

SECTION 2
TECHNICAL PART

2.4 Confirmation analysis by GC/C/IRMS

2.4.1 GC/MS analysis

TOPLEVEL PARAMETERS

Method Information For: C:\MSDCHEM\1\METHODS\MAN_52.M
Method Sections To Run:

- () Save Copy of Method With Data
- () MSTOP Pre-Run Cmd/Macro =
- () Instrument Control Pre-Run Cmd/Macro =
- () Data Analysis Pre-Run Cmd/Macro =
- (X) Data Acquisition
- (X) Data Analysis
- () MSTOP Post-Run Cmd/Macro =
- () Instrument Control Post-Run Cmd/Macro =
- () Data Analysis Post-Run Cmd/Macro =

Method Comments:
This is the default method

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

6890 GC METHOD

OVEN

Initial temp: 70 'C (On) Maximum temp: 325 'C
Initial time: 1.00 min Equilibration time: 0.50 min
Ramps:
Rate Final temp Final time
1 30.00 270 12.00
2 10.00 300 3.00
3 0.0(Off)
Post temp: 70 'C
Post time: 0.00 min
Run time: 25.67 min

FRONT INLET (SPLIT/SPLITLESS)

Mode: Splitless
Initial temp: 280 'C (On)
Pressure: 145.0 kPa (On)
Purge flow: 20.0 mL/min
Purge time: 2.00 min
Total flow: 25.4 mL/min
Gas saver: Off
Gas type: Helium

BACK INLET (UNKNOWN)

COLUMN 1

Capillary Column
Model Number: Agilent 19091s-433
Max temperature: 325 'C
Nominal length: 30.0 m
Nominal diameter: 250.00 um
Nominal film thickness: 0.25 um
Mode: constant pressure
Pressure: 145.0 kPa
Nominal initial flow: 2.3 mL/min
Average velocity: 56 cm/sec
Inlet: Front Inlet
Outlet: MSD
Outlet pressure: vacuum

COLUMN 2
(not installed)

Method: MAN_52.M

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FRONT DETECTOR ()

BACK DETECTOR ()

SIGNAL 1
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

SIGNAL 2
Data rate: 20 Hz
Type: test plot
Save Data: Off
Zero: 0.0 (Off)
Range: 0
Fast Peaks: Off
Attenuation: 0

COLUMN COMP 1
(No Detectors Installed)

COLUMN COMP 2
(No Detectors Installed)

THERMAL AUX 2
Use: MSD Transfer Line Heater
Description:
Initial temp: 300 'C (On)
Initial time: 0.00 min
Rate Final temp Final time
1 0.0(Off)

POST RUN
Post Time: 0.00 min

TIME TABLE
Time Specifier

Parameter & Setpoint

7673 Injector

Front Injector:
Sample Washes 0
Sample Pumps 3
Injection Volume 1.0 microliters
Syringe Size 10.0 microliters
PostInj Solvent A Washes 3
PostInj Solvent B Washes 3
Viscosity Delay 3 seconds
Plunger Speed Fast
PreInjection Dwell 0.00 minutes
PostInjection Dwell 0.00 minutes

Back Injector:
No parameters specified
Column 1 Inventory Number : ?
Column 2 Inventory Number :

MS ACQUISITION PARAMETERS

General Information

Tune File : atune.u
Acquisition Mode : Scan

MS Information

Solvent Delay : 9.00 min
EM Absolute : False
EM Offset : 0
Resulting EM Voltage : 1811.8

[Scan Parameters]

Low Mass : 50.0

Method: MAN_52.M

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igh Mass : 550.0
hreshold : 150
Sample # : 2 A/D Samples 4
lot 2 low mass : 50.0
lot 2 high mass : 550.0

[MSZones]

S Quad : 150 C maximum 200 C
S Source : 230 C maximum 250 C

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\MSDCHEM\1\METHODS\MAN_52.M

Percent Report Settings

Sort By: Signal

Output Destination
Screen: No
Printer: Yes
File: No

Integration Events: AutoIntegrate

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search Minimum Quality
C:\temp\IRMS.L 90

Integration Events: AutoIntegrate

Report Type: Summary

Output Destination
Screen: No
Printer: Yes
File: No

Generate Report During Run Method: No

Quantitative Report Settings

Method: MAN_52.M

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Report Type: Summary

Output Destination

Screen: Yes
Printer: No
File: No

Generate Report During Run Method: No

Calibration Last Updated:

Reference Window: 10.00 Percent
Non-Reference Window: 5.00 Percent
Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

*** Empty Quantitation Database ***

END OF DATA ANALYSIS PARAMETERS

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Method: MAN_52.M

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Additional Information for STER1FS.M
File created Fri Mar 05 17:08:58 2004

Method : C:\MSDCHEM\1\METHODS\DEFAULT.M
Renamed: C:\MSDCHEM\1\METHODS\STER1FS.M
Fri Mar 05 17:08:58 2004

Method : C:\MSDCHEM\1\METHODS\STER1FS.M
Renamed: C:\MSDCHEM\1\METHODS\STER1FSKETO.M
Tue Apr 20 14:52:43 2004

Method : C:\MSDCHEM\1\METHODS\STER1FS.M
Renamed: C:\MSDCHEM\1\METHODS\MAN_52.M
Wed Nov 09 18:43:55 2005

Method: MAN_52.M

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USADA 0128

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Sequence Name: C:\MSDCHEM\1\sequence\2006\JUL106\2307.S

Comment:

Operator: 49

Data Path: D:\MSD22\JUL106\2307\

Top Pre-Seq Cmd:
Instrument Control Pre-Seq Cmd:
Data Analysis Pre-Seq Cmd:

Top Post-Seq Cmd:
Instrument Control Post-Seq Cmd:
Data Analysis Post-Seq Cmd:

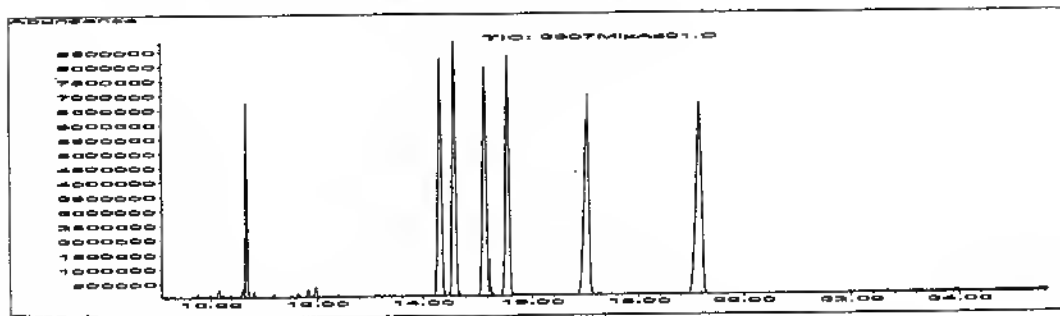
Method Sections To Run On A Barcode Mismatch
(X) Full Method (X) Inject Anyway
() Reprocessing Only () Don't Inject

Line Sample Name/Misc Info
1) Calibration 1 2307MixAc01
Datafile
Method MAN_52
2) Blank 2 blu1F3 MAN_52 Blu 1 F3
3) Sample 3 17807474F3
Datafile
Method MAN_52
4) Blank 4 blu1F1 MAN_52 Blu 1 F1
5) Sample 5 17807474F1
Datafile
Method MAN_52
6) Blank 6 blu1F2 MAN_52 Blu 1 F2
7) Sample 7 17807474F2
Datafile
Method MAN_52
8) Sample 7 17807474F2b
Datafile
Method MAN_52

Séquence vérifiée par :49.....

Remarques :

Data File Name 2307MixAc01.D
 Data File Path D:\Msd22\Juif06\2307\
 Operator 49
 Date Acquired 7/23/2006 10:19
 Acq. Method File MAN_52.M
 Sample Name Mix Ac 50
 Vial Number 1
 Misc Info Mix Acétate 001 50 ng injecté



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.69		258	10,892,705
Etiocholanolone AC	14.35	1.343	272	20,821,485
Androsterone AC	14.62	1.367	272	35,520,111
5b Androstan 3a 17b diol diAC	15.17	1.419	256	20,304,691
5a Androstan 3a 17b diol diAC	15.57	1.456	316	28,264,859
11 KetoEtiocholanolone AC	17.07	1.597	271	27,735,914
5b Pregnan 3a 20a diol diAC	19.20	1.796	284	35,625,886

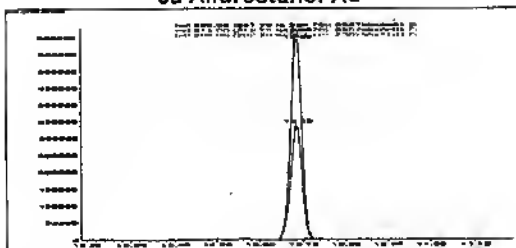
M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	11,496,221	105.5
Etiocholanolone AC	257	13,275,551	63.8
Androsterone AC	257	15,144,189	42.6
5b Androstan 3a 17b diol diAC	316	18,349,659	90.4
5a Androstan 3a 17b diol diAC	241	16,541,690	58.5
11 KetoEtiocholanolone AC	191	23,029,104	83.0
5b Pregnan 3a 20a diol diAC	269	20,053,051	56.3

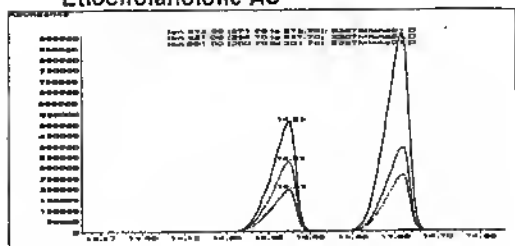
M3 signal

Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	6,139,162	56.4
Etiocholanolone AC	201	7,984,811	38.3
Androsterone AC	218	10,868,162	30.6
5b Androstan 3a 17b diol diAC	241	15,646,481	77.1
5a Androstan 3a 17b diol diAC	256	13,817,358	48.9
11 KetoEtiocholanolone AC	286	16,604,214	59.9
5b Pregnan 3a 20a diol diAC	344	9,962,084	28.0

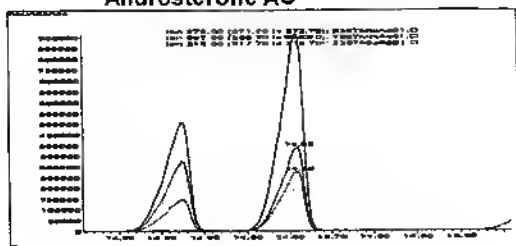
5a Androstanol AC



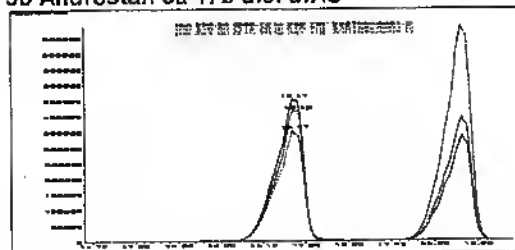
Etiocholanolone AC



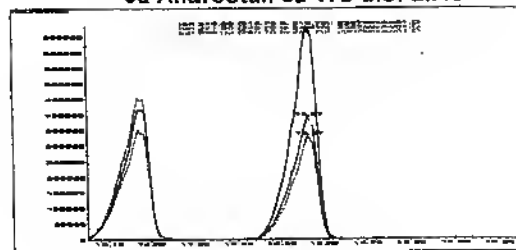
Androsterone AC



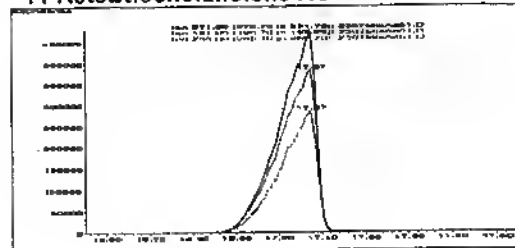
5b Androstan 3a 17b diol diAC



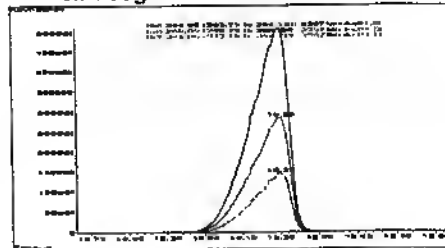
5a Androstan 3a 17b diol diAC



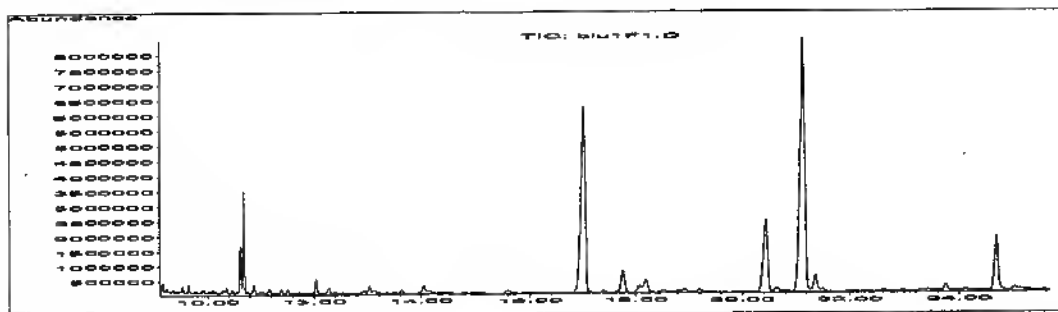
11 KetoEtiocholanolone AC



5b Pregnan 3a 20a diol diAC



Data File Name blu1F1.D
 Data File Path D:\Msd22\Juil06\2307\
 Operator 49
 Date Acquired 7/23/2006 12:10
 Acq. Method File MAN_52.M
 Sample Name Blu 1 F1
 Vial Number 4
 Misc Info Blanc urinaire 1 Pool 4 Fraction 1 dans 100µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.68		258	5,234,507
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0
11 KetoEtiocholanolone AC	17.04	1.596	271	23,056,534
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

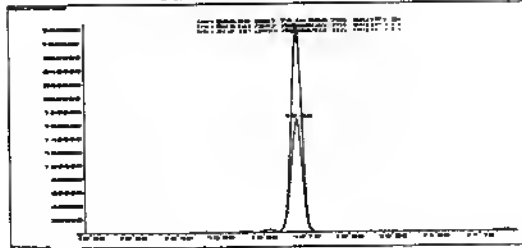
M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	5,447,955	104.1
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	0	0.0
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	19,045,346	82.6
5b Pregnan 3a 20a diol diAC	269	0	0.0

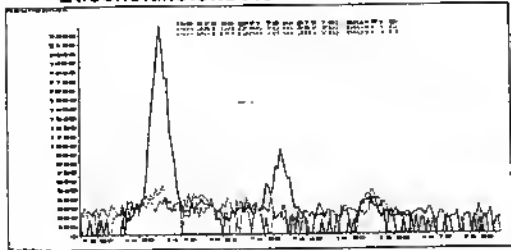
M3 signal

Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	2,943,791	56.2
Etiocholanolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC	256	0	0.0
11 KetoEtiocholanolone AC	286	13,914,444	60.3
5b Pregnan 3a 20a diol diAC	344	0	0.0

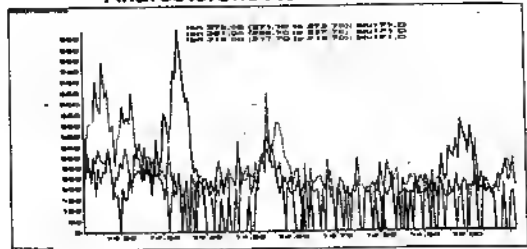
5a Androstanol AC



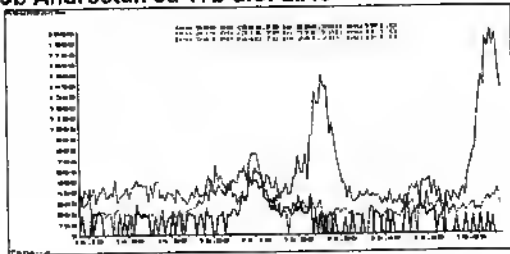
Etiocholanolone AC



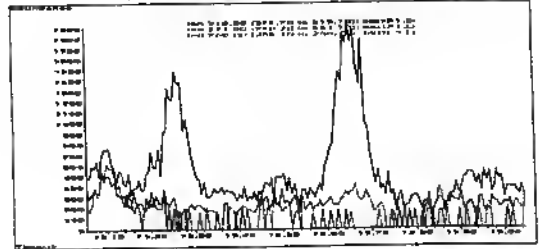
Androsterone AC



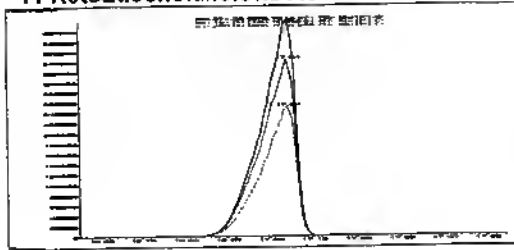
5b Androstan 3a 17b diol diAC



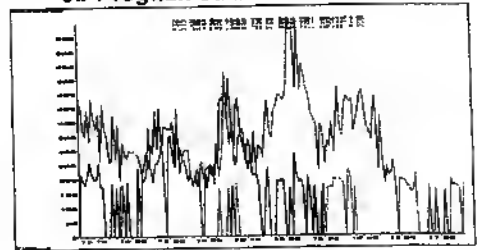
5a Androstan 3a 17b diol diAC



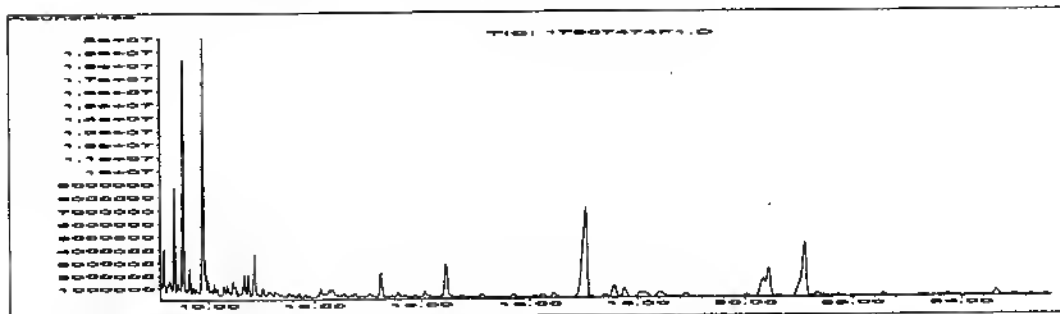
11 KetoEtiocholanolone AC



5b Pregnan 3a 20a diol diAC



Data File Name 17807474F1.D
 Data File Path D:\Msd22\Jul06\2307\
 Operator 49
 Date Acquired 7/23/2006 12:42
 Acq. Method File MAN_52.M
 Sample Name 178/07 995474 F1
 Vial Number 5
 Misc Info 178/07 995474 Fraction 1 dans 100µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.69		258	2,672,512
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0
11 KetoEtiocholanolone AC	17.05	1.596	271	27,520,882
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

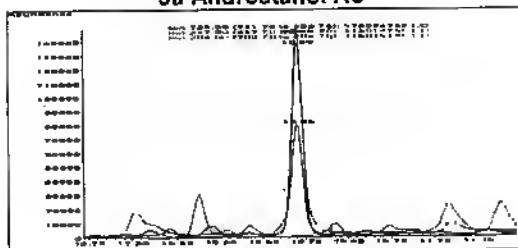
M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	2,977,015	111.4
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	0	0.0
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	22,323,512	81.1
5b Pregnan 3a 20a diol diAC	269	0	0.0

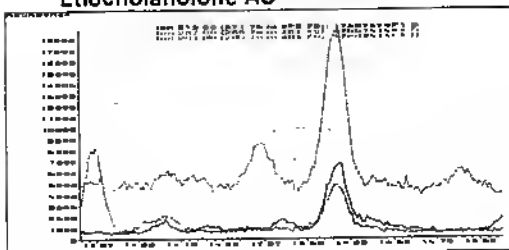
M3 signal

Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	1,719,112	64.3
Etiocholanolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC	256	0	0.0
11 KetoEtiocholanolone AC	286	16,694,242	60.7
5b Pregnan 3a 20a diol diAC	344	0	0.0

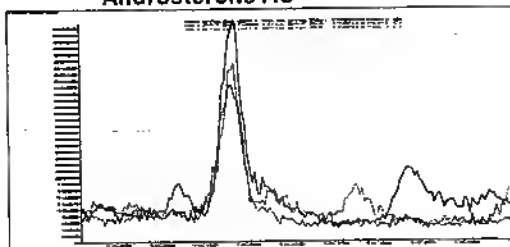
5a Androstanol AC



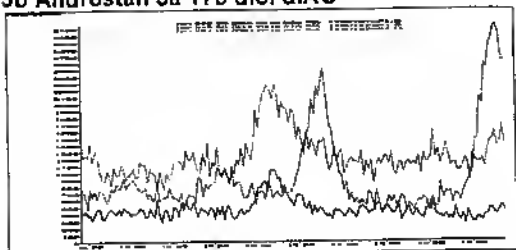
Etiocholanolone AC



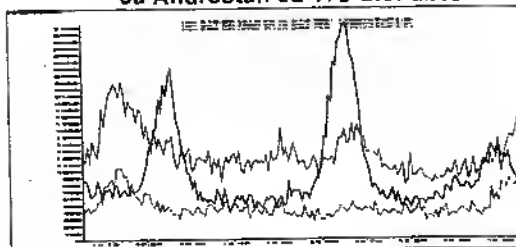
Androsterone AC



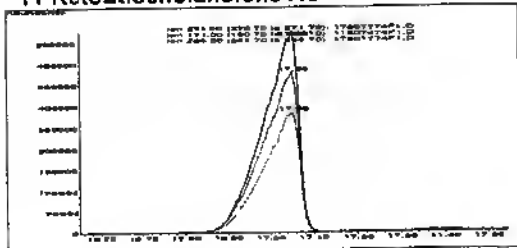
5b Androstan 3a 17b diol diAC



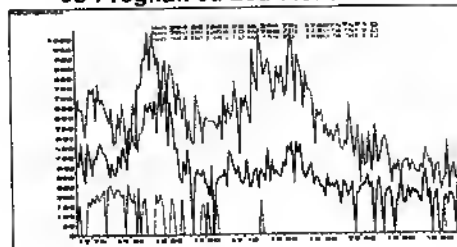
5a Androstan 3a 17b diol diAC



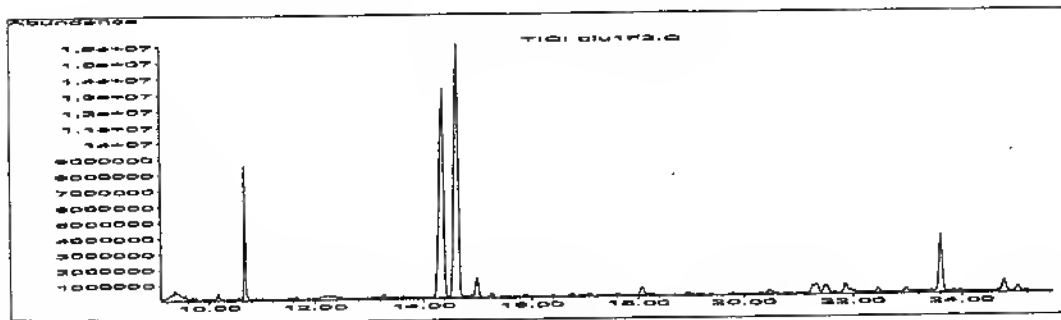
11 KetoEtiocholanolone AC



5b Pregnan 3a 20a diol diAC



Data File Name blu1F2.D
 Data File Path D:\Msd22\Jul06\2307\
 Operator 49
 Date Acquired 7/23/2006 13:15
 Acq. Method File MAN_52.M
 Sample Name Blu 1 F2
 Vial Number 6
 Misc Info Blanc urinaire 1 Pool 4 Fraction 2 dans 400µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.68		258	14,356,335
Etiocolanolone AC	14.39	1.347	272	46,218,242
Androsterone AC	14.66	1.373	272	92,159,652
5b Androstan 3a 17b diol diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0
11 KetoEtiocolanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

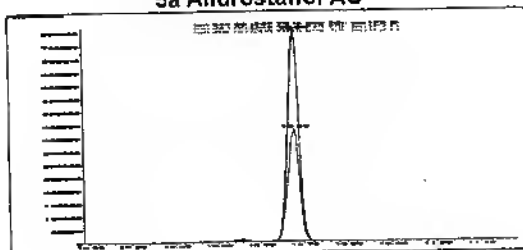
M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	14,913,181	103.9
Etiocolanolone AC	257	29,244,244	63.3
Androsterone AC	257	39,385,747	42.7
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocolanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	0	0.0

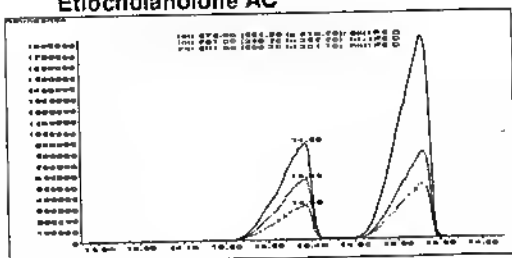
M3 signal

Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	7,892,527	55.0
Etiocolanolone AC	201	16,995,418	36.8
Androsterone AC	218	26,814,361	29.1
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC	256	0	0.0
11 KetoEtiocolanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	0	0.0

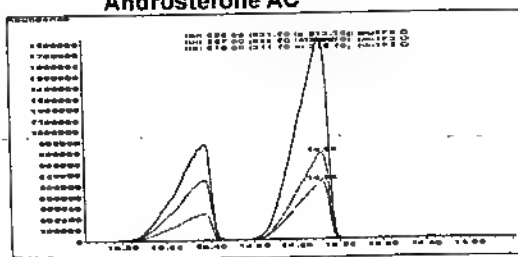
5a Androstanol AC



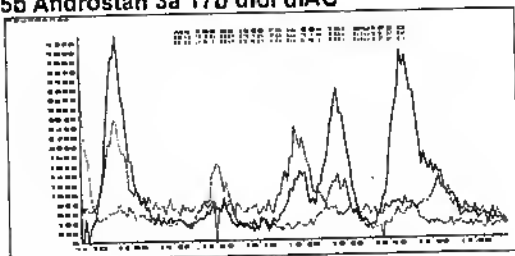
Etiocholanolone AC



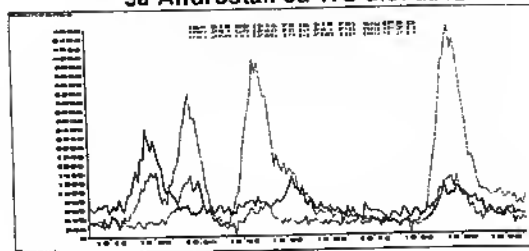
Androsterone AC



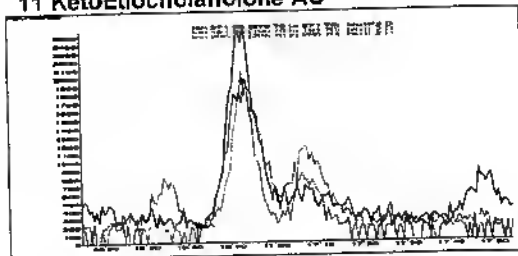
5b Androstan 3a 17b diol diAC



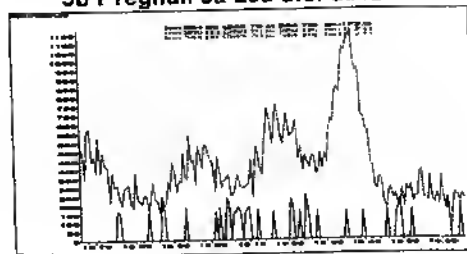
5a Androstan 3a 17b diol diAC



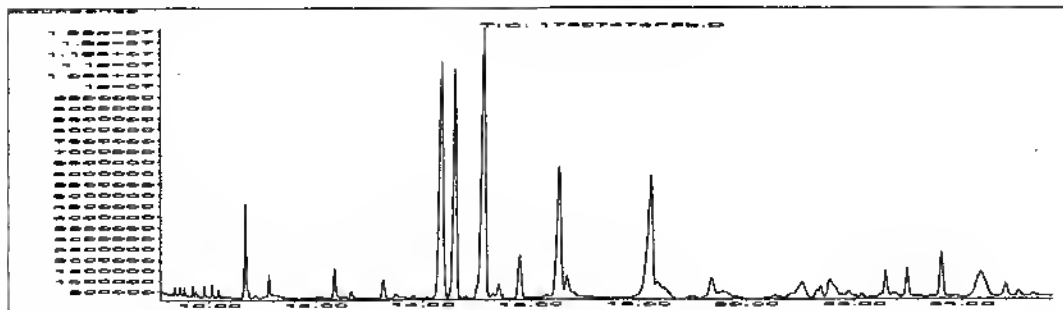
11 KetoEtiocholanolone AC



5b Pregnan 3a 20a diol diAC



Data File Name 17807474F2b.D
 Data File Path D:\Msd22\Juil06\2307\
 Operator 49
 Date Acquired 7/23/2006 14:33
 Acq. Method File MAN_52.M
 Sample Name 178/07 995474 F2
 Vial Number 7
 Misc Info 178/07 995474 Fraction 2 dans 400µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.69		258	6,790,329
Etiocholanolone AC	14.38	1.345	272	30,616,404
Androsterone AC	14.65	1.370	272	44,803,237
5b Androstan 3a 17b diol diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

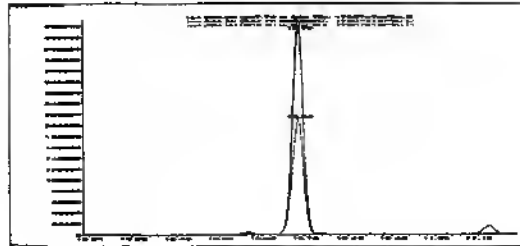
M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	7,142,048	105.2
Etiocholanolone AC	257	19,396,055	63.4
Androsterone AC	257	19,255,218	43.0
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	0	0.0

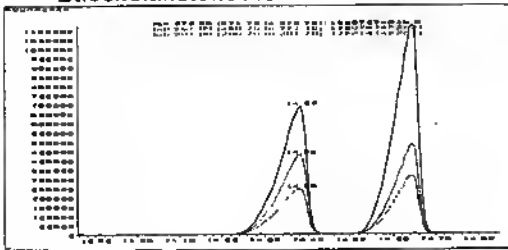
M3 signal

Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	3,868,152	57.0
Etiocholanolone AC	201	11,568,610	37.8
Androsterone AC	218	13,516,338	30.2
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC	256	0	0.0
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	0	0.0

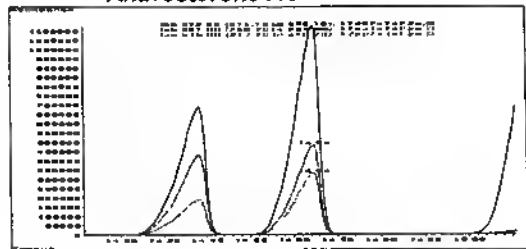
5a Androstanol AC



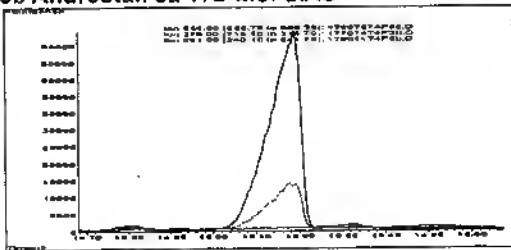
Etiocholanolone AC



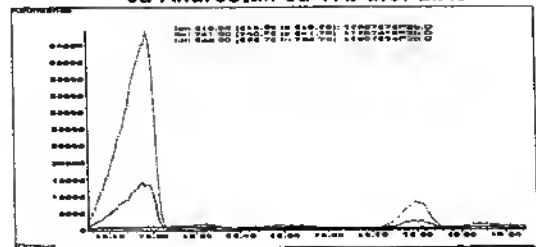
Androsterone AC



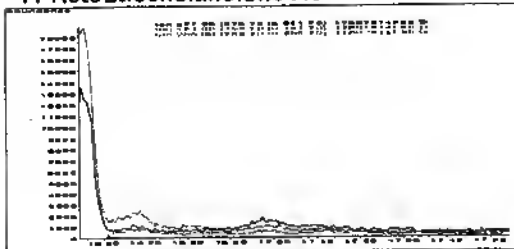
5b Androstan 3a 17b diol diAC



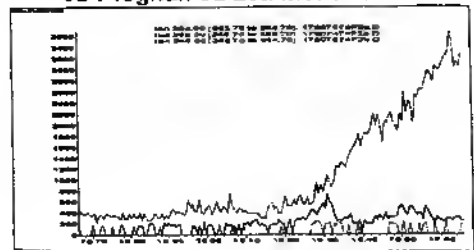
5a Androstan 3a 17b diol diAC



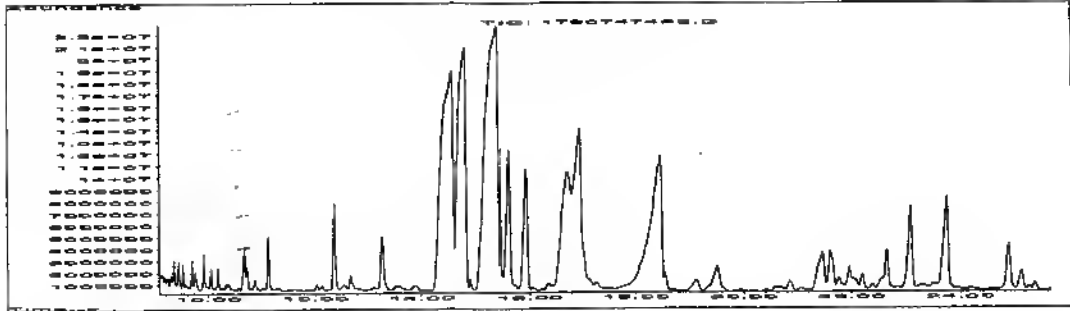
11 KetoEtiocholanolone AC



5b Pregnan 3a 20a diol diAC



Data File Name 17807474F2.D
 Data File Path D:\Msd22\Juil06\2307\
 Operator 49
 Date Acquired 7/23/2006 13:47
 Acq. Method File MAN_52.M
 Sample Name 178/07 995474 F2
 Vial Number 7
 Misc Info 178/07 995474 Fraction 2 dans 100µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.69		258	4,709,923
Etiocholanolone AC	14.58	1.365	272	169,848,822
Androsterone AC	14.84	1.388	272	213,976,918
5b Androstan 3a 17b diol diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

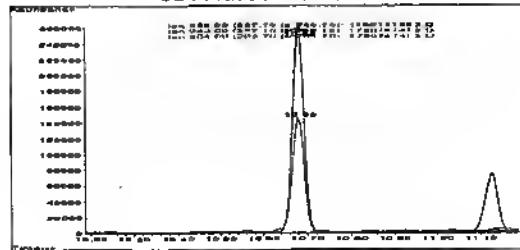
M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	5,011,709	106.4
Etiocholanolone AC	257	104,358,276	61.4
Androsterone AC	257	91,230,541	42.6
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	0	0.0

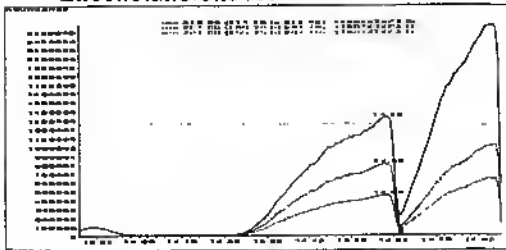
M3 signal

Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	2,896,716	61.5
Etiocholanolone AC	201	59,514,197	35.0
Androsterone AC	218	61,028,102	28.5
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC	256	0	0.0
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	0	0.0

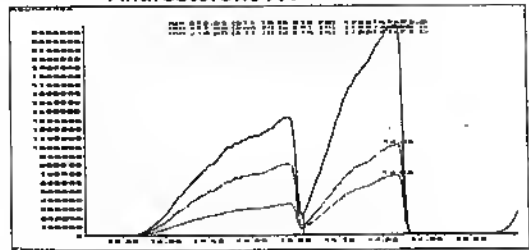
5a Androstanol AC



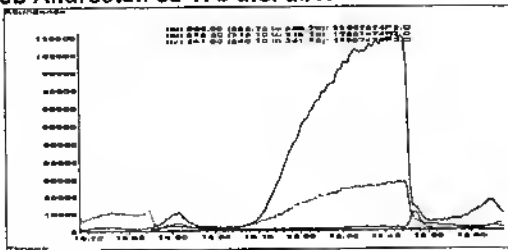
Etiocholanolone AC



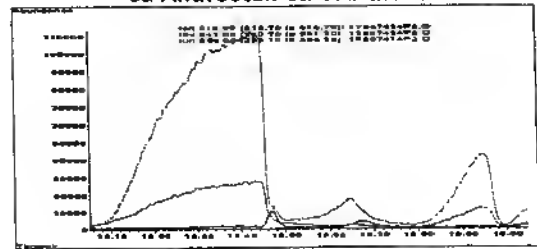
Androsterone AC



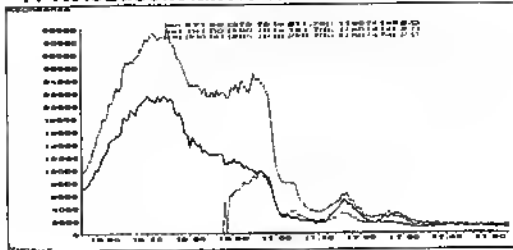
5b Androstan 3a 17b diol diAC



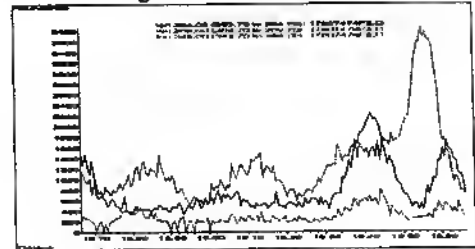
5a Androstan 3a 17b diol diAC



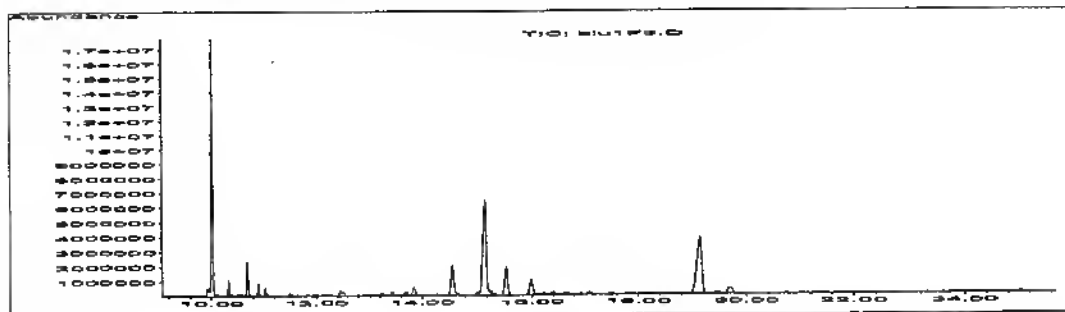
11 KetoEtiocholanolone AC



5b Pregnan 3a 20a diol diAC



Data File Name blu1F3.D
 Data File Path D:\Msd22\Juil06\2307\
 Operator 49
 Date Acquired 7/23/2006 11:00
 Acq. Method File MAN_52.M
 Sample Name Blu 1 F3
 Vial Number 2
 Misc Info Blanc urinaire 1 Pool 4 Fraction 3 dans 100µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.68		258	3,476,361
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	15.17	1.420	256	15,597,241
5a Androstan 3a 17b diol diAC	15.51	1.452	316	5,215,670
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	19.14	1.792	284	16,549,438

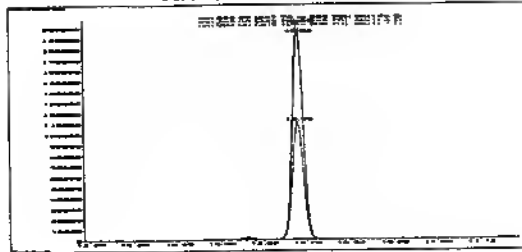
M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	3,650,999	105.0
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	0	0.0
5b Androstan 3a 17b diol diAC	316	14,170,551	90.9
5a Androstan 3a 17b diol diAC	241	3,187,095	61.1
11 KetoEtiocholanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	9,365,521	56.6

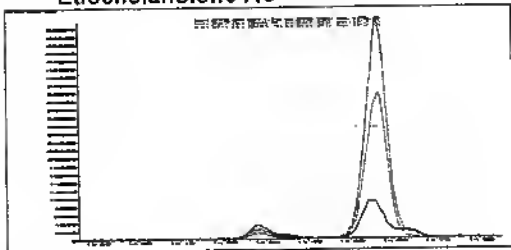
M3 signal

Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	1,954,169	56.2
Etiocholanolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	12,280,836	78.7
5a Androstan 3a 17b diol diAC	256	2,634,418	50.5
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	4,508,073	27.2

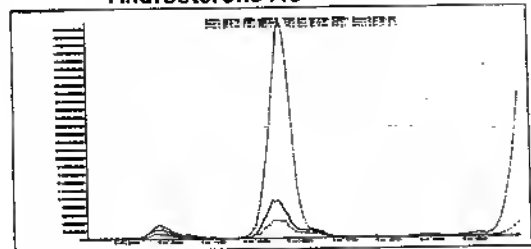
5a Androstanol AC



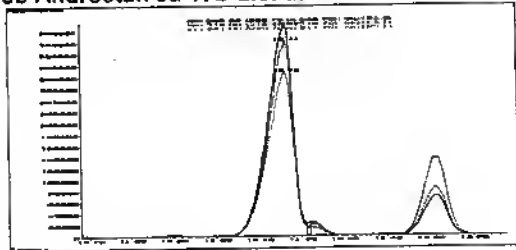
Etiocholanolone AC



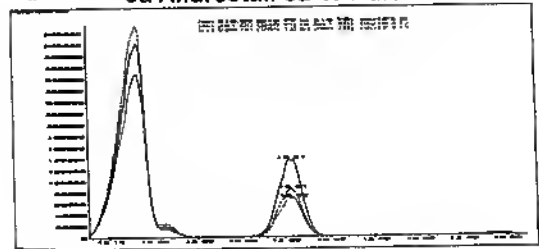
Androsterone AC



5b Androstan 3a 17b diol diAC



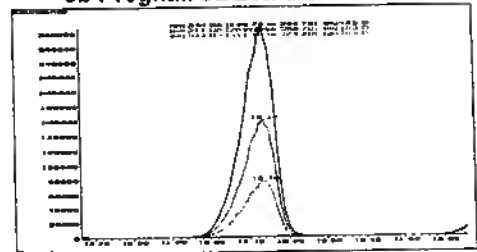
5a Androstan 3a 17b diol diAC



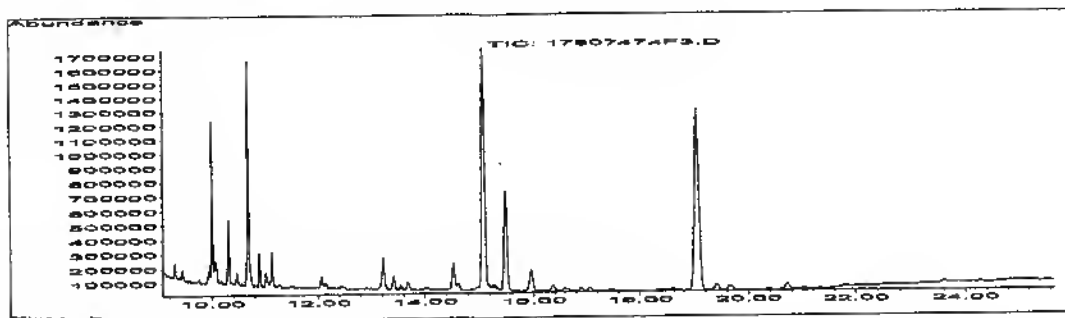
11 KetoEtiocholanolone AC



5b Pregnan 3a 20a diol diAC



Data File Name 17807474F3.D
 Data File Path D:\Msd22\Juil06\2307\
 Operator 49
 Date Acquired 7/23/2006 11:33
 Acq. Method File MAN_52.M
 Sample Name 178/07 995474 F3
 Vial Number 3
 Misc Info 178/07 995474 Fraction 3 dans 400µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.67		258	2,492,729
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	15.10	1.415	256	3,658,105
5a Androstan 3a 17b diol diAC	15.48	1.450	316	1,968,201
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	19.06	1.786	284	4,951,688

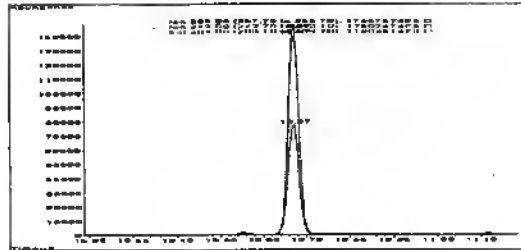
M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	2,615,044	104.9
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	0	0.0
5b Androstan 3a 17b diol diAC	316	3,242,469	88.6
5a Androstan 3a 17b diol diAC	241	1,199,926	61.0
11 KetoEtiocholanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	2,833,947	57.2

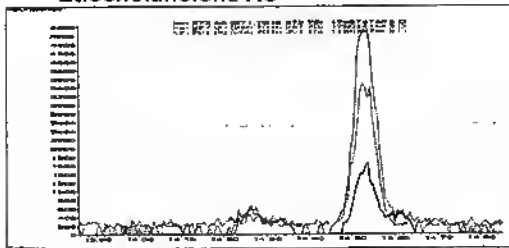
M3 signal

Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	1,395,444	56.0
Etiocholanolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	2,877,543	78.7
5a Androstan 3a 17b diol diAC	256	985,712	50.1
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	1,343,347	27.1

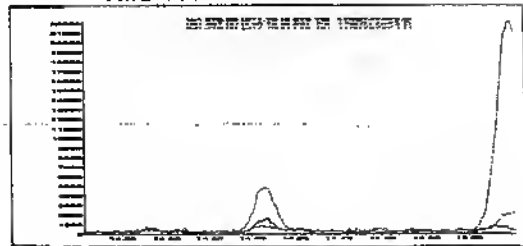
5a Androstanol AC



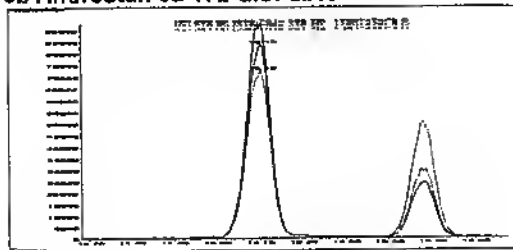
Etiocholanolone AC



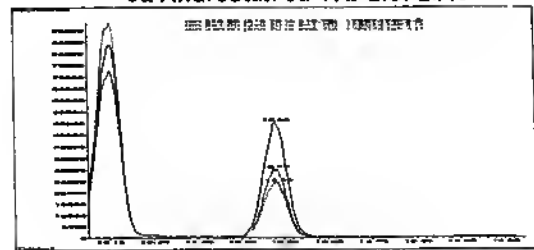
Androsterone AC



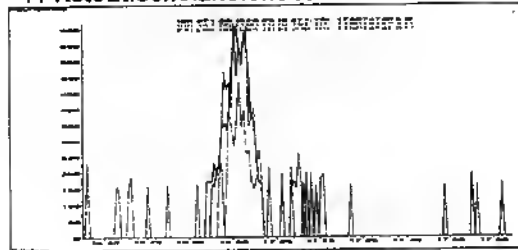
5b Androstan 3a 17b diol diAC



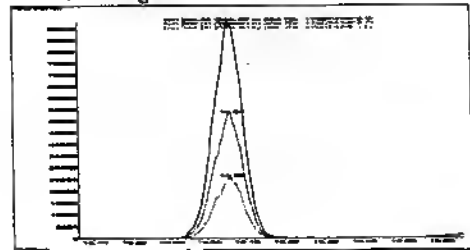
5a Androstan 3a 17b diol diAC



11 KetoEtiocholanolone AC



5b Pregnan 3a 20a diol diAC



LNDD	ENREGISTREMENT	Codification : E-CC-11 Version : B Date : 08/03/2006 1/1
VERIFICATION DES PERFORMANCES INSTRUMENTALES EN CG/SM (screening et confirmation)		

Numéro d'identification de l'appareil : MSD 230

Date : 23/07/06

1 - Source d'ionisation

		Oui	Non
MSD	Autotune : Ion 69 ou 219 majoritaire	<input checked="" type="checkbox"/>	<input type="checkbox"/>
	Autotune : Abondance de l'ion 502 > 3%	<input checked="" type="checkbox"/>	<input type="checkbox"/>
	Repeller < 35	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Polaris	Ion time > 2 ms	<input type="checkbox"/>	<input type="checkbox"/>

Observations :

2 - Etanchéité do système

		Oui	Non
MSD	18/69 (H2O), 28/69 (N2), 32/69 (O2), 44/69 (CO2) < 10%	<input checked="" type="checkbox"/>	<input type="checkbox"/>
	Polaris	Air et eau : Intensité ion 19 < intensité ion 18	<input type="checkbox"/>

Observations :

3 - Sensibilité

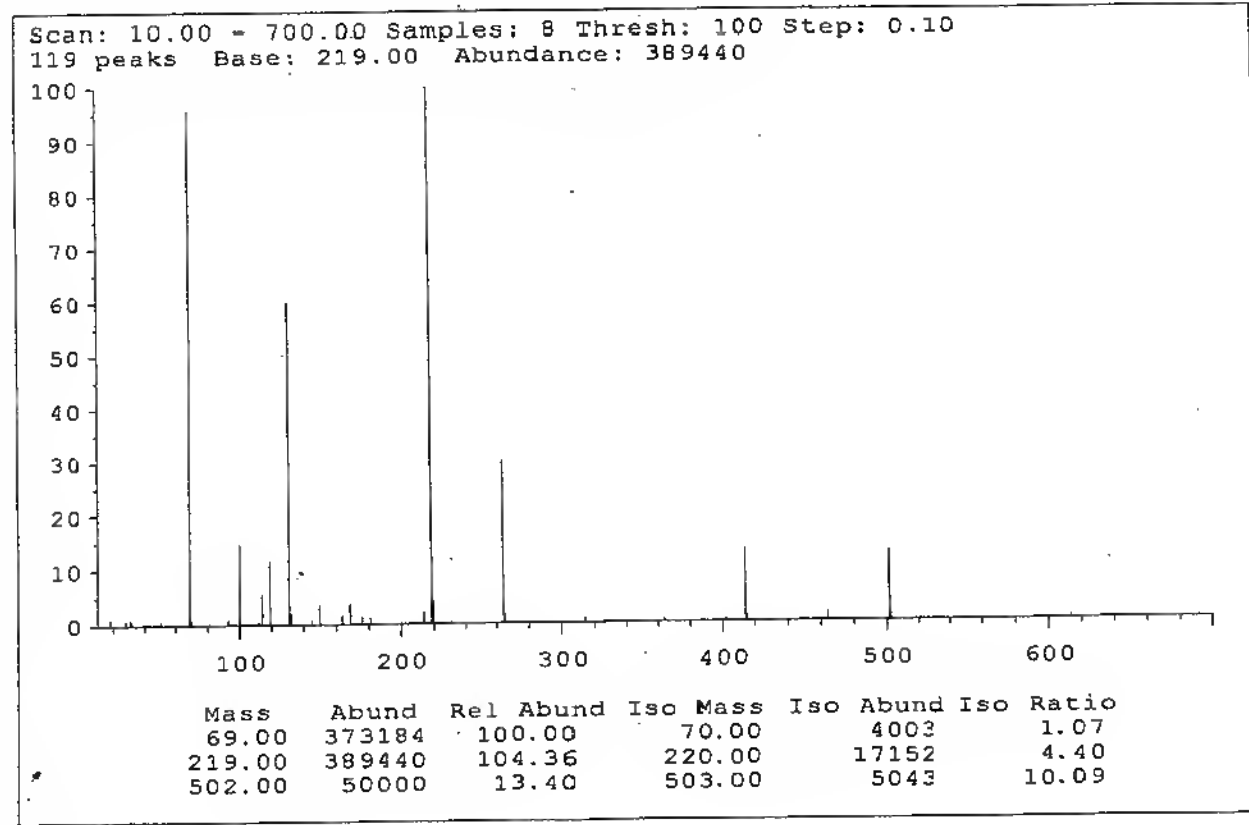
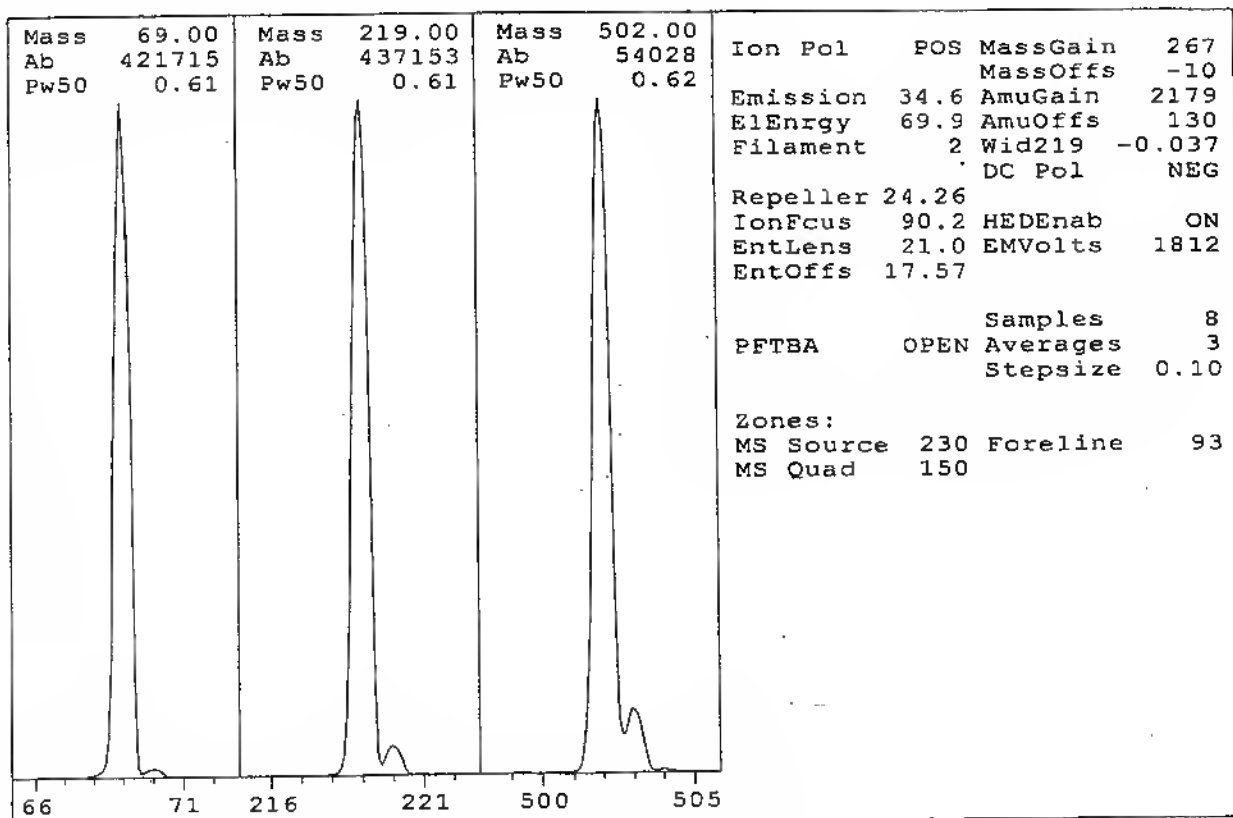
		Oui	Non
Screening	Recal / Mix conforme	<input type="checkbox"/>	<input type="checkbox"/>
Conf	TP conforme - Fichier : 2307 Mix Acc 01	<input checked="" type="checkbox"/>	<input type="checkbox"/>
	TP conforme - Fichier :	<input type="checkbox"/>	<input type="checkbox"/>
	TP conforme - Fichier :	<input type="checkbox"/>	<input type="checkbox"/>
	TP conforme - Fichier :	<input type="checkbox"/>	<input type="checkbox"/>

Observations :

Code opérateur et paraphe : 49 *[Signature]*

Cet enregistrement est à archiver dans le classeur C-MA-Ech de l'appareil

Sun Jul 23 10:11:55 2006
 C:\MSDCHEM\1\5973N\ATUNE.U



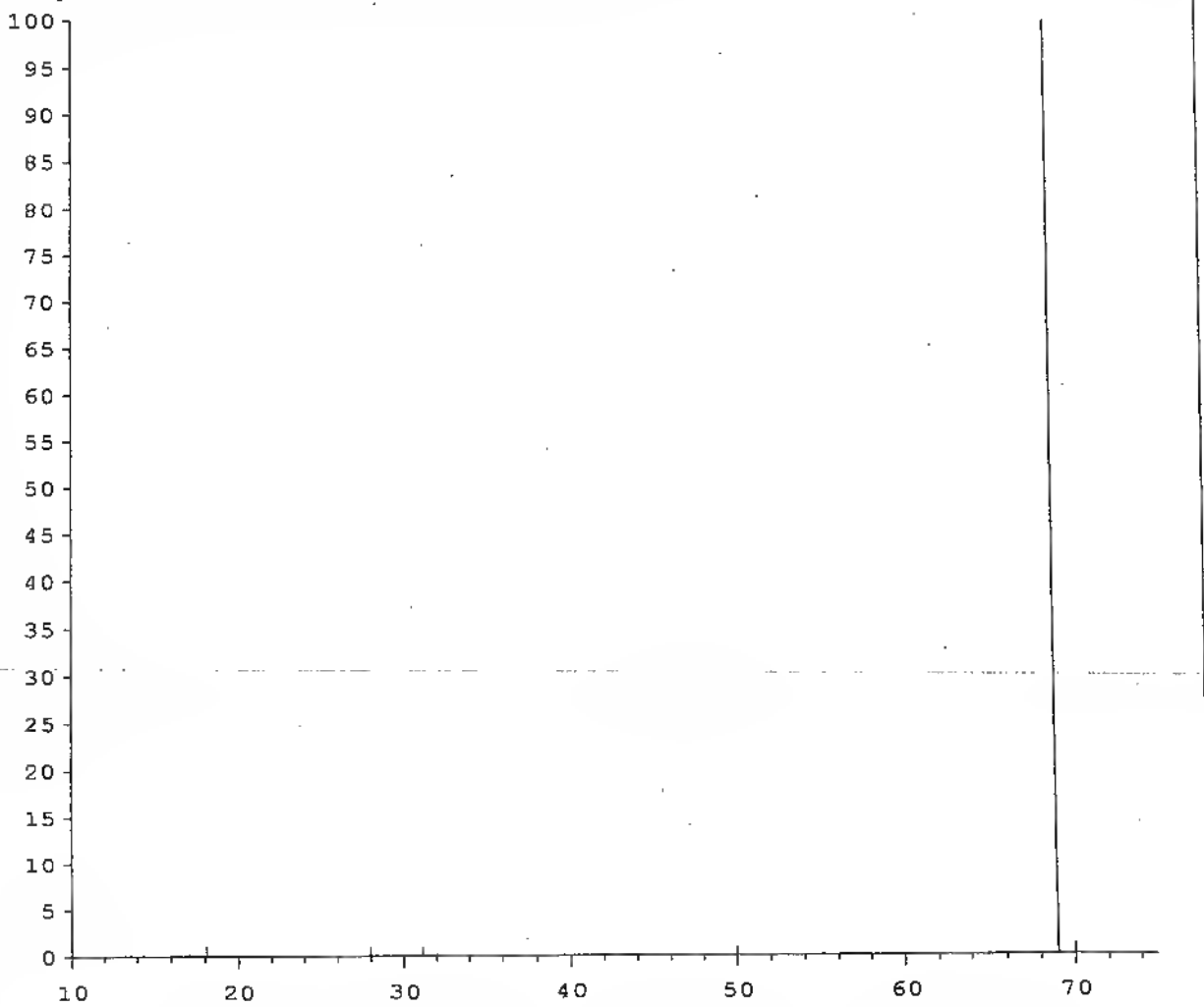
5973 Air and Water Check

Instrument: MSD22

Sun Jul 23 10:12:58 2006

C:\MSDCHEM\1\5973N\

Scan: 10.00 - 75.00 Samples: 8 Thresh: 0 Step: 0.10
 69 peaks Base: 69.00 Abundance: 322304



Mass	Abund	Rel Abund	Iso	Mass	Iso Abund	Iso Ratio
69.00	322304	100.00		70.00	3612	1.12
18.10	3336	1.04		19.00	34	1.02
28.00	3013	0.93		28.90	58	1.92

Current Params used: ATUNE.U

Relative abundances:

18/69 = 1.04	Water%
28/69 = 0.93	Nitrogen%
32/69 = 0.28	Oxygen%
44/69 = 0.17	Carbon Dioxide%
28/18 = 90.32	Nitrogen/Water%

LNDD	ENREGISTREMENT	Codification :	E-FCR-09
		Version :	A
		Date :	31/01/2006
			1/3
FICHE D'ANALYSE / RESULTAT ANALYSE QUALITATIVE GC/MS POUR CONFIRMATION GC/C/IRMS			

Echantillon :

Tolérances fixées par l'AMA (document : WADA Technical Document - TD2003IDCR)

Tolérances sur le tr et le trr : +/-1% ou +/- 0,2 min (prendre la plus faible des deux)

Pour les abondances relatives > 50% il est admis +/- 10% (en absolu) de variation
 25 < ab < 50% +/- 20% (en relatif)
 < 25% +/- 5% (en absolu)

Calcul des abondances en : Tabulation: Surface: Hauteur:

Standard Interne:

	Mix	Fraction F1	Fraction F2	Fraction F3
Tr (min)	10.69	10.69	10.69	10.67
Fichier	2307MixAc01	17807474F1	17807474F2b	17807474F3

Substance caractérisée : Fichier :

	Mix					Echantillon				
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)
	17.07	1.597	100	83	59.9	17.05	1.596	100	81.1	60.7
Tolérance basse	16.90	1.581		73	49.9					
Tolérance haute	17.24	1.613		93	69.9					

Concordance des Tr : oui non
 Concordance des Trr : oui non
 Concordance globale des abondances relatives : oui non

Substance caractérisée : Fichier :

	Mix					Echantillon				
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)
	14.35	1.343	100	63.8	42.6	14.38	1.345	100	63.4	37.8
Tolérance basse	14.21	1.330		53.8	34.08					
Tolérance haute	14.49	1.356		73.8	51.12					

Concordance des Tr : oui non
 Concordance des Trr : oui non
 Concordance globale des abondances relatives : oui non

LNDD	ENREGISTREMENT	Codification :	E-FCR-09
		Version :	A
		Date :	31/01/2006 2/3
FICHE D'ANALYSE / RESULTAT ANALYSE QUALITATIVE GC/MS POUR CONFIRMATION GC/CIRMS			

Substance caractérisée : **Androstérone AC** Fichier : **17807474F2b**

	Mix					Echantillon				
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)
	14.62	1.367	100	42.6	30.6	14.65	1.370	100	43	30.2
Tolérance basse	14.47	1.353		34.08	24.48					
Tolérance haute	14.77	1.381		51.12	36.72					

Concordance des Tr : oui non
 Concordance des Trr : oui non
 Concordance globale des abondances relatives : oui non

Substance caractérisée : **5b Androstane-3a,17b-diol diAC** Fichier : **17807474F3**

	Mix					Echantillon				
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)
	15.17	1.419	100	90.4	77.1	15.1	1.415	100	88.6	78.7
Tolérance basse	15.02	1.405		80.4	67.1					
Tolérance haute	15.32	1.433		100.4	87.1					

Concordance des Tr : oui non
 Concordance des Trr : oui non
 Concordance globale des abondances relatives : oui non

Substance caractérisée : **5a Androstane-3a,17b-diol diAC** Fichier : **17807474F3**

	Mix					Echantillon				
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)
	15.57	1.456	100	58.5	48.9	15.48	1.450	100	61	50.1
Tolérance basse	15.41	1.441		48.5	39.12					
Tolérance haute	15.73	1.471		68.5	58.68					

Concordance des Tr : oui non
 Concordance des Trr : oui non
 Concordance globale des abondances relatives : oui non

Substance caractérisée : **5b Prégnane-3a,20a-diol diAC** Fichier : **17807474F3**

	Mix					Echantillon				
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)
	19.2	1.796	100	56.3	28	19.06	1.786	100	57.2	27.1
Tolérance basse	19.01	1.778		46.3	22.4					
Tolérance haute	19.39	1.814		66.3	33.6					

Concordance des Tr : oui non
 Concordance des Trr : oui non
 Concordance globale des abondances relatives : oui non

LNDD	ENREGISTREMENT	Codification : E-FCR-09 Version : A Date : 31/01/2006 3/3
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FICHE D'ANALYSE / RESULTAT ANALYSE QUALITATIVE GC/MS POUR CONFIRMATION GC/C/IRMS

Substance caractérisée :

Fichier :

	Mix					Echantillon				
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)
			100					100		
Tolérance basse										
Tolérance haute										

Concordance des Tr : oui non

Concordance des Trr : oui non

Concordance globale des abondances relatives : oui non

Substance caractérisée :

Fichier :

	Mix					Echantillon				
	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%)
			100					100		
Tolérance basse										
Tolérance haute										

Concordance des Tr : oui non

Concordance des Trr : oui non

Concordance globale des abondances relatives : oui non

Paraphe et code opérateur :

Partie à remplir par le responsable :

Caractérisation formelle de tous les analytes : oui non

Paraphe et code opérateur :

Observations :

Cet enregistrement est à archiver dans le dossier de confirmation de l'échantillon