# 高性能ICPOES及其应用

- 高光晔
- 2018-9-21

ICPOES的发展过程

- 1961年Reed设计了在大气压下稳定的高频震荡放电（ICP）
- 1964年S.Greefield发表第一篇ICPOES应用报告
- 1975年第一台商用ICPOES（垂直火炬多通道）
- 1977年第一台单道扫描性ICPOES
- 1982年单道+多通道ICPOES
- 1990年第一台水平火炬ICPOES
- 1991年第一台全谱直读型ICPOES
- 1994年第一台双向观测ICPOES
- 2011年第一台平板等离子体ICPOES
- 2013年第一台垂直火炬双向观测ICPOES

LPG 21

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ICPOES硬件的变迁

功能模块	发展变化
ICP的布局及观测方式	垂直火炬径向观测→水平火炬轴向观测→水平火炬双向观测→垂直火炬双向观测
射频发生器	电子管放大型→固态放大型→变频固态放大型
分光系统	凹面光栅多道同时型→平面光栅单道扫描型→中阶梯光栅两维色散全谱直读型
检测器	光电倍增管（PMT）→固态检测器（CCD、CID）
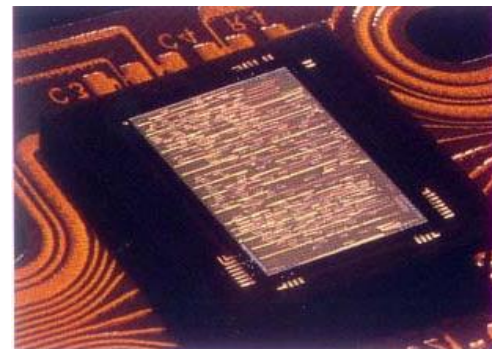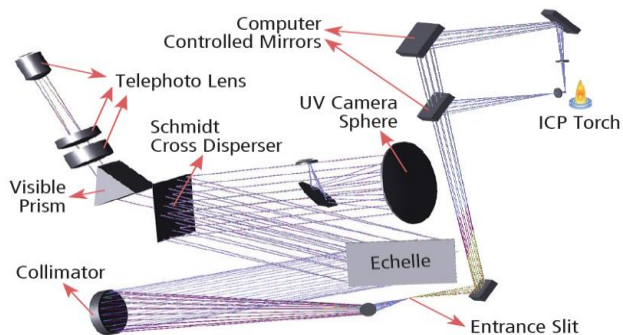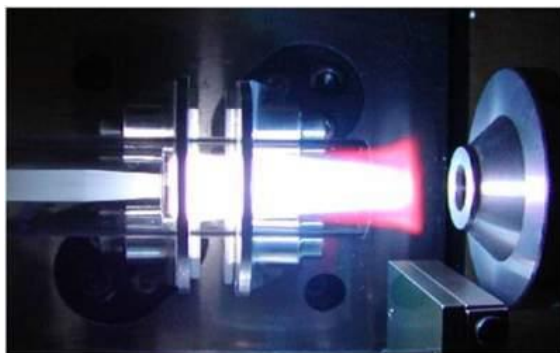
表1 各元素 ICP-AES 分析法的检测限(L. D. , μg/L)

Table 1 The detection limit for elements by ICP-AES(L. D. , μg/L)

分析元素	Ag	Al	As	Au	B	Ba	Be	Bi	Ca	Cd	Ce	Co	Cr	Cu	Dy	Er	Eu
L. D. 80 [1]	6.6	22	50	16	4.5	1.2	0.25	21	0.18	2.4	50	5.0	4.0	2.3	10	9.4	2.5
L. D. 99 [2]	0.2	0.2	0.9	0.6	0.3	0.04	0.05	2.6	0.02	0.09	2.0	0.2	0.2	0.2	1.0	0.7	0.2
分析元素	Fe	Ga	Gd	Ge	Hf	Hg	Ho	In	Ir	K	La	Li	Lu	Mg	Mn	Mo	Na
L. D. - 80 [1]	1.7	21	14	17	11	25	5.4	59	25	60	9.4	1.8	0.94	0.14	1.3	7.4	29
L. D. - 99 [2]	0.2	4	0.9	6.0	3.3	0.5	0.4	9	5	0.2	1	0.2	0.2	0.01	0.04	0.2	0.5
分析元素	Nb	Nd	Ni	Os	P	Pb	Pd	Pr	Pt	Re	Rh	Ru	S	Sb	Sc	Se	Si
L. D. - 80 [1]	39	47	9.4	0.34	73	40	40	36	28	57	40	28	-	17	-	70	9
L. D. - 99 [2]	5	2	0.3	0.13	1.5	1.5	3	2	4.7	3.3	5	6	9	2.0	0.09	1.5	1.5
分析元素	Sm	Sn	Sr	Ta	Tb	Te	Th	Ti	Tl	Tm	U	V	W	Y	Yb	Zn	Zr
L. D. - 80 [1]	40	25	0.4	24	22	39	61	3.5	39	4.9	240	4.6	28	3.2	1.7	1.7	6.6
L. D. - 99 [2]	2	1.3	0.01	5.3	2	10	5.4	0.05	1.0	0.6	15	0.2	2.0	0.3	0.3	0.2	0.3

[1] 为 20 世纪 80 年代文献上所发表的数据。引自文献[5];

[2] 为 20 世纪 90 年代末商品仪器的最好水平。引自各仪器制造公司所提供的技术资料。
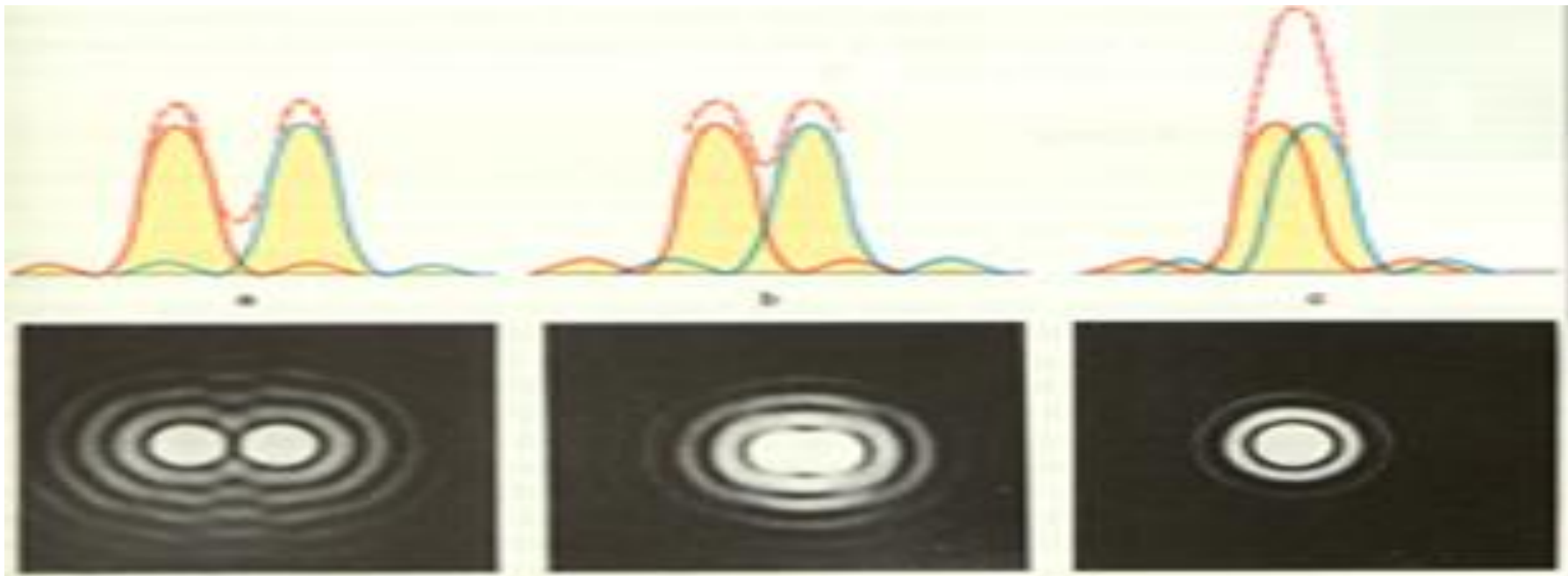
ICPOES性能的改进之二（光学分辨率）

0.006nm的光学分辨率可区分相差0.015nm的干扰线。如P213.617nm和Cu213.598nm

0.005-0.006nm   **0.007-0.008nm**.   **0.009-0.010nm**

ICPOES性能的改进之二（光学分辨率）

- 计算举例：**ICP5110**
- 光栅刻线94.74条/mm，计算得d=1mm/94.74=0.01056mm
- 闪耀角44.77°，计算得cos(β)=0.714
- 当λ=200nm时，光学级数74级，
- 根据光栅方程**m*λ=d*（sinα+sinβ ），**光栅的角色散率公式**dβ/dλ=m/(d*cosβ)**
- 角色散率=m/(d*cosβ)=74/(0.01056*0.714)=9814.5弧度/mm=0.0098弧度/nm
- 焦距为400毫米、像素宽度0.0125mm，
- 线色散率倒数=1/(角色散率*焦距)=1/(0.00098*400)=0.255nm/mm
- 仪器在200nm像素分辨率为：
- 像素分辨率=线色散率倒数*像素宽度=0.255*0.0125=**0.0032nm**

- **ICP5110在200nm处光学分辨率=2*像素分辨率=0.0032*2=**0.0064nm

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ICPOES的性能改进之三（稳定性）

（1）高频发生器的改进：由于 ICP 的电子密度和激发温度随频率的增加而减低，而光源的背景强度（Ar 的连续光谱）则与频率的平方成反比，随频率的提高要降低得多[6]。经比较[7]发现，采用 40.68MHz 的高频发生器，有较高的信背比（S/B），与 27.12MHz 发生器相比信背比有显著提高，检出限改善可达 4 倍。为了提高高频发生器的稳定性，采用具有很高的频率稳定性（优于 1 KHz）和输出功率稳定性（≤0.1 %）的固态发生器，等离子体阻抗可自动调节补偿，并由计算机控制工作参数、设定点火程序，可自动点火。因此，新型的商品仪器均已使用 40.68MHz 固态发生器，结合对样品的引入系统采取恒温，提高进样的稳定性，加上光学系统的恒温，热稳定性高（±0.1℃），使仪器预热时间大为缩短，开机 10min 即可达到稳定，使仪器的短时间稳定性可提高到 0.3 %RSD，长时间稳定性达 <1 %RSD。大大提高了 ICP-AES 法的分析精度和准确性。

Meinhard Type K1 and standard baffled cyclonic spray chamber
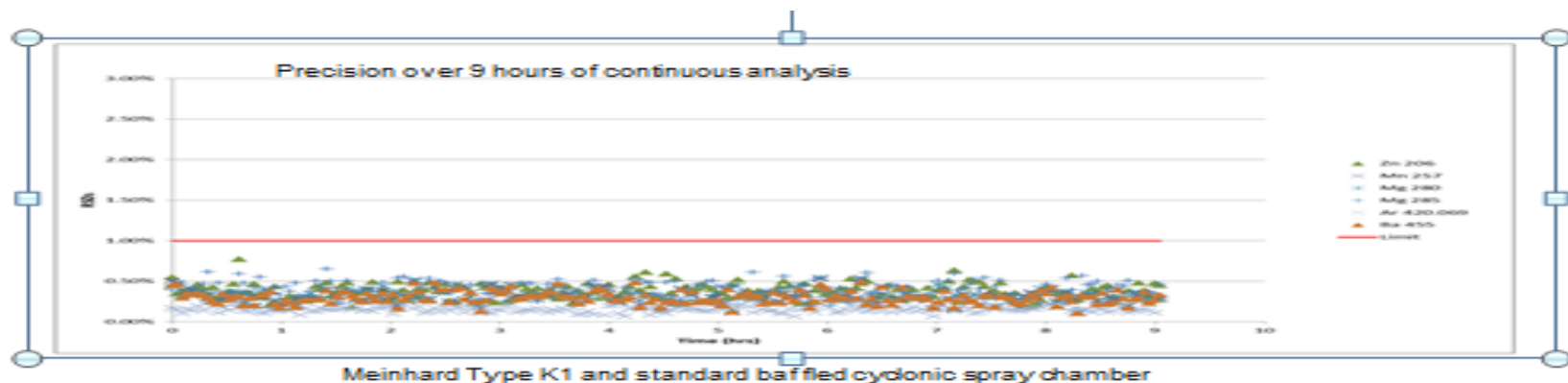
9-hour stability run results in <1% RSD

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主流（好的）ICPOES的基本要求

➤ 高灵敏度（检出限0.1-1ppb）
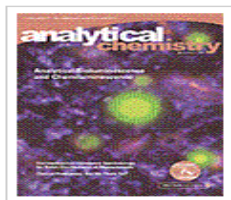➤ 高分辨率（200nm处优于0.007nm）— 好的光谱
➤ 高稳定性（短期Rsd%优于0.5%）

耐各种复杂基体样品 ⟶ 好的ICP

好的ICP光谱

实际样品测定对ICPOES的要求之一（氧化硼中Be测定）

期刊文献 Certification of Beryllium Mass Fraction in SRM 1877 Beryllium Oxide Powder Using High-Performance Inductively Coupled Plasma Optical Emission Spectrometry with Exact Matching

作者： Winchester, Michael R. mrw@nist.gov Turk, Gregory C. Butler, Therese A. Oatts, Thomas J. Coleman, Charles Nadratowski, Donald Sud, Ritu Hoover, Mark D. Stefaniak, Aleksandr B.

High-performance inductively coupled plasma optical emission spectrometry (HP-ICP-OES) was used to certify the Be mass fraction in National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 1877 Beryllium Oxide Powder. The certified value and expanded uncertainty expressed at a 95% confidence level is **(0.3576 ± 0.0024) g/g**.

- 二倍标准偏差0.6%，ICPOES短期RSD%必须低于0.3%

实际样品测定对ICPOES的要求之二（贵金属定值）

- **The Customer Request**
  - **Demonstrate that the Optima is capable of analyzing high accurate and precise determinations of PMGs**
- **The Elements**
  - **Au, Pt, Pd, Rh, Ir, Ag, Ru**
  - **e.g. ISO 11494 & 11495**
- **The Problem**
  - **Analytical Precision Relatively Poor : 0.20 - 0.40%**

$$\overline{m}_{\text{Pt,fin}} = \frac{1}{5}\left(\sum_{n=1}^{5} m_{\text{Pt}}\right)$$

The RSD of m_{Pt} shall not exceed 0,30 %.

INTERNATIONAL STANDARD

ISO 11494

Second edition 2014-12-01

Jewellery — Determination of platinum in platinum jewellery alloys — ICP-OES method using yttrium as internal standard element

Joaillerie, bijouterie — Dosage du platine dans les alliages de platine pour la joaillerie, bijouterie — Méthode par ICP-OES utilisant l'yttrium comme étalon interne

4.3.5 分析结果的表述

有效五氧化二磷（P_2O_5）含量 w_1 以质量分数（%）表示，按式（1）计算：

$$w_1 = \frac{(m_1 - m_2) \times 0.032\ 07}{m_{01} \times \frac{V_{01}}{250}} \times 100$$

$$= \frac{(m_1 - m_2) \times 801.75}{m_{01} V_{01}} \quad \dots\dots\dots\dots\dots\dots\dots(1)$$

式中：

m_1——测定时所得磷钼酸喹啉沉淀质量，单位为克（g）；

m_2——空白试验时，所得磷钼酸喹啉沉淀质量，单位为克（g）；

0.032 07——磷钼酸喹啉质量换算为五氧化二磷质量的系数；

m_{01}——试料的质量，单位为克（g）；

V_{01}——所取试样溶液的体积，单位为毫升（mL）；

250——试样溶液总体积，单位为毫升（mL）。

取平行测定结果的算术平均值为测定结果。

4.3.6 允许差

平行测定结果的绝对差值不大于 0.20%。

不同实验室测定结果的绝对差值不大于 0.30%。

实际样品测定对ICPOES的要求之四（标准溶液）

- NIST produces and certifies 70 Spectrometric Solution SRMs
  - 1000's sold annually for calibration of ICP, AAS
  - typically 10 mg/g
  - ~0.3% uncertainty
- transfer standards used for calibration

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- 化学分析的精度（RSD%＜0.2%），仪器分析的便捷

Anal. Chem. **2001**, 73, 4821−4829

Single-Element Solution Comparisons with a High-Performance Inductively Coupled Plasma Optical Emission Spectrometric Method

Marc L. Salit,* Gregory C. Turk, Abigail P. Lindstrom, Therese A. Butler, Charles M. Beck II, and Bruce Norman

Chemical Science and Technology Laboratory, National Institute of Standards and Technology, 100 Bureau Drive, Gaithersburg, Maryland 20899-8391

A solution-based inductively coupled plasma optical emission spectrometric (ICP-OES) method is described for elemental analysis with relative expanded uncertainties on the order of 0.1% relative. The single-element determinations of 64 different elements are presented, with aggregate performance results for the method and parameters for the determination of each element. The performance observed is superior to that previously reported for ICP-OES, resulting from a suite of technical strategies that exploit the strengths of contemporary spectrometers, address measurement and sample handling noise sources, and permit rugged operation with small uncertainty. Taken together, these strategies constitute high-performance ICP-OES.

quantitatively different from that previously reported for ICP-OES results. Different chemical information becomes available with this ability to discriminate between solutions of very similar composition—an improvement in *concentration resolution*. Additionally, at this level of uncertainty, uncertainty is dominated by effects other than spectroscopic measurement (e.g., sample handling and preparation).

Measurements are performed using unmodified commercially available equipment and are significantly less costly than classical analysis. The opportunity to achieve results of the quality expected of classical analysis with instrumental efficiency enables new analytical applications. In contrast to classical analysis, instrumental automation permits the analysis of multiple samples with little incremental cost per sample. Similar to classical analysis, this method is suitable for determination of "major" and "minor"

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信号波动=噪音+漂移

◦ 噪声是短期的
◦ 漂移是长期的

Drift is major source of uncertainty

noise

drift

signal

sample number
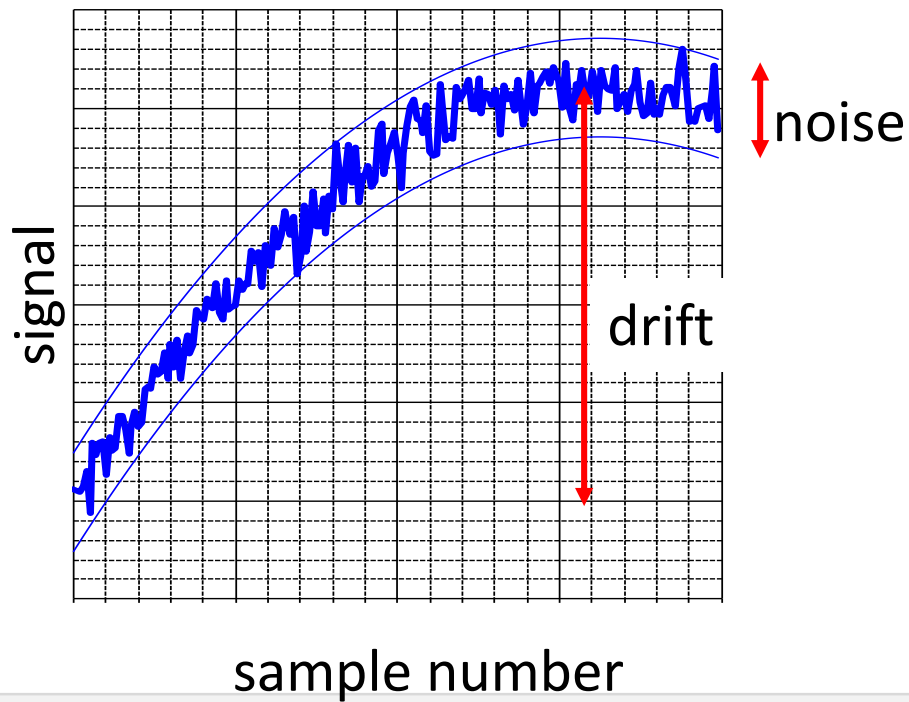
Drift Correction

$$S_{measured} = S_{truth} + \varepsilon_{drift} + \varepsilon_{noise}$$

- separate variables to estimate drift as f(t)
  - use grand mean for sample to estimate S_{truth}
    - create time series of deviations from mean
      - use *ALL* standards and samples
      - this is our estimate of ($\varepsilon_{drift} + \varepsilon_{noise}$)!
        - use relative deviations for multiplicative drift, absolute deviations for additive drift
  - fit smooth function to these deviations
    - this is our estimate of ε_{drift}!

Improved performance using internal standardization in inductively-coupled plasma emission spectroscopy

S. A. MYERS and D. H. TRACY

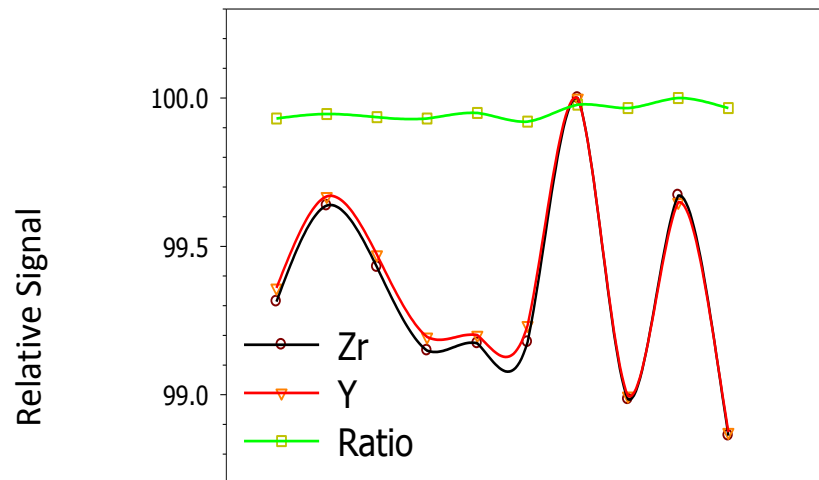Perkin–Elmer Corporation, Main Avenue, Norwalk, CT 06856, U.S.A.

Abstract—Significantly improved performance in ICP emission spectroscopy is possible using internal standardization given the proper choice of operating conditions. A dual monochromator detection system was used to study the correlation between two emission signals recorded simultaneously as several ICP parameters were varied. The behavior of the signal formed by dividing the analyte and reference signals was also measured.

The noise behavior of twenty analytically important elements was compared to that of a manganese ion line used as the internal standard for a single, compromise choice of ICP operating parameters (RF power = 1250 W, carrier gas flow rate = 0.7 or 0.9 l/min, and viewing height = 20 mm). The elements differed widely in excitation and ionization energies. Sample concentrations were chosen so that the analyte emission signals were sufficiently intense that the noise and drift contributions due to plasma background and shot noise could be ignored. Under these conditions very good signal correlation ($r \geq 0.95$) and similar noise proportionality factors were observed leading to noise reductions of $\geq 10 \times$ after signal division in almost every instance. After signal division the noise, defined as the relative standard deviation of the emission signal, (time constant = 1.0 s, $n = 20$) was less than 0.1 %. The closely similar noise behavior is attributed to sample density fluctuations arising from corresponding fluctuations of the sample aerosol density. Good correlation is also observed between fluctuations in the plasma background simultaneously detected at two widely separated wavelengths when appropriate steps are taken to reduce shot noise. At carrier gas flow rates above 1.0 l/min and at viewing heights below 15 mm or above 20 mm, the signal correlation is reduced or absent, yielding little or no improvement using internal standardization.

The use of internal standardization leads to other improvements in analytical performance in addition to noise reduction. Drift is reduced, and nebulizer performance is improved in that it is less sensitive to variations in liquid sample uptake rate, or nebulizer instabilities associated with high solids content sample introduction. The divided signal takes much less time to reach equilibrium following introduction of a new sample.
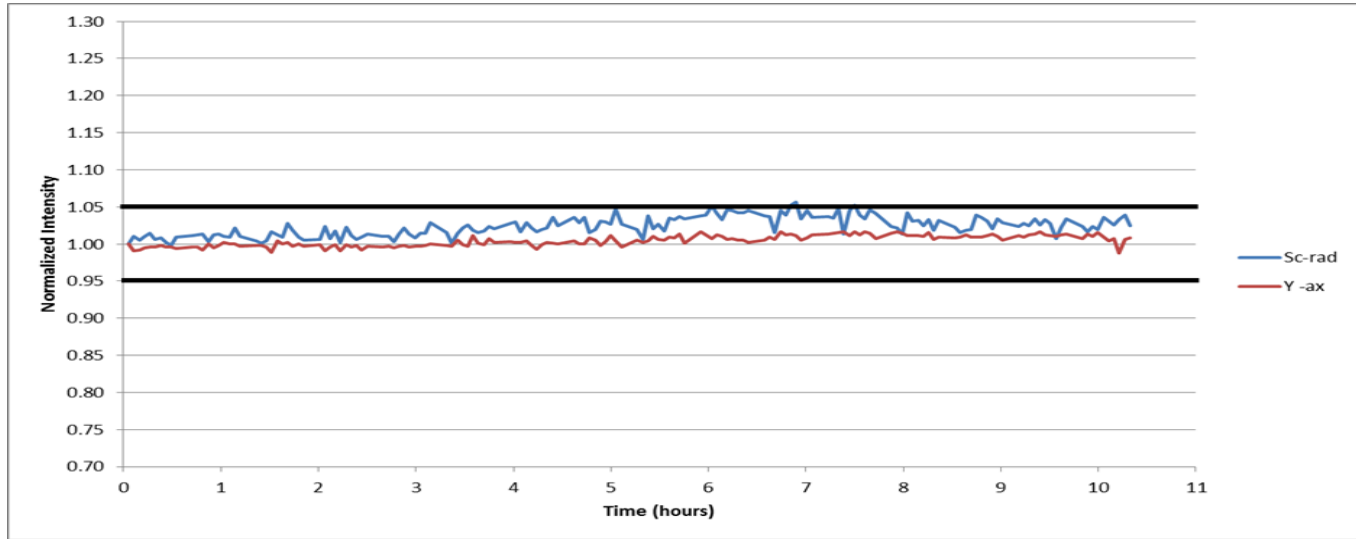
Establishing Traceability to NIST SRMs
with High Performance ICP-OES
Marc Salit, NIST and Sue Evans, VHG

- precision enhancement
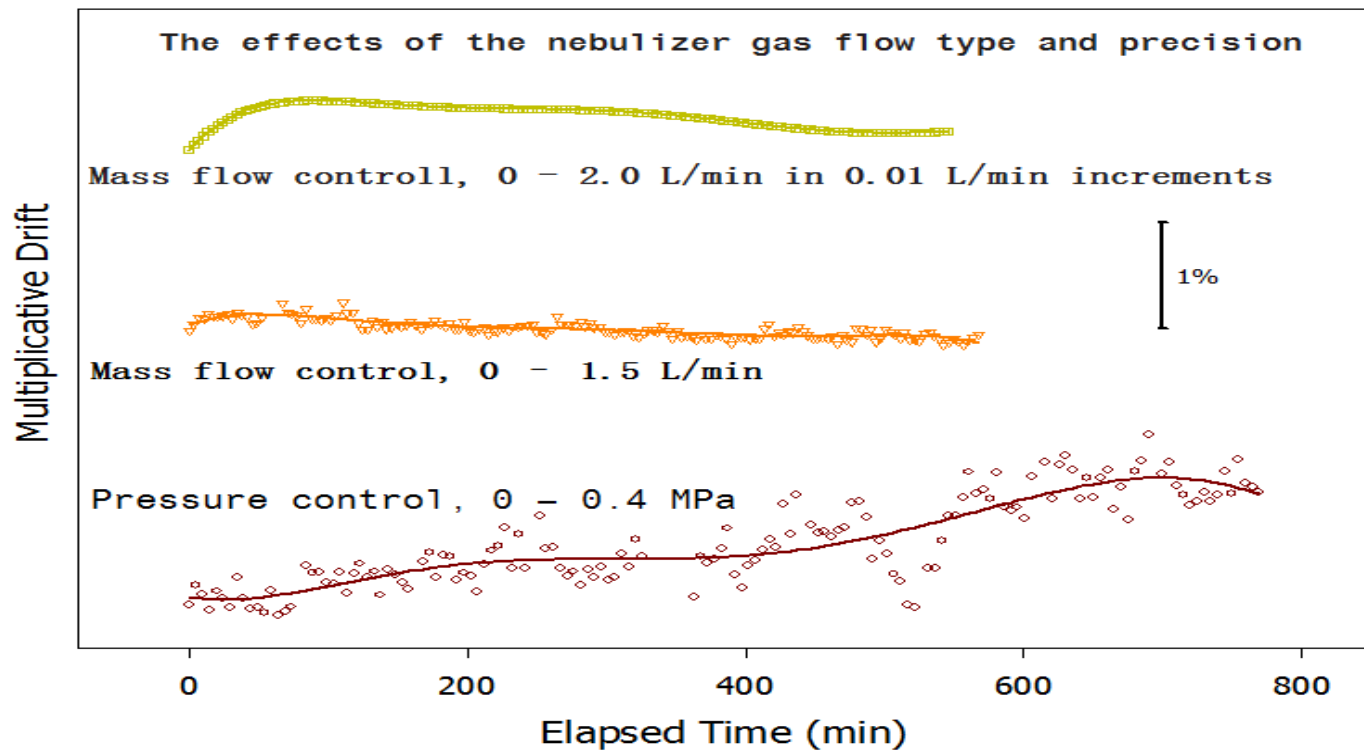 ◦ matched internal standard
  ◦ can be ~20x improvement

Results: Stability

- Monitor internal standards to check for instrument drift
  - ◦ Sc in radial mode, Y in axial mode

Variations of 5% over 10 hours
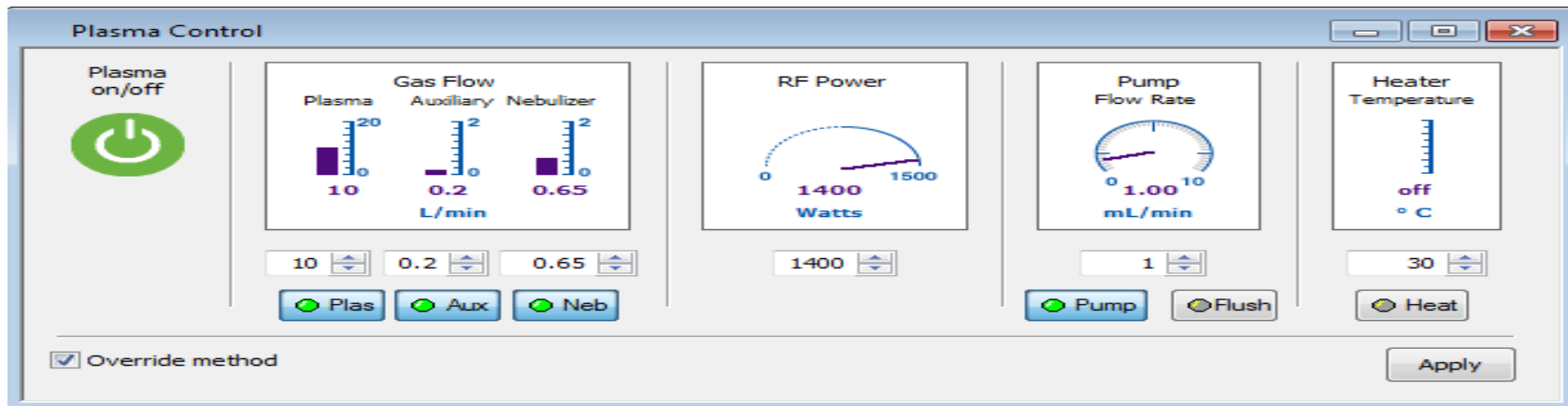
The effects of the nebulizer gas flow type and precision

Mass flow controll, 0 — 2.0 L/min in 0.01 L/min increments

Mass flow control, 0 — 1.5 L/min

Pressure control, 0 — 0.4 MPa

Multiplicative Drift

Elapsed Time (min)

1%

噪音来自蠕动泵、雾化气、电压波动、温度波动…
降低噪音的办法？

噪音来源	处理办法	效果
气体压力波动	使用质量流量计控制雾化器气、冷却气、辅助气流量	有显著效果
电压波动	使用稳压电压	效果不明显
温度变化	配置空调，提高光室恒温精度	有效果

噪音来自蠕动泵、雾化气、电压波动、温度波动。。。

Make a simple Argon diagnostic method and monitor the RSD.

Run a 2v/v% nitric acid solution with four different conditions:

1. Pump on and nebulizer on
2. Pump on and nebulizer off
3. Pump off and nebulizer on
4. Pump off and nebulizer off
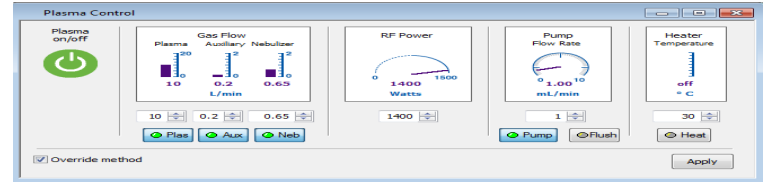
	A	B	C	D	E	F	G	H	I
	Exp_5 Ar-diagnose test								
	Plasma power:	1350		Nebulizer type:		CF	resolution		normal
	Nebulizer flow:	0.85		Nebulizer tubing:		B/B	profiling		no
	Auxiliary flow:	0.2		Injector diameter:		2.0 mm	Replicates		10
	Plasma flow:	15		Peak Algorithm:		Area			
	Pump speed:	1.5		Points per peak:		3	torch position		-3
	methode:		Exp_5		Results file:		Exp_5		
	Read parameters	min 1	max 5						
	test	Ar 420.069 % RSD							
	pump on neb on	0.28							
	pump on neb uit	0.18							
	pump off neb on	0.14							
	pump off neb out	0.03							

Biggest contribution is the PERISTALTIC PUMP!!

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消除噪音，改进RSD到0.1%的关键是消除蠕动泵脉动

- 同步内标校正


```
--------------------------------------------------------------------------------
Sequence No.: 1                          Autosampler Location:
Sample ID: Sample002                     Date Collected: 2017/10/18 16:47:10
Analyst:                                 Data Type: Reprocessed on 2017/10/19 14:03:11
Logged In Analyst (Original) : lenovo
Initial Sample Wt:                       Initial Sample Vol:
Dilution: 1144X                          Sample Prep Vol:
Wash Time (before sample):      分别使用Y和Sc作为Co和Mn的内标
--------------------------------------------------------------------------------

Mean Data: Sample002
                  Mean Corrected        Calib.                   Sample
Analyte             Intensity      Conc. Units    Std.Dev.    Conc. Units    Std.Dev.    RSD
Sc 357.253           362735.4                                               1750.31     0.48%
Y 324.227            201340.0                                               1088.82     0.54%
Y 360.073            260931.3                                               1393.02     0.53%
Sc 357.634           178463.4                                                908.55     0.51%
Co 228.616†            5569.9     16.17 mg/L      0.011      18500 mg/L       12.59     0.07%
Co 238.892†           31495.3     15.99 mg/L      0.011      18300 mg/L       12.19     0.07%
Co 236.380†           11857.8     16.08 mg/L      0.008      18400 mg/L        8.90     0.05%
Mn 257.610†          130261.2     23.89 mg/L      0.010      27330 mg/L       11.81     0.04%
Mn 259.372†          270620.9     23.76 mg/L      0.016      27180 mg/L       18.03     0.07%
Mn 260.568†           98557.7     23.73 mg/L      0.019      27140 mg/L       22.25     0.08%
```

实时内标校正保证 RSD<0.1%

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同步内标校正关键是分析线与内标线有完全相同的曝光时间

Results

```
================================================================================
Sequence No.: 1                        Autosampler Location:
Sample ID: Stand High                  Date Collected: 12/18/2014 10:08:54 AM
Analyst:                               Data Type: Reprocessed on 12/30/2014 2:41:03 PM
Logged In Analyst (Original) : ICP1
Initial Sample Wt: 1 g                 Initial Sample Vol:
Dilution:                              Sample Prep Vol: 100 mL
Wash Time:
--------------------------------------------------------------------------------
Auto-Integration Report

Analyte                    Integration        Number of          Read
                           Time (s)           Integrations       Time (s)
Be 313.042 Ax              0.010              1000               10.000
Be 313.042 Rad            0.100               100                10.000
Be 313.107 Ax             0.010               1000               10.000
Be 313.107 Rad            0.100               100                10.000
Sc 361.383 Ax             0.010               1000               10.000
Sc 361.383 Rad            0.500               20                 10.000
Y 371.029 Ax              0.010               1000               10.000
Y 371.029 Rad             0.500               20                 10.000
Yb 289.138 Ax             0.100               100                10.000
Yb 289.138 Rad            0.500               20                 10.000
Pt 193.700 Rad            0.500               20                 10.000
Pt 204.937 Rad            0.500               20                 10.000
Pt 214.423 Rad            0.500               20                 10.000
Pt 265.945 Ax             0.010               1000               10.000
Pt 265.945 Rad            0.100               100                10.000
Ru 240.272 Ax             0.100               100                10.000
Ru 279.535 Ax             0.100               100                10.000
Ru 349.894 Ax             0.100               100                10.000
--------------------------------------------------------------------------------
```

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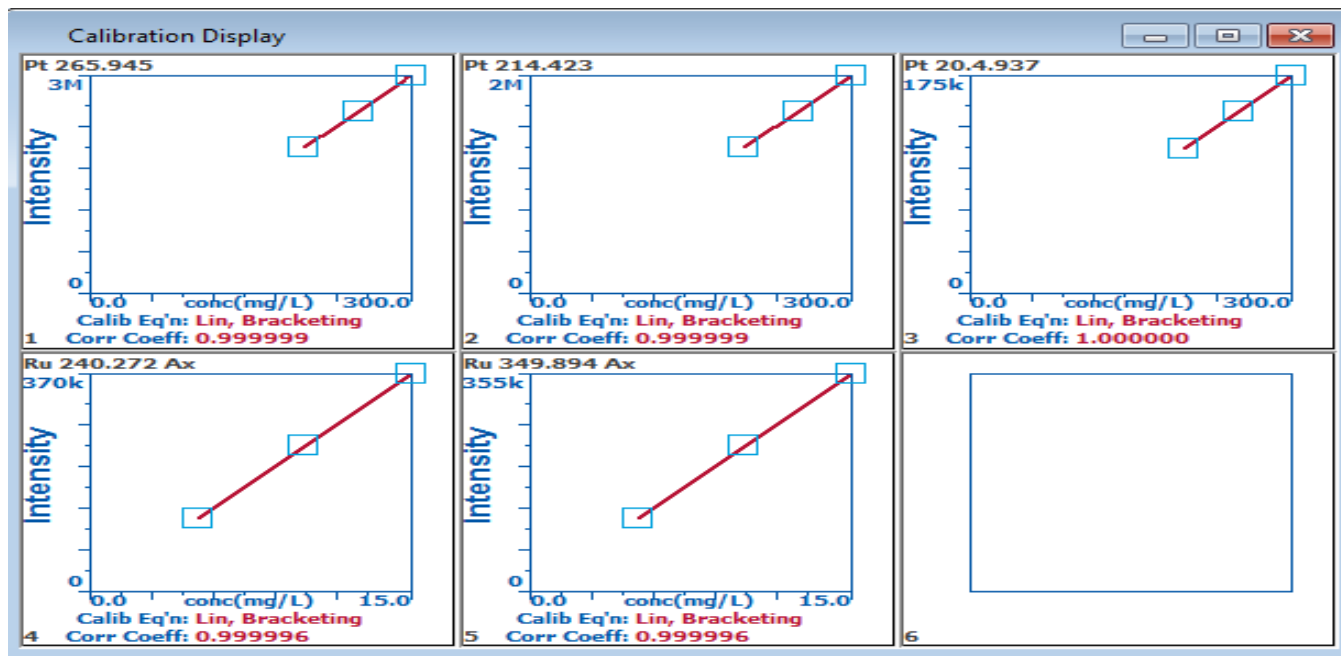
Full Compliance with ISO11494, 11495, etc.

高性能ICPOES应用实例一：贵金属检测
Pt、Ru检测结果RSD%＜0.1%

```
-----------------------------------------------------------------------------
Sequence No.: 3                          Autosampler Location: 3
Sample ID: Std2                          Date Collected: 12/18/2014 2:02:20 PM
Analyst:                                 Data Type: Reprocessed on 12/30/2014 3:08:56 PI
Logged In Analyst (Original) : ICP1
Initial Sample Wt:                       Initial Sample Vol:
Dilution:                                Sample Prep Vol:
Wash Time:


-----------------------------------------------------------------------------
Mean Data: Std2
                  Mean Corrected                              Calib
Analyte             Intensity      Std.Dev.     RSD      Conc. Units
Be 313.107 Ax      2119890.3       7221.66     0.34%     98.67 %
Be 313.042 Ax      4285765.2      14410.54     0.34%     98.72 %
Y  371.029 Ax      1337847.3       3817.57     0.29%     98.38 %
Yb 289.138 Ax       154247.0        448.19     0.29%     98.23 %
Sc 361.383 Ax      1485809.9       4170.77     0.28%     98.30 %
Pt 265.945†        2529182.3        818.69     0.03%     [250] mg/L
Pt 214.423†        1699057.0        632.78     0.04%     [250] mg/L
Pt 20.4.937†        145684.1         63.67     0.04%     [250] mg/L
Ru 240.272 Ax†      247572.0        177.19     0.07%      [10] mg/L
Ru 349.894 Ax†      236982.8         20.39     0.01%      [10] mg/L
```

内标

分析物

24

- comparison of NIST SRM against…
  - EMPA distilled Zn
  - high purity metals
- calibration curve for 20 solutions
  - 800 min expt
  - 0.0082% solution-to-solution variability

measured at EMPA, June 1999
standard deviation of the mean = 0.0082%
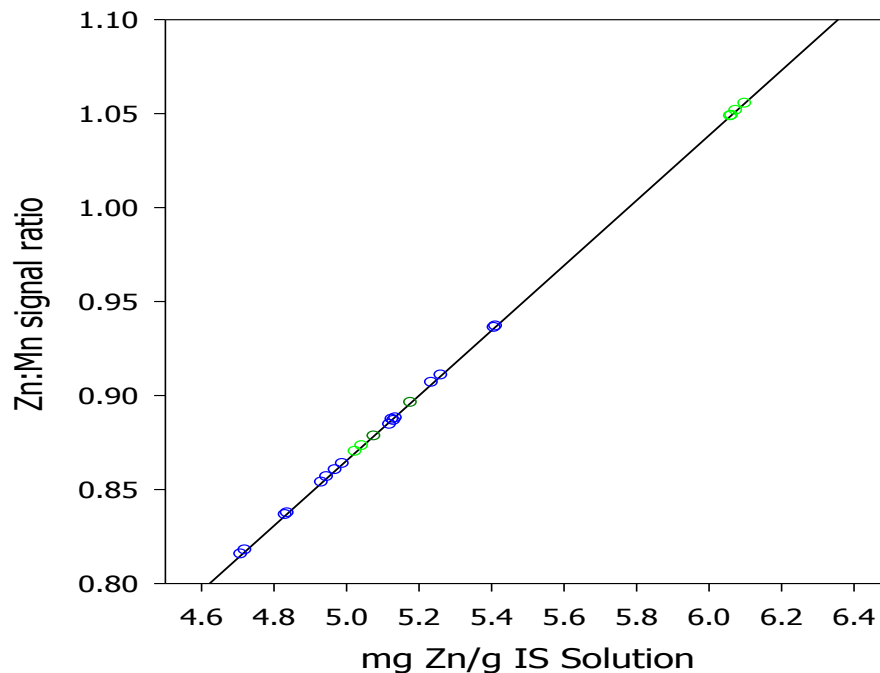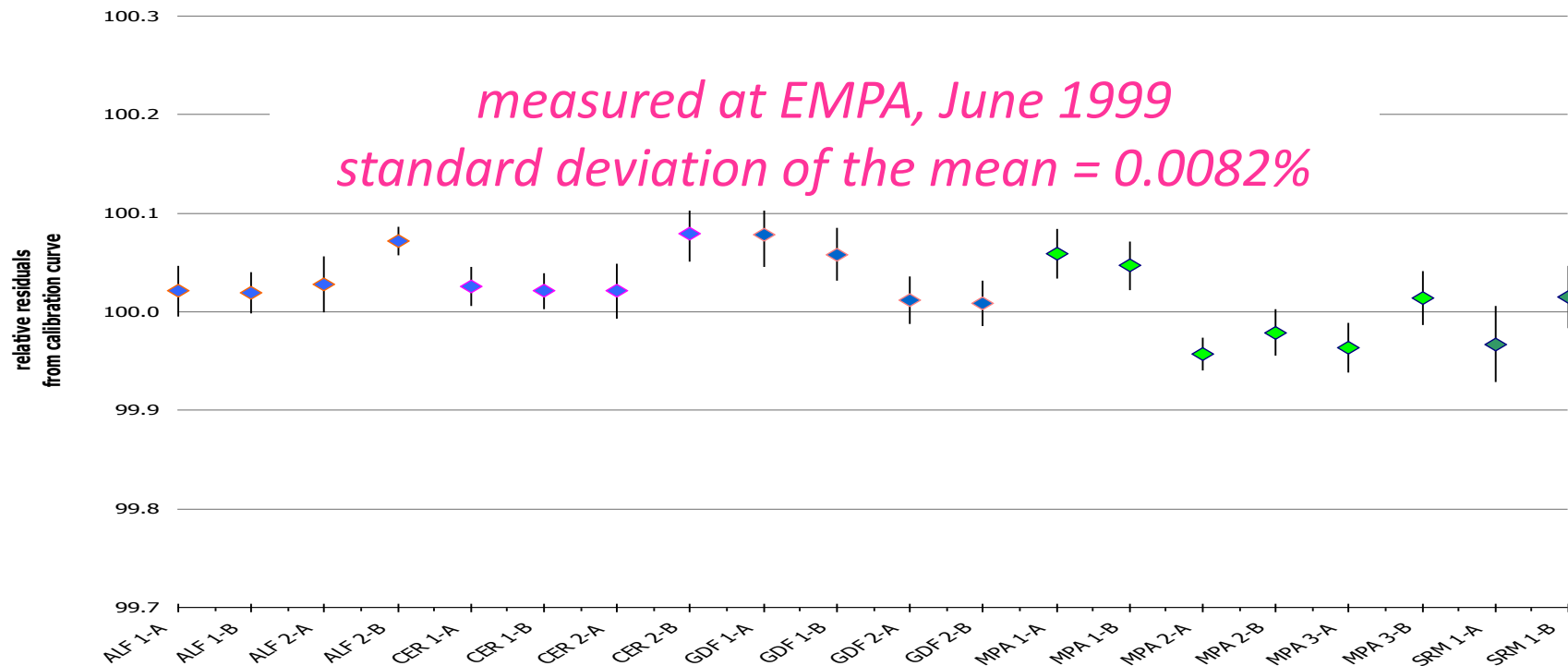
高性能ICPOES应用实例三：NdFeB合金主量元素分析

ICP–AES 法高精度测定钕铁硼合金中主量元素

徐　　静[*]，李明来，李世鹏，彭海军，赵　斌，于敦波

（北京有色金属研究总院稀土材料国家工程研究中心，有研稀土新材料股份有限公司，北京 100088）

Table 7　Precisions（$n = 10$）and recoveries of the method

Component	Found $w/(\%,$ mass fraction)	Average $w/(\%,$ mass fraction)	RSD/ %	Added $w/(\%,$ mass fraction)	Found $w/(\%,$ mass fraction)	Recovery/ %
Nd	29.90, 29.88, 29.84, 29.90, 29.87, 29.88, 29.85, 29.86, 29.81, 29.85	29.86	0.09	30.0	58.1	97
Fe	69.05, 69.07, 69.11, 69.05, 69.08, 69.07, 69.10, 69.09, 69.14, 69.09	69.08	0.04	70.0	142.95	103
B	1.051, 1.050, 1.045, 1.050, 1.051, 1.047, 1.053, 1.046, 1.053, 1.055	1.050	0.31	1.0	2.01	98

➤ PerkinElmer 独有的扣除光谱干扰功能，解决了 ICP-OES分析复杂基体样品中的谱线干扰问题，其原理是利用现代化学计量学和多元线性回归算法，在分析复杂基体样品时，通过数学模型将分析谱线从干扰光谱中剥离出来。只需要保持峰型稳定，所以该模型通常与样品浓度及特定的离子状况等无关，

　　因此实际应用非常方便，只需要空白溶液、待测元素溶液及干扰元素溶液即可。
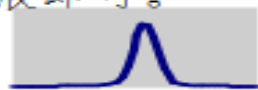
Analyte
纯分析元素

Blank
空白

Matrix
纯基体

=

Measured Spectrum
实际测定的光谱图

PerkinElmer
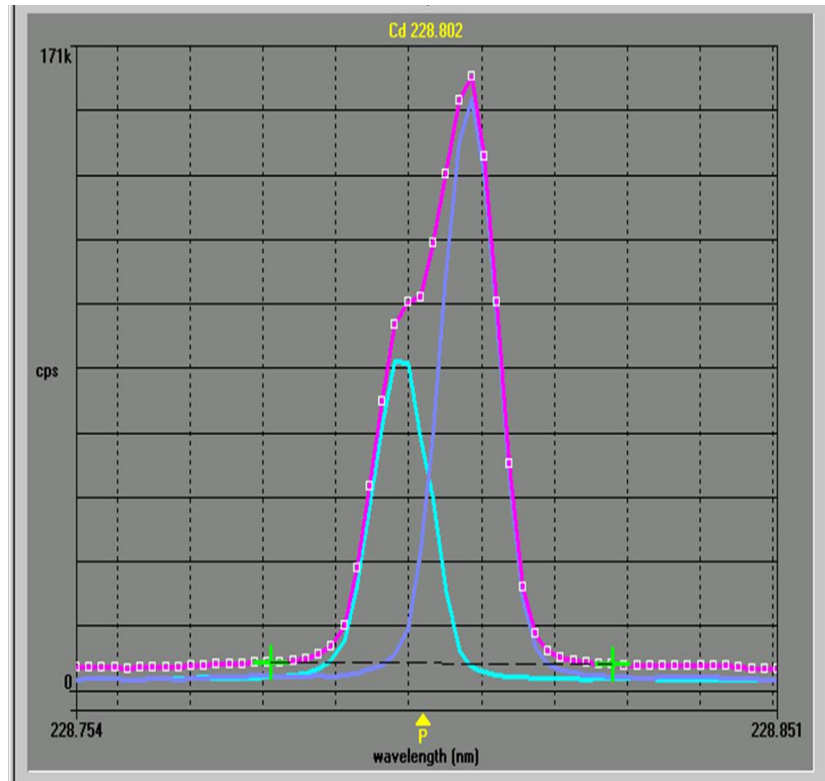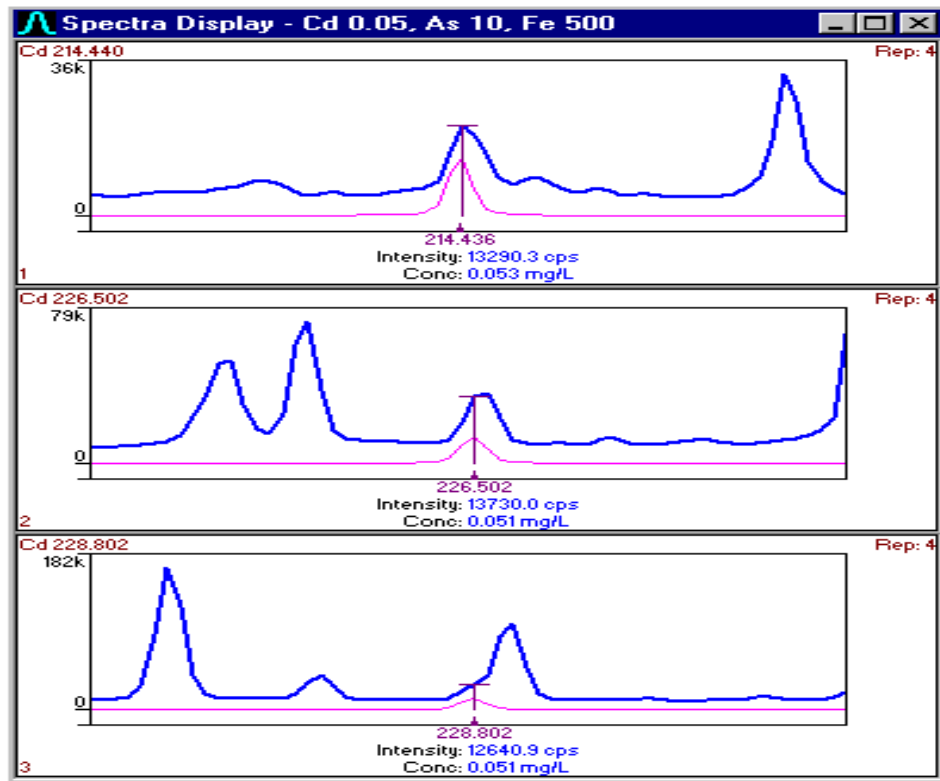
MSF模型解析用于Be的测定(干扰Co、Mo、Fe、Cu)
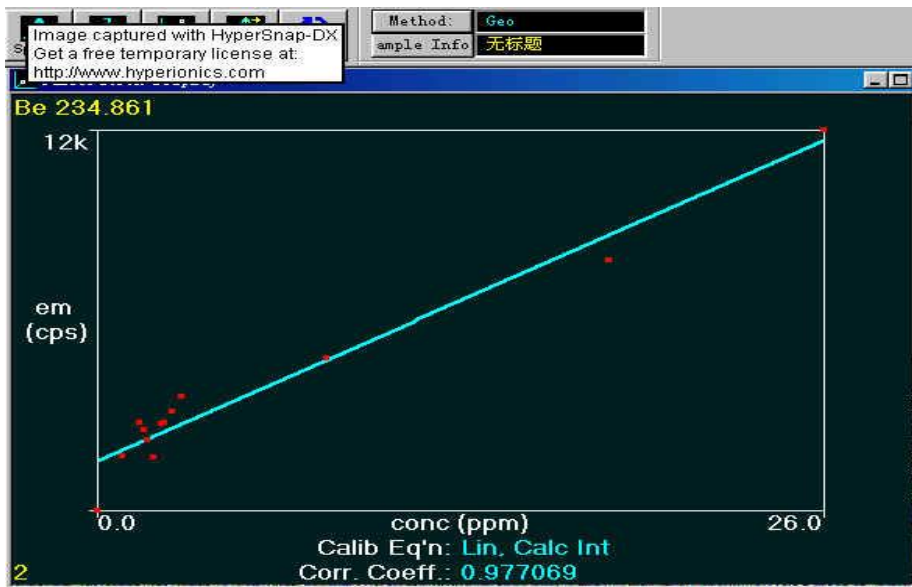
没有使用MSF解析的拟合曲线
r=0.977

使用MSF解析后的拟合曲线
r>0.999

PerkinElmer

高性能ICPOES其他要求：复杂样品分析能力
复杂样品得到理想检测结果

Stainless Steel   Type 304L
B.S. 81G   Values expressed as weight%

Element and Wavelength	Reference Value	Avio500 Value
B 249.772	0.0005 ±0.001	0.00049 ±0.000005
Co 228.616	0.29 ±0.01	0.296 ±0.003
Cr 283.563	18.65 ±0.08	18.73 ±0.075
Cu 324.752	0.54 ±0.02	0.543 ±0.004
Mn 294.920	1.69 ±0.03	1.72 ±0.013
Mo 203.845	0.69 ±0.02	0.685 ±0.006

双向观测的ICP-OES，才可以同时分析出百分含量的主成分和固体中ppm量级的痕量成分。

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10000 Mo 10sec
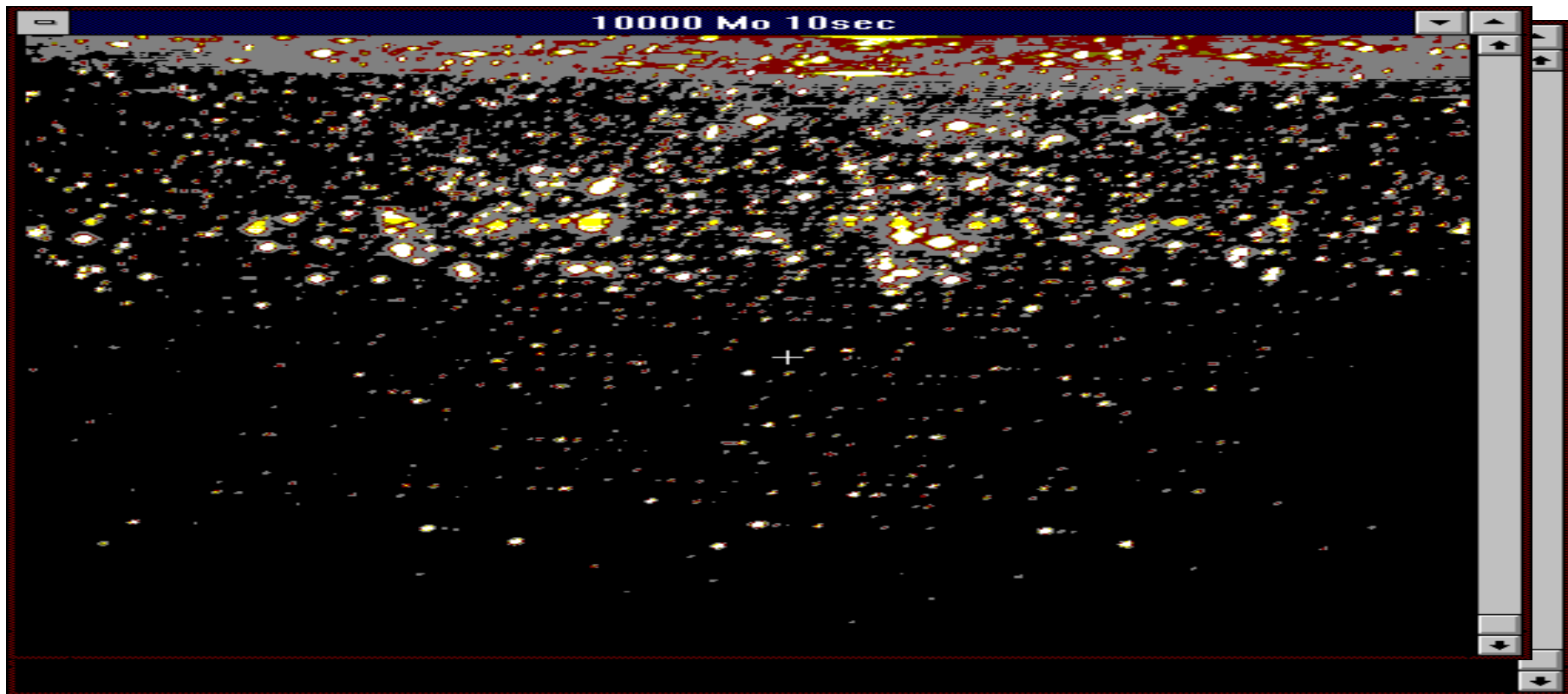
中阶梯光栅与跨级干扰（1）

- 中阶梯光栅虽然有非常好的光学分辨能力，但由于使用大级数分光，需要注意光谱级次间的干扰，如：

- Pb220.353nm，本来不会受到Cu的干扰，最近的Cu谱线波长也会相差0.3nm。
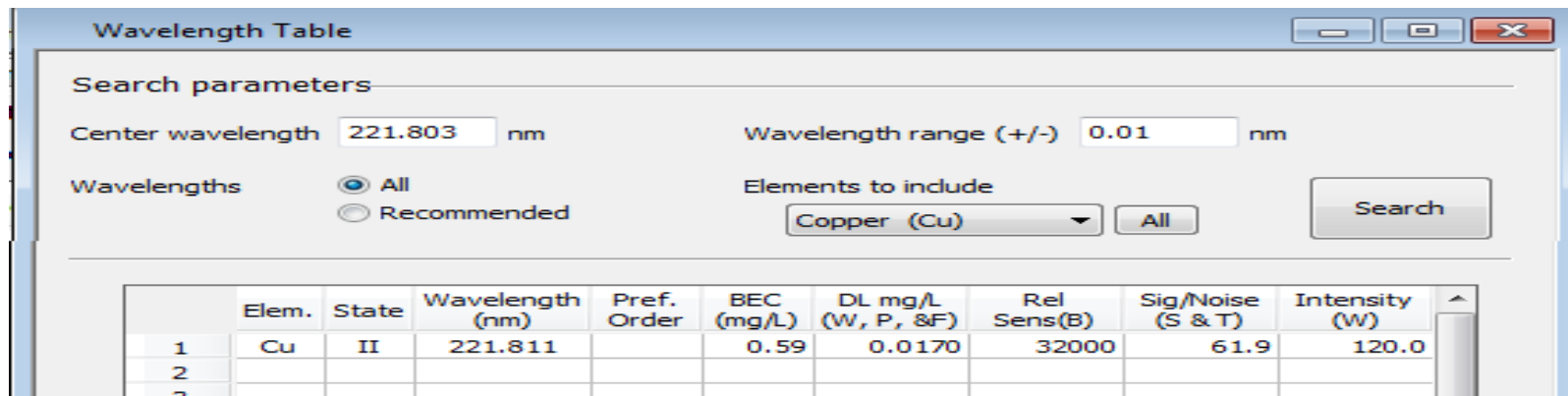
- 但某型号仪器使用的光学级次为153级，会受到152级次上的Cu谱线的严重干扰。

中阶梯光栅与跨级干扰（2）

- 接上页：
- Pb220.353nm（153级）在152级对应的波长为：
- 221.803nm（220.353nm*153/152），与221.811nm的Cu线相近

- 221.811nm（152级）相当于220.361nm（153），将对Pb220.353nm（153）产生严重的干扰，使含铜(如青铜）样品中Pb的测定出现大问题！

高性能ICPOES其他要求：复杂样品分析能力
需要处理跨光谱级次的干扰

- 1、检测器上的处理。
- 2、光学级次的选择。
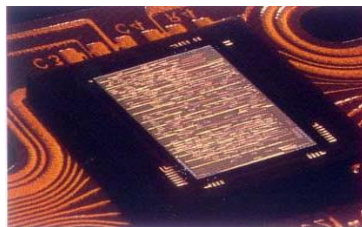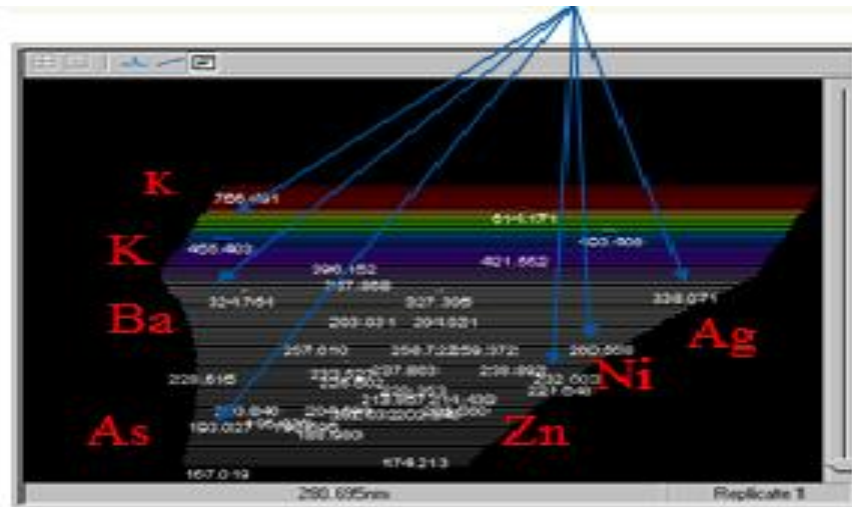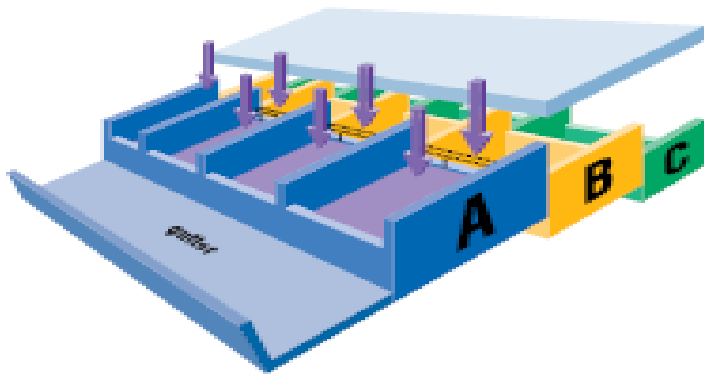
- 记录全部的光学信息而不是用户选择的几条谱线的信息。
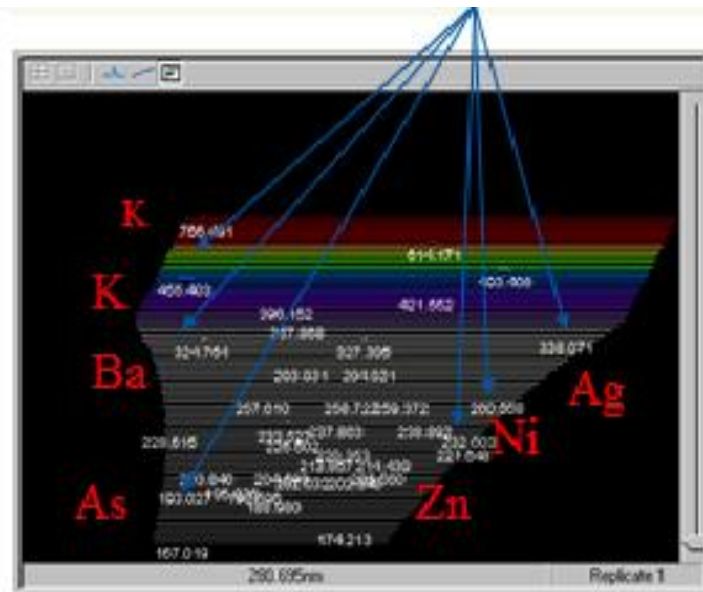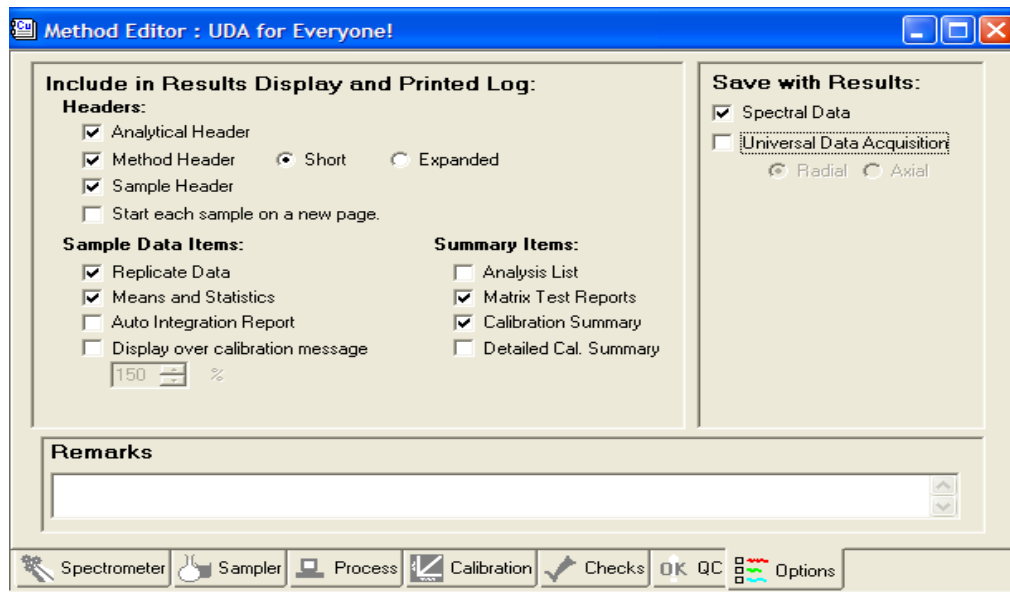
- 在检测结果中请注意：
  - Fe的回收率较低
  - Co 的结果为负值
  - Sn 的结果很高

- 那么!
  - 通常，重做吧！
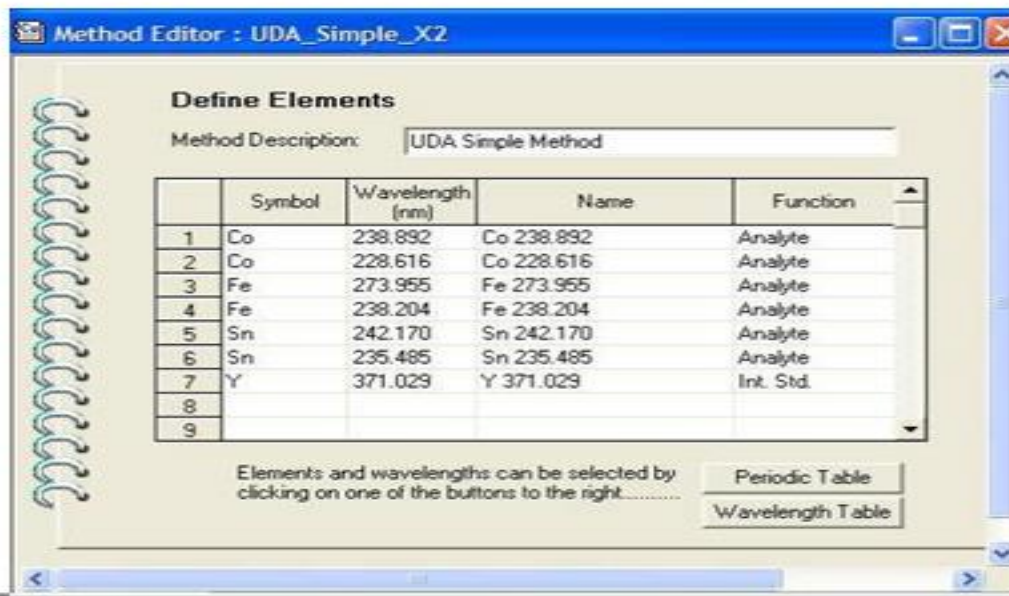
高性能ICPOES其他要求：得到完整的光谱学信息
全谱直读的作用：解决谱线选择错误

➤ 对于全谱直读仪器，那么你可以随时随地在检测方法中加入其他谱线并直接再处理数据即可。

数据再处理

添加 Co 228, Fe 273, Sn242

Method Editor : UDA_Simple_X2

Define Elements

Method Description: UDA Simple Method

	Symbol	Wavelength (nm)	Name	Function
1	Co	238.892	Co 238.892	Analyte
2	Co	228.616	Co 228.616	Analyte
3	Fe	273.955	Fe 273.955	Analyte
4	Fe	238.204	Fe 238.204	Analyte
5	Sn	242.170	Sn 242.170	Analyte
6	Sn	235.485	Sn 235.485	Analyte
7	Y	371.029	Y 371.029	Int. Std.
8				
9				

Elements and wavelengths can be selected by clicking on one of the buttons to the right..........
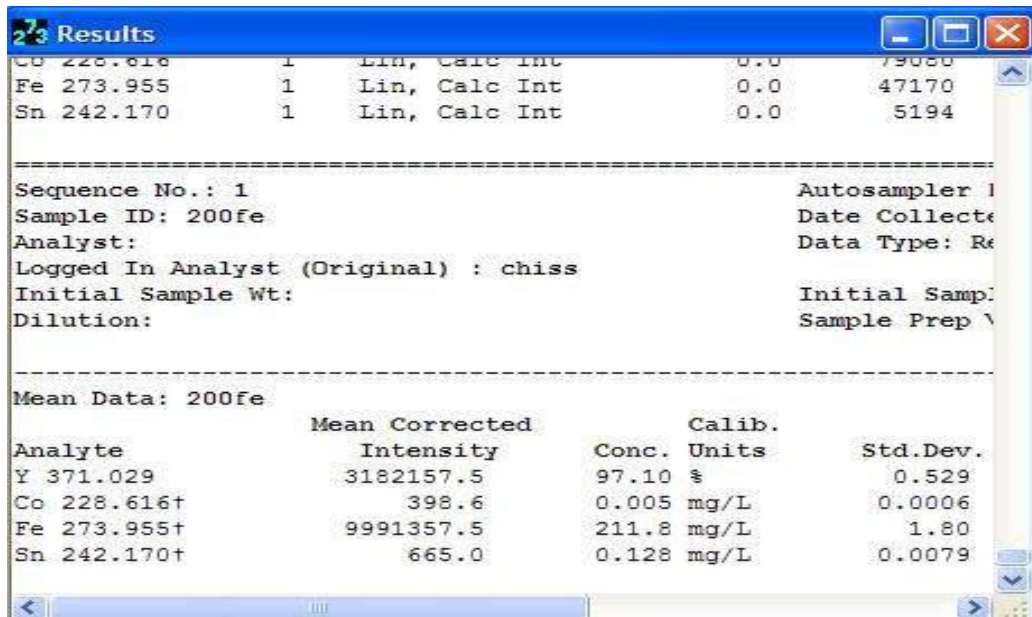
Periodic Table

Wavelength Table

高性能ICPOES其他要求：得到完整的光谱学信息
全谱直读的作用：解决谱线选择错误

- 现在请注意：Fe的结果更加的接近200ppm的真值，同时Co和Sn的结果也已经消除了干扰。

- 可以对任何元素，任何干扰进行处理，不需要重新分析！！！！

- ## UDA 校准溶液包
  - ○ 共4瓶各100ml的溶液

    - 10 ppm

    - 稀释1:10

    - 1 ppm

    - 元素周期表中所有元素

PerkinElmer UDA Standards Kit
Part no. N9300685

高性能ICPOES的要求：得到完整光谱学，不能缺少重要谱线
检测器与光学系统的匹配问题

- 某仪器：
- 光栅刻线52.91条/mm，闪耀角63.5°。光学系统焦距为383mm。
- 检测器参数：像素大于290000（540*540），像素宽度0.028mm（15mm/540）
- Order 82时：
- 中心波长λ=2*(1/52.91)*sin63.5°*1000000/82=2*0.0189*0.895*1000000/82=412.57nm
- Order 83时：
- 中心波长λ=2*(1/52.91)*sin63.5°*1000000/83=2*0.0189*0.895*1000000/83=407.6nm
- Order 84时：
- 中心波长λ=2*(1/52.91)*sin63.5°*1000000/84=2*0.0189*0.895*1000000/84=402.75nm
- **所以当光学级数为83时，检测器需要覆盖的波长为（412.57-402.75）/2=4.91nm**
- 根据光栅方程计算：
- 中心波长λ=2*(1/52.91)*sin63.5°*1000000/83=2*0.0189*0.895*1000000/83=407.6nm
- 光栅角色散率= m/(d*cosβ)=83/(0.0189*0.446)=9846.49.85弧度/mm=0.0098弧度/nm
- 光学系统线色散率倒数=1/(角色散率*焦距)=1/(0.0098*383)=0.2652nm/mm
- 检测器像素分辨率=线色散率倒数*像素宽度=0.2652*0.028=0.0074nm
- **检测器每行覆盖波长=每个像素覆盖波长*每行像素数=0.0074*540=4.01nm**

- **严重后果：该仪器没有La408.672nm和La398.852ı**

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