

## Analysis of Lead-Based Paint Using the Teledyne Leeman Labs Dual-View Prodigy Plus ICP-OES

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### Introduction

One of the most common uses of lead is in pigments for paint. Lead (II) chromate ( $\text{PbCrO}_4$ ) creates a color commonly known as “chrome yellow” and a “white lead” color is created using lead (II) carbonate ( $\text{PbCO}_3$ ). Lead is also added to improve the general performance of paint by reducing drying time, improving durability and increasing resistance to moisture and mildew.



Almost all homes constructed before 1970 contain some lead-based paint, which until 1950, could contain up to 50% lead (500,000 ppm). By the late 1960s, paints containing more than 1% (10,000 ppm) were still in use. Lead is also used in the toy manufacturing process and can be particularly concentrated in toys made from plastic. Toys may be painted with lead-based paint and plastic toys may use lead to increase flexibility and resist warping.

When lead-based paint is adhered to the surface it is painted on, it poses little or no health risk, but once it begins deteriorating it can become a serious human health hazard, due to exposure by inhalation or ingestion. Considering lead’s known effect on childhood mental development and adolescent hand-to-mouth activity, the presence of lead containing products has become an ever-increasing environmental concern.

In 1978, the US Consumer Product Safety Commission (USCPSC) set the maximum allowable lead content in paint for residential use to 0.06% (600 ppm) as outlined in 16 CFR § 1303.1. In 2009, the maximum allowable lead content was reduced to 0.009% (90 ppm). While lead has been banned from all paint used for domestic purposes, the use of lead in plastics is still permitted.

This application note will demonstrate the ability of the Teledyne Leeman Labs Prodigy Plus High Dispersion ICP to analyze lead-based paint samples. The sensitivity and large linear dynamic range of the instrument in the axial view mode will be used to determine a wide range of lead concentrations in several different paint samples.

## Instrumentation

A Prodigy Plus High-Dispersion ICP-OES system equipped with a dual-view torch and 120-position CETAC ASX-280 autosampler (Figure 1) was used to generate the data for this application note.

**Figure 1** Prodigy Plus ICP-OES and ASX-280 Autosampler



The Prodigy Plus is a compact benchtop simultaneous ICP-OES system featuring an 800 mm focal length Echelle optical system coupled with a mega-pixel large format CMOS detector. At 28 x 28 mm, the active area of the CMOS detector is significantly larger than any other solid-state detector currently used for ICP-OES. This combination allows the Prodigy Plus to achieve higher optical resolution and dispersion than any other solid-state detector-based ICP systems. The detector also provides continuous wavelength coverage from 165 to 1100 nm, permitting measurement over the entire ICP spectrum in a single reading, without sacrificing wavelength range or resolution. The detector design is inherently anti-blooming and is capable of random access, non-destructive readout that results in a dynamic range of more than six orders of magnitude. The Prodigy Plus uses a 40.68 MHz rugged, free-running RF generator, allowing it to handle the most difficult sample matrices, as well as common organic solvents.

## Sample Introduction

A high-sensitivity sample introduction system ensured that sufficient and steady emission signals were delivered to the spectrometer. The sample introduction system consisted of:

- Cyclonic Spray Chamber with Center Knockout Tube
- Glass Expansion Conikal™ Aqueous Nebulizer
- Four-Channel Peristaltic Pump

The volume of the cyclonic spray chamber is low allowing for fast washout between samples, while its knockout tube (or baffle) efficiently reduces the amount of sample aerosol that reaches the torch.

The Prodigy Plus torch is mounted using an innovative twist-lock auto-aligning sample introduction system, shown in Figure 2. This design permits operators to remove and replace the torch to the exact same position, providing day-to-day reproducibility and simplified training. Additionally, the twist-lock design automatically connects the coolant and auxiliary gas flows, eliminating potential errors.

**Figure 2** Axial Twist Lock Sample Introduction System


## Method

For the analyte of interest, background correction was performed simultaneously with the peak measurement, resulting in improved detection limits. The Prodigy Plus instrument operating parameters are shown in [Table I](#).

Table I Instrument Operating Parameters		
Parameter	Value	Part Number
RF Power	1.2 kW	-
Coolant Flow	15 L/min	-
Auxiliary Flow	0 L/min	-
Plasma Configuration	Axial	-
Nebulizer Pressure	36 psi	-
Pump Rate	35 rpm	-
Torch	Demountable Quartz (Axial, Radial and Dual)	318-00167-1
Injector	2.5 mm Bore Quartz Demountable	318-00161-AQ1
Sample Uptake Time	35 s	-
Nebulizer	Glass Expansion Conikal™ Aqueous Nebulizer	120-00463-1
Spray Chamber	Cyclonic with Knock-out	120-00393-1
Rinse Time	20 s	-
Integration Time	Axial 30 s	-
Optical Purge Rate	0.7 L/min	-

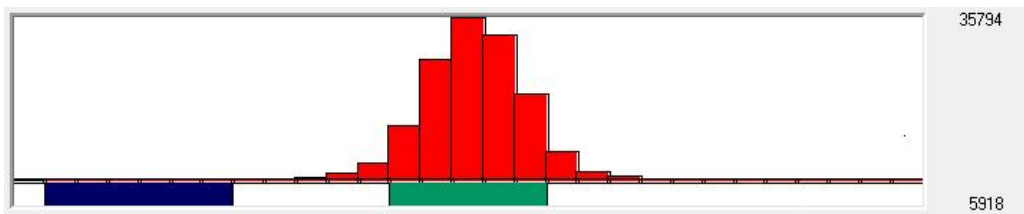
## Wavelengths

The Prodigy Plus uses a 5 x 29 pixel subarray, centered on the wavelength of interest, to collect data for each analyte. The analytical peaks and background correction points are defined in each subarray with pixel position and width values. The wavelength and background correction points used in this method are listed in [Table II](#). For background correction points that were more than 1 pixel wide, the background position indicates the starting position for the set of pixels used. The starting position and pixel width used for the analytical peak was 7 and 3, respectively. For the analyte of interest, background correction was performed simultaneously with the peak measurement. Additionally, all pixel data is saved allowing for future data recalculation.

Table II Wavelength, Background Correction Points and Integration Times Used						
Element	Wavelength (nm)	Left Background Pixel Position	Left Background Width	Right Background Pixel Position	Right Background Width	Simultaneous Integration Time (s)
Pb	220.353	2	6	None	-	5

An example of the data collection for each subarray is illustrated graphically in [Figure 3](#). This figure represents the data collected for Std 1 at the Pb 220 nm line. [Figure 3](#) shows the left background correction point at pixel position 2 with a width of 6 pixels. No background correction was used on the right side of the peak, due to a noisy baseline observed in the paint samples. The pixels used for integrating the analytical peak are at pixel positions 13 through 17. The thin black line that runs across the subarray underneath the peak illustrates the calculated background based on the background correction points chosen.

**Figure 3** Graphical Representation of the Pb 220 nm Subarray



### Sample Preparation

Two paint reference materials and two real-world paint samples were analyzed. The certified reference materials (CRM) included NIST SRM 2582 Powdered Paint Nominal 200 mg/kg Lead and RTC CRM 013-050 Trace Metals in Paint Chips (Lot # AW13). Both CRMs were weighed and digested as received. Real-world paint samples included paint chips scraped from the porch of a local house and from a yellow-painted plastic toy car. Both samples were ground with a mortar and pestle prior to weighing for digestion.

All paint samples were digested using a microwave accelerated reaction system (MARS®) 5, closed vessel system (CEM Corporation, Matthews, North Carolina) according to the EPA Method SW846 3051A procedure:

1. Weigh 0.3 – 0.5 g of sample directly into a PTFE vessel.
2. Add 10 mL of concentrated nitric acid (HNO<sub>3</sub>).
3. Seal the vessel and heat in the microwave using the heating program in [Table III](#).

Table III Microwave Heating Program				
Step	Power (W)	Ramp (°C/min)	Temp (°C)	Hold (min)
1	600	5.5	175	5.0

4. Once cool, the seals on the vessels were broken and the samples were allowed to sit inside a fume hood until HNO<sub>3</sub> acid fumes were no longer visible.
5. The contents of each vessel were then filtered through 0.4 μm filter paper directly into a 100 mL volumetric flask and brought to volume with deionized water.

**Note:** 5 mL of HNO<sub>3</sub> was used to digest the NIST powdered paint standard. The final volume was brought to 50 mL to avoid diluting the sample below the concentration of the lowest calibration standard and to maintain the same acid concentration as the calibration standards.

### Calibration Standards

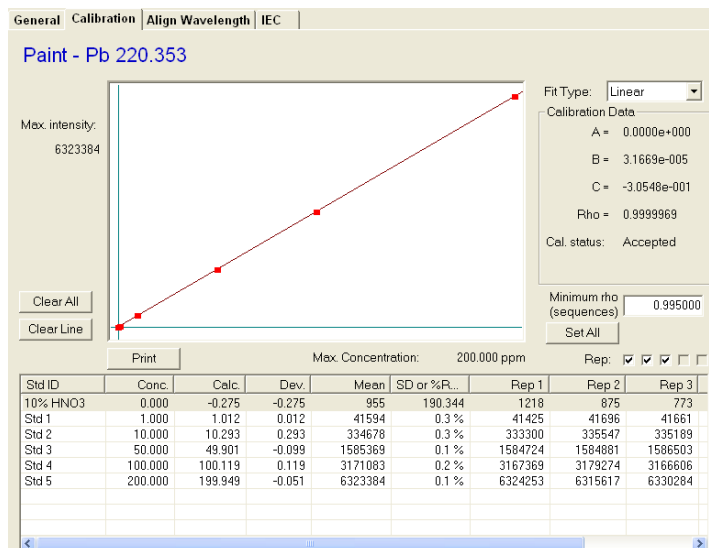
Calibration standards were made from single element VHG Labs (Manchester, NH) ICP standards containing either 1000 or 10,000 ppm Pb. The final acid concentration in all standards was 10% HNO<sub>3</sub> to match the acid concentration in the digested samples. The concentrations used for calibration are shown in Table IV. The instrument was calibrated over a wide range to accommodate the expected Pb concentrations in a wide variety of lead-based paints.

Table IV Calibration Standard Concentration (in 10% HNO <sub>3</sub> ), ppm					
Element	STD1	STD2	STD3	STD4	STD5
Pb	0	1	10	50	100

### Calibration Results

After igniting the plasma and allowing 15 minutes for the instrument to warm up, the instrument was calibrated using the calibration blank and standards listed in Table IV. Calibration results are shown in Figure 4.

**Figure 4** Typical Calibration Curve for Pb 220.353 nm



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## Sample Results

Following calibration, the CRMs were analyzed. Results from the NIST and RTC standards are presented in [Table V](#) and [Table VI](#), respectively. The NIST standard was prepared and analyzed in duplicate and the % difference between each sample preparation was calculated. The RTC standard was also prepared in duplicate, however, the results from both sample preparations were averaged together to compensate for the inhomogeneous nature of the sample (paint chips of varying colors, textures and sizes).

Table V Results from the Analysis of NIST SRM 2582						
Element	Wavelength (nm)	Measured Pb Conc (mg/kg)	St Dev (mg/kg)	%RSD	Certified Conc (mg/kg)	% Difference Between Result and Duplicate Prep
<b>Pb</b>	220.353	212.4	2.4	1.1	208.8 ±4.9	1.2

Table VI Results from the Analysis of RTC CRM 013-050					
Element	Wavelength (nm)	Avg Measured Conc (mg/kg)	Avg.% RSD	Certified Conc (mg/kg)	Acceptable Conc Range (mg/kg)
<b>Pb</b>	220.353	637.3	0.5	643 ±129.4	513.6-772.4

Results for the real-world porch paint and plastic toy car paint samples are shown in [Table VII](#) and [Table VIII](#), respectively. Note that the results in [Table VII](#) are listed in units of elemental wt %, while the results in [Table VIII](#) are listed in units of mg/kg. Because no detectable Pb was measured in the plastic toy car paint samples, 10 ppm Pb was spiked into the samples to verify that Pb could be measured at relatively low concentrations in that sample matrix. As [Table VIII](#) indicates, both spikes were measured within ±10% of the spiked concentration.

Table VII Results from the Analysis of Paint Chips from a Porch					
Element	Wavelength (nm)	Measured Pb Conc (wt %)	St. Dev. (wt %)	% RSD	% Difference Between Result and Duplicate Prep
Pb	220.353	5.49	0.01	0.2	1.2

Table VIII Results from the Analysis of Paint Scraped from a Plastic Toy					
Sample ID	Wavelength (nm)	Measured Pb Conc (mg/kg)	% RSD	Spike Conc (µg/mL)	Spike Rec (%)
Toy Paint, Prep 1	220.353	None Detected	-	10.0	102.6
Toy Paint, Prep 2	220.353	None Detected	-	10.0	102.7

## Conclusion

The analysis of Pb in a variety of lead-based paint samples was successfully performed using the Teledyne Leeman Labs Prodigy Plus High Dispersion ICP. The sensitivity of the instrument in the axial view mode, combined with its wide linear dynamic range, allowed the instrument to be calibrated over a broad range of concentrations (100 ppm to 6%) using a single instrument method. The image stabilized plasma and the simultaneous data collection of both peak and background data combine to provide exceptionally precise and stable results.