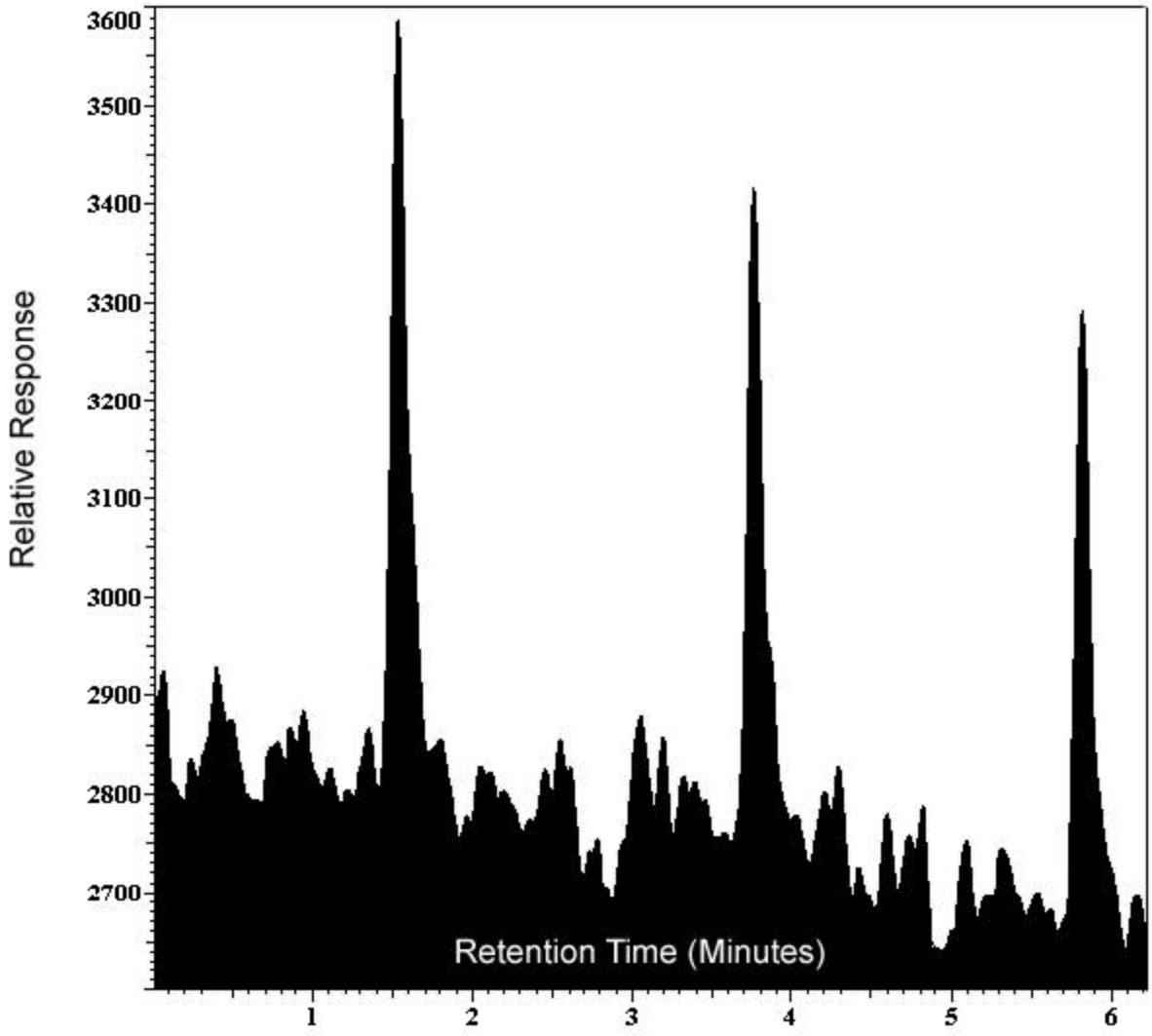

Figure 1. Triplicate Injection of 1 pg Testosterone On-Column