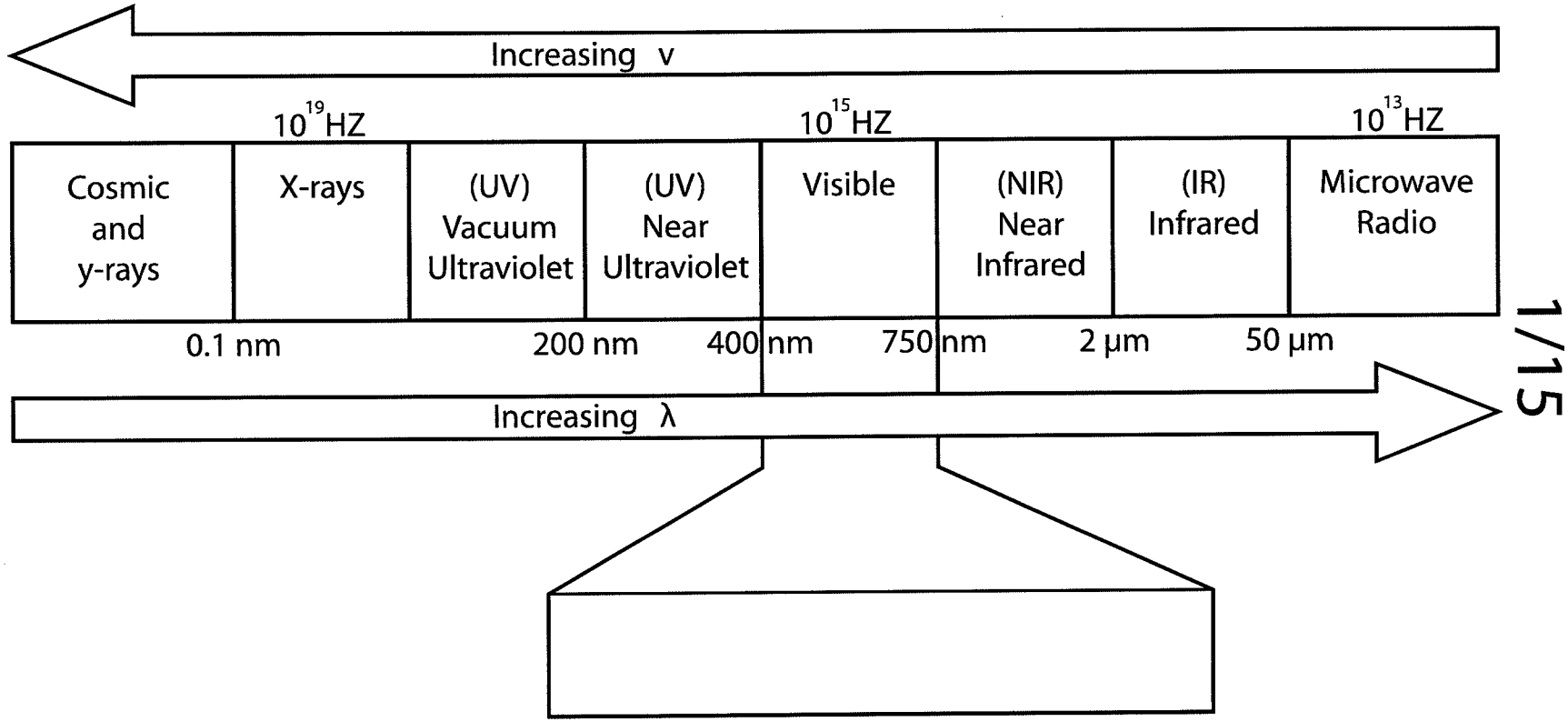

Increasing v
10^19 HZ
10^15 HZ
10^13 HZ
Cosmic and y-rays
X-rays
(UV) Vacuum Ultraviolet
(UV) Near Ultraviolet
Visible
(NIR) Near Infrared
(IR) Infrared
Microwave Radio
0.1 nm
200 nm
400 nm
750 nm
2 µm
50 µm
Increasing λ

FIG. 1

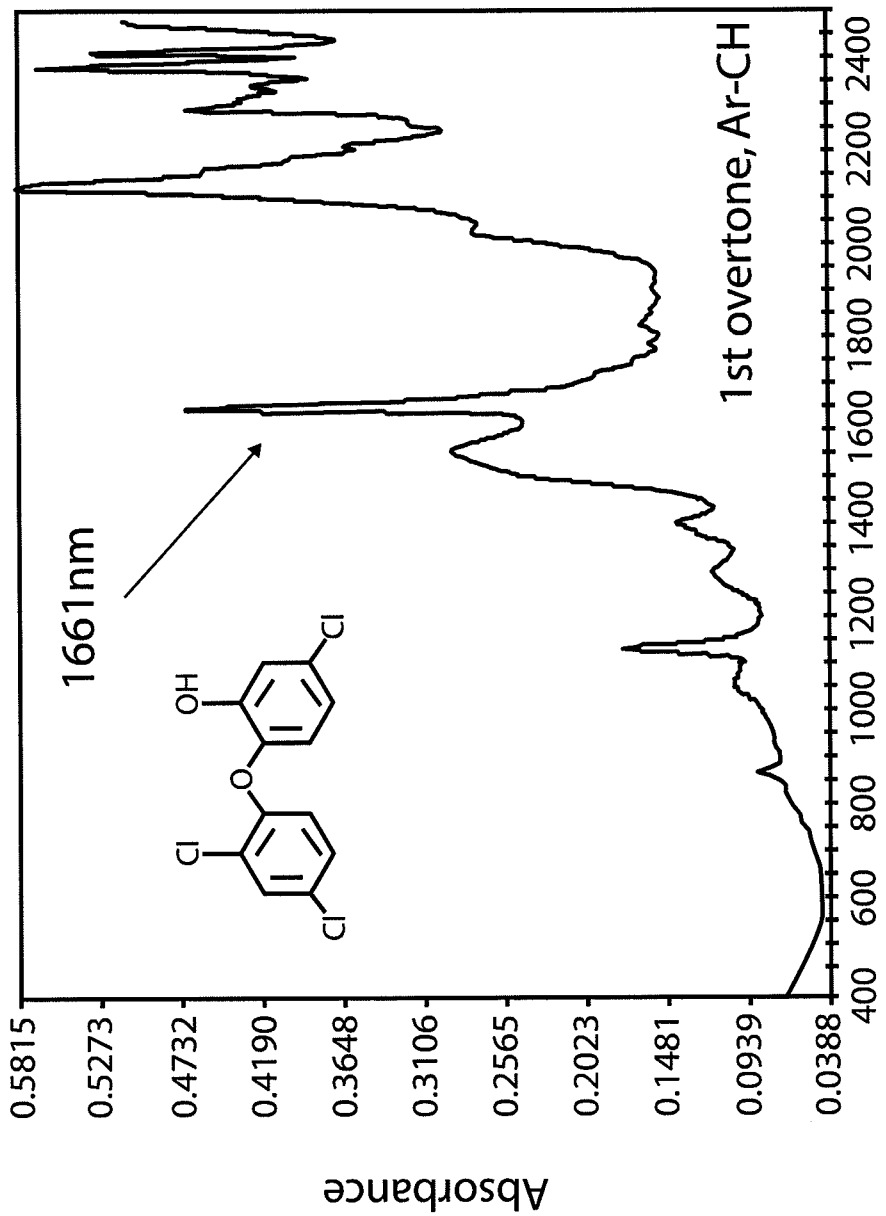

0.5815
0.5273
0.4732
0.4190
0.3648
0.3106
0.2565
0.2023
0.1481
0.0939
0.0388
Absorbance
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
Wavelength
1661nm
1st overtone, Ar-CH
OH
Cl
O
Cl
Cl

FIG. 2

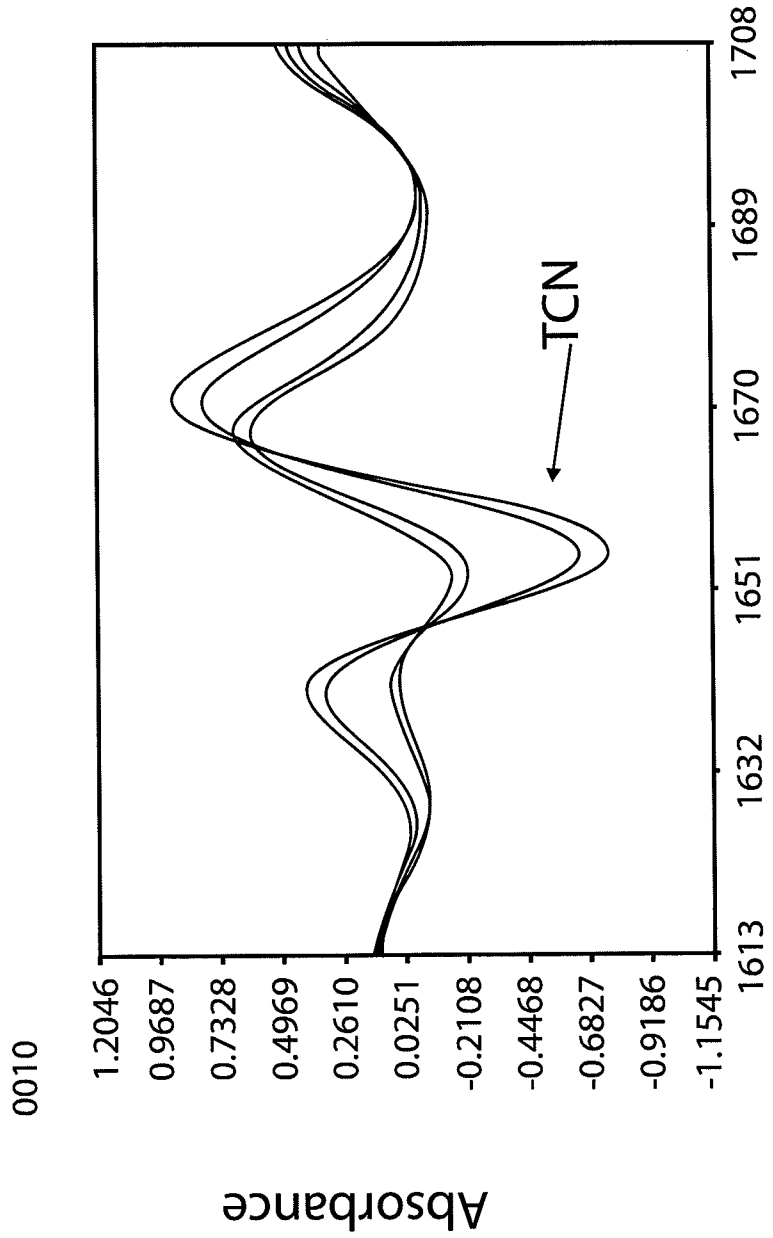

0010
1.2046
0.9687
0.7328
0.4969
0.2610
0.0251
-0.2108
-0.4468
-0.6827
-0.9186
-1.1545
Absorbance
1613
1632
1651
1670
1689
1708
Wavelength
TCN

FIG. 3

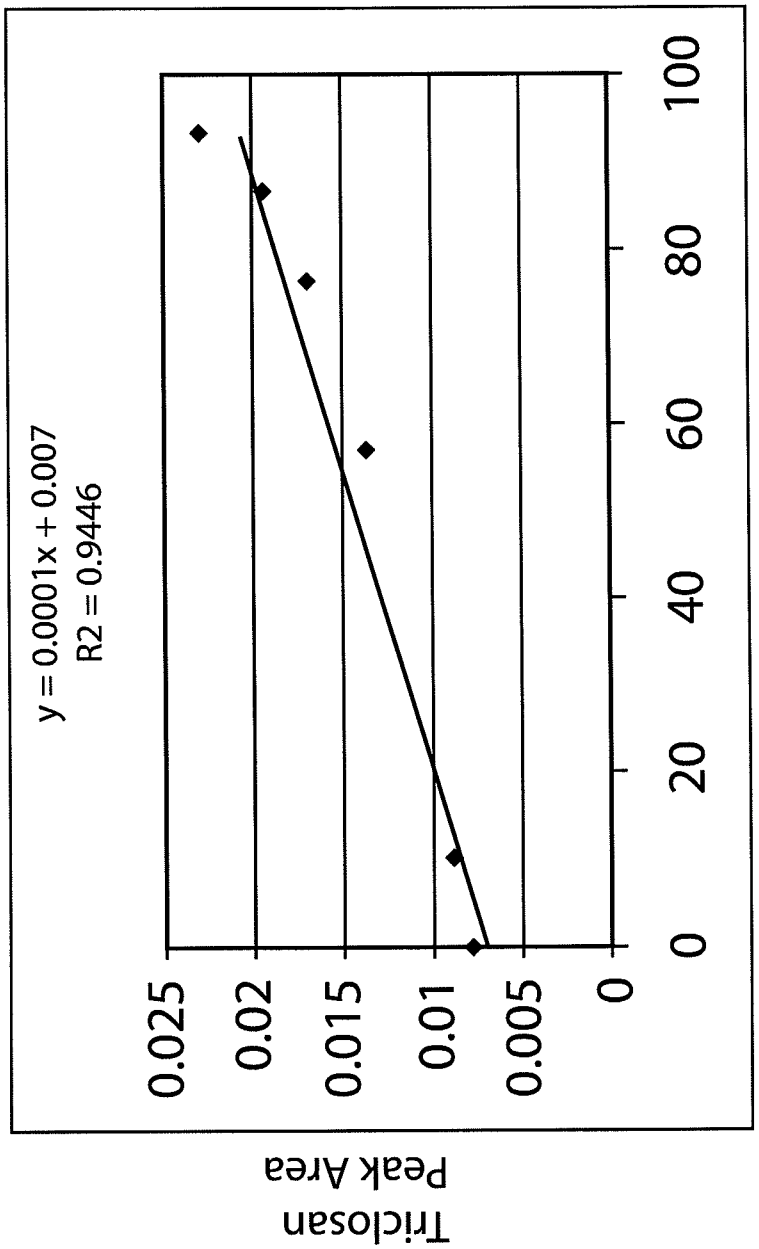

y = 0.0001x + 0.007
R2 = 0.9446
Triclosan
Peak Area
0.025
0.02
0.015
0.01
0.005
0
0
20
40
60
80
100
HPLC Concentration, ppm

FIG. 4

Sample	Triclosan Deposited, ppm
Delivery System 1, Disk 1	30.7
Delivery System 1, Disk 2	35.0
Delivery System 1, Disk 3	35.1
Delivery System 2, Disk 1	94.3
Delivery System 2, Disk 2	80.0
Delivery System 2, Disk 3	85.1

FIG. 5

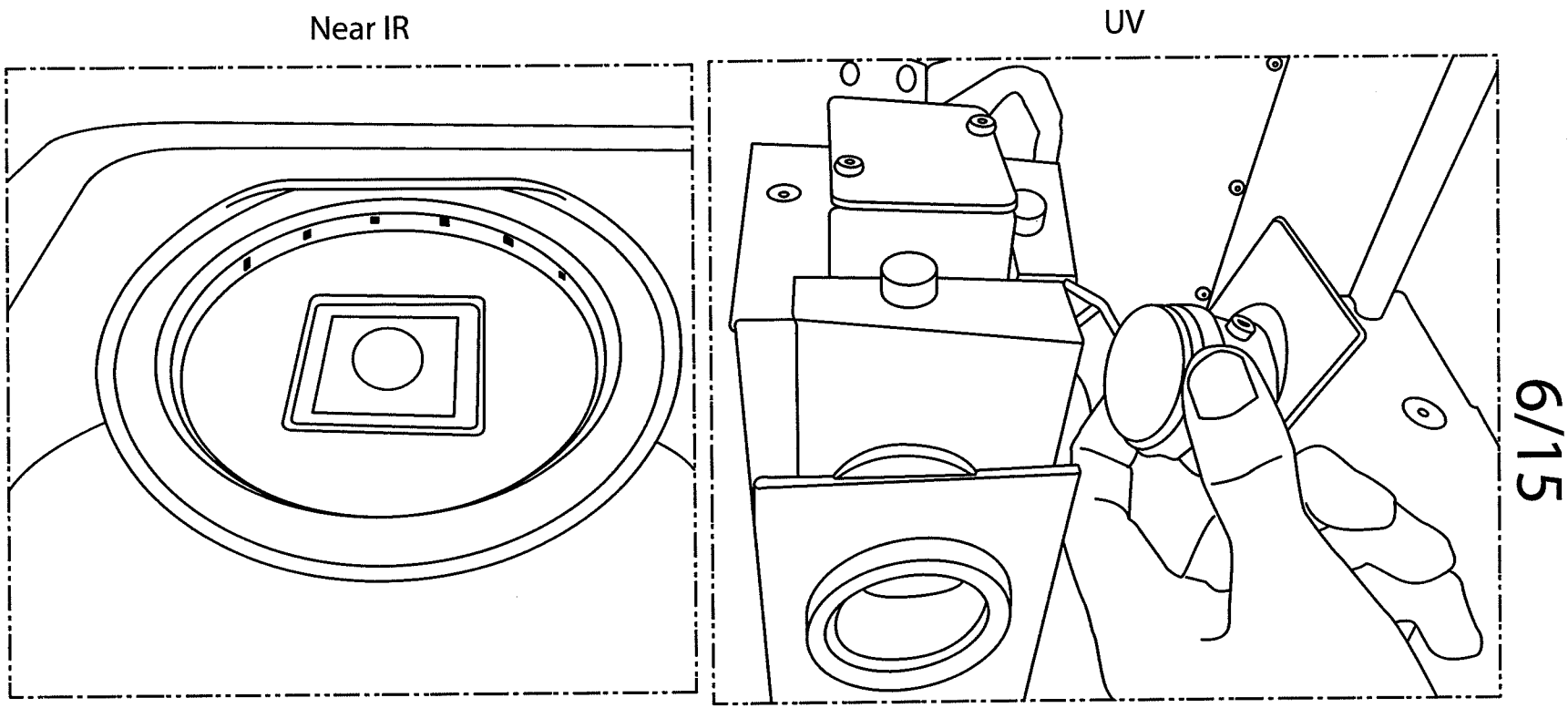


FIG. 6

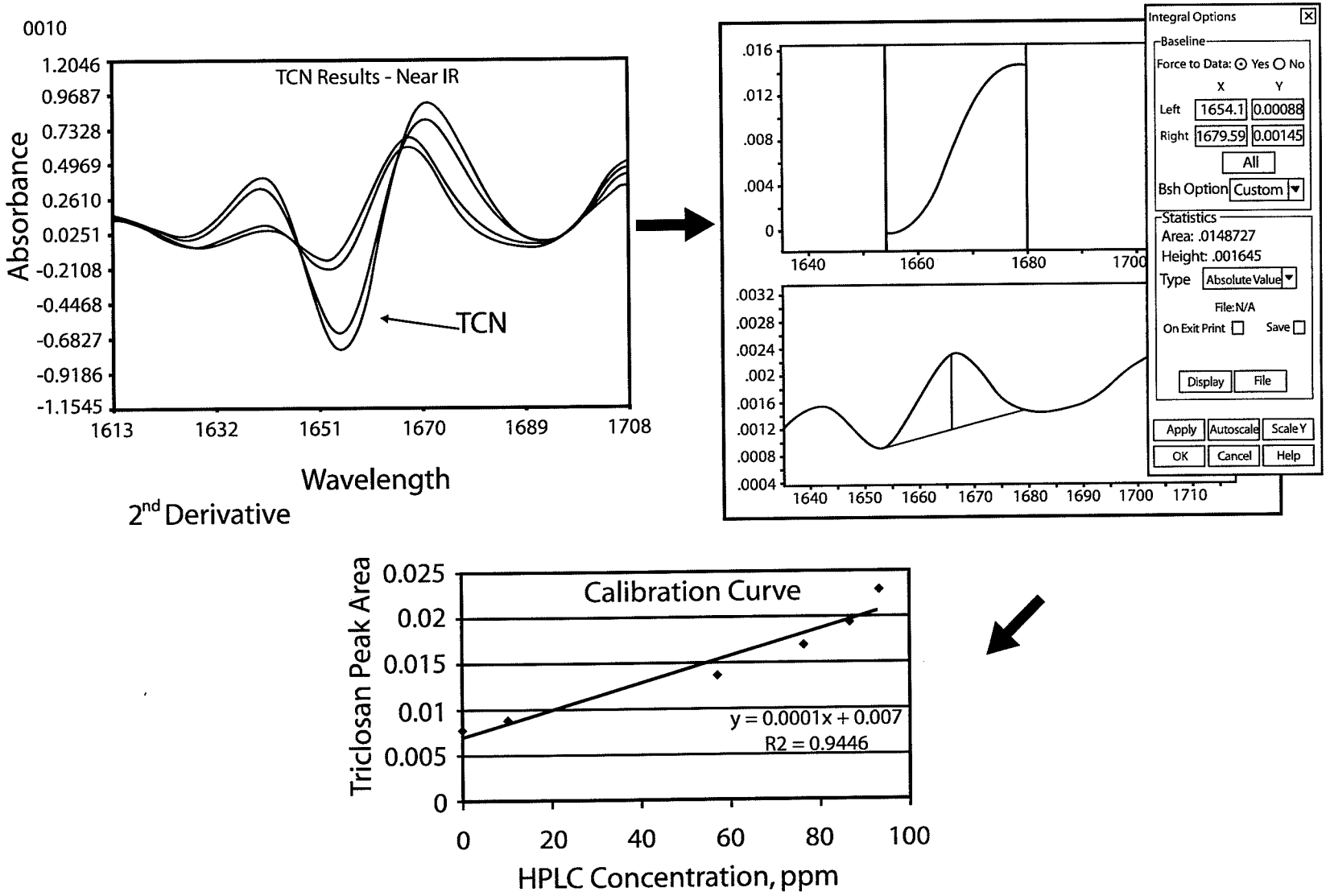

0010
TCN Results - Near IR
Absorbance
1.2046
0.9687
0.7328
0.4969
0.2610
0.0251
-0.2108
-0.4468
-0.6827
-0.9186
-1.1545
1613
1632
1651
1670
1689
1708
TCN
Wavelength
2nd Derivative
.016
.012
.008
.004
0
1640
1660
1680
1700
.0032
.0028
.0024
.002
.0016
.0012
.0008
.0004
1640
1650
1660
1670
1680
1690
1700
1710
Integral Options
Baseline
Force to Data: Yes No
X
Y
Left
1654.1
0.00088
Right
1679.59
0.00145
All
Bsh Option
Custom
Statistics
Area: .0148727
Height: .001645
Type
Absolute Value
File:N/A
On Exit Print
Save
Display
File
Apply
Autoscale
Scale Y
OK
Cancel
Help
Calibration Curve
Triclosan Peak Area
0.025
0.02
0.015
0.01
0.005
0
y = 0.0001x + 0.007
R2 = 0.9446
0
20
40
60
80
100
HPLC Concentration, ppm

FIG. 7

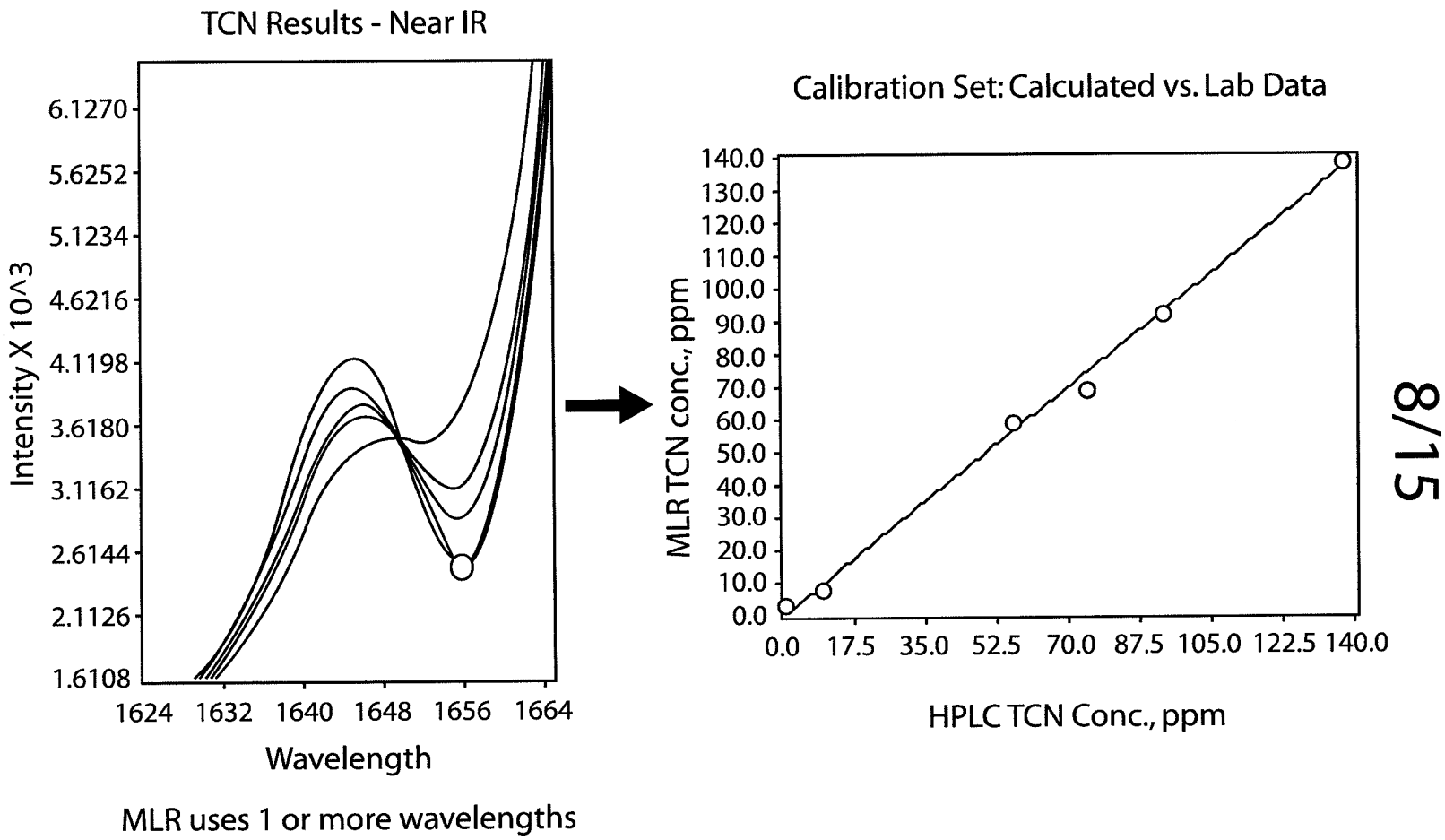

TCN Results - Near IR
Intensity X 10^3
6.1270
5.6252
5.1234
4.6216
4.1198
3.6180
3.1162
2.6144
2.1126
1.6108
1624
1632
1640
1648
1656
1664
Wavelength
MLR uses 1 or more wavelengths
Calibration Set: Calculated vs. Lab Data
MLR TCN conc., ppm
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
0.0
17.5
35.0
52.5
70.0
87.5
105.0
122.5
140.0
HPLC TCN Conc., ppm

FIG. 8

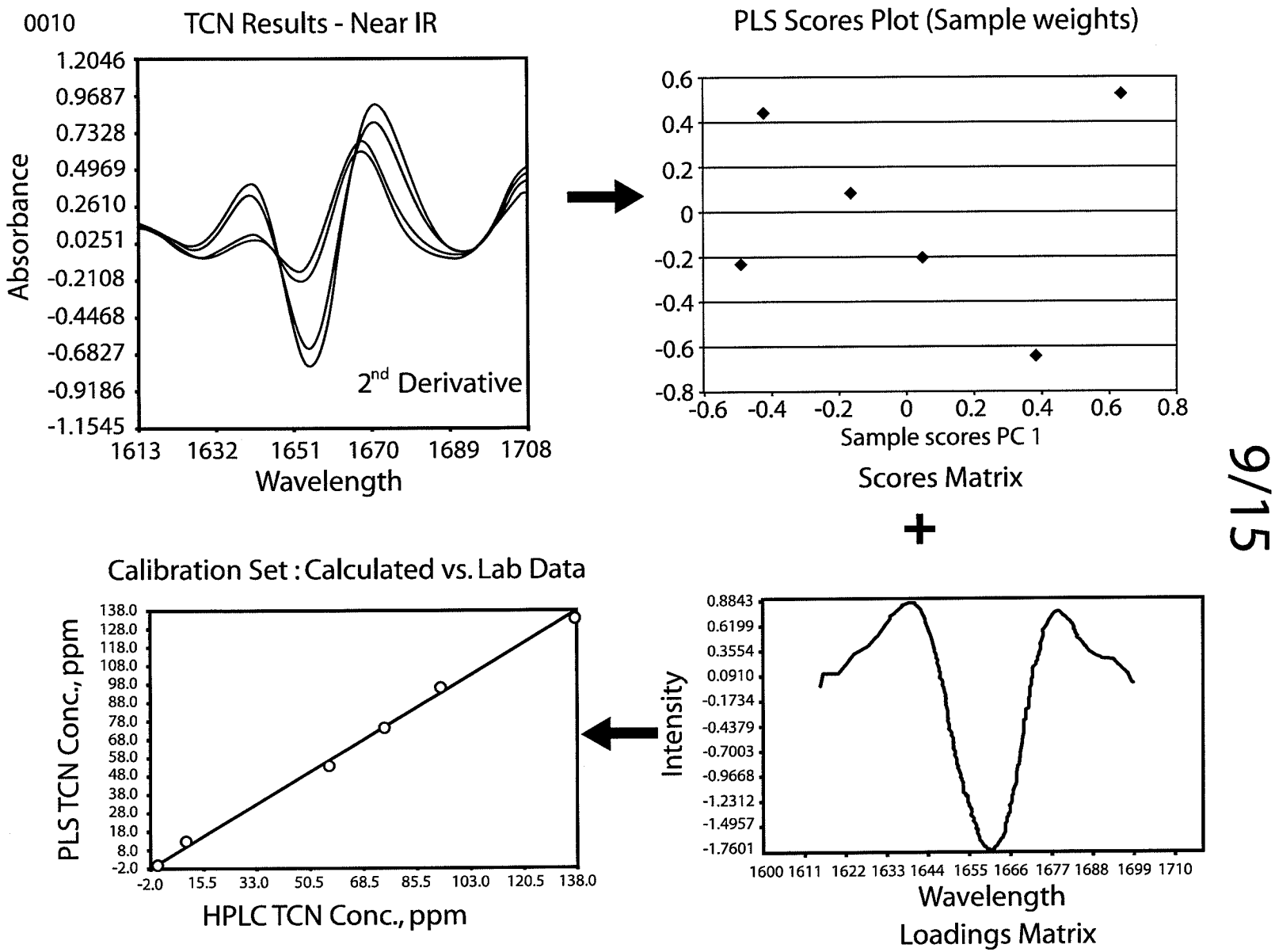
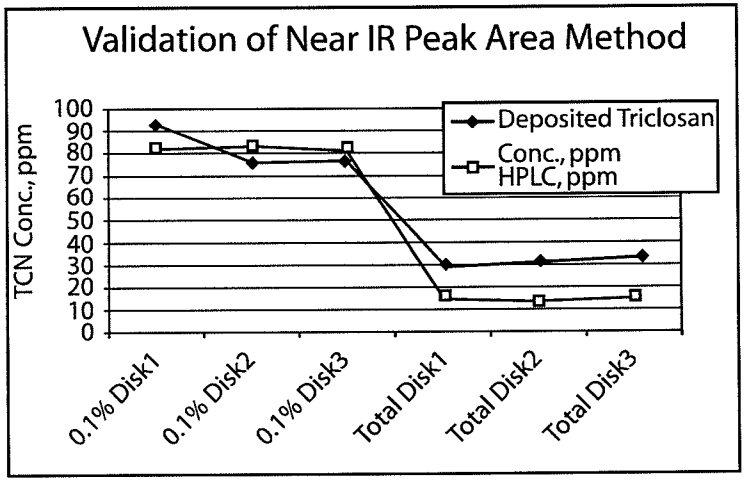
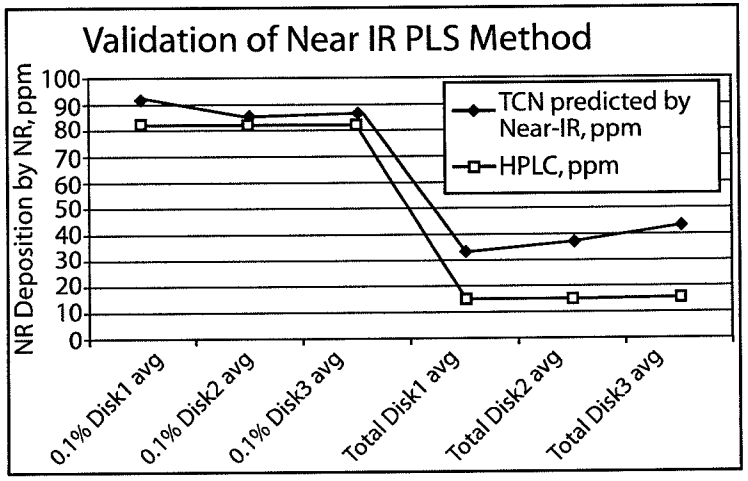

0010
TCN Results - Near IR
Absorbance
1.2046
0.9687
0.7328
0.4969
0.2610
0.0251
-0.2108
-0.4468
-0.6827
-0.9186
-1.1545
2nd Derivative
1613 1632 1651 1670 1689 1708
Wavelength
PLS Scores Plot (Sample weights)
0.6
0.4
0.2
0
-0.2
-0.4
-0.6
-0.8
-0.6 -0.4 -0.2 0 0.2 0.4 0.6 0.8
Sample scores PC 1
Scores Matrix
+
Calibration Set : Calculated vs. Lab Data
PLS TCN Conc., ppm
138.0
128.0
118.0
108.0
98.0
88.0
78.0
68.0
58.0
48.0
38.0
28.0
18.0
8.0
-2.0
-2.0 15.5 33.0 50.5 68.5 85.5 103.0 120.5 138.0
HPLC TCN Conc., ppm
Intensity
0.8843
0.6199
0.3554
0.0910
-0.1734
-0.4379
-0.7003
-0.9668
-1.2312
-1.4957
-1.7601
1600 1611 1622 1633 1644 1655 1666 1677 1688 1699 1710
Wavelength
Loadings Matrix

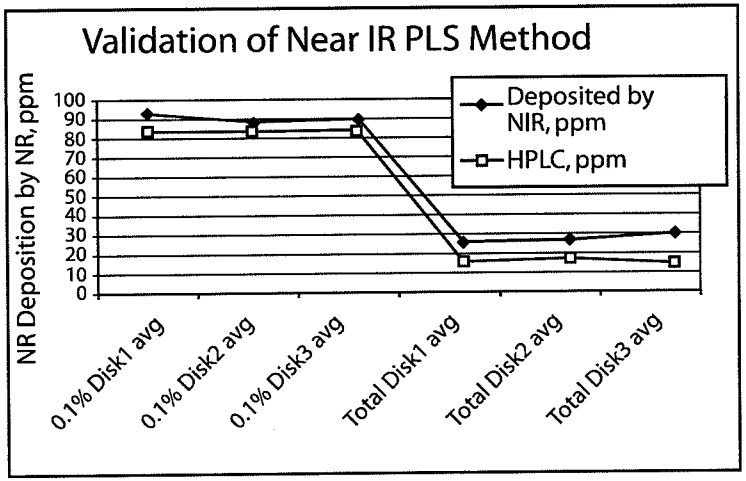
FIG. 9



Peak Area SEP ± 10.5 ppm

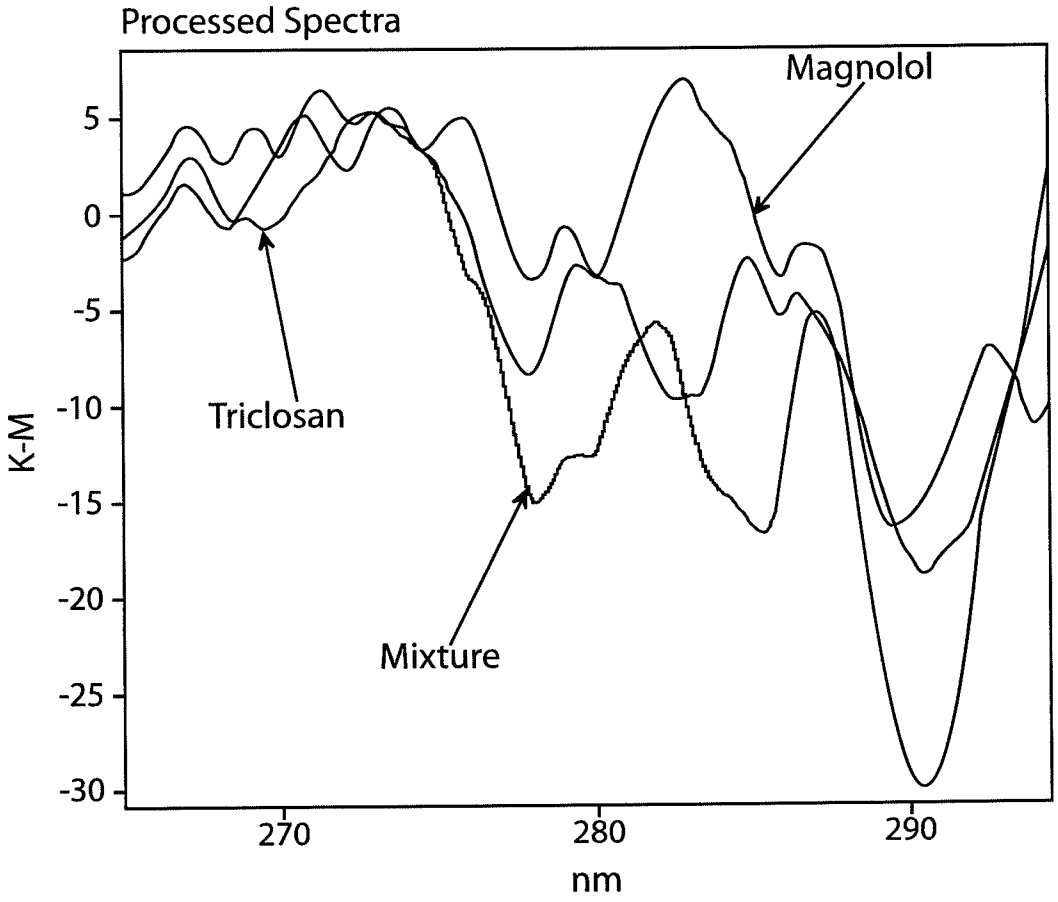


PLS SEP ± 8.6 ppm



MLR SEP ± 2.8 ppm

FIG. 10



Sample	Triclosan Peak Area	Mixed Active (1:1)	
5 min	4.4 units	52% Triclosan	48% Magnolol
30 min	18.1 units		

FIG. 11

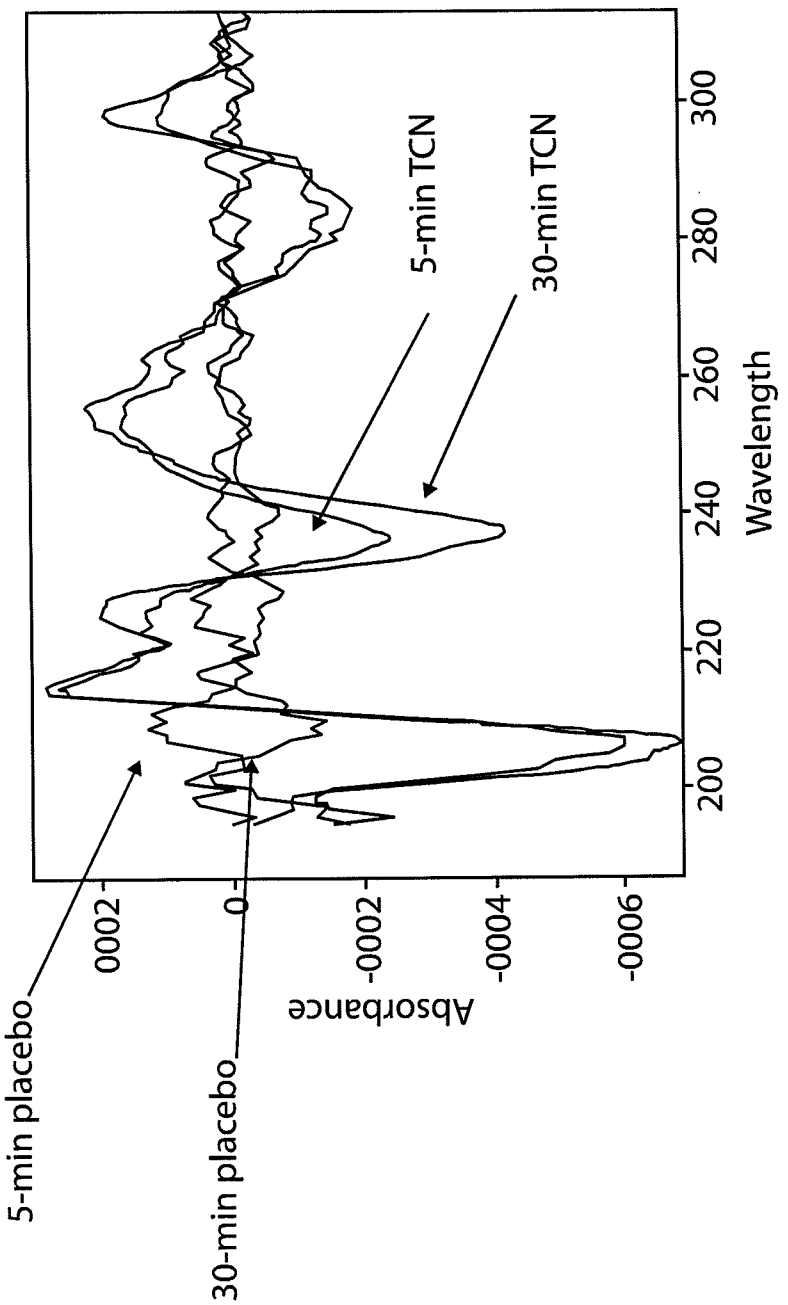

5-min placebo
30-min placebo
5-min TCN
30-min TCN
0002
0
-0002
-0004
-0006
Absorbance
200
220
240
260
280
300
Wavelength

FIG. 12

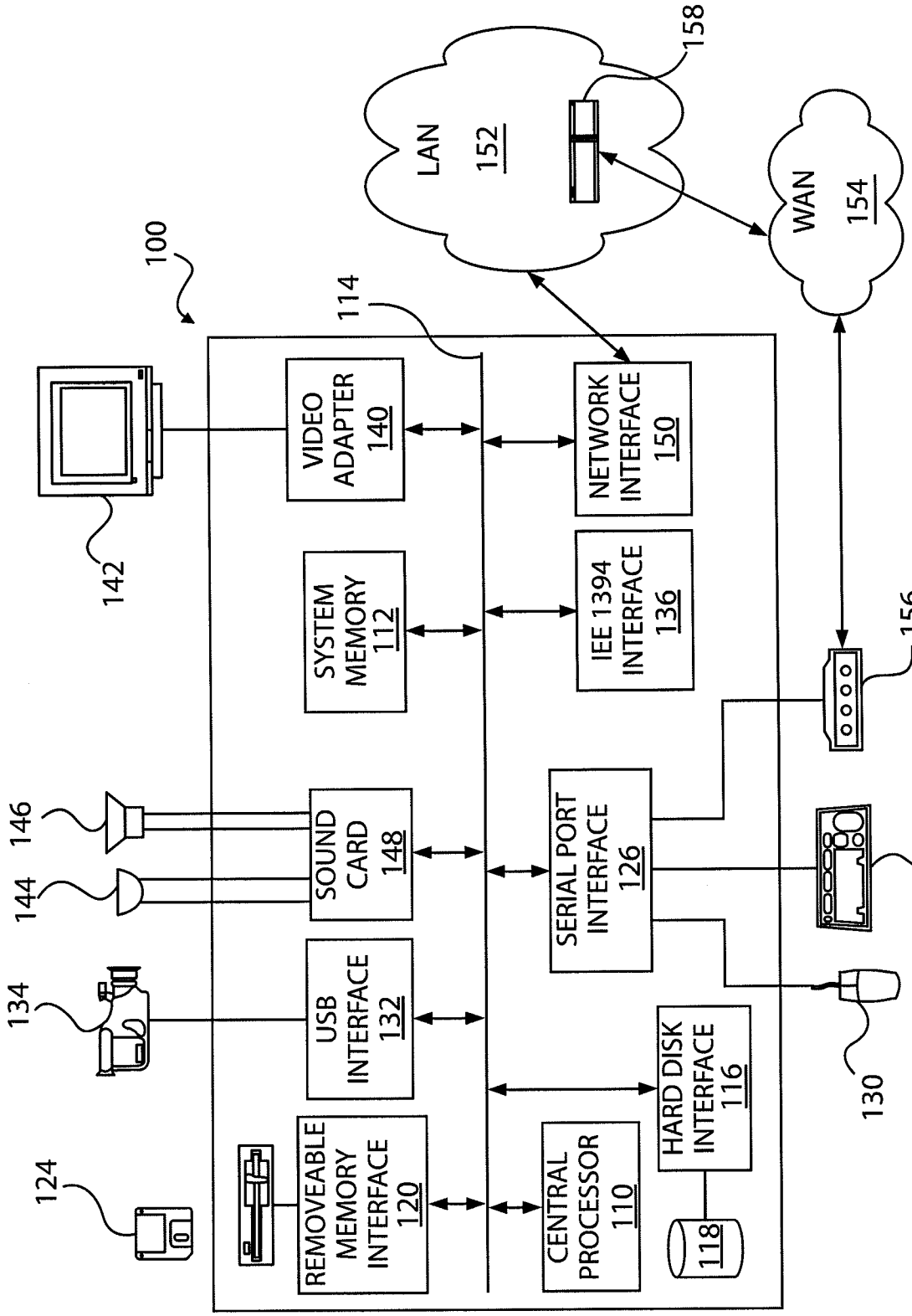

100
114
LAN
152
158
WAN
154
VIDEO ADAPTER
140
NETWORK INTERFACE
150
142
SYSTEM MEMORY
112
IEE 1394 INTERFACE
136
156
146
144
SOUND CARD
148
SERIAL PORT INTERFACE
126
128
134
USB INTERFACE
132
HARD DISK INTERFACE
116
130
124
REMOVEABLE MEMORY INTERFACE
120
CENTRAL PROCESSOR
110
118

FIG. 13

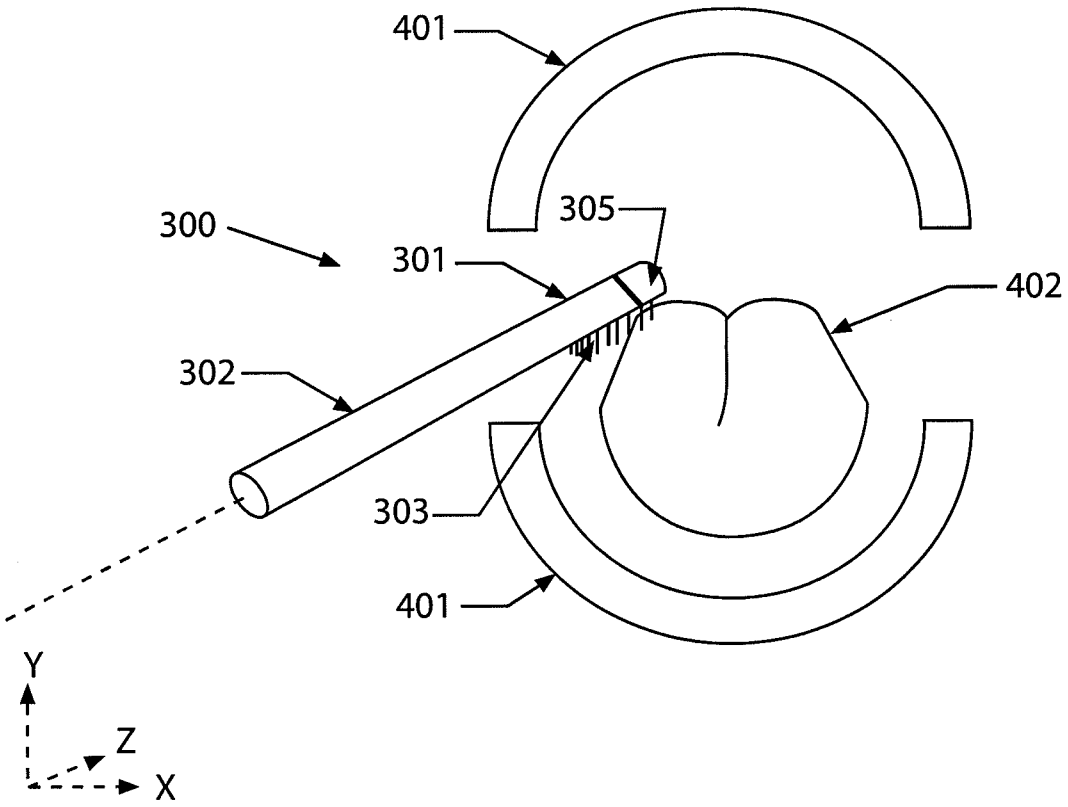


FIG. 14

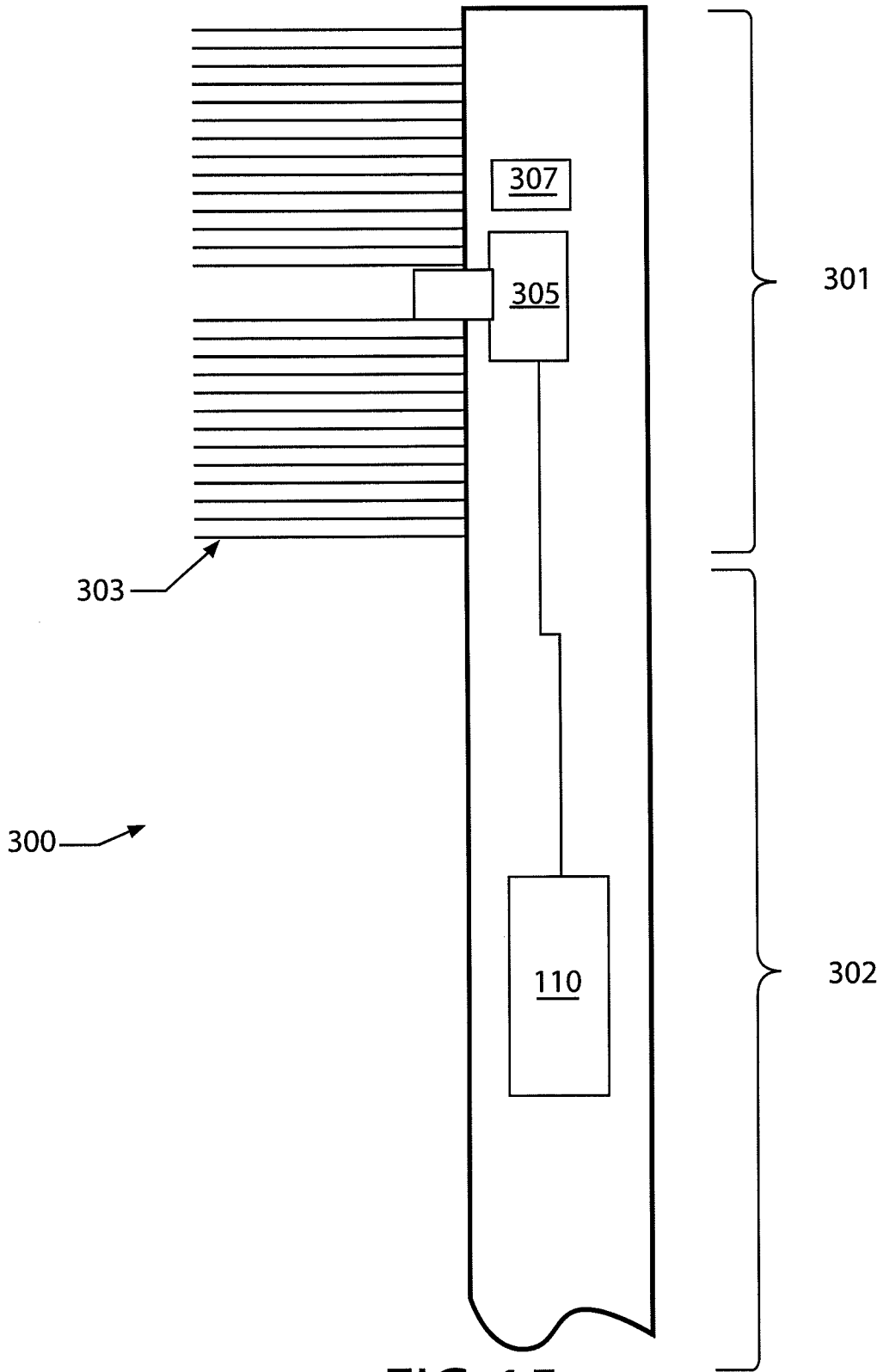


FIG. 15

WO 2009/124310 PCT/US2009/039651

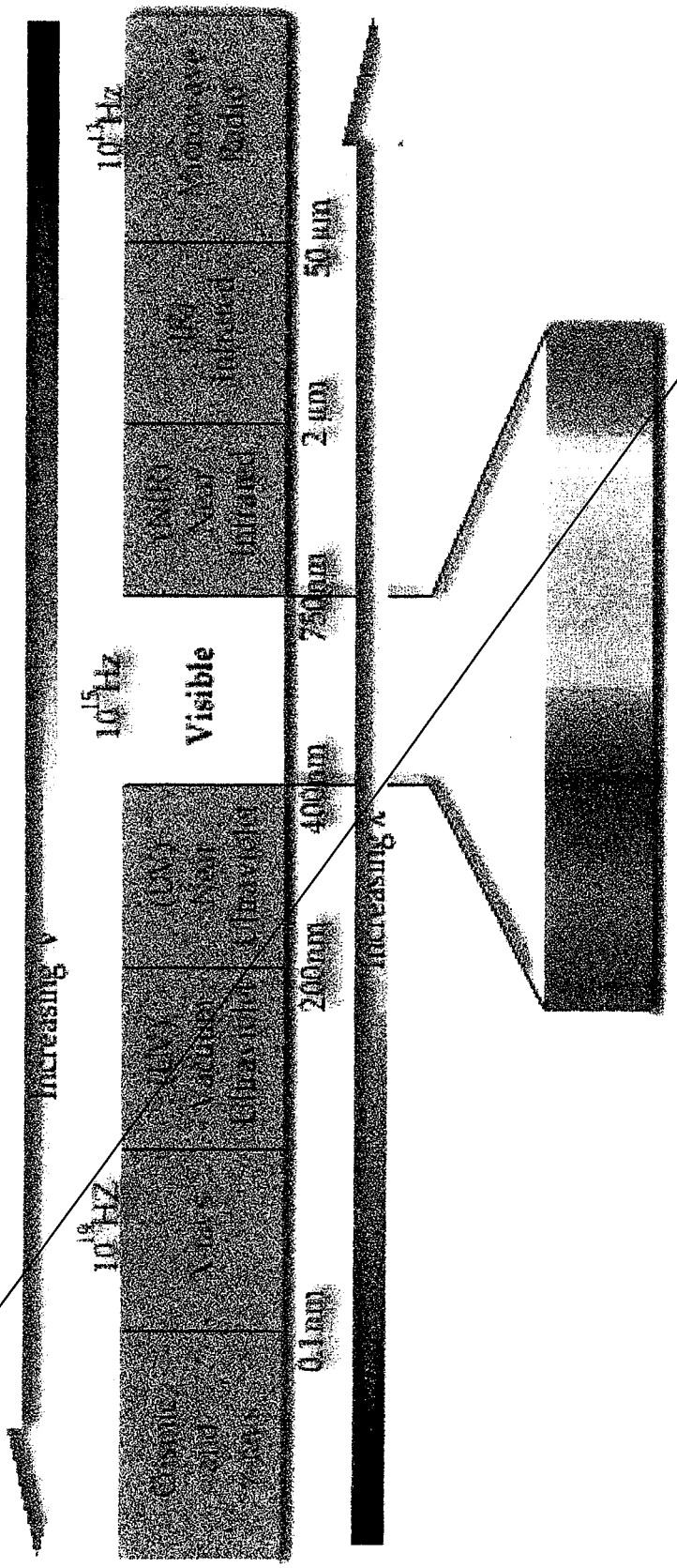


Fig. 1

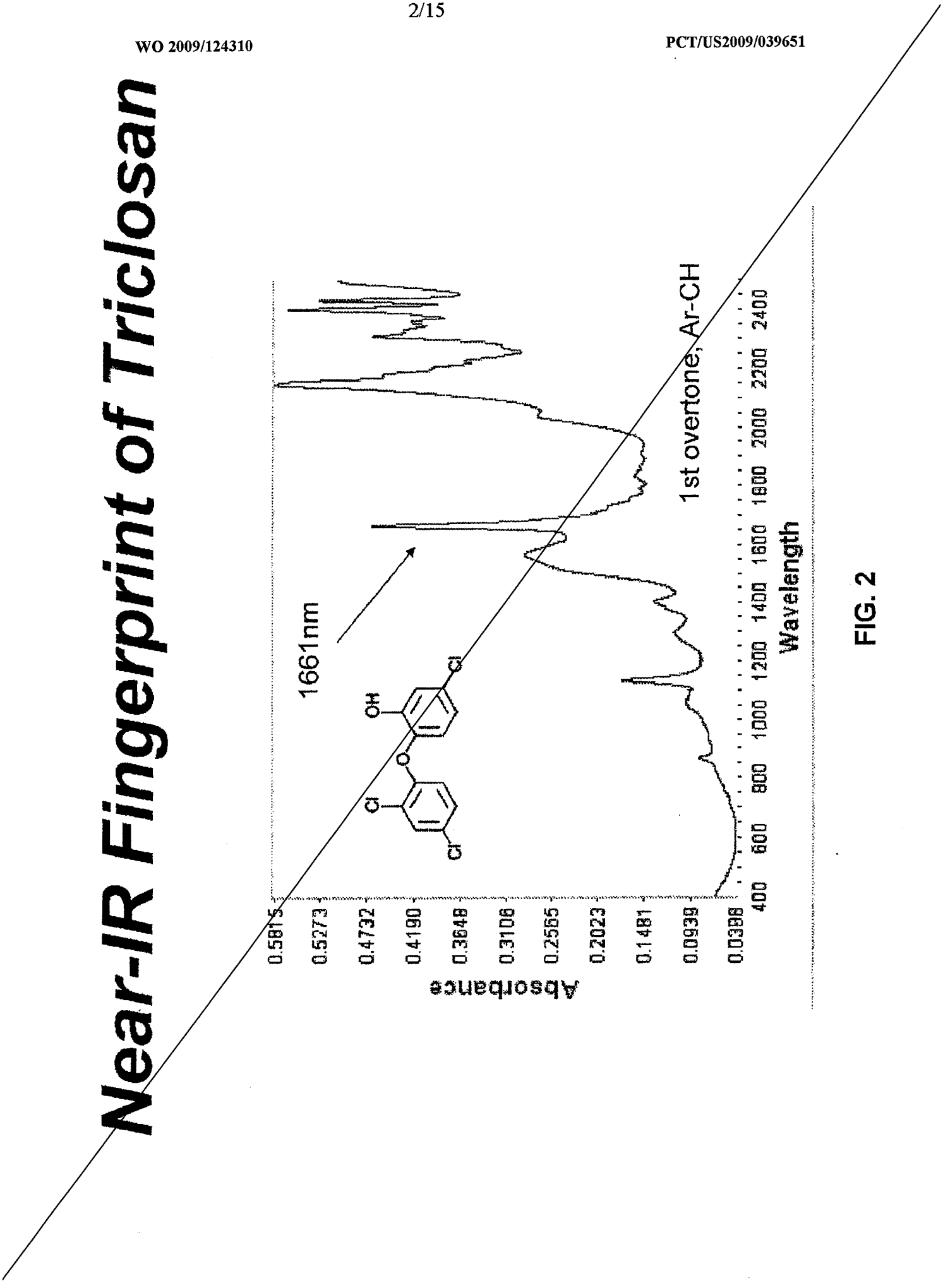

Near-IR Fingerprint of Triclosan
1661nm
1st overtone, Ar-CH
Absorbance
Wavelength
0.5815
0.5273
0.4732
0.4190
0.3648
0.3106
0.2565
0.2023
0.1481
0.0939
0.0398
400
600
800
1000
1200
1400
1600
1800
2000
2200
2400
OH
O
Cl
Cl
Cl

FIG. 2

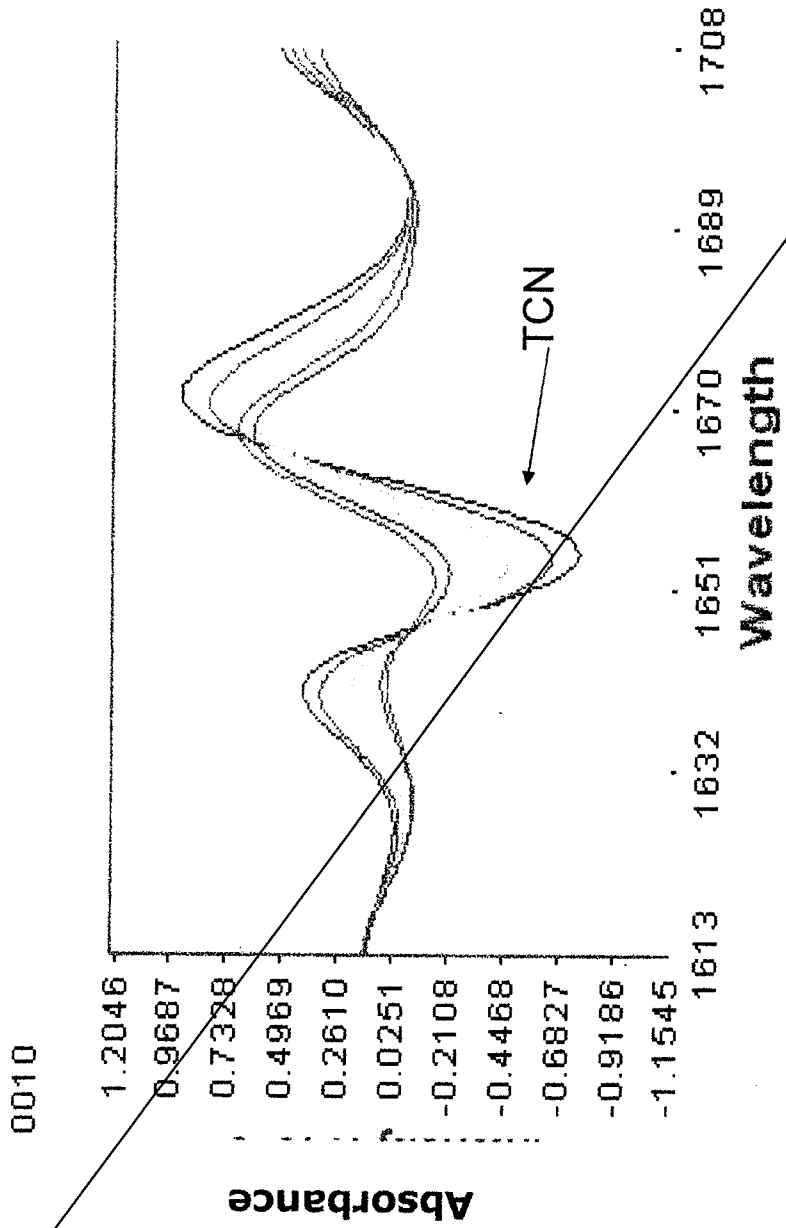

0010
1.2046
0.9687
0.7328
0.4969
0.2610
0.0251
-0.2108
-0.4468
-0.6827
-0.9186
-1.1545
Absorbance
1613
1632
1651
1670
1689
1708
Wavelength
TCN

Fig. 3

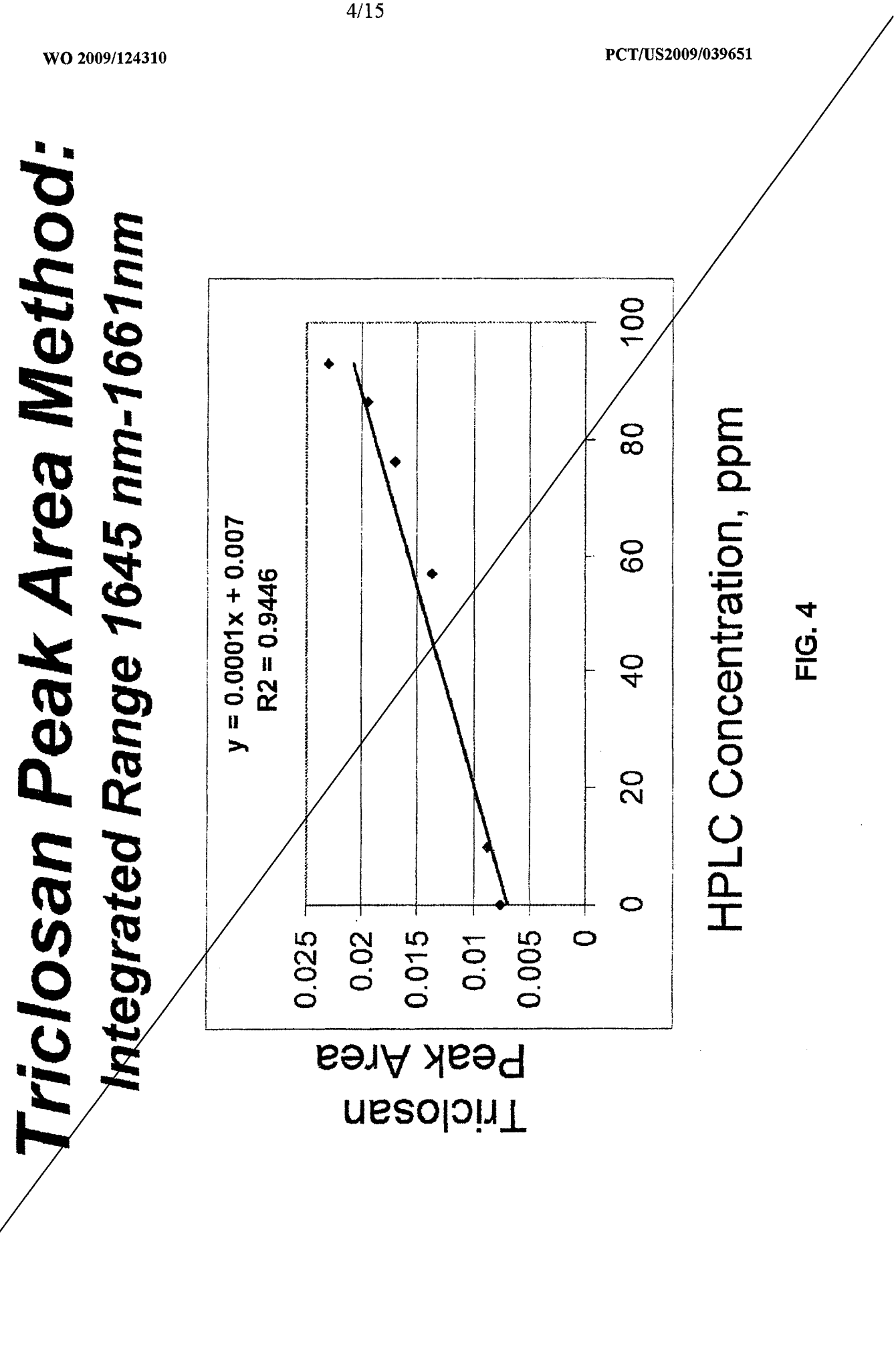

Triclosan Peak Area Method:
Integrated Range 1645 nm-1661nm
y = 0.0001x + 0.007
R2 = 0.9446
Triclosan Peak Area
0.025
0.02
0.015
0.01
0.005
0
0
20
40
60
80
100
HPLC Concentration, ppm

FIG. 4

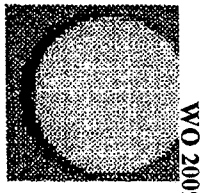
Validation of Near-IR Method

Sample	Triclosan Deposited, ppm
Delivery System 1, Disk 1	30.7
Delivery System 1, Disk 2	35.0
Delivery System 1, Disk 3	35.1
Delivery System 2, Disk 1	94.3
Delivery System 2, Disk 2	80.0
Delivery System 2, Disk 3	85.1

FIG. 5

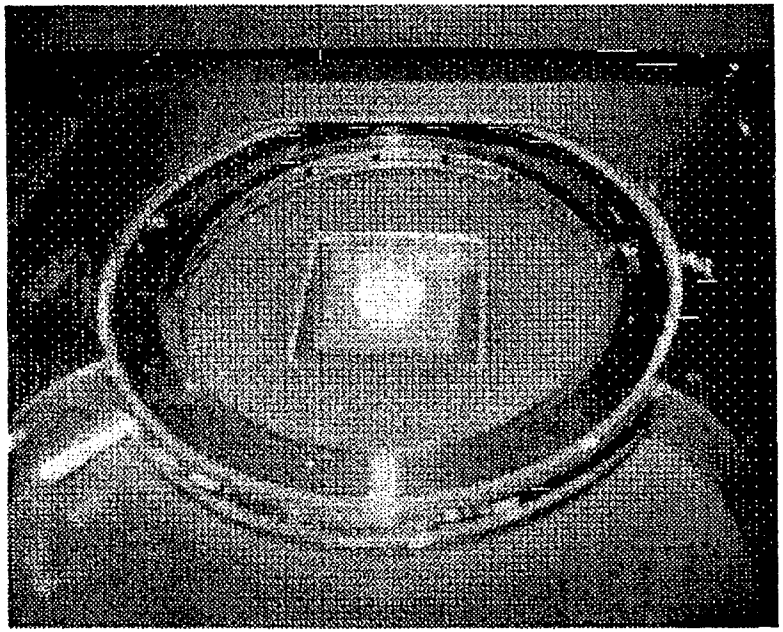
FIG. 6

Experimental Procedure

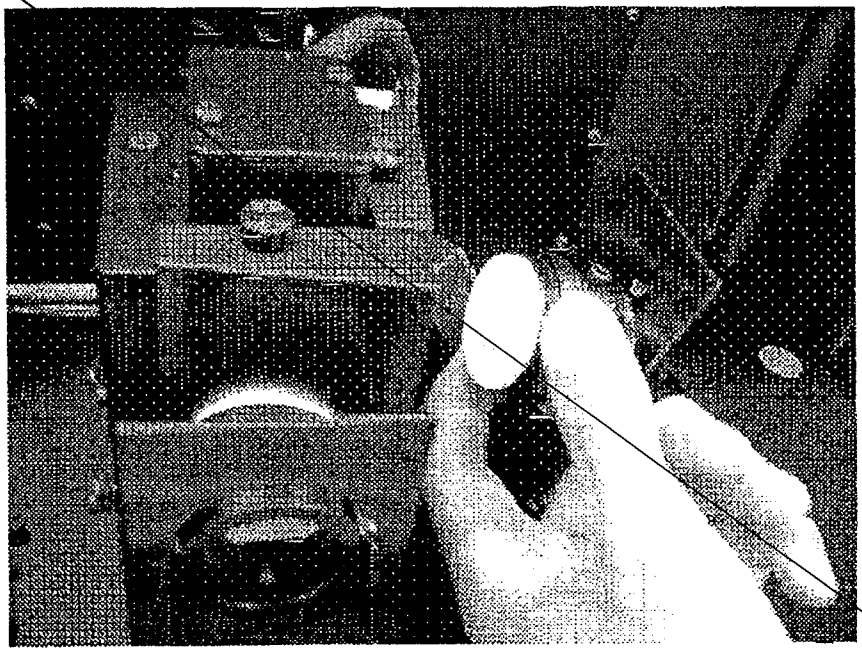


- Hydroxyapatite (HAP) disks are treated with solutions containing actives and measured:
  - Direct measurements with Near IR and UV
  - Conventional measurement by extraction and HPLC

Near IR



UV



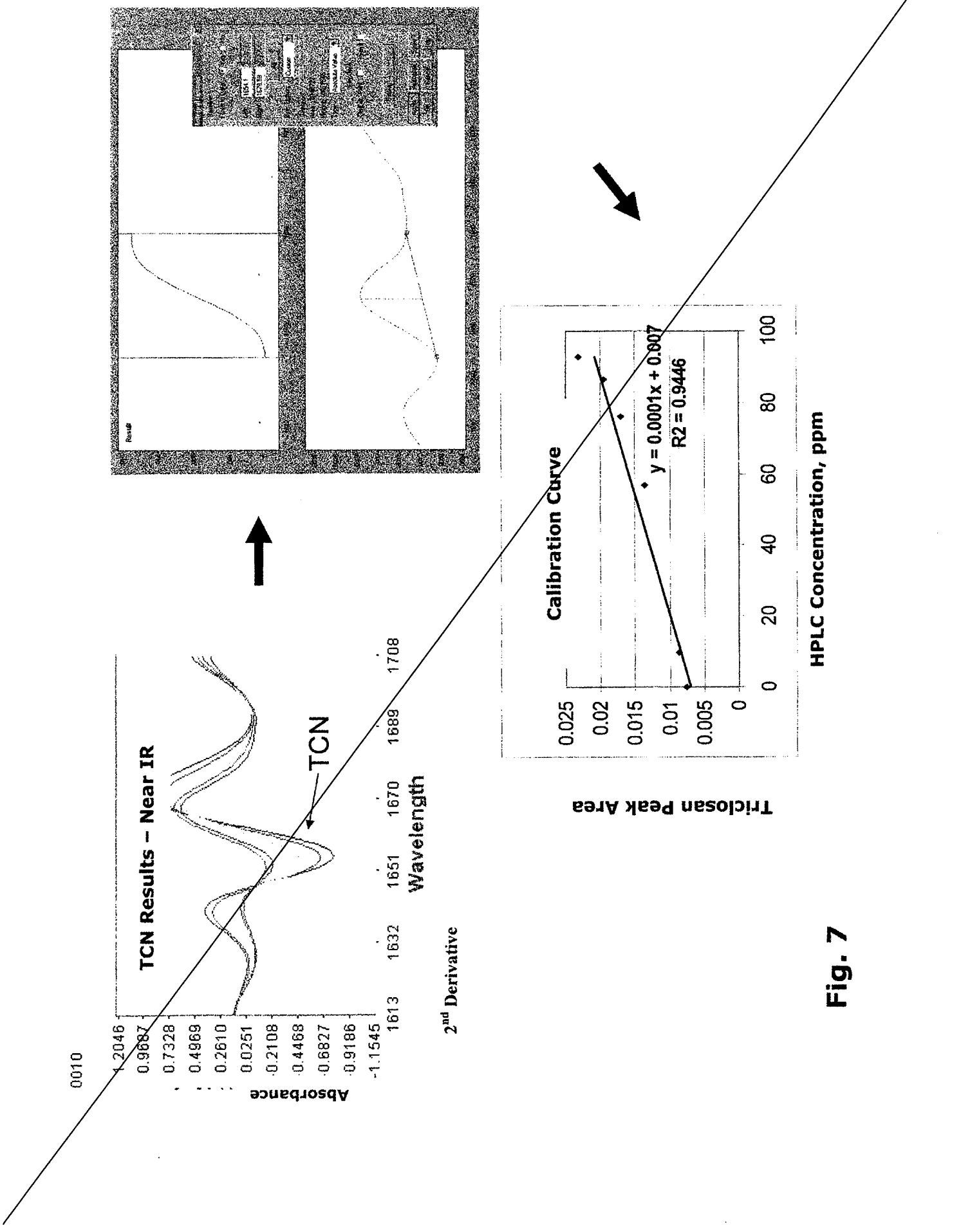

TCN Results – Near IR
TCN
Absorbance
Wavelength
2nd Derivative
Calibration Curve
y = 0.0001x + 0.007
R2 = 0.9446
Triclosan Peak Area
HPLC Concentration, ppm

Fig. 7

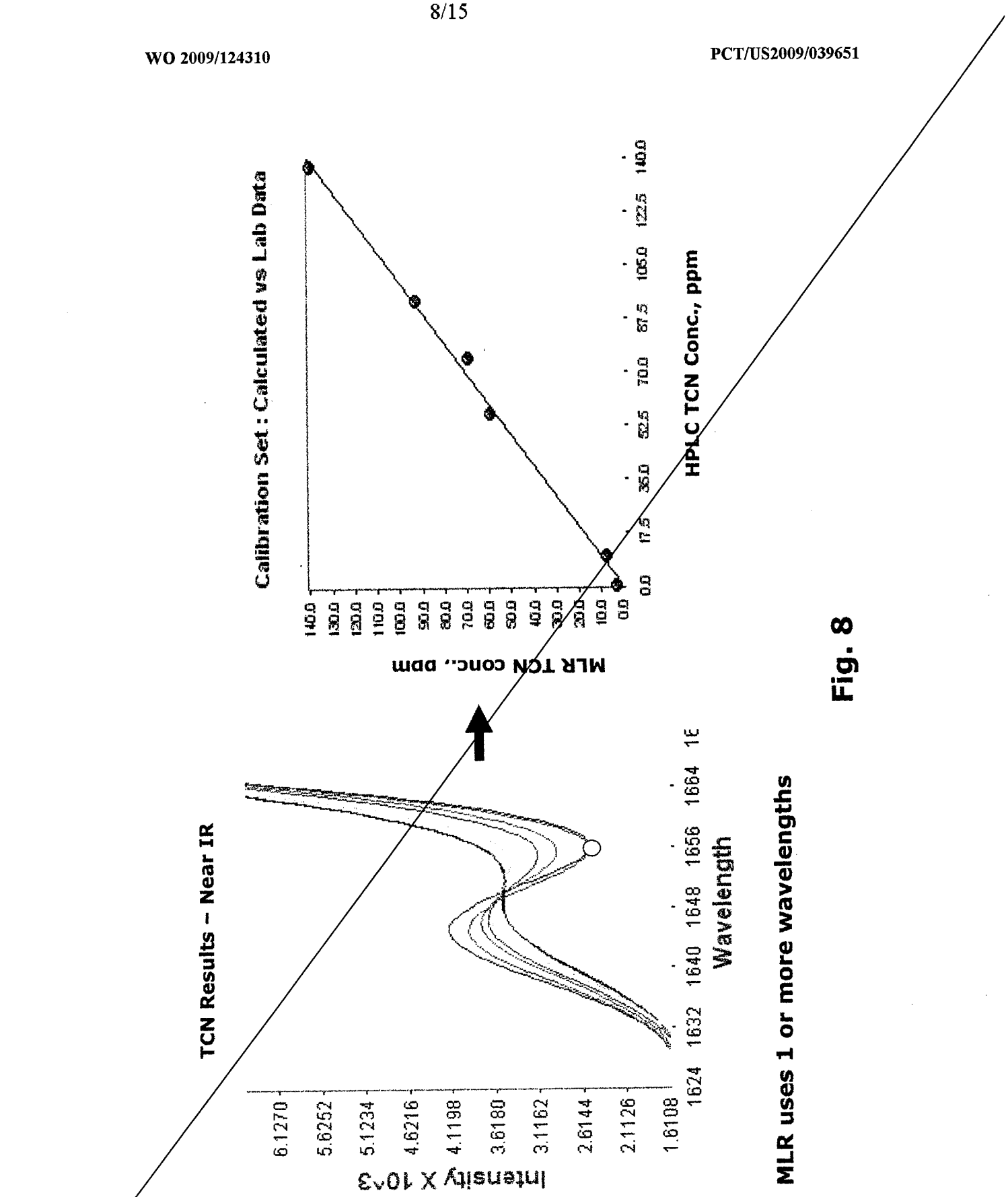

TCN Results – Near IR
Intensity X 10^3
6.1270
5.6252
5.1234
4.6216
4.1198
3.6180
3.1162
2.6144
2.1126
1.6108
1624
1632
1640
1648
1656
1664
Wavelength
MLR uses 1 or more wavelengths
MLR TCN conc., ppm
Calibration Set : Calculated vs Lab Data
HPLC TCN Conc., ppm

Fig. 8

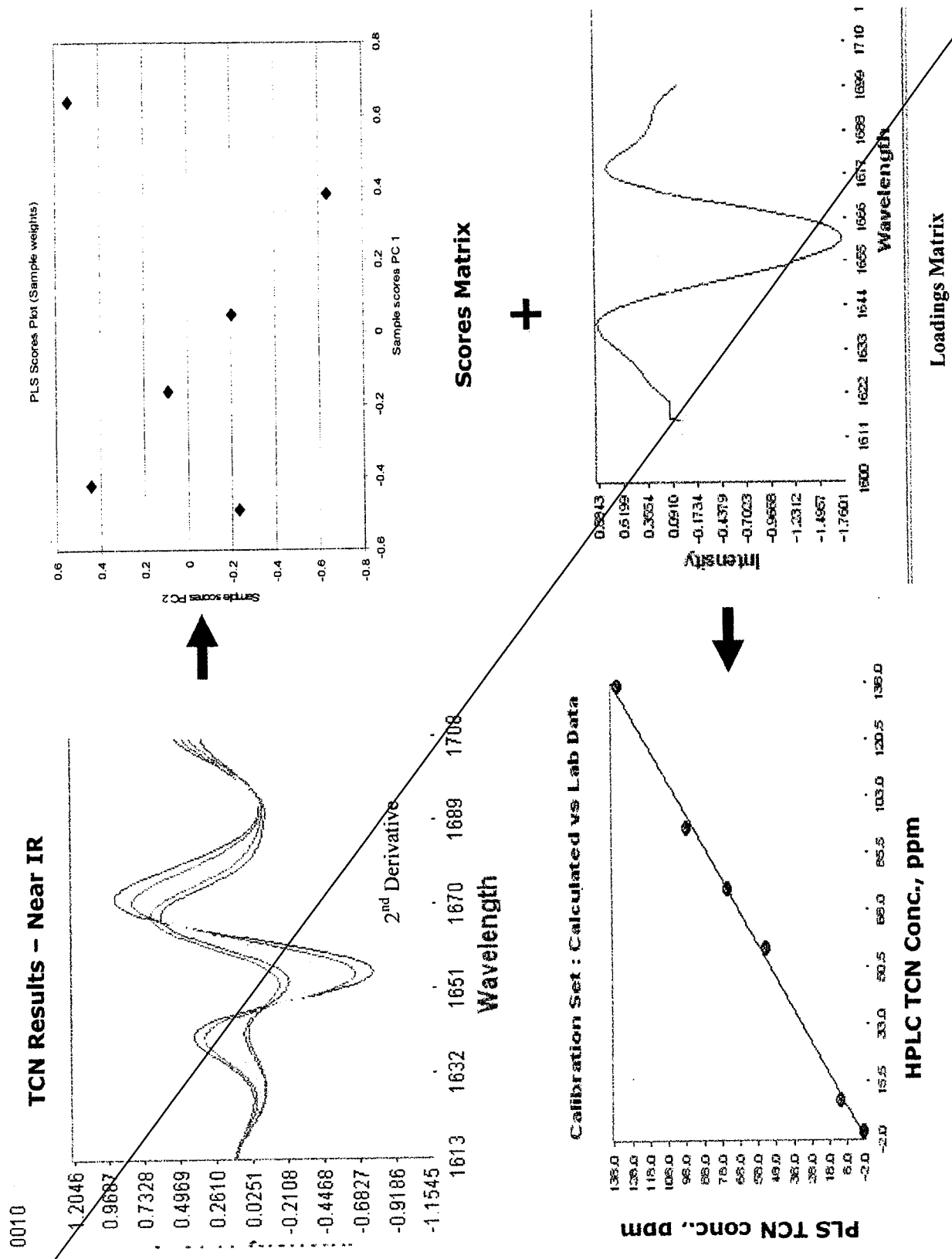
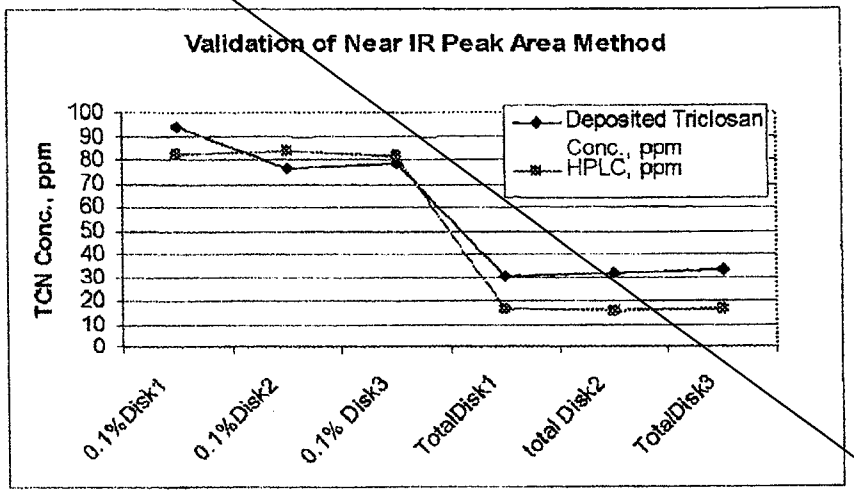

TCN Results – Near IR
Absorbance
Wavelength
2nd Derivative
Scores Matrix
PLS Scores Plot (Sample weights)
Sample scores PC 2
Sample scores PC 1
Loadings Matrix
Intensity
Wavelength
Calibration Set : Calculated vs Lab Data
PLS TCN conc., ppm
HPLC TCN Conc., ppm

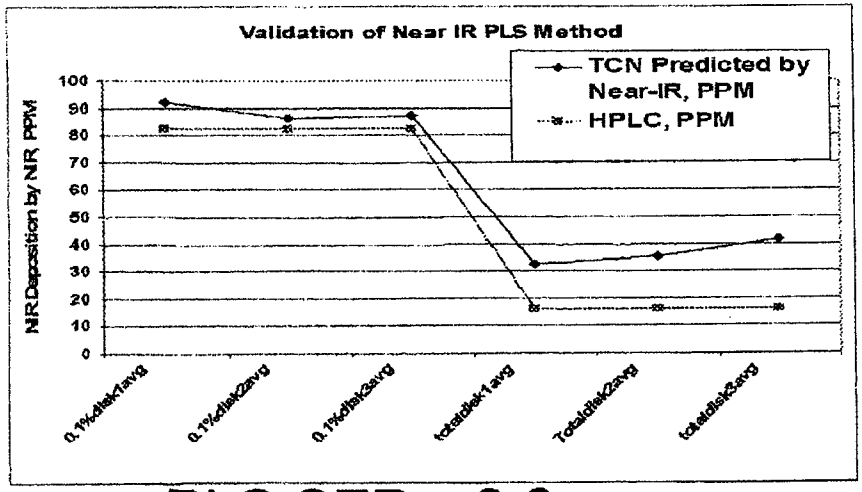
FIG. 9

Near IR Validation

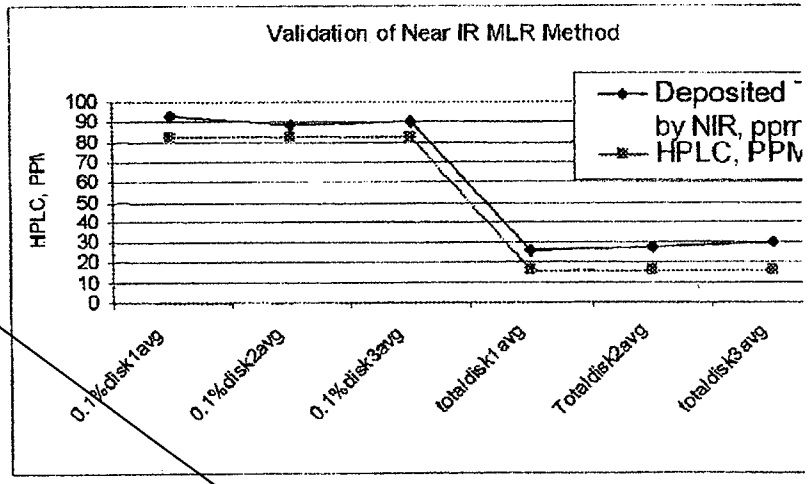
MLR was more effective than PLS & area methods



Peak Area SEP ± 10.5 ppm



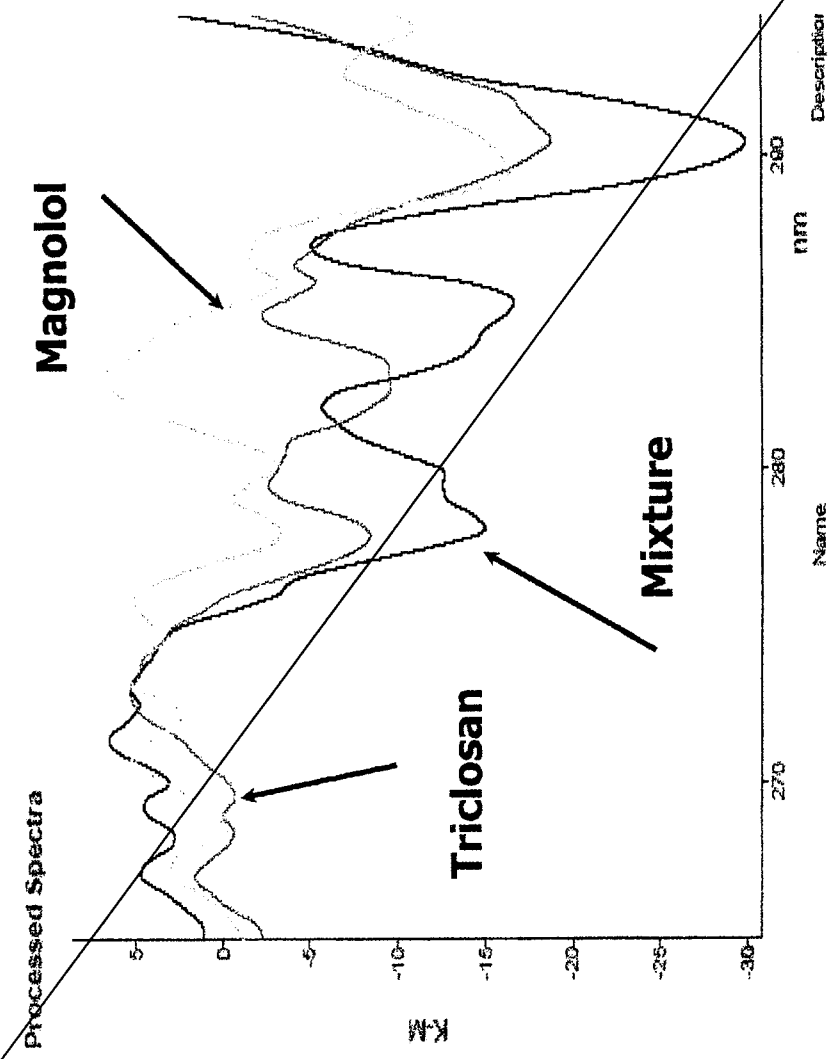
PLS SEP ± 8.6ppm



MLR SEP ± 2.8ppm

FIG. 10

Sample	Triclosan Peak Area	Mixed Active (1:1)	
5 min	4.4 units	52% Triclosan	48% Magnolol
30 min	18.1 units		



2nd Derivative of Actives

Fig. 11

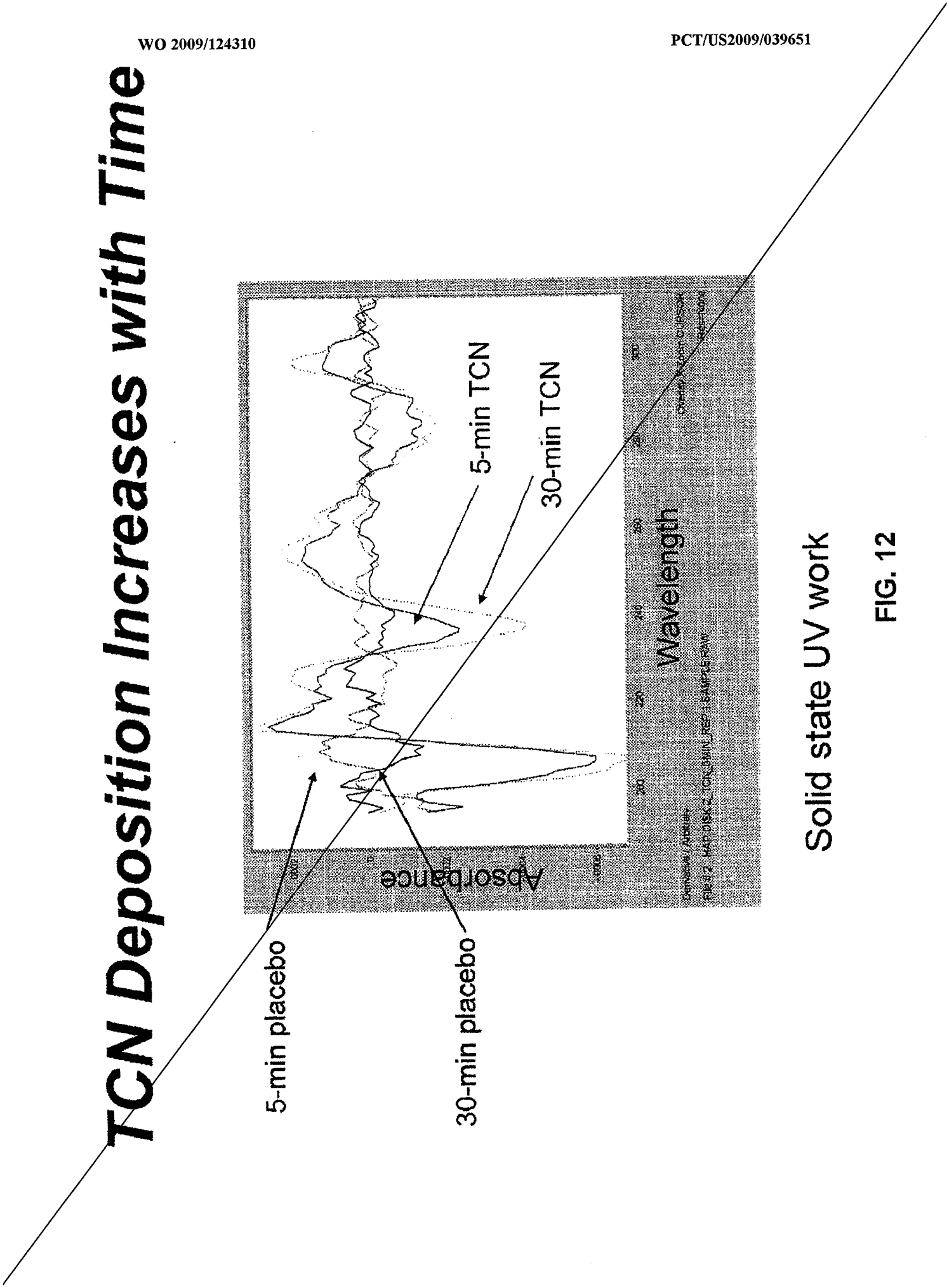

TCN Deposition Increases with Time
5-min TCN
30-min TCN
5-min placebo
30-min placebo
Absorbance
Wavelength
Solid state UV work

FIG. 12

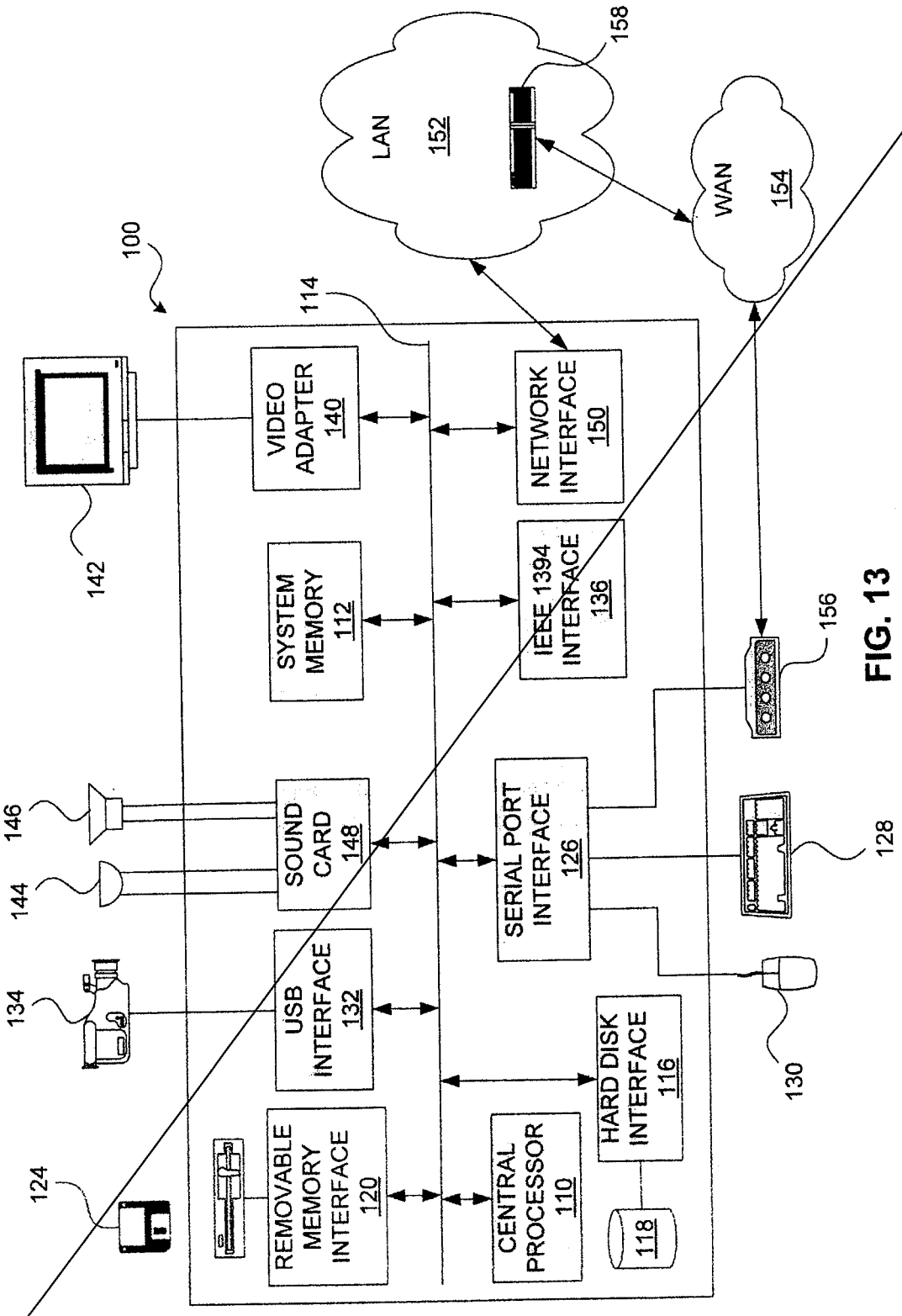
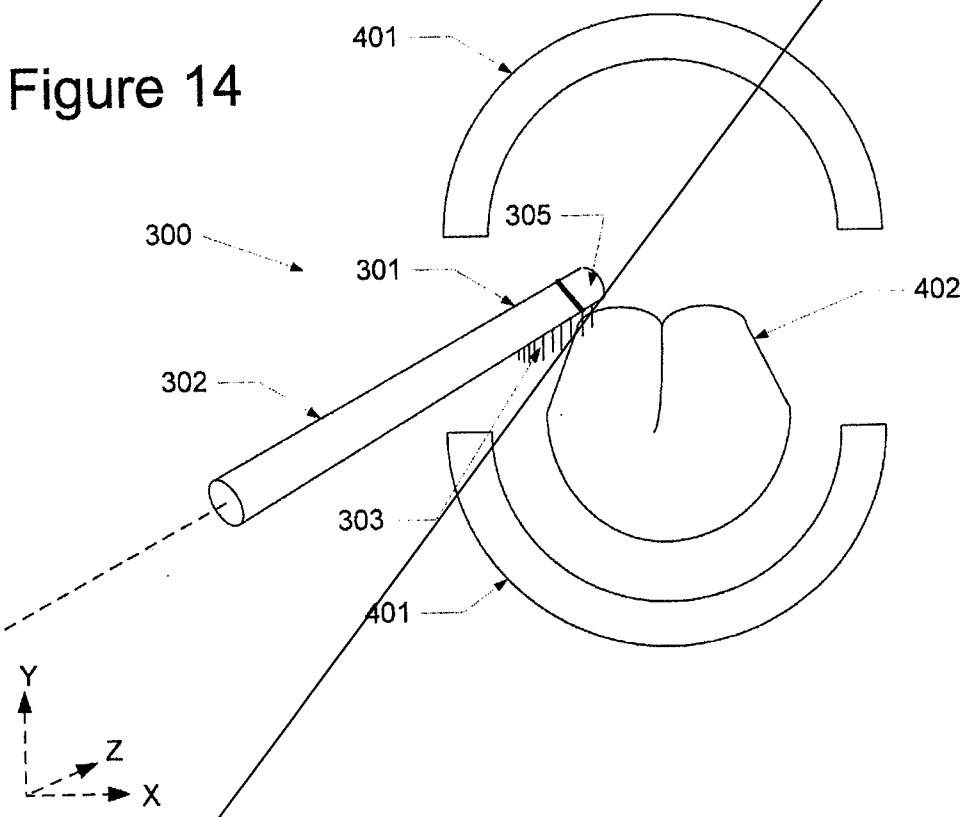

100
114
LAN
152
158
WAN
154
VIDEO ADAPTER 140
NETWORK INTERFACE 150
142
SYSTEM MEMORY 112
IEEE 1394 INTERFACE 136
156
146
SOUND CARD 148
SERIAL PORT INTERFACE 126
128
144
134
USB INTERFACE 132
HARD DISK INTERFACE 116
130
124
REMOVABLE MEMORY INTERFACE 120
CENTRAL PROCESSOR 110
118

FIG. 13

Figure 14



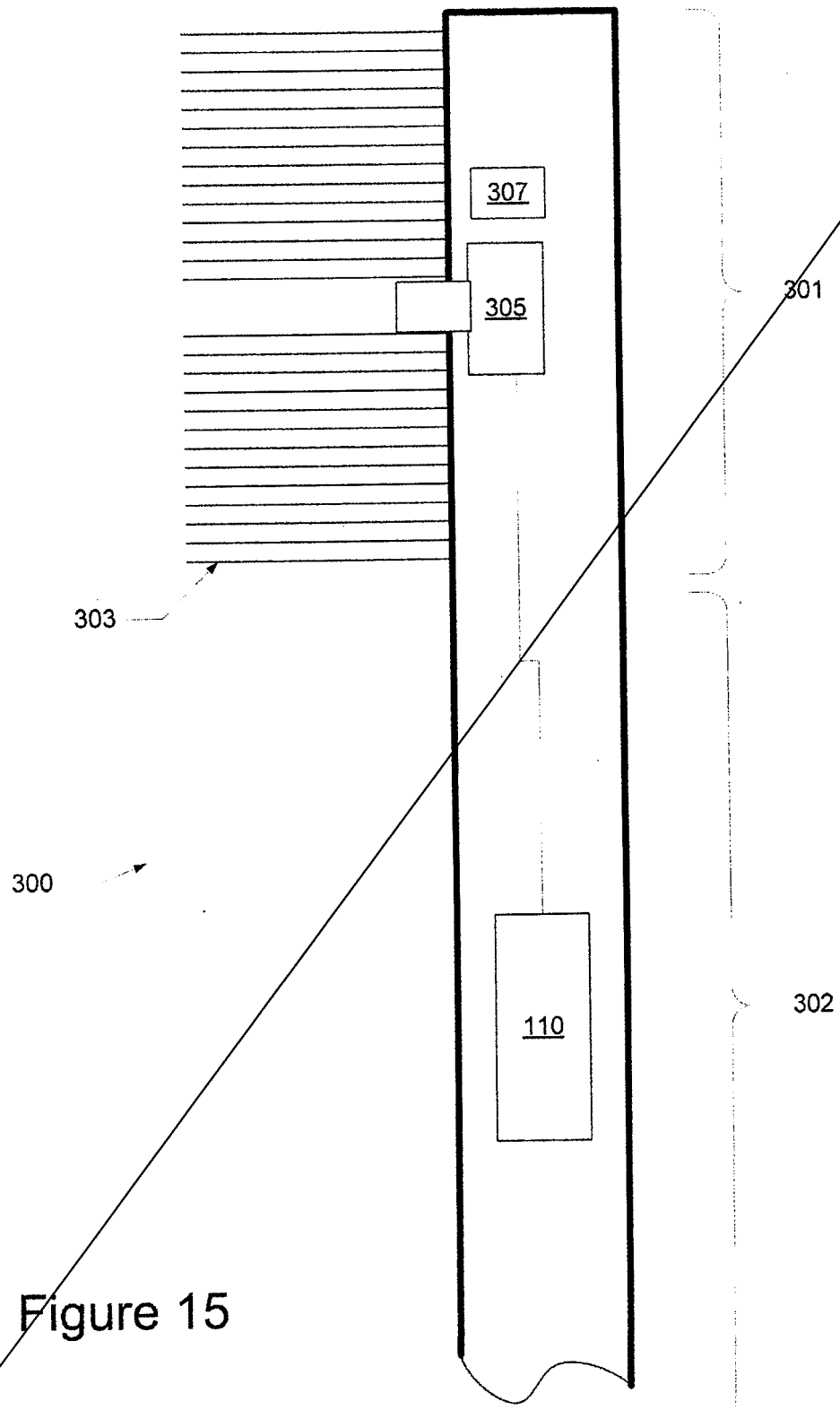


Figure 15