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Caroline Bouvier, Sebastiaan van Nuffel, Alain Brunelle. ToF-SIMS spectra of historical inorganic pigments: Lead-based pigments in negative polarity. *Surface Science Spectra*, 2024, 31 (1), pp.015005. 10.1116/6.0003507 . hal-04595993

HAL Id: hal-04595993

<https://hal.science/hal-04595993>

Submitted on 31 May 2024

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RESEARCH ARTICLE | MAY 30 2024

ToF-SIMS spectra of historical inorganic pigments: Lead-based pigments in negative polarity

Special Collection: [ToF-SIMS Spectra of Historical Inorganic Pigments](#)

Caroline Bouvier  ; Sebastiaan Van Nuffel  ; Alain Brunelle  



Surf. Sci. Spectra 31, 015005 (2024)

<https://doi.org/10.1116/6.0003560>



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- ▶ partial pressure measurement and control of process gases
- ▶ reactive sputter process control
- ▶ vacuum diagnostics
- ▶ vacuum coating process monitoring

ToF-SIMS spectra of historical inorganic pigments: Lead-based pigments in negative polarity

Cite as: Surf. Sci. Spectra 31, 015005 (2024); doi: 10.1116/6.0003560

Submitted: 20 February 2024 · Accepted: 13 May 2024 ·

Published Online: 30 May 2024



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Note: This paper is part of the 2024 Special Topic Collection on ToF-SIMS Spectra of Historical Inorganic Pigments.

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ABSTRACT

ToF-SIMS is increasingly used to analyze cultural heritage materials because it can simultaneously detect organic and inorganic materials while mapping them on a surface. The precise identification of a pigment in a specific layer of a painting or of remaining color on a statue can inform about the technique used or the time of manufacture as well as expose possible forgeries when anachronistic ingredients are identified. Reference spectra are required to confidently identify a given pigment using ToF-SIMS. This database focuses on eight lead-based historical inorganic pigments, manufactured following traditional recipes. Lead pigments have been widely used in painting until the late 19th century. Here, the negative polarity ToF-SIMS reference spectra using a Bi³⁺ primary ion species are presented.

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Accession #: 01919, 01920, 01921, 01922, 01923, 01924, 01925, 01926

Technique: SIMS

Specimen: Lead tin yellow I, Naples yellow, Lead white, Flake white 10 μm, Flake white, Cerussite, Minium, Litharge

Instrument: IONTOF, TOF SIMS IV

Major Species in Spectra: O, H, Si, S, Al, C, Na, Pb, K, Cl, Sb, Sn

Minor Species in Spectra: Hydrocarbon contamination

Published Spectra: 8

Spectral Category: Reference

INTRODUCTION

ToF-SIMS imaging can detect a wide range of elements, so colored minerals can be identified and mapped, given that reference spectra are available for reliable assignment. Here, we provide a ToF-SIMS spectral database for eight lead-based inorganic pigments. Lead-based pigments have been commonly used in all painting techniques until the late 19th century and, therefore, are very likely to be found when analyzing a historical painting sample. In particular, the pigment generally referred to as “lead white” can have various compositions depending on the manufacturing process, and this paper compares a selection of them (namely, #01921, #01922, #01923, and #01924). For each, a list of their fragmentation products is given.

The reference materials were supplied by specialized manufacturers selling pigments reproducing historical recipes as closely as

possible, such as Kremer Pigmente GmbH, Master Pigments, Laverdure, and Sennelier. Pigments were available as finely ground powder of variable particle size (between one and a few tens of μm) conditioned in glass sealed vials. In paintings, pigment particles are dispersed and not dissolved, so all references were analyzed without any preparation treatment. Natural and synthetic pigments may contain impurities, and specimen compositions described below are the major components as found on the material data sheets provided by the suppliers.

A stainless-steel spatula was cleaned with propan-2-ol and the powder was directly deposited on a 1 × 1 mm² piece of conductive double-sided tape (3M) already fixed to a 1 cm × 1 cm × 0.125 mm stainless steel plate (17-7PH, Goodfellow Cambridge Limited, UK) fitting the dimensions of the sample holder. A hand press covered

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with clean aluminum foil was applied onto the powder to fix it to the tape. This allowed us to minimize the risk of dust formation when operating in the analysis chamber under ultrahigh vacuum (10^{-8} to 10^{-9} hPa) while ensuring a flat surface.

All spectra were obtained on an IONTOF TOF SIMS IV equipped with a 25 keV bismuth liquid metal ion gun (LMIG) and an Argon gas cluster ion beam (GCIB). Before all analyses, a $500 \times 500 \mu\text{m}^2$ surface was cleaned using 1500–2000 Ar clusters with a kinetic energy of 20 keV (sputter ion dose 2×10^{15} ions/cm²). Analyses were then performed on a $250 \times 250 \mu\text{m}^2$ area rastered over 128×128 pixels using the high current bunched mode. Primary ion dose (25 keV Bi₃⁺) was 4.35×10^{11} ions/cm². Surface potential was compensated.

Data were calibrated using a set of peaks appearing in all spectra, namely, the peaks O₂⁻ at m/z 31.99, Cl⁻ at m/z 34.97, PbO₂H⁻ at m/z 241.98, and ²⁰⁶PbPbO₃H⁻ at m/z 462.94, as well as ²⁰⁶PbPb₂O₄H⁻ at m/z 686.94 for all data except #01919. This calibration list allowed for a complete coverage of the mass range of interest, and the lead-containing calibration ions could easily be verified using their isotopic pattern.

Figures and tables presented below contain the major monoisotopic peaks for negative polarity. Sn, Sb, and Pb clusters have isotopic patterns with often more than 5–10 peaks and all of them should be present for accurate identification. Full data are available within the supplementary material. Each pigment is uniquely identified, thanks to an accession number and they are grouped according to their composition and described in the following section. Their historical relevance is briefly described based on the current literature.^{1–5} The Spectrum ID# refer to that number.

SPECIMEN DESCRIPTION (ACCESSION # 01919)

Specimen: Lead Tin Yellow I (Kremer Pigmente ref. 10100)

CAS Registry #: 12143-43-0

Specimen Characteristics: Homogeneous; solid; unknown crystallinity; dielectric; inorganic compound; powder;

Chemical Name: Lead stannate

Source: Kremer Pigmente GmbH

Specimen Composition: Pb₂SnO₄

Form: Ground pigment powder

History and Significance: A light-yellow pigment that was widely used in oil painting from the Middle Ages to the dawn of the 19th century. Prohibited today because of its lead content.

As Received Condition: Ground pigment powders were stored in a sealed glass vial in a wooden box, protected from daylight. Stored at room temperature.

Analyzed Region: Areas of $250 \times 250 \mu\text{m}^2$

Ex Situ Preparation/Mounting: Powder was deposited and pressed onto conductive double-sided tape attached to stainless steel $1 \times 1 \text{ cm}^2$ plates. The resulting deposit of powder had a flat surface and was fixed in a suitable way for the vacuum chamber. The plate was directly mounted on a “backmount” sample holder (IONTOF).

In Situ Preparation: None

Charge Control Conditions and Procedures: A low energy electron flood gun was used. Bias voltage between 20 and –30 V. Flood gun filament current 2.35 A. Surface potential was corrected (sample dependent).

Temp. During Analysis: 300 K

Pressure During Analysis: Between 1×10^{-5} and 1×10^{-6} Pa

Pre-analysis Beam Exposure: Ar_{1500–2000}, 20 keV, 2×10^{15} ions/cm²

SPECIMEN DESCRIPTION (ACCESSION # 01920)

Specimen: Naples Yellow (Kremer Pigmente ref. 43130)

CAS Registry #: 13510-89-9

Specimen Characteristics: Homogeneous; solid; unknown crystallinity; dielectric; inorganic compound; powder;

Chemical Name: Lead antimonate

Source: Kremer Pigmente GmbH

Specimen Composition: Pb₂Sb₂O₇ [and Pb₃(SbO₄)₂]

Form: Ground pigment powder

History and Significance: A yellow pigment used on Renaissance palettes, which is synthesized by heating a lead compound (oxide or nitrate) with an antimony compound (oxide, sulfide, or potassium antimonate). Particle size is in the micrometer range.

As Received Condition: Ground pigment powders were stored in a sealed glass vial in a wooden box, protected from daylight. Stored at room temperature.

Analyzed Region: Areas of $250 \times 250 \mu\text{m}^2$

Ex Situ Preparation/Mounting: Powder was deposited and pressed onto conductive double-sided tape attached to stainless steel $1 \times 1 \text{ cm}^2$ plates. The resulting deposit of powder had a flat surface and was fixed in a suitable way for the vacuum chamber. The plate was directly mounted on a “backmount” sample holder (IONTOF).

In Situ Preparation: None

Charge Control Conditions and Procedures: A low energy electron flood gun was used. Bias voltage between 20 and –30 V. Flood gun filament current 2.35 A. Surface potential was corrected (sample dependent).

Temp. During Analysis: 300 K

Pressure During Analysis: Between 1×10^{-5} and 1×10^{-6} Pa

Pre-analysis Beam Exposure: Ar_{1500–2000}, 20 keV, 2×10^{15} ions/cm²

SPECIMEN DESCRIPTION (ACCESSION # 01921, 01922, 01923, 01924)

Specimen: (Accession #01921) Lead white (Kremer Pigmente ref. 46000), (Accession #01922) Flake white $10 \mu\text{m}$ (Masters Pigments ref. 901010), (Accession #01923) Flake white (Sennelier PW no.108), (Accession #01924) Cerussite (Masters Pigments ref. 951010)

CAS Registry #: (Accession #01921, #01924) 598-63-0, (Accession #01922, #01923) 1319-46-6

Specimen Characteristics: Inhomogeneous; solid; unknown crystallinity; dielectric; inorganic compound; powder;

Chemical Name: (Accession #01921, #01924) Lead (II) carbonate, synth., (Accession #01922, #01923) Lead (II) carbonate, basic

Source: (Accession #01921) Kremer Pigmente GmbH, (Accession #01922, #01924) Masters Pigments, USA, (Accession #01923) Sennelier, France

Specimen Composition: (Accession #01921, #01924) PbCO₃, (Accession #01922, #01923) 2PbCO₃·Pb(OH)₂

Form: Ground pigment powder

History and Significance: The most widely used white pigment was progressively replaced during the beginning of the 20th century with newly available less toxic alternatives. A mixture of cerussite (lead carbonate) and hydrocerussite (basic lead carbonate) with proportions varying according to the manufacturing process can inform about the origin of the pigment. It is synthesized using lead strips, vinegar, CO₂, and heat. The latter two are usually provided by a fermenting material. In the presence of acetic acid vapors, the lead surface corrodes to lead acetate, which is then converted to basic lead carbonate in the presence of CO₂. This stable process has been used over centuries, and its origins date back to Ancient Greece and China. It was also used as a cosmetic since ancient times.

As Received Condition: Ground pigment powders were stored in a sealed glass vial in a wooden box, protected from daylight. Stored at room temperature.

Analyzed Region: Areas of $250 \times 250 \mu\text{m}^2$

Ex Situ Preparation/Mounting: Powder was deposited and pressed onto conductive double-sided tape attached to stainless steel $1 \times 1 \text{ cm}^2$ plates. The resulting deposit of powder had a flat surface and was fixed in a suitable way for the vacuum chamber. The plate was directly mounted on a “backmount” sample holder (IONTOF).

In Situ Preparation: None

Charge Control Conditions and Procedures: A low energy electron flood gun was used. Bias voltage between 20 and -30 V . Flood gun filament current 2.35 A. Surface potential was corrected (sample dependent).

Temp. During Analysis: 300 K

Pressure During Analysis: Between 1×10^{-5} and $1 \times 10^{-6} \text{ Pa}$

Pre-analysis Beam Exposure: Ar_{1500–2000}, 20 keV, 2×10^{15} ions/cm²

SPECIMEN DESCRIPTION (ACCESSION # 01925)

Specimen: Minium/Red Lead (Kremer Pigmente ref. 42500)

CAS Registry #: 1314-41-6

Specimen Characteristics: Inhomogeneous; solid; unknown crystallinity; dielectric; inorganic compound; powder;

Chemical Name: Lead tetraoxide

Source: Kremer Pigmente GmbH

Specimen Composition: Pb₃O₄

Form: Ground pigment powder

History and Significance: This naturally occurring lead oxide has been synthesized since early times by heating lead carbonates. Its color approaches that of cinnabar, providing a cheaper alternative. Used since ancient Greek times as a rust inhibitor for metals, it remained its main use in the 20th century until it was banned in the 1990s.

As Received Condition: Ground pigment powders were stored in a sealed glass vial in a wooden box, protected from daylight. Stored at room temperature.

Analyzed Region: Areas of $250 \times 250 \mu\text{m}^2$

Ex Situ Preparation/Mounting: Powder was deposited and pressed onto conductive double-sided tape attached to stainless steel $1 \times 1 \text{ cm}^2$ plates. The resulting deposit of powder had a flat surface and was fixed in a suitable way for the vacuum chamber. The plate was directly mounted on a “backmount” sample holder (IONTOF).

In Situ Preparation: None

Charge Control Conditions and Procedures: A low energy electron flood gun was used. Bias voltage between 20 and -30 V . Flood gun filament current 2.35 A. Surface potential was corrected (sample dependent).

Temp. During Analysis: 300 K

Pressure During Analysis: Between 1×10^{-5} and $1 \times 10^{-6} \text{ Pa}$

Pre-analysis Beam Exposure: Ar_{1500–2000}, 20 keV, 2×10^{15} ions/cm²

SPECIMEN DESCRIPTION (ACCESSION # 01926)

Specimen: Massicot, Litharge (Kremer Pigmente ref. 43010)

CAS Registry #: 1317-36-8

Specimen Characteristics: Inhomogeneous; solid; unknown crystallinity; dielectric; inorganic compound; powder;

Chemical Name: Lead (II) oxide

Source: Kremer Pigmente GmbH

Specimen Composition: PbO

Form: Ground pigment powder

History and Significance: Litharge and massicot are lead monoxide polymorphs with the formula PbO, respectively, red and yellow orange in appearance. They are both formed by heating lead or lead white. Litharge plays a key role in the preparation of drying oil for painting: when mixed, it accelerates polymerization of the film.

As Received Condition: Ground pigment powders were stored in a sealed glass vial in a wooden box, protected from daylight. Stored at room temperature.

Analyzed Region: Areas of $250 \times 250 \mu\text{m}^2$

Ex Situ Preparation/Mounting: Powder was deposited and pressed onto conductive double-sided tape attached to stainless steel $1 \times 1 \text{ cm}^2$ plates. The resulting deposit of powder had a flat surface and was fixed in a suitable way for the vacuum chamber. The plate was directly mounted on a “backmount” sample holder (IONTOF).

In Situ Preparation: None

Charge Control Conditions and Procedures: A low energy electron flood gun was used. Bias voltage between 20 and -30 V . Flood gun filament current 2.35 A. Surface potential was corrected (sample dependent).

Temp. During Analysis: 300 K

Pressure During Analysis: Between 1×10^{-5} and $1 \times 10^{-6} \text{ Pa}$

Pre-analysis Beam Exposure: Ar_{1500–2000}, 20 keV, 2×10^{15} ions/cm²

INSTRUMENT CONFIGURATION

Manufacturer and Model: IONTOF, TOF SIMS IV

Analyzer Type: Time-of-flight

Sample Rotation: No

Rotation Rate: 0 rpm

Oxygen Flood Source: None

Oxygen Flood Pressure: N/A

Other Flood Source: None

Other Flood Pressure: N/A

Unique Instrument Features Used: None

Energy Acceptance Window: 20 eV

Post-acceleration Voltage: 10 000 eV

Sample Bias: 10–20 eV

Specimen Normal-to-Analyzer (Θ_e): 0°

■ Ion Sources

Ion Source 1 of 2

Purpose of This Ion Source: Analysis beam

Ion Source Manufacturer: IONTOF GmbH

Ion Source Model: LMIG with bismuth cluster (25 keV)

Beam Mass Filter: Yes

Beam Species and Charge State: Bi_3^+

Beam Gating Used: Double pulsed + Bunched

Additional Beam Comments: None

Beam Voltage: 25 000 eV

Net Beam Voltage (impact voltage): 25 000 eV

Ion Pulse Width: 0.8–1.2 ns

Ion Pulse Rate: 5–10 kHz

DC Beam Current: ~ 10 nA

Pulsed Beam Current: ~ 0.0004 nA

Current Measurement Method: Faraday cup

Beam Diameter: ~ 2 μm

Beam Raster Size: 250×250 μm^2

Raster Pixel Dimensions: 128×128

Beam Incident Angle: 45°

Source-to-Analyzer Angle: 45°

Ion Source 2 of 2

Purpose of this Ion Source: Sputtering beam

Ion Source Manufacturer: IONTOF GmbH

Ion Source Model: Argon GCIB

Beam Mass Filter: Yes

Beam Species and Charge State: Ar_n^+ with $n = 1500$ – 2000

Beam Gating Used: Wien filter

Additional Beam Comments: Used as sputter gun, not analysis mode.

Beam Voltage: 20 000 eV

Net Beam Voltage (impact voltage): 20 000 eV

Ion Pulse Width: N/A

Ion Pulse Rate: N/A

DC Beam Current: ~ 10 nA

Pulsed Beam Current: N/A

Current Measurement Method: Faraday cup

Beam Diameter: ~ 50 μm

Beam Raster Size: 500×500 μm^2

Raster Pixel Dimensions: N/A

Beam Incident Angle: 45°

Source-to-Analyzer Angle: 45°

ACKNOWLEDGMENTS

This work was financially supported by the Agence Nationale de la Recherche (Grant No. ANR-2015-CE29-0007 DEFIMAGE). C.B. has received funding from the European Union's Horizon Europe Research and Innovation Programme under Marie Skłodowska-Curie Grant Agreement No. 101108506. The authors thank Sven Kayser and Matthias Kleine-Boymann (IONTOF GmbH) for providing extended access to the software SURFACELAB 7.3 that allowed thorough processing of the data.

AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Caroline Bouvier: Investigation (lead); Writing – original draft (lead). **Sebastiaan Van Nuffel:** Investigation (equal); Writing – review & editing (lead). **Alain Brunelle:** Conceptualization (lead); Supervision (equal); Writing – review & editing (equal).

DATA AVAILABILITY

The data that support the findings of this study are available within the article and its supplementary material.

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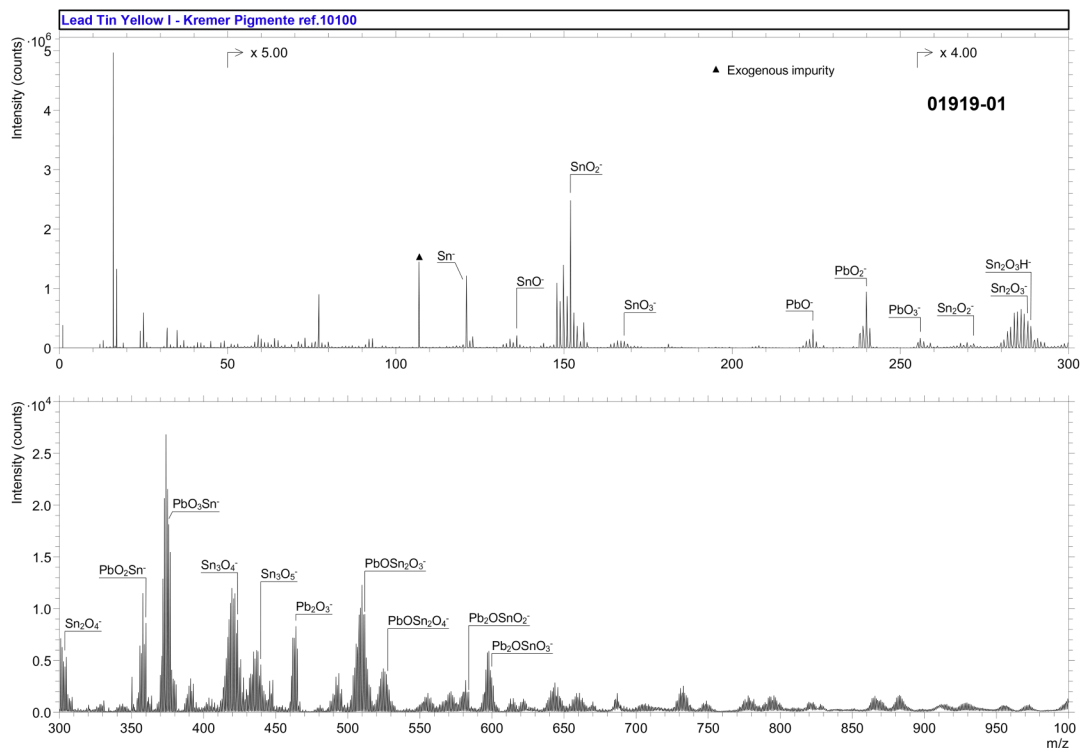
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SPECTRAL FEATURES TABLE

Lead Tin Yellow I—Kremer Pigmente ref. 10100—Negative polarity

Spectrum ID #	Mass (Δm), Da	Species	Peak Assignment
01919-01	119.900 (−25.6)	Sn^-	Pb_2SnO_4
	135.898 (5.42)	SnO^-	Pb_2SnO_4
	151.896 (22.0)	SnO_2^-	Pb_2SnO_4
	167.890 (14.0)	SnO_3^-	Pb_2SnO_4
	168.899 (21.6)	SnO_3H^-	Pb_2SnO_4
	223.974 (9.20)	PbO^-	Pb_2SnO_4
	239.966 (6.39)	PbO_2^-	Pb_2SnO_4
	255.956 (−23.7)	PbO_3^-	Pb_2SnO_4
	271.801 (22.0)	Sn_2O_2^-	Pb_2SnO_4
	287.798 (29.4)	Sn_2O_3^-	Pb_2SnO_4
	303.792 (22.9)	Sn_2O_4^-	Pb_2SnO_4
	359.877 (21.6)	PbO_2Sn^-	Pb_2SnO_4
	375.875 (28.5)	PbO_3Sn^-	Pb_2SnO_4
	423.702 (35.7)	Sn_3O_4^-	Pb_2SnO_4
	439.688 (14.6)	Sn_3O_5^-	Pb_2SnO_4
	463.940 (3.85)	Pb_2O_3^-	Pb_2SnO_4
	511.783 (41.8)	$\text{PbOSn}_2\text{O}_3^-$	Pb_2SnO_4
	527.777 (40.3)	$\text{PbOSn}_2\text{O}_4^-$	Pb_2SnO_4
	583.860 (32.4)	$\text{Pb}_2\text{OSnO}_2^-$	Pb_2SnO_4
	599.853 (29.6)	$\text{Pb}_2\text{OSnO}_3^-$	Pb_2SnO_4

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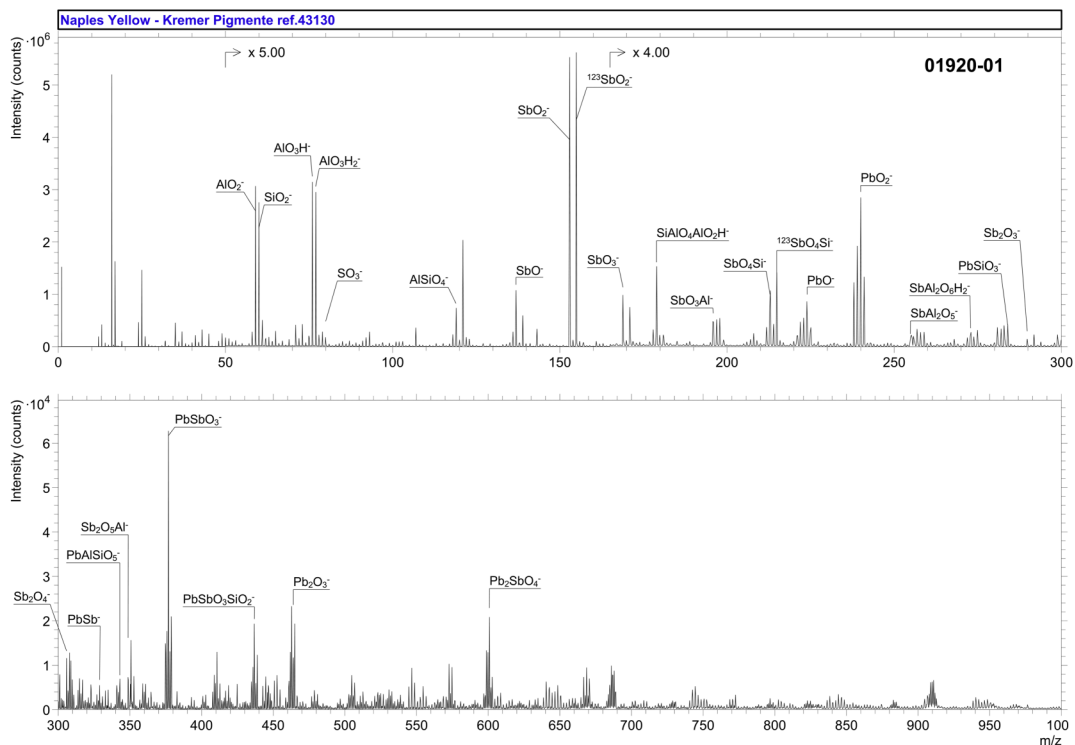
Accession #:	01919-01
Specimen:	Lead Tin Yellow I—Kremer Pigmente ref. 10100
Technique:	SIMS
Instrument:	IONTOF, TOF SIMS IV
Analyzer Type:	Time-of-flight
Analyzer Mass Resolving Power:	2800 m/Δm
Mass Resolving Power Determined at:	25 Da
Species Used for Mass Calibration:	O ₂ ⁻ , Cl ⁻ , PbO ₂ H ⁻ , ²⁰⁶ PbPbO ₃ H ⁻
Specimen Normal-to-Analyzer:	0°
Source-to-Analyzer Angle:	45°
Primary Beam Ion Gun:	LMIG
Primary Ion Species:	Bi ³⁺
Primary Ion Dose:	4.34 × 10 ¹¹ ion/cm ²
Primary Ion Pulse Width:	0.8–1.2 ns
Primary Ion Pulse Rate:	5 kHz
Net Beam Voltage:	25 000 eV
Pulsed Beam Current:	0.000 21 nA
DC Beam Current:	14.4 nA
Beam Diameter:	~2 μm
Beam Raster Size:	250 × 250 μm ²
Beam Incident Angle:	45°
Secondary Source Polarity:	Negative
Mass Range Minimum:	0 Da
Mass Range Maximum:	3545 Da
Spectrum Dead Time Corrected:	Yes
Total Spectral Acquisition Time:	207 s
Comment:	Distinctive isotopic pattern for Sn-containing ions

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SPECTRAL FEATURES TABLE

Spectrum ID #	Mass (Δm), Da	Species	Peak Assignment
01920-01	58.972 (8.57)	AlO_2^-	From manufacturing process
	59.970 (40.5)	SiO_2^-	From manufacturing process
	75.975 (-2.10)	AlO_3H^-	From manufacturing process
	76.983 (9.38)	AlO_3H_2^-	From manufacturing process
	79.960 (36.9)	SO_3^-	From manufacturing process
	118.943 (37.9)	AlSiO_4^-	From manufacturing process
	136.898 (-12.6)	SbO^-	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$
	152.899 (29.0)	SbO_2^-	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$
	168.890 (6.46)	SbO_3^-	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$
	178.918 (0.95)	$\text{AlSiO}_4\text{AlO}_2\text{H}^-$	From manufacturing process
	195.876 (27.6)	SbAlO_3^-	From manufacturing process
	212.869 (36.5)	SbOSiO_3^-	From manufacturing process
	223.970 (-10.9)	PbO^-	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$
	239.965 (-18.9)	PbO_2^-	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$
	254.846 (13.9)	$\text{SbAl}_2\text{O}_5^-$	From manufacturing process
	272.859 (21.8)	$\text{SbAl}_2\text{O}_6\text{H}_2^-$	From manufacturing process
	283.947 (28.4)	PbSiO_3^-	From manufacturing process
	289.800 (23.4)	Sb_2O_3^-	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$
	305.794 (18.5)	Sb_2O_4^-	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$
	328.890 (26.3)	PbSb^-	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$
	342.921 (31.5)	PbAlSiO_5^-	From manufacturing process
	348.771 (20.1)	$\text{Sb}_2\text{O}_5\text{Al}^-$	From manufacturing process
	376.874 (21.1)	PbSbO_3^-	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$
	436.845 (28.7)	$\text{PbSbO}_3\text{SiO}_2^-$	From manufacturing process
	463.933 (-12.6)	Pb_2O_3^-	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$
	600.860 (38.0)	$\text{Pb}_2\text{SbO}_4^-$	$\text{Pb}_2\text{Sb}_2\text{O}_7/\text{Pb}_3(\text{SbO}_4)_2$

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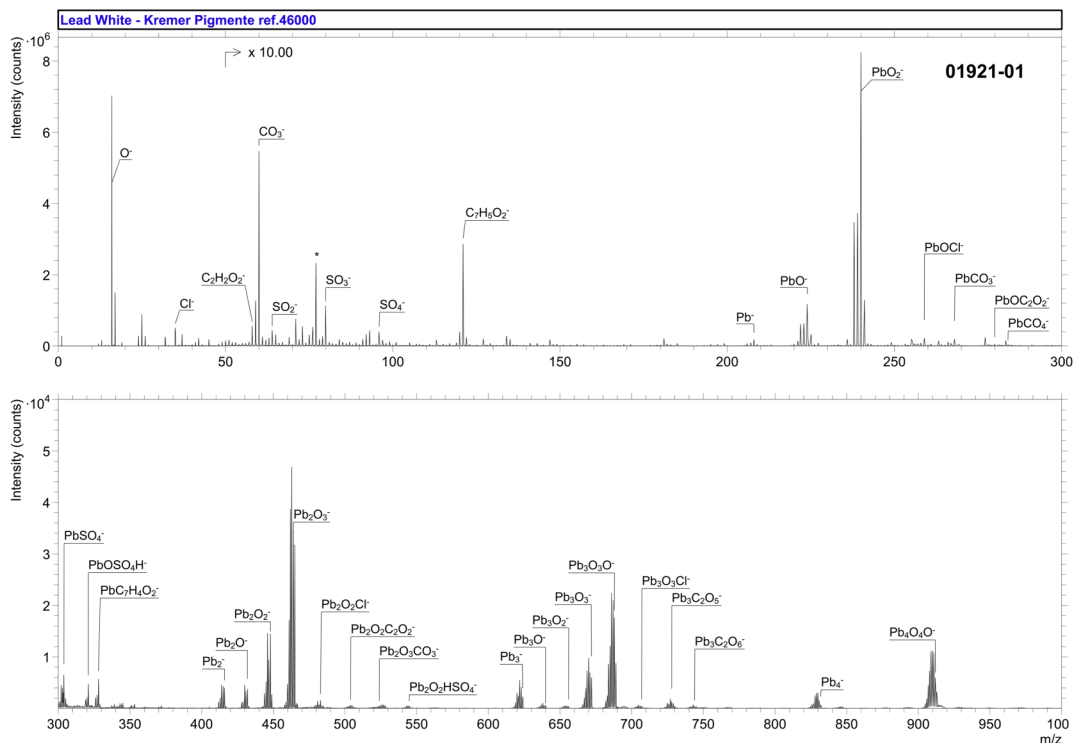
Accession #:	01920-01
■ Specimen:	Naples Yellow—Kremer Pigmente ref. 43130
■ Technique:	SIMS
Instrument:	IONTOF, TOF SIMS IV
Analyzer Type:	Time-of-flight
Analyzer Mass Resolving Power:	2900 m/Δm
Mass Resolving Power Determined at:	25 Da
Species Used for Mass Calibration:	O ₂ ⁻ , Cl ⁻ , PbO ₂ H ⁻ , ²⁰⁶ PbPbO ₃ H ⁻ , ²⁰⁶ PbPb ₂ O ₄ H ⁻
Specimen Normal-to-Analyzer:	0°
Source-to-Analyzer Angle:	45°
Primary Beam Ion Gun:	LMIG
Primary Ion Species:	Bi ₃ ⁺
Primary Ion Dose:	4.34 × 10 ¹¹ ion/cm ²
Primary Ion Pulse Width:	0.8–1.2 ns
Primary Ion Pulse Rate:	5 kHz
Net Beam Voltage:	25 000 eV
Pulsed Beam Current:	0.000 21 nA
DC Beam Current:	14.6 nA
Beam Diameter:	~2 μm
Beam Raster Size:	250 × 250 μm ²
Beam Incident Angle:	45°
Secondary Source Polarity:	Negative
Mass Range Minimum:	0 Da
Mass Range Maximum:	3545 Da
Spectrum Dead Time Corrected:	Yes
Total Spectral Acquisition Time:	210 s
Comment:	Distinctive isotopic pattern for Sb-containing ions

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SPECTRAL FEATURES TABLE

Spectrum ID #	Mass (Δm), Da	Species	Peak Assignment
01921-01	34.970 (17.5)	Cl ⁻	—
	58.008 (36.5)	C ₂ H ₂ O ₂ ⁻	Acetate from manufacturing process
	59.986 (5.70)	CO ₃ ⁻	PbCO ₃
	63.965 (38.9)	SO ₂ ⁻	Sulfate from manufacturing process
	79.962 (51.3)	SO ₃ ⁻	Sulfate from manufacturing process
	95.954 (16.7)	SO ₄ ⁻	Sulfate from manufacturing process
	121.035 (42.9)	C ₆ H ₄ CO ₂ H ⁻	Phthalate contamination
	207.973 (-21.0)	Pb ⁻	PbCO ₃
	223.969 (-15.9)	PbO ⁻	PbCO ₃
	239.964 (-11.6)	PbO ₂ ⁻	PbCO ₃
	258.943 (8.45)	PbOCl ⁻	—
	267.957 (-19.6)	PbCO ₃ ⁻	PbCO ₃
	279.959 (-9.12)	PbOC ₂ O ₂ ⁻	PbCO ₃
	283.962 (18.4)	PbCO ₄ ⁻	PbCO ₃
	303.944 (49.6)	PbSO ₄ ⁻	Sulfate from manufacturing process
	320.951 (60.1)	PbOHSO ₄ ⁻	Sulfate from manufacturing process
	328.007 (26.4)	PbC ₇ H ₄ O ₂ ⁻	—
	415.955 (3.08)	Pb ₂ ⁻	PbCO ₃
	431.941 (-18.8)	Pb ₂ O ⁻	PbCO ₃
	447.936 (-16.6)	Pb ₂ O ₂ ⁻	PbCO ₃
	463.931 (-16.8)	Pb ₂ O ₃ ⁻	PbCO ₃
	464.937 (-20.6)	Pb ₂ O ₂ OH ⁻	PbCO ₃
	482.913 (0.10)	Pb ₂ O ₂ Cl ⁻	—
	503.939 (11.6)	Pb ₂ O ₂ C ₂ O ₂ ⁻	PbCO ₃
	523.931 (15.4)	Pb ₂ O ₃ CO ₃ ⁻	PbCO ₃
	544.911 (14.6)	Pb ₂ O ₂ HSO ₄ ⁻	Sulfate from manufacturing process
	623.952 (33.7)	Pb ₃ ⁻	PbCO ₃
	639.939 (20.8)	Pb ₃ O ⁻	PbCO ₃
	655.933 (18.9)	Pb ₃ O ₂ ⁻	PbCO ₃
	671.933 (26.1)	Pb ₃ O ₃ ⁻	PbCO ₃
	687.932 (31.3)	Pb ₃ O ₃ O ⁻	PbCO ₃
	688.933 (22.3)	Pb ₃ O ₃ OH ⁻	PbCO ₃
	706.907 (31.8)	Pb ₃ O ₃ Cl ⁻	—
	727.926 (28.5)	Pb ₃ C ₂ O ₅ ⁻	PbCO ₃
	743.915 (19.5)	Pb ₃ C ₂ O ₆ ⁻	PbCO ₃
	744.917 (12.0)	Pb ₃ C ₂ O ₆ H ⁻	PbCO ₃
	831.920 (15.2)	Pb ₄ ⁻	PbCO ₃
	911.901 (21.5)	Pb ₄ O ₄ O ⁻	PbCO ₃
	912.902 (13.1)	Pb ₄ O ₄ OH ⁻	PbCO ₃
	1039.907 (22.8)	Pb ₅ ⁻	PbCO ₃
	1119.906 (42.2)	Pb ₅ O ₅ ⁻	PbCO ₃
	1135.879 (23.1)	Pb ₅ O ₆ ⁻	PbCO ₃
	1136.888 (23.8)	Pb ₅ O ₅ OH ⁻	PbCO ₃
	1247.875 (11.7)	Pb ₆ ⁻	PbCO ₃
	1343.865 (25.8)	Pb ₆ O ₆ ⁻	PbCO ₃
1359.860 (25.8)	Pb ₆ O ₆ O ⁻	PbCO ₃	
1360.853 (15.2)	Pb ₆ O ₆ OH ⁻	PbCO ₃	

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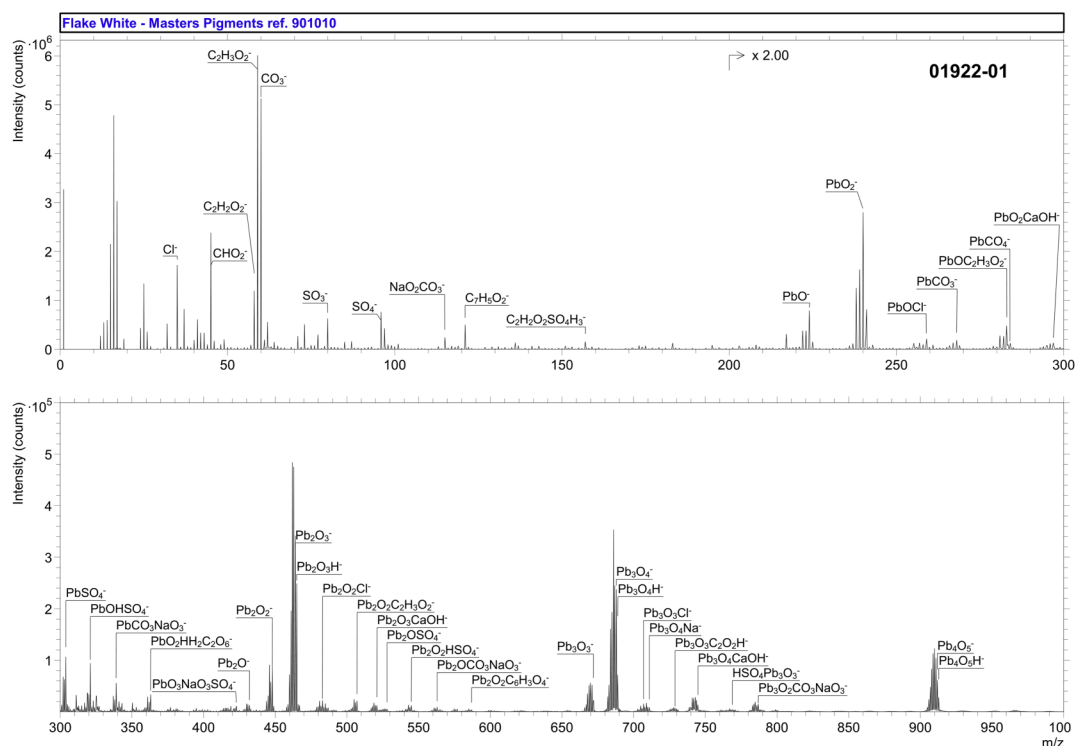
Accession #:	01921-01
■ Specimen:	Lead white—Kremer Pigmente ref. 46000
■ Technique:	SIMS
Instrument:	IONTOF, TOF SIMS IV
Analyzer Type:	Time-of-flight
Analyzer Mass Resolving Power:	3500 m/Δm
Mass Resolving Power Determined at:	25 Da
Species Used for Mass Calibration:	O_2^- , Cl^- , PbO_2H^- , $^{206}PbPbO_3H^-$, $^{206}PbPb_2O_4H^-$
Specimen Normal-to-Analyzer:	0°
Source-to-Analyzer Angle:	45°
Primary Beam Ion Gun:	LMIG
Primary Ion Species:	Bi_3^+
Primary Ion Dose:	4.37×10^{11} ion/cm ²
Primary Ion Pulse Width:	0.8–1.2 ns
Primary Ion Pulse Rate:	5 kHz
Net Beam Voltage:	25 000 eV
Pulsed Beam Current:	0.000 16 nA
DC Beam Current:	15.7 nA
Beam Diameter:	~2 μm
Beam Raster Size:	250 × 250 μm ²
Beam Incident Angle:	45°
Secondary Source Polarity:	Negative
Mass Range Minimum:	0 Da
Mass Range Maximum:	3545 Da
Spectrum Dead Time Corrected:	Yes
Total Spectral Acquisition Time:	250 s
Comment:	None

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SPECTRAL FEATURES TABLE

Spectrum ID #	Mass (Δm), Da	Species	Peak Assignment
01922-01	34.971 (33.8)	Cl ⁻	—
	44.998 (5.24)	CHO ₂ ⁻	Acetate from manufacturing process
	58.008 (33.3)	C ₂ H ₂ O ₂ ⁻	Acetate from manufacturing process
	59.016 (31.1)	C ₂ H ₃ O ₂ ⁻	Acetate from manufacturing process
	59.987 (27.9)	CO ₃ ⁻	2PbCO ₃ ·Pb(OH) ₂
	79.962 (53.3)	SO ₃ ⁻	Sulfate from manufacturing process
	95.954 (14.5)	SO ₄ ⁻	Sulfate from manufacturing process
	114.971 (53.4)	NaO ₂ CO ₃ ⁻	—
	121.036 (49.9)	C ₇ H ₅ O ₂ ⁻	Phthalate contamination
	156.986 (32.9)	C ₂ H ₂ O ₂ SO ₄ H ₃ ⁻	From manufacturing process
	223.970 (-10.1)	PbO ⁻	2PbCO ₃ ·Pb(OH) ₂
	239.965 (-8.51)	PbO ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	258.928 (-50.2)	PbOCl ⁻	—
	267.946 (-59.3)	PbCO ₃ ⁻	2PbCO ₃ ·Pb(OH) ₂
	282.981 (-17.1)	PbOC ₂ H ₃ O ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	283.955 (-6.83)	PbCO ₄ ⁻	2PbCO ₃ ·Pb(OH) ₂
	296.930 (9.08)	PbO ₂ CaOH ⁻	—
	303.934 (15.9)	PbSO ₄ ⁻	Sulfate from manufacturing process
	320.942 (31.3)	PbOH ₂ SO ₄ ⁻	Sulfate from manufacturing process
	338.943 (19.7)	PbCO ₃ NaO ₃ ⁻	—
	362.968 (21.0)	PbO ₂ H(HCO ₃) ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	422.891 (7.38)	PbO ₃ NaO ₃ SO ₄ ⁻	—
	431.947 (-3.27)	Pb ₂ O ⁻	2PbCO ₃ ·Pb(OH) ₂
	447.946 (5.95)	Pb ₂ O ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	463.947 (17.7)	Pb ₂ O ₃ ⁻	2PbCO ₃ ·Pb(OH) ₂
	464.949 (5.13)	Pb ₂ O ₃ H ⁻	2PbCO ₃ ·Pb(OH) ₂
	482.916 (7.54)	Pb ₂ O ₂ Cl ⁻	—
	506.970 (25.1)	Pb ₂ O ₂ C ₂ H ₃ O ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	520.902 (-3.07)	Pb ₂ O ₃ CaOH ⁻	—
	527.912 (21.9)	Pb ₂ OSO ₄ ⁻	Sulfate from manufacturing process
	544.898 (-9.24)	Pb ₂ O ₂ HSO ₄ ⁻	Sulfate from manufacturing process
	562.891 (-30.3)	Pb ₂ OCO ₃ NaO ₃ ⁻	—
	586.951 (32.7)	Pb ₂ O ₃ H(HCO ₃) ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	671.913 (-3.39)	Pb ₃ O ₃ ⁻	2PbCO ₃ ·Pb(OH) ₂
	687.912 (2.40)	Pb ₃ O ₄ ⁻	2PbCO ₃ ·Pb(OH) ₂
	688.918 (-0.38)	Pb ₃ O ₄ H ⁻	2PbCO ₃ ·Pb(OH) ₂
	706.887 (4.16)	Pb ₃ O ₃ Cl ⁻	—
	710.909 (13.2)	NaPb ₃ O ₄ ⁻	—
	730.937 (10.9)	Pb ₃ O ₃ C ₂ H ₃ O ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	744.880 (5.46)	Pb ₃ O ₄ CaOH ⁻	—
	768.888 (17.2)	HSO ₄ Pb ₃ O ₃ ⁻	—
	786.891 (13.9)	Pb ₃ O ₂ CO ₃ NaO ₃ ⁻	—
	911.888 (7.90)	Pb ₄ O ₅ ⁻	2PbCO ₃ ·Pb(OH) ₂
	912.895 (6.11)	Pb ₄ O ₅ H ⁻	2PbCO ₃ ·Pb(OH) ₂

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Accession #:

01922-01

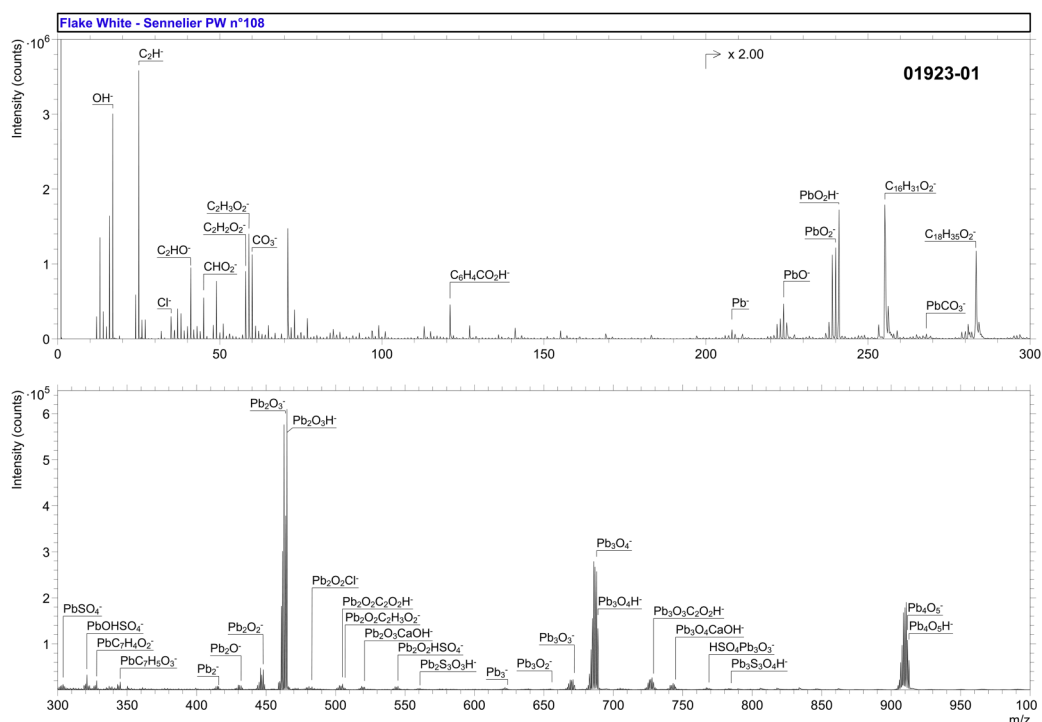
<p> Specimen: Technique: Instrument: Analyzer Type: Analyzer Mass Resolving Power: Mass Resolving Power Determined at: Species Used for Mass Calibration: Specimen Normal-to-Analyzer: Source-to-Analyzer Angle: Primary Beam Ion Gun: Primary Ion Species: Primary Ion Dose: Primary Ion Pulse Width: Primary Ion Pulse Rate: Net Beam Voltage: Pulsed Beam Current: DC Beam Current: Beam Diameter: Beam Raster Size: Beam Incident Angle: Secondary Source Polarity: Mass Range Minimum: Mass Range Maximum: Spectrum Dead Time Corrected: Total Spectral Acquisition Time: Comment: </p>	<p> Flake White 10 μm—Masters Pigments ref. 901010 SIMS IONTOF, TOF SIMS IV Time-of-flight 3600 m/Δm 25 Da O_2^-, Cl^-, PbO_2H^-, $^{206}\text{PbPbO}_3\text{H}^-$, $^{206}\text{PbPb}_2\text{O}_4\text{H}^-$ 0° 45° LMIG Bi_3^+ 4.37×10^{11} ion/cm^2 0.8–1.2 ns 8.3 kHz 25 000 eV 0.000 38 nA 1.9 nA ~2 μm $200 \times 200 \mu\text{m}^2$ 45° Negative 0 Da 1994 Da Yes 161 s None </p>
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SPECTRAL FEATURES TABLE

Spectrum ID #	Mass (Δm), Da	Species	Peak Assignment
01923-01	17.003 (-21.5)	OH ⁻	—
	34.970 (18.0)	Cl ⁻	—
	41.004 (21.1)	C ₂ HO ⁻	—
	44.998 (-9.10)	CHO ₂ ⁻	Acetate from manufacturing process
	58.008 (26.0)	C ₂ H ₂ O ₂ ⁻	Acetate from manufacturing process
	59.017 (48.7)	C ₂ H ₃ O ₂ ⁻	Acetate from manufacturing process
	59.986 (5.88)	CO ₃ ⁻	2PbCO ₃ ·Pb(OH) ₂
	121.038 (67.5)	C ₇ H ₅ O ₂ ⁻	Phthalate contamination
	207.974 (-13.8)	Pb ⁻	2PbCO ₃ ·Pb(OH) ₂
	223.968 (-19.8)	PbO ⁻	2PbCO ₃ ·Pb(OH) ₂
	239.966 (-6.10)	PbO ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	240.973 (-8.00)	PbO ₂ H ⁻	2PbCO ₃ ·Pb(OH) ₂
	255.238 (18.9)	C ₁₆ H ₃₁ O ₂ ⁻	Fatty acid from contamination
	267.945 (-64.6)	PbCO ₃ ⁻	2PbCO ₃ ·Pb(OH) ₂
	283.269 (15.3)	C ₁₈ H ₃₅ O ₂ ⁻	Fatty acid from contamination
	296.930 (-8.05)	PbO ₂ CaOH ⁻	—
	303.939 (33.6)	PbSO ₄ ⁻	Sulfate from manufacturing process
	320.942 (31.5)	PbOHSO ₄ ⁻	Sulfate from manufacturing process
	328.001 (34.0)	PbC ₇ H ₄ O ₂ ⁻	—
	345.019 (53.3)	PbC ₇ H ₅ O ₃ ⁻	—
	415.950 (-10.0)	Pb ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	431.945 (-9.12)	Pb ₂ O ⁻	2PbCO ₃ ·Pb(OH) ₂
	447.940 (-7.74)	Pb ₂ O ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	463.942 (9.16)	Pb ₂ O ₃ ⁻	2PbCO ₃ ·Pb(OH) ₂
	464.950 (6.75)	Pb ₂ O ₃ H ⁻	2PbCO ₃ ·Pb(OH) ₂
	482.914 (3.43)	Pb ₂ O ₂ Cl ⁻	—
	504.933 (-17.5)	Pb ₂ O ₂ C ₂ O ₂ H ⁻	2PbCO ₃ ·Pb(OH) ₂
	506.950 (-13.6)	Pb ₂ O ₂ C ₂ H ₃ O ₂ ⁻	—
	520.896 (-14.6)	Pb ₂ O ₃ CaOH ⁻	—
	544.887 (-30.1)	Pb ₂ O ₂ HSO ₄ ⁻	Sulfate from manufacturing process
	560.865 (4.97)	Pb ₂ S ₃ O ₃ H ⁻	Sulfate from manufacturing process
	623.930 (-0.47)	Pb ₃ ⁻	2PbCO ₃ ·Pb(OH) ₂
	639.916 (-14.5)	Pb ₃ O ⁻	2PbCO ₃ ·Pb(OH) ₂
	655.905 (-23.4)	Pb ₃ O ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	671.905 (-15.4)	Pb ₃ O ₃ ⁻	2PbCO ₃ ·Pb(OH) ₂
	687.911 (0.54)	Pb ₃ O ₄ ⁻	2PbCO ₃ ·Pb(OH) ₂
	688.915 (-4.55)	Pb ₃ O ₄ H ⁻	2PbCO ₃ ·Pb(OH) ₂
	706.876 (-11.8)	Pb ₃ O ₃ Cl ⁻	—
	728.898 (-20.4)	Pb ₃ O ₃ C ₂ O ₂ H ⁻	2PbCO ₃ ·Pb(OH) ₂
	744.875 (0.01)	Pb ₃ O ₄ CaOH ⁻	—
768.879 (5.91)	HSO ₄ Pb ₃ O ₃ ⁻	Sulfate from manufacturing process	
784.858 (30.0)	Pb ₃ S ₃ O ₄ H ⁻	Sulfate from manufacturing process	
911.881 (-1.02)	Pb ₄ O ₅ ⁻	2PbCO ₃ ·Pb(OH) ₂	
912.879 (-11.5)	Pb ₄ O ₅ H ⁻	2PbCO ₃ ·Pb(OH) ₂	

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Accession #:

01923-01

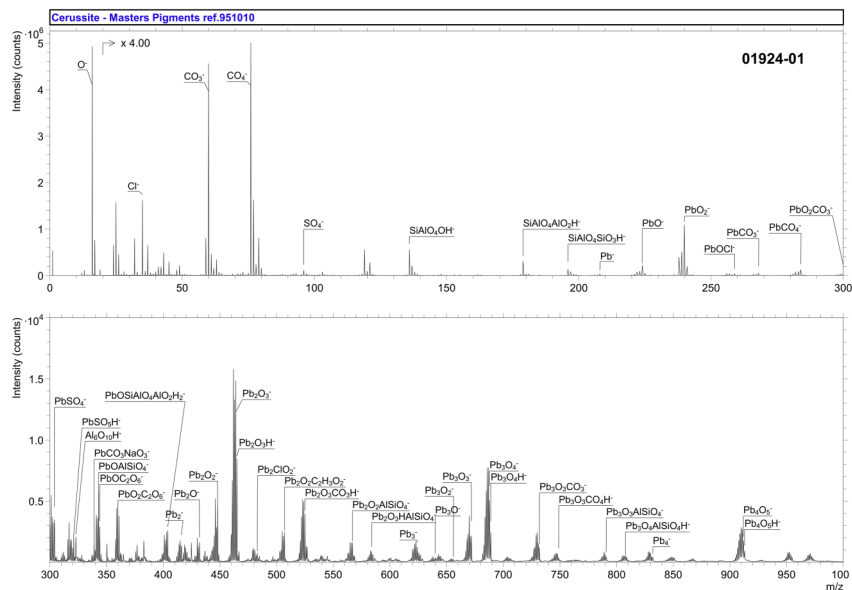
<p> ■ Specimen: ■ Technique: Instrument: Analyzer Type: Analyzer Mass Resolving Power: Mass Resolving Power Determined at: Species Used for Mass Calibration: Specimen Normal-to-Analyzer: Source-to-Analyzer Angle: Primary Beam Ion Gun: Primary Ion Species: Primary Ion Dose: Primary Ion Pulse Width: Primary Ion Pulse Rate: Net Beam Voltage: Pulsed Beam Current: DC Beam Current: Beam Diameter: Beam Raster Size: Beam Incident Angle: Secondary Source Polarity: Mass Range Minimum: Mass Range Maximum: Spectrum Dead Time Corrected: Total Spectral Acquisition Time: Comment: </p>	<p> Flake White—Sennelier PW no.108 SIMS IONTOF, TOF SIMS IV Time-of-flight 3100 m/Δm 25 Da O_2^-, Cl^-, PbO_2H^-, $^{206}PbPbO_3H^-$, $^{206}PbPb_2O_4H^-$ 0° 45° LMIG Bi_3^+ 4.37×10^{11} ion/cm² 0.8–1.2 ns 8.3 kHz 25 000 eV 0.000 38 nA 1.9 nA ~2 μm 200 × 200 μm² 45° Negative 0 Da 1994 Da Yes 161 s Contamination with a fatty material, probably from manufacturing process </p>
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SPECTRAL FEATURES TABLE

Spectrum ID #	Mass (Δm), Da	Species	Peak Assignment
01924-01	34.970 (9.2)	Cl ⁻	—
	95.955 (32.1)	SO ₄ ⁻	Sulfate from manufacturing process
	135.939 (-20.2)	SiAlO ₅ H ⁻	Aluminosilicate (possible filler)
	178.919 (6.84)	SiAl ₂ O ₆ H ⁻	Aluminosilicate (possible filler)
	195.913 (22.3)	Si ₂ AlO ₇ H ⁻	Aluminosilicate (possible filler)
	207.971 (-28.0)	Pb ⁻	PbCO ₃
	223.968 (-18.5)	PbO ⁻	PbCO ₃
	239.965 (-8.89)	PbO ₂ ⁻	PbCO ₃
	258.950 (34.1)	PbOCl ⁻	—
	267.955 (-29.8)	PbCO ₃ ⁻	PbCO ₃
	283.961 (15.6)	PbCO ₄ ⁻	PbCO ₃
	299.954 (8.88)	PbO ₂ CO ₃ ⁻	PbCO ₃
	303.945 (53.5)	PbSO ₄ ⁻	Sulfate from manufacturing process
	320.949 (54.4)	PbSO ₅ H ⁻	Sulfate from manufacturing process
	322.850 (10.1)	Al ₆ O ₁₀ H ⁻	Aluminosilicate (possible filler)
	338.937 (0.28)	PbCO ₃ NaO ₃ ⁻	—
	342.923 (36.9)	PbOAlSiO ₄ ⁻	Aluminosilicate (possible filler)
	343.941 (-1.17)	PbOC ₂ O ₆ ⁻	PbCO ₃
	359.936 (-0.71)	PbO ₂ C ₂ O ₆ ⁻	PbCO ₃
	403.910 (60.4)	PbAl ₂ SiO ₆ H ₂ ⁻	Aluminosilicate (possible filler)
	415.964 (23.7)	Pb ₂ ⁻	PbCO ₃
	431.939 (-23.3)	Pb ₂ O ⁻	PbCO ₃
	447.942 (-3.53)	Pb ₂ O ₂ ⁻	PbCO ₃
	463.935 (-8.36)	Pb ₂ O ₃ ⁻	PbCO ₃
	464.949 (5.76)	Pb ₂ O ₃ H ⁻	PbCO ₃
	482.917 (7.15)	Pb ₂ ClO ₂ ⁻	—
	506.949 (-16.7)	Pb ₂ O ₂ C ₂ H ₃ O ₂ ⁻	2PbCO ₃ ·Pb(OH) ₂
	524.948 (32.0)	Pb ₂ O ₃ CO ₃ H ⁻	PbCO ₃
	566.890 (14.1)	Pb ₂ O ₂ AlSiO ₄ ⁻	Aluminosilicate (possible filler)
	583.904 (34.0)	Pb ₂ O ₃ HAlSiO ₄ ⁻	Aluminosilicate (possible filler)
	623.940 (15.8)	Pb ₃ ⁻	PbCO ₃
	639.934 (13.0)	Pb ₃ O ⁻	PbCO ₃
	655.914 (-10.1)	Pb ₃ O ₂ ⁻	PbCO ₃
	671.928 (19.4)	Pb ₃ O ₃ ⁻	PbCO ₃
	687.929 (27.1)	Pb ₃ O ₄ ⁻	PbCO ₃
	688.944 (37.3)	Pb ₃ O ₄ H ⁻	PbCO ₃
	731.915 (21.0)	Pb ₃ O ₃ CO ₃ ⁻	PbCO ₃
	748.922 (25.8)	Pb ₃ O ₃ CO ₄ H ⁻	PbCO ₃
	790.883 (37.9)	Pb ₃ O ₃ AlSiO ₄ ⁻	Aluminosilicate (possible filler)
	807.885 (36.2)	Pb ₃ O ₄ AlSiO ₄ H ⁻	Aluminosilicate (possible filler)
	831.926 (23.1)	Pb ₄ ⁻	PbCO ₃
	911.896 (15.5)	Pb ₄ O ₅ ⁻	PbCO ₃
	912.922 (36.0)	Pb ₄ O ₅ H ⁻	PbCO ₃
	952.896 (12.1)	Pb ₄ C ₂ O ₆ H ⁻	PbCO ₃

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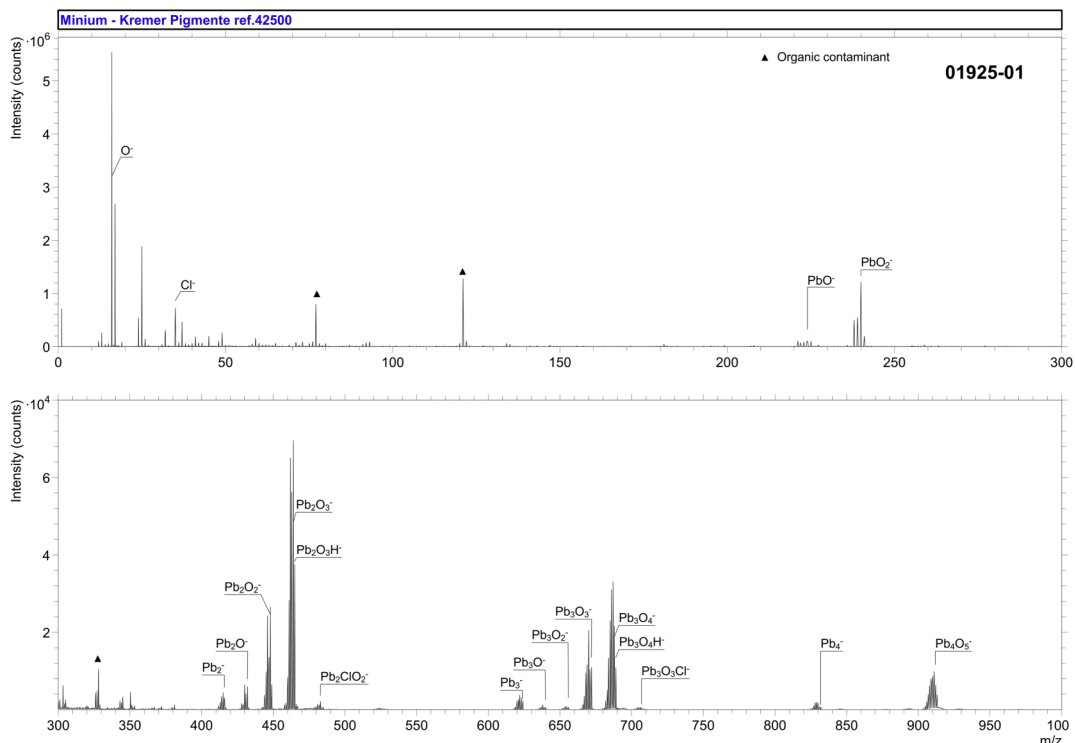
Accession #:	01924-01
Specimen:	Cerussite—Masters Pigments ref. 951010
Technique:	SIMS
Instrument:	IONTOF, TOF SIMS IV
Analyzer Type:	Time-of-flight
Analyzer Mass Resolving Power:	2200 m/Δm
Mass Resolving Power Determined at:	25 Da
Species Used for Mass Calibration:	O_2^- , Cl^- , PbO_2H^- , $^{206}PbPbO_3H^-$, $^{206}PbPb_2O_4H^-$
Specimen Normal-to-Analyzer:	0°
Source-to-Analyzer Angle:	45°
Primary Beam Ion Gun:	LMIG
Primary Ion Species:	Bi_3^+
Primary Ion Dose:	4.35×10^{11} ion/cm ²
Primary Ion Pulse Width:	0.8 - 1.2 ns
Primary Ion Pulse Rate:	5 kHz
Net Beam Voltage:	25 000 eV
Pulsed Beam Current:	0.000 23 nA
DC Beam Current:	7.2 nA
Beam Diameter:	~2 μm
Beam Raster Size:	250 × 250 μm ²
Beam Incident Angle:	45°
Secondary Source Polarity:	Negative
Mass Range Minimum:	0 Da
Mass Range Maximum:	3542 Da
Spectrum Dead Time Corrected:	Yes
Total Spectral Acquisition Time:	194 s
Comment:	The peak at <i>m/z</i> 59.98 can be attributed to CO_3^- along with a near isobaric interference from SiO_2^- , thanks to the ion images. The mass range attributed to CO_3^- exhibits the same spatial distribution as lead-containing ions, while the mass range attributed to SiO_2^- exhibits the same spatial distribution as the other aluminosilicate ions

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SPECTRAL FEATURES TABLE

Spectrum ID #	Mass (Δm), Da	Species	Peak Assignment
01925-01	15.996 (11.9)	O ⁻	(saturated)
	34.970 (24.1)	Cl ⁻	—
	207.971 (-29.5)	Pb ⁻	Pb ₃ O ₄
	223.971 (-6.51)	PbO ⁻	Pb ₃ O ₄
	239.966 (-6.29)	PbO ₂ ⁻	Pb ₃ O ₄
	415.954 (0.12)	Pb ₂ ⁻	Pb ₃ O ₄
	431.936 (-28.9)	Pb ₂ O ⁻	Pb ₃ O ₄
	447.938 (-13.6)	Pb ₂ O ₂ ⁻	Pb ₃ O ₄
	463.931 (-17.1)	Pb ₂ O ₃ ⁻	Pb ₃ O ₄
	464.938 (-17.5)	Pb ₂ O ₃ H ⁻	Pb ₃ O ₄
	482.907 (-10.8)	Pb ₂ ClO ₂ ⁻	—
	623.947 (26.1)	Pb ₃ ⁻	Pb ₃ O ₄
	639.939 (21.3)	Pb ₃ O ⁻	Pb ₃ O ₄
	655.932 (17.3)	Pb ₃ O ₂ ⁻	Pb ₃ O ₄
	671.930 (22.0)	Pb ₃ O ₃ ⁻	Pb ₃ O ₄
	687.929 (27.6)	Pb ₃ O ₄ ⁻	Pb ₃ O ₄
	688.937 (27.3)	Pb ₃ O ₄ H ⁻	Pb ₃ O ₄
	706.904 (28.7)	Pb ₃ O ₃ Cl ⁻	—
	831.916 (10.8)	Pb ₄ ⁻	Pb ₃ O ₄
	911.908 (28.9)	Pb ₄ O ₅ ⁻	Pb ₃ O ₄
	1039.903 (18.2)	Pb ₅ ⁻	Pb ₃ O ₄
	1119.886 (24.9)	Pb ₅ O ₅ ⁻	Pb ₃ O ₄
	1135.888 (30.9)	Pb ₅ O ₆ ⁻	Pb ₃ O ₄

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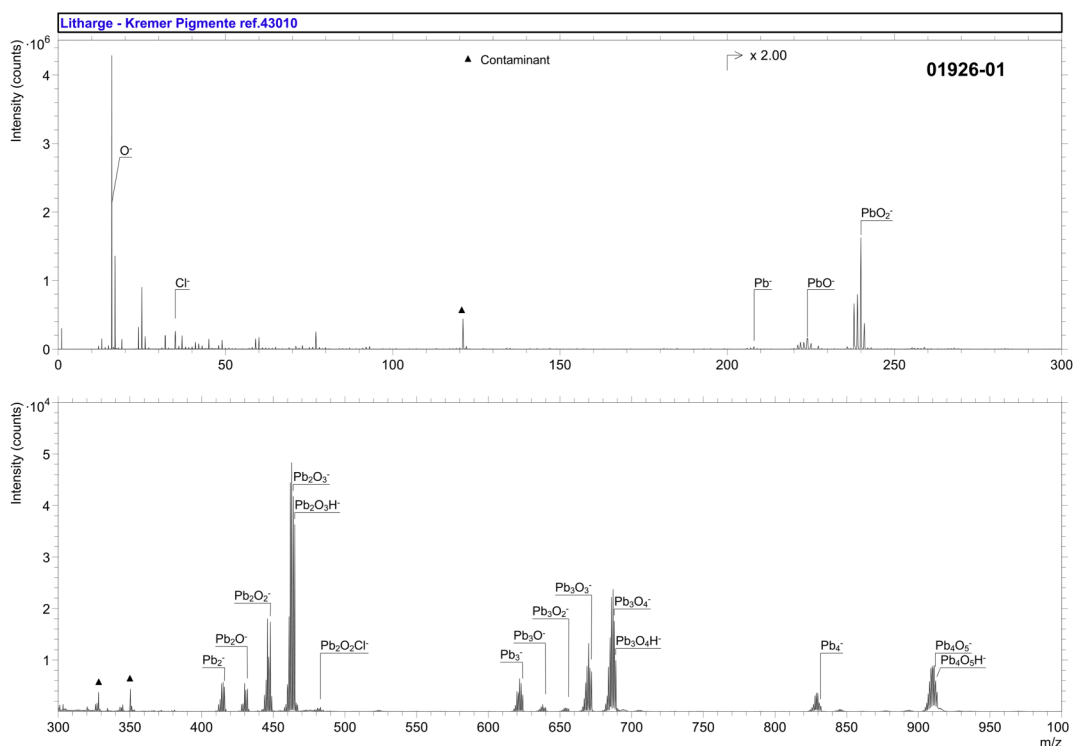


Accession #:	01925-01
■ Specimen:	Minium—Kremer Pigmente ref.42500
■ Technique:	SIMS
Instrument:	IONTOF, TOF SIMS IV
Analyzer Type:	Time-of-flight
Analyzer Mass Resolving Power:	4000 m/Δm
Mass Resolving Power Determined at:	25 Da
Species Used for Mass Calibration:	O ₂ ⁻ , Cl ⁻ , PbO ₂ H ⁻ , ²⁰⁶ PbPbO ₃ H ⁻ , ²⁰⁶ PbPb ₂ O ₄ H ⁻
Specimen Normal-to-Analyzer:	0°
Source-to-Analyzer Angle:	45°
Primary Beam Ion Gun:	LMIG
Primary Ion Species:	Bi ₃ ⁺
Primary Ion Dose:	4.35 × 10 ¹¹ ion/cm ²
Primary Ion Pulse Width:	0.8–1.2 ns
Primary Ion Pulse Rate:	5 kHz
Net Beam Voltage:	25 000 eV
Pulsed Beam Current:	0.000 23 nA
DC Beam Current:	7.2 nA
Beam Diameter:	~2 μm
Beam Raster Size:	250 × 250 μm ²
Beam Incident Angle:	45°
Secondary Source Polarity:	Negative
Mass Range Minimum:	0 Da
Mass Range Maximum:	3542 Da
Spectrum Dead Time Corrected:	Yes
Total Spectral Acquisition Time:	194 s
Comment:	Specific ion Pb ₄ O ₄ ⁻ is barely detected in minium

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SPECTRAL FEATURES TABLE

Spectrum ID #	Mass (Δm), Da	Species	Peak Assignment
01926-01	34.971 (31.9)	Cl ⁻	—
	207.970 (-34.3)	Pb ⁻	PbO
	223.966 (-28.2)	PbO ⁻	PbO
	239.960 (-31.2)	PbO ₂ ⁻	PbO
	415.955 (3.48)	Pb ₂ ⁻	PbO
	431.932 (-39.3)	Pb ₂ O ⁻	PbO
	447.940 (-8.66)	Pb ₂ O ₂ ⁻	PbO
	463.932 (-13.3)	Pb ₂ O ₃ ⁻	PbO
	464.942 (-8.61)	Pb ₂ O ₃ H ⁻	PbO
	482.911 (-4.13)	Pb ₂ O ₂ Cl ⁻	—
	623.950 (30.5)	Pb ₃ ⁻	PbO
	639.931 (9.00)	Pb ₃ O ⁻	PbO
	655.932 (17.2)	Pb ₃ O ₂ ⁻	PbO
	671.922 (10.4)	Pb ₃ O ₃ ⁻	PbO
	687.925 (21.3)	Pb ₃ O ₄ ⁻	PbO
	688.937 (27.4)	Pb ₃ O ₄ H ⁻	PbO
	831.917 (12.1)	Pb ₄ ⁻	PbO
	911.903 (23.1)	Pb ₄ O ₅ ⁻	PbO
	912.910 (22.5)	Pb ₄ O ₅ H ⁻	PbO



Accession #:

01926-01

<p>■ Specimen: ■ Technique: Instrument: Analyzer Type: Analyzer Mass Resolving Power: Mass Resolving Power Determined at: Species Used for Mass Calibration: Specimen Normal-to-Analyzer: Source-to-Analyzer Angle: Primary Beam Ion Gun: Primary Ion Species: Primary Ion Dose: Primary Ion Pulse Width: Primary Ion Pulse Rate: Net Beam Voltage: Pulsed Beam Current: DC Beam Current: Beam Diameter: Beam Raster Size: Beam Incident Angle: Secondary Source Polarity: Mass Range Minimum: Mass Range Maximum: Spectrum Dead Time Corrected: Total Spectral Acquisition Time: Comment:</p>	<p>Litharge—Kremer Pigmente ref. 43010 SIMS IONTOF, TOF SIMS IV Time-of-flight 2300 m/Δm 25 Da O_2^-, Cl^-, PbO_2H^-, $^{206}PbPbO_3H^-$, $^{206}PbPb_2O_4H^-$ 0° 45° LMIG Bi_3^+ 4.35×10^{11} ion/cm² 0.8–1.2 ns 5 kHz 25 000 eV 0.000 23 nA 7.2 nA ~2 μm 250 × 250 μm² 45° Negative 0 Da 3542 Da Yes 194 s Specific ion $Pb_4O_4^-$ is barely detected in litharge</p>
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