

樂盟季刊

The introduction of Thermo Scientific iCAP Q ICP-MS and iCAP 7000 ICP-OES with ESI prepFAST Autodilution system 智能自動化配製檢量線及自動稀釋樣品的自動取樣器介紹

關鍵字

prepFAST、食品添加劑、洛克沙砷、Apple leaves

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- Thermo Scientific iCAP 7000 Series ICP-OES在食品添加劑分析上的應用
- 利用HPLC-ICP-OES測定洛克沙砷及其代謝物
- 利用Thermo Scientific iCAP Qc ICP-MS 分析NIST SRM標準品驗證食品五重檢測實驗方法
- Automated, Intelligent Sample Preparation: Integration of the ESI prepFAST Auto-Dilution system with the Thermo Scientific iCAP Q ICP-MS

前言

本期主刊以ESI prepFAST 自動化系統做詳細的介紹，prepFAST 具有高樣品通量、快速取樣、快速清洗、自動配製檢量線與自動稀釋樣品等功能，並且具有良好的穩定性、準確性及精密性。ESI prepFAST與Thermo iCAP Q及iCAP 7400能完美的進行連結，因此一推出即得到熱烈迴響，prepFAST不僅提升樣品分析的效率更能够有效降低實驗室成本，將成為分析實驗室未來發展的重要工具。

近年來食品風暴襲擊全台各類食品，自原料端到產品端的每個環節一一被放大檢視，食品的安全儼然成為全民關注的重要議題。本期副刊利用ICP-OES分析食品添加劑-人工食用色素，分析數種常見的人工食用色素中金屬元素含量是否符合法規標準，在享受美食時也多了一份保障。HPLC串聯ICP-OES可用來測定洛克沙砷及其代謝物，雖ICP-OES感度不如ICP-MS高，但此次分析利用HPLC串聯Thermo Scientific iCAP 7000仍可得到不錯的偵測極限。最後利用Thermo Scientific iCAP Q ICP-MS，分析NIST SRM1515蘋果葉及SRM 1568b米粉標準品，依兩種標準物質的COA比對實際分析結果，來驗證食品中五大重金屬檢測方法的準確性。

樂盟科技在今年七月於臺北、新竹和台南三地舉辦了綜合應用研討會，我們以食品、藥品、環境分析及可提升分析效率並降低人為分析污染的自動樣品配製系統為主題，分享分析儀器最新技術的演進與應用實例，感謝大家熱情參與，讓此活動圓滿落幕。樂盟也在研討會後舉辦員工旅遊“雷射槍戰”活動，此活動不僅能舒解壓力也促進員工之間的情誼，樂盟永遠秉持著「快樂」、「健康」和「人性」的宗旨持續為客戶服務，感謝大家的支持。

Reed的話

要為客戶建立完整服務體系，團隊是成功的鑰匙。

大團隊是指一個一個任務有二到三人可以相互支援執行同一個任務。

團隊的成長及與市場結合的訓練是重點。

我們團隊：自己加油，穩步向前，過程快樂，結果小心經營，成功就在加油小心快樂及經營的路途中。

路途上，我們需要許多，道理簡單，萬物生而不足，依自然環境，群體協助而成長。

萬物成長有時有序，日月星辰山川流水，週而復始，妳你我他，卻是並非按時依序。哭笑唏噓卻時時佔據我們的生命週期，在分分秒秒中建立自己的能力，我們的心思與企圖心，最終也影響了那最終的結果。

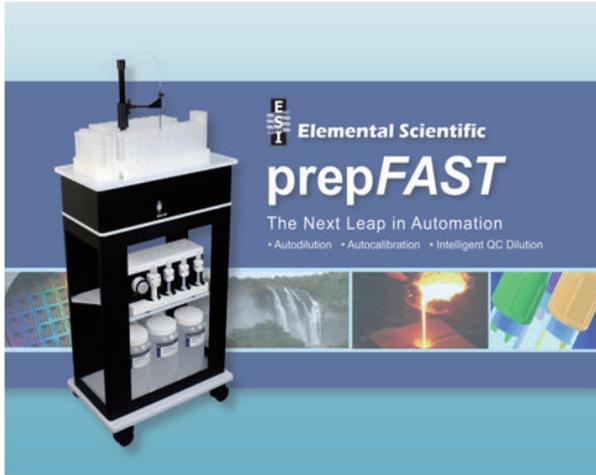
各位，我們攜手，快樂，成長成為一個大樂盟團隊。



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應用工程師 張凱媛

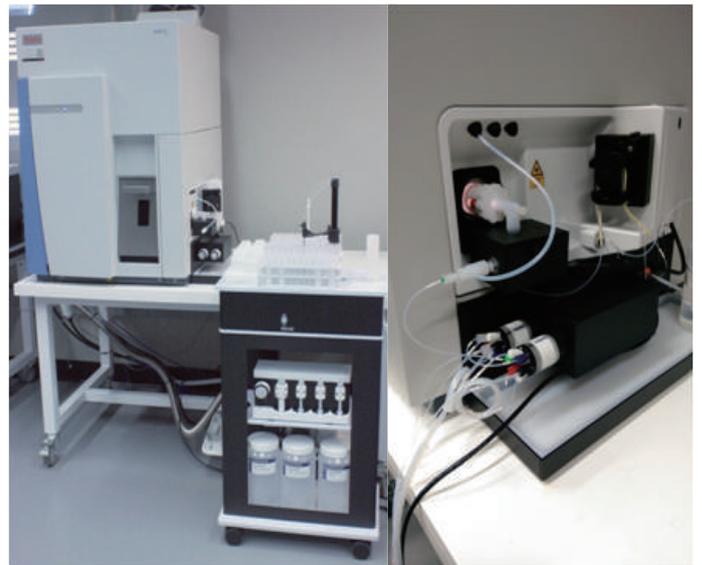
Email: carriechang@joytech.com.tw



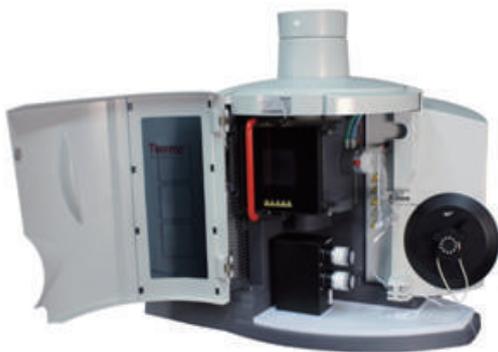
在ICP-MS 或 ICP-OES分析的前置作業上，需依待測元素配製成至少包含3種不同濃度的檢量線，並且檢量線之線性需合乎規範。待測物的前處理也是樣品分析中不可輕忽的流程，例如未知樣品需要經過一定倍數的稀釋處理後才能上機分析，或是經微波消化的樣品，考量到樣品基質的差異性，而需要再稀釋適當的倍數才能上機分析，若分析數據中待測元素濃度超過檢量線的線性範圍，抑或基質影響超乎預期，則需要執行額外的樣品稀釋。雖然這些繁複的動作皆可由受過良好訓練的使用者來完成，但需消耗較多的人力及時間。隨著科技不斷的進步，在追求自動化的條

件下，由Elemental Scientific Ins. (ESI)推出的新一代自動取樣器- prepFAST，具備自動配製檢量線及自動稀釋樣品的功能，亦能將背景值維持在一定的水準，可與Thermo iCAP Q 和 iCAP 7400完美連結，並且透過Qtegra的ISDS(Intelligent Scientific Data Solution)智能資料處理系統自動判斷分析數據，當樣品濃度超過檢量線範圍時，可自動計算出合適的稀釋倍率並自動稀釋樣品再次執行分析，也能自動稀釋內標超過規範的樣品，使樣品分析的確效能符合規範。

ESI prepFAST自動化系統能避免人為汙染、減少樣品稀釋產生的誤差並且節省大量的人力成本，在與Thermo Scientific儀器完美的連結下，將成為實驗分析上不可或缺的最佳配備。



圖一 & 圖二：Thermo iCAP Q連接ESI prepFAST II



圖三&圖四：Thermo iCAP 7400 連接prepFAST II

prepFAST 運作簡介

ESI prepFAST自動取樣器是結合兩個多項閥與4個獨立的注射幫浦(syringe pump)-S400V(圖五)進行運作，較傳統的蠕動幫浦能提供更高的精密度與準確性。每個注射幫浦流速可控制在1~20000 $\mu\text{L}/\text{min}$ 的範圍內，且每個注射針具有不同的功能，S1為潤洗(Rinse)，S2為載流(Carrier)，S3為稀釋(Dilute)，S4為添加內標準品。S400V透過控制S2及S3注射的流速比例，搭配多項閥在數秒內即可完成樣品稀釋，並且S4能同時以穩定的速率添加內標準品至樣品中。prepFAST兩個多項閥結合S400V的運作模式彙總為下列4個步驟(圖六)：



圖五：S400V ESI syringe pump

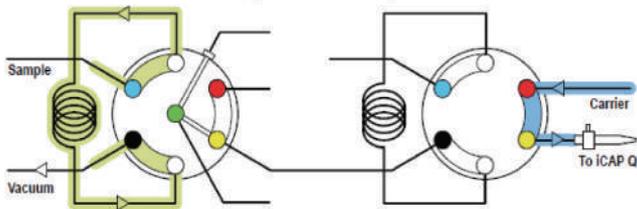
- 步驟1：樣品透過真空幫浦於5秒內充滿左邊多項閥上的sample loop。
- 步驟2：透過注射幫浦將左邊Sample Loop中的樣品和稀釋液混合並同時加入內標，注射至右邊多項閥的Sample Loop中。
- 步驟3：已稀釋的樣品注入霧化器的同時，Sample Loop進行進行清洗的動作。
- 步驟4：清洗兩邊的多項閥及Sample Loop，即完成一個完整的樣品自動稀釋分析流程。

prepFAST快速分析

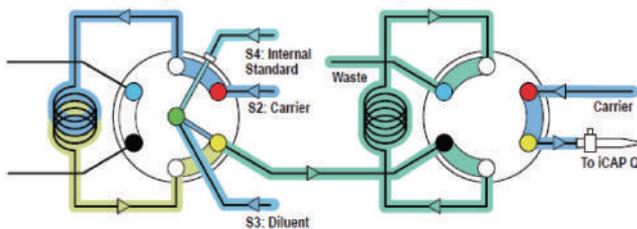
prepFAST系統透過真空幫浦可快速吸取樣品(<5秒)並且快速清洗Sample loop，有效地縮短每個樣品uptake及rinse時間。

一般分析與快速分析所花費的時間示意圖如圖七所示，分析相同的樣品，快速分析能節省樣品吸取和管路清洗的時間，故在樣品測量時間固定的情況下，可提升2-3倍的分析效率。

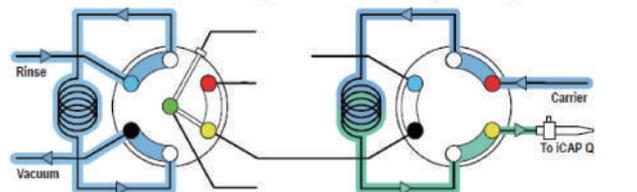
1. Vacuum load sample into loop



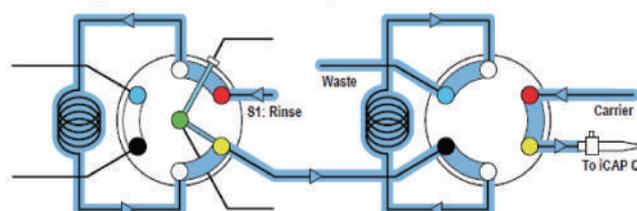
2. Syringes add internal standard and dilute sample into second loop



3. Diluted sample is injected and sample loop is cleaned

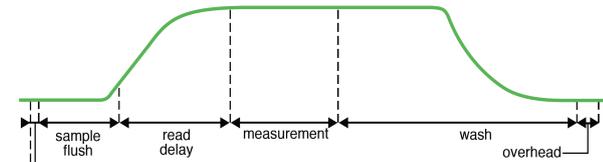


4. Sample and dilution loops are cleaned

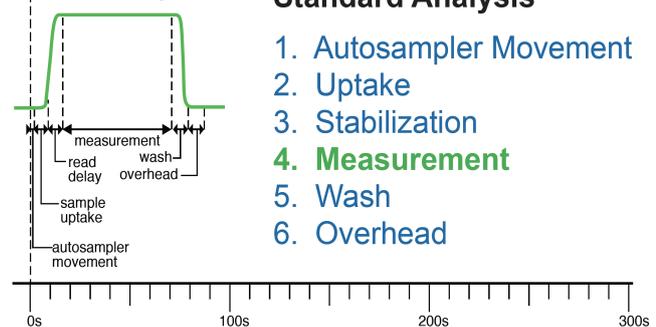


圖六：prepFAST系統中兩個多項閥的運作示意圖

Normal Analysis



FAST Analysis



Six Steps in a Standard Analysis

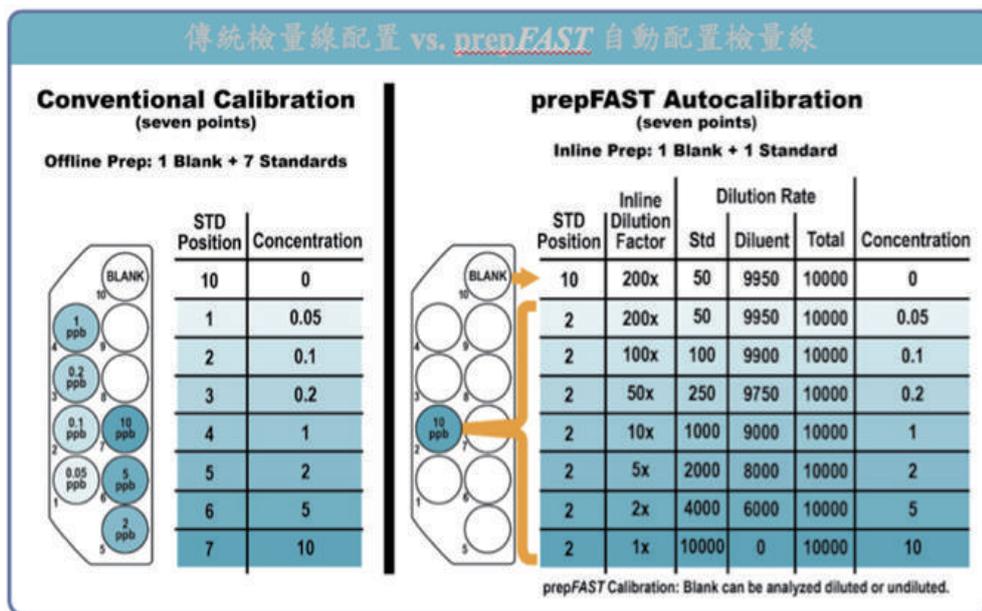
1. Autosampler Movement
2. Uptake
3. Stabilization
4. Measurement
5. Wash
6. Overhead

圖七：一般樣品分析時間與連接FAST系統的樣品分析時間比較

prepFAST自動配製檢量線

分析樣品的前置作業中，需依據待測元素取標準品配置至少包含3種不同濃度的檢量線，甚至為求準確而配置7種不同的濃度，不僅花費時間與人力，還須避免人工配置可能造成的污染與誤差。然而透過prepFAST自動配製檢量線的功能，使用者僅需配置一高濃度的待測元素標準品，即可依所需之倍率自動配置出指定濃度的檢量線，傳統配置檢量線與prepFAST自動配置檢量線之比較如圖八所示。

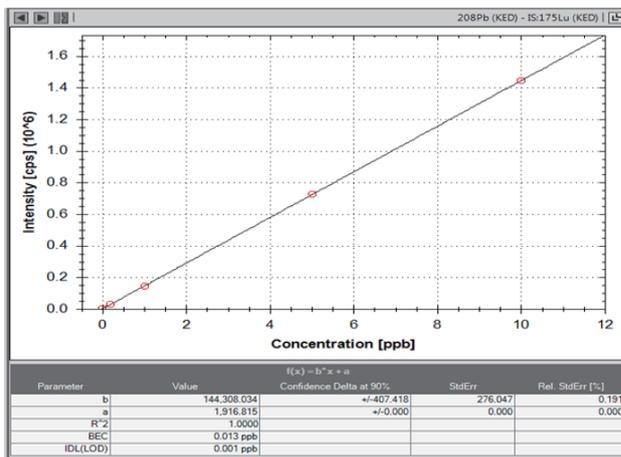
prepFAST在Qtegra軟體上的操作，僅需在Vial Numbers欄位中輸入空白溶液及高濃度標準品所擺放的位置，並在prepFAST DF 欄位中輸入1-200倍適當的稀釋倍數，即可輕鬆完成檢量線配製的設定，如圖九所示。圖十為人工配置10 ppb的Pb標準品，並由prepFAST自動配置且分析繪成0、0.5、1、5、10 ppb的檢量線，以及圖十一人工配置100 ppt的In標準品，由prepFAST自動配置且分析繪成0、2、5、10、50、100 ppt的檢量線，從圖中可看出prepFAST自動配製成的檢量線，其線性(R²)皆趨近為1，顯示出其具有高度的準確性。prepFAST自動配置檢量線的功能只需要準備一種高濃度的標準品，並且具有人性化的操作介面，可大幅降分析時耗費的人力與時間等成本，並且建立的檢量線具有高度的準確性與穩定性，將能成為分析實驗室不可或缺的設備。



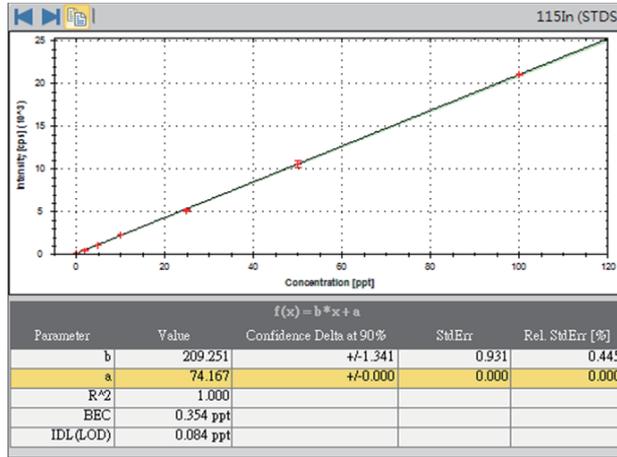
圖八：傳統手動配製檢量線與透過prepFAST自動配置檢量線之比較

| Label | Main Runs | Sample Type | Standard | Rack Number | Vial Numbers | prepFAST DF |
|-----------------------|-----------|-------------|---------------|-------------|--------------|-------------|
| 1 Stabilization Blank | 3 | UNKNOWN | | | 0 | 1 |
| 2 Cal Blank | 3 | BLK | | | 0 | 1 |
| 3 0.05 ppb | 3 | STD | 10 ppb Enviro | 0 | 2 | 200 |
| 4 0.1 ppb | 3 | STD | 10 ppb Enviro | 0 | 2 | 100 |
| 5 0.2 ppb | 3 | STD | 10 ppb Enviro | 0 | 2 | 50 |
| 6 0.5 ppb | 3 | STD | 10 ppb Enviro | 0 | 2 | 20 |
| 7 1 ppb | 3 | STD | 10 ppb Enviro | 0 | 2 | 10 |
| 8 2 ppb | 3 | STD | 10 ppb Enviro | 0 | 2 | 5 |
| 9 5 ppb | 3 | STD | 10 ppb Enviro | 0 | 2 | 2 |
| 10 10 ppb | 3 | STD | 10 ppb Enviro | 0 | 2 | 1 |

圖九：於Qtegra軟體上設定prepFAST自動配製檢量線



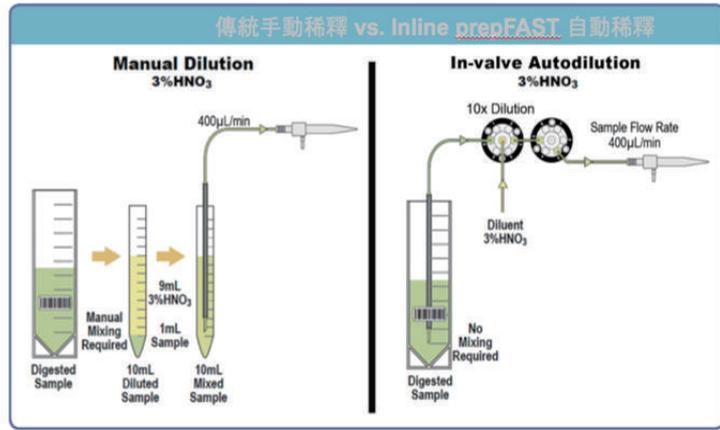
圖十：208 Pb 檢量線線性，從10 ppb標準品自動配製，檢量線範圍0, 0.5, 1, 5, 10 ppb



圖十一：115 In檢量線線性，從100 ppt標準品自動配製，檢量線範圍0, 2, 5, 10, 50, 100 ppt

prepFAST自動樣品稀釋

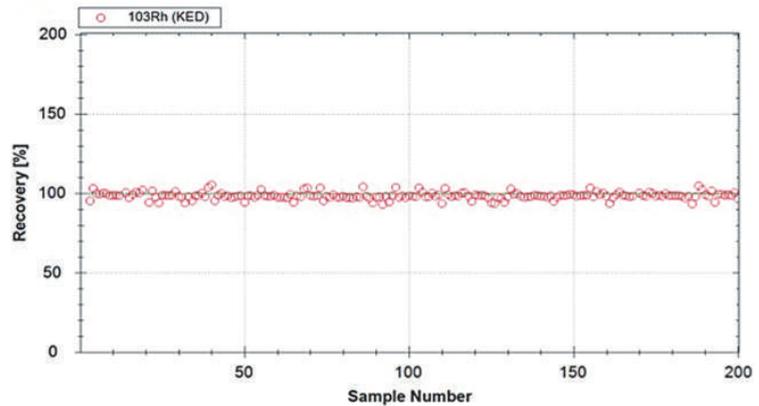
樣品上機前通常需要經過適當的稀釋才能進行分析，或是經微波消化的樣品額外稀釋10-100倍，使樣品基質與檢量線基質有所匹配。如圖十二所示，傳統人工稀釋時，需依賴操作者依序將樣品稀釋成適當倍數，耗費人力、時間與分析成本，而透過prepFAST使用者僅需將待稀釋之樣品放置於prepFAST自動取樣器上，並於Qtetra軟體中輸入欲稀釋的倍數，即可自動完成樣品的稀釋及分析。



圖十二：傳統手動稀釋樣品與prepFAST自動稀釋樣品的比較

prepFAST自動添加內標元素

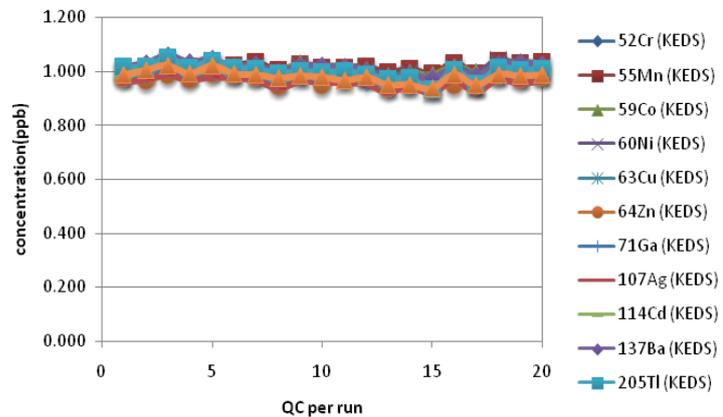
透過prepFAST自動化系統，可設定自動添加一定量的內標元素於檢量線及樣品中，如圖十三，測量200個樣品並添加內標103Rh，其內標回收率皆落在100%的範圍內，證明prepFAST添加內標有良好的穩定性及準確度。



圖十三：長時間測量200個樣品的內標穩定性

prepFAST重複稀釋測試

實際測試prepFAST執行自動稀釋的穩定性，配置10 ppb的標準品與空白溶液，設定自動稀釋成1 ppb並重複測量20次，分析結果如圖十四，顯示分析數據均落在1 ppb的範圍，證實利用prepFAST稀釋樣品具有優良的穩定性及準確性。



圖十四：prepFAST從10 ppb標準品自動配製成1 ppb重複分析20次

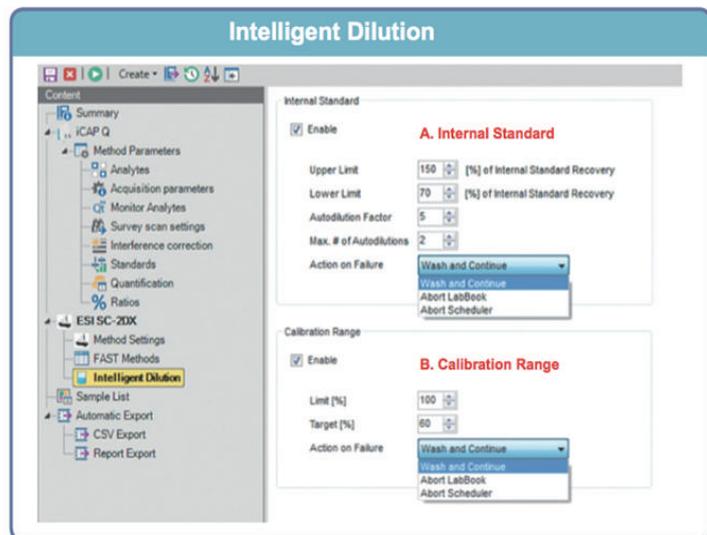
prepFAST智能自動稀釋

ESI prepFAST系統可與Thermo的儀器有良好的連結，不需額外安裝軟體，使用Thermo Qtetra軟體即可輕易的操控prepFAST系統。

透過Qtetra的ISDS(Intelligent Scientific Data Solution)智能資料處理系統，可根據樣品實際分析數據進行判讀並自動執行樣品稀釋並重新分析，Qtetra中智能稀釋的設定畫面如圖十五，設定自動稀釋的參數包含了下列兩項影響分析確效的因素：

A. Internal Standard 內標超過規範：

於Qtetra軟體中，可依照現行規範設定內標回收率的上限值與下限值，如圖十五，設定樣品內標回收率介於70~150%之間，因此當樣品分析時，其內標回收率超過所設定的範圍，prepFAST會自動依所設定的稀釋倍率，重新稀釋樣品並再次執行分析，實際執行數據如圖十六所示。



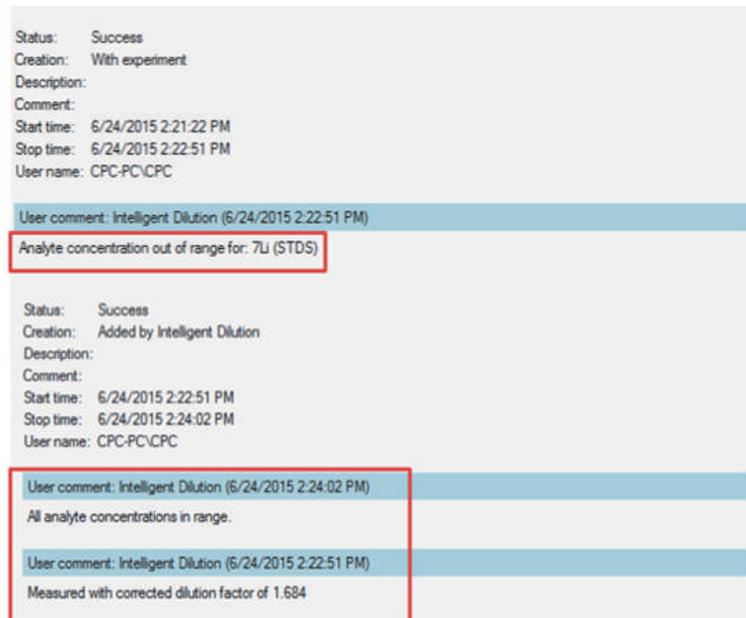
圖十五：Qtetra軟體中智能稀釋功能，分成A. Internal Standard 及 B. Calibration Range

| | Comment | Evaluate | Sample Type | Dilution Factor | Total Dilution Factor | Rock Number | Val Numbers | prepFASTDF |
|---|-----------|----------|-------------|-----------------|-----------------------|-------------|-------------|------------|
| 3 | <Comment> | ✓ | UNKNOWN | 1 | 1 | 0 | 1 | 1 |
| 3 | <Comment> | ✓ | BLK | 1 | 1 | 0 | 1 | 1 |
| 3 | 0.1 | ✓ | STD | 10 ppb | 100 | 0 | 2 | 100 |
| 3 | 0.5 | ✓ | STD | 10 ppb | 20 | 0 | 2 | 20 |
| 3 | 1 | ✓ | STD | 10 ppb | 10 | 0 | 2 | 10 |
| 3 | 2 | ✓ | STD | 10 ppb | 5 | 0 | 2 | 5 |
| 3 | 5 | ✓ | STD | 10 ppb | 2 | 0 | 2 | 2 |
| 3 | 10 | ✓ | STD | 10 ppb | 1 | 0 | 2 | 1 |
| 3 | <Comment> | ✓ | UNKNOWN | 1 | 10 | 0 | 2 | 10 |
| 3 | <Comment> | ✓ | UNKNOWN | 1 | 1 | 0 | 2 | 1 |
| 3 | <Comment> | ✓ | UNKNOWN | 1 | 1.684 | 0 | 2 | 1.684 |
| 3 | <Comment> | ✓ | UNKNOWN | 1 | 200 | 0 | 2 | 200 |
| 3 | <Comment> | ✓ | UNKNOWN | 1 | 341.781 | 0 | 2 | 341.781 |
| 3 | <Comment> | ✓ | UNKNOWN | 1 | 200 | 0 | 2 | 200 |
| 3 | <Comment> | ✓ | UNKNOWN | 1 | 10 | 0 | 2 | 10 |
| 3 | <Comment> | ✓ | UNKNOWN | 1 | 150 | 0 | 2 | 150 |

圖十六：內標回收率超過規範而自動執行樣品稀釋與分析。

B. Calibration Range 樣品濃度超過檢量線範圍

分析未知樣品時常會發生樣品濃度超過檢量線線性範圍的狀況，透過Qtegra的ISDS會自動計算適當的稀釋倍率，並自動重新稀釋及分析樣品，使樣品再次分析的濃度能落在檢量線範圍中，Qtegra的ISDS執行的畫面如圖十七所示。



圖十七：樣品分析濃度超過檢量線範圍，Qtegra軟體自動計算出適合的稀釋倍率，並自動執行稀釋與分析。

結 論

ESI prepFAST 自動化系統，有著基本高樣品通量、快速取樣及快速清洗的功能，並可自動配製檢量線，自動稀釋樣品，有良好的穩定性、準確性及精密性。ESI prepFAST與Thermo iCAP Q及 iCAP 7400可完美連結，透過Qtegra軟體即可輕鬆操作，Qtegra ISDS還具備智能稀釋樣品的功能，當樣品超過檢量線範圍或內標回收率超出規範時，可自動計算合適的稀釋倍率，並重新稀釋後再分析，將可有效地提升樣品分析的效率，降低實驗室成本，是現代化實驗室分析上不可或缺的好幫手。

Thermo Scientific iCAP 7000 Series ICP-OES

在食品添加劑分析上的應用

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食品添加劑
Food Additive

食品業內人士常說一句話：「食品添加劑是食品工業的靈魂。」那食品添加劑到底是什麼？食品添加劑可用來改善食品的色、香、味，以及因防腐、保鮮和加工工藝的需求而加入食品中的人工合成或者天然物質，例如糕點中的防腐劑、酸奶裏的增稠劑等，一種食品中往往就摻雜四五種以上的添加劑。各大食品企業的研發部門都在竭盡所能尋找最合理的配方，既滿足工藝需要、符合消費者的口味又能控制成本，而食品添加劑正是能實現這些目的的重要因素。我們日常生

活中早以攝入大量的添加劑而不自知，既然無法避免攝入，而檢視其安全性就是一個重要的課題。目前市面上最普遍的添加劑應當就屬食用色素了，例如糖果、餅乾、飲料...等，在這些食品裡面添加食用色素能為食品增添鮮豔的色彩，那「食用色素」又是什麼呢？

「食用色素」簡單來說，就是食物的著色材料，可以讓食物顏色更鮮豔、豐富，不僅能提升食用者的食慾，還可以增添感官享受。食用色素可以分為「人工」和「天然」2大類別，天然食用色素的來源非常廣泛，包括植物色素、動物色素、維生素色素和焦糖色素等，透過物理方法提取精製而成，然而卻因此產生較高的製造成本。目前法定的人工色素有八種：藍色1號、藍色2號、綠色3號、黃色4號、黃色5號、紅色6號、紅色7號與紅色40號，在食品添加物規範中屬於第(九)類-著色劑，可於各類食品中適量使用，但禁止於生鮮肉類、生鮮魚貝類、生鮮豆類、生鮮蔬菜、生鮮水果、味噌、醬油、海帶、海苔、茶等。本文利用Thermo Scientific iCAP 7000 Series ICP-OES分析合法的人工食用色素中金屬元素的含量是否符合法規標準。

儀器設定與實驗方法

1. 儀器設定

本實驗使用Thermo Scientific iCAP 7000 Series ICP-OES進行分析，詳細的儀器進樣選擇與參數設定如表A。

表A、儀器參數設定

| Instrument | | |
|---------------------------|--------------------|-----|
| Nebulizer | Aerosalt Nebulizer | |
| Spray Chamber | Quartz | |
| Centre Tube | Ceramic 2mm ID | |
| Torch | Duo Torch | |
| Parameter | | |
| Exposure Time(s) | UV | VIS |
| | 15 | 5 |
| View Modes | Axial | |
| RF Power(W) | 1150 | |
| Nebulizer Gas Flow(L/min) | 0.5 | |
| Auxiliary Gas Flow(L/min) | 0.5 | |
| Coolant Gas Flow(L/min) | 12 | |
| Pump Speed(RPM) | 50 | |



2. 實驗方法

本次實驗分析元素除了法規規範之Fe、Cr、Zn三種元素外，尚分析Ag、Al、As、Bi、Cd、Cu、Hg、Mo、Ni、Pb、Sb和Sn總共十五個元素。本次實驗的檢量線濃度為0、50、100、200、500、1000和2000 ppb(註:只有鐵到2000 ppb)，另外As、Hg的檢量線濃度則為0、5、10、20、50和100 ppb。分析樣品的過程中，同時以Y型管導入1 ppm Y，作為修飾基質的內標準品。

3. 樣品前處理

本實驗以合法的人工色素為樣品，選用食用藍色1號(B1)、食用藍色2號(B2)、食用黃色4號(Y4)、食用黃色5號(Y5)、食用紅色6號(R6)、食用紅色7號(R7)及食用紅色40號(R40)等七件樣品。精稱0.5 g 色素樣品於消化瓶中以超純水溶解後定量至25 mL；另取1消化瓶，加入25 mL超純水作為方法空白。

實驗數據及結果

1. 檢量線線性與方法偵測極限

本實驗檢量線線性R²值皆大於0.999，具有良好的線性。儀器偵測極限(IDL)乘上稀釋倍率(50倍)即為方法偵測極限(MDL)，其檢量線線性迴歸、方法偵測極限(MDL)、背景值(BEC)如表B所示。

表B、檢量線線性及方法偵測極限和背景值

| Element | 線性迴歸(R ²) | BEC(ppb) | MDL(ppb) |
|---------|-----------------------|----------|----------|
| Ag | 0.9991 | 3.378 | 75.90 |
| Al | 1.0000 | 5.646 | 100.7 |
| As | 0.9996 | -1.291 | 134.2 |
| Bi | 0.9999 | 0.430 | 260.1 |
| Cd | 0.9999 | 0.042 | 13.70 |
| Cr | 1.0000 | 0.153 | 41.55 |
| Cu | 1.0000 | -0.0003 | 53.25 |
| Fe | 1.0000 | 0.933 | 38.70 |
| Hg | 1.0000 | 0.557 | 32.75 |
| Mo | 1.0000 | 0.220 | 35.25 |
| Ni | 0.9999 | -0.400 | 36.00 |
| Pb | 0.9999 | 3.520 | 148.8 |
| Sb | 1.0000 | 1.466 | 146.3 |
| Sn | 1.0000 | 0.565 | 34.45 |
| Zn | 1.0000 | 0.141 | 35.15 |

2. 樣品分析濃度

本實驗分析Ag、Al、As、Bi、Cd、Cr、Cu、Fe、Hg、Mo、Ni、Pb、Sb、Sn、Zn等十五個元素，除了Al、As、Cr、Cu、Fe、Ni、Sn、Zn高於IDL外，其餘元素都在IDL以下。ICP-OES具有相當低的偵測極限，足以偵測出樣品中重金屬的濃度，樣品分析濃度詳見表C。

表C、樣品分析濃度

| Element | IDL | QC STD-3 | MBK | B1 | B2 | Y4 | Y5 | R6 | R7 | R40 |
|------------|-------|-------------|-----|-------|-------|-------|-------|-------|-------|-------|
| Unit : ppb | | | | | | | | | | |
| Ag | 1.518 | 216.9 | <DL | <DL | <DL | <DL | <DL | <DL | <DL | <DL |
| Al | 2.014 | 197.7 | <DL | 21.77 | 140.1 | 13.98 | 8.833 | 56.57 | <DL | 222.1 |
| As | 2.684 | 20.26 | <DL | 6.204 | 2.934 | 3.256 | 3.148 | 5.251 | 3.595 | <DL |
| Bi | 5.202 | 202.7 | <DL | <DL | <DL | <DL | <DL | <DL | <DL | <DL |
| Cd | 0.274 | 202.0 | <DL | <DL | <DL | <DL | <DL | <DL | <DL | <DL |
| Cr | 0.831 | 201.9 | <DL | <DL | 164.1 | 2.396 | 7.645 | 3.599 | <DL | <DL |
| Cu | 1.065 | 201.7 | <DL | 1.313 | 3.377 | <DL | 2.000 | 6.086 | <DL | 2.937 |
| Fe | 0.774 | 201.7 | <DL | 89.33 | 763.3 | 139.7 | 144.5 | 187.2 | 19.13 | 33.29 |
| Hg | 0.655 | 19.49 | <DL | <DL | <DL | <DL | <DL | <DL | <DL | <DL |
| Mo | 0.705 | 199.8 | <DL | <DL | <DL | <DL | <DL | <DL | <DL | <DL |
| Ni | 0.720 | 202.3 | <DL | <DL | 7.717 | 1.397 | 8.689 | 5.738 | <DL | 2.603 |
| Pb | 2.975 | 205.1 | <DL | <DL | <DL | <DL | <DL | <DL | <DL | <DL |
| Sb | 2.925 | 199.1 | <DL | <DL | <DL | <DL | <DL | <DL | <DL | <DL |
| Sn | 0.689 | 200.1 | <DL | 6.189 | 4.224 | 3.464 | 4.507 | 3.529 | 3.220 | 4.277 |
| Zn | 0.703 | 202.0 | <DL | 0.794 | 12.27 | 6.658 | 2.653 | 4.091 | 4.615 | 17.25 |

3. 樣品實際濃度

表D為樣品實際濃度以及其法規限量。Ag、Bi、Cd、Hg、Mo、Pb、Sb等元素皆小於MDL，另外Al、As、Cr、Cu、Fe、Ni、Sn、Zn雖有檢出，結果也在法規值內。

表D、樣品實際濃度

| Element | 藍色一號 | | 藍色二號 | | 黃色四號 | | 黃色五號 | | 紅色六號 | | 紅色七號 | | 紅色四十號 | |
|------------|-------|------|-------|------|-------|------|-------|------|-------|------|-------|------|-------|------|
| | 樣品含量 | 法規限量 |
| Unit : ppm | | | | | | | | | | | | | | |
| Ag | <MDL | - |
| Al | 1.084 | - | 6.974 | - | 0.693 | - | 0.437 | - | 2.757 | - | <MDL | - | 11.01 | - |
| As | 0.309 | 2 | 0.146 | 2 | 0.161 | 2 | 0.156 | 2 | 0.256 | 2 | 0.178 | 2 | <MDL | 3 |
| Bi | <MDL | - |
| Cd | <MDL | - |
| Cr | <MDL | 50 | 8.169 | 25 | 0.119 | 25 | 0.378 | - | 0.175 | 25 | <MDL | 25 | <MDL | - |
| Cu | 0.065 | - | 0.168 | - | <MDL | - | 0.099 | - | 0.297 | - | <MDL | - | 0.146 | - |
| Fe | 4.449 | 500 | 37.99 | 1000 | 6.927 | 500 | 7.140 | - | 9.125 | 500 | 0.950 | 500 | 1.650 | - |
| Hg | <MDL | - |
| Mo | <MDL | - |
| Ni | <MDL | - | 0.384 | - | 0.069 | - | 0.429 | - | 0.280 | - | <MDL | - | 0.129 | - |
| Pb | <MDL | - | <MDL | - | <MDL | - | <MDL | 2 | <MDL | - | <MDL | - | <MDL | 2 |
| Sb | <MDL | - |
| Sn | 0.308 | - | 0.210 | - | 0.172 | - | 0.223 | - | 0.172 | - | 0.160 | - | 0.212 | - |
| Zn | 0.040 | 200 | 0.611 | 200 | 0.330 | 200 | 0.131 | - | 0.199 | 200 | 0.229 | 200 | 0.855 | - |
| *Total | 0.683 | 20 | 0.524 | 20 | 0.333 | 20 | 0.477 | - | 0.724 | 20 | 0.338 | 20 | 0.358 | - |

*註：Total(比色法以鉛計)Ag、Pb、As、Cu、Cd、Hg、Mo、Bi、Sn和Sb共10個元素加總值

表E、樣品回收率測試

| Element | Y4 (ppb) | *Y4 +200ppb/20ppb | Recovery (%) |
|---------|----------|-------------------|--------------|
| Ag | <DL | 187.0 | 94 |
| Al | 13.98 | 210.2 | 98 |
| As | 3.256 | 23.66 | 102 |
| Bi | 2.522 | 183.4 | 90 |
| Cd | 0.109 | 185.9 | 93 |
| Cr | 2.396 | 203.4 | 101 |
| Cu | 0.628 | 211.5 | 105 |
| Fe | 139.7 | 337.9 | 99 |
| Hg | <DL | 17.84 | 92 |
| Mo | <DL | 197.1 | 99 |
| Ni | 1.397 | 185.2 | 92 |
| Pb | <DL | 182.1 | 91 |
| Sb | <DL | 193.3 | 98 |
| Sn | 3.464 | 186.0 | 91 |
| Zn | 6.658 | 196.7 | 95 |

*註：以食用黃色4號樣品消化液添加200 ppb Ag、Al、Bi、Cd、Cr、Cu、Fe、Mo、Ni、Pb、Sb、Sn、Zn，As、Hg添加20 ppb

4. 樣品添加回收率測試

樣品分析時需做回收率測試，藉此確認分析結果之加成性。本次實驗以食用黃色4號樣品消化液添加200 ppb(As、Hg添加20 ppb)濃度進行樣品添加回收率測試，分析結果顯示各元素回收率介於90~110%之間，表示此次分析方法具有實驗加成性，詳見表E。

結 論

以Thermo Scientific iCAP 7000 Series ICP-OES分析重金屬元素，只需要一個方法便能分析多種元素，且Thermo ICP-OES具有世界上最小的光室設計，在分析167~200 nm的元素波長擁有良好的儀器感度及最低之偵測極限，此外還有線性範圍廣的特性，不同元素其高低濃度可一次分析完成。

由實驗結果可知，這七種人工食用色素中金屬元素含量相當低，並皆符合法規規範。雖然食物添加人工色素，看起來真的很「吸睛」，好吃度瞬間提升，不過最怕不肖商人添加其他非法食用色素，如此一來安全性就會大打折扣！

利用HPLC-ICP-OES測定洛克沙砷及其代謝物

應用工程師 黃盟順 張僑宏

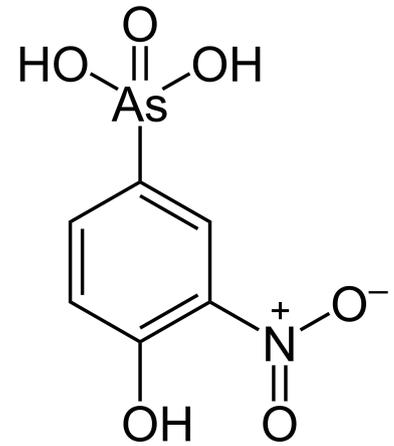
Email: mountainhuang@joytech.com.tw / simon@joytech.com.tw



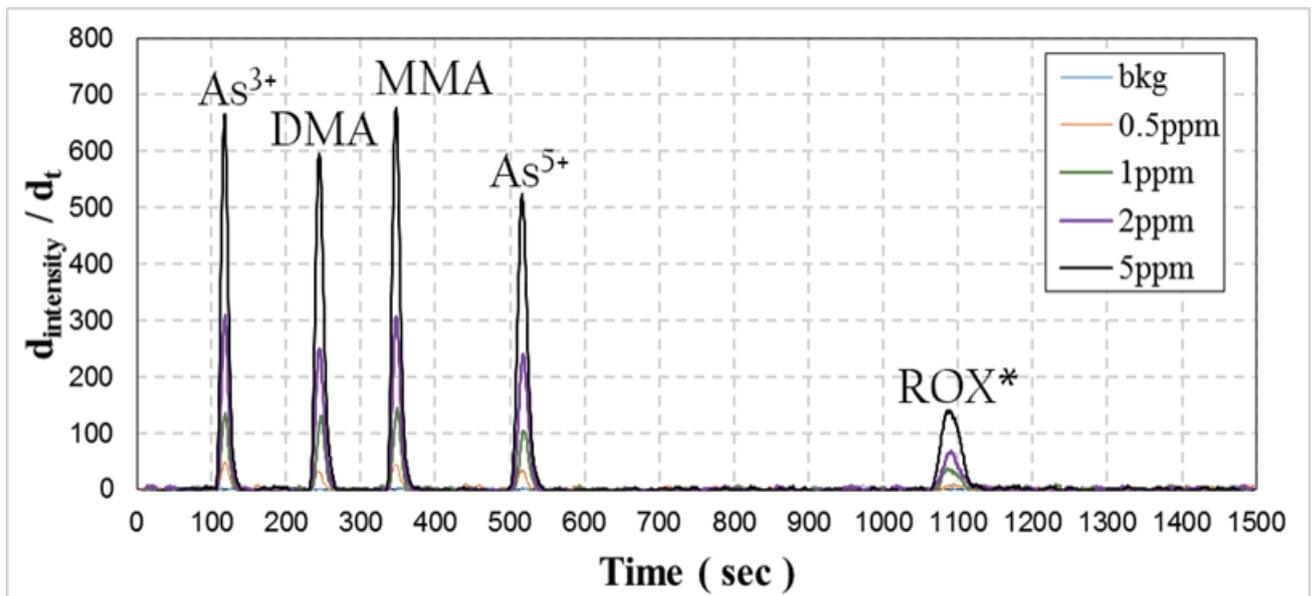
洛克沙砷 (Roxarsone, ROX) 常被用來做為畜禽飼料的添加劑之一，主要是因為其被認為是一種低毒性的殺菌劑及生長促進劑。雖然畜禽對於ROX的吸收性很低，且大部分會以原始形態隨糞便或尿液排出。但ROX進入環境後，經過轉化及降解等作用後，便會形成其他型態的砷化合物，如：無機砷 (As^{3+} 與 As^{5+})、單甲基砷酸 (monomethylarsonic acid, MMA)、二甲基砷酸 (dimethylarsinic acid, DMA) 等。傳統上認為，砷在生物體內的毒性為 $As^{3+} > As^{5+} > 有機砷$ 。此外，亦有研究指出DMA和MMA可能會引起動物及人類器官的腫瘤發生。

目前針對不同型態砷化合物的檢測方法有液相層析法 (LC)、氣相層析質譜法 (GC-MS)、液相層析質譜法 (LC-MS) 及液相層析串連感應耦合電漿質譜分析儀 (ICP-MS) 或光譜儀 (ICP-OES)，其中又以LC-ICP-MS最為常見。一般來說，ICP-OES的感度不及ICP-MS，且利用OES來分析砷亦有感度較差的問題存在。但本次實驗中，我們將使用LC-ICP-OES，嘗試建立一個 As^{3+} 、 As^{5+} 、DMA、MMA及ROX等5種砷化合物的分析方法。

圖二為 As^{3+} 、 As^{5+} 、DMA、MMA及ROX等5種砷化合物的層析圖譜，使用之波長為189.0 nm。此外，儀器偵測極限以空白溶液重覆分析3次，計算其3倍之標準偏差濃度，即為儀器偵測極限 (IDL)。 As^{3+} 、DMA、MMA、 As^{5+} 及ROX之IDL分別為0.05、0.02、0.1、0.01及0.09 ppm。



圖一、洛克沙砷結構式。



圖二、5種砷化合物之層析圖譜，其中ROX之濃度分別為0.5、1、2及5 ppm。

利用Thermo Scientific iCAP Qc ICP-MS 分析NIST SRM 標準品驗證食品五重檢測實驗方法

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近年食品安全問題事件層出不窮，像是違法添加三聚氫胺、塑化劑、農藥殘留或重金屬殘留等問題，造成民眾人心惶惶，使得食品安全與食品檢驗格外受到重視。一般容易累積於食物鍊且具有毒性的元素包括砷、鎘、汞、鉛及銅，其中銅雖然為生物體中的必需微量元素，但當攝取過量還是會對人體造成危害，而砷、鎘、汞和鉛為易累積於生物體且毒性非常強的元素，例如攝取受到鎘污染的食物，會造成骨骼的病變，引起骨軟化、骨質疏鬆、甚至是癱瘓或死亡。因此，食品中重金屬的檢測已成為食品安全相當重要的一環。衛福部依據各種不同食品規定有害元素的檢出限量，而檢測方法必須遵循相關規範，回收率測試即是其中一種間接確認分析方法是否可信的方式之一，而最具公信力的方式便是向NIST (National Institute of Standards and Technology)購買類似樣品基質的標準參考物質來分析，以驗證實驗方法的準確與否。本篇利用Thermo Scientific iCAP Q ICP-MS感應耦合電漿質譜儀，分析NIST SRM1515蘋果葉及SRM 1568b米粉標準物質，依此兩種標準物質的COA比對實際分析結果，驗證食品中五大重金屬檢測方法的準確性。

儀器設定與實驗分析方法

1. 儀器設定

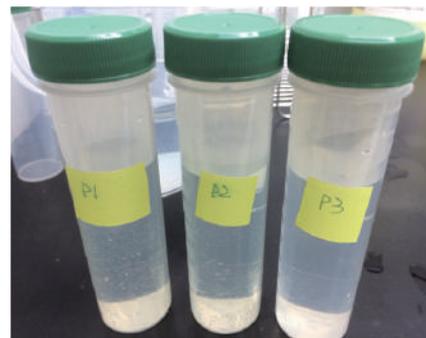
本實驗使用的儀器為Thermo Scientific iCAP Q ICP-MS，可分析質量範圍從4到250amu。因前處理時使用少許HF為消化溶劑，所以進樣系統則選用抗氫氟酸之藍寶石中心管，並採用PFA霧化室及霧化器。儀器具備碰撞氣體的模式，可利用單一氣體(氬氣)有效地去除干擾，並且同時保有測定時的高靈敏度，利用單一分析模式(KED)即可完成多種元素的分析。詳細的儀器進樣系統選擇及儀器參數設定如表一。

表一、儀器進樣系統及參數設定

| Introduction Components | |
|--------------------------------|---------------------------------|
| Spray Chamber | PFA Cyclonic Spray Chamber |
| Injector | 2.0 mm Sapphire Injector |
| Sample Cone | Platinum (Pt) |
| Skimmer Cone | Platinum (Pt) |
| Nebulizer | PFA-ST-400 Micro-flow Nebulizer |
| Instrumental Parameters | |
| Operation Mode | KED |
| RF Power | 1550W |
| Nebulizer Flow (L/min) | 0.88 |
| CCT Gas Flow (He) (mL/min) | 4.721 |
| Spray Chamber Temperature (°C) | 2.7 |

2. 樣品前處理方法

本實驗使用NIST購買之SRM標準物質，分別為SRM1515 Apple Leaves 及 SRM 1568b Rice Flour。精秤0.2 g烘乾SRM標準物質各4瓶，分別加入6 mL 70% HNO₃及1 mL 49% HF，並且加入40 uL 100 ppm Au，鎖緊瓶蓋，放入微波消化器中消化，待消化完成後，冷卻至室溫，再以DIW定量至40 mL，上機時再稀釋2.5倍進行分析，標準品總稀釋倍率為500倍。



圖一、樣品經微波消化並定量後實際圖

3. 檢量線配製

檢量線配製需與樣品上機基質匹配，因此本實驗以6% HNO₃及1% HF為檢量線基質，配製檢量線範圍如表二，並添加1 ppb Rh & Bi當作內標修正。

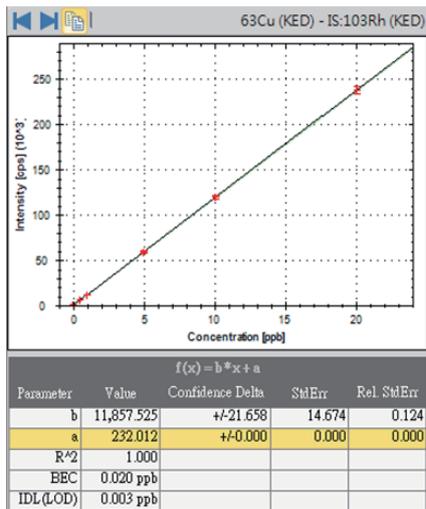
表二、Cu、As、Cd、Hg、Pb檢量線範圍

| Elements | Unit | STD1 | STD2 | STD3 | STD4 | STD5 |
|--------------|------|--------|-------|-------|------|------|
| As / Cd / Pb | ppb | 0.05 | 0.1 | 0.5 | 1 | 2 |
| Cu | ppb | 0.5 | 1.0 | 5.0 | 10 | 20 |
| Hg | ppb | 0.0125 | 0.025 | 0.125 | 0.25 | 0.5 |

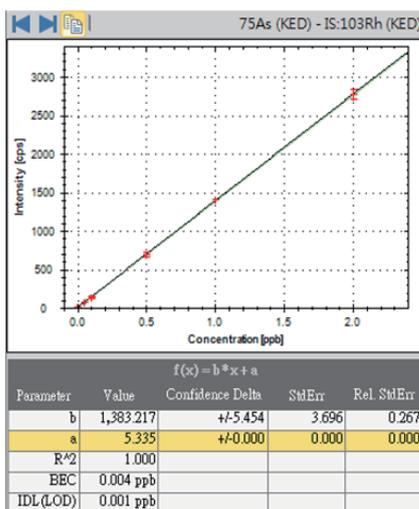
實驗數據及結果

1. 檢量線線性

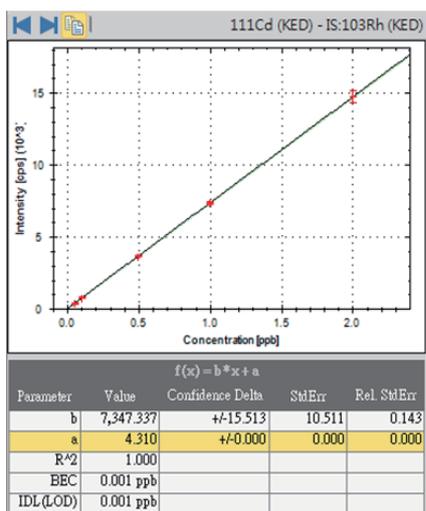
本實驗以單一模式(KED)分析食品中常需檢測的五大重金屬含量，所得到的檢量線線性皆大於0.999，具有良好的線性，Cu、As、Pb、Cd和Hg檢量線圖顯示於圖二至圖六。



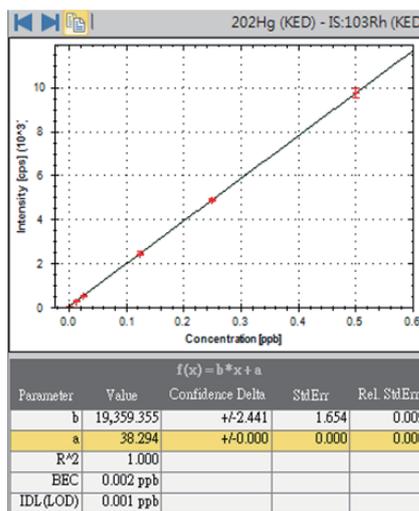
圖二、Cu Calibration (R2>0.999)



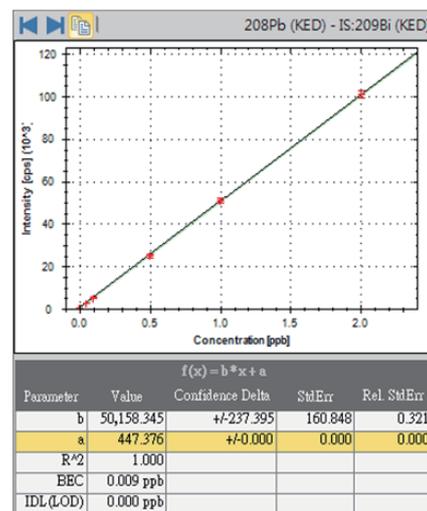
圖三、As Calibration (R2>0.999)



圖四、Cd Calibration (R2>0.999)



圖五、Hg Calibration (R2>0.999)



圖六、Pb Calibration (R2>0.999)

2. 偵測極限

偵測極限以空白溶液(6% HNO₃ + 1% HF)重複分析7次，計算其3倍之標準偏差，得知其儀器偵測極限(IDL)皆小於等於20 ppt，有良好的偵測極限，乘上稀釋倍數500倍後，即可得方法偵測極限(MDL)，個元素的IDL與MDL如表三所示。

表三、各元素儀器與方法偵測極限值

| Elements | Unit | BEC | IDL | MDL |
|----------|------|-------|-------|-------|
| Cu | ppb | 0.020 | 0.003 | 1.312 |
| As | ppb | 0.004 | 0.001 | 0.650 |
| Cd | ppb | 0.001 | 0.001 | 0.315 |
| Hg | ppb | 0.002 | 0.001 | 0.628 |
| Pb | ppb | 0.009 | 0.000 | 0.154 |

3. 樣品實際濃度

表四為本次實驗所分析之米粉標準物質SRM1568b之結果，表五為分析蘋果葉標準物質SRM1515之結果，米粉測量Cu、As和Cd三種元素，蘋果葉測量Cu、Cd、Hg和Pb四種元素，其結果皆有落在SRM的認證值範圍內，顯示本實驗方法具有相當的準確性。

表四、NIST 1568b-Rice Flour 實際分析結果

| Element | 1568b-1 | 1568b-2 | 1568b-3 | 1568b-4 | Average | RSD (%) | Measured | Certified |
|----------------------|---------|---------|---------|---------|---------|---------|----------------------|---------------|
| -----Unit : ppb----- | | | | | | | -----Unit : ppm----- | |
| Cu | 2283.32 | 2281.23 | 2262.50 | 2247.30 | 2268.59 | 0.75 | 2.269 | 2.35±0.16 |
| As | 278.97 | 279.75 | 272.84 | 283.96 | 278.88 | 1.64 | 0.279 | 0.285±0.014 |
| Cd | 21.84 | 21.21 | 22.31 | 22.17 | 21.88 | 2.24 | 0.022 | 0.0224±0.0013 |

表五、NIST 1515-Apple Leaves 實際分析結果

| Element | 1515-1 | 1515-2 | 1515-3 | 1515-4 | Average | RSD(%) | Measured | Certified |
|----------------------|---------|---------|---------|---------|---------|--------|----------------------|-------------|
| -----Unit : ppb----- | | | | | | | -----Unit : ppm----- | |
| Cu | 5694.99 | 5760.88 | 5543.73 | 5704.29 | 5675.97 | 1.64 | 5.676 | 5.64±0.24 |
| Cd | 12.86 | 13.19 | 14.18 | 14.05 | 13.57 | 4.77 | 0.014 | 0.013±0.002 |
| Hg | 42.09 | 41.08 | 43.97 | 45.35 | 43.12 | 4.43 | 0.043 | 0.044±0.004 |
| Pb | 485.81 | 457.83 | 467.21 | 458.66 | 467.38 | 2.78 | 0.467 | 0.470±0.024 |

4. 樣品添加回收率

表六為米粉標準物質添加STD4的回收率測試，表七為蘋果葉標準物質添加STD4的回收率測試，所有元素之回收率皆介於90~110%之間，顯示此分析方法皆有良好的加成性。

表六、NIST 1568b-Rice Flour 添加回收率測試

| Elements | Unit | STD4 | SRM1568b | SRM1568b+spike | Recovery(%) |
|----------|------|------|----------|----------------|-------------|
| Cu | ppb | 5 | 4.585 | 9.360 | 95.5 |
| As | ppb | 0.5 | 0.560 | 1.047 | 97.4 |
| Cd | ppb | 0.5 | 0.044 | 0.530 | 97.3 |

表七、NIST 1515-Apple Leaves 添加回收率測試

| Elements | Unit | STD4 | SRM1515 | SRM1515+spike | Recovery(%) |
|----------|------|-------|---------|---------------|-------------|
| Cu | ppb | 5 | 11.568 | 16.621 | 101.1 |
| Cd | ppb | 0.5 | 0.026 | 0.541 | 103.0 |
| Hg | ppb | 0.125 | 0.082 | 0.219 | 109.1 |
| Pb | ppb | 0.5 | 0.919 | 1.463 | 108.8 |

結 論

利用Thermo Scientific iCAP Q ICP-MS以單一分析模式(KED)，不但有良好去除干擾物質的效果，並且可快速準確的分析五大重金屬的含量，分析米粉與蘋果葉標準物質中五大重金屬的含量，結果皆落在標示的範圍內，且回收率皆在90-110%，驗證依照此方法來分析食品中的重金屬含量有良好的準確性，實驗方法是可被信賴的。

樂盟科技2015年度研討會

樂盟科技2015年綜合應用研討會已於七月中旬分別在臺北、新竹及臺南三地圓滿結束。在今年的研討會中，我們特別以食品、藥品、環境分析，及可提升分析效率並降低人為分析污染的自動樣品配製系統為主題，期望能以貼近客戶需求的角度提供各位關於實驗分析上可能需要的最新資訊。

Thermo Fisher Scientific的Arthur van Strien為我們帶來樂盟科技代理Thermo Fisher Scientific原子吸收光譜儀（AA）、感應耦合電漿發射光譜儀（ICP-OES）、感應耦合電漿質譜儀（ICP-MS）、元素分析儀（EA）等產品線的最新訊息；國立臺灣海洋大學水產品產銷履歷驗證暨檢驗中心冉繁華主任以深入淺出的方式介紹ICP-MS應用於水產品中重金屬分析與食品安全的關係；SGS臺灣檢驗科技食品服務部萬乃容主任簡單介紹了穩定同位素分析技術於食品摻偽檢測上的潛在應用範疇與目前在臺灣市場的發展狀況；最後則由我們樂盟科技同仁介紹高效能液相層析系統（HPLC）及ESI prepFAST自動樣品配製系統等周邊配備與Thermo Fisher Scientific ICP-OES/ICP-MS產品結合的可能應用與樣品分析經驗分享。感謝各位的熱情參與，讓樂盟科技能與大家的實驗室一同成長。



與文化大學黃鵬林院長合影



研討會—台北場



研討會—新竹場



研討會—新竹場



研討會—台南場

活動花絮



樂盟科技國內旅遊—雷射鎗戰

Automated, Intelligent Sample Preparation: Integration of the ESI prepFAST Auto-Dilution System with the Thermo Scientific iCAP Q ICP-MS

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Key Words

Auto-Dilution, EPA 200.8, ICP-MS, USP <233>

Goal

To demonstrate the increase in and reduce manual tasks through automation of the analytical workflow. Through an integrated combination of intelligent software and hardware, demonstrate how auto-calibration and auto-dilution can simplify routine elemental analysis.

Introduction

Similar to most solution based techniques, elemental quantification by inductively coupled plasma mass spectrometry (ICP-MS) involves numerous dilution steps before the analytical run can commence.

A series of calibration standards have to be supplied at concentration levels designed to span the expected content in the unknowns. These standards are usually prepared by serial dilution from one or more stock standard solutions. Depending on the required calibration range, several dilutions between 10 to 200-fold are usually required. The majority of samples for elemental analysis by ICP-MS are supplied as solids that have to be first brought into solution, for example, by mineral acid digestion. Depending on the sample and digestion procedure required, the samples would then have to be diluted before analysis – usually between 10 to 100-fold.

During the analytical run however, samples that do not meet the requirements defined by laboratory standard operating procedures (SOPs) may have to be removed from the autosampler rack for additional dilution and subsequent repeat analysis. For example to:

- Confine measured concentrations to within the calibrated concentration range.
- Eliminate the effect of variable, sample dependent matrix suppression of the analytical signal.

While these dilution steps can be manually performed by a skilled laboratory technician it is both tedious and time consuming.



Figure 1. The Thermo Scientific iCAP Q ICP-MS with integrated ESI prepFAST Auto-dilution System.

The ESI prepFAST™ Auto-dilution System in combination with the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software offers the following capabilities for routine trace elemental analysis by the Thermo Scientific™ iCAP™ Q ICP-MS:

- Prescriptive dilution of a single stock standard to generate multiple standards for calibration curves directly from the autosampler rack.
- Per analysis prescriptive dilution of samples directly from the autosampler rack.
- Over calibration range results for samples or QC analyses trigger intelligent, automated dilution to restrict measured concentrations to within a defined range.
- Internal standard recoveries outside of defined limits trigger intelligent, automated dilution to eliminate manual reruns of unexpectedly high matrix samples.

These features allow for fully automated sample preparation and per-analysis data review. Large analysis batches can now be confidently processed without any supervision, freeing up laboratory personnel for other tasks.

prepFAST Operation

The prepFAST Auto-dilution System is based on dual FAST valves operated in combination with a bank of four syringe pumps (S1–S4) that provide improved precision and accuracy over peristaltic pumps. With flow rates of between 1 to 20,000 $\mu\text{L}\cdot\text{min}^{-1}$ the S2 (carrier) and S3 (diluent) syringes can perform dilutions in seconds while the S4 syringe adds internal standard at a constant rate. Speed of dilution is independent of dilution factor (1 to 400) ensuring exact injection timing for all solution regardless of dilution. The operation of the prepFAST is summarised in the 4 steps shown in Figure 2: 1) Vacuum loading of the loop, 2) syringe dilution (S2 & S3) and addition of internal standard (S4), 3) sample injection and 4) loop rinsing (S4).

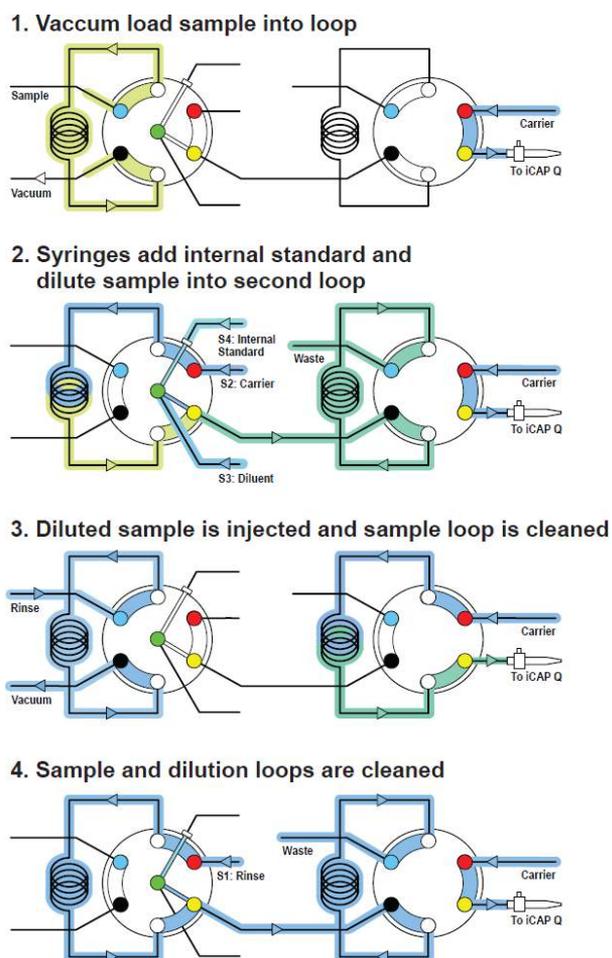


Figure 2. The 4 main steps of prepFAST operation, showing the combination of the 7 and 6-port FAST valves. The prepFAST Auto-dilution System is installed directly beneath the iCAP Q ICP-MS sample introduction system (Figure 1) to provide the shortest sample transfer distance reducing uptake delays to just a few seconds, thereby improving sample throughput.

Since the sample pathway is completely inert problems of cross sample contamination due to the use of peripump tubing are avoided. In the same way prepFAST applications are not limited to aqueous samples but can be applied to organic solvent based samples.

The ESI prepFAST Auto-dilution System provides important advantages for routine analysis with the iCAP Q ICP-MS:

- **High throughput:** Samples are vacuum loaded (~4s) into the prepFAST loop that is mounted directly beneath the sample introduction system. Sample uptake and washout delays are therefore significantly reduced, leading to tangible reductions in complete, sample to sample analysis times.
- **High purity:** Vacuum loading of samples through a fluoropolymer flow path onto the FAST loop is much cleaner than traditional transfer by peristaltic pump tubing, minimizing cross contamination and carry-over.
- **Inline sample preparation:** Internal standard addition and any subsequent dilution is performed inside the valve reducing contamination, enabling high linearity calibration curves for even the lowest concentrations.
- **Automation:** All dilutions are automated, eliminating any errors introduced by manual dilution.
- **Auto-dilution:** Dilution factors of up to 400-fold are reliably and accurately performed with all flows controlled by high precision syringe pumps.
- **Auto-calibration:** Calibration curves with high accuracy and linearity are effortlessly generated direct from a stock standard solution in the autosampler rack.
- **Auto-quality control (QC):** Samples can be individually diluted in position on the autosampler rack without being removed and re-added to/from the rack or analysis queue, eliminating any errors from manual sample handling or data entry.

Software Support of the prepFAST

All aspects of prepFAST Auto-dilution System operation are controlled by a dedicated Qtegra ISDS Software plug-in without having to use any external, secondary software. A series of FAST methods to address common sample handling applications are supplied; all are selectable and editable from within the Qtegra ISDS interface. By fully integrating prepFAST Auto-dilution System into the standard workflow, auto-dilution becomes as easy and routine to use as a standard autosampler.

Automated Calibration

The flexible prescriptive dilution capabilities of the prepFAST Auto-dilution System allow the analyst to choose the appropriate calibration strategy for each application.

- In the simplest approach a single stock standard can be used to create a complete multi-point calibration line (Figure 4).
- For methods that require sample specific calibration ranges (e.g. USP <233>), appropriate standard curves can be generated from a single stock.
- Multiple stocks can be used to create combined calibration curves over extended concentration ranges.
- Separate calibration curves for incompatible elements can be easily created from separate stock solutions.
- Since multiple standard vials are not required, contamination via atmospheric deposition (e.g. B from filters) is minimized.

All of these approaches are possible through the flexible definition of analysis specific prescriptive dilution factors in the Qtegra ISDS Software sample list.

In Figure 3, for example, a 10-point calibration requires a single autosampler rack and vial position, freeing up rack space for additional samples and QC analyses. Note the use of prescriptive dilution factors (prepFAST DF column entries) to define the calibration range.

| | Label | Sample Type | Standard | Rack | Vial | prepFAST DF |
|----|-----------|-------------|-------------|------|------|-------------|
| 1 | Blank | AVERAGE BLK | | 3 | 1 | 1 |
| 2 | Blank | AVERAGE BLK | | 3 | 1 | 1 |
| 3 | Blank | AVERAGE BLK | | 3 | 1 | 1 |
| 4 | 0.010 ppb | STD | 4 ppb Stock | 3 | 2 | 400 |
| 5 | 0.013 ppb | STD | 4 ppb Stock | 3 | 2 | 300 |
| 6 | 0.020 ppb | STD | 4 ppb Stock | 3 | 2 | 200 |
| 7 | 0.040 ppb | STD | 4 ppb Stock | 3 | 2 | 100 |
| 8 | 0.080 ppb | STD | 4 ppb Stock | 3 | 2 | 50 |
| 9 | 0.160 ppb | STD | 4 ppb Stock | 3 | 2 | 25 |
| 10 | 0.4 ppb | STD | 4 ppb Stock | 3 | 2 | 10 |
| 11 | 0.8 ppb | STD | 4 ppb Stock | 3 | 2 | 5 |
| 12 | 2 ppb | STD | 4 ppb Stock | 3 | 2 | 2 |
| 13 | 4 ppb | STD | 4 ppb Stock | 3 | 2 | 1 |

Figure 3. Generation of a 10-point calibration through the use of prescriptive dilution factors (prepFAST DF column entries) in the Qtegra ISDS Software Sample List.

Figure 4 illustrates the basic approach of generating a 10-point calibration curve. A correlation coefficient of 0.99996 illustrates the dilution accuracy of prepFAST Auto-dilution System at low ng·mL⁻¹ concentrations.

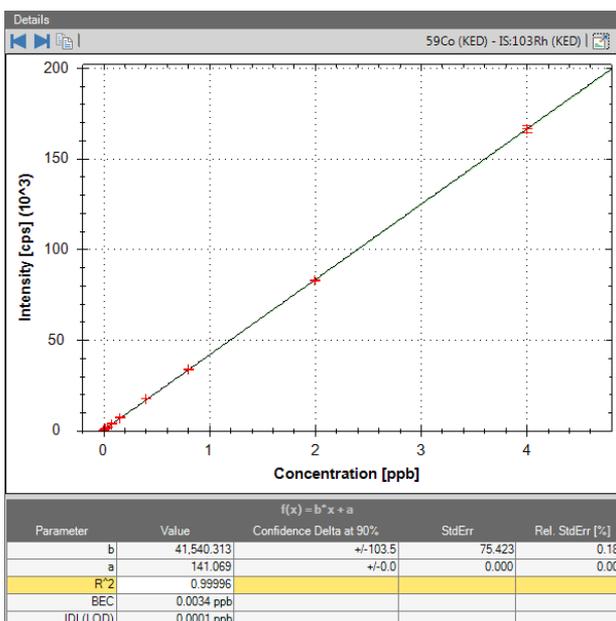


Figure 4. A 10-point calibration generated from a single stock solution using prescriptive dilution.

Automated Sample Dilution

As defined in USP <233> Elemental Impurities – Procedures,¹ all samples have to be diluted “with an appropriate solvent to obtain a final concentration of the Target elements of not more than 2 J” where J is defined by the daily dose of a drug and 2 J is the highest calibration point. To support this sample specific prescriptive dilution factors (prepFAST DF column entries, Figure 3) can be entered in the Qtegra ISDS Sample List (from a text file or LIMS import) and are seamlessly implemented by the prepFAST Auto-dilution System to eliminate manual sample preparation.

Automated Intelligent Dilution

The Thermo Scientific Qtegra ISDS Software supports thirteen separate QC analysis types in addition to any user defined tests. Depending on the protocol defined criteria, Qtegra ISDS can trigger a series of actions in order to address the observed data quality issue. If the QC test requires a new dilution Qtegra automatically inserts a new analysis to the acquisition queue and instructs the prepFAST Auto-dilution System to dilute the sample by an intelligently determined factor.

Over Calibration Range Auto-Dilution

While it is generally considered good laboratory practice to bracket measured concentrations within the calibrated concentration range, this is specifically mandated in some protocols. For example the US EPA states: “Samples with analyte concentrations above the calibration range should have been diluted and reanalyzed.”² While this could be achieved by manual dilution it is often impractical without prior knowledge of the sample and over-range samples have to be manually removed from the autosampler rack, diluted off-line, given a new position in the rack and added again to the analysis queue. Each step in the process is prone to error and potentially costly in terms of materials and time. With Qtegra ISDS Software support of the prepFAST Auto-dilution System however, measured concentrations can be restricted to within the calibration range with intelligent auto-dilution eliminating “reruns.”

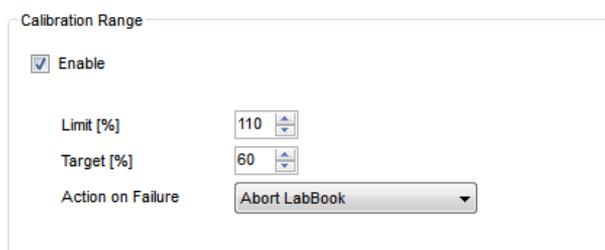


Figure 5. Calibration Range auto-dilution in the Qtegra ISDS Software prepFAST plug-in.

For example, Figure 5 shows how auto-dilution for over calibration range analyses are defined in Qtegra ISDS Software. With the Calibration Range Limit set to 110% (10% above the top standard concentration), any sample or QC analysis with at least one readback value over this limit will be automatically diluted by the prepFAST Auto-dilution System (to give a Target concentration of 60% of the top standard) and reanalyzed.

Internal Standard Range Auto-Dilution

All ICP-MS based instruments suffer from signal suppression when high levels of dissolved solids enter the plasma. Internal standards (IS) are employed in most methods to track signal response. For the analysis of drinking and waste waters, EPA Method 200.8² defines an acceptable IS recovery range of 60–125%.

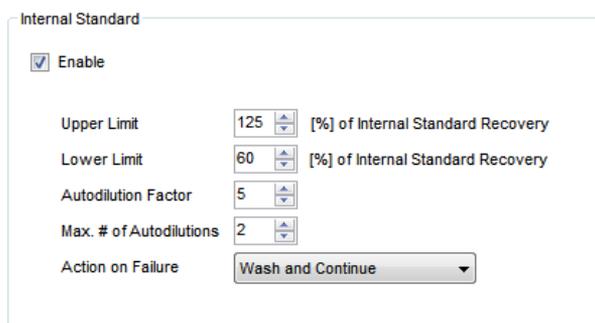


Figure 6. Internal Standard auto-dilution in the Qtegra ISDS Software prepFAST Auto-dilution System plug-in.

Figure 6, for example, shows how auto-dilution for internal standard recoveries are defined in the Thermo Scientific Qtegra ISDS Software. Samples with recoveries outside of the defined range would initially be 5-fold diluted followed by a new 10-fold dilution if required. With routine laboratories facing large numbers of often widely differing samples for 200.8 based analyses, intelligent auto-dilution offers significant throughput improvements.

Conclusion

Automation of the lab workflow has taken a step forward with the integration of auto-dilution. The ESI prepFAST Auto-dilution system has been demonstrated to be a powerful, flexible and robust tool in routine trace elemental analyses by the iCAP Q ICP-MS. The Thermo Scientific Qtegra ISDS based control of the complete system provides a single, simple integrated workflow, eliminating manual dilution in both prescriptive and intelligent, fully automated, analyses. Eliminating manual intervention increases productivity, prevents re-runs and reduces cost of ownership.

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1. United States Pharmacopeia General Chapter <233> Elemental Impurities – Procedures: Second Supplement to USP 35-NF 30.
2. US EPA – Solutions to Analytical Chemistry Problems with Clean Water Act Methods (EPA-821-R-07-002, March 2007).
3. US EPA Method 200.8 – Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma Mass Spectrometry (ICP-MS).

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