

EXPERT RESEARCH PROTOCOL

from 08/22/2015

Code: 15-08-20-1 (109)
Customer:
Number of samples: 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 – 240, 225, 254 nm;
Detector – MSD, ionization method APCI Positive/Negative, SCAN (100 - 500 m/z)
Number of samples: 1
Subject: Nandrolone decanoate

The solvent for the sample: Propanol/Chloroform (1/1).

Mobile phase: A - MeOH (93%), B - H₂O (7%).

The elution mode is isocratic.

The flow rate through the column: 0.3 ml/min. A thermostat: 30°C.

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

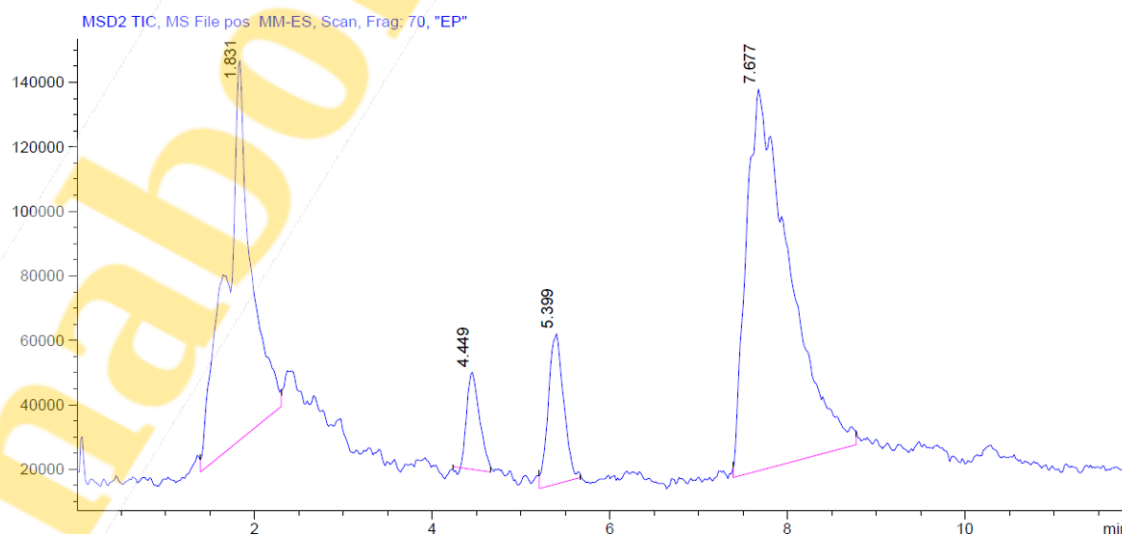


Fig.1. The component output chromatogram of the sample, detector MS in EP

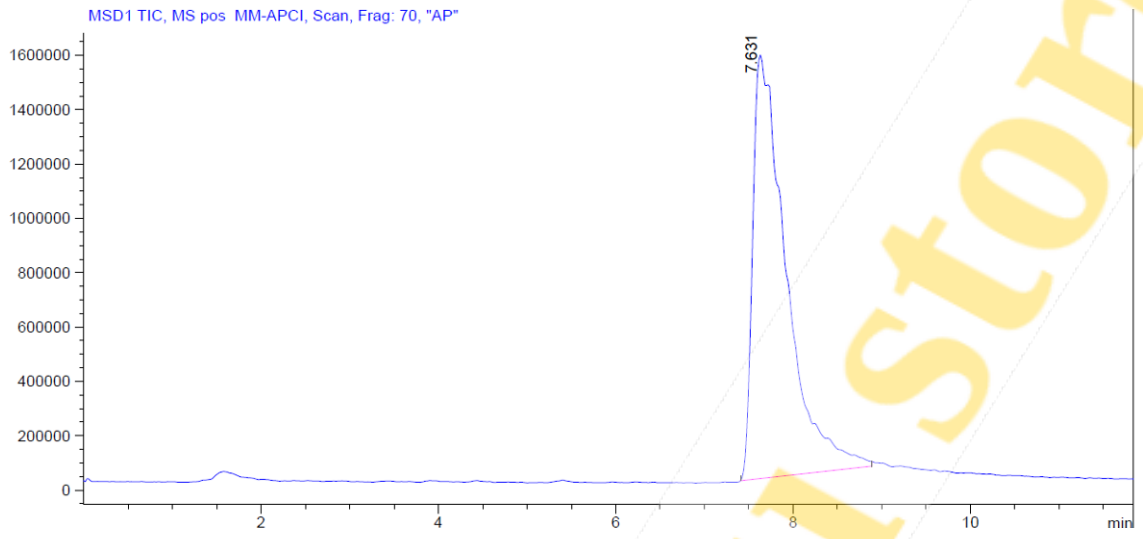


Fig.2. The component output chromatogram of the sample, detector MS in AP

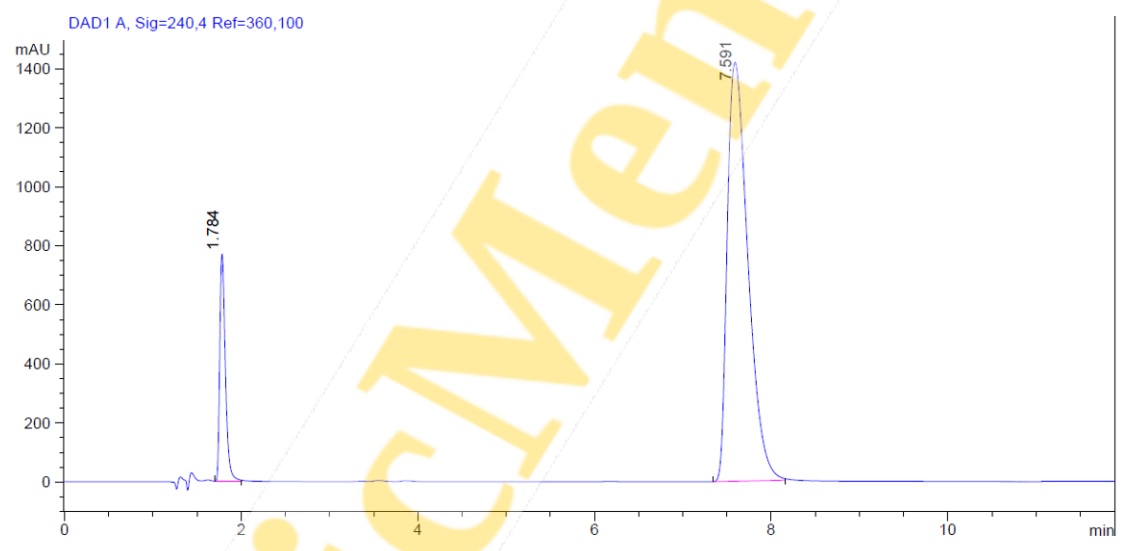


Fig.3. The component output chromatogram of the sample, detector DA

Tbl. 1. The calculation results of the peak areas on Fig. 1-3

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                          Area Percent Report
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution      :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: MSD1 TIC, MS File
Peak RetTime Type Width Area Height Area
# [min] [min] [min] [min] [%]
-----|-----|-----|-----|-----|-----
1 7.631 BB 0.3367 4.08562e7 1.55908e6 100.0000
Totals : 4.08562e7 1.55908e6
  
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Signal 2: MSD2 TIC, MS File

Peak #	RetTime [min]	Type	Width [min]	Area	Height	Area %
1	1.831	MM	0.3248	2.31901e6	1.18998e5	32.1863
2	4.449	MM	0.1679	3.05549e5	3.03288e4	4.2408
3	5.399	MM	0.1992	5.60087e5	4.68656e4	7.7737
4	7.677	MM	0.5645	4.02030e6	1.18694e5	55.7992

Totals : 7.20494e6 3.14887e5

Signal 3: DAD1 A, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.784	BB	0.0684	3389.02734	769.10114	12.8364
2	7.591	BB	0.2501	2.30126e4	1419.83484	87.1636

Totals : 2.64017e4 2188.93597

The analysis results of the received peaks by detector DA are shown on Fig. 4

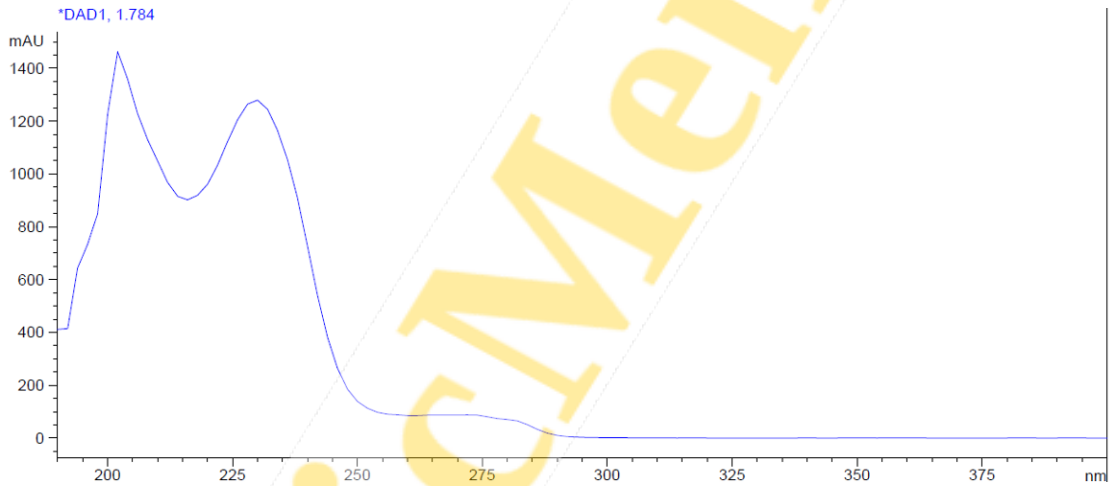


Fig. 4. The analysis of the peak 1, DA detector

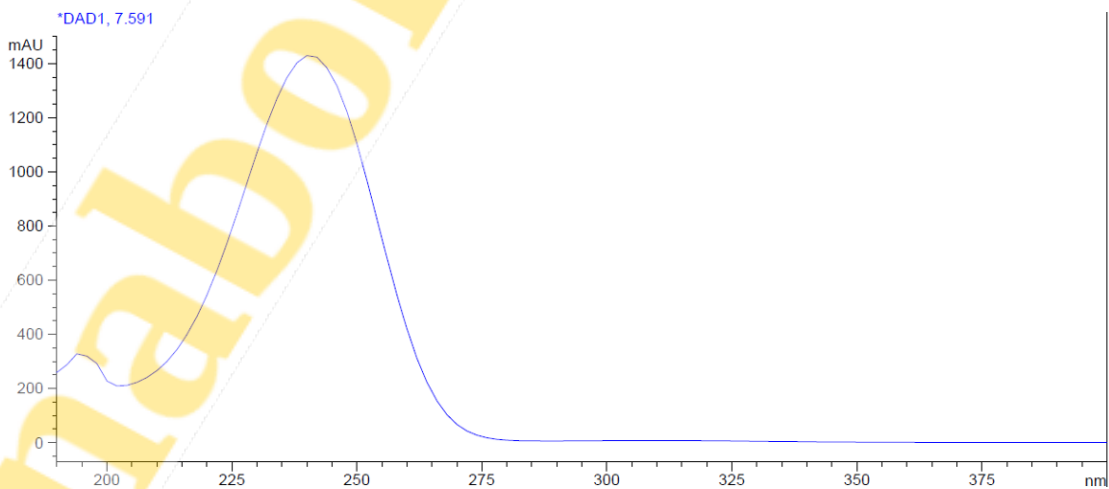


Fig. 5. The analysis of the peak 2, DA detector

The analysis results of the received peaks by detector MS are shown on Fig. 6-7

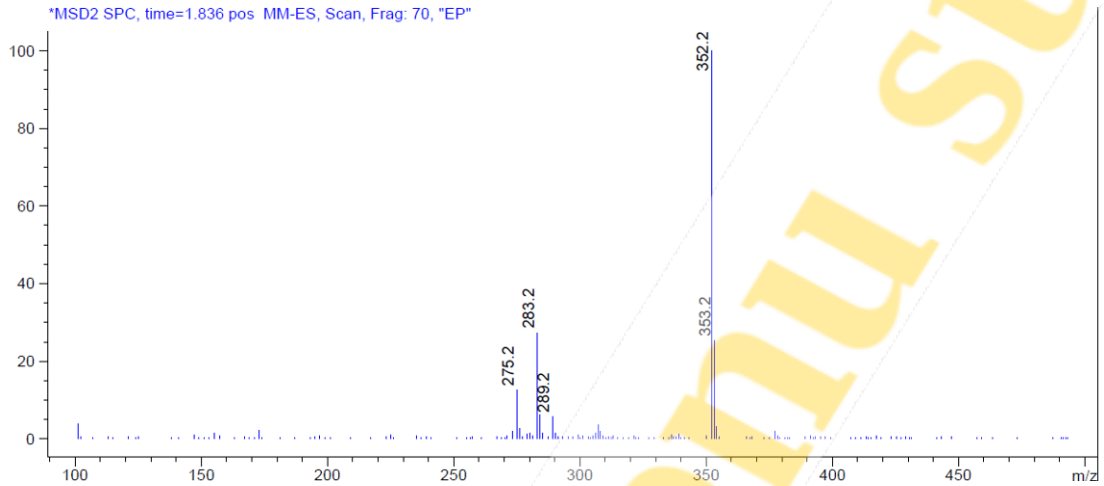


Fig. 6. The analysis of the peak 1, MS detector (mode EP)

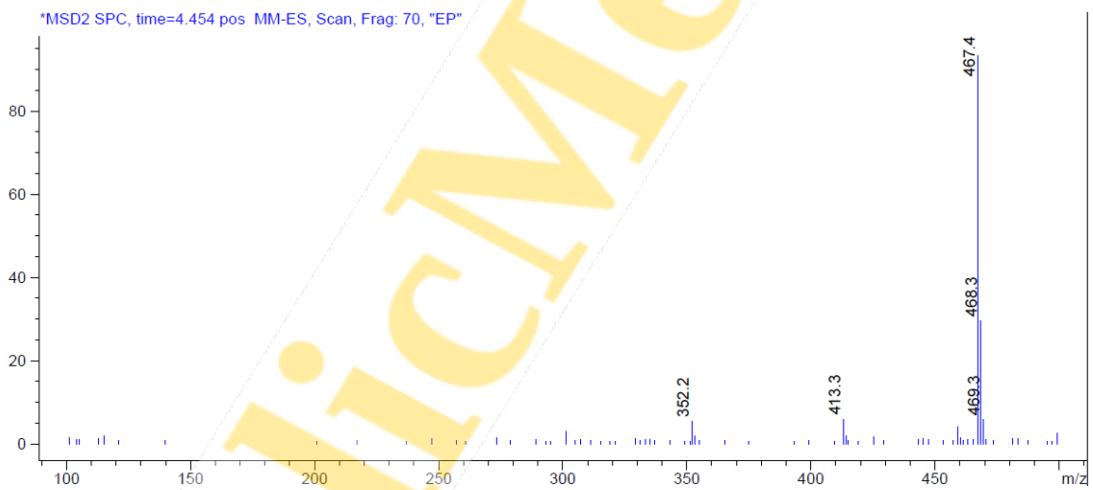


Fig. 6. The analysis of the peak 2, MS detector (mode EP)

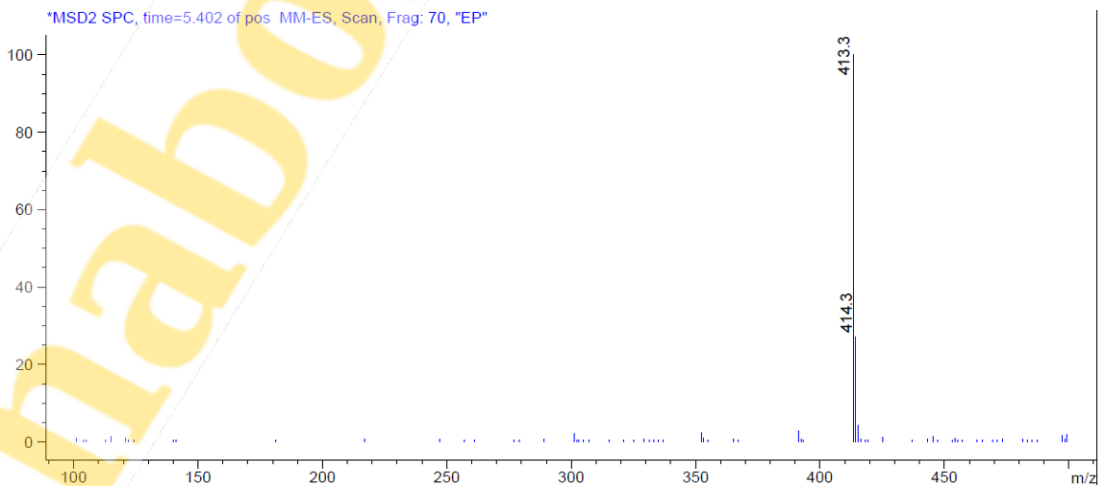


Fig. 6. The analysis of the peak 3, MS detector (mode AP)

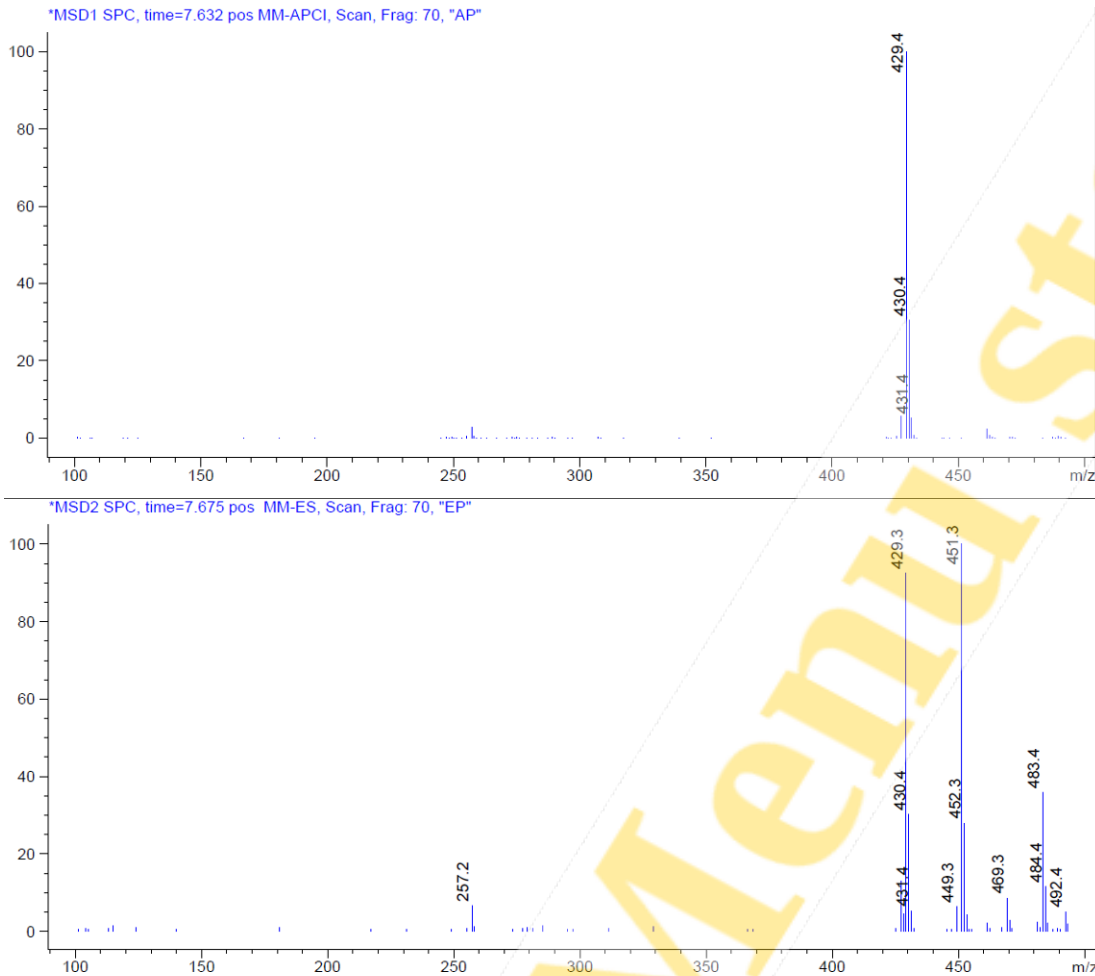


Fig. 7. The analysis of the peak 4 and 1, MS detector (mode EP and AP)

The received data of the analytical comparison results of MS and DA detectors with the calculated data on the test substance allow us to state that peak 2 refers to Nandrolone decanoate (mw 428.6). The chromatographic purity of Nandrolone decanoate by DA detectors is 87.2%.

Remarks:

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