



ANALYSIS OF SILDENAFIL CITRATE IN VIAGRA AND AN OFFSHORE FORMULATION

INTRODUCTION

Due to the popularity of Pfizer's Viagra for the treatment of erectile dysfunction, several alternative sources of sildenafil citrate (the active ingredient in Viagra) have become available on the internet. Several companies based in Belize sell sildenafil citrate tablets manufactured offshore. There is also recent work in which dietary supplements in China were analyzed to determine if they were adulterated with sildenafil citrate (1). The extraction procedure used in this work was conducted on a Pfizer Viagra tablet and a sildenafil citrate tablet procured on the internet. Both tablets were supposed to contain 100 mg of active ingredient. Both extracts were analyzed by High Temperature Liquid Chromatography (HTLC) using a thermal gradient.

EXPERIMENTAL CONDITIONS

The extraction protocol was based on work in (1). Each tablet was ground in a mortar and pestle and the ground sample was placed in a 40 mL vial. Each sample was extracted with 30 mL of 50:50 methanol:water by sonicating the vials for 20 min. The samples were then centrifuged at 3000 rpm for 15 minutes. The supernatant was collected and transferred to 100 mL Class A volumetric flasks. The extracts were diluted to the mark with 50:50 methanol:water and filtered through 0.45 μm nylon prior to HTLC analysis.

HTLC conditions are summarized in Table 1. A Milton Roy CM4000 pump, Alltech vacuum degasser, Thermo Separations UV2000 variable wavelength detector and Alcott autosampler were used in conjunction with a Selerity Technologies Series 8000 programmable HTLC oven.

RESULTS AND CONCLUSIONS

Figure 1 shows the analysis of sildenafil citrate in Viagra (blue) and the offshore tablet (red).

To determine the level of sildenafil citrate in the offshore tablet, peak areas of the two analyses were compared and corrected for the amount of tablet used. It was assumed that the 100 mg Viagra tablet contained 100 mg of sildenafil citrate. Using this method, it was determined that the offshore tablet only contained about 80 mg of sildenafil citrate, although it was sold as having 100 mg of active ingredient. Note that there is no evidence of degradation of the active ingredient by performing the analysis at elevated temperature.

TABLE 1 : HTLC CONDITIONS FOR ANALYSIS OF SILDENAFIL CITRATE	
COLUMN:	SELERITY BLAZE C ₈ , 100 x 4.6 mm, 3 μm
MOBILE PHASE:	35:65 ACETONITRILE:WATER WITH 0.1% TFA
FLOW:	1.0 mL/MIN
DETECTION:	UV @ 220 nm
INJECTION:	5 μL
TEMPERATURE:	40°C (HOLD TWO MINUTES) RAMP TO 100°C AT 15°/MIN, HOLD SIX MIN

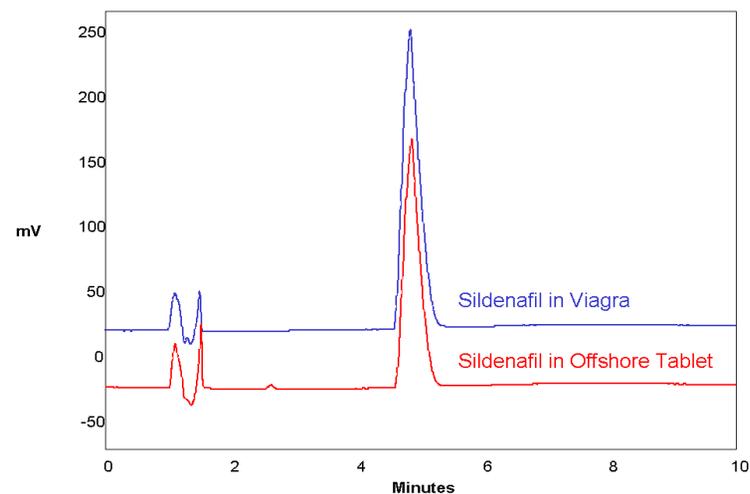


FIGURE 1 :
Separation of sildenafil citrate in Viagra and an offshore tablet using a thermal gradient from 40°C to 100°C.

REFERENCE

(1) Tseng and Lin, *J. of Food and Drug Analysis*, 10, 112-119 (2002)