石墨炉原子吸收法直接测定油品中金属元素——砷和铅

作者

PerkinElmer, Inc.

引言

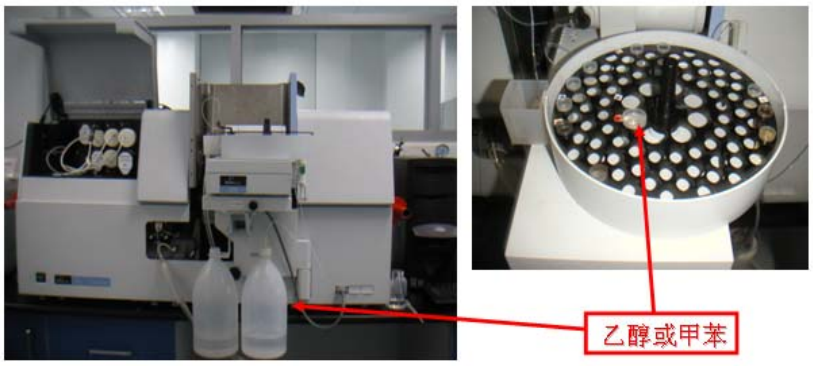
油品中的金属杂质，对加工或油品品质往往造成有害的影响，如：油品中的铅、砷等会导致催化剂中毒。因此，对油品中的痕量元素铅、砷的测定要求很高。目前，主要方法还是需要对油品进行湿法消化，这易导致油品损失，使结果偏低。

本方法采用甲苯稀释，用 PE AA600/800 原子吸收光谱仪，测定时，加钯和硝酸镁作为基体改进剂，同时在石墨炉干燥和灰化阶段中，加入一步通空气技术，消除积碳干扰，直接测定油品中的铅、砷等杂质元素。

实验结果表明：该方法无论在灵敏度、分析速度、准确度和操作等方面，都可以获得非常满意的结果。

仪器

使用 PerkinElmer AAnalyst600/800 原子吸收光谱仪，配以 AS800 自动进样器和 PerkinElmer 专用石墨管，WinLab32 for AA 软件，特殊通空气程序，消

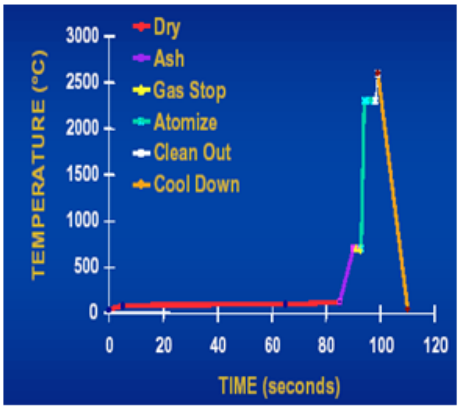


PerkinElmer

原 理

样品经进样器进样后，在石墨管中通过干燥、碳化、灰化、原子化等步骤，使样品蒸发离解形成原子蒸气，对来自光源的特征电磁辐射产生吸收，将测得的样品吸光度和标准吸光度进行比较，确定样品中被测元素的含量。

优点：无需消化，可以直接分析原油、石脑油、乙醇汽油等。检出限低ppb级，所需样品量少等。



特 点

1.THGA(横向加热)石墨炉

2.专利的纵向交流zeeman效应背景校正

3.新型内置直流ASCOM石墨炉电源

4.专利的TTC(真实温度控制)技术,石墨炉温度准确到±10℃, 重现性好

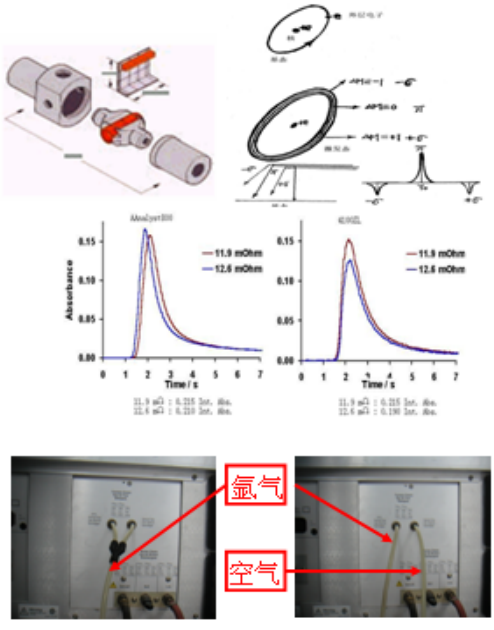
5.气动式锁紧石墨管,石墨管损坏自动报警

6.管内外气流分开,原子化阶段内管自动停气氩气耗量 <0.7L/min

7.自动节气停水技术

8.独特的通空气技术，消除有机物、氯化物等的干扰

9.专利的BOC(自动基线调零)技术,每次测定读数前，自动进行零点漂移校正, 克服由于灯、电路漂移或石墨炉两边石英窗变脏引的零点变化，长时间测定稳定性好



准 备

石墨炉分析

a) 特征质量

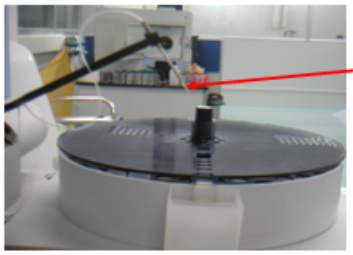
b) 灵敏度检查

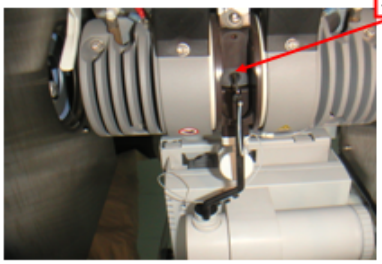
c) 基体改进剂配制

d) 进样针调节

e) 仪器推荐条件

f) STPF





标准和样品制备

1、无机标样配制：称取一定量水标样，用乙醇稀释至浓度为 50ng/g

2、有机标样配制：称取一定量有机标样，用甲苯稀释至 浓度为 50ng/g

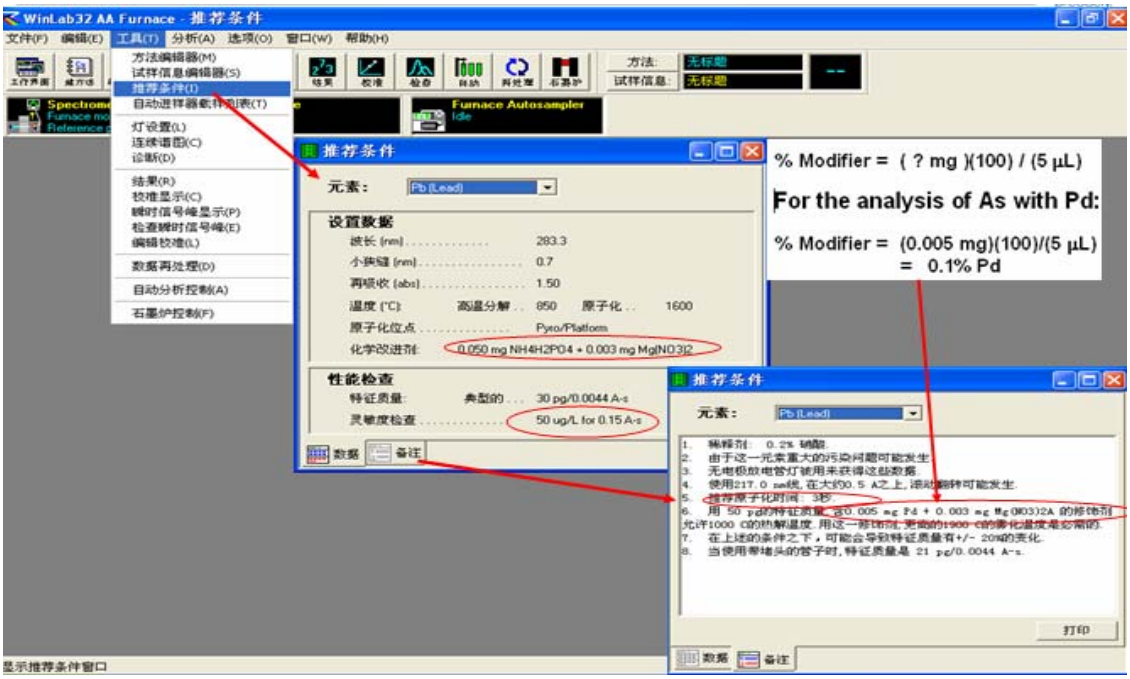
3、基体改进剂：称取一定量的 1%Pd 和 1%Mg(NO3)2，用乙醇稀释至浓度为 0.1%Pd 和 0.06%Mg(NO3)2

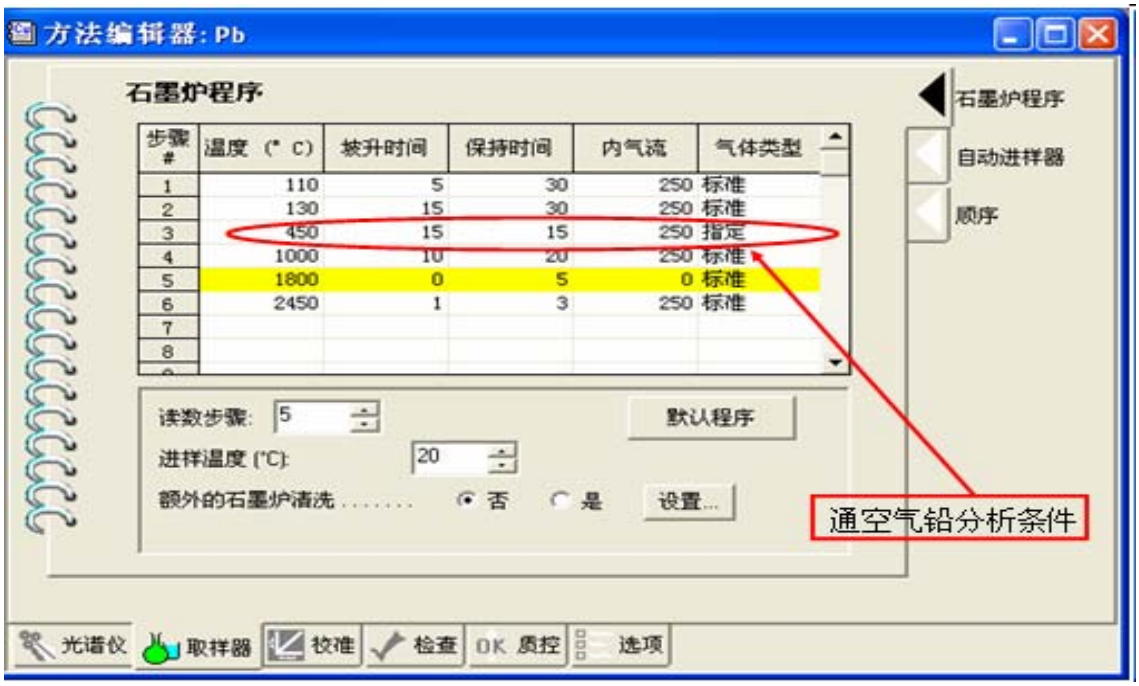
4、稀释液和清洗液：分析乙醇汽油样品时用乙醇，分析原油等样品时用甲苯

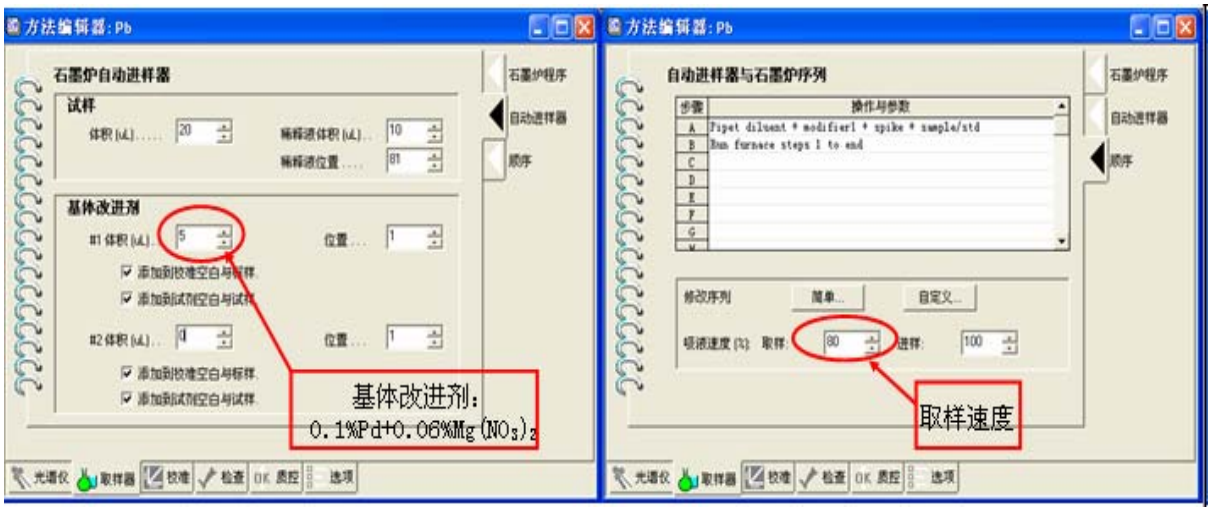
分析条件和结果

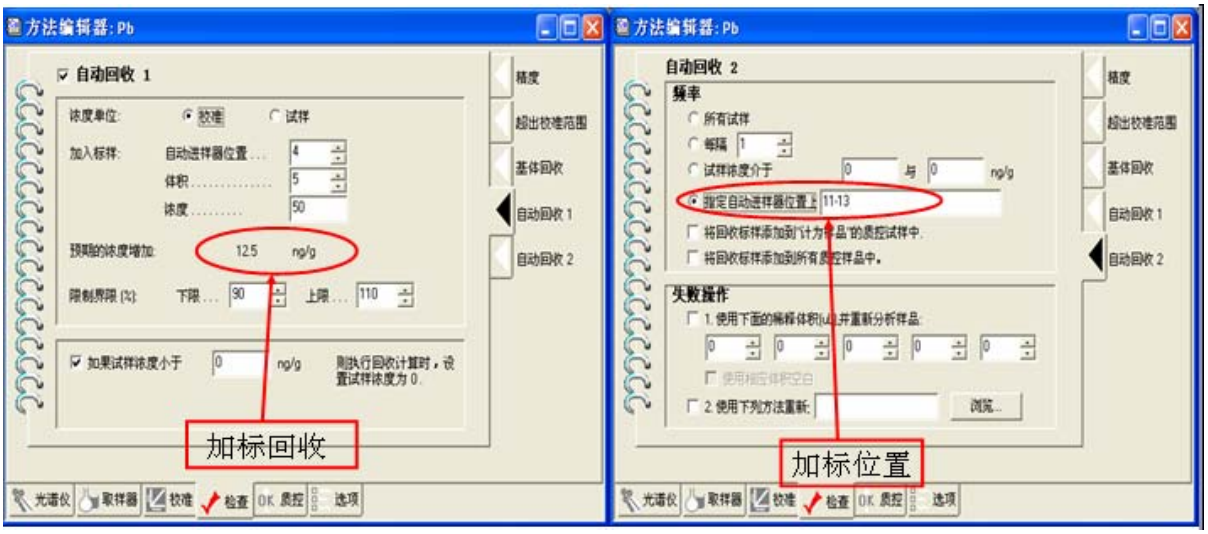
1、油品中铅的分析条件和结果

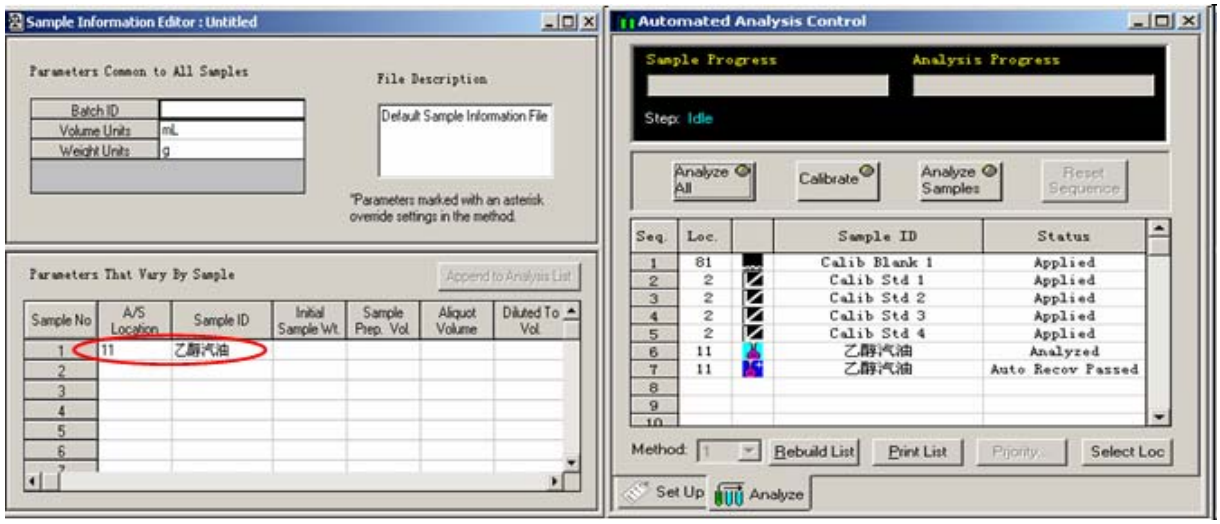
1.1、铅分析条件

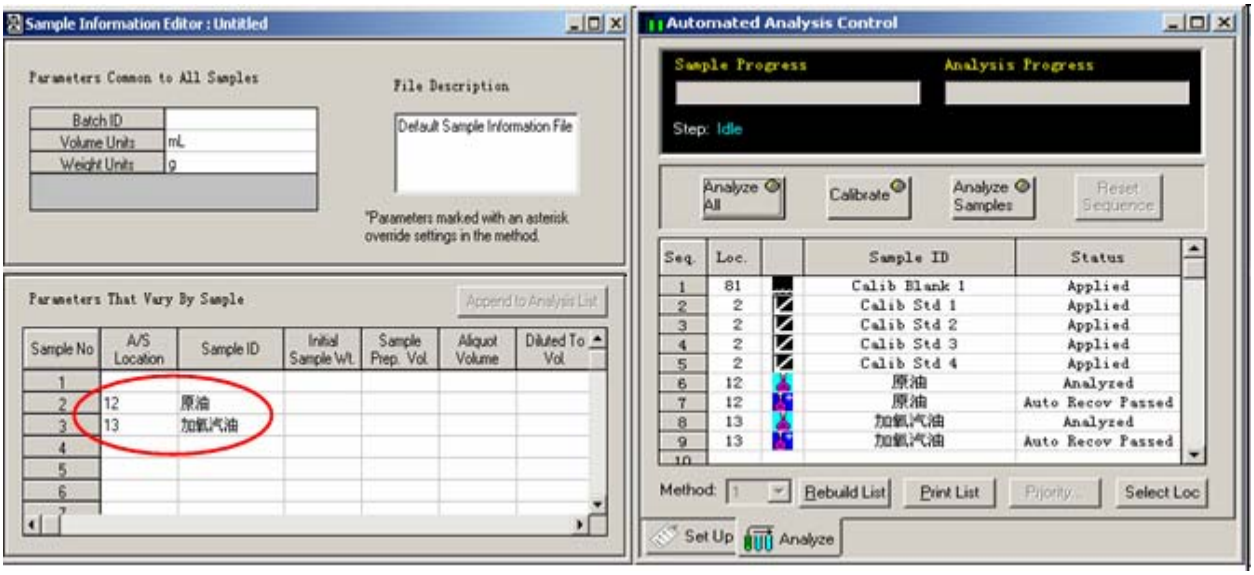





方法编辑器: Pb
石墨炉自动进样器
试样
体积 (uL)
基体改进剂
#1 体积 (uL)
基体改进剂：
0.1%Pd+0.06%Mg(NO3)2
方法编辑器: Pb
自动进样器与石墨炉序列
修改序列
简单...
自定义...
吸液速度 (%): 取样:
进样:
取样速度


方法编辑器: Pb
自动回收 1
浓度单位:
加入标样:
预期的浓度增加:
12.5
ng/g
限制界限 (%):
加标回收
方法编辑器: Pb
自动回收 2
频率
所有试样
每隔
指定自动进样器位置上
11-13
失败操作
加标位置

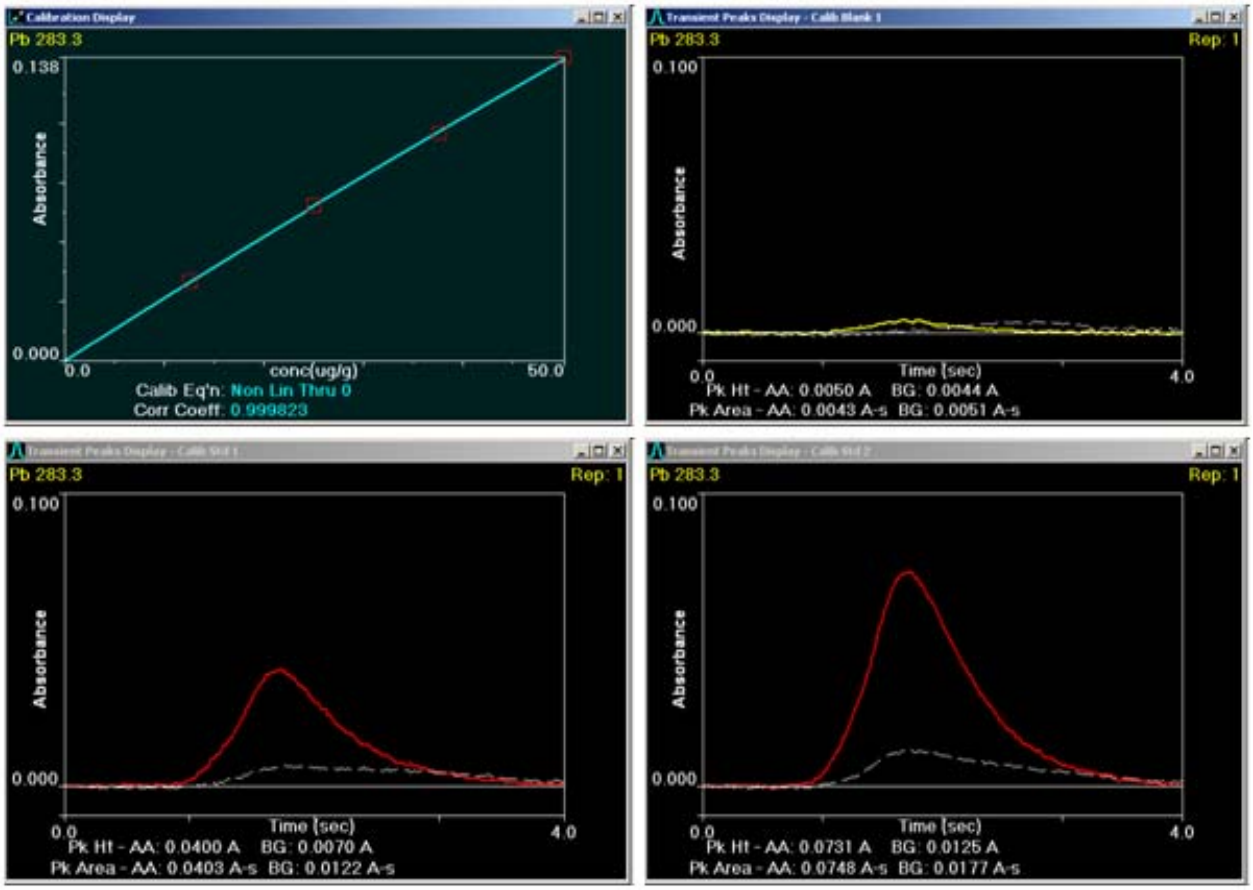

Sample Information Editor : Untitled
Parameters Common to All Samples
Batch ID
Volume Units
mL
Weight Units
g
File Description
Default Sample Information File
*Parameters marked with an asterisk override settings in the method.
Parameters That Vary By Sample
Append to Analysis List
Sample No
A/S Location
Sample ID
Initial Sample Wt.
Sample Prep. Vol.
Aliquot Volume
Diluted To Vol.
1
11
乙醇汽油
Automated Analysis Control
Sample Progress
Analysis Progress
Step: Idle
Analyze All
Calibrate
Analyze Samples
Reset Sequence
Seq.
Loc.
Sample ID
Status
1 81 Calib Blank 1 Applied
2 2 Calib Std 1 Applied
3 2 Calib Std 2 Applied
4 2 Calib Std 3 Applied
5 2 Calib Std 4 Applied
6 11 乙醇汽油 Analyzed
7 11 乙醇汽油 Auto Recov Passed
Method:
Rebuild List
Print List
Priority...
Select Loc
Set Up
Analyze

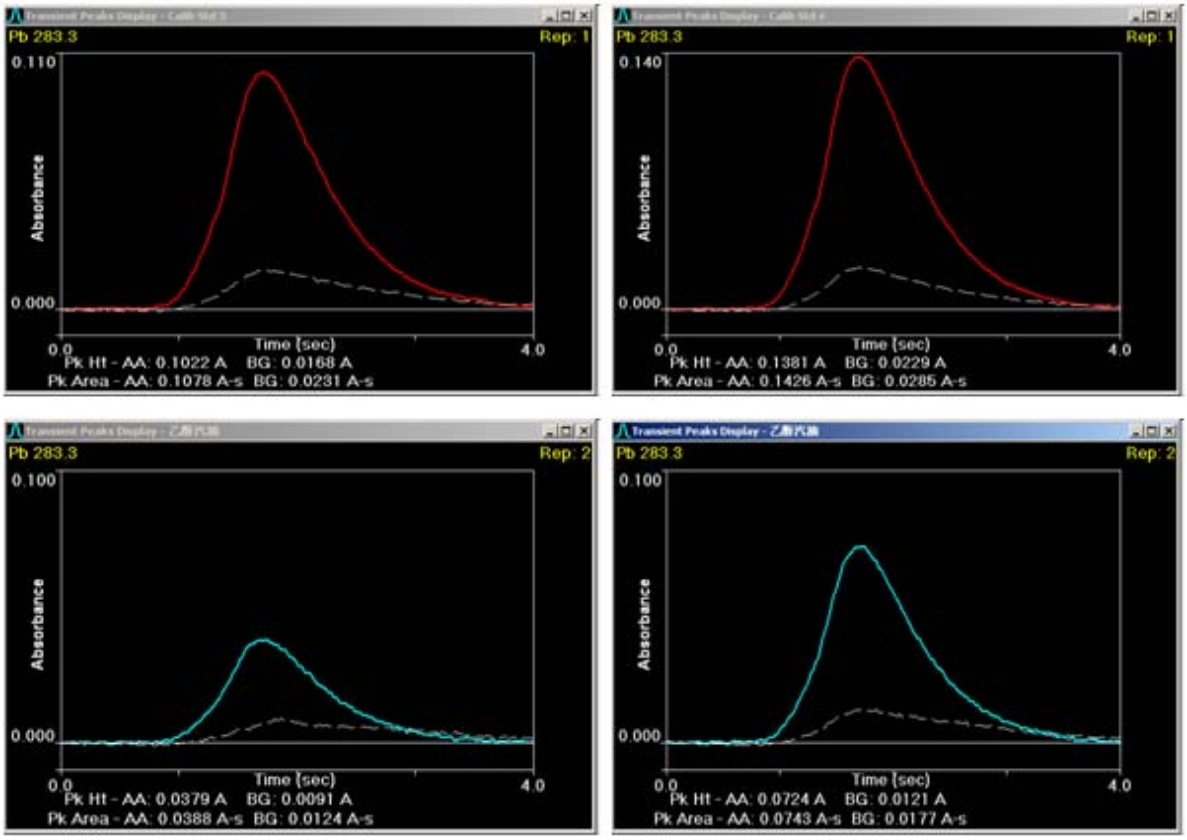


1.2 铅结果

1.2.1 乙醇汽油铅图谱和结果

1.2.1.1 乙醇汽油铅图谱

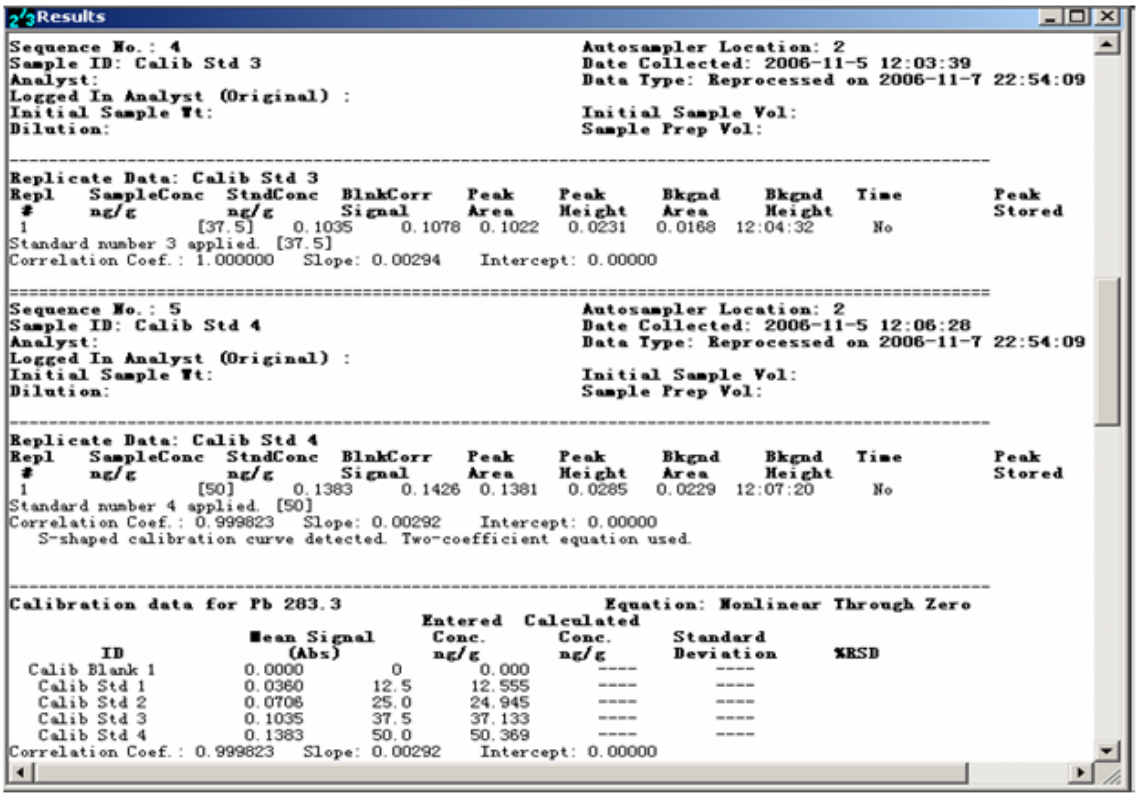




1.2.1.2 乙醇汽油铅结果

Results

```
Sequence No.: 1                                  Autosampler Location: 81
Sample ID: Calib Blank 1                         Date Collected: 2006-11-5 11:55:03
Analyst:                                         Data Type: Reprocessed on 2006-11-7 22:54:09
Logged In Analyst (Original) :
Initial Sample Wt:                               Initial Sample Vol:
Dilution:                                        Sample Prep Vol:

-------------------------------------------------------------------------------------------------
Replicate Data: Calib Blank 1
Repl   SampleConc  StndConc  BlnkCorr   Peak     Peak     Bkgnd    Bkgnd   Time          Peak
 #       ng/g       ng/g      Signal    Area     Height   Area     Height                Stored
 1                [0.00]     0.0043    0.0043   0.0050   0.0051   0.0044  11:56:07       No
Auto-zero performed.

=================================================================================================
Sequence No.: 2                                  Autosampler Location: 2
Sample ID: Calib Std 1                           Date Collected: 2006-11-5 11:58:03
Analyst:                                         Data Type: Reprocessed on 2006-11-7 22:54:09
Logged In Analyst (Original) :
Initial Sample Wt:                               Initial Sample Vol:
Dilution:                                        Sample Prep Vol:

-------------------------------------------------------------------------------------------------
Replicate Data: Calib Std 1
Repl   SampleConc  StndConc  BlnkCorr   Peak     Peak     Bkgnd    Bkgnd   Time          Peak
 #       ng/g       ng/g      Signal    Area     Height   Area     Height                Stored
 1              [12.5]       0.0360    0.0403   0.0400   0.0122   0.0070  11:58:55       No
Standard number 1 applied. [12.5]
Correlation Coef.: 1.000000   Slope: 0.00288    Intercept: 0.00000

=================================================================================================
Sequence No.: 3                                  Autosampler Location: 2
Sample ID: Calib Std 2                           Date Collected: 2006-11-5 12:00:51
Analyst:                                         Data Type: Reprocessed on 2006-11-7 22:54:09
Logged In Analyst (Original) :
Initial Sample Wt:                               Initial Sample Vol:
Dilution:                                        Sample Prep Vol:

-------------------------------------------------------------------------------------------------
Replicate Data: Calib Std 2
Repl   SampleConc  StndConc  BlnkCorr   Peak     Peak     Bkgnd    Bkgnd   Time          Peak
 #       ng/g       ng/g      Signal    Area     Height   Area     Height                Stored
 1              [25]         0.0706    0.0748   0.0731   0.0177   0.0125  12:01:43       No
Standard number 2 applied. [25]
```



Results

Sequence No.: 4
Sample ID: Calib Std 3
Analyst:
Logged In Analyst (Original) :
Initial Sample Wt:
Dilution:

Autosampler Location: 2
Date Collected: 2006-11-5 12:03:39
Data Type: Reprocessed on 2006-11-7 22:54:09

Initial Sample Vol:
Sample Prep Vol:

Replicate Data: Calib Std 3

Repl #	SampleConc ng/g	StndConc ng/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	[37.5]	0.1035	0.1078	0.1022	0.0231	0.0168	12:04:32		No

Standard number 3 applied. [37.5]
Correlation Coef.: 1.000000 Slope: 0.00294 Intercept: 0.00000

Sequence No.: 5
Sample ID: Calib Std 4
Analyst:
Logged In Analyst (Original) :
Initial Sample Wt:
Dilution:

Autosampler Location: 2
Date Collected: 2006-11-5 12:06:28
Data Type: Reprocessed on 2006-11-7 22:54:09

Initial Sample Vol:
Sample Prep Vol:

Replicate Data: Calib Std 4

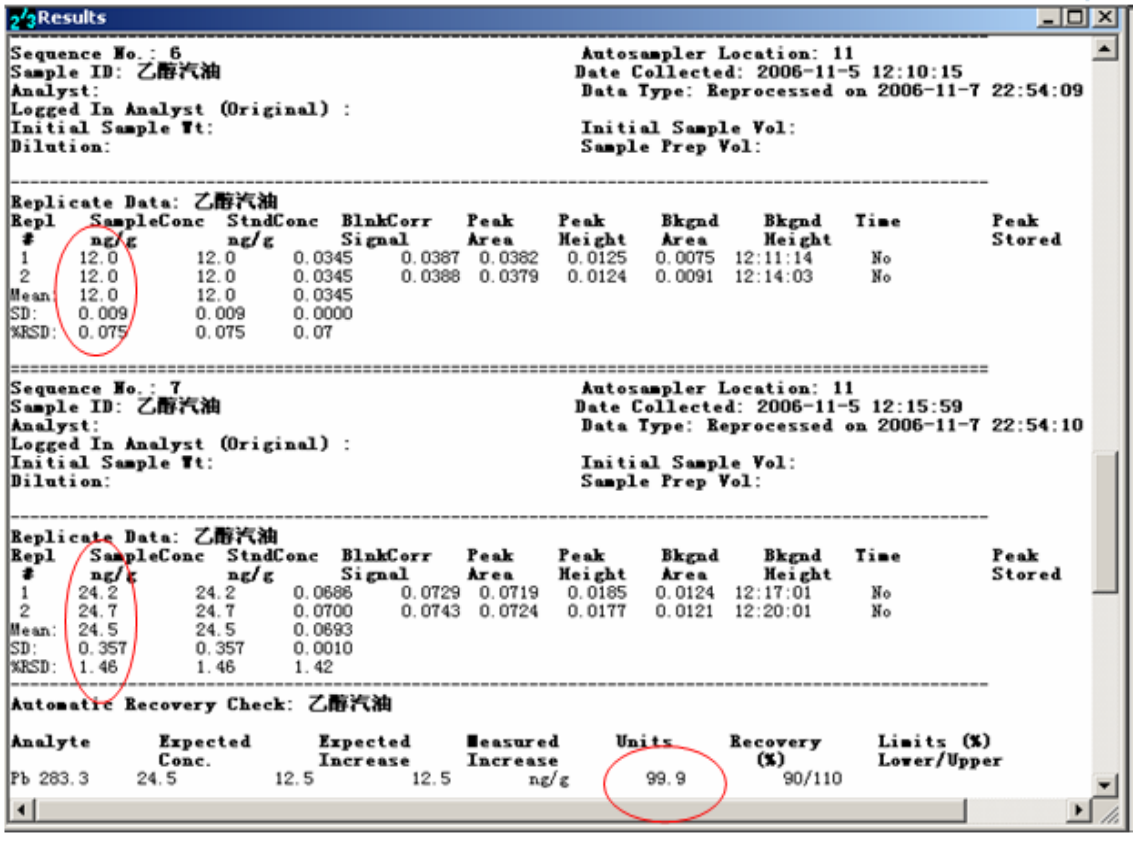
Repl #	SampleConc ng/g	StndConc ng/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	[50]	0.1383	0.1426	0.1381	0.0285	0.0229	12:07:20		No

Standard number 4 applied. [50]
Correlation Coef.: 0.999823 Slope: 0.00292 Intercept: 0.00000
S-shaped calibration curve detected. Two-coefficient equation used.

Calibration data for Pb 283.3 Equation: Nonlinear Through Zero

ID	Mean Signal (Abs)	Entered Conc. ng/g	Calculated Conc. ng/g	Standard Deviation	%RSD
Calib Blank 1	0.0000	0	0.000	----	----
Calib Std 1	0.0360	12.5	12.555	----	----
Calib Std 2	0.0706	25.0	24.945	----	----
Calib Std 3	0.1035	37.5	37.133	----	----
Calib Std 4	0.1383	50.0	50.369	----	----

Correlation Coef.: 0.999823 Slope: 0.00292 Intercept: 0.00000



Results

Sequence No.: 6
Sample ID: 乙醇汽油
Analyst:
Logged In Analyst (Original) :
Initial Sample Wt:
Dilution:

Autosampler Location: 11
Date Collected: 2006-11-5 12:10:15
Data Type: Reprocessed on 2006-11-7 22:54:09

Initial Sample Vol:
Sample Prep Vol:

Replicate Data: 乙醇汽油

Repl #	SampleConc ng/g	StndConc ng/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	12.0	12.0	0.0345	0.0387	0.0382	0.0125	0.0075	12:11:14	No
2	12.0	12.0	0.0345	0.0388	0.0379	0.0124	0.0091	12:14:03	No
Mean:	12.0	12.0	0.0345						
SD:	0.009	0.009	0.0000						
%RSD:	0.075	0.075	0.07						

Sequence No.: 7
Sample ID: 乙醇汽油
Analyst:
Logged In Analyst (Original) :
Initial Sample Wt:
Dilution:

Autosampler Location: 11
Date Collected: 2006-11-5 12:15:59
Data Type: Reprocessed on 2006-11-7 22:54:10

Initial Sample Vol:
Sample Prep Vol:

Replicate Data: 乙醇汽油

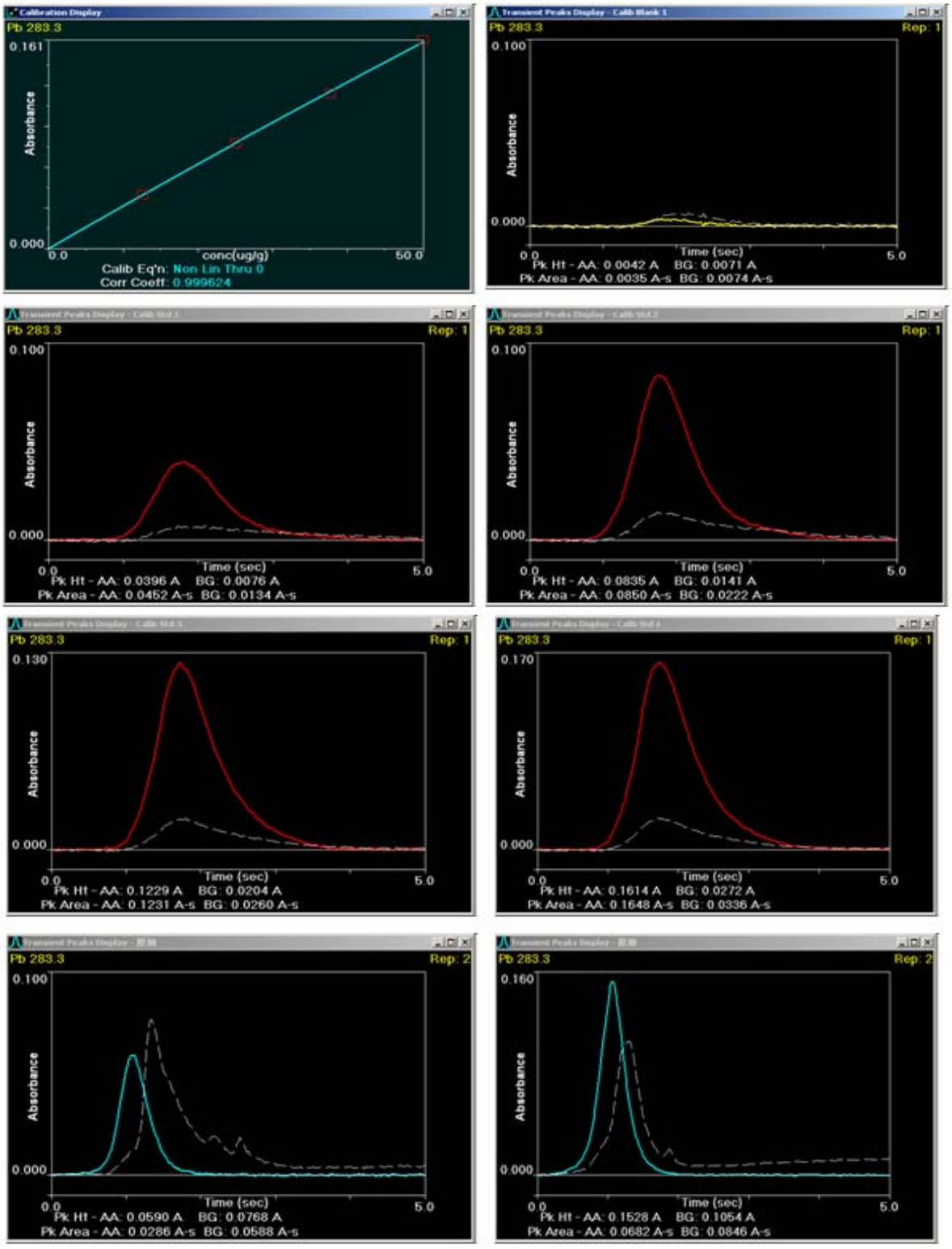
Repl #	SampleConc ng/g	StndConc ng/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	24.2	24.2	0.0686	0.0729	0.0719	0.0185	0.0124	12:17:01	No
2	24.7	24.7	0.0700	0.0743	0.0724	0.0177	0.0121	12:20:01	No
Mean:	24.5	24.5	0.0693						
SD:	0.357	0.357	0.0010						
%RSD:	1.46	1.46	1.42						

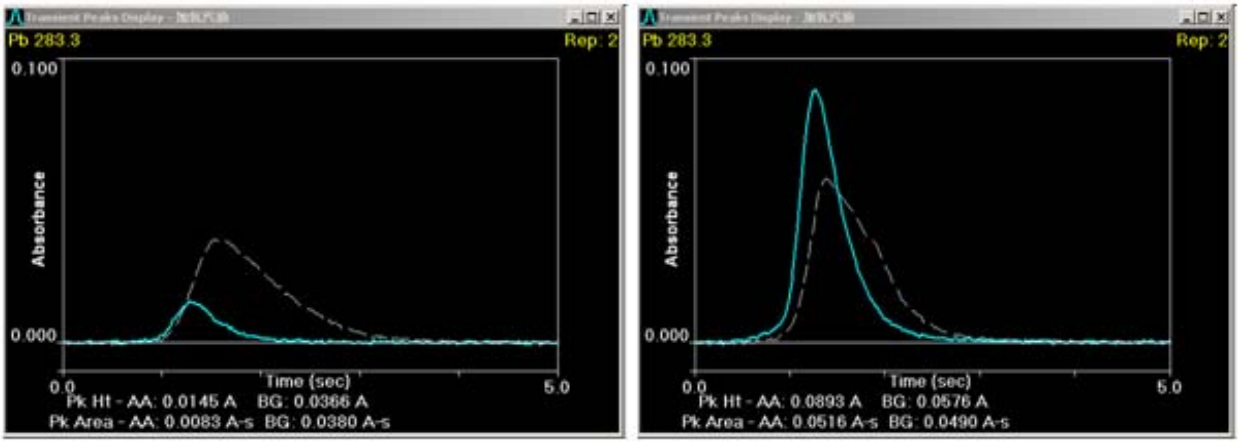
Automatic Recovery Check: 乙醇汽油

Analyte	Expected Conc.	Expected Increase	Measured Increase	Units	Recovery (%)	Limits (%) Lower/Upper
Pb 283.3	24.5	12.5	12.5	ng/g	99.9	90/110

1.2.2 原油铅图谱和结果

1.2.2.1 原油铅图谱

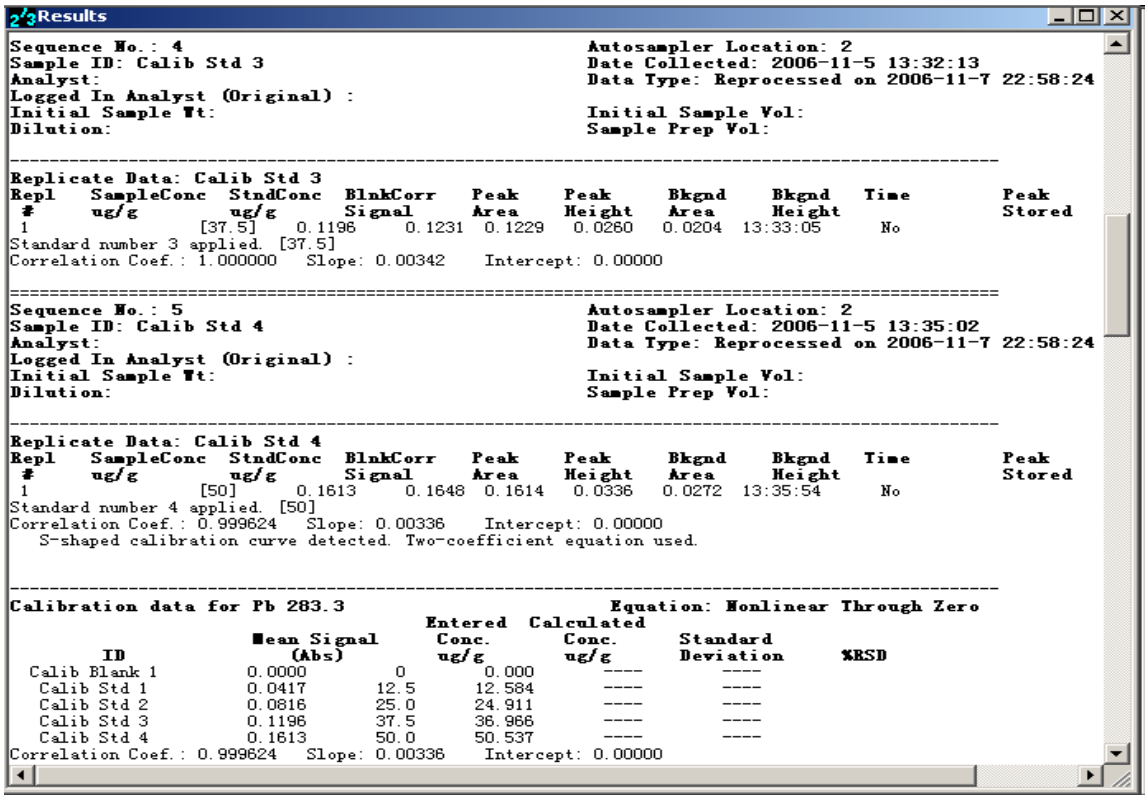




1.2.2.2 原油铅图谱

Results

```
--------------------------------------------------------------------------------------------------------
Replicate Data: Calib Blank 1
Repl   SampleConc  StndConc  BlnkCorr   Peak     Peak     Bkgnd    Bkgnd   Time         Peak
 #       ug/g        ug/g      Signal    Area     Height   Area     Height               Stored
 1              [0.00]    0.0035     0.0035  0.0042   0.0074   0.0071  13:24:38      No
Auto-zero performed.

========================================================================================================
Sequence No.: 2                                  Autosampler Location: 2
Sample ID: Calib Std 1                           Date Collected: 2006-11-5 13:26:35
Analyst:                                         Data Type: Reprocessed on 2006-11-7 22:58:23
Logged In Analyst (Original) :
Initial Sample Wt:                               Initial Sample Vol:
Dilution:                                        Sample Prep Vol:

--------------------------------------------------------------------------------------------------------
Replicate Data: Calib Std 1
Repl   SampleConc  StndConc  BlnkCorr   Peak     Peak     Bkgnd    Bkgnd   Time         Peak
 #       ug/g        ug/g      Signal    Area     Height   Area     Height               Stored
 1             [12.5]    0.0417     0.0452  0.0396   0.0134   0.0076  13:27:27      No
Standard number 1 applied. [12.5]
Correlation Coef.: 1.000000   Slope: 0.00334    Intercept: 0.00000

========================================================================================================
Sequence No.: 3                                  Autosampler Location: 2
Sample ID: Calib Std 2                           Date Collected: 2006-11-5 13:29:24
Analyst:                                         Data Type: Reprocessed on 2006-11-7 22:58:23
Logged In Analyst (Original) :
Initial Sample Wt:                               Initial Sample Vol:
Dilution:                                        Sample Prep Vol:

--------------------------------------------------------------------------------------------------------
Replicate Data: Calib Std 2
Repl   SampleConc  StndConc  BlnkCorr   Peak     Peak     Bkgnd    Bkgnd   Time         Peak
 #       ug/g        ug/g      Signal    Area     Height   Area     Height               Stored
 1              [25]      0.0816     0.0850  0.0835   0.0222   0.0141  13:30:16      No
Standard number 2 applied. [25]
Correlation Coef.: 1.000000   Slope: 0.00342    Intercept: 0.00000
```



Results

Sequence No.: 4 — Autosampler Location: 2
Sample ID: Calib Std 3 — Date Collected: 2006-11-5 13:32:13
Analyst: — Data Type: Reprocessed on 2006-11-7 22:58:24
Logged In Analyst (Original) :
Initial Sample Wt: — Initial Sample Vol:
Dilution: — Sample Prep Vol:

Replicate Data: Calib Std 3

Repl #	SampleConc ug/g	StndConc ug/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	[37.5]	0.1196	0.1231	0.1229	0.0260	0.0204	13:33:05	No	

Standard number 3 applied. [37.5]
Correlation Coef.: 1.000000 Slope: 0.00342 Intercept: 0.00000

Sequence No.: 5 — Autosampler Location: 2
Sample ID: Calib Std 4 — Date Collected: 2006-11-5 13:35:02
Analyst: — Data Type: Reprocessed on 2006-11-7 22:58:24
Logged In Analyst (Original) :
Initial Sample Wt: — Initial Sample Vol:
Dilution: — Sample Prep Vol:

Replicate Data: Calib Std 4

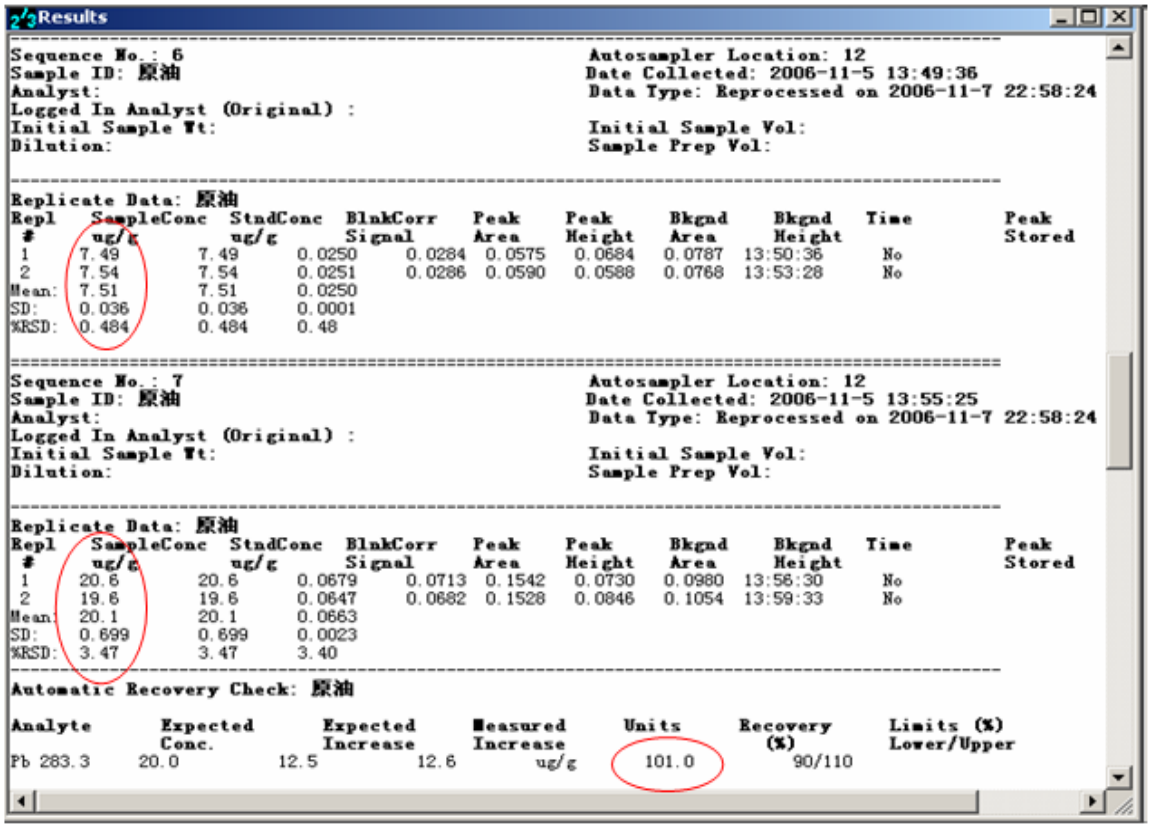
Repl #	SampleConc ug/g	StndConc ug/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	[50]	0.1613	0.1648	0.1614	0.0336	0.0272	13:35:54	No	

Standard number 4 applied. [50]
Correlation Coef.: 0.999624 Slope: 0.00336 Intercept: 0.00000
S-shaped calibration curve detected. Two-coefficient equation used.

Calibration data for Pb 283.3 — Equation: Nonlinear Through Zero

ID	Mean Signal (Abs)	Entered Conc. ug/g	Calculated Conc. ug/g	Standard Deviation	%RSD
Calib Blank 1	0.0000	0	0.000	----	----
Calib Std 1	0.0417	12.5	12.584	----	----
Calib Std 2	0.0816	25.0	24.911	----	----
Calib Std 3	0.1196	37.5	36.966	----	----
Calib Std 4	0.1613	50.0	50.537	----	----

Correlation Coef.: 0.999624 Slope: 0.00336 Intercept: 0.00000



Results

Sequence No.: 6 — Autosampler Location: 12
Sample ID: 原油 — Date Collected: 2006-11-5 13:49:36
Analyst: — Data Type: Reprocessed on 2006-11-7 22:58:24
Logged In Analyst (Original) :
Initial Sample Wt: — Initial Sample Vol:
Dilution: — Sample Prep Vol:

Replicate Data: 原油

Repl #	SampleConc ug/g	StndConc ug/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	7.49	7.49	0.0250	0.0284	0.0575	0.0684	0.0787	13:50:36	No
2	7.54	7.54	0.0251	0.0286	0.0590	0.0588	0.0768	13:53:28	No
Mean:	7.51	7.51	0.0250						
SD:	0.036	0.036	0.0001						
%RSD:	0.484	0.484	0.48						

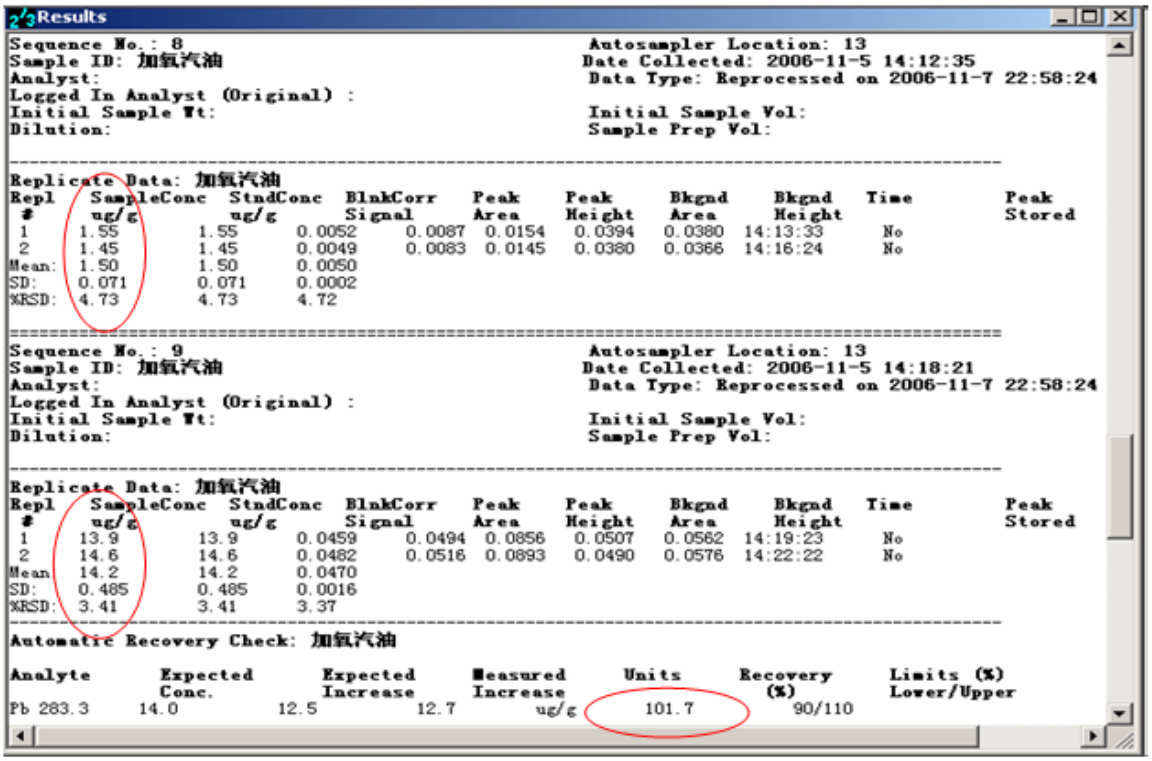
Sequence No.: 7 — Autosampler Location: 12
Sample ID: 原油 — Date Collected: 2006-11-5 13:55:25
Analyst: — Data Type: Reprocessed on 2006-11-7 22:58:24
Logged In Analyst (Original) :
Initial Sample Wt: — Initial Sample Vol:
Dilution: — Sample Prep Vol:

Replicate Data: 原油

Repl #	SampleConc ug/g	StndConc ug/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	20.6	20.6	0.0679	0.0713	0.1542	0.0730	0.0980	13:56:30	No
2	19.6	19.6	0.0647	0.0682	0.1528	0.0846	0.1054	13:59:33	No
Mean:	20.1	20.1	0.0663						
SD:	0.699	0.699	0.0023						
%RSD:	3.47	3.47	3.40						

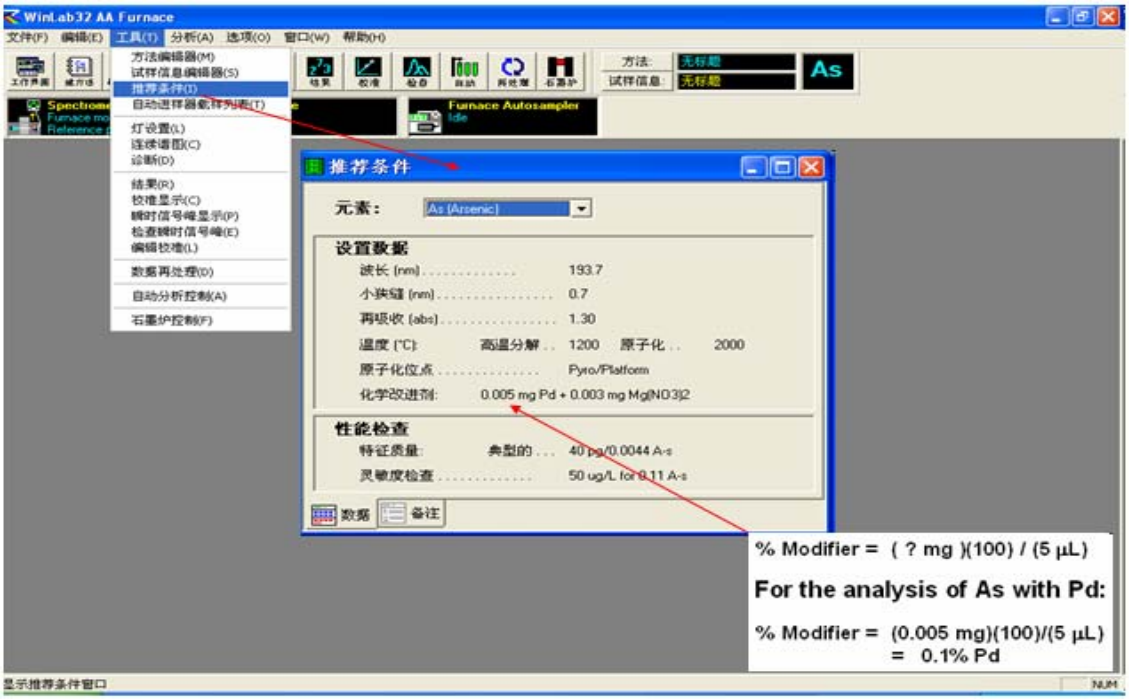
Automatic Recovery Check: 原油

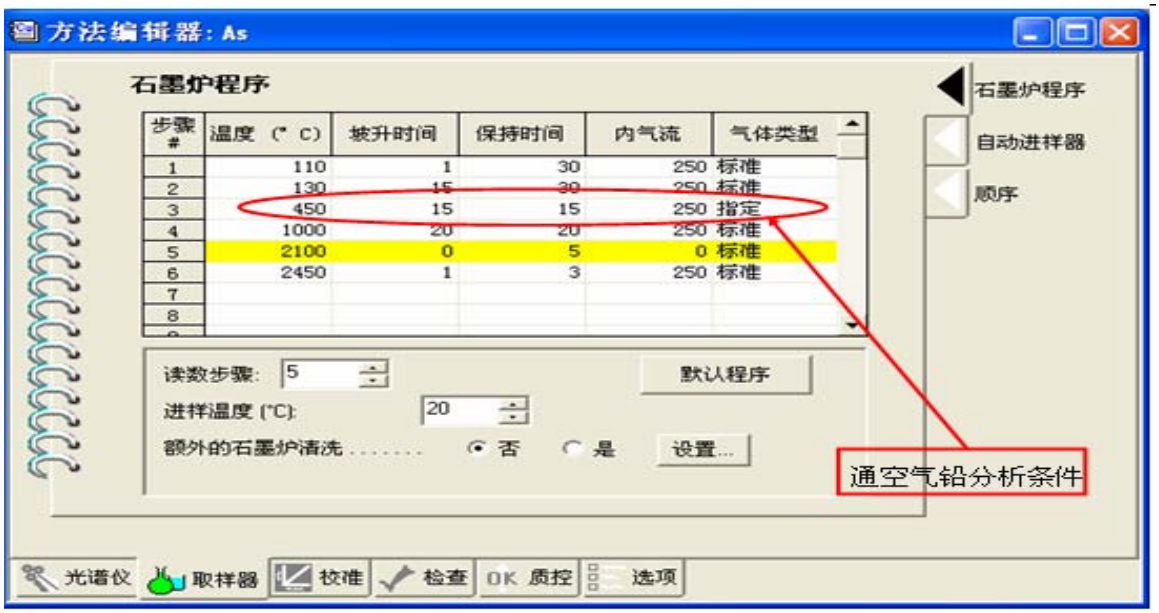
Analyte	Expected Conc.	Expected Increase	Measured Increase	Units	Recovery (%)	Limits (%) Lower/Upper
Pb 283.3	20.0	12.5	12.6	ug/g	101.0	90/110



2、油品中砷的分析条件和结果

2.1、砷分析条件

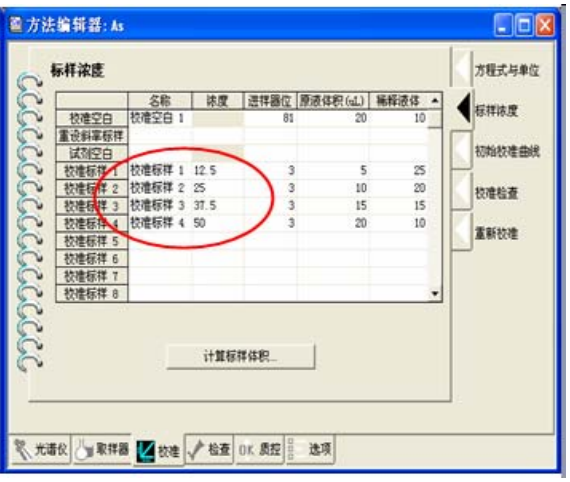


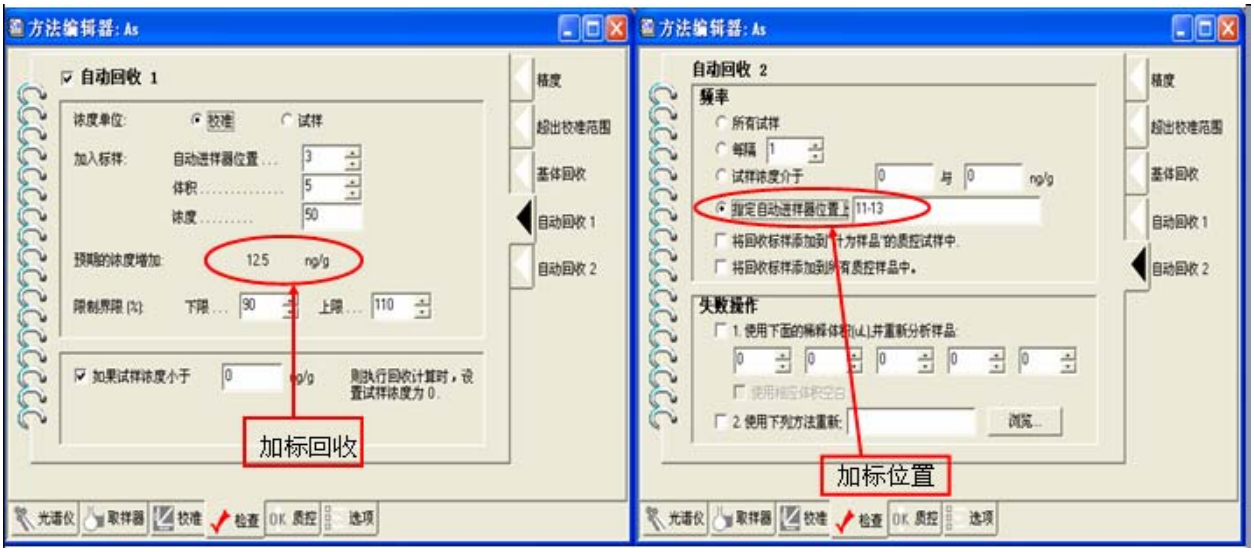

方法编辑器: As
石墨炉程序
步骤 # | 温度 (° C) | 坡升时间 | 保持时间 | 内气流 | 气体类型
1 | 110 | 1 | 30 | 250 | 标准
2 | 130 | 15 | 30 | 250 | 标准
3 | 450 | 15 | 15 | 250 | 指定
4 | 1000 | 20 | 20 | 250 | 标准
5 | 2100 | 0 | 5 | 0 | 标准
6 | 2450 | 1 | 3 | 250 | 标准
读数步骤: 5
默认程序
进样温度 (°C): 20
额外的石墨炉清洗 . . . . . 否 是 设置...
石墨炉程序
自动进样器
顺序
通空气铅分析条件
光谱仪 取样器 校准 检查 OK 质控 选项

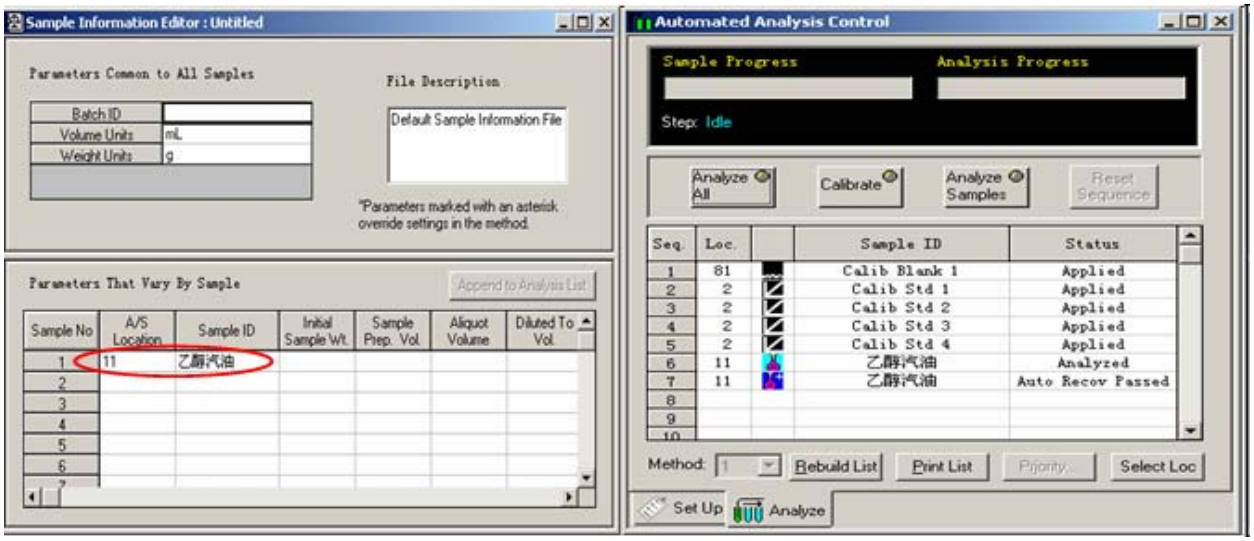

方法编辑器: As
石墨炉自动进样器
试样
体积 (uL) 20
稀释液体积 (uL) . . 10
稀释液位置 81
基体改进剂
#1 体积 (uL) . . 5
位置 . . . 1
添加到校准空白与标样
添加到试剂空白与试样
#2 体积 (uL) . . 0
位置 . . . 1
添加到校准空白与标样
添加到试剂空白与试样
基体改进剂
0.1%Pd+0.06%Mg(NO3)2
石墨炉程序
自动进样器
顺序
光谱仪 取样器 校准 检查 OK 质控 选项

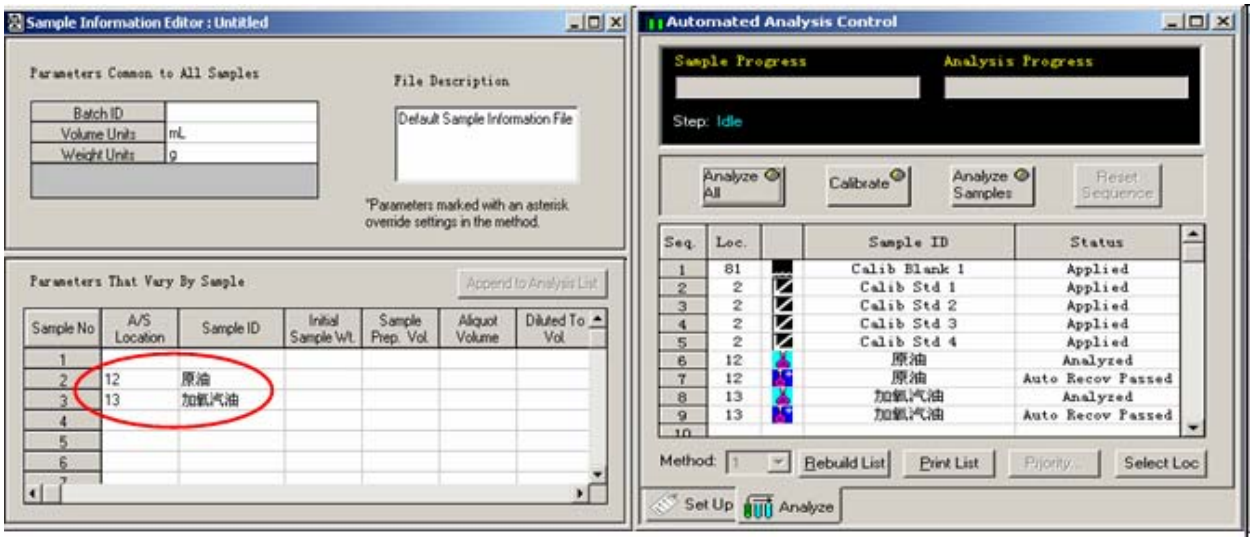

方法编辑器: As
自动进样器与石墨炉序列
步骤 | 操作与参数
A | Pipet diluent + modifier1 + spike + sample/std
B | Run furnace steps 1 to end
C
D
E
F
G
H
修改序列 简单... 自定义...
吸液速度 (%): 取样: 80 进样: 100
取样速度
石墨炉程序
自动进样器
顺序
光谱仪 取样器 校准 检查 OK 质控 选项


方法编辑器: As
校准方程式
方程式 . . . . . 非线性通过零点
最大小数点位数: 3
最大有效数字位数: 3
加入法 . . . . . 系统准备 分析准备
单位
校准 . . . . . ng/g
试样 . . . . . ng/g
方程式与单位
标样浓度
初始校准曲线
校准检查
重新校准
光谱仪 取样器 校准 检查 OK 质控 选项


方法编辑器: As
标样浓度
名称 | 浓度 | 进样器位 | 原液体积 (uL) | 稀释液体
校准空白 | 校准空白 1 | | 81 | 20 | 10
重设斜率标样
试剂空白
校准标样 1 | 校准标样 1 | 12.5 | 3 | 5 | 25
校准标样 2 | 校准标样 2 | 25 | 3 | 10 | 20
校准标样 3 | 校准标样 3 | 37.5 | 3 | 15 | 15
校准标样 4 | 校准标样 4 | 50 | 3 | 20 | 10
校准标样 5
校准标样 6
校准标样 7
校准标样 8
计算标样体积...
方程式与单位
标样浓度
初始校准曲线
校准检查
重新校准
光谱仪 取样器 校准 检查 OK 质控 选项


方法编辑器: As
自动回收 1
浓度单位:
校准
试样
加入标样:
自动进样器位置...
体积.....
浓度.....
预期的浓度增加:
125
ng/g
限制界限 (%):
下限...
90
上限...
110
如果试样浓度小于
0
ng/g
则执行回收计算时，设置试样浓度为 0.
精度
超出校准范围
基体回收
自动回收 1
自动回收 2
加标回收
光谱仪
取样器
校准
检查
OK 质控
选项
方法编辑器: As
自动回收 2
频率
所有试样
每隔
1
试样浓度介于
0
与
0
ng/g
指定自动进样器位置上
11-13
将回收标样添加到计为样品'的质控试样中.
将回收标样添加到所有质控样品中.
失败操作
1. 使用下面的稀释体积(uL)并重新分析样品:
浏览...
2. 使用下列方法重新:
加标位置
精度
超出校准范围
基体回收
自动回收 1
自动回收 2
光谱仪
取样器
校准
检查
OK 质控
选项

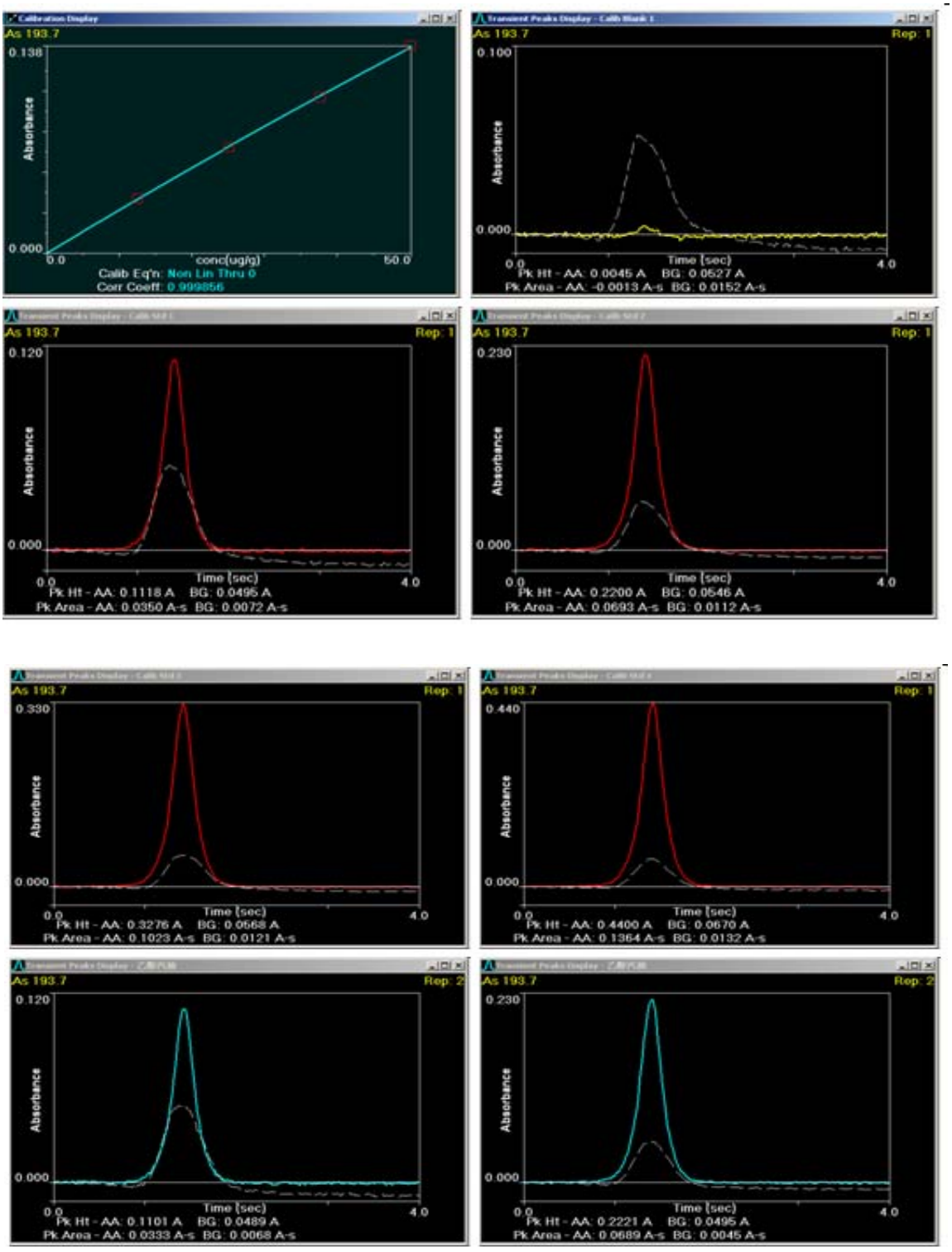

Sample Information Editor : Untitled
Parameters Common to All Samples
Batch ID
Volume Units
mL
Weight Units
g
File Description
Default Sample Information File
*Parameters marked with an asterisk override settings in the method.
Parameters That Vary By Sample
Append to Analysis List
Sample No
A/S Location
Sample ID
Initial Sample Wt.
Sample Prep. Vol.
Aliquot Volume
Diluted To Vol.
1
11
乙醇汽油
Automated Analysis Control
Sample Progress
Analysis Progress
Step: Idle
Analyze All
Calibrate
Analyze Samples
Reset Sequence
Seq.
Loc.
Sample ID
Status
1 81 Calib Blank 1 Applied
2 2 Calib Std 1 Applied
3 2 Calib Std 2 Applied
4 2 Calib Std 3 Applied
5 2 Calib Std 4 Applied
6 11 乙醇汽油 Analyzed
7 11 乙醇汽油 Auto Recov Passed
Method:
Rebuild List
Print List
Priority...
Select Loc
Set Up
Analyze


Sample Information Editor : Untitled
Parameters Common to All Samples
Batch ID
Volume Units
mL
Weight Units
g
File Description
Default Sample Information File
*Parameters marked with an asterisk override settings in the method.
Parameters That Vary By Sample
Append to Analysis List
Sample No
A/S Location
Sample ID
Initial Sample Wt.
Sample Prep. Vol.
Aliquot Volume
Diluted To Vol.
2 12 原油
3 13 加氧汽油
Automated Analysis Control
Sample Progress
Analysis Progress
Step: Idle
Analyze All
Calibrate
Analyze Samples
Reset Sequence
Seq.
Loc.
Sample ID
Status
1 81 Calib Blank 1 Applied
2 2 Calib Std 1 Applied
3 2 Calib Std 2 Applied
4 2 Calib Std 3 Applied
5 2 Calib Std 4 Applied
6 12 原油 Analyzed
7 12 原油 Auto Recov Passed
8 13 加氧汽油 Analyzed
9 13 加氧汽油 Auto Recov Passed
Method:
Rebuild List
Print List
Priority...
Select Loc
Set Up
Analyze

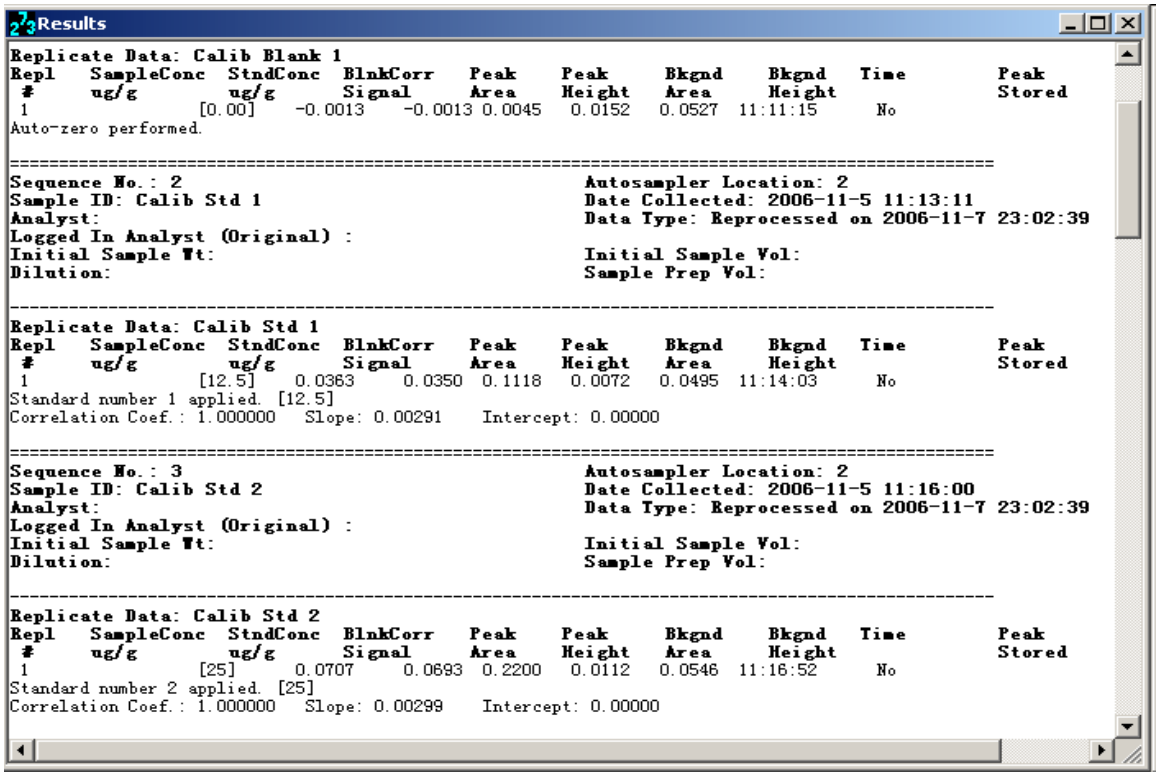
2.2 砷结果

2.2.1 乙醇汽油砷图谱和结果

2.2.1.1 乙醇汽油砷图谱



2.2.1.2 乙醇汽油砷结果



Results

Replicate Data: Calib Blank 1

Repl #	SampleConc ug/g	StndConc ug/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	[0.00]	-0.0013	-0.0013	0.0045	0.0152	0.0527	11:11:15		No

Auto-zero performed.

Sequence No.: 2 — Autosampler Location: 2
Sample ID: Calib Std 1 — Date Collected: 2006-11-5 11:13:11
Analyst: — Data Type: Reprocessed on 2006-11-7 23:02:39
Logged In Analyst (Original) :
Initial Sample Wt: — Initial Sample Vol:
Dilution: — Sample Prep Vol:

Replicate Data: Calib Std 1

Repl #	SampleConc ug/g	StndConc ug/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	[12.5]	0.0363	0.0350	0.1118	0.0072	0.0495	11:14:03		No

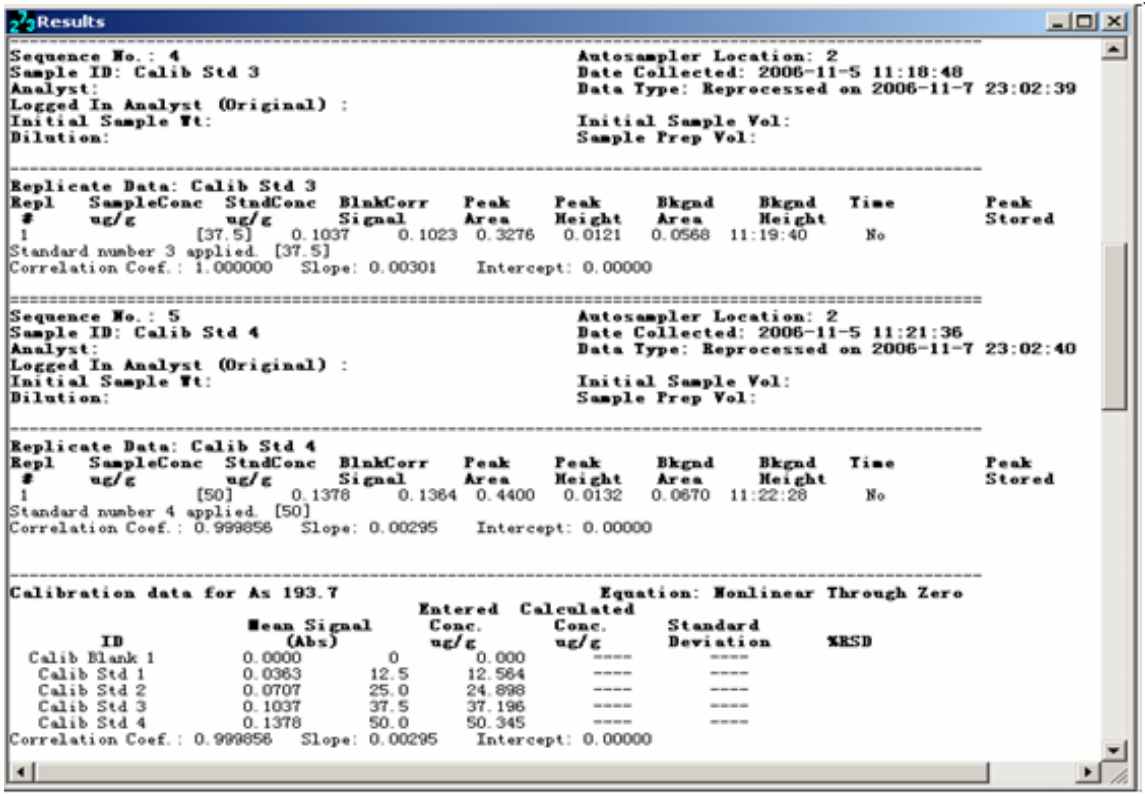
Standard number 1 applied. [12.5]
Correlation Coef.: 1.000000 Slope: 0.00291 Intercept: 0.00000

Sequence No.: 3 — Autosampler Location: 2
Sample ID: Calib Std 2 — Date Collected: 2006-11-5 11:16:00
Analyst: — Data Type: Reprocessed on 2006-11-7 23:02:39
Logged In Analyst (Original) :
Initial Sample Wt: — Initial Sample Vol:
Dilution: — Sample Prep Vol:

Replicate Data: Calib Std 2

Repl #	SampleConc ug/g	StndConc ug/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	[25]	0.0707	0.0693	0.2200	0.0112	0.0546	11:16:52		No

Standard number 2 applied. [25]
Correlation Coef.: 1.000000 Slope: 0.00299 Intercept: 0.00000



Results

Sequence No.: 4 — Autosampler Location: 2
Sample ID: Calib Std 3 — Date Collected: 2006-11-5 11:18:48
Analyst: — Data Type: Reprocessed on 2006-11-7 23:02:39
Logged In Analyst (Original) :
Initial Sample Wt: — Initial Sample Vol:
Dilution: — Sample Prep Vol:

Replicate Data: Calib Std 3

Repl #	SampleConc ug/g	StndConc ug/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	[37.5]	0.1037	0.1023	0.3276	0.0121	0.0568	11:19:40		No

Standard number 3 applied. [37.5]
Correlation Coef.: 1.000000 Slope: 0.00301 Intercept: 0.00000

Sequence No.: 5 — Autosampler Location: 2
Sample ID: Calib Std 4 — Date Collected: 2006-11-5 11:21:36
Analyst: — Data Type: Reprocessed on 2006-11-7 23:02:40
Logged In Analyst (Original) :
Initial Sample Wt: — Initial Sample Vol:
Dilution: — Sample Prep Vol:

Replicate Data: Calib Std 4

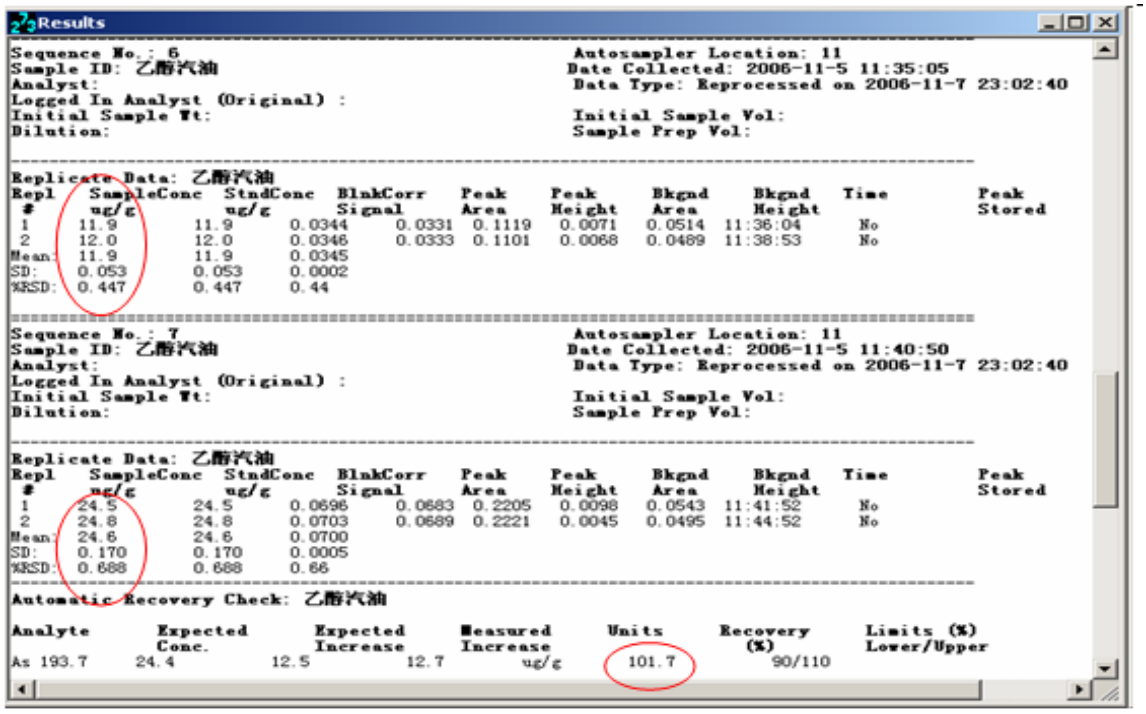
Repl #	SampleConc ug/g	StndConc ug/g	BlnkCorr Signal	Peak Area	Peak Height	Bkgnd Area	Bkgnd Height	Time	Peak Stored
1	[50]	0.1378	0.1364	0.4400	0.0132	0.0670	11:22:28		No

Standard number 4 applied. [50]
Correlation Coef.: 0.999856 Slope: 0.00295 Intercept: 0.00000

Calibration data for As 193.7 — Equation: Nonlinear Through Zero

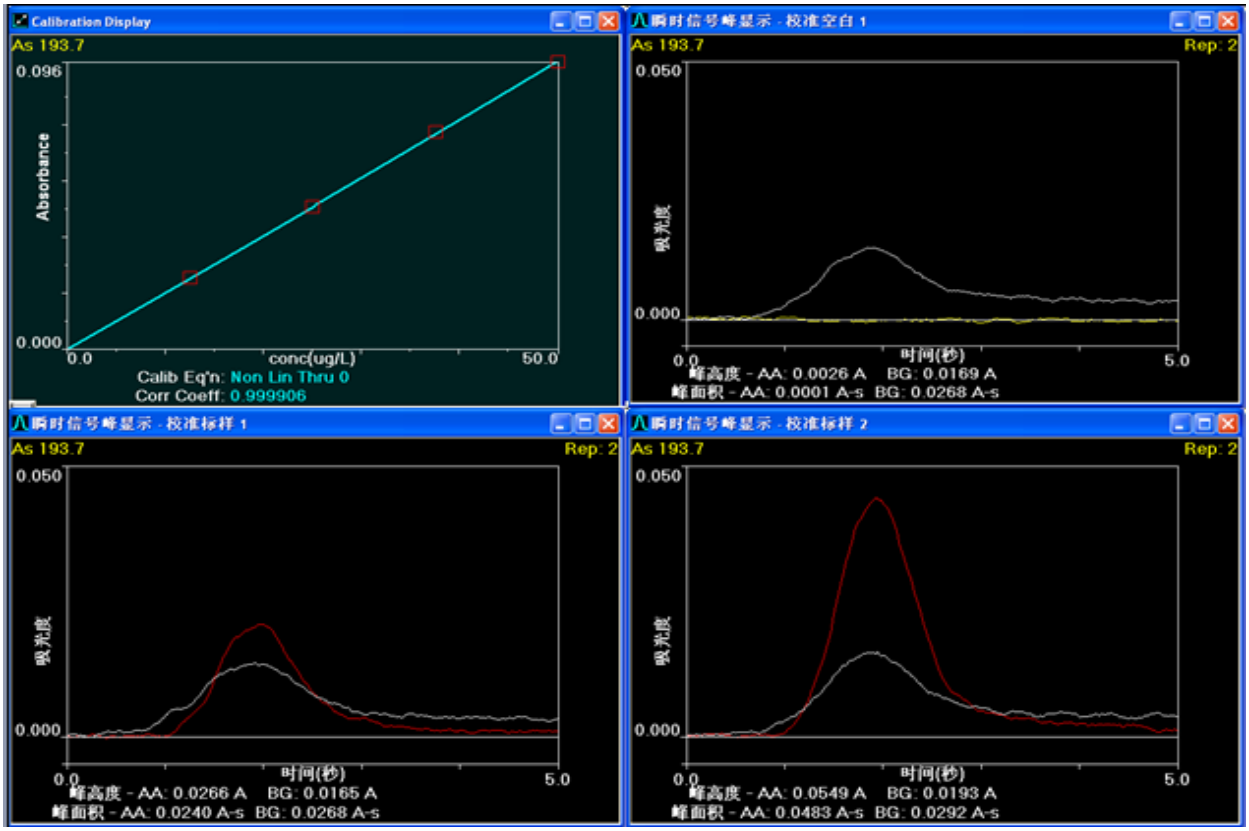
ID	Mean Signal (Abs)	Entered Conc. ug/g	Calculated Conc. ug/g	Standard Deviation	%RSD
Calib Blank 1	0.0000	0	0.000	----	----
Calib Std 1	0.0363	12.5	12.564	----	----
Calib Std 2	0.0707	25.0	24.898	----	----
Calib Std 3	0.1037	37.5	37.196	----	----
Calib Std 4	0.1378	50.0	50.345	----	----

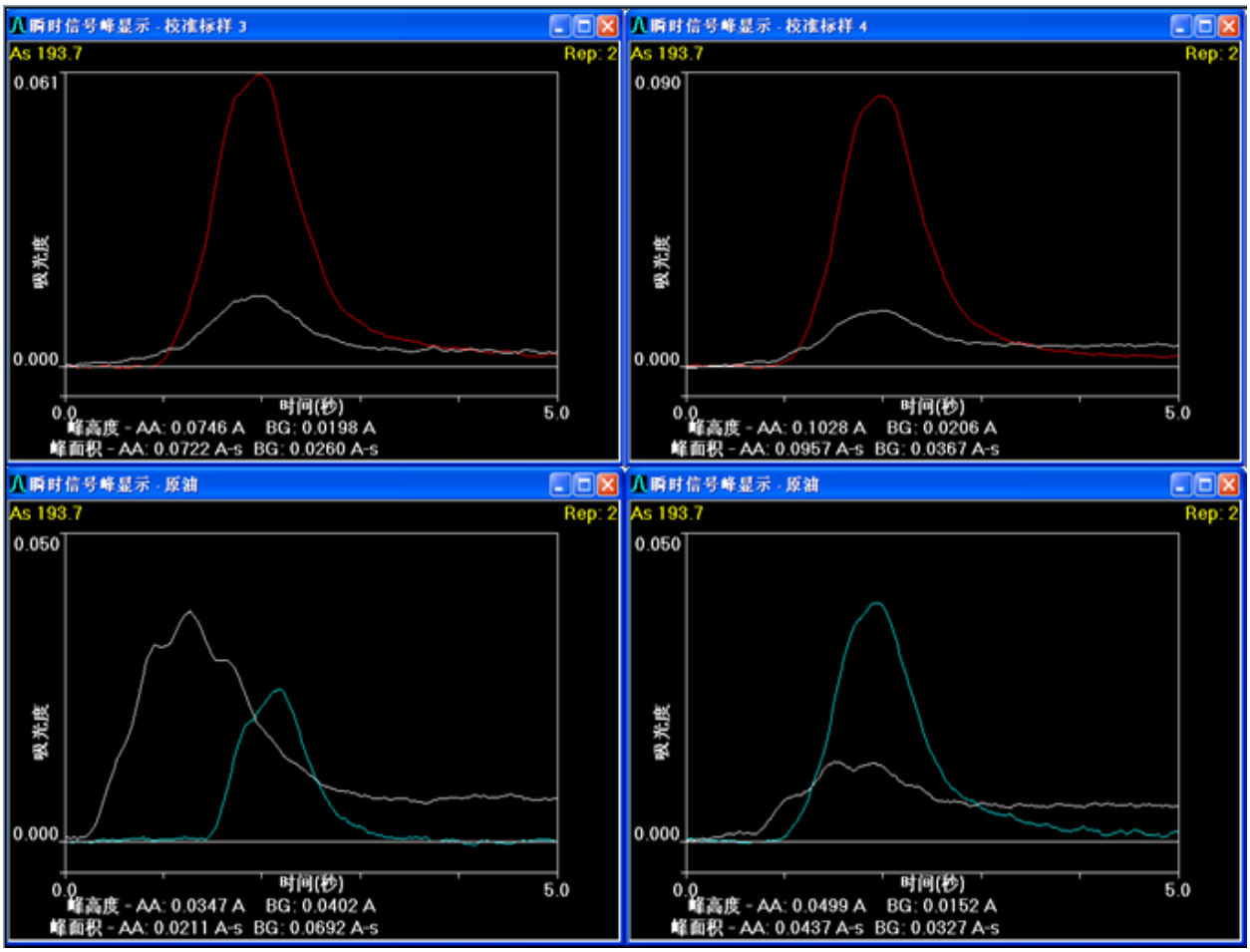
Correlation Coef.: 0.999856 Slope: 0.00295 Intercept: 0.00000

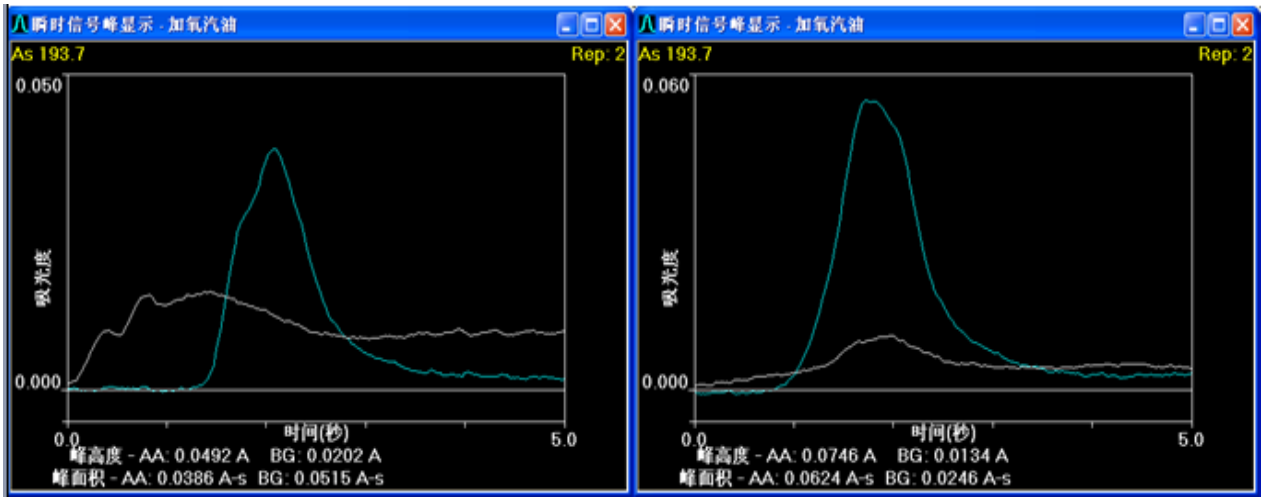


2.2.2 原油砷图谱和结果

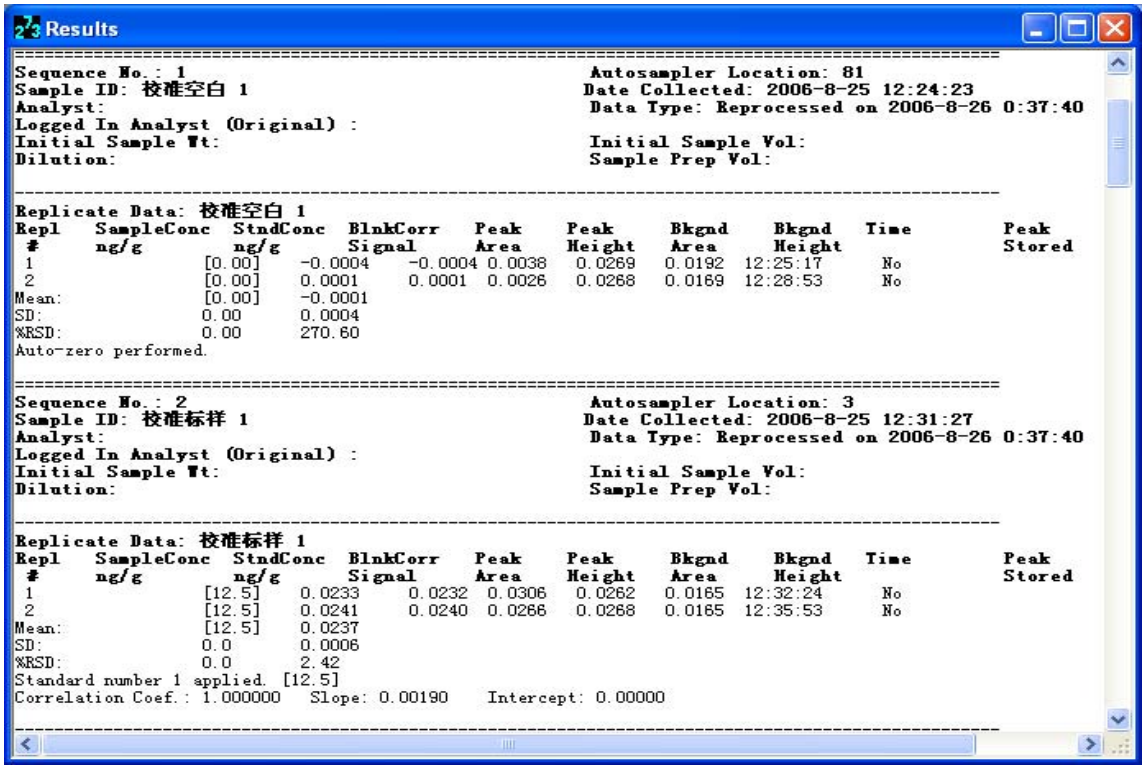
2.2.2.1 原油砷图谱




瞬时信号峰显示 - 校准标样 3
As 193.7
Rep: 2
0.061
吸光度
0.000
0.0
时间(秒)
5.0
峰高度 - AA: 0.0746 A BG: 0.0198 A
峰面积 - AA: 0.0722 A-s BG: 0.0260 A-s
瞬时信号峰显示 - 校准标样 4
As 193.7
Rep: 2
0.090
吸光度
0.000
0.0
时间(秒)
5.0
峰高度 - AA: 0.1028 A BG: 0.0206 A
峰面积 - AA: 0.0957 A-s BG: 0.0367 A-s
瞬时信号峰显示 - 原油
As 193.7
Rep: 2
0.050
吸光度
0.000
0.0
时间(秒)
5.0
峰高度 - AA: 0.0347 A BG: 0.0402 A
峰面积 - AA: 0.0211 A-s BG: 0.0692 A-s
瞬时信号峰显示 - 原油
As 193.7
Rep: 2
0.050
吸光度
0.000
0.0
时间(秒)
5.0
峰高度 - AA: 0.0499 A BG: 0.0152 A
峰面积 - AA: 0.0437 A-s BG: 0.0327 A-s

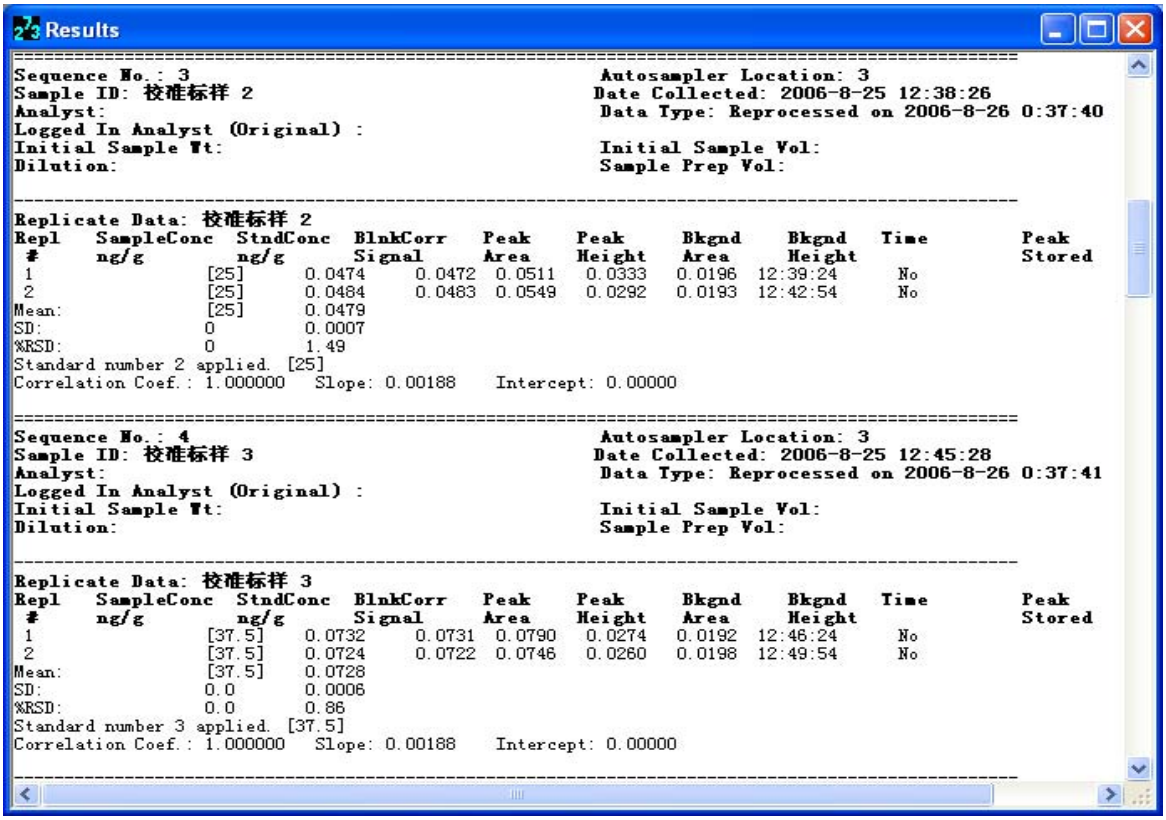

瞬时信号峰显示 - 加氧汽油
As 193.7
Rep: 2
0.050
吸光度
0.000
0.0
时间(秒)
5.0
峰高度 - AA: 0.0492 A BG: 0.0202 A
峰面积 - AA: 0.0386 A-s BG: 0.0515 A-s
瞬时信号峰显示 - 加氧汽油
As 193.7
Rep: 2
0.060
吸光度
0.000
0.0
时间(秒)
5.0
峰高度 - AA: 0.0746 A BG: 0.0134 A
峰面积 - AA: 0.0624 A-s BG: 0.0246 A-s

2.2.2.2 原油砷结果



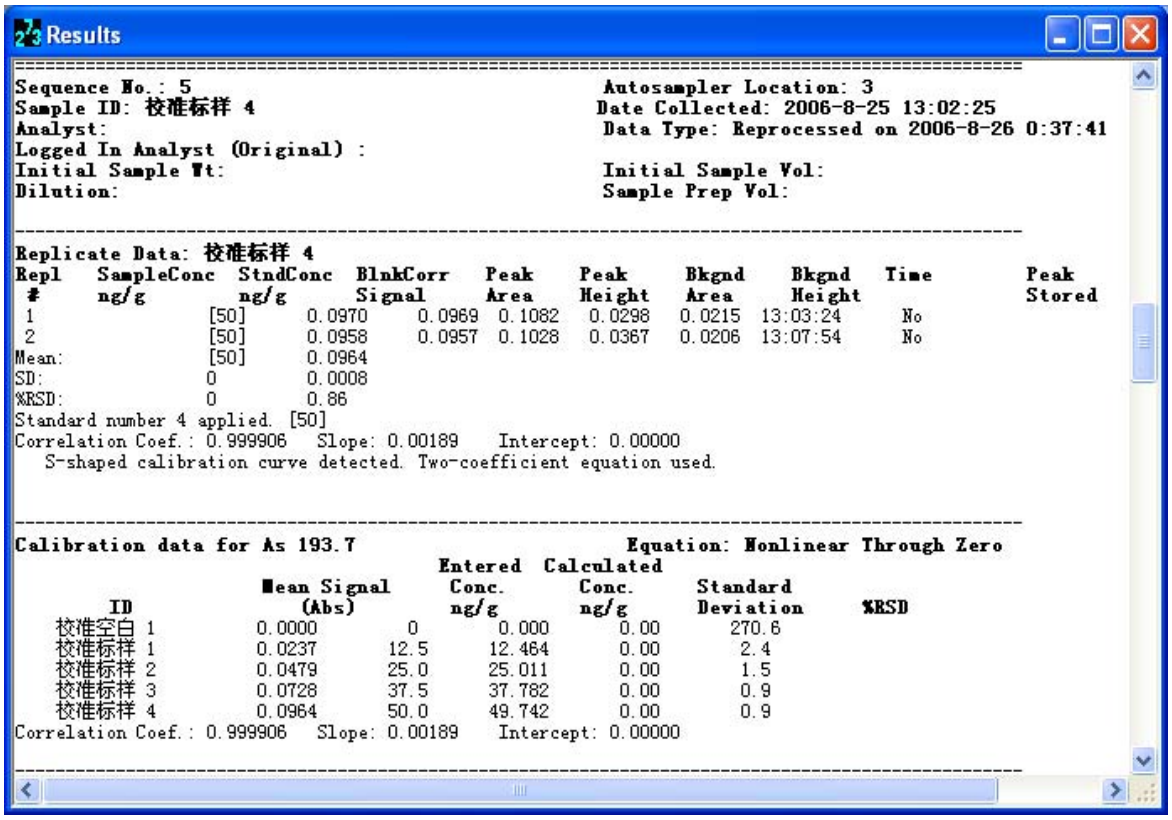
Results

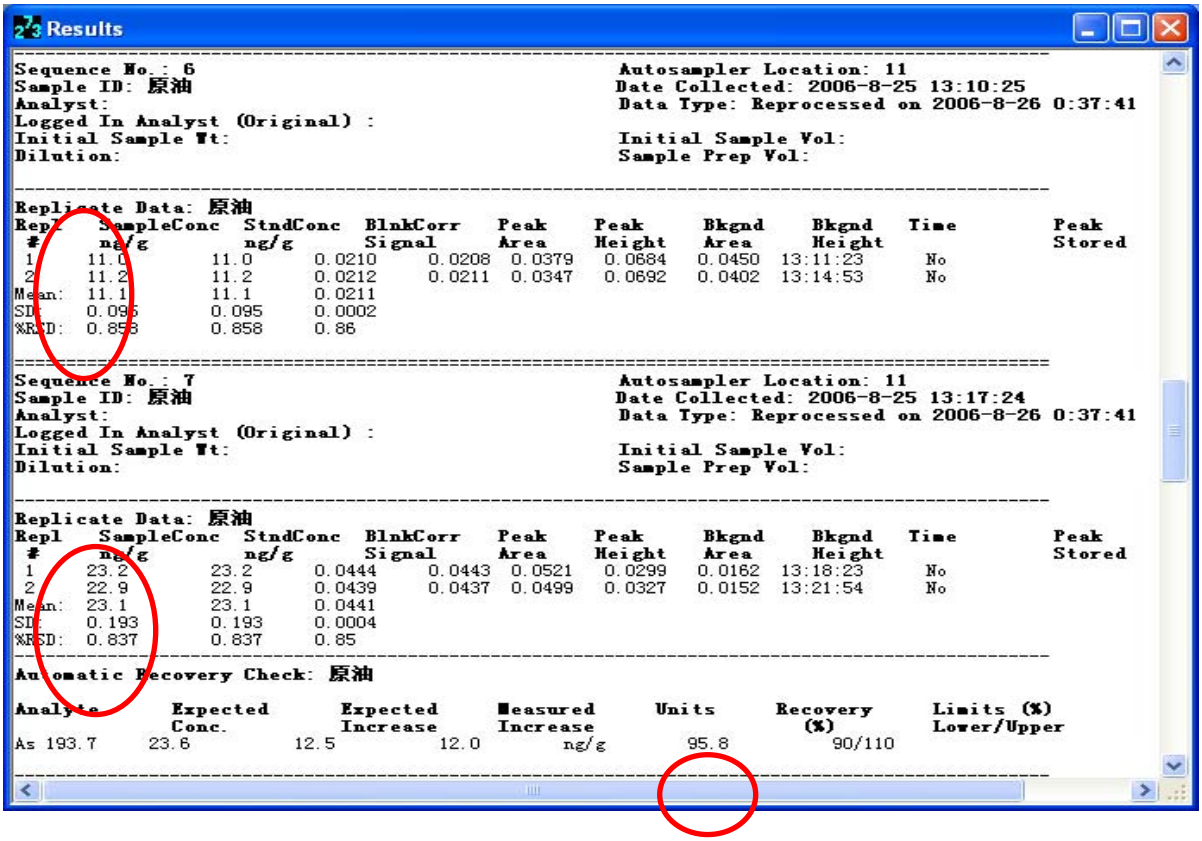
```
Sequence No.: 1                                   Autosampler Location: 81
Sample ID: 校准空白 1                              Date Collected: 2006-8-25 12:24:23
Analyst:                                          Data Type: Reprocessed on 2006-8-26 0:37:40
Logged In Analyst (Original) :
Initial Sample Wt:                                Initial Sample Vol:
Dilution:                                         Sample Prep Vol:

Replicate Data: 校准空白 1
Repl  SampleConc  StndConc  BlnkCorr   Peak     Peak     Bkgnd    Bkgnd   Time        Peak
 #      ng/g        ng/g      Signal    Area    Height    Area    Height               Stored
 1              [0.00]    -0.0004    -0.0004  0.0038   0.0269   0.0192  12:25:17      No
 2              [0.00]     0.0001     0.0001  0.0026   0.0268   0.0169  12:28:53      No
Mean:           [0.00]    -0.0001
SD:             0.00       0.0004
%RSD:           0.00      270.60
Auto-zero performed.

Sequence No.: 2                                   Autosampler Location: 3
Sample ID: 校准标样 1                              Date Collected: 2006-8-25 12:31:27
Analyst:                                          Data Type: Reprocessed on 2006-8-26 0:37:40
Logged In Analyst (Original) :
Initial Sample Wt:                                Initial Sample Vol:
Dilution:                                         Sample Prep Vol:

Replicate Data: 校准标样 1
Repl  SampleConc  StndConc  BlnkCorr   Peak     Peak     Bkgnd    Bkgnd   Time        Peak
 #      ng/g        ng/g      Signal    Area    Height    Area    Height               Stored
 1             [12.5]     0.0233     0.0232  0.0306   0.0262   0.0165  12:32:24      No
 2             [12.5]     0.0241     0.0240  0.0266   0.0268   0.0165  12:35:53      No
Mean:          [12.5]     0.0237
SD:            0.0        0.0006
%RSD:          0.0        2.42
Standard number 1 applied. [12.5]
Correlation Coef.: 1.000000   Slope: 0.00190    Intercept: 0.00000
```

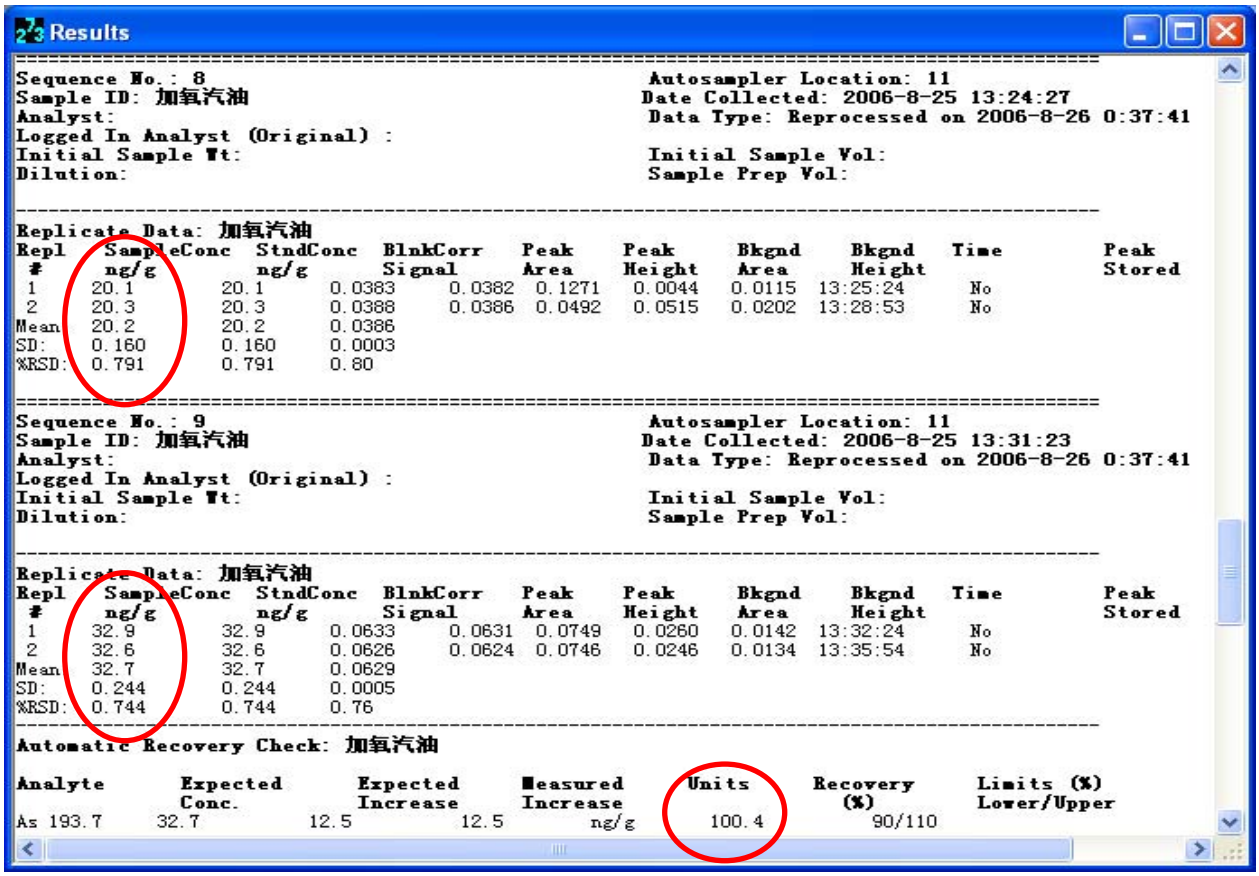


Results

```
Sequence No.: 3                                   Autosampler Location: 3
Sample ID: 校准标样 2                              Date Collected: 2006-8-25 12:38:26
Analyst:                                          Data Type: Reprocessed on 2006-8-26 0:37:40
Logged In Analyst (Original) :
Initial Sample Wt:                                Initial Sample Vol:
Dilution:                                         Sample Prep Vol:

Replicate Data: 校准标样 2
Repl  SampleConc  StndConc  BlnkCorr   Peak     Peak     Bkgnd    Bkgnd   Time        Peak
 #      ng/g        ng/g      Signal    Area    Height    Area    Height               Stored
 1              [25]      0.0474     0.0472  0.0511   0.0333   0.0196  12:39:24      No
 2              [25]      0.0484     0.0483  0.0549   0.0292   0.0193  12:42:54      No
Mean:           [25]      0.0479
SD:             0         0.0007
%RSD:           0         1.49
Standard number 2 applied. [25]
Correlation Coef.: 1.000000   Slope: 0.00188    Intercept: 0.00000

Sequence No.: 4                                   Autosampler Location: 3
Sample ID: 校准标样 3                              Date Collected: 2006-8-25 12:45:28
Analyst:                                          Data Type: Reprocessed on 2006-8-26 0:37:41
Logged In Analyst (Original) :
Initial Sample Wt:                                Initial Sample Vol:
Dilution:                                         Sample Prep Vol:

Replicate Data: 校准标样 3
Repl  SampleConc  StndConc  BlnkCorr   Peak     Peak     Bkgnd    Bkgnd   Time        Peak
 #      ng/g        ng/g      Signal    Area    Height    Area    Height               Stored
 1             [37.5]     0.0732     0.0731  0.0790   0.0274   0.0192  12:46:24      No
 2             [37.5]     0.0724     0.0722  0.0746   0.0260   0.0198  12:49:54      No
Mean:          [37.5]     0.0728
SD:            0.0        0.0006
%RSD:          0.0        0.86
Standard number 3 applied. [37.5]
Correlation Coef.: 1.000000   Slope: 0.00188    Intercept: 0.00000
```


Results
Sequence No.: 5
Sample ID: 校准标样 4
Analyst:
Logged In Analyst (Original) :
Initial Sample Wt:
Dilution:
Autosampler Location: 3
Date Collected: 2006-8-25 13:02:25
Data Type: Reprocessed on 2006-8-26 0:37:41
Initial Sample Vol:
Sample Prep Vol:
Replicate Data: 校准标样 4
Repl # | SampleConc ng/g | StndConc ng/g | BlnkCorr Signal | Peak Area | Peak Height | Bkgnd Area | Bkgnd Height | Time | Peak Stored
1 [50] 0.0970 0.0969 0.1082 0.0298 0.0215 13:03:24 No
2 [50] 0.0958 0.0957 0.1028 0.0367 0.0206 13:07:54 No
Mean: [50] 0.0964
SD: 0 0.0008
%RSD: 0 0.86
Standard number 4 applied. [50]
Correlation Coef.: 0.999906 Slope: 0.00189 Intercept: 0.00000
S-shaped calibration curve detected. Two-coefficient equation used.
Calibration data for As 193.7
Equation: Nonlinear Through Zero
ID | Mean Signal (Abs) | Entered Conc. ng/g | Calculated Conc. ng/g | Standard Deviation | %RSD
校准空白 1 0.0000 0 0.000 0.00 270.6
校准标样 1 0.0237 12.5 12.464 0.00 2.4
校准标样 2 0.0479 25.0 25.011 0.00 1.5
校准标样 3 0.0728 37.5 37.782 0.00 0.9
校准标样 4 0.0964 50.0 49.742 0.00 0.9
Correlation Coef.: 0.999906 Slope: 0.00189 Intercept: 0.00000


Results
Sequence No.: 6
Sample ID: 原油
Analyst:
Logged In Analyst (Original) :
Initial Sample Wt:
Dilution:
Autosampler Location: 11
Date Collected: 2006-8-25 13:10:25
Data Type: Reprocessed on 2006-8-26 0:37:41
Initial Sample Vol:
Sample Prep Vol:
Replicate Data: 原油
Repl # | SampleConc ng/g | StndConc ng/g | BlnkCorr Signal | Peak Area | Peak Height | Bkgnd Area | Bkgnd Height | Time | Peak Stored
1 11.0 11.0 0.0210 0.0208 0.0379 0.0684 0.0450 13:11:23 No
2 11.2 11.2 0.0212 0.0211 0.0347 0.0692 0.0402 13:14:53 No
Mean: 11.1 11.1 0.0211
SD: 0.095 0.095 0.0002
%RSD: 0.858 0.858 0.86
Sequence No.: 7
Sample ID: 原油
Analyst:
Logged In Analyst (Original) :
Initial Sample Wt:
Dilution:
Autosampler Location: 11
Date Collected: 2006-8-25 13:17:24
Data Type: Reprocessed on 2006-8-26 0:37:41
Initial Sample Vol:
Sample Prep Vol:
Replicate Data: 原油
Repl # | SampleConc ng/g | StndConc ng/g | BlnkCorr Signal | Peak Area | Peak Height | Bkgnd Area | Bkgnd Height | Time | Peak Stored
1 23.2 23.2 0.0444 0.0443 0.0521 0.0299 0.0162 13:18:23 No
2 22.9 22.9 0.0439 0.0437 0.0499 0.0327 0.0152 13:21:54 No
Mean: 23.1 23.1 0.0441
SD: 0.193 0.193 0.0004
%RSD: 0.837 0.837 0.85
Automatic Recovery Check: 原油
Analyte | Expected Conc. | Expected Increase | Measured Increase | Units | Recovery (%) | Limits (%) Lower/Upper
As 193.7 23.6 12.5 12.0 ng/g 95.8 90/110



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