

EXPERT RESEARCH PROTOCOL

from 08/22/2015

Code: 15-08-20-1 (102)
Customer: [REDACTED]
Type of analysis: Qualitative analysis of the sample, determination of purity
Methods: Agilent 1200, High-performance liquid chromatography (HPLC);
Column: Zorbax SB-C18 150 mm×2.1 mm, 3 mkm;
Detector – DAD, wavelength – 245, 225, 254 nm;
Detector – MSD, ionization method APCI Positive, ESI Positive, SCAN (100-500 m/z)
Number of samples: 1
Subject: Halotestine (Fluoxymesterone)

Mobile phase: A — MeOH (70%), B - H₂O (30%).

The elution mode is isocratic.

The flow rate through a column: 0.3 ml/min. Thermostat: 30°C.

Single quadrupole mass analyzer is used for identification of the chemical elements. The samples were ionized at the electrostatic spraying (ESI) and atmospheric pressure with chemical ionization (APCI) mode with fixed positive and negative ions.

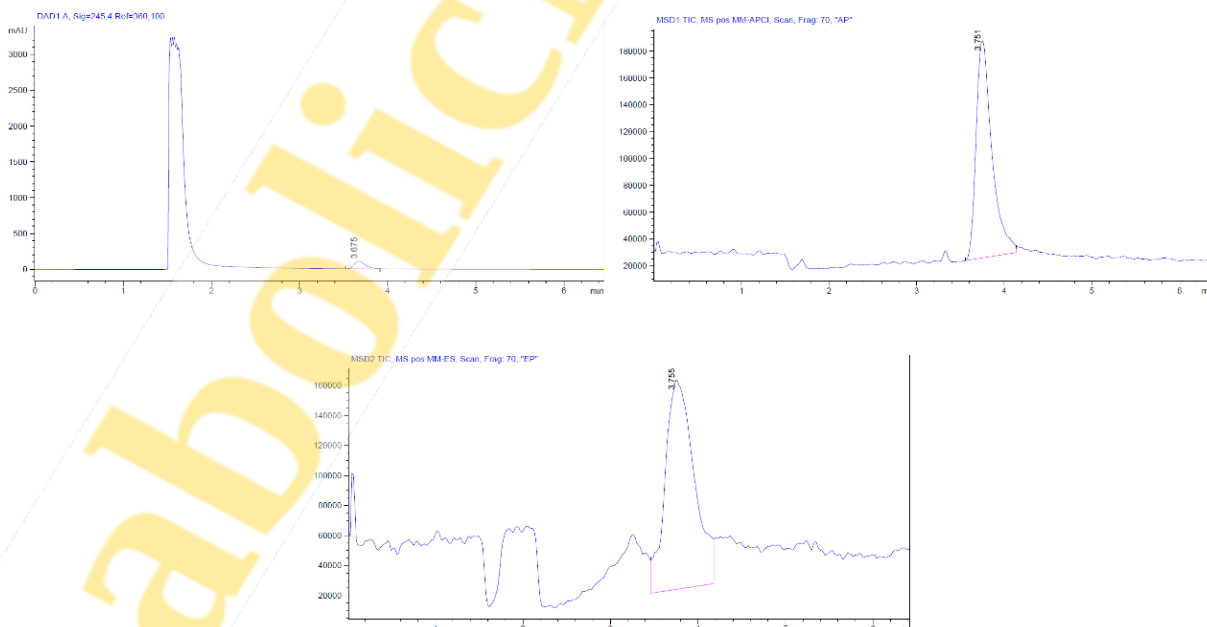


Fig.1. The component output chromatogram of the sample, detector DA, MS in AP, MS in EP

Tbl. 1. The calculation results of the peak areas on Fig. 1

Area Percent Report						
Sorted By	:	Signal				
Multiplier	:	1.0000				
Dilution	:	1.0000				
Use Multiplier & Dilution Factor with ISTDs						
Signal 1: DAD1 A, Sig=245,4 Ref=360,100						
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.675	BB	0.1334	920.28729	104.87843	100.0000
Totals :				920.28729	104.87843	
Signal 2: MSD1 TIC, MS File						
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	3.751	BB	0.1820	2.02863e6	1.62292e5	100.0000
Totals :				2.02863e6	1.62292e5	
Signal 3: MSD2 TIC, MS File						
Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	3.755	VV	0.2945	3.22673e6	1.40113e5	100.0000
Totals :				3.22673e6	1.40113e5	

The analysis results of the received peaks by detector DA are shown in Fig. 2

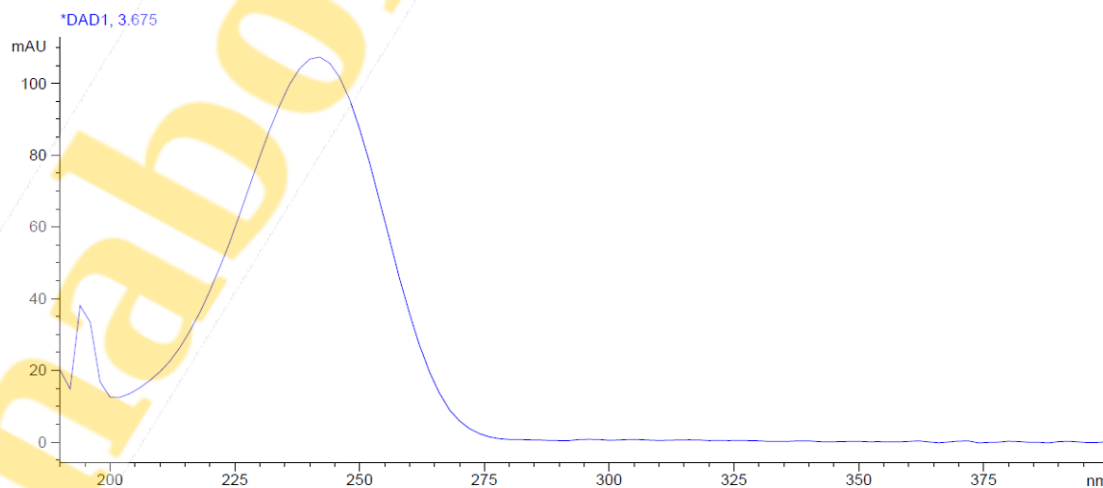


Fig. 2. The analysis of the peak 1, DA detector

The analysis results of the received peaks by detector MS are shown on Fig. 3



Fig. 3. The analysis of the peak 1, MS detector (modes AP and EP)

The received data of the analytical comparison results of MS detectors with the calculated data on the test substance allow us to state that peak 1 refers to Fluoxymesterone (mw 336.441).

The chromatographic purity of Fluoxymesterone by MS and DA detectors is 98%

Remarks:

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